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Graphical Abstract

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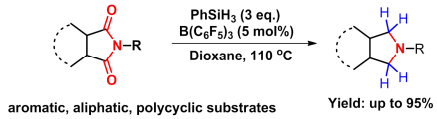

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ABSTRACT

$B(C_6F_5)_3$-catalyzed hydrosilylation of cyclic imides afforded an efficient synthetic method of pyrrolidines. In the presence of 5 mol% $B(C_6F_5)_3$, various aromatic, aliphatic and polycyclic imides were smoothly reduced by $PhSiH_3$ to generate the corresponding pyrrolidines in high yields. The reaction profiles monitored by ^1H NMR spectroscopy disclosed the reduction process of cyclic imides and the effect of difference structure of the hydrosilanes on the hydrosilylation .

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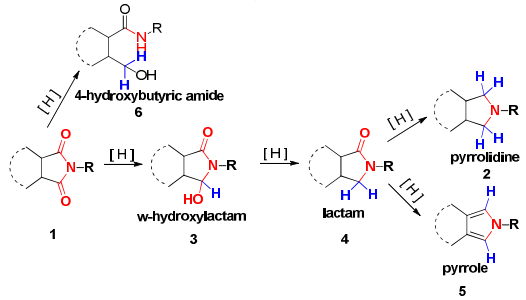
1. Introduction

The reduction of carbonyl compounds is one of the most fundamental and widely employed transformations in synthetic organic chemistry.[1] Hydrosilanes are air- and moisture-stable hydride sources and can be readily activated under mild conditions. It is attractive to develop a catalytic reduction system of the carbonyl compounds with hydrosilanes which enable the fine-tuning of activity to improve chemo- and regioselectivity.[2] Various catalysts had been explored for the chemo-, regio-, and/or stereoselective hydrosilylation of aldehydes, ketones, esters, amides, carboxylic acid derivatives and nitriles.[2] Among these catalysts, boron based catalytic systems are important for the hydrosilylations.[3] For example, Tris(pentafluorophenyl)borane ($B(C_6F_5)_3$) shows high effective for the hydrosilylation of alcohols,[4] olefins,[5] imines,[6] ethers,[7] amides,[8] carboxylic acids and their derivatives.[9] Moreover, the mechanisms of $B(C_6F_5)_3$ catalyzed hydrosilylations were different from typical Lewis acid catalyzed hydrosilylations in which $B(C_6F_5)_3$ activated hydrosilanes by an unusual η^1 coordination rather than carbonyl groups.[10] However, there is no report about boron catalyzed the reduction of cyclic imides with hydrosilanes.

The reduction of available cyclic imides gives several nitrogen containing compounds such as ω-hydroxylactams,[11] lactams,[12] substituted pyrroles[13] or pyrrolidines[14] which are versatile building blocks for organic synthesis (Scheme 1).[13d,15] The classical reduction process employing metal hydrides always resulted in the formation of byproducts and suffered from practical issues due to the moisture sensitive nature of metal hydrides.[1,16] Therefore, the reduction of the cyclic imides with hydrosilanes is an appealing method. $Zn(OAc)_2$ catalyzed reduction of cyclic imides with polymethylhydrosiloxane (PMHS) provided effectively ω-hydroxylactams.[17] Lewis base catalyzed hydrosilylations of cyclic imides can afford selectively lactams or ω-hydroxylactams.[18] Pyrrolidines as the important reduction products of cyclic imides are present in numerous natural products, biomacromolecules and pharmaceuticals with diverse biological activities such as moxifloxacin and mitiglinide calcium.[19] There are only two literature examples about the hydrosilylation of cyclic imides to pyrrolidines. Ito and coworkers gave one example of Rh-catalyzed hydrosiylation of *N*-benzyl succinimide with Ph_2SiH_2 to *N*-benzylpyrrolidine in 70% yield.[20] Beller and coworkers reported a sequential reduction of *N*-benzylphthalimide to 2-benzylisoindoline by *n*-Bu_4NF/PMHS and $Fe_3(CO)_{12}$/PMHS in moderate yield.[18b] Therefore, it is attractive to develop a convenient and effective method for the reduction of cyclic imides to pyrrolidines. Due to boron catalytic systems allowing for deoxygenation of the amide functionality[8], we envision that boron based catalytic systems will display high chemoselectivity for the reduction of the cyclic imides to pyrrolidines. Herein, we report an efficient reduction of cyclic imides to the corresponding pyrrolidines by a convenient tri(penta-flurophenyl)borane catalyzed hydrosilylation.

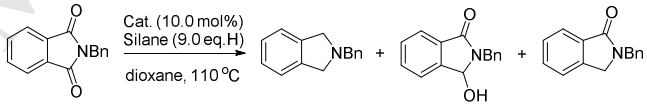


Scheme 1. The reduction of cyclic imides

2. Results and Discussion

N-benzyl phthalimide (**1a**) was chosen as the model substrate to optimize boron catalyzed reduction of cyclic imides with hydrosilanes. $BF_3{\cdot}Et_2O$ was initially examined in the reduction which was efficient for the reduction of aldehydes and ketones with hydrosilanes.[21] Nevertheless, no reduction occurred (Table 1, entry 1). The catalyst was changed to $B(C_6F_5)_3$ that was commercially available Lewis acid of comparable strength to BF_3 but without the problems associated with reactive B-F bonds.[22] Gratifyingly, the pyrrolidine (**2a**) was obtained as the sole product in 95% yield (Table 1, entry 2). Replacing $B(C_6F_5)_3$ with the moderately Lewis acidic triphenylborane (BPh_3) which was found to catalyze the selective hydrosilylation of CO_2 and tertiary amides,[23] no desired product (**2a**) was generated (Table 1, entry 3). In the cases of organo-borane reagents, benzo[b]thiophen-2-ylboronic acid and $PhB(OH)_2$, also any reduced products did not observed (Table 1, entries 4 and 5). Other metal or Lewis acid catalyzed the reduction could not afford selectively the pyrrolidine (**2a**) (Table 1, entries 6 - 8). Consequently, $B(C_6F_5)_3$ was an efficient catalyst for the hydrosilylation of the imides to the pyrrolidine (**2a**). When $B(C_6F_5)_3$ was used as the catalyst, less active silanes like Ph_2SiH_2 or PHMS led to the desired pyrrolidine (**2a**) albeit in lower yield of 71% and 76%, respectively (Table 1, entries 9 and 11). The high yield of **2a** was also obtained as TMDS (tetramethyldisiloxane) was used (Table 1, entry 10), however 5% of isoindole (**5a**) as the byproduct was observed in the reduction products. Reduced dosage of $PhSiH_3$ resulted in a decreased yield of **2a**, wherein the mono-reduction product (**4a**) was detected in 10% yield (Table 1, entry 12).

Table 1. The effect of catalysts and hydrosilanes on the reduction of cyclic imides [a]



1a 2a 3a 4a

Entry	Cat.	Silane	Conv. (%)	Yield (%)		
				2a	**3a**	**4a**
1	$BF_3{\cdot}Et_2O$	$PhSiH_3$	0	--	--	--
2	$B(C_6F_5)_3$	$PhSiH_3$	100	95	0	0
3	BPh_3	$PhSiH_3$	0	0	0	0
4	$PhB(OH)_2$	$PhSiH_3$	0	0	0	0
5	benzo[b]thiophen-2-yl boronic acid	$PhSiH_3$	0	0	0	0
6	$Zn(OAc)_2{\cdot}2H_2O$	$PhSiH_3$	100	0	88	13
7	$Ti(OiPr)_4$	$PhSiH_3$	25	0	18	0
8	$In(OAc)_2$	$PhSiH_3$	20	0	15	0
9	$B(C_6F_5)_3$	Ph_2SiH_2	100	71	0	0
10[b]	$B(C_6F_5)_3$	TMDS	100	92	0	0
11	$B(C_6F_5)_3$	PMHS	94	76	0	0
12[c]	$B(C_6F_5)_3$	$PhSiH_3$	85	75	0	10

[a] Reaction conditions: *N*-benzyl phthalimide (**1a**, 1.0 mmol), Silane (9.0 equiv. H), catalyst (10.0 mol%), dioxane (2.0 mL), 110 °C, 16 h; conversion and yields were determined by ^1H NMR analysis (internal standard: 4,4'-di-tert-butyl-1,1'-biphenyl).

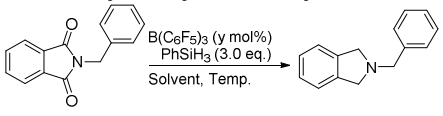
[b] 5% of isoindole (**5a**) as the byproduct was observed.

[c] The amount of $PhSiH_3$ is 2.0 eq. (2.0 mmol), 10% of **4a** as the byproduct was observed.

Next, the influence of solvents (Table 2, entris 1-5), the catalyst loading (Table 2, entries 6 and 7) and temperature (Table 2, entries 8 and 9) on the $B(C_6F_5)_3$ catalyzed hydrosilylation of

the imides with $PhSiH_3$ were explored. Highest activity was found in dioxane and toluene (Table 2, entries 1 and 2). Incomplete-reduced product was observed when $CHCl_3$ was used as the solvent (Table 2, entry 4). Quantitative yields of **2a** were generated with a lower catalyst loading of 5 mol % (Table 2, entry 6). Lowering the reaction temperature caused a decrease in yield (Table 2, entries 8 and 9), so the reduction was performed well at 110 °C.

Table 2. The effect of solvent, catalyst loading and temperature on the hydrosilylation of cyclic imides [a]



1a **2a**

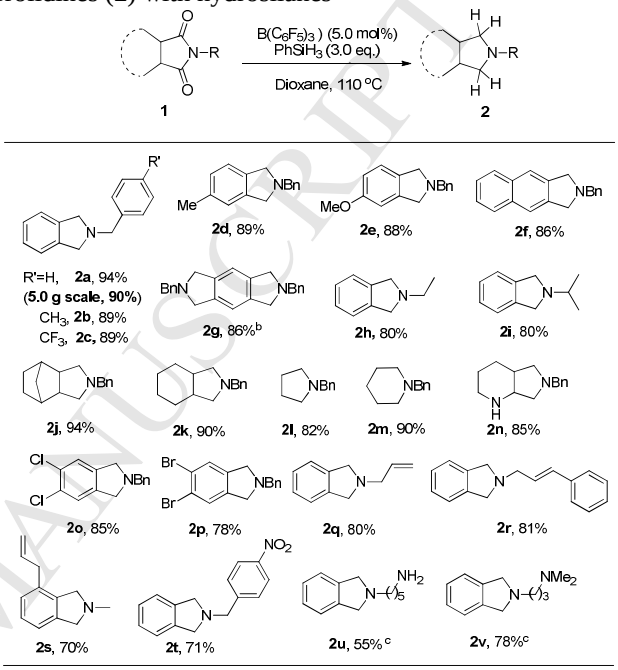
Entry	Solvent	$B(C_6F_5)_3$ (y mol%)	Temp. (°C)	Conv. (%)	Yield (%) **2a**
1	Dioxane	10	110	100	95
2	Toluene	10	110	100	95
3	$CH_3OCH_2CH_2OCH_3$	10	85	54	52
4[b]	$CHCl_3$	10	65	90	76
5	$ClCH_2CH_2Cl$	10	85	58	58
6	Dioxane	5.0	110	100	95
7	Dioxane	2.5	110	100	92
8	Dioxane	5.0	80	90	89
9	Dioxane	5.0	25	20	20

[a] Reaction condition: *N*-phenmethyl phthalimide (**1a**, 1 mmol), $PhSiH_3$ (3.0 eq., 3.0 mmol), $B(C_6F_5)_3$ (y mol%), solvent (2 mL), 110 °C, 16 h; Yields were determined by ^1H NMR analysis (internal standard: 4,4'-di-tert-butyl-1,1'-biphenyl).

[b] 8% of the lactam (**4a**) was observed in this condition.

Under the above optimized conditions, the scope of the $B(C_6F_5)_3$-catalyzed reduction of cyclic imides (**1**) to pyrrolidines (**2**) with $PhSiH_3$ was probed. As shown in Table 3, $B(C_6F_5)_3$-catalyzed hydrosilylation afforded effectively a range of the cyclic amines (**2**). The reduction system displayed the high efficiency for the reduction of various aryl imides (**1**) to the corresponding isoindolines (**2a-2i**). The effect of the electronic nature of the substituents at aromatic rings was not obvious on the reductions. The methoxyl substituted phthalimide (**1e**) and the naphthalinic imide (**1f**) were reacted in excellent yields. Notably, full reduction of pyromellitic diimide (**1g**) to the corresponding product (**2g**) was achieved in excellent yield without increasing the catalyst loading. The reduction of *N*-alkyl phthalimides (**1h and 1i**) also afforded the isoindolines (**2h and 2i**) in good yield. Moreover, the reduction of aliphatic imides (**1j-1n**) was executed smoothly with this reduction system. The polycyclic substrate (**1j**) was reduced well in 94% yield, and the less sterically hindered *N*-benzyl succinimide (**1l**) was reduced in 82% isolated yield. The catalyst system was also applicable for the reduction of six-membered cyclic imide (**1m**), in which 1-benzylpiperidine (**2m**) was obtained in 90% yield. The nitrogenous heterocyclic substrate (**1n**) was reacted efficiently to afford *N*-benzyl-2,8-diazabicyclo[4.3.0]-nonane (**2n**) in 85% yield which is an important building block for the synthesis of moxifloxacin.[19e] The reduction system also tolerated aryl halides, alkenes and nitro groups. *N*-benzyl 5,6-dibromo phthalimide (**1p**) and *N*-benzyl 5,6-dichlorophthalimide (**1o**) gave the isoindolines (**2p** and **2o**) in 78% and 85% yield, respectively. For *N*-allylic and cinnamyl imides (**1q**, **1r** and **1s**), the carbonyl groups were reduced selectively to afford the corresponding isoindolines with the alkene unaffected.[5] However, the steric hindrance of allyl group at C4 position of cyclic imides resulted the yield of **2s** to drop slightly. The reduction of *N*-(4-nitrobenzyl) phthalimide (**1t**) afforded the corresponding isoindoline (**2t**) in 71% yield. For the cyclic imides (**1u**) with cyano group, the reduction of both the imide and cyano group was observed, and the amino substituent isoindolines (**2u**) was obtained in moderate yield. Similarly, the reduction of the cyclic imide with amide group (**1v**) generated the diamine (**2v**) in good yields. Regrettably, when ethyl 4-phthalimido-butyric ester (**1w**) was reduced under the catalytic system, only little of the object product was observed and most of the imide (**1w**) was recovered. In addition, the scalability of this procedure was exemplified by the reduction of 5 gram of *N*-benzyl phthalimide (**1a**) with $PhSiH_3$ which afforded a 90% isolated yield of **2a**.

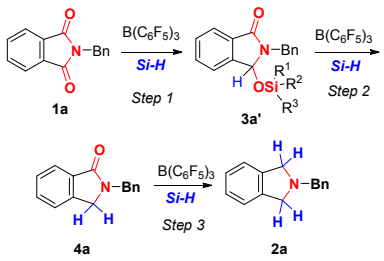
Table 3. $B(C_6F_5)_3$-catalyzed reduction of cyclic imides (**1**) to pyrrolidines (**2**) with hydrosilanes [a]



[a] Reaction condition: cyclic imide (**1**, 1.0 mmol), $PhSiH_3$ (3.0 mmol), $B(C_6F_5)_3$ (5.0 mol%), dioxane (2.0 ml), 110 °C, 16 h, isolated yield.

[b] $PhSiH_3$ (6.0 eq., 6.0 mmol).

[c] $PhSiH_3$ (4.0 eq., 4.0 mmol), $B(C_6F_5)_3$ (15.0 mol%), 130 °C, 48 h.

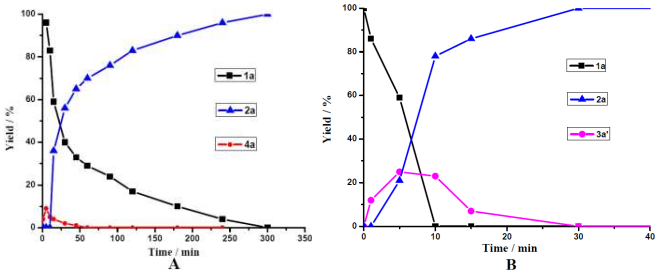


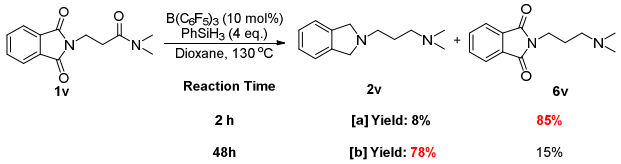
Scheme 2. The possible reduction process of cyclic imides catalyzed by $B(C_6F_5)_3$

The reduction of cyclic imides to pyrrolidines may go through several intermediates as shown in Scheme 2. To investigate the reaction pathway of $B(C_6F_5)_3$-catalyzed hydrosilylation of cyclic imides, the reaction profile for the reduction of **1a** with the hydrosilane was monitored by ^1H NMR spectroscopy (Figure 1) (^1H NMR spectra copies shown in the Supporting Information, Figure S1). In $PhSiH_3/B(C_6F_5)_3$ reduction system (**A**), only the lactam (**4a**) was observed in a low concentration at the initial stage of the reaction, and it disappeared after 60 minutes, while the nearly complete conversion of **1a** to the pyrrolidine (**2a**) is achieved after 5.0 h. These results suggested that in the reduction system the conversion of the intermediate (**4a**) was faster than that of the imide (**1a**). Furthermore, the hypothesis can be demonstrated by the intramolecular competitive reduction of the

amide group and the imide group (Scheme 3). When the cyclic imide with amide group (**1t**) was reacted in $PhSiH_3/B(C_6F_5)_3$ reduction system, the preferential reduction of the amide group was observed, and the compound (**6t**) was generated in 85% yield after 2 hrs. Then the yield of full reduced product (**2t**) increased gradually after the catalyst and the hydrosilane were added supplementally. However, in $TMDS/B(C_6F_5)_3$ reduction system (**B,** Figure 1), ω-hydroxy lactam derivative (**3a'**) was the only observed intermediate during the reduction, and the build-up and decay of **3a'** is evident. This phenomenon indicated that the conversion of **3a**' might be slower than that of imide (**1a**) and the intermediates (**4a**).

Figure 1. Reaction profile of $B(C_6F_5)_3$ catalyzed reduction of **1a** with $PhSiH_3$ and TMDS (A: $PhSiH_3$ reduction system; B: TMDS reduction system).





Scheme 3. $B(C_6F_5)_3$-catalyzed reduction of the cyclic imide (**1t**) with an amide group with $PhSiH_3$

[a] Reaction conditions: **1t** (1.0 mmol), $PhSiH_3$ (4.0 mmol), $B(C_6F_5)_3$ (10.0 mol%), dioxane (2.0 mL), 130 °C; yields were determined by ^1H NMR analysis (internal standard: 4,4'-di-tert-butyl-1,1'-biphenyl).

[b]After 5 hr, $B(C_6F_5)_3$ (5.0 mol%) and $PhSiH_3$ (2.0 mmol) were added again.

These results demonstrated the reduction of cyclic imides to pyrrolidines processed *via* three steps. Firstly, the hydrosilane was activated by $B(C_6F_5)_3$ and then added to the carbonyl group of **1a** to generate ω-hydroxy lactam derivative (**3a'**). Further, **3a'** was reduced to lactam (**4a**). Finally, $B(C_6F_5)_3$ catalyzed the reduction of **4a** with the hydrosilane to pyrrolidine (**2a**). Moreover, the difference structure of the hydrosilanes may affect the reaction rate of the three steps and result that the different intermediates was observed in the two reduction systems. The synergetic effect[24] of $PhSiH_3$, poly-hydrogen atoms attached to the same silicon atom, may result in prompt transformation of the possible intermediate (**3a'**) to the lactam (**4a**), so **3a'** can not be observed in the $PhSiH_3/B(C_6F_5)_3$ reduction reaction.

3. Conclusions

In summary, we have developed a convenient and efficient reduction method of cyclic imides to pyrrolidines via $B(C_6F_5)_3$-catalyzed hydrosilylations. Various cyclic imides, including aromatic, aliphatic, polycyclic substrates, were effectively reduced to the corresponding cyclic amines in high yield with $PhSiH_3/B(C_6F_5)_3$ reduction system. This catalytic protocol showed good tolerance for alkenes, halogens, nitro groups and heterocyclic compounds. The reaction profiles monitored by ^1H NMR spectroscopy displayed $B(C_6F_5)_3$-catalyzed reduction process of cyclic imides and the effect of difference structure of the hydrosilanes on the hydrosilylation.

4. Experimental section

4.1. General information

Unless otherwise noted, all reactions were carried out under an atmosphere of nitrogen using standard Schlenk techniques. Materials were purchased from commercial suppliers and used without further purification. Anhydrous dioxane was freshly distilled from Sodium. ^1H NMR and ^{13}C NMR spectra were recorded on a 400 MHz spectrometer. The chemical shifts for ^1H NMR were recorded in ppm downfield from tetramethylsilane (TMS) with the solvent resonance as the internal standard. The chemical shifts for ^{13}C NMR were recorded in ppm downfield using the central peak of deuterochloroform (77.16 ppm) as the internal standard. Coupling constants (*J*) are reported in Hz and refer to apparent peak multiplications. HRMS were obtained on an ESI-TOF mass spectrometer. Flash column chromatography was performed on silica gel (300-400 mesh).

*4.2. General experimental procedures of tri(pentaflurophenyl)-borane-catalyzed reduction of cyclicimides (**1**)*

To the mixture of $B(C_6F_5)_3$ (5.0 mol%) and cyclic imides (1.0 mmol) in dioxane, was added $PhSiH_3$ (3.0 mmol) slowly under an atmosphere of nitrogen. The reaction mixture was stirred and refluxed at 110 °C under an atmosphere of nitrogen. After the imide was consumed completely (detected by TLC) the mixture was added with aqueous ammonia (15 mL) and extracted with CH_2Cl_2 (10 mL×3). The combined organic phase was dried over Na_2SO_4, after removing the solvent under vacuum, the residue was purified by column chromatography to give the product.

*4.2.1. 2-Benzylisoindoline (**2a**)*[18b]

Reddish brown solid, 196.7 mg, yield: 94%, Mp: 33.0 – 35.3 °C. ^1H NMR (400 MHz, $CDCl_3$): δ 7.44 – 7.42 (m, 2H), 7.38 – 7.34 (m, 2H), 7.31 – 7.26 (m, 1H), 7.18 (s, 4H), 3.94 (s, 4H), 3.92 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.3, 139.2, 128.9, 128.5, 127.2, 126.7, 122.4, 60.4, 59.0.

*4.2.2. 2-(4-Methylbenzyl)isoindoline (**2b**)*[25]

Reddish brown solid, 198.8 mg, yield: 89%, Mp: 33.0 – 35.3 °C. ^1H NMR (400 MHz, $CDCl_3$): δ 7.32 – 7.30 (m, 2H), 7.20 – 7.16 (m, 6H), 3.92 (s, 4H), 3.88 (s, 2H), 2.36 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.3, 136.7, 136.1, 129.1, 128.8, 126.7, 122.3, 60.0, 58.9, 21.2.

*4.2.3. 2-(4-(Trifluoromethyl)benzyl)isoindoline (**2c**)*

Brown solid, 246.8 mg, yield: 89%, Mp: 50.2 – 52.8 °C. ^1H NMR (400 MHz, $CDCl_3$): δ 7.62 – 7.60 (m, 2H), 7.55 – 7.53 (m, 2H), 7.20 (s, 4H), 3.97 (s, 2H), 3.94 (s, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.5, 140.1, 129.5 (q, J = 32.1 Hz), 129.0, 126.9, 125.5 (q, J = 3.7 Hz), 124.4 (q, J = 270.1 Hz), 122.5, 59.9, 59.1. HRMS-ESI (m/z): Calculated for $C_{16}H_{15}F_3N$ $(M + H)^+$:278.1157, Found: 278.1156.

*4.2.4. 2-Benzyl-5-methylisoindoline (**2d**)*

Brown solid, 196.7 mg, yield: 89%, Mp: 51.8 – 53.7 °C. ^1H NMR (400 MHz, $CDCl_3$): δ 7.43 – 7.42 (m, 2H), 7.38 – 7.34 (m, 2H), 7.31 – 7.26 (m, 1H), 7.08 – 7.06 (m, 1H), 7.00 – 6.99 (m, 2H), 3.91 (s, 2H), 3.90 (s, 4H), 2.33 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.4, 139.2, 137.3, 136.3, 128.8, 128.4, 127.4, 127.1, 123.0, 122.1, 60.4, 58.9, 58.8, 21.4. HRMS-ESI (m/z): Calculated for $C_{16}H_{18}N$ $(M + H)^+$:224.1439, Found: 224.1439.

*4.2.5. 2-Benzyl-5-methoxyisoindoline (**2e**)*

Reddish brown solid, 210.6 mg, yield: 88%, Mp: 64.8 – 66.8 °C. ^1H NMR (400 MHz, $CDCl_3$): δ 7.45 – 7.43 (m, 2H), 7.39 – 7.35 (m, 2H), 7.32 – 7.26 (m, 1H), 7.10 – 7.08 (m, 1H), 6.76 – 6.75 (m, 2H), 3.93 (s, 4H), 3.90 (s, 2H), 3.79 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.1, 141.7, 139.1, 132.3, 128.9, 128.5,

127.2, 123.0, 112.6, 108.2, 60.4, 59.2, 58.4, 55.5. HRMS-ESI (m/z): Calculated for $C_{16}H_{18}NO$ $(M + H)^+$: 240.1388, Found: 240.1389.

*4.2.6. 2-Benzyl-2,3-dihydro-1H-benzo[f]isoindole (**2f**)*

Yellow solid, 228.3 mg, yield: 88%, Mp: 138.2 – 140.3 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81 – 7.79 (m, 2H), 7.63 (s, 2H), 7.49 – 7.39 (m, 6H), 7.35 – 7.32 (m, 1H), 4.06 (s, 4H), 3.97 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.4, 139.1, 133.1, 129.0, 128.5, 127.8, 127.3, 125.4, 120.6, 60.6, 58.7. HRMS-ESI (m/z): Calculated for $C_{19}H_{18}N$ $(M + H)^+$: 260.1439, Found:260.1439.

*4.2.7. 2,6-Bibenzyl-1,2,3,5,6,7-hexahydropyrrolo[3,4-f]isoindole (**2g**)*

Brown solid, 295.6 mg, yield: 86%, Mp: 137.8 – 140.1 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41 – 7.39 (m, 4H), 7.36 – 7.32 (m, 4H), 7.29 – 7.27 (m, 2H), 6.97 (s, 2H), 3.89 (s, 4H), 3.87 (s, 8H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.2, 139.0, 128.9, 128.5, 127.2, 116.5, 60.5, 58.9. HRMS-ESI (m/z): Calculated for $C_{24}H_{25}N_2$ $(M + H)^+$: 341.2018, Found: 341.2016.

*4.2.8. 2-Ethylisoindoline (**2h**)[26]*

Reddish brown oil, 117.8 mg, yield: 80%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.22 – 7.18 (m, 4H), 3.93 (s, 4H), 2.78 (q, *J* = 7.2 Hz, 2H), 1.21 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.1, 126.8, 122.3, 58.9, 50.1, 14.1.

*4.2.9. 2-Isopropylisoindoline (**2i**)[14b]*

Brown oil, 129.3 mg, yield: 80%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.22 – 7.17 (m, 4H), 3.97 (s, 4H), 2.77 – 2.71 (m, 1H), 1.20 (d, *J* = 6.0 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.1, 126.7, 122.3, 57.2, 54.5, 21.9.

*4.2.10. 2-Benzyloctahydro-1H-4,7-methanoisoindole (**2j**)*

Yellow oil, 211.5 mg, yield: 93%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36 – 7.28 (m, 4H), 7.24 – 7.21 (m, 1H), 3.52 (s, 2H), 2.76 (d, *J* = 10.0 Hz, 2H), 2.35 – 2.34 (m, 2H), 2.11 (s, 2H), 2.02 – 1.98 (m, 2H), 1.77 – 1.75 (m, 2H), 1.41 – 1.33 (m, 2H), 1.26 – 1.24 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.5, 128.5, 128.2, 126.7, 60.7, 55.4, 44.1, 42.3, 41.4, 24.1. HRMS-ESI (m/z): Calculated for $C_{16}H_{22}N$ $(M + H)^+$: 228.1752, Found: 228.1751.

*4.2.11. 2-Benzyloctahydro-1H-isoindole (**2k**)*

Yellow oil, 193.8 mg, yield: 90%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38 – 7.32 (m, 4H), 7.29 – 7.24 (m, 1H), 3.76 (s, 2H), 2.83 – 2.79 (m, 2H), 2.57 – 2.53 (m, 2H), 2.21 – 2.16 (m, 2H), 1.60 – 1.46 (m, 6H), 1.38 – 1.32 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.9, 128.8, 128.3, 126.9, 61.4, 58.4, 37.3, 27.0, 23.0. HRMS-ESI (m/z): Calculated for $C_{15}H_{22}N$ $(M + H)^+$:216.1752, Found: 216.1751.

*4.2.12. 1-Benzylpyrrolidine(**2l**)*

Yellow oil, 193.8 mg, yield: 82%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 – 7.23 (m, 5H), 3.65 (s, 2H), 2.55 – 2.53 (m, 4H), 1.82 – 1.78 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.2, 129.0, 128.3, 127.0, 60.8, 54.2, 23.5. HRMS-ESI (m/z): Calculated for $C_{11}H_{16}N$ $(M + H)^+$:162.1283, Found: 162.1283.

*4.2.13. 1-Benzylpiperidine(**2m**)[27]*

Yellow oil, 157.6 mg, yield: 90%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.23 (m, 5H), 3.49 (s, 2H), 2.46 – 2.32 (m, 4H), 1.61 – 1.55 (m, 4H), 1.47 – 1.38 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.6, 129.4, 128.2, 127.0, 64.0, 54.6, 26.1, 24.5.

*4.2.14. 6-Benzyloctahydro-1H-pyrrolo[3,4-b]pyridine (**2n**)*

Yellow oil, 183.7 mg, yield: 85%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34 – 7.29 (m, 4H), 7.27 – 7.22 (m, 1H), 3.82 – 3.69 (m, 2H), 3.28 – 3.26 (m, 1H), 3.04 – 3.00 (m, 1H), 2.87 – 2.84 (m, 1H), 2.80 – 2.75 (m, 1H), 2.71 – 2.55 (m, 3H), 2.29 – 2.21 (m, 1H), 1.69 – 1.64 (m, 2H), 1.52 – 1.43 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.0, 128.7, 128.2, 126.9, 60.5, 59.8, 56.1, 55.0, 43.8, 36.2, 23.9, 21.4. HRMS-ESI (m/z): Calculated for $C_{14}H_{21}N_2$ $(M + H)^+$: 217.1705, Found: 217.1705.

*4.2.15. 2-Benzyl-5,6-dichloroisoindoline (**2o**)*

Yellow solid, 236.4 mg, yield: 85%, Mp: 59.8 – 61.9 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39 – 7.26 (m, 5H), 7.25 (s, 2H), 3.89 (s, 2H), 3.87 (s, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.6, 138.6, 130.5, 128.7, 128.5, 127.3, 124.3, 60.0, 58.2. HRMS-ESI (m/z): Calculated for $C_{15}H_{14}{}^{35}Cl_2N$ $(M + H)^+$: 278.0503, Found: 278.0502.

*4.2.16. 2-Benzyl-5,6-dibromoisoindoline (**2p**)*

Yellow solid, 285.1 mg, yield: 78%, Mp: 75.9 – 78.1 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42 – 7.29 (m, 7H), 3.88 (s, 2H), 3.85 (s, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.7, 138.6, 128.8, 128.6, 127.5, 127.4, 122.5, 60.0, 58.1. HRMS-ESI (m/z): Calculated for $C_{15}H_{14}{}^{79}Br_2N$ $(M + H)^+$: 365.9493, Found: 365.9491.

*4.2.17. 2-Allylisoindoline (**2q**)*

Brown oil, 128.0 mg, yield: 80%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.20 (s, 4H), 6.05 – 5.94 (m, 1H), 5.29 (dd, *J* = 17.2, 1.6 Hz, 1H), 5.18 (dd, *J* = 10.4, 1.6 Hz, 1H), 3.97 (s, 4H), 3.40 (d, *J* = 6.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.2, 136.0, 126.8, 122.4, 117.3, 59.0, 58.9. HRMS-ESI (m/z): Calculated for $C_{11}H_{14}N$ $(M + H)^+$: 160.1126, Found: 160.1127.

*4.2.18. 2-Cinnamylisoindoline (**2r**)*

Reddish brown oil, 190.5 mg, yield: 81% (with reaction system **B**). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42 – 7.40 (m, 2H), 7.34 – 7.30 (m, 2H), 7.24 – 7.22 (m, 1H), 7.20 (s, 4H), 6.63 (d, *J* = 16.0 Hz, 1H), 6.46 – 6.35 (m, 1H), 4.00 (s, 4H), 3.55 (dd, *J* = 6.4, 1.2 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.0, 137.1, 132.4, 128.7, 127.6, 127.3, 126.8, 126.5, 122.4, 58.9, 58.2. HRMS-ESI (m/z): Calculated for $C_{17}H_{18}N$ $(M + H)^+$:236.1439, Found: 236.1437.

*4.2.19. 4-allyl-2-methylisoindoline (**2s**)*

Reddish brown oil, 120.8 mg, yield: 70%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.21 – 7.18 (m, 1H), 7.09 – 7.04 (m, 2H), 5.92 – 5.84 (m, 1H), 5.08 – 5.00 (m, 2H), 4.13 (s, 2H), 4.09 (s, 2H), 3.31 (d, *J* = 6.5 Hz, 2H), 2.72 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 138.4, 137.7, 135.7, 134.4, 128.0, 127.9, 120.5, 116.4, 61.0, 59.6, 42.6, 37.9. HRMS-ESI (m/z): Calculated for $C_{12}H_{16}N$ $(M + H)^+$:174.1283, Found: 174.1288.

*4.2.20. 2-(4-nitrobenzyl)isoindoline (**2t**)*

Pale yellow solid, Mp: 63.5 – 65.8 °C. 180.1 mg, yield: 71%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (d, *J* = 8.4 Hz, 2H), 7.60 (d, *J* = 8.4 Hz, 2H), 7.21 (s, 4H), 4.02 (s, 2H), 3.97 (s, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.3, 147.2, 139.9, 129.3, 127.0, 123.8, 122.4, 59.6, 59.2. HRMS-ESI (m/z): Calculated for $C_{15}H_{15}N_2O_2$ $(M + H)^+$:255.1134, Found: 255.1135.

*4.2.21. 5-(Isoindolin-2-yl)pentan-1-amine (**2u**)*

Red oil, 133.2 mg, yield: 55%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.18 (s, 4H), 3.91 (s, 4H), 3.69 (br, 2H), 2.74 – 2.69 (m, 4H), 1.63 – 1.50 (m, 4H), 1.44 – 1.37 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.8, 126.8, 122.3, 59.0, 55.9, 40.4, 29.9, 28.4, 24.5. HRMS-ESI (m/z): Calculated for $C_{13}H_{21}N_2$ $(M + H)^+$: 205.1705, Found: 205.1708.

*4.2.22. 3-(Isoindolin-2-yl)-N,N-dimethylpropan-1-amine (**2v**)*

Yellow oil, 159.2 mg, yield: 78%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.17 (s, 4H), 3.90 (s, 4H), 2.74 (t, *J* = 7.2 Hz, 2H), 2.36 (t, *J* = 7.6 Hz, 2H), 2.23 (s, 6H), 1.79 – 1.72 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.1, 126.7, 122.3, 59.2, 57.8, 54.3, 45.6, 27.2 HRMS-ESI (m/z): Calculated for $C_{13}H_{21}N_2$ $(M + H)^+$: 205.1705, Found: 205.1701.

*4.2.23. 2-Benzyl-3-hydroxyisoindolin-1-one (**3a**)[17]*

White solid, Mp: 141.7 – 143.8 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 – 7.77 (m, 1H), 7.58 – 7.56 (m, 2H), 7.52 – 7.48 (m, 1H), 7.33 – 7.25 (m, 5H), 5.63 (d, *J*=11.6 Hz, 1H), 5.03 (d, *J*=14.8 Hz, 1H), 4.35 (d, *J*=14.8 Hz, 1H), 2.63 (d, *J*=11.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.4, 144.0, 136.6, 132.3, 131.0, 130.0, 128.7, 128.4, 127.6, 123.4, 123.2, 80.9, 42.4.

*4.2.24. 2-Benzylisoindolin-1-one (**4a**)*[18a]

Pale yellow solid, Mp: 83.3 –84.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 – 7.89 (m, 1H), 7.54 – 7.45 (m, 2H), 7.39 – 7.36 (m, 1H), 7.34 – 7.26 (m, 5H), 4.81 (s, 2H), 4.27 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.5, 141.3, 137.1, 132.7, 131.4, 128.8, 128.2, 128.1, 127.7, 123.9, 122.8, 49.5, 46.4.

*4.2.25. 2-Benzyl-2H-isoindole (**5a**)*[28]

White solid yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52 – 7.45 (m, 2H), 7.27 – 7.22 (m, 3H), 7.15 – 7.03 (m, 4H), 6.92 – 6.88 (m, 2H), 5.20 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.4, 128.8, 128.0, 127.3, 124.6, 120.9, 119.7, 111.3, 54.8.

*4.2.26. 2-(3-(Dimethylamino)propyl)isoindoline-1,3-dione (**6v**)*[29]

Yellow oil, 204.3 mg, yield: 88%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 – 7.83 (m, 2H), 7.72 – 7.70 (m, 2H), 3.74 (t, J = 6.8 Hz, 2H), 2.45 (t, J = 7.2 Hz, 2H), 2.28 (s, 6H), 1.93 – 1.86 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.3, 134.0, 132.1, 123.2, 56.3, 44.4, 36.1, 25.8.

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Supplementary Material

Supplementary data related to this article can be found at

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