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Effect of the ancillary ligand in *N*-heterocyclic carbene iridium(III) catalyzed *N*-alkylation of amines with alcohols

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ABSTRACT

A series of air-stable *N*-heterocyclic carbene (NHC) Ir(III) complexes (**Ir1**-**6**), bearing various combinations of chlorine, pyridine and NHC ligands, were assayed for the *N*-alkylation of amines with alcohols. It was found that **Ir3**, with two monodentate 1,3-bis-methyl-imidazolylidene (IMe) ligands, emerged as the most active complex. A large variety of amines and primary alcohols were efficiently converted into mono-*N*-alkylated amines in 53–96% yields. As a special highlight, for the challenging MeOH, selective *N*-monomethylation could be achieved using KOH as a base under an air atmosphere. Moreover, this catalytic system was successfully applied to the gram-scale synthesis of some valuable compounds.

1. Introduction

N-Alkylated amines are one of the most valuable types of compounds, having versatile applications in polymer materials, pharmaceutical and synthetic industries [1–3]. The conventional method for the preparation of *N*-alkylated amines involves a nucleophilic substitution reaction of amines with an alkylating agent, such as an alkyl halide. However, this procedure suffers from some draw-backs, such as overalkylation, using toxic alkylating agents, low atom economy and the generation of harmful by-products. Alternatively, the hydrogen auto-transfer (HA) or borrowing hydrogen (BH) strategy (Scheme 1a) is one of the most appealing synthetic approaches for the synthesis of selective *N*-alkylated amines [4–6]. Notably, this transformation is a green and sustainable reaction, using abundant alcohols as coupling reagents and producing water as the only by-product.

To date, various transition metal (TM) catalyst systems based on noble metals, such as Ru [7–10] and Ir [11–13], or non-noble metals, such as Co [14–16], Fe [17–20], Ni [21–23] and Mn [24–26], as well as TM-free catalyst systems [27–30] have been developed in these fields. Especially, Ir complexes with NHC ligands have emerged as one of the most effective and practical catalysts [31]. For instance, in 2012, Martín-Matute *et al.* developed the Cp*Ir(III) complex **A** (Scheme 1b), bearing a functional alcohol/alkoxide-tethered-NHC ligand, which exhibited highly catalytic activity for the *N*-alkylation of anilines with alcohols under base-free conditions [32]. In 2013, Andersson *et al.* developed the bidentate phosphine-NHC-Ir(I) complex **B** (Scheme 1b) and realized the *N*-alkylation of anilines with alcohols at room temperature [33]. In 2017, Royo *et al.* reported the amide-functionalized NHC-Cp*Ir(III) complex **C** (Scheme 1b), which exhibited high activity for the *N*-alkylation of amines with alcohols in aqueous media and under base-free conditions [34]. In 2019, Ke *et al.* demonstrated that NHC-Ir(III) complex **D** (Scheme 1b), with a 2-hydroxypyridine ligand, acted as a metal–ligand bifunctional catalyst for the *N*-alkylation of aromatic amines and poor nucleophilic sulfonamides with alcohols in aqueous media [11]. Additionally, NHC-Ir complexes have been developed for more challenging reactions, such as the *N*-alkylation of amines with methanol, the *N*-alkylation of amides with alcohols and the *N*-alkylation of aqueous ammonia with alcohols [12,35–38]. In 2017, Tu *et al.* reported the NHC-Ir coordination polymer **E** (Scheme 1b) for the selective *N*-methylation of anilines with methanol at 130 °C [36]. In 2018, Fujita *et al.* developed the NHC-Ir complex **F** (Scheme 1b), which exhibited high catalytic performance for the *N*-alkylation of aqueous ammonia with alcohols at 150 °C [12]. Despite these great efforts, high temperature (above 100 °C) or high catalyst loadings (above 2 mol%) are usually required to obtained a satisfactory yield. In addition, the effect of the NHC ligands on the activity of Ir compounds has not been studied systematically. Therefore, the development of NHC-Ir catalysts remains a major driving force in these fields.

Very recently, our group has developed a bis-NHC-Mn complex, which provided the first example of a room temperature non-noble metal homogeneous system that catalyzes the selective *N*-alkylation of anilines with alcohols [26]. Continuing in this field, we became interested in a systematic study of the effects of the NHC ligands of Cp*Ir(III) complexes on the reaction of the *N*-alkylation

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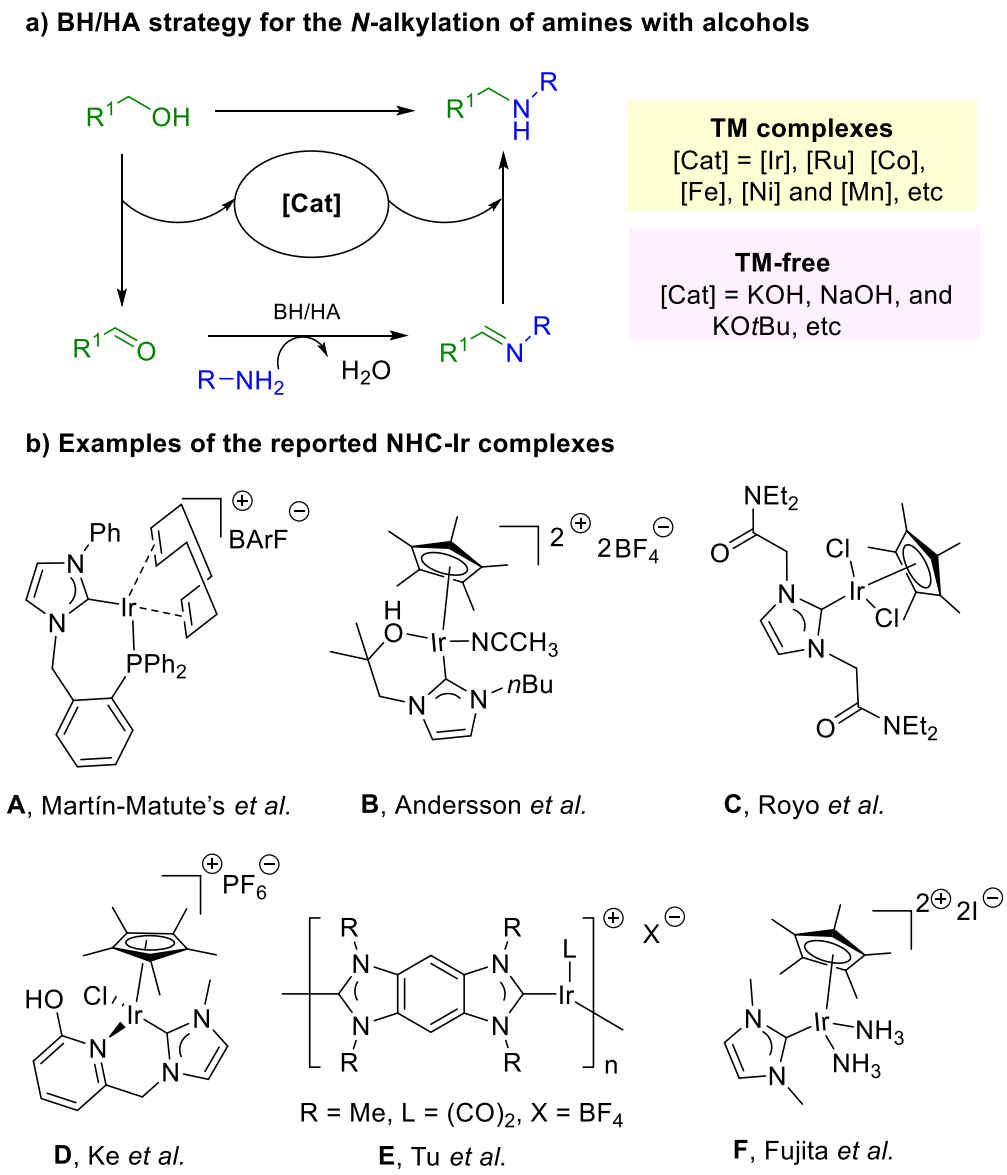


Scheme 1. Efficient NHC-Ir complexes for the *N*-alkylation of amines with alcohols.

with alcohols. Here, a small library of Cp*Ir(III) complexes (**Ir1**-**6**, Table 1) with various combinations of chlorine, pyridine and NHC ligands were prepared. To our delight, **Ir3**, containing two monodentate NHC ligands, showed high catalytic performance for the *N*-alkylation of aromatic amines with a wide range of primary alcohols, including the challenging case of methanol.

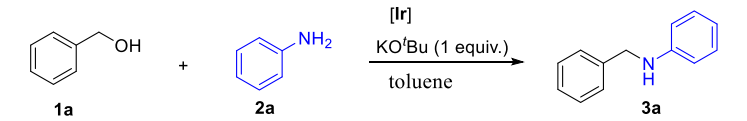
2. Results and discussion

Ir1-**6** (Table 1) were designed as catalyst precursors to probe the influence of chlorine versus pyridine versus NHC (**Ir1** vs **Ir3** vs **Ir6**, **Ir2** vs **Ir4**), monodentate versus chelating (**Ir2** vs **Ir3**, **Ir4** vs **Ir5** vs **Ir6**) and linkage patterns (**Ir4** vs **Ir5**). All the compounds were obtained via the procedures published in the literature and were isolated in good to excellent yields as air- and moisture-stable solids [39,40].

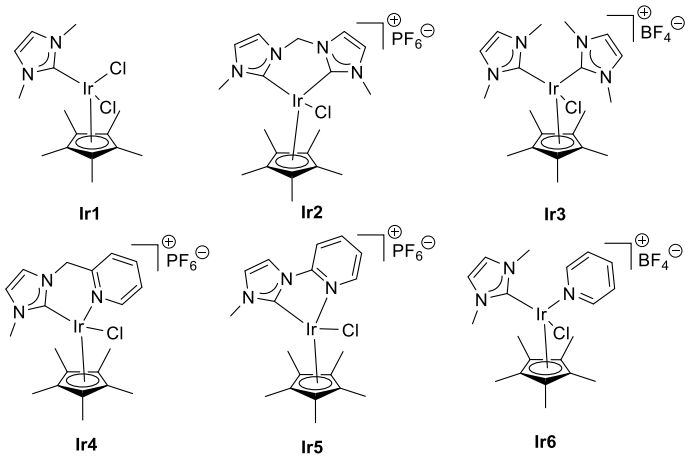
Firstly, the activities of the Ir complexes for the *N*-alkylation of amines with alcohols were investigated. The coupling of benzyl alcohol (**1a**) and aniline (**2a**) in toluene at 110 °C to afford *N*-benzylaniline (**3a**) was chosen as the benchmark reaction (Table 1). Interestingly, **Ir3** (entry 5), bearing two monodentate IMe ligands, emerged as the most active complex, surprisingly superior to the one mono-NHC complex **Ir1** (entry 3) and chelating bis-NHC congener **Ir2** (entry 4). **Ir4**-**6** (entries 6–8), bearing a labile pyridine ligand, gave inferior results compared with that of **Ir3** under identical conditions. These results indicated that the two monodentate NHC ligands are more prone to facilitate precatalyst activation and thus enhance the catalytic performance.

Thus, **Ir3** was selected as the catalyst precursor for further investigations. It is sufficient to get an excellent yield of up to 96% with 0.5 mol% catalyst loading (entries 9–10). A quick screening of different bases (entries 11–16) was also examined. When the reaction was conducted in the absence of a base (entry 11) or a weak base (entry 13), only a trace of the desired product was observed. Other bases such as KOH (entry 12), NaOH (entry 14) and Cs_2CO_3 (entry 15), provided very low yields. The amount of base could be further reduced to 0.75 equiv. without a significant decrease in the product yield (entries 16–17). However, the yield was reduced to 80% when the reaction was performed under an air atmosphere (entry 18). The temperature could be lowered to 80 or 50 °C, without diminishing the yields (entries 19 and 20).

Table 1
Screening of Ir catalysts for dehydrogenative coupling of 1a and 2a.[a, b]



1a 2a 3a



Entry[a]	cat. (mol %)	base (equiv.)	Temp. (°C)	Yield[b] (%)
1	–	KO*t*Bu (1)	110	–
2	$[Cp^*IrCl_2]_2$ (0.5)	KO*t*Bu (1)	110	39
3	**Ir1** (1)	KO*t*Bu (1)	110	72
4	**Ir2** (1)	KO*t*Bu (1)	110	52
5	**Ir3** (1)	KO*t*Bu (1)	110	99
6	**Ir4** (1)	KO*t*Bu (1)	110	58
7	**Ir5** (1)	KO*t*Bu (1)	110	75
8	**Ir6** (1)	KO*t*Bu (1)	110	64
9	**Ir3** (0.5)	KO*t*Bu (1)	110	96
10	**Ir3** (0.25)	KO*t*Bu (1)	110	76
11	**Ir3** (0.5)	KO*t*Bu (0)	110	trace
12	**Ir3** (0.5)	KOH (1)	110	45
13	**Ir3** (0.5)	K_2CO_3 (1)	110	trace
14	**Ir3** (0.5)	NaOH (1)	110	28
15	**Ir3** (0.5)	Cs_2CO_3 (1)	110	43
16	**Ir3** (0.5)	KO*t*Bu (0.75)	110	95
17	**Ir3** (0.5)	KO*t*Bu (0.5)	110	70
18[c]	**Ir3** (0.5)	KO*t*Bu (0.75)	110	80
19[d]	**Ir3** (0.5)	KO*t*Bu (0.75)	80	98(95%)[e]
20[d]	**Ir3** (0.5)	KO*t*Bu (0.75)	50	96

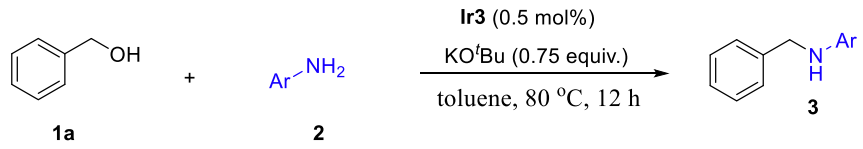
[a]*N*-alkylation reaction conditions: 0.5 mmol **1a**, 0.5 mmol **2a**, 1.0 mL toluene, 110 °C, 12 h. [b]GC yields. [c]under Air. [d]0.25 mL toluene. [e]isolated yield.

Having established the optimal reaction conditions, the scope of the **Ir3** catalyzed *N*-alkylation of amines with alcohols was explored. A large variety of anilines bearing different steric and electronic properties were successfully alkylated with benzyl alcohol in high yields at 80 °C (Table 2, entries 1–16). Halogen-substituted anilines were found to be a viable coupling partner, delivering the desired products in 87–93% yields (entries 4–6 and 8–9). More importantly, in all of these cases, no dehalogenation products were detected. Sterically encumbered anilines with *ortho*-substituted groups such as Me, Br, Ph and *t*Bu also could be converted to the corresponding products in 72–91% yields (entries 9–12). Moreover, heteroaromatic amines such as 2-aminopyridine and 3-aminopyridine were well-tolerated, as well (entries 15–16).

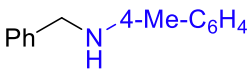
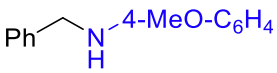
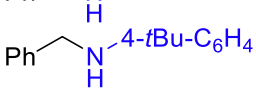
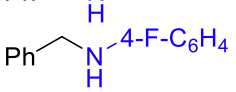
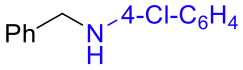
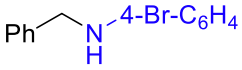
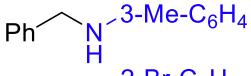
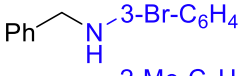
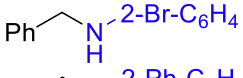
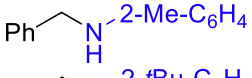
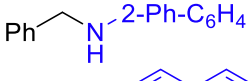
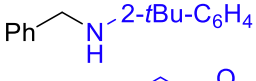
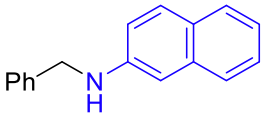
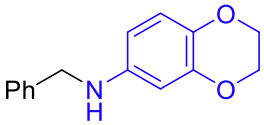
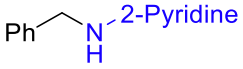
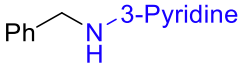
Next, we studied the reactions of aniline with different alcohols (Table 3). To our delight, the reaction of both electron-rich and electron-deficient benzylic alcohols proceeded smoothly, and furnished the desired products in excellent yields (entries 1–9, 83–94%). A series of functional groups such as the methoxy group (entry 2), halide groups (entries 3–5 and 8) and the trifluoromethyl group (entry 9) were compatible. Alcohols with a heteroaromatic group, such as 3-pyridinemethanol, 2-pyridinemethanol and 2-thienylmethanol, could convert to the desired products (entries 10–12) in 65–92% yields. Aliphatic alcohols, such as phenylpropanol, *n*-hexanol and *n*-butanol, were also alkylated to the corresponding products (entries 14–16) in 80–94% isolated yields. Notably, no dialkylation products were observed in all the cases.

Encouraged by these results, we further investigated the *N*-alkylation of amines with methanol (Tables 4 and 5). Methanol is intrinsically an extremely challenging alkylating agent in BH processes, owing to its high dehydrogenation energy compared with that of other alcohols, such as ethanol and higher alcohols. When the reaction was performed at 80 °C, low yields were obtained with different amounts of MeOH (entries 1–3). To our delight, at 100 °C, in the presence of 5 equiv. MeOH and without toluene, the yield increased to 99% (entry 4). However, attempts to lower the catalyst or base loading resulted in a decrease of the yield (entries 5–7). Gratifyingly, the reaction could be performed under an air atmosphere (entry 8). To further optimize the reaction

Table 2
Amine scope: *N*-alkylation of amines with benzyl alcohol catalyzed by **Ir3**.[a]

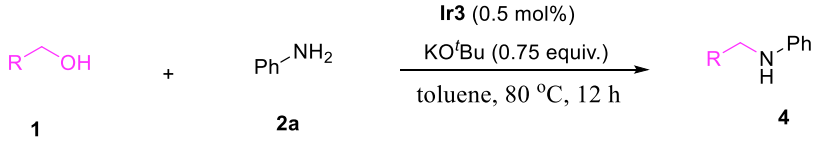


1a **2** **3**

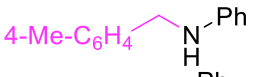
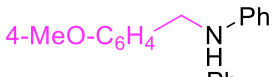
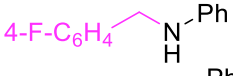
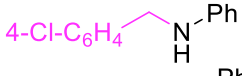
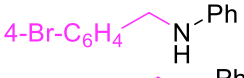
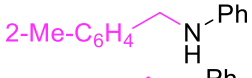
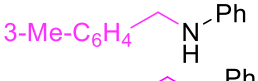
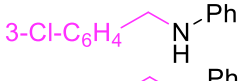
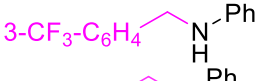
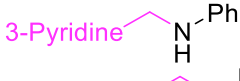
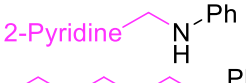
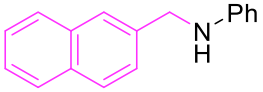
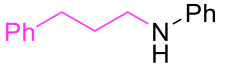
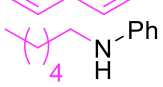
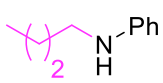
Entry	Product	Yield (%)	Entry	Product	Yield (%)
1		85	9		88
2		84	10		91
3		93	11		86
4		92	12		72
5		88	13		91
6		93	14		84
7		90	15		92
8		87	16		94

[a]*N*-alkylation reaction conditions: 0.5 mmol **1a**, 0.5 mmol **2**, KO*t*Bu (75 mol%), 1.0 mL toluene, 80 °C, 12 h.

Table 3
Alcohol scope; *N*-alkylation of aniline with alcohols catalyzed by **Ir3**.[a]

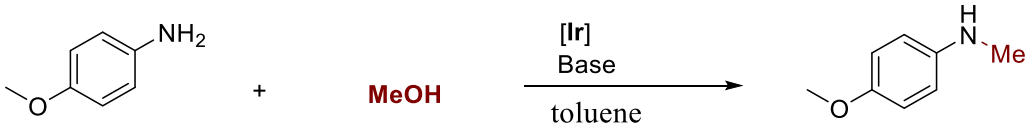


1 **2a** **4**

Entry	Product	Yield (%)	Entry	Product	Yield (%)
1		93	9		92
2		92	10		92
3		83	11		80
4		88	12		65
5		87	13		84
6		93	14		80
7		94	15		90
8		84	16		94

[a]*N*-alkylation reaction conditions: 0.5 mmol **1**, 0.5 mmol **2a**, KO*t*Bu (75 mol%), 1.0 mL toluene, 80 °C, 12 h.

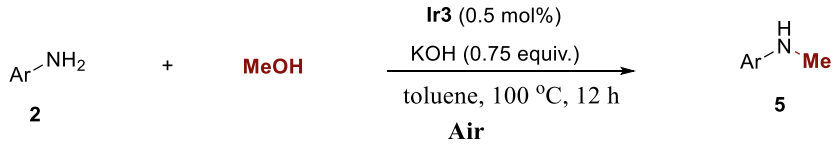
Table 4
Screening of Ir catalysts for dehydrogenative coupling of **2a** and MeOH.[a, b]

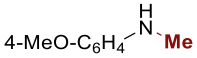
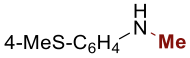
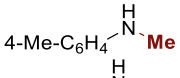
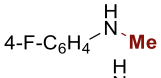
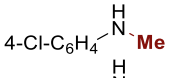
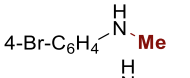
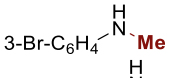
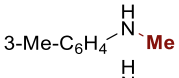
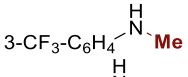
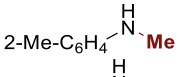
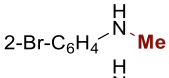
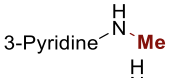
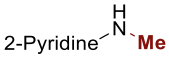
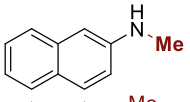
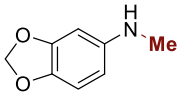
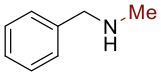


Entry[a]	cat. (mol %)	Base (equiv.)	MeOH (μL)	Toluene (mL)	Temp. (°C)	Yield[b] (%)
1	**Ir3** (0.5)	KO*t*Bu (0.75)	20 (1 equvi.)	0.25	80	12
2	**Ir3** (0.5)	KO*t*Bu (0.75)	200 (10 equvi.)	0.25	80	60
3	**Ir3** (0.5)	KO*t*Bu (0.75)	400 (20 equvi.)	0.25	80	34
4	**Ir3** (0.5)	KO*t*Bu (0.75)	100 (5 equvi.)	–	100	99
5	**Ir3** (0.25)	KO*t*Bu (0.75)	100 (5 equvi.)	–	100	87
6	**Ir3** (0.13)	KO*t*Bu (0.75)	100 (5 equvi.)	–	100	65
7	**Ir3** (0.5)	KO*t*Bu (0.5)	100 (5 equvi.)	–	100	77
8[c]	**Ir3** (0.5)	KO*t*Bu (0.75)	100 (5 equvi.)	–	100	99
9[c]	**Ir3** (0.5)	KOH (0.75)	100 (5 equvi.)	–	100	99
10[c]	**Ir3** (0.5)	K_2CO_3 (0.75)	100 (5 equvi.)	–	100	trace
11[c]	**Ir3** (0.5)	NaOH (0.75)	100 (5 equvi.)	–	100	85
12[c]	–	KOH (0.75)	100 (5 equvi.)	–	100	trace
13[c]	$[Cp^*IrCl_2]_2$ (0.25)	KOH (0.75)	100 (5 equvi.)	–	100	52
14[c]	**Ir1** (0.5)	KOH (0.75)	100 (5 equvi.)	–	100	46
15[c]	**Ir2** (0.5)	KOH (0.75)	100 (5 equvi.)	–	100	63
16[c]	**Ir4** (0.5)	KOH (0.75)	100 (5 equvi.)	–	100	34
17[c]	**Ir5** (0.5)	KOH (0.75)	100 (5 equvi.)	–	100	35
18[c]	**Ir6** (0.5)	KOH (0.75)	100 (5 equvi.)		100	40

[a]*N*-alkylation reaction conditions: *p*-anisidine (0.5 mmol), base (x equiv.), MeOH (x μL), toluene (x μL), temp., 12 h. [b]GC yields. [c]under Air.

Table 5
Amine scope; *N*-alkylation of amines with MeOH catalyzed by **Ir3**.[a]



Entry	Product	Yield (%)	Entry	Product	Yield (%)
1		95	9		91
2		53	10		74
3		94	11		65
4		93	12		90
5		92	13		94
6		88	14		80
7		94	15		82
8		82	16		0

[a]*N*-alkylation reaction conditions: 0.5 mmol **2a**, MeOH (100 μL), KOH (75 mol%), 100 °C, 12 h.

conditions, different bases (entries 9–11) and catalysts were evaluated. With KOH as the base, the yield was kept at 99% (entry 9). The control experiment showed that the *N*-methylation reaction did not proceed well in the absence of the catalyst (entry 12). It is clear that **Ir3** showed the highest catalytic performance compared to $[Cp^*IrCl_2]_2$, **Ir1**-**2** and **Ir4**-**6** (entries 13–18).

With the optimized conditions in hand, we subsequently explored the substrate scope for the *N*-methylation of amines. As

shown in Table 5, a series of anilines were converted into the corresponding *N*-methylated

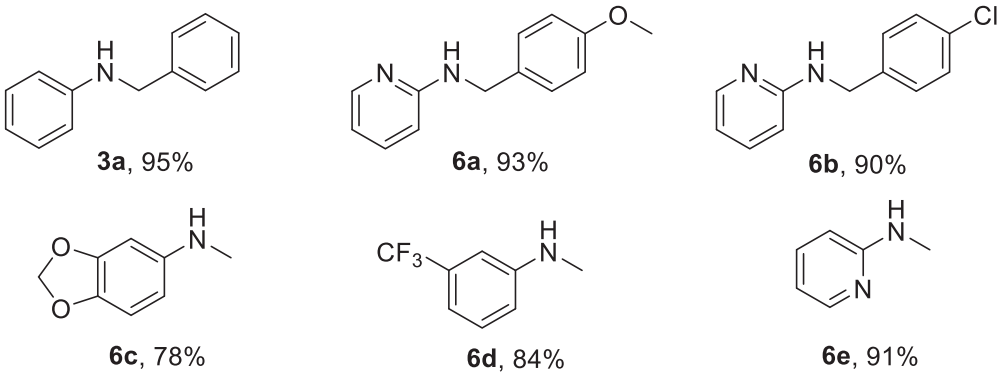
products in good to excellent isolated yields (entries 1–15: 53–95%). Importantly, anilines bearing a methoxy group (entry 1), a thioether group (entry 2), halide groups (entries 4–7 and 11) and a trifluoromethyl group (entry 9) were tolerated, and the desired products were obtained in 53–95% yields. The system was also applied to heteroaromatic amines and bulky anilines, giving the desired products in 90–94% yields (entries 12–15). However, attempts at the *N*-methylation of benzylamine with MeOH was unsuccessful (entry 16).

To establish the synthetic application of this catalytic process, the preparative scale syntheses of several compounds were conducted (scheme 2). Following the optimized reaction conditions, the corresponding products **3a** and **6a**-**e** were obtained in 78–95% yields in 10 mmol scale reactions. It should be noted that the catalytic system was highly successfully in converting 2-aminopyridine into the intermediates of the pharmaceutically active molecules mepyramine (**6a**) and chloropyramine (**6b**), extensively used for their antihistamine activity.

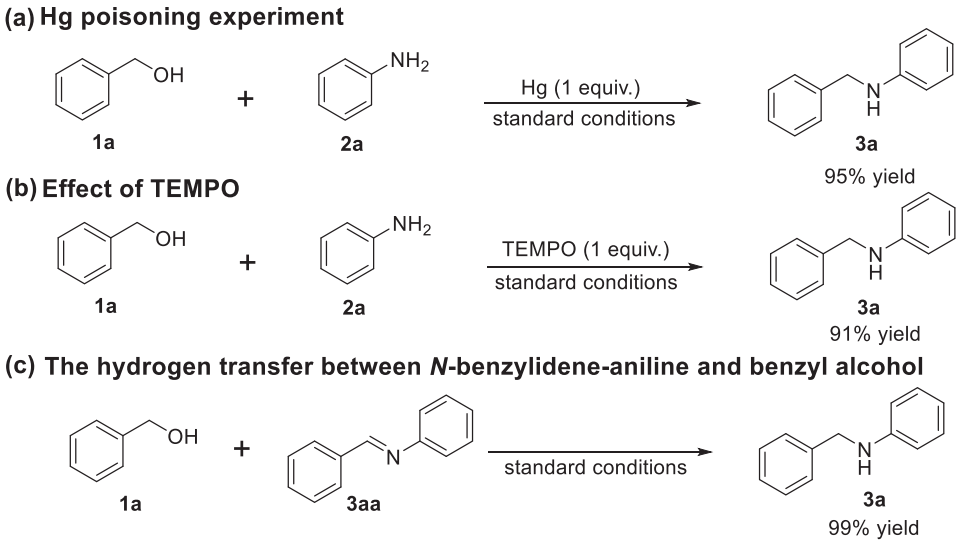
To gain insight into the mechanism of this reaction, some control experimental studies were carried out. Initially, the homogeneity of this catalytic system was confirmed by a mercury poisoning experiment (Scheme 3a). The addition of elemental mercury (1.0 equiv.) at the start of the reaction led to no inhibition of the reaction and 95% of the *N*-alkylation product formed. Then, addition of 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO, 1.0 equiv.) resulted in no inhibition of catalysis, which excludes a radical pathway (Scheme 3b). The transfer hydrogenation of the imine **3aa** with benzylic alcohol **1a** as the hydrogen source under the optimal conditions gave **3a** in quantitative yield (Scheme 3c). In addition, **3aa** was observed in the reaction process. These results indicate that the imine is a key intermediate and the reaction follows the BH/HA pathway.

3. Conclusion

In summary, we have applied a family of NHC-Ir(III) complexes for the *N*-alkylation of amines with alcohols. The different combinations of chlorine, pyridine and NHC ligands of these complexes have a great influence on their catalytic activity. To our delight, **Ir3**, containing two monodentate NHC ligands, is the most active catalyst, which can realize the reaction under mild conditions. A large variety of (hetero)aromatic amines react efficiently with various primary alcohols, including the challenging case of methanol. Notably, we have demonstrated the application potential of this protocol in the gram-scale synthesis of some valuable compounds. Initial mechanistic studies reveal that the reaction proceeds though the BA/HA pathway. These results imply the potential of the NHC-Ir complexes in BA/HA or other tandem catalytic reactions.



Scheme 2. Preparative scale synthesis of different compounds using **Ir3**.



Scheme 3. Preliminary mechanistic studies.

4. Experimental section

4.1. General procedures and materials

Non-halogenated solvents were dried over sodium benzophenone ketyl and halogenated solvents over CaH_2. All other commercial reagents were used without further purification.

NMR spectra were recorded using a Bruker 400 MHz spectrometer and chemical shifts are reported relative to TMS for ^1H and ^{13}C. GC analyses were recorded in a Shimadzu GC-2014C device equipped with a Wondacap 1 column.

4.2. General method for the N-alkylation of amines with alcohols

To a 15 mL reaction tube in a glovebox, was added complex **Ir3** (0.5 mol%), KO*t*Bu (75 mol%), the alcohol (0.5 mmol) and the amine (0.5 mmol) at room temperature. Then the tube was closed and removed from the glovebox. The reaction mixture was stirred at 80 °C for 12 h. After cooling to room temperature, the reaction mixture was diluted with ethyl acetate, filtered and dried under vacuum. The product was purified by column chromatography over silica-gel (300–400 mesh) with an appropriate mixture of petroleum ether and ethyl acetate (80:1).

4.3. General method for the N-methylation of anilines with methanol

To a 15 mL sealing tube, was added the amine (0.5 mmol), MeOH (100 μL), **Ir3** (0.5 mol%) and KOH (75 mol%), and the tube was closed with a screw-top cap. Next, the reaction mixture was stirred for 12 h at 100 °C. After cooling to room temperature, the mixture was diluted with ethyl acetate and filtered through a short pad of silica (2 cm in a Pasteur pipette). The silica was washed with ethyl acetate. The filtrate was evaporated and the crude residue was purified by column chromatography (SiO_2, petroleum ether: ethyl acetate = 80:1).

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgment

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.poly.2021.115289.

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