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Highly efficient gold(III)-catalyzed intermolecular hydroarylation of unactivated alkenes with arenes under mild conditions

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ABSTRACT

A simple and efficient method for functionalization of electron-rich arenes and heteroarenes with unactivated alkenes by Au(III)-catalyzed intermolecular hydroarylation under mild reaction conditions was developed. This method features a short reaction time (5 h) under mild conditions and has a broad substrate scope, including electron-rich arenes and heteroarenes, terminal and internal substituted aryl alkenes, and unactivated aliphatic alkenes.

1. Introduction

Au(I) and Au(III) complexes have increasingly been used as catalysts in a variety of organic transformations, and the majority of such transformations draw on the propensity of gold ions to activate alkynes toward nucleophilic addition [1]. In recent years, gold-catalyzed organic transformations have also been developed into a number of protocols to activate C=C bonds toward nucleophilic attack, and indeed, gold-catalyzed inter- and intramolecular addition of unactivated alkenes to oxygen- [2], nitrogen- [3], or active methylene-nucleophiles [4] have been documented in the literatures. However, to the best of our knowledge, there have been few examples on gold-catalyzed intermolecular hydroarylation of unactivated alkenes [5], which can be applied to a variety of alkenes and aryl substrates. Although Liu and co-workers have reported the use of $AuCl_3$/AgOTf or triflic acid as the catalyst for hydroarylation of alkenes, this reaction was confined to indole substrates [6].

Intermolecular hydroarylation of alkenes is an efficient and atom-economic approach to synthesize a variety of alkylated arenes and heteroarenes, which are important structural motifs commonly found in many pharmaceuticals, agro-, and fine chemicals [7]. Transition metal catalysts for such transformation reactions are of particular importance due to their high selectivity, synthetic efficiency and environmental friendliness [8–10]. In the literatures, hydroarylations of styrene derivatives with electron-rich aryl and heterocyclic compounds have also been carried out with $FeCl_3$ [11], $Bi(OTf)_3$ [12], or $BiCl_3$ [13] as the catalyst for the synthesis of a variety of 1,1-diarylalkanes. However, these reported intermolecular hydroarylation reactions usually require high reaction temperature (80–110 °C) and/or prolonged reaction time (~24 h). Furthermore, the alkene substrates of most of the reported intermolecular hydroarylations are confined to styrene derivatives [11–13]. Thus, a catalyst that induces addition of both aryl and heterocyclic compounds to both styrenes and aliphatic alkenes in a relatively short reaction time at lower temperature would be highly desirable. Owing to the ability of gold(I) and gold(III) to activate aryl C–H bonds [14], we envisioned that gold complexes might be able to catalyze C–C bond formation through activation of arenes toward attack by unactivated alkenes. As part of our program to develop gold-catalyzed C–O, C–N, and C–C bond formation reactions [15], herein we report a highly efficient gold(III)-catalyzed intermolecular hydroarylation of unactivated alkenes with arenes or heteroarenes under mild reaction conditions and have extended the substrate scope of alkenes to those having different steric and electronic properties.

2. Results and discussion

To identify optimal reaction conditions for gold-catalyzed hydroarylation of alkenes, a number of Ag^I, Au^I, and Au^{III} catalysts in different organic solvents at 50 °C were tested for the reaction of

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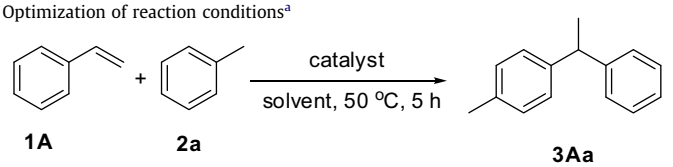
styrene (**1A**) with toluene (**2a**) (Table 1). The metal salts $AgSbF_6$, $Au(OAc)_3$, $AuCl_3$ and $Au(PPh_3)Cl$ failed to catalyze this reaction (Table 1, entries 1–4). In the presence of 5 mol% of $Au(PPh_3)Cl/AgSbF_6$ or $AuBr_3/AgSbF_6$, reaction of **1A** with **2a** in dichloroethane for 5 h gave the corresponding product **3Aa** in low yield (~30%) and poor regioselectivity ((2–5):1) (Table 1, entries 5 and 6). To our delight, using a combination of 5 mol% of $AuCl_3$ and 15 mol% of $AgSbF_6$ as a catalyst, the hydroarylation product **3Aa** was formed in 86% yield with good regioselectivity (9:1) (Table 1, entry 7). A dramatic decrease in the yield of **3Aa** was found when the amount of $AgSbF_6$ was reduced from 15 to 5 mol% (Table 1, entries 7–9). Other counteranions such as NO_3^-, BF_4^-, PF_6^- did not lead to hydroarylation activity, perhaps due to their good binding ability to gold(III) ion (Table 1, entries 10–12). Noncoordinating anion such as SbF_6^- was found to be the best counteranion for the reaction (Table 1, entry 7). The effect of solvent was also examined. Halogenated solvent gave better results than non-halogenated solvents (Table 1, entries 7 and 15–17), and the yield of **3Aa** remained almost the same under solvent-free conditions (Table 1, entry 18). The use of an excess amount of **2a** could effectively suppress some side reactions, and the yield of **3Aa** could be increased significantly from 41% to 86% (Table 1, entries 7, 19, and 20). When a 10:1 ratio of **2a** and **1A** was used, the formation of by-products became negligible and the yield of **3Aa** was the best. After optimization of reaction conditions, the protocol with 5 mol% of $AuCl_3$ and 15 mol% of $AgSbF_6$ at 50 °C in dichloroethane for 5 h gave the product in 86% yield.

Table 1
Optimization of reaction conditions[a]



Entry	Catalyst	Solvent	Selectivity (*p*-:*o*-)[b]	Yield (%)[b]
1	15 mol% $AgSbF_6$	$ClCH_2CH_2Cl$	–	Trace
2	5 mol% $Au(OAc)_3$	$ClCH_2CH_2Cl$	–	Trace
3	5 mol% $AuCl_3$	$ClCH_2CH_2Cl$	–	Trace
4	5 mol% $Au(PPh_3)Cl$	$ClCH_2CH_2Cl$	–	Trace
5	5 mol% $Au(PPh_3)Cl$/5 mol% $AgSbF_6$	$ClCH_2CH_2Cl$	2:1	41
6	5 mol% $AuBr_3$/15 mol% $AgSbF_6$	$ClCH_2CH_2Cl$	5:1	33
7	5 mol% $AuCl_3$/15 mol% $AgSbF_6$	$ClCH_2CH_2Cl$	9:1	86[c]
8	5 mol% $AuCl_3$/10 mol% $AgSbF_6$	$ClCH_2CH_2Cl$	9:1	24
9	5 mol% $AuCl_3$/5 mol% $AgSbF_6$	$ClCH_2CH_2Cl$	9:1	19
10	5 mol% $AuCl_3$/15 mol% $AgNO_3$	$ClCH_2CH_2Cl$	–	Trace
11	5 mol% $AuCl_3$/15 mol% $AgBF_4$	$ClCH_2CH_2Cl$	–	Trace
12	5 mol% $AuCl_3$/15 mol% $AgPF_6$	$ClCH_2CH_2Cl$	–	Trace
13	5 mol% $AuCl_3$/15 mol% AgOTf	$ClCH_2CH_2Cl$	9:1	27
14	5 mol% $AuCl_3$/15 mol% $AgClO_4$	$ClCH_2CH_2Cl$	7:1	51
15	5 mol% $AuCl_3$/15 mol% $AgSbF_6$	CH_2Cl_2	10:1	64[d]
16	5 mol% $AuCl_3$/15 mol% $AgSbF_6$	*c*-Hexane	–	Trace
17	5 mol% $AuCl_3$/15 mol% $AgSbF_6$	THF	–	Trace
18	5 mol% $AuCl_3$/15 mol% $AgSbF_6$	Toluene	9:1	88[e]
19	5 mol% $AuCl_3$/15 mol% $AgSbF_6$	$ClCH_2CH_2Cl$	9:1	55[f]
20	5 mol% $AuCl_3$/15 mol% $AgSbF_6$	$ClCH_2CH_2Cl$	9:1	41[g]

[a] Reaction conditions: 0.5 mmol of styrene, 5 mmol of toluene, 12 mL of solvent, 50 °C, 5 h.
[b] Yield and selectivity determined by ^1H NMR (internal standard: trimethyl(phenyl)silane).
[c] Yield of isolated product is based on **1A**.
[d] Reaction conditions: 0.5 mmol of styrene, 12 mL of CH_2Cl_2, 40 °C, 5 h.
[e] Reaction conditions: 0.5 mmol of styrene, 12 mL of toluene, 50 °C, 5 h.
[f] Reaction conditions: 0.5 mmol of styrene, 3 mmol of toluene, 12 mL of solvent, 50 °C, 5 h.
[g] Reaction conditions: 0.5 mmol of styrene, 2 mmol of toluene, 12 mL of solvent, 50 °C, 5 h.

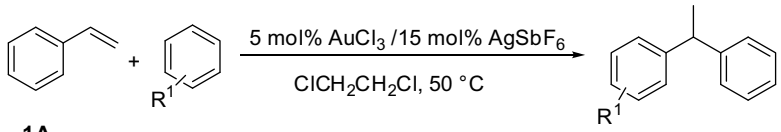
Having established the optimal conditions, we next investigated the effect of arenes in their reactions with styrene (**1A**) (Table 2). A wide range of arenes including aryl and heterocyclic variants could be used, leading to the formation of desired adducts in high yields and good regioselectivities. Regardless of the substituent on the aryl ring, arene derivatives furnished a variety of 1,1-diarylalkanes in 58–96% yields (Table 2, entries 1–5). In all cases, the *para*-substituted product was obtained as the major product, except in the case of benzene (**2b**) and *p*-xylene (**2d**). It should be noted that steric hindrance has little effect on this reaction. For instance, with mesitylene (**2f**) as the substrate, the product **3Af** was obtained in a good yield (79%) under the same reaction conditions (Table 2, entry 6). This Au(III)-catalyzed reaction allows for the synthesis of heteroarene derivatives. Treatment of thiophene (**2g**) with $AuCl_3$/$AgSbF_6$ furnished a 4.5:1 mixture of the regioisomeric product **3Ag** in 87% yield (Table 2, entry 7). This contrasts to the formation of similar heteroarenes in <60% yields from thiophene in other similar transition metal-catalyzed reactions [11–13]. This protocol can be scaled up to gram-scale synthesis of arene derivatives. A coupling reaction of **1A** (10 mmol) with **2a** (100 mmol) in the presence of 2 mol% of $AuCl_3$ and 6 mol% of $AgSbF_6$ afforded **3Aa** (1.6 g) in 78% yield for a reaction time of 7 h (Table 2, entry 8).

Following the successful gold(III)-catalyzed hydroarylation of styrene **(1A)** using various arenes, we decided to examine a series of unactivated alkenes in their reactions with anisole (**2c**) (Table 3). Generally, the reaction worked well for a range of styrene derivatives with substituents having different steric and electronic properties, and coupling of anisole (**2c**) with styrenes **1B**-**G** bearing electron-rich *p*-Me substituent and electron-withdrawing halogen-substitutents (F, Cl, Br) gave the corresponding adducts in 85–90% yields (Table 3, entries 1–6). Interestingly, the Au(III)-catalyzed reactions of disubstituted aryl alkenes also afforded the highly substituted arenes in good yields (Table 3, entries 8–10). For example, the styrene derivative **1I** having geminal substituent was transformed to the corresponding product **3Ic** containing a quaternary carbon center in 89% yield (Table 3, entry 8). Using indene (**1J**) and dihydronaphthalene (**1K**) as the substrates, aryldihydroindene and tetrahydronaphthalene derivatives **3Jc** and **3Kc**, the key moieties of which are commonly found in different products such as agrochemicals and fungicides, and useful compounds such as nafenopin, a peroxisome proliferator [12], were obtained in excellent yields (Table 3, entries 9 and 10).

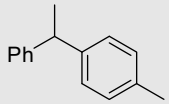
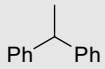
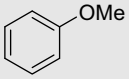
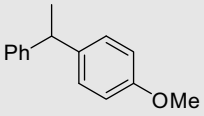
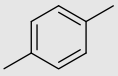
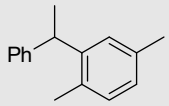
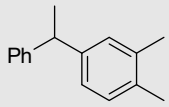
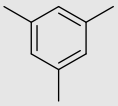
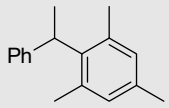
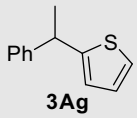
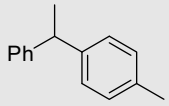
In the literatures, most addition of arenes to alkenes was conducted with aryl alkenes [11–13]. However, this gold(III)-catalyzed hydroarylation reaction could also be applicable to unactivated aliphatic alkenes. Norbornene (**1L**) underwent hydroarylation, producing *exo*-addition product **3Lc** stereoselectively in 93% yield, albeit with a low regioselectivity (1:1.1) (Table 3, entry 11). When unactivated 2-methyl-2-butene (**1M**) and 2,3-dimethyl-2-butene (**1N**) were used as the substrates in their reactions with anisole (**2c**) under the same conditions, alkylated arenes **3Mc** and **3Nc** were obtained in 95% and 93% yields, respectively, with excellent regioselectivities (>20:1) (Table 3, entries 12 and 13). The yield of **3Nc** remained almost the same upon decreasing catalyst loading from 5 to 1 mol% (Table 3, entry 13). It is interesting that **3Nc** could also be obtained by coupling of 3,3-dimethyl-1-butene (**1O**) with **1c** (Table 3, entry 14). This may be due to *in situ* conversion of **1O** to **1N** via a gold(III)-catalyzed methyl group migration reaction [16], and the **1N** in situ coupled with **1c** to give **3Nc**. Under similar conditions, gold-catalyzed couplings of a series of unactivated aliphatic cycloalkenes (**1P-S**) with anisole were accomplished to give the corresponding products **3Pc**-**Sc** in 59–93% yields (Table 3, entries 15–18).

For previously proposed gold(III)-catalyzed intermolecular hydroarylation of dienes with phenols in gold(III)-catalyzed phenol-diene annulations [5], the reported mechanism includes

Table 2
Scope of arenes of the intermolecular hydroarylation of styrene (**1A**)[a]

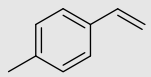
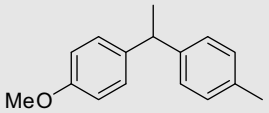
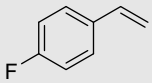
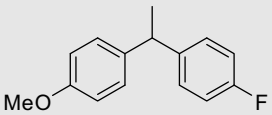
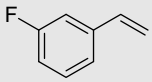
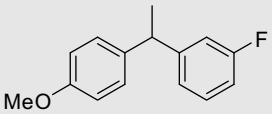
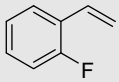
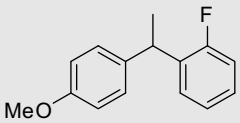
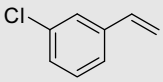
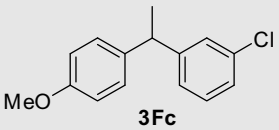
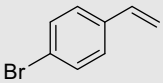
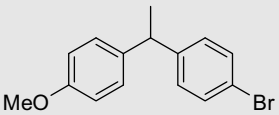
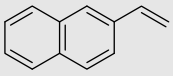
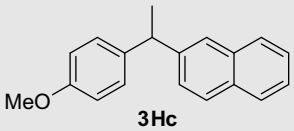
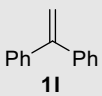
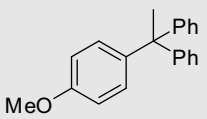
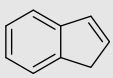
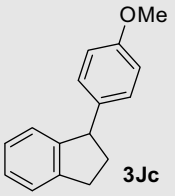


1A **2a-g** **3Aa-Ag**

Entry	Arene	Selectivity (*p*-:*o*-)[b]	Major product	Yield (%)[c]
1	 **2a**	9:1	 **3Aa**	86
2[d]	 **2b**	–	 **3Ab**	78
3	 **2c**	5.0:1	 **3Ac**	96 (91[e])
4	 **2d**	–	 **3Ad**	58
5	 **2e**	>20:1	 **3Ae**	79
6	 **2f**	–	 **3Af**	79
7	 **2g**	4.5:1	 **3Ag**	87
8[f]	 **2a**	9:1	 **3Aa**	78

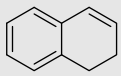
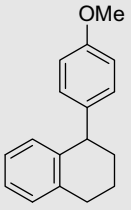
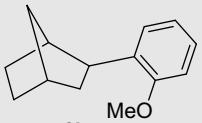
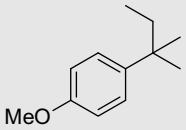
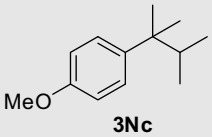
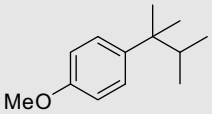
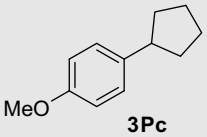
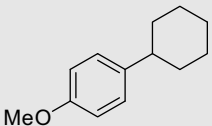
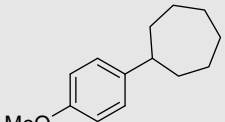
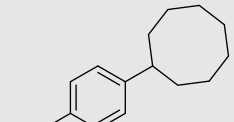
[a] Reaction conditions: 0.5 mmol of styrene, 5 mmol of arene, 12 mL of solvent, 50 °C, 5 h.
[b] Selectivity determined by ^{1}H NMR.
[c] Yield of isolated products is based on **1A**.
[d] Benzene was used as solvent.
[e] Reaction conditions: 1 mmol of styrene, 10 mmol of anisole, 1 mol% of catalyst loading, 24 mL of solvent, 50 °C, 7 h.
[f] Reaction conditions: **1A** (10 mmol), **2a** (100 mmol), 2 mol% of catalyst loading.

Table 3
Scope of unactivated alkenes of the intermolecular hydroarylation[a]

Entry	Alkene	Selectivity (*p*-:*o*-)[b]	Major product	Yield[c] (%)
1	 **1B**	5.3:1	 **3Bc**	86
2	 **1C**	4.4:1	 **3Cc**	90
3	 **1D**	1.6:1	 **3Dc**	88
4	 **1E**	2.0:1	 **3Ec**	85
5	 **1F**	1.8:1	 **3Fc**	87
6	 **1G**	5.4:1	 **3Gc**	90
7	 **1H**	7.5:1	 **3Hc**	78
8	 **1I**	>20:1	 **3Ic**	89
9	 **1J**	7.0:1	 **3Jc**	98

(*continued on next page*)

Table 3 (*continued*)

Entry	Alkene	Selectivity (*p*-:*o*-)[b]	Major product	Yield[c] (%)
10	 **1K**	4.9:1	 **3Kc**	97
11	 **1L**	1:1.1	 **3Lc**	93
12	 **1M**	>20:1	 **3Mc**	95
13	 **1N**	>20:1	 **3Nc**	93 (90[d])
14	 **1O**	>20:1	 **3Nc**	79
15	 **1P**	2.4:1	 **3Pc**	59
16	 **1Q**	1.8:1	 **3Qc**	85
17	 **1R**	1.6:1	 **3Rc**	90
18	*Cis*-  **1S**	1.0:1	 **3Sc**	93

[a] Reaction conditions: 0.5 mmol of alkene, 5 mmol of anisole, 12 mL of solvent, 50 °C, 5 h.
[b] Selectivity determined by ^{1}H NMR.
[c] Yield of isolated products is based on **1**.
[d] Reaction conditions: 1 mmol of alkene, 10 mmol of anisole, 24 mL of solvent, 1 mol% of catalyst loading, 50 °C, 7 h.

coordination of a diene double bond to Au(III), followed by formation of a C–Au bond through intermolecular addition of the C–H bond, with subsequent protonolysis of the C–Au bond to give the hydroarylation intermediate [5]. A similar mechanism as depicted in Scheme 1 can be proposed for the catalytic hydroarylation of unfunctionalized alkenes reported herein. Another mechanism, which could not be excluded, involves the metalation of an arene by cationic gold(III) to form an arylgold(III) intermediate and reaction of the arylgold(III) intermediate with alkene to form the hydroarylation product [14b,17].

3. Conclusion

In summary, we have developed a simple and efficient method for the functionalization of arenes and heteroarenes with product yields up to 98% and good regioselectivities under mild reaction conditions. This method features a short reaction time (5 h) under mild conditions and a broad substrate scope, including electron-rich arenes and heteroarenes, terminal and internal substituted aryl alkenes, and unactivated aliphatic alkenes.

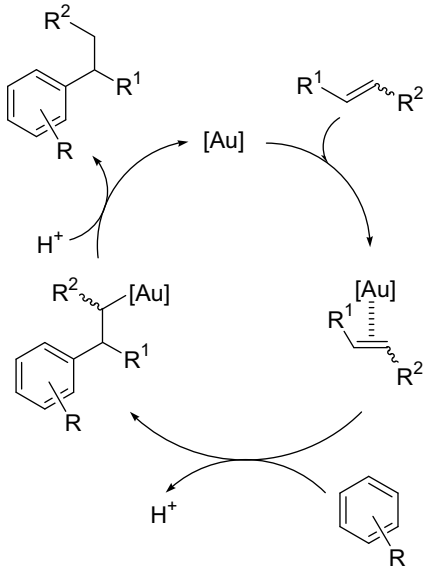
4. Experimental

4.1. General

All reagents were obtained commercially and used without further purification unless otherwise noted. All experiments were carried out under an inert atmosphere of Argon in the dark. All anhydrous solvents used in the reactions were dried and freshly distilled. NMR spectra were recorded in $CDCl_3$ on a Varian Mercury 300 spectrometer at 25 °C with TMS as an internal standard. Mass spectra were obtained on a HP5989A spectrometer (EI) or Agilent 5973N MSD spectrometer (EI). IR spectra were recorded, using KBr discs, on a Bio-Rad FTS-185 spectrometer.

4.2. General procedure for gold(III)-catalyzed intermolecular hydroarylation

$AuCl_3$ (7.6 mg, 0.025 mmol) and $AgSbF_6$ (25.6 mg, 0.075 mmol) were added into dichloroethane (4 mL) under Argon. The mixture was stirred at room temperature for 2 h. Arene (5 mmol) was added into the solution and the solution was heated to 50 °C. Alkene (0.5 mmol) dissolved in dichloroethane (8 mL) was introduced into the mixture of reaction system by syringe pump over 4 h under 50 °C. The mixture was stirred at 50 °C for additional 1 h, and the solvent was removed under reduced pressure. The residuum was subjected to flash chromatography with silica gel to give the product.



Scheme 1.

4.2.1. 1-Methyl-4-(1-phenylethyl)benzene (**3Aa**) [18]

Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.30–7.11 (m, 9H), 4.12 (q, J = 7.2 Hz, 1H), 2.31 (s, 3H), 1.62 (d, J = 7.2 Hz, 3H); IR (neat): 3084, 2966, 2928, 2873, 1602, 1513, 1494, 1452, 1374, 822, 722, 761, 699 cm^{-1}; MS (EI): m/z = 196([M$^+$], 37), 181(100), 165(38), 166(32), 182(16), 77(13), 89(12), 178(9).

4.2.2. 1,1-Diphenylethane (**3Ab**) [19]

Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.31–7.12 (m, 10H), 4.15 (q, J = 7.5 Hz, 1H), 1.64 (d, J = 7.5 Hz, 3H); IR (neat): 3206, 2962, 2928, 1602, 1495, 1448, 767, 701 cm^{-1}; MS (EI): m/z = 182([M$^+$], 36), 167(100), 165(40), 152(19), 115(19), 77(18), 166(16), 168(15).

4.2.3. 1-Methoxy-4-(1-phenylethyl)benzene (**3Ac**) [20]

Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.30−7.13 (m, 7H), 6.83 (m, d, J = 8.1 Hz, 2H), 4.57 (q, J = 7.2 Hz, 1H), 3.78 (s, 3H), 1.61 (d, J = 7.2 Hz, 3H); IR (neat): 3067, 2966, 1612, 1512, 1245, 1178, 1028, 833, 699 cm^{-1}; MS (EI): m/z = 212([M$^+$], 41), 197(100), 91(34), 165(24), 153(20), 152(16), 77(15), 198(15).

4.2.4. 1,4-Dimethyl-2-(1-phenylethyl)benzene (**3Ad**) [21]

Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.30–7.23 (m, 2H), 7.18–7.17 (m, 3H), 7.03 (m, 1H), 7.03–6.93 (m, 2H), 4.28 (q, J = 7.2 Hz, 1H), 2.32 (s, 3H), 2.18 (s, 3H), 1.59 (d, J = 7.2 Hz, 3H); IR (neat): 3025, 2967, 2929, 2873, 1602, 1495, 1451, 1377, 1158, 1031, 810, 699 cm^{-1}; MS (EI): m/z = 210([M$^+$], 42), 195(100), 165(32), 180(28), 196(19), 178(18), 179(16), 77(15).

4.2.5. 1,2-Dimethyl-4-(1-phenylethyl)benzene (**3Ae**) [11]

Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.31–7.15 (m, 5H), 7.07–7.04 (m, 1H), 7.00–6.95 (m, 2H), 4.09 (q, J = 7.2 Hz, 1H), 2.15 (s, 6H), 1.61 (d, J = 7.2 Hz, 3H); IR (neat): 3026, 2968, 2931, 1602, 1504, 1451, 823, 699 cm^{-1}; MS (EI): m/z = 210([M$^+$], 37), 195(100), 165(30), 180(24), 196(17), 178(16), 179(14), 89(14).

4.2.6. 1-(2,4,6-Trimethylphenyl)-1-phenylethane (**3Af**) [13]

Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.28–7.16 (m, 5H), 6.82 (s, 2H), 4.63 (q, J = 7.2 Hz, 1H), 2.26 (s, 3H), 2.11 (s, 6H), 1.65 (d, J = 7.2 Hz, 3H); IR (neat): 3002, 2965, 2930, 1611, 1511, 1427, 1036, 833, 816, 751 cm^{-1}; MS (EI): m/z = 224([M$^+$], 43), 209(100), 179(26), 178(20), 210(18), 194(17), 115(17), 193(14).

4.2.7. 2-(1-Phenylethyl)thiophene (**3Ag**) [22]

Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.31–7.22 (m, 5H), 7.15 (m, 1H), 6.97–6.92 (m, 1H), 6.83 (m, 1H), 4.35 (q, J = 7.5 Hz, 1H), 1.70 (d, J = 7.5 Hz, 3H); IR (neat): 3064, 3028, 2968, 2929, 2872, 1602, 1494, 1452, 1375, 1272, 1027, 851, 698 cm^{-1}; MS (EI): m/z = 188([M$^+$], 46), 173(100), 129(14), 174(13), 128(9), 171 (9), 77(8), 111(7).

4.2.8. 1-(1-(4-Methoxyphenyl)ethyl)-4-methylbenzene (**3Bc**) [12]

Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.15–7.05 (m, 6H), 6.83–6.81 (m, 2H), 4.07 (q, J = 7.2 Hz, 1H), 3.78 (s, 3H), 2.30 (s, 3H), 1.59 (d, J = 7.2 Hz, 3H); IR (neat): 3063, 2955, 2927, 1601, 1494, 1453, 1376, 1026, 754, 699 cm^{-1}; MS (EI): m/z = 226([M$^+$], 33), 211(100), 105(23), 212(17), 165(13), 152(10), 153(10), 91(9).

*4.2.9. 1-Fluoro-4-(1-(4-methoxyphenyl)ethyl)benzene (**3Cc**) [12]*

Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.18–7.10 (m, 4H), 6.98–6.92 (m, 2H), 6.83 (d, J = 7.8 Hz, 2H), 4.09 (q, J = 7.2 Hz, 1H), 3.78 (s, 3H), 1.59 (d, J = 7.2 Hz, 3H); IR (neat): 3036, 2965, 2932, 2875, 1886, 1608, 1584, 1509, 1463, 1303, 1247, 1180, 1159, 1035, 831, 753 cm^{-1}; MS (EI): m/z = 230([M$^+$], 31), 215(100), 171(20), 216(16), 183(14), 109(12), 170(11), 172(8).

*4.2.10. 1-Fluoro-3-(1-(4-methoxyphenyl)ethyl)benzene (**3Dc**)*

Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.24–7.17 (m, 1H), 7.12–7.10 (m, 2H), 6.98–6.96 (m, 1H), 6.90–6.81 (m, 4H), 4.08 (q, J = 7.2 Hz, 1H), 3.75 (s, 3H), 1.58 (d, J = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 164.51, 161.26, 157.96, 149.49, 149.39, 137.72, 129.71, 129.60, 128.41, 123.19, 123.15, 114.47, 114.19, 113.80, 113.37, 112.87, 112.59, 65.42, 55.14, 43.66, 43.64, 21.83, 19.05; IR (neat): 2968, 2934, 2837, 1614, 1590, 1512, 1490, 1448, 1375, 1303, 1247, 1180, 1144, 1124, 1034, 909, 872, 832, 786, 753, 693, 559 cm^{-1}; MS (EI): m/z = 230([M$^+$], 47), 215(100), 109(37), 183(21), 171(19), 216(16), 170(13), 18(9). HRMS(EI) Calcd. For [$C_{15}H_{15}OF$] 230.1107, Found 230.1103.

*4.2.11. 1-Fluoro-2-(1-(4-methoxyphenyl)ethyl)benzene (**3Ec**) [12]*

Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.21–7.02 (m, 6H), 6.85–6.82 (m, 2H), 4.43 (q, J = 7.2 Hz, 1H), 3.78 (s, 3H), 1.60 (d, J = 7.2 Hz, 3H); IR (neat): 2969, 2934, 2877, 2837, 1612, 1584, 1513, 1489, 1455, 1247, 1227, 1180, 1035, 832, 755 cm^{-1}; MS (EI): m/z = 230([M$^+$], 12), 115(100), 57(86), 114(51), 215(35), 216(25), 89(15), 132(12).

*4.2.12. 1-Chloro-3-(1-(4-methoxyphenyl)ethyl)benzene (**3Fc**) [12]*

Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.18–7.07 (m, 6H), 6.85–6.83 (m, 2H), 4.07 (q, J = 6.9 Hz, 1H), 3.78 (s, 3H), 1.59 (d, J = 6.9 Hz, 3H); IR (neat): 2967, 2934, 2836, 1612, 1595, 1512, 1478, 1464, 1303, 1248, 1180, 1035, 832, 786, 695 cm^{-1}; MS (EI): m/z = 246([M$^+$], 1), 57(100), 115(96), 114(50), 216(19), 89(15), 132(12), 63(10), 116(10).

*4.2.13. 1-Bromo-4-(1-(4-methoxyphenyl)ethyl)benzene (**3Gc**) [23]*

Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.39 (d, J = 8.4 Hz, 2H), 7.12–7.09 (m, 4H), 6.83 (d, J = 8.4 Hz, 2H), 4.06 (q, J = 7.2 Hz, 1H), 3.78 (s, 3H), 1.58 (d, J = 7.2 Hz, 3H); IR (neat): 2967, 2933, 2835, 1611, 1513, 1484, 1404, 1378, 1303, 1244, 1177, 1072, 1028, 1008, 842, 782, 742, 545 cm^{-1}; MS (EI): m/z = 290([M$^+$], 36), 275(100), 277(99), 292(35), 152(29), 153(22), 165(20), 276(17).

*4.2.14. 2-(1-(4-Methoxyphenyl)ethyl)naphthalene (**3Hc**) [24]*

Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.80–7.68 (m, 5H), 7.45–7.34 (m, 2H), 7.19–7.16 (m, 2H), 6.85–6.82 (m, 2H), 4.27 (q, J = 6.9 Hz, 1H), 3.78 (s, 3H), 1.70 (d, J = 6.9 Hz, 3H); IR (neat): 3055, 2965, 2932, 2835, 1633, 1610, 1584, 1511, 1463, 1440, 1303, 1246, 1179, 1034, 857, 832, 820, 751, 551 cm^{-1}; MS (EI): m/z = 262([M$^+$], 1), 57(100), 115(97), 114(50), 216(16), 89(15), 74(13), 132(12), 63(11).

*4.2.15. 1-(1,1-Diphenylethyl)-4-methoxybenzene (**3Ic**) [12]*

Yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.29–7.20 (m, 6H), 7.11–7.10 (m, 4H), 7.00 (d, J = 9.0 Hz, 2H), 6.80 (d, J = 9.0 Hz, 2H), 3.80 (s, 3H), 2.17 (s, 3H); IR (neat): 3062, 3014, 2975, 2933, 2834, 1959, 1888, 1609, 1596, 1512, 1490, 1461, 1442, 1376, 1294, 1248, 1183, 1029, 913, 855, 809, 788, 765, 700 cm^{-1}; MS (EI): m/z = 288([M$^+$], 4), 247(100), 262(41), 57(38), 115(37), 215(31), 202(26), 141(25), 273(23).

*4.2.16. 1-(4-Methoxyphenyl)-2,3-dihydro-1 H-indene (**3Jc**) [12]*

Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.30–7.26 (m, 1H), 7.20–7.10 (m, 4H), 6.96–6.93 (m, 1H), 6.87–6.84 (m, 2H), 4.29 (t, J = 8.1 Hz, 1H), 3.80 (s, 3H), 3.04–2.88 (m, 2H), 2.61–2.50 (m, 1H), 2.08–1.98 (m, 1H); IR (neat): 3067, 3020, 2954, 2836, 2064, 1612, 1585, 1512, 1478, 1457, 1441, 1344, 1303, 1245, 1178, 1107, 1038, 829, 763 cm^{-1}; MS (EI): m/z = 224([M$^+$], 100), 173(86), 193(66), 115(53), 223(52), 188(41), 127(37), 116(32).

*4.2.17. 1-(4-Methoxyphenyl)-1,2,3,4-tetrahydronaphthalene (**3Kc**) [12]*

Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.12 (m, 2H), 7.08–7.00 (m, 3H), 6.86–6.81 (m, 3H), 4.07 (t, J = 6.0 Hz, 1H), 3.79 (s, 3H), 2.94–2.80 (m, 2H), 2.17–2.09 (m, 1H), 1.88–1.74 (m, 3H); IR (neat): 3003, 2922, 2851, 1612, 1585, 1514, 1491, 1454, 1302, 1263, 1241, 1176, 1110, 1032, 835, 817, 771, 741 cm^{-1}; MS (EI): m/z = 238([M$^+$], 32), 130(100), 129(30), 115(28), 165(22), 179(20), 178(19), 210(17).

*4.2.18. exo-2-(o-Methoxyphenyl)bicyclo[2.2.1]heptane (**3Lc**) [25]*

Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.15–7.05 (m, 2H), 6.93–6.81 (m, 2H), 3.82 (s, 3H), 3.00–2.95 (m, 1H), 2.34–2.31 (m, 2H), 1.82–1.74 (m, 1H), 1.67–1.12 (m, 7H); IR (neat): 2952, 2870, 2834, 1612, 1583, 1513, 1491, 1249, 1180, 1108, 1037, 824, 752 cm^{-1}; MS (EI): m/z = 202([M$^+$], 3), 149(100), 121(28), 147(20), 91(16), 190(11), 150(11), 134(8), 77(8).

*4.2.19. 1-Methoxy-4-tert-pentylbenzene (**3Mc**) [26]*

Colorless oil . ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.24 (d, J = 9.0 Hz, 2H), 6.84 (d, J = 9.0 Hz, 2H), 3.79 (s, 3H), 1.61 (q, J = 7.2 Hz, 1H), 1.26 (s, 6H), 0.67 (t, J = 7.2 Hz, 3H); IR (neat): 3103, 3063, 3029, 2965, 2878, 2836, 1612, 1582, 1515, 1465, 1443, 1385, 1363, 1305, 1251, 1185, 1117, 1039, 1010, 828, 807, 775, 649 cm^{-1}; MS (EI): m/z = 178([M$^+$], 13), 149(100), 121(31), 150(14), 109(10), 91(9), 163(6), 77(5).

*4.2.20. 2,3-Dimethyl-3-(4-methoxyphenyl)butane (**3Nc**) [27]*

Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.24 (d, J = 8.4 Hz, 2 H), 6.83 (d, J = 8.4 Hz, 2H), 3.81 (s, 3H), 1.90–1.81 (m, 1H), 1.22 (s, 6H), 0.75 (d, J = 6.6 Hz, 6H); IR (neat): 2963, 2876, 2835, 1612, 1582, 1514, 1486, 1442, 1378, 1296, 1253, 1186, 1149, 1118, 1086, 1039, 1011, 830, 808, 762, 559 cm^{-1}; MS (EI): m/z = 192([M$^+$], 3.8), 148(100), 121(19), 150(11), 91(11), 109(8), 77(6), 147(5).

*4.2.21. 1-Cyclopentyl-4-methoxybenzene (**3Pc**) [28]*

Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.16 (d, J = 8.7 Hz, 2H), 6.83 (d, J = 8.7 Hz, 2H), 3.79 (s, 3H), 2.94 (m, 1H), 2.08–2.00 (m, 2H), 1.84–1.50 (m, 6H). IR (neat): 2997, 2954, 2869, 2835, 1612, 1584, 1514, 1464, 1442, 1304, 1246, 1179, 1109, 1040, 826, 808, 753 cm^{-1}; MS (EI): m/z = 176([M$^+$], 47), 147(100), 121(42), 91(34), 134(28), 148(19), 119(11), 77(9).

*4.2.22. 1-Cyclohexyl-4-methoxybenzene (**3Qc**) [29]*

Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.13 (d, J = 6.9 Hz, 2H), 6.84 (d, J = 6.9 Hz, 2H), 3.78 (s, 3H), 2.50–2.38 (m, 1H), 1.90–1.69 (m, 5H), 1.46–1.16 (m, 5H). IR (neat): 2996, 2926, 2852, 1611, 1583, 1514, 1449, 1285, 1178, 1105, 1040, 825, 796, 751 cm^{-1}; MS (EI): m/z = 190([M$^+$], 57), 147(100), 121(61), 149(51), 91(37), 134(25), 77(15), 148(14).

*4.2.23. 1- Cycloheptyl-4-methoxy-benzene (**3Rc**)*

Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$/TMS): δ 7.12 (d, J = 8.7 Hz, 2H), 6.83 (d, J = 8.7 Hz, 2H), 3.79 (s, 3H), 2.66–2.60 (m, 1H), 1.92–1.53 (m, 12H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 157.36, 142.28, 127.46, 113.59, 55.20, 46.16, 37.03, 27.90, 27.10; IR (neat):

3064, 3030, 2925, 2855, 2836, 1890, 1600, 1586, 1512, 1492, 1463, 1328, 1291, 1241, 1176, 1100, 1054, 1035, 949, 819, 751, 740 cm^{-1}; MS (EI): m/z = 204([M^+], 87), 147(100), 121(68), 91(54), 134(35), 119(23), 205(16), 122(12). HRMS(EI) Calc. For [$C_{14}H_{20}O$]: 204.1514. Found: 204.1513.

4.2.24. 1- Cyclooctyl-4-methoxy-benzene (**3Sc**) [30]

Colorless oil. ^1H NMR (300 MHz, $CDCl_3$/TMS): δ 7.11 (d, J = 8.1 Hz, 2H), 6.82 (d, J = 8.1 Hz, 2H), 3.78 (s, 3H), 2.75–2.69 (m, 1H), 2.08–2.01 (m, 1H), 1.83–1.53 (m, 13H). IR (neat): 2923, 2854, 1600, 1585, 1492, 1465, 1447, 1290, 1241, 1184, 1131, 1055, 1034, 751, 738 cm^{-1}; MS (EI): m/z = 218([M^+], 55), 147(100), 121(76), 91(54), 134(53), 119(32), 259(20), 258(20).

4.3. Procedure for large scale hydroarylation

$AuCl_3$ (60.6 mg, 0.2 mmol) and $AgSbF_6$ (206.2 mg, 0.6 mmol) were added into dichloroethane (20 mL) under Argon. The mixture was stirred at room temperature for 2 h. Toluene (9.32 g, 100 mmol) was added into the solution and the solution was heated to 50 °C. Styrene (1.092 g, 10.48 mmol) was dissolved in dichloroethane (40 mL) and introduced into the mixture of reaction system by syringe pump over 6 h under 50 °C. The mixture was stirred at 50 °C for additional 1 h, and the solvent was removed under reduced pressure. The residuum was subjected to flash chromatography with silica gel to give the product (1.610 g, 8.20 mmol, yield: 78%).

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