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Ruthenium-catalyzed alkylarylation of alkenes *via para*-selective C-H functionalization of unprotected anilines

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ABSTRACT

Metal-catalyzed alkylarylation of two carbon−carbon (C-C) bonds across vicinal C(sp^2) sites in olefins can provide access to diverse combinatorial chemical space. Pre-activated arenes are often used as aryl sources, but the direct utilization of Csp^2−H bond instead of pre-activated arenes remains underdeveloped. Herein, ruthenium-catalyzed alkylarylation of alkenes *via para*-selective C-H functionalization of unprotected aniline has been reported. It bears good functional group tolerance and enables late functionalization of complex natural products. Evaluation of cone angle and minimum percent buried volume help to determine the optimal ligand. The mechanism studies reveal the dissociation of *p*-cymene during the catalytic cycle which triggers the redox processes for radical generation.

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Metal-catalyzed formation of two carbon–carbon (C-C) bonds across vicinal C(sp^2) sites in olefins is a powerful method to construct complex molecules rapidly from readily available starting materials [1–8]. Despite the great advances, the development of such a method is generally hindered by β-H elimination from β-H−C(sp^3)-[M] intermediates [9–15] and regioselectivity placing two discrete carbon sources across the vicinal C(sp^2) sites [1,16–20]. To address these issues, a coordinating group is normally required either by preinstallation or a native functional group to induce a proximity bias at the vicinal sites for regiocontrol and subsequently generate transient metallacycles for addressing β-H elimination [21–25]. An alternative approach that proceeds by reductive coupling *via* radical intermediates [26] is also emerging as a powerful complementary protocol [27]. In these strategies, pre-activated arenes are often used as aryl sources, including aryl metal reagents, [13,28–32] aryldiazonium salts [33,34] and aryl halides (Scheme 1a) [35–37]. Despite significant progress, the direct utilization of Csp^2−H bond as carbon sources instead of pre-activated arenes remains elusive.

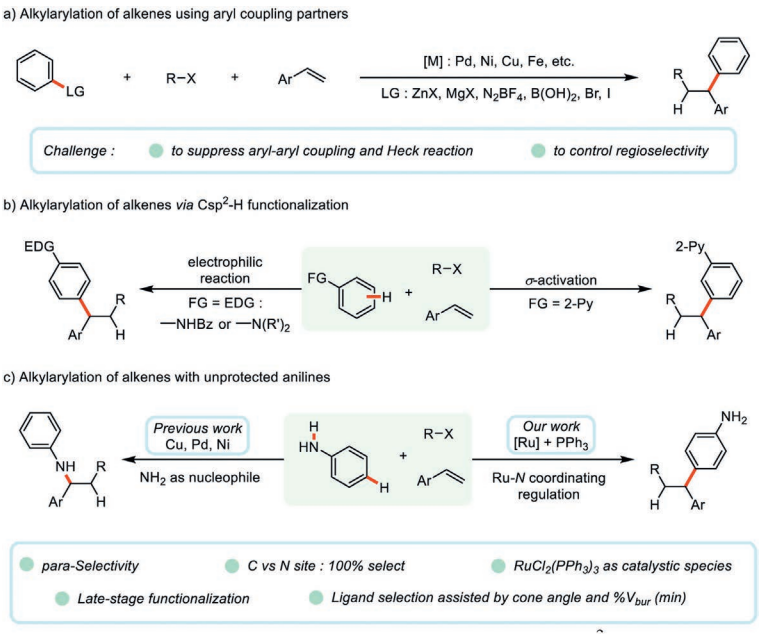
With the development of C-H activation catalyzed by transition metals, several types of arenes with special structures have been removed from dependence on leaving groups, and can directly undergo the alkylarylation of alkenes, with atomic economy and step economy, and with good site selectivity (Scheme 1b). Besides the electron rich *N*-heteroarenes (*e.g.*, indole) [38–43], 8-aminoquinoline [44] and *N*,*N*-disubstituted aniline derivatives [45] successfully afforded 1,2-alkylarylation products from olefins *via* combination of radical pathways with electrophilic substitution at arenes. Additionally, σ-activation through inner-sphere electron transfer, and cyclometallation enables the difunctionalization of alkene using 2-phenylpyridine/pyrimidine [46–52]. Despite these progresses, Csp^2−H coupling sources are limited and alkylarylation of alkenes using this protocol remains underdeveloped.

Aniline is one of the important amines and has a very wide range of uses in industry and manufacturing as well as in the pharmaceutical field [53–57]. Modification of anilines by site-selective C-H activation has attracted great attention to tune their function and activity [58–61]. Due to the good nucleophilicity of amino group and low steric hindrance of aniline, the alkylarylation of alkenes involving unprotected aniline normally led to alkylamination products [62–66], and alkylarylation of alkenes *via para*-C-H activation of unprotected aniline remains unsolved (Scheme 1c). Recently, Ru-catalyzed *para*-C-H alkylation of unprotected aniline *via* σ-activation was disclosed [67–74]. We herein successfully achieved the radical alkylarylation of alkenes *via para*-C-H activation of unprotected aniline. Cone angle and minimum percent buried volume (%V_{bur}) were used to evaluate the ligand effect. The

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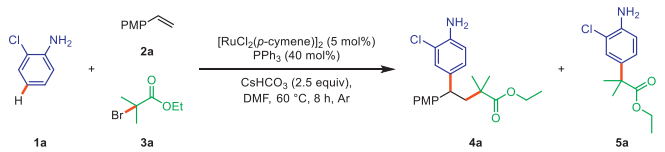
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Scheme 1. Strategies for alkylarylation across vicinal C(sp^2) sites in alkenes.

Table 1
Optimization studies for three-component reactions of alkenes with anilines.[a]



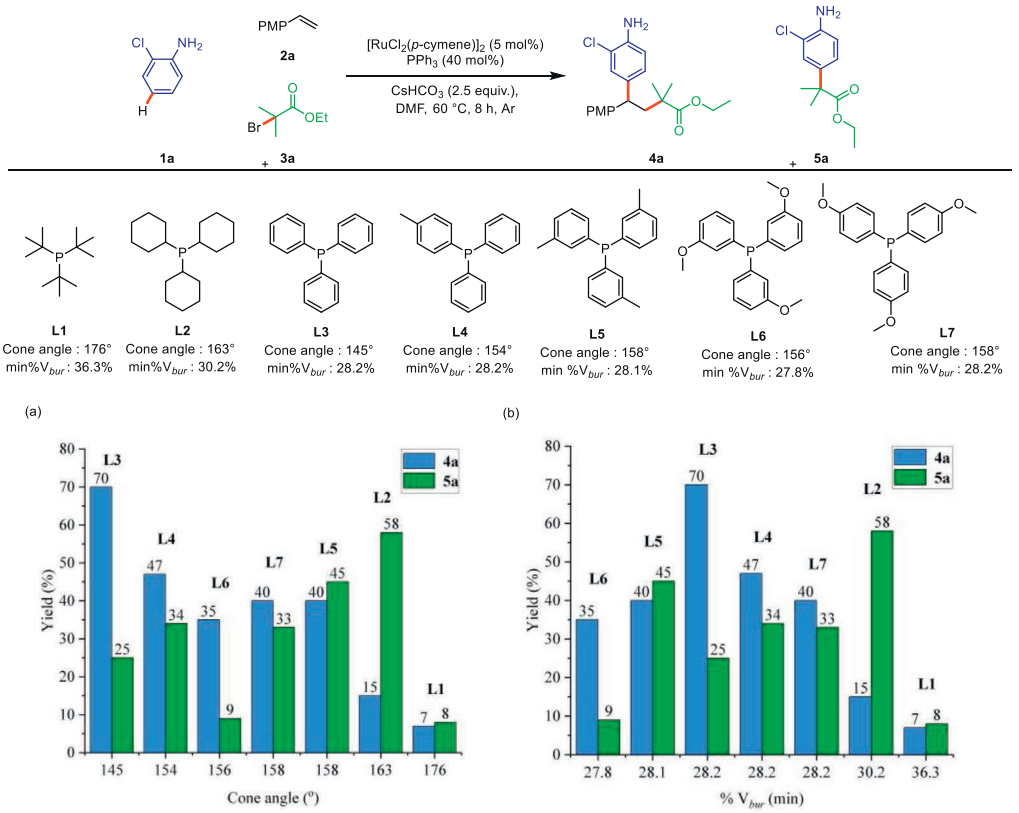
Entry	Deviation from standard conditions	**4a** (%)	**5** (%)
1	None	70	25
2	Without PPh_3	NR	NR
3	Without $[RuCl_2(p\text{-cymene})]_2$	NR	NR
4	Without Ar	NR	NR
5	$RuCl_3{\cdot}3H_2O$ instead of $[RuCl_2(p\text{-cymene})]_2$	65	17
6	$Ru_3(CO)_{12}$ instead of $[RuCl_2(p\text{-cymene})]_2$	Trace	Trace
7	$KHCO_3$ instead of $CsHCO_3$	40	9
8	Cs_2CO_3 instead of $CsHCO_3$	Trace	Trace

[a] Standard conditions: **1a** (0.6 mmol, 3 equiv.), **2a** (0.6 mmol, 3 equiv.), **3a** (0.2 mmol, 1 equiv.), $[RuCl_2(p\text{-cymene})]_2$ (5 mol%), PPh_3 (40 mol%), $CsHCO_3$ (2.5 equiv.), DMF (1.0 mL), 60 °C, 8 h, under Ar. Isolated yields. PMP = *para*-methoxyphenyl.

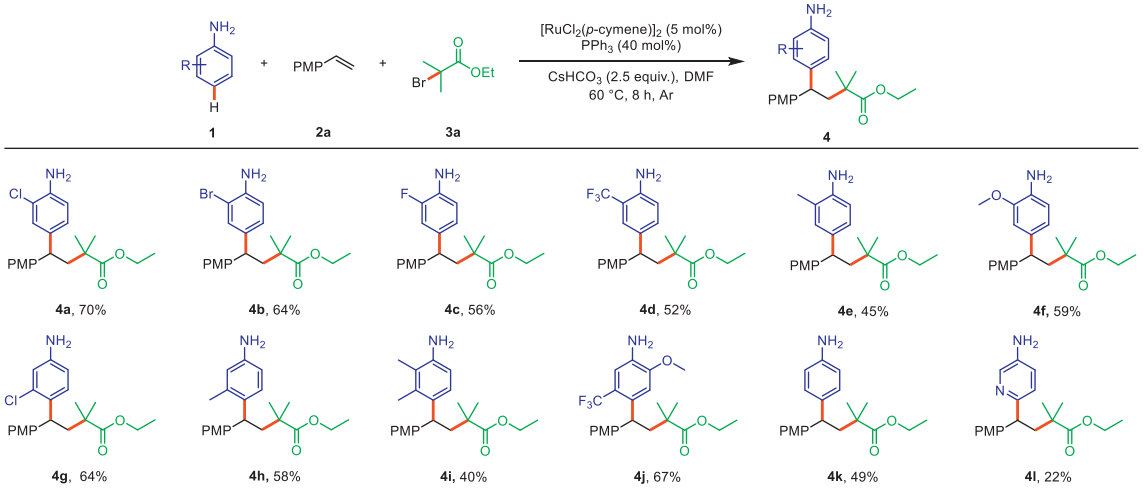
protocol allows absolute C *vs.* N site selectivity and good functional group tolerance, which enables the late-stage functionalization of nature products. The mechanism studies reveal a dissociation of *p*-cymene during the catalytic processes, which is distinct from most previous σ-activation pathways.

We commenced our study by investigating the alkylarylation of alkene **2a** with aniline **1a** and α–bromo alkane **3a**. After rigorous screening of the reaction conditions (For detail screening, see Supporting information), the optimal catalytic system with $[RuCl_2(p\text{-cymene})]_2$ (5 mol%), PPh_3 (40 mol%), and $CsHCO_3$ (2.5 equiv.) in DMF successfully afforded **4a** in 70% isolated yield (Table 1, entry 1). The control experiments revealed that the reaction did not proceed in the absence of PPh_3 (entry 2), which is consistent with previous phosphine ligand involving catalytic systems [75]. To elucidate the influence of different phosphine ligands, we correlated the experimental selectivities and yields with key descriptors of phosphine ligands, the cone angle (min) and the minimum percent buried volume (min $\%V_{bur}$). In Scheme 2, we generated plots illustrating the yields of **4a** (blue) and **5** (green) as a function of these parameters. The analysis revealed that phosphines with cone angles within a relatively narrow range (145°-156°) afforded slightly better selectivity for the three-component product **4a** over **5**. Increasing the cone angles led to the *para*-alkylated **5** as predominant product. Additionally, when the min $\%V_{bur}$ of the phosphine was approximately 28.2%, the reaction exhibited better combined yield of **4a** and **5**. The selectivity of the reaction with different phosphine ligands at the same min $\%V_{bur}$ differ greatly, which might be attributed to their different cone angles. Thus, we proposed in this catalytic system, cone angle might regulate the regioselectivity while min $\%V_{bur}$ would control the combined yields. The highest overall yield of and selectivity for **4a** is observed with PPh_3, ligand that have cone angle and min $\%V_{bur}$ in the optimal range (145° and 28.2%, respectively) along with three component products. The reaction failed to proceed in the absence of $[RuCl_2(p\text{-cymene})]_2$ underscoring the critical role of Ru catalyst in the reaction (entry 3). Inert gas protection was also required, suggesting that a radical pathway was plausible (entry 4). Substituting $[RuCl_2(p\text{-cymene})]_2$ with $RuCl_3{\cdot}3H_2O$ slightly decreased the reaction yield with better selectivity (entry 5). It suggests the reaction may proceed *via* Ru catalyst without *p*-cymene. In contrast, Ru(0) catalyst was not effective for the reaction (entry 6). Further optimization revealed that bases play an important role in this transformation (entries 7 and 8). $KHCO_3$ displayed catalytic activity, but Cs_2CO_3 obtained product with trace yield.

Under the optimized conditions, we systematically evaluated the functional group compatibility of this transformation using diverse anilines (Scheme 3). Both electron-withdrawing and electron-donating substituents including halogens (**4a**-**4c**, **4g**), trifluoromethyl (**4d**), methyl (**4e**, **4h**), and methoxy groups (**4f**) delivered 1,1-diarylation products **4a**-**4h** in 45%–70% yields. Notably, *ortho*-position substitution with bulkier groups (such as CH_3) rather than halogen led to lower yields. This was likely because the bulky substituent interfered with the coordination between the aniline and the Ru catalyst, thereby affecting the reaction efficiency. The protocol also accommodated disubstituted substrates (**4i**-**4j**). *N*–Heteroarenes were also accessible substrates, delivering **4l** in 22% yield.

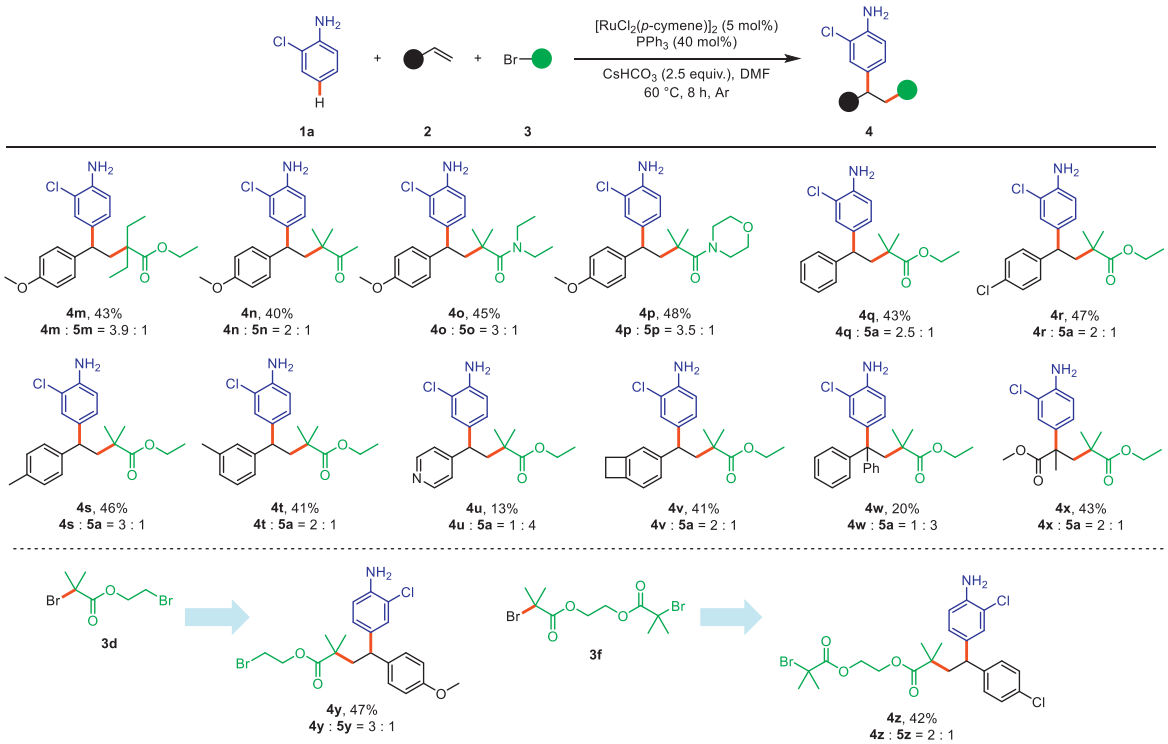


Scheme 2. Analysis of alkylarylation of **2a** using steric parameters (a) cone angle (°) and (b) minimum percent buried volume (%V_{bur}). Standard conditions: **1a** (0.6 mmol, 3 equiv.), **2a** (0.6 mmol, 3 equiv.), **3a** (0.2 mmol, 1 equiv.), [$RuCl_2$(*p*-cymene)]$_2$ (5 mol%), PPh_3 (40 mol%), $CsHCO_3$ (2.5 equiv.), DMF (1.0 mL), 60 °C, 8 h, under Ar. Isolated yields. PMP = *para*-methoxyphenyl.



Scheme 3. Substrate scope of anilines. Standard conditions: **1** (0.6 mmol, 3 equiv.), **2a** (0.6 mmol, 3 equiv.), **3a** (0.2 mmol, 1 equiv.), [$RuCl_2$(*p*-cymene)]$_2$ (5 mol%), PPh_3 (40 mol%), $CsHCO_3$ (2.5 equiv.), DMF (1.0 mL), 60 °C, 8 h, under Ar. Isolated yields. PMP = *para*-methoxyphenyl.

Substrate scope for α–bromo alkane derivatives and alkenes was subsequently investigated (Scheme 4). Sterically hindered α–bromo esters (**4m**), α–bromo ketones (**4n**), and α–bromo amides (**4o-4p**) proved to be compatible substrates. The reaction scope was further extended to styrene derivatives and α,β-unsaturated alkenes. *para*-Substituted styrenes with both electron-donating (**4s**) and electron-withdrawing groups (**4r**) exhibited good reactivity. *meta*-Substituents also demonstrated similar reactivity in three-component reaction (**4t**). The styrenes bearing pyridine moiety were also compatible with the reaction conditions to afford **4u** Strained four-membered ring was maintained during the reaction of 4-vinylbenzocyclobutene (**4v**), further proving the mildness of the protocol. The reaction of the 1,1-diphenylethylene proceeded, yielding a three-component adduct (**4w**) rather than a simple radical adduct. α,β-Unsaturated esters (**4x**) were also viable substrates for 1,2-alkylarylation. Notably, in contrast to other substates, 4-vinylpyridine and 1,1-diphenylethylene afforded *para*-substituted anilines as main products. Besides monobromo substrates, the substrates bearing dibromo substituents were synthesized and subjected to the reaction conditions. **3d** exhibited reactivity at α-position of ester group instead of primary bromo site (**4y**). When multiple α-positions of ester group were present in the polybromi-



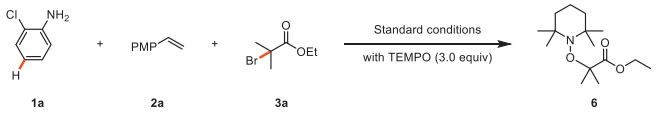
Scheme 4. Structure scope of α-alkyl bromides and alkenes. Standard conditions: **1a** (0.6 mmol, 3 equiv.), **2** (0.6 mmol, 3 equiv.), **3** (0.2 mmol, 1 equiv.), $[RuCl_2(p\text{-cymene})]_2$ (5 mol%), PPh_3 (40 mol%), $CsHCO_3$ (2.5 equiv.), DMF (1.0 mL), 60 °C, 8 h, under Ar. Isolated yields. PMP = *para*-methoxyphenyl.

nated substrate, only monosubstituted products were obtained under our reaction conditions (**4z**).

1,1-Diarylalkyl moiety is important building blocks in natural products and drugs [76]. Switching different 1,1-diarylalkyl moieties might improve the physicochemical properties *via* regulating the steric configuration, hydrophobicity, hydrogen bonding, *etc.* [77–79]. The robustness of this protocol was further demonstrated by the successful late-stage functionalization of α–bromo esters derived from complex pharmaceuticals and natural products by introducing the 1,1-diarylalkyl moieties (Scheme 5). The scope of the reaction was striking: α–bromo esters containing piperonyl alcohol (**4aa**), terpenoid (**4ab**, **4ad** and **4af**), sesquiterpenoid (**4ae**), steroid (**4ac** and **4ag**), vitamin E (**4ai**), or glucose derivatives (**4ah** and **4aj**) were shown to be compatible with our system. More specifically, sensitive functional groups, such as ethers (**4aa**), ketone (**4ac**), alkenes (**4ae** and **4ag**), ketal (**4aa**, **4ah**, **4aj**) and aryl group (**4ai**) were retained. Additionally, the structures of products **4** were explicitly established by single-crystal X-ray diffraction analyses of representative **4ad** (CCDC number: 2441323). Notably, the protocol also leaves the intact amino handle for further conversion of these nature product derivatives to antibody drug conjugate similar to previous reports [80].

To elucidate the radical generation of this reaction, we carried out a series free radical trapping experiments. In the presence of 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO), the reaction was completely inhibited and a radical adduct **6** of **3a** and TEMPO was obtained (Table 2, entry 1), indicating that the reaction involved a radical process. The absence of **1a** led to less production of **6** (entry 2). According to previous literatures [46,48–51,67,69,73,81–86], styrene is not readily coordinate with Ru in this σ-activation reaction. Indeed, the yield of **6** is not decreased without styrene **2a** (entry 3). Notably, **6** was not generated in the absence of PPh_3 or Ru (entries 4 and 5), highlighting PPh_3 and Ru played a crucial role in the production of free radicals. Thus, we considered that Ru and

Table 2
Radical-trapping experiments of three-component reaction.[a]

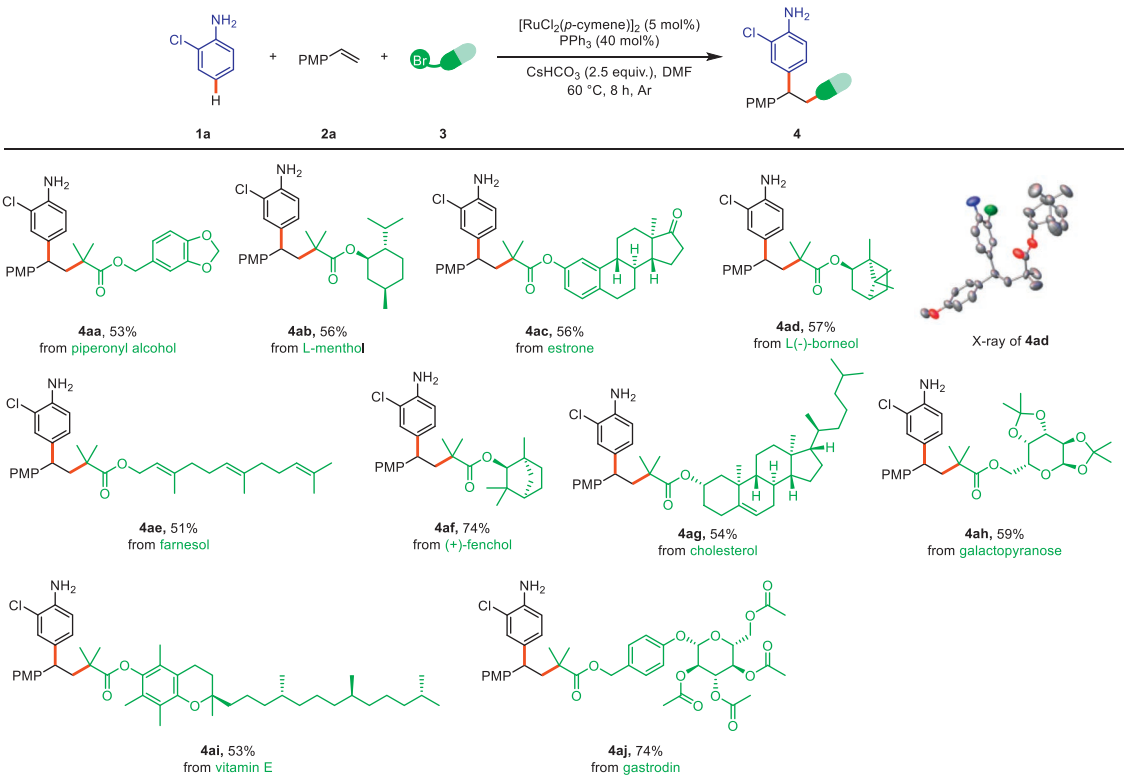


Entry	Deviation from standard conditions with TEMPO	**6** (%)
1	None	25
2	Without **1a**	11
3	Without **2a**	25
4	Without PPh_3	NR
5	Without $[RuCl_2(p\text{-cymene})]_2$	NR
6	10 mol% $[RuCl_2(PPh_3)_3]$ replace $[RuCl_2(p\text{-cymene})]_2$	31

[a] Standard conditions: **1a** (0.6 mmol, 3 equiv.), **2a** (0.6 mmol, 3 equiv.), **3a** (0.2 mmol, 1 equiv.), $[RuCl_2(p\text{-cymene})]_2$ (5 mol%), PPh_3 (40 mol%), $CsHCO_3$ (2.5 equiv.), DMF (1.0 mL), 60 °C, 8 h, under Ar. Isolated yields. PMP = *para*-methoxyphenyl.

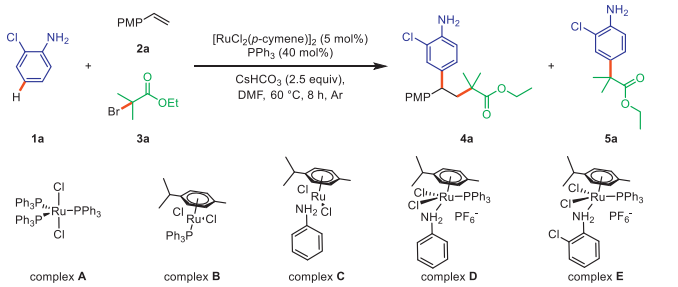
PPh_3 would form a complex which is responsible for reduction of alkyl bromide **3a** to generate corresponding radical. Furthermore, complex **A**, $RuCl_2(PPh_3)_3$, which is readily generated from $RuCl_3$ and PPh_3 proved more effective in radical generation (entry 6), which is consistent with our optimal results in Table 1 (entry 5). It also suggests *p*-cymene might not be involved in the catalytic Ru species as ligands.

According to literatures [87–89], C-H functionalization using $[RuCl_2(p\text{-cymene})]_2$ normally undergoes two pathways: (1) *p*-cymene dissociates from $[RuCl_2(p\text{-cymene})]_2$ and remaining Ru complex coordinate with substrates [90–95]; (2) $[RuCl_2(p\text{-cymene})]_2$ directly coordinates with substrates [67,69,82–84,86]. To further identify the catalytic Ru species generated from $[RuCl_2(p\text{-cymene})]_2$, a series of ruthenium complexes including complexes **B**-**E** with *p*-cymene were synthesized to compare with complex **A**, $RuCl_2(PPh_3)_3$ (Table 3, and Supporting information for de-



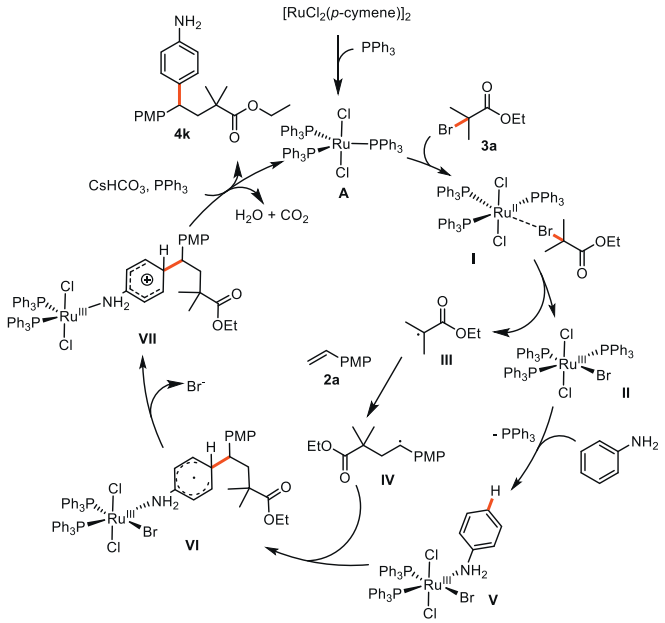
Scheme 5. Reaction with natural products and drugs. Standard conditions: **1a** (0.6 mmol, 3 equiv.), **2a** (0.6 mmol, 3 equiv.), **3** (0.2 mmol, 1 equiv.), $[RuCl_2(p\text{-cymene})]_2$ (5 mol%), PPh_3 (40mol%), $CsHCO_3$ (2.5 equiv.), DMF (1.0 mL), 60 °C, 8 h, under Ar. Isolated yields. PMP = *para*-methoxyphenyl.

Table 3
Control expenriments.[a]



Entry	Catalyst	PPh_3 (mol%)	**4a** (%)	**5** (%)
1	$[RuCl_2(p\text{-cymene})]_2$ [b]	40	70	25
2	Complex **B**	-	34	20
3	Complex **C**	-	NR	NR
4	Complex **D**	-	17	NR
5	Complex **E**	-	11	NR
6	Complex **A**	-	67	29
7	Complex **B**	30	54	13
8	Complex **C**	30	58	13
9	Complex **E**	30	29	NR

[a] Reaction conditions: **1a** (0.6 mmol, 3 equiv.), **2a** (0.6 mmol, 3 equiv.), **3a** (0.2 mmol, 1 equiv.), catalyst (10 mol%), PPh_3 (40 mol%), $CsHCO_3$ (2.5 equiv.), DMF (1.0 mL), 60 °C, 8 h, under Ar. Isolated yields.
[b] 5 mol% $[RuCl_2(p\text{-cymene})]_2$ was used. PMP = *para*-methoxyphenyl.



Scheme 6. Plausible catalytic cycle.

tail of their synthesis). When subjecting to the reaction without additional PPh_3, complexes **B**-**E** exhibited poor catalytic efficiency (entries 2–5). Despite the increase of the yield of **4a** by **B** and **C** after the addition of PPh_3 (entries 7 and 8), the complex **A**, $RuCl_2(PPh_3)_3$, without additional PPh_3 almost afforded the same reaction yield to the optimal conditions (entry 6). Therefore, complex **A** without *p*-cymene was more likely to be a catalytic species in the reaction process, which is transformed from $[RuCl_2(p\text{-cymene})]_2$. Notably, similar dissociation of *p*-cymene from $[RuCl_2(p\text{-cymene})]_2$ was observed in many visible-light mediated processes [90–95].

Based on these results and previous literatures [67], a plausible mechanism for this three-component reaction was proposed, as illustrated in Scheme 6. The reaction initiates with the dissoci-

ation of *p*-cymene to form complex **A**. Subsequently, **A** reacts with alkyl halide **3a** to generate intermediate **I**, which is further converted to alkyl radical **III** and a ruthenium(III) intermediate **II** *via* ligand-to-metal charge transfer (LMCT) processes. The alkyl radical **III** promptly adds to alkene **2a**, resulting in the formation of benzyl radical **IV**. Meanwhile, the ruthenium(III) intermediate **II** coordinates with aniline to form complex **V**. The benzyl radical **IV** attacks the aromatic moiety *para* to the ruthenium center, leading to the formation of intermediate **VI**. This intermediate undergoes a LMCT process to yield complex **VII**. Finally, rearomatization of **VII** occurs, releasing the difunctionalization product **4k** and regenerating the catalytically active ruthenium(II) species **A**.

In summary, we report a mild ruthenium-catalyzed three-component reaction enabling alkylarylation of alkenes *via para*-selective C-H activation of unprotected anilines. Substrate scope encompasses electron-rich/depleted anilines, hindered α–bromo derivatives, and styrenes, with late-stage functionalization for complex natural products. Mechanistic studies reveal a dissociation of *p*-cymene during the catalytic processes, which is distinct from most previous σ-activation pathways. The method circumvents traditional limitations (*e.g.*, protecting groups, harsh conditions) and offers applicability in pharmaceutical and natural product modification.

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.

CRediT authorship contribution statement

Xin-Juan Wang: Writing – review & editing, Writing – original draft, Investigation. **Yao-Hang Cheng:** Investigation. **Xin-Yan Lv:** Investigation. **Li-Ye Liang:** Investigation. **Guang-Hui An:** Writing – review & editing, Writing – original draft, Supervision, Methodology, Investigation, Funding acquisition, Conceptualization. **Guang-Ming Li:** Supervision, Investigation.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2025.112154.

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