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Transition-metal-catalyzed remote *meta*-C–H alkylation and alkynylation of aryl sulfonic acids enabled by an indolyl template

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ABSTRACT

Transition-metal-catalyzed remote sp^2C–H functionalization of aryl sulfonic acids was hardly ever realized owing to competitive *ortho*-C–H functionalization of aryl sulfonates and electron-deficient nature of phenyl ring. Herein, with the assistance of a practical biaryl indolyl directing template, palladium-catalyzed remote sp^2C–H alkylation of aryl sulfonic acids have been achieved in moderate to good yields with exclusive *meta* selectivity. Moreover, remote *meta*-selective C–H alkynylation of aryl sulfonic acids was also accomplished with a rhodium catalyst. These *meta*-C–H functionalized products proved to be the superior synthetic precursors, which are difficult to access using the conventional strategy.

Transition-metal-catalyzed C–H functionalization has proved a powerful synthetic strategy that converts inert C–H bonds into a diverse range of C–C and C–heteroatom bonds [1]. One of the significant challenges for this field is to achieve the site selectivity of C–H activation, *i.e.,* selective recognition of a target C–H bond where many enthalpically and entropically equivalent C–H bonds coexists in the molecules [2]. Directing group (DG)-assisted transition-metal-catalyzed C–H activation proved a successful vehicle for this process [3–15]. By means of proximity-driven cyclometalation process, activation of *ortho*-C–H bonds in the benzene rings *via* a conformationally rigid five- or six-membered metallacycle transition state (TS) was accomplished comprehensively [3,6,10,16–19]. In sharp contrast, transition-metal-catalyzed distal C–H bond functionalization in arenes still remains an enormous challenge [20–25]. The pioneering work by Yu provided a breakthrough solution in regard to this question [26]. Through using a nitrile or *N*-heterocycle, such as pyridine and pyrimidine, embedded in the template that serves as an end-on directing group, distal C–H bonds that locate at the *meta* or *para* position to the existing functional group in the arenes are successfully recognized by tuning the spatial and geometric parameters of directing templates [22,27–56]. These directing templates were commonly attached to the substrate by an amide or ester linkage, accommodating the substrate and catalyst to assemble a macrocyclophane (MCP) pre-transition state in the C–H activation step.

Despite these progresses, the remote *meta*-C–H functionalization of conjugated aryl sulfonic acids substantially suffers from the substrate-imposed challenges to date. The inherently directed *ortho*-C–H functionalization by the sulfonate group, *e.g.,* the sulfonamide group, is shown to be the mainly competitive reaction with regard to the target *meta*-C–H activation. The electron-deficient characteristic of the phenyl ring further retards the sp^2C–H metalation process significantly. Moreover, the contiguous sulfur(VI) center makes the whole molecule far more rigid than the related long-chain congeners, not beneficial to the assembly of MCP pre-transition state.

In light of the ubiquity of aryl sulfonic acids and the related sulfonamides as the key structural units in pharmaceuticals, agrochemicals and fine chemicals (Fig. 1a), synthesis and modification of aryl sulfonates by selective C–H functionalization appear particularly attractive. Different from transition-metal-catalyzed *ortho*-C–H functionalization of aryl sulfonate substrates (Fig. 1b) [57–73], direct *meta*-C–H bond functionalization of aryl sulfonate derivatives still remains rarely explored owing to the aforementioned limitations. Based on a recent statistical analysis [74] on the relationship between the site-selectivity and MCP-like pre-transition state, our research group, in collaborated with Yu's group, have successfully developed a palladium-catalyzed inter-

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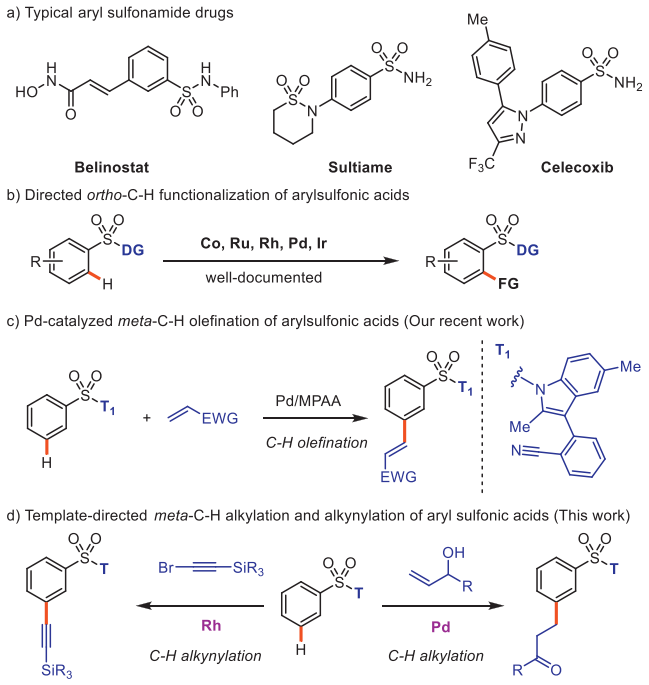


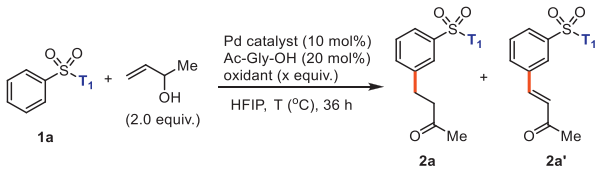
Fig. 1. Transition-metal-catalyzed sp^2C–H functionalization of aryl sulfonic acids.

and intramolecular *meta*-selective C–H olefination reactions of aryl sulfonic acids using an indole-derived nitrile directing template (**T_1**) very recently (Fig. 1c) [75].

To the best of our knowledge, the alkyl-substituted arenes as the core units have been widely applied for active pharmaceutical ingredients, fine chemicals, and material science. The alkylation reaction of arenes is an extremely important chemical transformation in organic synthesis. Direct alkylation reaction of aryl sulfonates, such as Friedel–Crafts reaction or radical reaction, often suffers from low reactivity, poor site selectivity, and limited substrate scope [76]. Therefore, we envisioned the incorporation of an alkyl side chain into aryl sulfonic acid *via* remote *meta*-selective C–H functionalization by using the aforementioned indolyl directing template. Herein, we disclosed a palladium-catalyzed *meta*-selective sp^2C–H alkylation of aryl sulfonic acid using the allyl alcohols as an alkylation agents (Fig. 1d). In addition, with the assistance of this type of directing template, rhodium-catalyzed *meta*-C–H alkynylation of aryl sulfonic acids was also demonstrated to be viable. The introduced alkynyl groups were shown to be readily converted into the diverse substituents, for example, the alkyl, alkenyl, acyl groups.

At outset, to achieve *meta*-C–H alkylation of aryl sulfonic acids using the established directing template, various reaction parameters including catalyst, oxidant, temperature, and time, *etc.*, were thus optimized with but–3-en-2-ol as an alkylated reagent (Table 1). Palladium pivalate and silver nitrate were demonstrated to be the optimal catalyst and oxidant, respectively. $AgNO_3$ was indispensable for this transformation and the catalytic process was almost halted when solely using 1,4-benzoquinone (2 equiv.) in the presence of O_2 (1 atm) as the oxidant (Table 1, entry 16). However, excess silver nitrate (2.5 equiv.) caused the production of olefination product **2a′** (Table 1, entry 4), which were probably resulted from coupling with methyl vinyl ketones produced by oxidation of the allyl alcohols. Ultimately, with palladium pivalate as the catalyst, the use of a combination of silver nitrate (0.5 equiv.), 1,4-benzoquinone (2 equiv.) and molecular oxygen (1 atm) as the oxidant gave the highest yield (Table 1, entry 14). Notably, palladium-catalyzed C–H alkylation reaction gave the desired products **2a** with exclusive *meta* selectivity (generally *meta*:others > 20:1) using the directing template (**T_1**).

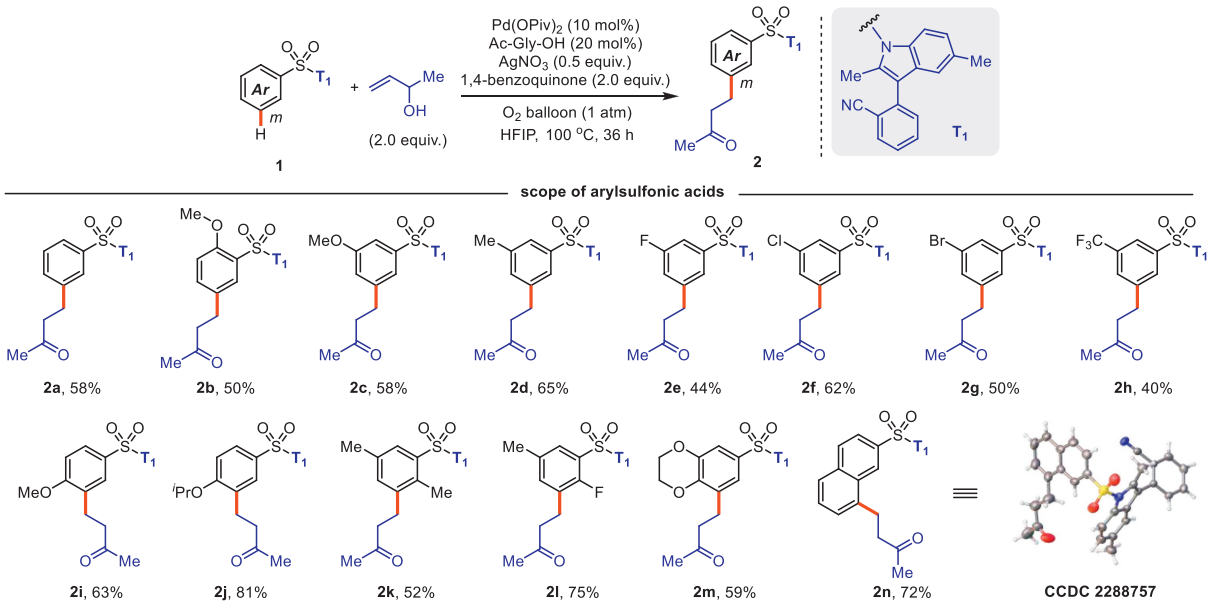
Table 1
Screening of reaction conditions for Pd-catalyzed *meta*-C–H alkylation of aryl sulfonic acids.[a]



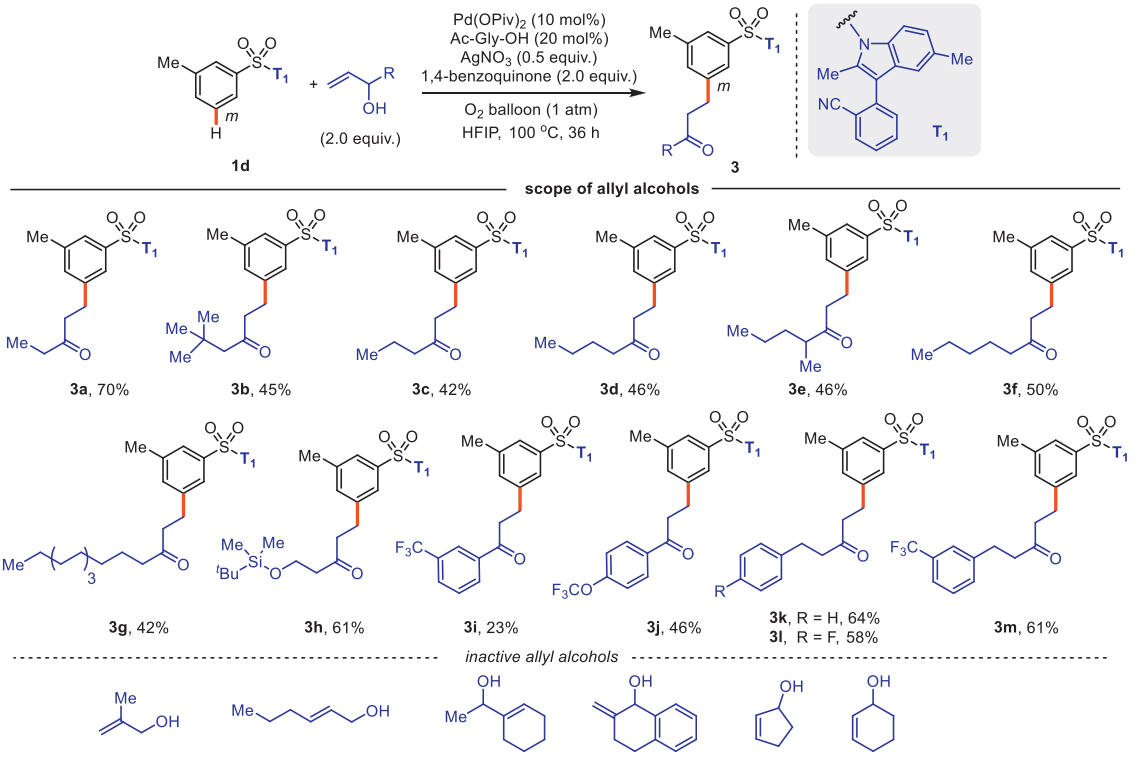
Entry	Pd catalyst	Oxidant (equiv.)	Temp (°C)	Yield of **2a**/**2a'** (%)
1	$Pd_2(dba)_3$	$AgNO_3$ (2.5)	100	36/4
2	$Pd(OAc)_2$	$AgNO_3$ (2.5)	100	25/4
3	$Pd(PPh_3)_2Cl_2$	$AgNO_3$ (2.5)	100	20/4
4	$Pd(OPiv)_2$	$AgNO_3$ (2.5)	100	40/8
5	$Pd(dppf)Cl_2$	$AgNO_3$ (2.5)	100	n.r.
6	$Pd(OPiv)_2$	$AgNO_3$ (2.5)	90	30/4
7	$Pd(OPiv)_2$	$AgNO_3$ (2.5)	110	36/8
8	$Pd(OPiv)_2$	$AgNO_3$ (1.5)	100	48/6
9	$Pd(OPiv)_2$	Ag_2CO_3 (1.5)	100	38/24
10	$Pd(OPiv)_2$	AgOAc (1.5)	100	19/14
11	$Pd(OPiv)_2$	$PhCO_2Ag$ (1.5)	100	24/12
12	$Pd(OPiv)_2$	$AgNO_3$ (0.5)[b]	100	40/10
13	$Pd(OPiv)_2$	$AgNO_3$ (0.5)[c]	100	32/12
14	$Pd(OPiv)_2$	$AgNO_3$ (0.5)[d]	100	58/6
15	$Pd(OPiv)_2$	$AgNO_3$ (0.25)[d]	100	24/2
16	$Pd(OPiv)_2$	–[e]	100	12/n.d.

[a] Reaction conditions: $PhSO_2$-**T_1** (0.1 mmol), but–3-en-2-ol (0.2 mmol), Pd catalyst (10 mmol%), Ac-Gly-OH (20 mmol%), oxidant (*x* equiv.), isolated yield was reported. n.r.: no reaction. n.d.: not detected.
[b] In the presence of O_2 (1 atm).
[c] 1,4-benzoquinone (0.2 mmol) was used.
[d] 1,4-benzoquinone (0.2 mmol) was used in the presence of O_2 (1 atm).
[e] Without $AgNO_3$, only 1,4-benzoquinone (0.2 mmol) in the presence of O_2 (1 atm) as the oxidant.

The scope of aryl sulfonic acids in palladium-catalyzed *meta*-selective C–H alkylation reaction was subsequently surveyed under the optimal reaction conditions (Scheme 1). With but–3-en-2-ol as the coupling partner, electron-donating substituents including the methoxy, methyl, and isopropanoxyl groups in aryl sulfonates were generally conducive to the C–H alkylation reaction, providing the desired *meta* alkylated products (**2c**, **2i**, **2d** and **2j**) in remarkably higher yields (58%–81%). Reaction with substrate bearing an *o*-OMe group delivered the product (**2b**) in slightly decreased yield (50%), probably due to the unfavored conformation caused by the steric repulsion between the methoxy group and tetrahedrally coordinated sulfonate group. The halo-substituted aryl sulfonate substrates were suitable for the protocol, giving the *meta*-alkylated products (**2e–2g**) in moderate yields (44%–62%). In particular, the product (**2g**) owning a bromo group was tolerated in this reaction, which is synthetically useful for further derivation of other functional groups. The presence of intrinsic sulfonic group makes the phenyl ring of aryl sulfonates highly electron-deficient. Incorporation of the additional electron-withdrawing functional groups into aryl sulfonates further augmented this trend, obviously not conducive to the C–H activation process. However, different from remote *meta*-C–H functionalization of benzoic acids [51,52], aryl sulfonates bearing a diverse set of electron-withdrawing groups, *e.g.*, CF_3 group, could still deliver the alkylated product (**2h**), albeit in lower yield (40%). Di-substituted aryl sulfonates in different substitution patterns were able to afford the *meta*-alkylated products (**2k–2m**) in 52%–75% yields. In addition, reaction with 2-naphthalenesulfonate is also feasible to afford the desired product in 72% yield, and C–H alkylation selectively occurred at the C_8 position, which was comprehensively confirmed by X-ray diffraction



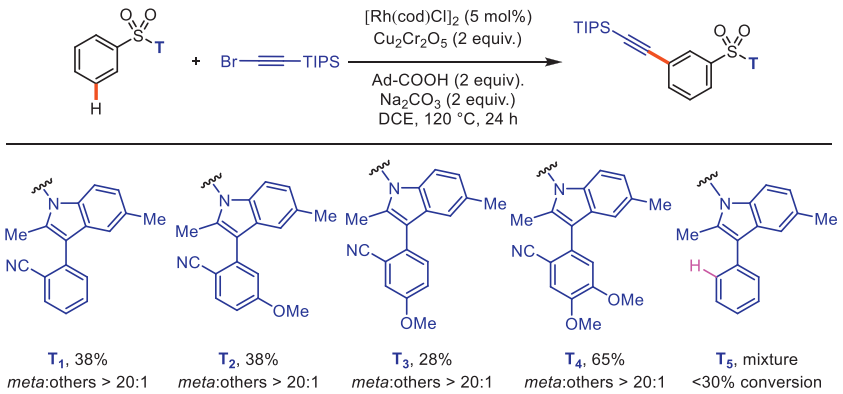
Scheme 1. Scope of aryl sulfonic acids for palladium-catalyzed *meta*-C–H alkylation. Reaction conditions: $ArSO_2$-**T_1** (0.1 mmol), but–3-en-2-ol (0.2 mmol), $Pd(OPiv)_2$ (10 mol%), Ac-Gly-OH (20 mol%), $AgNO_3$ (0.05 mmol), 1,4-benzoquinone (0.2 mmol), O_2 balloon (1 atm), HFIP (1.5 mL), 100 °C, 36 h. Isolated yields were reported.



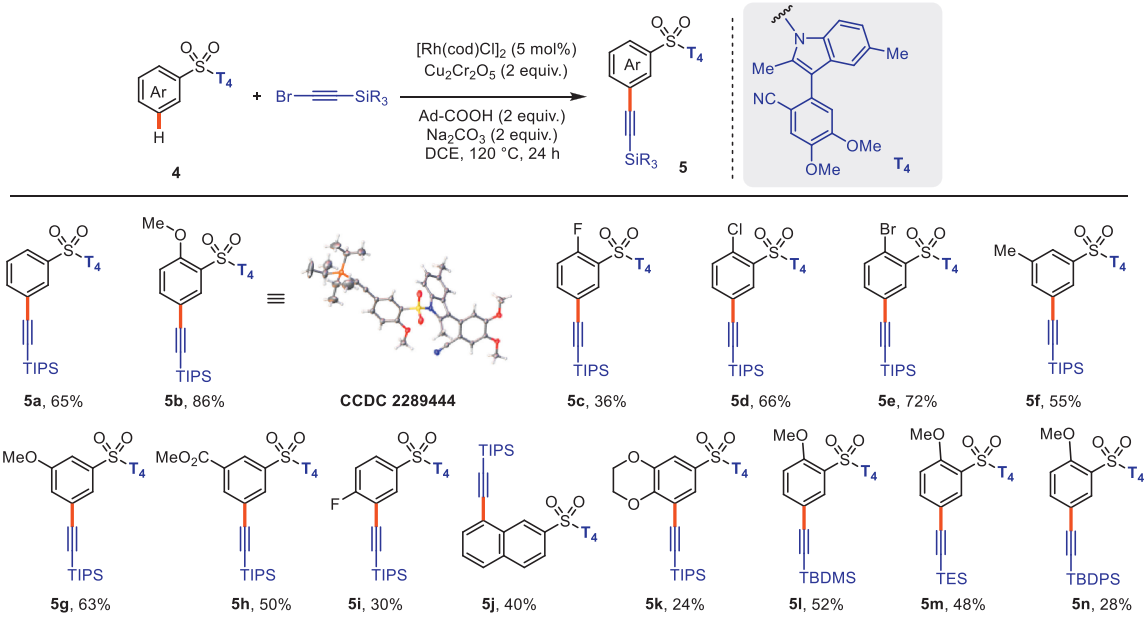
Scheme 2. Scope of allyl alcohols for palladium-catalyzed *meta*-C–H alkylation. Reaction conditions: $ArSO_2$-**T_1** 1d (0.1 mmol), allyl alcohol (0.2 mmol), $Pd(OPiv)_2$ (10 mol%), Ac-Gly-OH (20 mol%), $AgNO_3$ (0.05 mmol), 1,4-benzoquinone (0.2 mmol), O_2 balloon (1 atm), HFIP (1.5 mL), 100 °C, 36 h. Isolated yields were reported.

analysis of product **2n**. Notably, with the assistance of template **T_1**, exclusive *meta* selectivity (generally *meta*:others > 20:1) were observed with all of the substrates outlined in Scheme 1.

Next, we further investigated the compatibility of template-directed *meta*-C–H alkylation of aryl sulfonates with the diverse allyl alcohols (Scheme 2). The protocol revealed a widely synthetic utility, tolerating a range of allyl alcohols bearing various alkyl chains. Commonly, the desired products (**3a–3g**) were isolated in 42%–70% yields. Notably, allyl alcohol containing a terminal TBDMS-protected ether group was also suitable alkylated agent in this protocol, providing the *β*–hydroxy ketoneproduct (**3h**) in 61% yield. This allows for synthesis of selectively mono-protected 1,3-diol derivatives from product **3h**. Moreover, *meta*-C–H alkylation with 1-phenylprop-2-en-3-ol is also feasible, successfully affording the aromatic ketones (**3i** and **3j**). The terminal aryl groups in the allyl alcohol chains were well tolerated, successfully delivering the aliphatic ketones (**3k–3m**) in 58%–64% yields. As observed in Scheme 1, C–H alkylation of *m*-methyl benzenesulfonic acid-



Scheme 3. Screening of directing template for rhodium-catalyzed *meta*-C–H alkynylation. Reaction conditions: $ArSO_2$-**T** (0.1 mmol), (bromoethynyl)triisopropylsilane (0.6 mmol), $[Rh(cod)Cl]_2$ (5 mol%), $Cu_2Cr_2O_5$ (0.2 mmol), 1-Ad-COOH (0.2 mmol), Na_2CO_3 (0.2 mmol), DCE (1.5 mL), 120 °C, 24 h. Isolated yields were reported.



Scheme 4. Substrate scope for rhodium-catalyzed *meta*-C–H alkynylation. Reaction conditions: $ArSO_2$-$\mathbf{T_4}$ (0.1 mmol), bromoacetylene (0.6 mmol), $[Rh(cod)Cl]_2$ (5 mol%), $Cu_2Cr_2O_5$ (0.2 mmol), 1-Ad-COOH (0.2 mmol), Na_2CO_3 (0.2 mmol), DCE (1.5 mL), 120 °C, 24 h. Isolated yields were reported.

derived substrate **1d** with the different allyl alcohols also gave the desired products with exclusive *meta* selectivity. Unfortunately, this protocol is incompatible with the cyclic allyl alcohols.

Alkynes are among the most useful motifs in bioactive and material molecules [61]. The alkynyl group is also one of the most valuable synthons in synthetic organic chemistry [21]. Encouraged by our success in template-directed *meta*-C–H alkylation, we further embarked on transition-metal-catalyzed *meta*-C–H alkynylation of aryl sulfonates. Through using (bromoethynyl)triisopropylsilane as the alkynylated reagent, reaction conditions of template-directed *meta*-selective C–H alkynylation of aryl sulfonic acids were extensively evaluated (For reaction condition optimization, see Supporting information for details). Pleasingly, with $\mathbf{T_1}$ as the directing template, rhodium(I)-catalyzed C–H alkynylation successfully afforded the desired products in 38% yield under the optimal conditions. Notably, the presence of 1-Ad-CO_2H significantly accelerated the C–H activation/deprotonation process as observed previously by L. Ackermann [3,77]. Moreover, the electronic density of benzene ring significantly influences the coordination potential between the nitrile group and rhodium catalyst. As a consequence, with $\mathbf{T_4}$ as a directing template, the yield of alkynylation reaction drastically increased to 65% (Scheme 3).

With the optimal template ($\mathbf{T_4}$) in hand, we subsequently investigated the generality of rhodium-catalyzed *meta*-selective C–H alkynylation. It was found that substrates owning the methoxy and halo functional groups at *ortho* position were well tolerated under the present C–H alkynylation reaction conditions, providing the desired *meta* alkynylated products (**5b**–**5e**) in modest to good yields. The site-selectivity of C–H alkynylation was comprehensively confirmed by X-ray diffraction analysis of compound **5b**. Aryl sulfonamides bearing a diverse set of electron-withdrawing or electron-donating substituents, such as methoxy, methyl, and ester group at *meta* position, are all compatible to provide the target products (**5f–5h**) in 50%–63% yields. Additionally, C–H alkynylation reaction with 2-naphthalenesulfonate-derived substrate is also feasible, occurring at C_8-position (**5j**) selectively. Except for (bromoethynyl)triisopropylsilane, (bromoethynyl)(*tert*–butyl)dimethylsilane, (bromoethynyl)triethylsilane, and (bromoethynyl)(*tert*–butyl)diphenylsilane were tolerated under the present reaction conditions, producing the corresponding

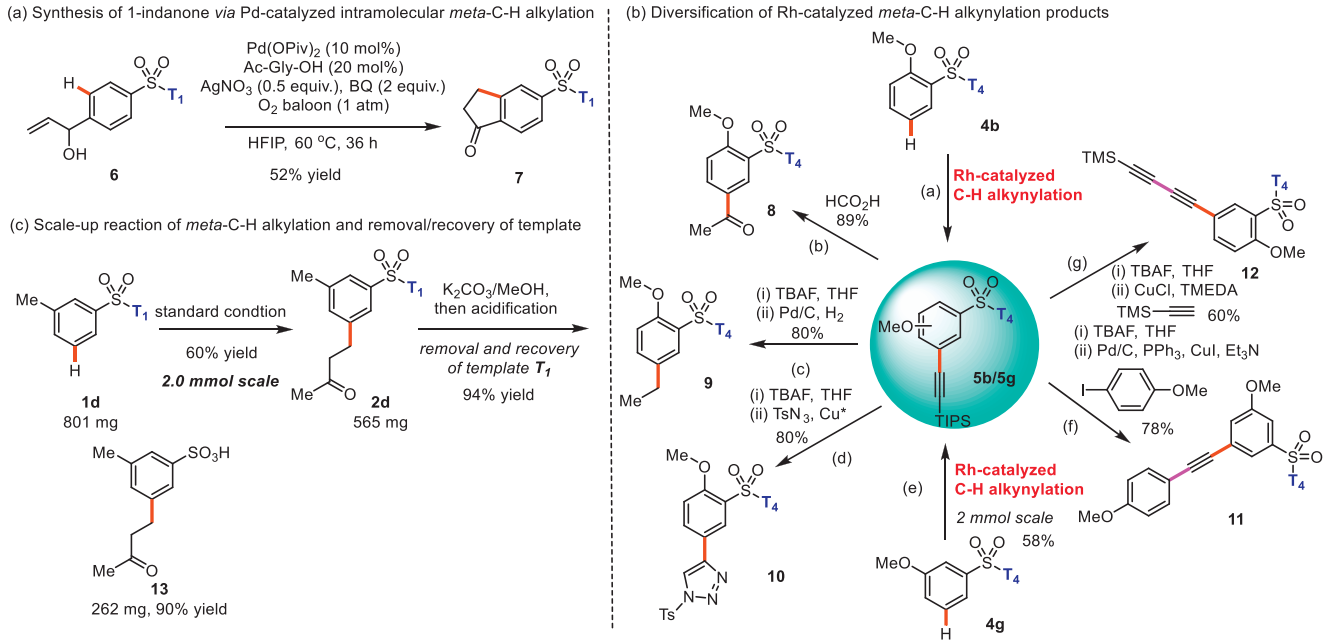


Fig. 2. Synthetic application of transition-metal-catalyzed *meta*-C–H functionalization of aryl sulfonic acids.

alkynylated products (**5l**–**5n**) in 28%–52% yields (Scheme 4). As validated by the aforementioned X-ray diffraction analysis, rhodium-catalyzed C–H alkynylation of benzenesulfonic acids also afforded the corresponding products **5a**–**5n** in exclusive *meta*-selectivity (generally *meta*:others > 20:1).

As the vital drug moiety, 1-indanones were widely served as a type of intermediates to synthesize anti-inflammatory, anti-malarial, anti-tumor, anti-viral and other bioactive drugs, such as Indinavir and Paucifloral F(III). Access to the substituted 1-indanone framework *via* intramolecular remote *meta*-C–H functionalization has not been achieved previously. To our delight, Pd-catalyzed intramolecular *meta*-C–H alkylation successfully afforded 5-sulfonate group-substituted 1-indanone **7** in 52% isolated yield with the present directing template $\mathbf{T_1}$ (Fig. 2a). Additionally, the introduced *meta* alkynyl groups in aryl sulfonates were conveniently converted into the various functional groups, further demonstrating the synthetic utility of the present remote *meta*-C–H functionalization strategy (Fig. 2b).

Finally, we successfully scaled up the *meta*-C–H reaction to 2.0 mmol scale (Fig. 2c). Palladium-catalyzed *meta*-C–H alkylation of aryl sulfonate **1d** provided the target mono-product **2d** in 60% isolated yield. The template $\mathbf{T_1}$ was easily removed under mild conditions (K_2CO_3, MeOH) and recovered in 94% yield, together with the desired *m*-alkylated arylsulfonic acid **13** in 90% yield.

In summary, we have successfully achieved transition-metal-catalyzed *meta*-selective C–H alkylation and alkynylation reactions of aryl sulfonic acids by means of a practical indolyl directing template developed by us very recently. Synthetic applications of the strategy were demonstrated in the synthesis of significant intermediate 1-indanone *via* a palladium-catalyzed intramolecular *meta*-C–H alkylation process. Further investigation on this template-assisted *meta*-C–H functionalization is underway in our laboratory.

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.

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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.2023.109361.

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