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Base-Mediated O-Arylation of Alcohols and Phenols by Triarylsulfonium Triflates

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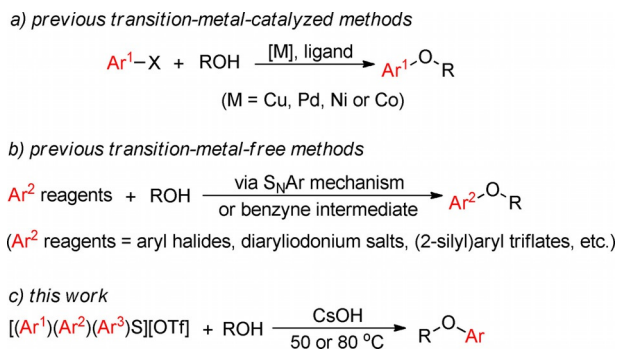
Abstract: A mild and efficient protocol for O-arylation of alcohols and phenols (ROH) by triarylsulfonium triflates was developed under transition-metal-free conditions. Various alcohols, including primary, secondary and tertiary, and phenols bearing either electron-donating or electron-withdrawing groups on the aryl rings were smoothly converted to form the corresponding aromatic ethers in moderate to excellent yields. The reactions were conducted at 50 or 80 °C for 24 h in the presence of a certain base and showed good functional group tolerance. The base-mediated arylation with asymmetric triarylsulfonium salts could selectively transfer the aryl groups of sulfoniums to ROH, depending on their inherent electronic nature. The mechanistic studies revealed that the reaction might proceed through the nucleophilic attack of the in situ formed alkoxy or phenoxy anions at the aromatic carbon atoms of the C–S bonds of triarylsulfonium cations to furnish the target products.

Introduction

Aromatic ethers represent an important class of structural motifs that are widely present in natural products, pharmaceuticals, agrochemicals, and functional materials.[1] A large number of synthetic routes to aromatic ethers have been developed under transition-metal-catalyzed or -free conditions (Scheme 1).[2–6] The Cu-, Pd-, Ni- and Co-catalyzed reactions of phenols/alcohols with (hetero)aryl halides or arenes have proved to be the most effective approaches to construct aromatic ethers, some of which, however, suffered from narrow substrate scopes, high reaction temperatures, large catalyst loadings, expensive ligands (especially when the sterically hindered alcohols were coupled with the aryl halides), need for directing groups (arenes), and/or use of large excess of oxidants (arenes).[2–5] As a result, the transition-metal-free O-arylations of alcohols/phenols with aryl halides, diaryliodonium salts, *o*-silylaryl triflates and arenes via S_NAr pathways, aryne intermediates or photocatalytic alkoxylation have been harnessed.[6] Although these strategies are attractive in the fields of applications where the heavy metal residuals in products need to be strictly controlled (e.g. the pharmaceutical industry), the low efficiency, the poor regioselectivity and/or the harsh reaction conditions significantly limit the uses of these transition-metal-free methods in practice.



Scheme 1. Transition-metal-catalyzed or -free synthesis of aromatic ethers from alcohols and phenols.

On the other hand, arylsulfonium salts have been confirmed as versatile reagents in organic synthesis.[7] Both triaryl- and alkylarylsulfonium salts are useful cross-coupling partners in the palladium-catalyzed reactions, including Suzuki, Heck, Sonogashira, Negishi, sulfonylation, carbonylation, and borylation reactions.[8] The photoredox catalysis enables the homolytic cleavages of the C-S bonds and couplings of triarylsulfonium salts ($[Ar_3S]X$, X = OTf, BF_4, PF_6, etc.) with alkenes, halides, trifluoromethylthiolate, triphenylphosphite, and cyanide at ambient temperature through aryl radical intermediates to form complex small molecules.[9] The transition-metal-free reactions of $[Ar_3S][OTf]$ with different nucleophiles (e.g. amines, fluorides) provides direct access to a variety of important arylated compounds.[10] All these achievements have verified the great usefulness of arylsulfonium salts in the carbon-carbon and carbon-heteroatom bonds formation reactions. Despite the fact that transition-metal-free alkaline decomposition of triarylsulfonium salts gave diverse products via aromatic nucleophilic substitution, aryne intermediates, or radical processes, which were dependent upon the nature of bases and anions of the sulfonium salts,[11] the synthetic utility of these transformations was not recognized, possibly owing to the complicated product mix-

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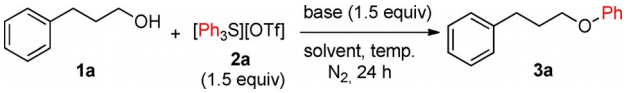
tures generated from the reported reactions. As a class of promising aryl transfer sources, triarylsulfonium salts have featured several advantages such as non-volatility, easy preparation, non-toxicity, modest reactivity, good thermal stability, and broad structural diversity. In this work, we disclosed a transition-metal-free synthesis of aromatic ethers from alcohols and phenols using triarylsulfonium triflates as arylation reagents (Scheme 1 c). In contrast to the elusive reactions with triarylsulfonium halides and the much strong bases,[11] our new method using triarylsulfonium triflates together with the weaker bases made the reactions synthetically useful, which expanded the substrate scope and accomplished good selectivity, high efficiency, and excellent functional groups tolerance.

Results and Discussion

As described in Table 1, we initially tested the reaction of 3-phenylpropanol (**1a**) with triphenylsulfonium triflate (**2a**, 1.5 equiv) and CsOH (1.5 equiv) in toluene at 80 °C under a nitrogen atmosphere for 24 h, which gave 3-phenylpropyl phenyl ether (**3a**) in 95% yield (entry 1). Further investigation showed that the choice of bases had a considerable impact on the O-phenylation. When CsOH was replaced by KOH, NaOH, and LiOH in the same reaction, **3a** was formed in 85%, 71%, and <1% yields, respectively (entries 2–4), suggesting that the alkali metal cations of the hydroxides greatly affected the conversion. NaH had a comparable effect as CsOH, affording **3a** in 93% yield (entry 5). *t*BuOK was unexpectedly less effective, providing **3a** in 66% yield (entry 6). It was noteworthy that the relatively weak bases such as Cs_2CO_3, K_3PO_4 and CsF were also amenable to the reaction, which gave **3a** in 37–64% yields (entries 7–9). Other bases such as K_2CO_3, Na_2CO_3, DBU, and NEt_3 were almost totally ineffective, yielding **3a** in only trace amounts (see the supporting information). It should be mentioned that reaction of **1a** and **2a** in toluene without base at 80 °C for 24 h gave no desired product (entry 10). Furthermore, a survey of solvents for the reactions of **1a**, **2a** and CsOH at 80 °C or 60 °C showed that *m*-xylene, THF, 1,4-dioxane, and dichloromethane were similarly effective as toluene and that CH_3CN, NMP, DMF, and DMSO were less effective than toluene (entries 11–12 and Table S2). The reaction temperature also had an influence on the reaction, the tendency of which was varied by changing the solvents (Table S3). Consequently, reaction of **1a** with **2a** (1.5 equiv) and CsOH (1.5 equiv) in toluene at 100 °C for 24 h or in 1,4-dioxane at 50 °C for 24 h gave **3a** in >99% or 97% yield as the optimal results (entry 13 or 14). Besides, the molar ratios of reactants interestingly impacted the preparation of **3a** (Table S6). Deviation of the molar ratio of **1a**/ **2a**/ CsOH from 1:1.5:1.5 to 2:1:1, 1.2:1:1, 1:1:0.5, 1:1:1, 1:1:1.2, 1:1.2:1, 1:1.2:1.5, and 1:1.5:2 in the reactions of **1a**, **2a**, and CsOH in 1,4-dioxane at 50 °C for 24 h led to **3a** in 34%, 77%, 33%, 72%, 85%, 74%, 89% and 90% yields, respectively. The data indicated that the equivalents of CsOH relative to **1a** might have a great effect on the reaction and that a proper combination of excess **2a** and CsOH benefited the production of **3a**. In addition, reaction of **1a**, **2a** (1.5 equiv), and CsOH (1.5 equiv) in 1,4-dioxane in air at 50 °C for 24 h provided **3a** in 86% yield (entry 13 or 14). Similar reaction of **1a**, **2a**, and CsOH in undried 1,4-dioxane under a N_2 atmosphere furnished **3a** in 93% yield. These results suggested that the base-mediated O-phenylation of **1a** by **2a** was not sensitive to air or moisture.

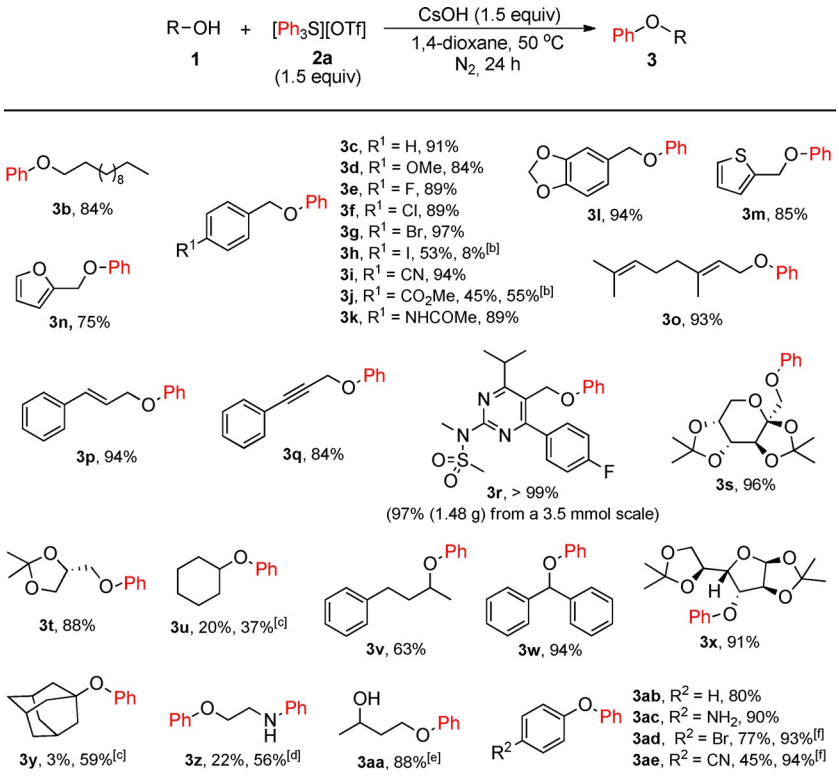
Table 1. Screening of the optimal reaction conditions for O-arylation of alcohol (**1a**) by triphenylsulfonium triflate (**2a**).[a]



Entry	Base	Solvent	T [°C]	Yield [**3a**, %][a]
1	CsOH	toluene	80	95
2	KOH	toluene	80	85
3	NaOH	toluene	80	71
4	LiOH	toluene	80	<1
5	NaH	toluene	80	93
6	*t*-BuOK	toluene	80	66
7	Cs_2CO_3	toluene	80	64
8	K_3PO_4	toluene	80	42
9	CsF	toluene	80	37
10	none	toluene	80	0
11	CsOH	THF	80	93
12	CsOH	1,4-dioxane	80	92
13	CsOH	toluene	100	>99
14	CsOH	1,4-dioxane	50	97 (94[b])
15[c]	CsOH	1,4-dioxane	50	86%
16[d]	CsOH	1,4-dioxane	50	93%

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.3 mmol), base (0.3 mmol), solvent (2 mL), 50 °C or 80 °C or 100 °C, N_2, 24 h. The yields were determined by HPLC using **3a** as an external standard ($t_R = 8.280$ min, $\lambda_{max} =$ 213 nm, water/ methanol = 10/ 90 (v/ v)). [b] Isolated yield. [c] The reaction was run in air. [d] The reaction was performed in undried 1,4-dioxane.

Taking an assembly of **1**, **2a** (1.5 equiv), CsOH (1.5 equiv), 1,4-dioxane, 50 °C, N_2 and 24 h as one of the optimal reaction conditions, the substrate scope of the O-arylation was examined (Scheme 2). To our delight, various primary alcohols such as dodecyl alcohol (**1b**) and benzylic alcohols (**1c**–**1k**) were readily converted to form the target aromatic ethers (**3b**–**3k**) in good to excellent yields (45–97%). The electron-donating groups (e.g., OMe (**1d**) and NHAc (**1k**)) and the electron-withdrawing groups (e.g., F (**1e**), Cl (**1f**), Br (**1g**), I (**1h**), CN (**1i**), and CO_2Me (**1j**)) on the aryl rings of benzylic alcohols were well tolerated in the reaction, which supplied the corresponding benzyl phenyl ethers (**3d**–**3i** and **3k**) in satisfactory yields. For the reaction of **1j** bearing an ester group, the use of Cs_2CO_3 instead of CsOH could further improve the yield of **3j**, probably due to the better survival of the ester group from the relatively weak base (Cs_2CO_3) than the strong one (CsOH). Other (hetero)arylmethanols like benzo[*d*][1,3]dioxol-5-ylmethanol (**1l**), thiophen-2-ylmethanol (**1m**), and furan-2-ylmethanol (**1n**) underwent smooth O-phenylation with **2a** to afford the respective phenylated products (**3l**–**n**) in 75–94% yields. Allyl alcohols such as (*E*)-3,7-dimethylocta-2,6-dien-1-ol (**1o**) and (*E*)-3-phenylprop-2-en-1-ol (**1p**) and propargyl alcohol like 3-phenylprop-2-yn-1-ol (**1q**) reacted successfully with **2a** under the standard conditions to furnish **3o**–**3q** in 84–94% yields, implying good compatibility of the carbon-carbon double bonds



Scheme 2. O-Phenylation of alcohols and phenols (**1**) by triphenylsulfonium triflate (**2a**).[a] [a] Reaction conditions: **1** (0.2 mmol), **2a** (0.3 mmol), CsOH (0.3 mmol), 1,4-dioxane (2 mL), 50 °C, N_2, 24 h. Isolated yield. [b] Cs_2CO_3 was used as a base. [c] NaH was used as a base. [d] **1**:**2a** : CsOH = 1:3:3. [e] 0.4 mmol scale. [f] 1:**2a** : CsOH = 1:1.5:3.

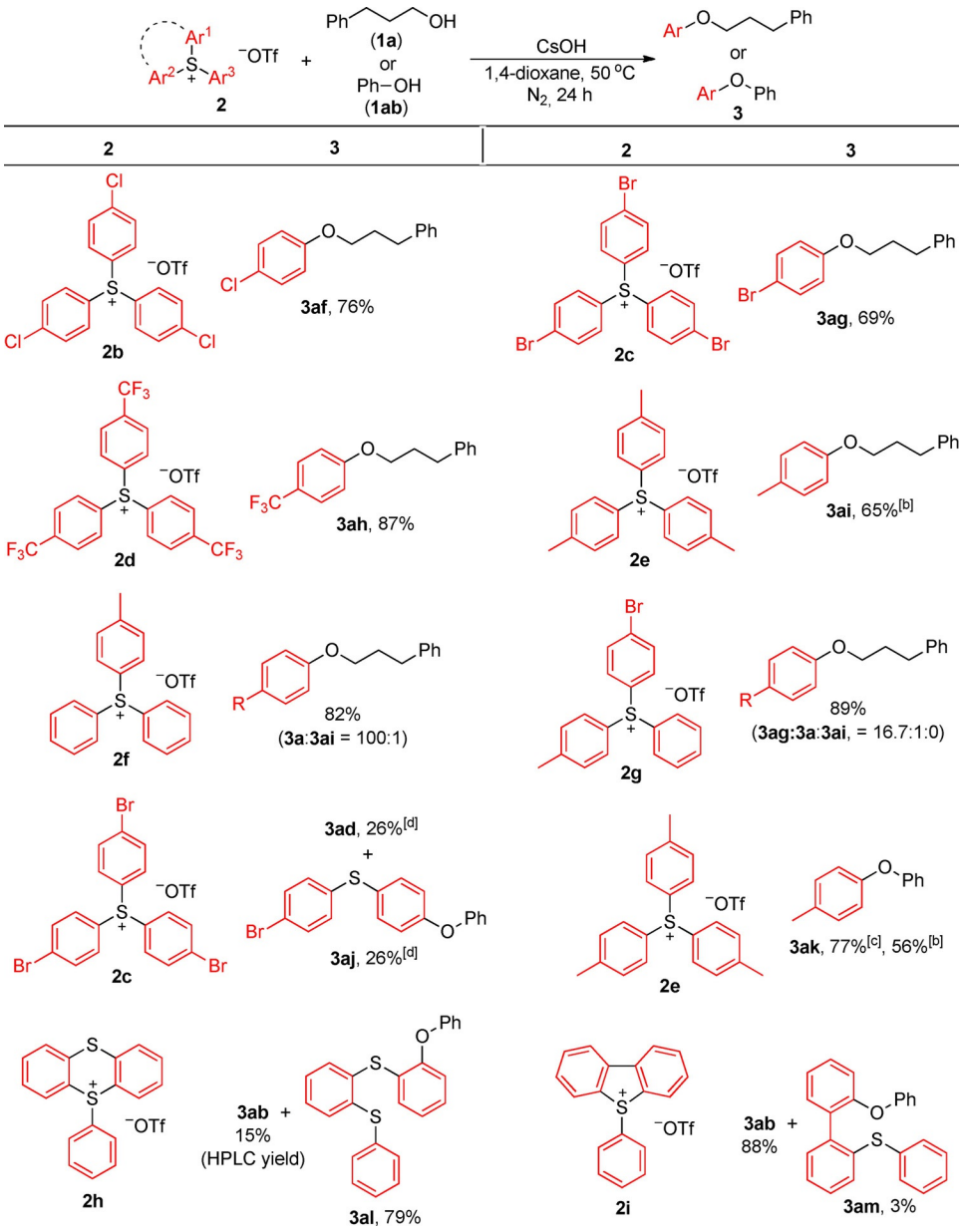
and triple bond in the reactions. Moreover, *N*-(4-(4-fluorophenyl)-5-(hydroxymethyl)-6-isopropyl-4,5-dihydropyrimidin-2-yl)-*N*-methylmethanesulfonamide (**1r**), an important synthetic intermediate for Rosuvastatin,[12] was a suitable substrate in this reaction, achieving almost quantitative yield of the O-phenylated product (**3r**, >99%). The reaction was also applicable to saccharide derivatives. Treatment of 2,3:4,5-di-*O*-isopropylidene-β-D-fructopyranose (**1s**) with **2a** and CsOH at 50 °C for 24 h gave **3s** in 96% yield, and reaction of (4*R*)-2,2-dimethyl-1,3-dioxolane-4-methanol (**1t**) with **2a**/CsOH under the same conditions formed **3t** in 88% yield. All these results suggested the great potentials of this method in modification of complex structures.

Additionally, the secondary alcohols such as cyclohexanol (**1u**), 4-phenylbutan-2-ol (**1v**), and diphenylmethanol (**1w**) reacted with **2a**/CsOH under the standard conditions to construct (cyclohexyloxy)benzene (**3u**), (3-phenoxybutyl)benzene (**3v**), and (phenoxymethylene)dibenzene (**3w**) in 20–94% yields. Likewise, treatment of 1,2:5,6-di-*O*-isopropylidene-α-L-glucofuranose (**1x**) with **2a** and CsOH furnished **3x** in 91% yield. Using NaH instead of CsOH in the reaction of **1u** and **2a** could slightly promote the formation of **3u** (37%). Tertiary alcohol such as (3s,5s,7s)-adamantan-1-ol (**1y**) was also amenable to the reaction. Under the standard conditions, the desired product (**3y**) was obtained in only 3% yield; however, use of NaH as a base in the same reaction led to 59% of **3y**. The less effective production of **3u** and **3y** in the presence of CsOH might be attributed to the relatively poorer acidity of the starting alcohols as well as their steric hindrance, which required stronger base such as NaH for the conversion. Since the free NH_2 groups could be arylated by $[Ar_3S][OTf]$ in the presence of bases,[10a] the reaction of ethanolamine (**1z**) with **2a** (1.5 or 3 equiv) and CsOH (1.5 or 3 equiv) under the standard conditions formed bis(arylated) product **3z** in 22% or 56% yield, respectively, accompanied by a trace amount or 19% of tri(arylated) product (*N*-(2-phenoxyethyl)diphenylamine, **3z′**). Notably, when 1,3-butanediol (**1aa**) reacted with **2a**/CsOH at 50 °C for 24 h, 4-phenoxy-2-butanol (**3aa**) was formed in 88% yield with the secondary hydroxyl group unchanged, suggesting that the O-phenylation could selectively occur at the primary alcoholic-OH site.

To further verify the generality of this method, we next applied the protocol to phenol systems. It was found that reactions of phenol derivatives with triphenylsulfonium triflate (**2a**) at 50 °C for 24 h produced diaryl ethers in good yields. 4-Aminophenol (**1ac**) reacted with **2a** and CsOH under the standard conditions to surprisingly afford 4-aminodiphenyl ether (**3ac**) in 90% yield as the sole product, indicating the preference of phenylation on aromatic OH group rather than aromatic NH_2 group. This result was different from that of the aliphatic analogue (**1z**), wherein no selectivity was observed. The selective phenylation of aromatic OH group over aromatic NH_2 group might be accounted for by the poorer nucleophilicity of the free aromatic NH_2 group than the phenoxy anion that was in situ formed by reacting with base. In addition, reaction of 4-bromophenol (**1ad**) with **2a**/CsOH under the standard condi-

tions constructed **3 ad** in 77% yield. If 3.0 equivalents of CsOH were employed in this reaction, **3 ad** was formed in 93% yield and **1 ad** was completely transformed. Analogously, treatment of 4-hydroxybenzonitrile (**1 ae**) with **2 a** (1.5 equiv)/CsOH (1.5 equiv) supplied 4-phenoxybenzonitrile (**3 ae**) in 45% yield, and increasing the equivalents of CsOH from 1.5 equiv to 3.0 equiv led to **3 ae** in 94% yield. It appeared that use of excess CsOH could improve the O-phenylation of electron-deficient phenols.

Other triarylsulfonium triflates were also reliable reagents in this base-mediated O-arylation of alcohol and phenol (Scheme 3). Tris(4-chlorophenyl)sulfonium triflate (**2 b**) reacted with **1 a** in the presence of CsOH under the standard conditions to furnish 1-chloro-4-(3-phenylpropoxy)benzene (**3 af**) in 76% yield. Analogously, reactions of tris(4-bromophenyl)sulfonium triflate (**2 c**) and tris(4-(trifluoromethyl)phenyl)sulfonium triflate (**2 d**) with **1 a**/CsOH gave 1-bromo-4-(3-phenylpropoxy)-benzene (**3 ag**) in 69% yield and 1-(3-phenylpropoxy)-4-(trifluoromethyl)benzene (**3 ah**) in 87% yield. Treatment of tris(4-toly)sulfonium triflate (**2 e**) with **1 a** and CsOH in toluene at 80 °C for 24 h provided 1-methyl-4-(3-phenylpropoxy)benzene (**3 ae**) in 65% yield. It seemed that the electron-donating CH_3 group on the aryl rings of **2 e** slowed down the arylation as the same reaction run in 1,4-dioxane at 50 °C for 24 h afforded no desired product. When diphenyl(4-tolyl)sulfonium triflate (**2 f**) was mixed with **1 a**/CsOH under the standard conditions, **3 a** was obtained in 82% yield accompanied by a trace amount of **3 ai** (**3 a** : **3 ai** = 100:1). If (4-bromophenyl)(phenyl)(4-tolyl)sul-

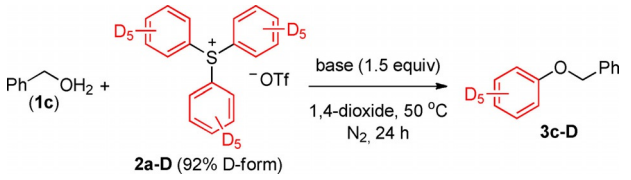


Scheme 3. O-Arylation of 3-phenylpropanol (**1 a**) and phenol (**1 ab**) by triarylsulfonium triflates in the presence of CsOH.[a] [a] Reaction conditions: **1 a** or **1 ab** (0.2 mmol), **2** (0.3 mmol), CsOH (0.3 mmol), 1,4-dioxane (2 mL), 50 °C, N_2, 24 h. Isolated yield. [b] Toluene (2 mL) as solvent and at 80 °C. [c] 80 °C. [d] 0.4 mmol scale (**1 ab**).

fonium triflate (**2g**) was treated with **1a**/CsOH in the same reaction, a mixture of **3ag** and **3a** was formed (89%) and no **3ai** was detected (**3ag**:**3a**:**3ai** = 16.7:1:0). The results suggested that the relatively electron-poor aryl groups of asymmetric sulfonium salts were much more easily transformed under the reaction conditions (e.g. phenyl vs. 4-tolyl (**2f**), 4-bromophenyl vs. phenyl (**2g**)), indicating good selectivity of the arylation with these salts. In a similar manner, reaction of **2c** with phenol (**1ab**) and CsOH at 50 °C for 24 h supplied 1-bromo-4-phenoxybenzene (**3ad**) in 26% yield. The formation of (4-bromophenyl)(4-phenoxyphenyl)sulfane (**3aj**) as a byproduct implied the competitive breakage of the C–Br bond with the C–S bond in the aryl rings of **2c**, which might subsequently undergo C–S bond cleavage to yield the side product. Again, treatment of **2e** with phenol (**1ab**) and CsOH at 80 °C for 24 h afforded **3ak** in good yield (77% or 56%) in either 1,4-dioxane or toluene. Intriguingly, 5-phenyl-5*H*-thianthren-5-ium trifluoromethanesulfonate (**2h**) reacted with **1ab**/CsOH under the standard conditions to form the ring opening products **3al** (79%) as the main product, while the similar reaction of 5-phenyl-5*H*-dibenzo[*b*,*d*]thiophen-5-ium trifluoromethanesulfonate (**2i**) with **1ab** and CsOH afforded the phenylated product (**3ab**, 88%) as the major product. The reasons for such differences remained unclear. It should be mentioned that there were no *meta*-isomers of the O-arylation products isolated in the above reactions, which might exclude the aryne intermediates.

Moreover, reactions of **1c** with deuterated triphenylsulfonium triflate (**2a-D**, 92% D-form) in 1,4-dioxane at 50 °C in the presence of different bases for 24 h provided **3c-D** in 23–90% yields (Table 2). Despite the yields varied, the percentages of

Table 2. O-Phenylation of **1c** by deuterated triphenylsulfonium triflate (**2a-D**) in the presence of different bases.[a]



2a-D (92% D-form)

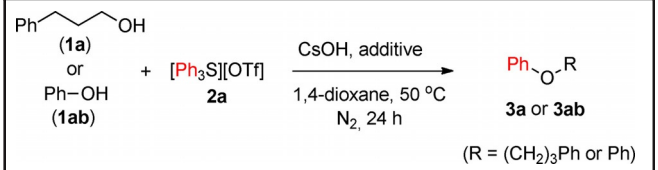
Entry	Base	Yield [**3c-D**, %]
1	CsOH	90, 92[b]
2	NaH	78, 92[b]
3	KOH	78, 92[b]
4	*t*-BuOK	23, 91[b]

[a] Reaction conditions: **1c** (0.2 mmol), **2a-D** (0.3 mmol), base (0.3 mmol), 1,4-dioxane (2 mL), 50 °C, N_2, 24 h. Isolated yield. [b] The percentage of deuterated form (D-form) (%). [c] CsOH (0.6 mmol).

the *ortho*-D forms of **3c-D** in these cases were rarely changed in comparison with that of **2a-D**, which were determined by ^{1}H NMR analysis of the isolated products. These data suggested again that the reaction might not proceed through an aryne intermediate even with diverse bases such as CsOH, NaH, KOH, and *t*BuOK.

In the previous alkali-initiated decomposition of triarylsulfonium salts, three possible mechanisms have been documented in the literatures:[11] a S_NAr mechanism, a benzyne intermediate, and a radical process. In our recent work, the aryne species have proved to be the major reactive intermediates in transition-metal-free *N*-arylation of amines by triarylsulfonium triflates in the presence of *t*BuOK or KOH.[10a] However, in this work the investigations revealed that the aryne intermediates might not be involved in the base-mediated O-arylation of alcohols and phenols by triarylsulfonium triflates. To further probe the possible reaction mechanisms, several control experiments were carried out (Table 3). Reactions of **1a** or **1ab**

Table 3. The standard reactions of **1a** and **1ab** in the presence of different radical inhibitors.[a]

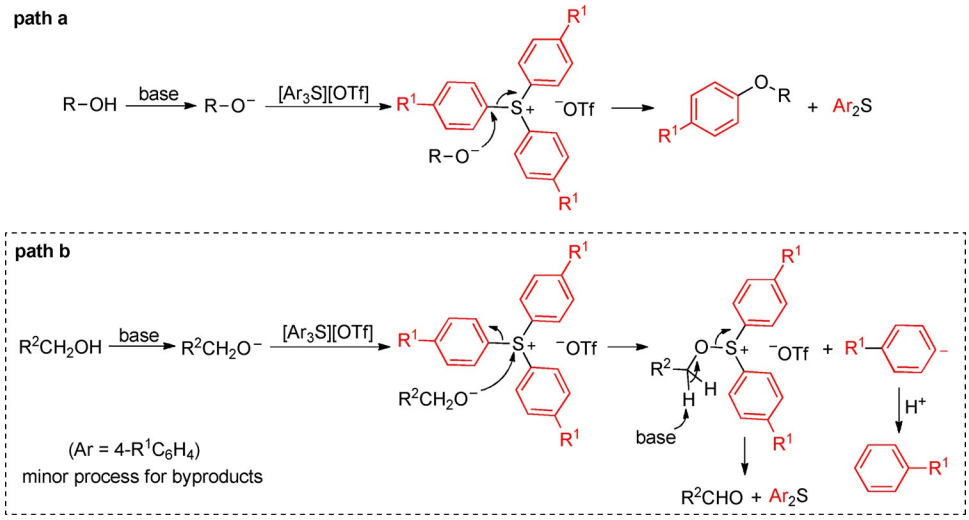


Entry	Additive	**3a** or **3ab**, Yield [%]
1	TEMPO	90 or 90
2	diallyl-PTSA	95 or 93
3	1,1-diphenylethylene	95 or 94
4	none	97 or 93
5[b]	none	97 or –

[a] Reaction conditions: **1a** or **1ab** (0.2 mmol), **2a** (0.3 mmol), CsOH (0.3 mmol), additive (0.3 mmol), 1,4-dioxane (2 mL), 50 °C, N_2, 24 h. The yields of **3a** were determined by HPLC using (3-phenoxypropyl)benzene as an external standard (t_R = 8.280 min, λ_{max} = 213 nm, water/ methanol = 10/ 90 (v/ v)). The yields of **3ab** were determined by HPLC using oxydibenzene as an external standard (t_R = 20.980 min, λ_{max} = 205 nm, water/ methanol = 20/ 80 (v/ v)). [b] The reaction of **1a** was run in the darkness. "-": The reaction was not conducted.

with **2a** (1.5 equiv) and CsOH (1.5 equiv) under the standard conditions in the presence of radical traps such as TEMPO, diallyl-PTSA, and 1,1-diphenylethylene gave **3a** or **3ab** in yields close to those without using inhibitors (entries 1–3 vs. entry 4). Furthermore, treatment of **1a** and **2a** with CsOH at 50 °C for 24 h in the darkness provided **3a** in 97% yield (entry 5). All these circumstances supported a non-radical pathway for both alcohols and phenols in the reactions.

Based on the above results and previous report,[11] a plausible nucleophilic substitution mechanism was suggested for this O-arylation (Scheme 4). Initially, alkoxide or phenoxide is derived from alcohol or phenol in the presence of CsOH. Then, nucleophilic attack of the alkoxy or phenoxy anion at the aromatic carbon atom of the C-S bond of triarylsulfonium cation gives O-arylated product and releases diaryl sulfide (**path a**). Since phenylmethanol (**1c**) reacted with **2a** (1.5 equiv) and CsOH (1.5 equiv) in 1,4-dioxane at 50 °C for 24 h to provide not only 91% of **3c** but also small amounts of benzene (4%) and benzaldehyde (4%), which were determined by HPLC analysis of the reaction mixtures, the alkaline decomposition of triarylsulfonium cation via nucleophilic attack of the alkoxide ion at the sulfur center of the C-S bond is also rational (**path b**). However, this process was not considered to be the predominant



Scheme 4. A proposed reaction mechanism for O-arylation of alcohols and phenols by triarylsulfonium triflates in the presence of bases.

pathway in the reaction as the side products (benzene and benzaldehyde) were formed in trace amounts. Likewise, the interaction of phenol and triarylsulfonium triflate via the nucleophilic attack of the phenoxy anion at the sulfur atom of the C–S bond in triarylsulfonium cation couldn't be excluded either.

Conclusions

In conclusion, we have developed a transition-metal-free method for O-arylation of alcohols and phenols by using triarylsulfonium triflates as arylation reagents and CsOH as a base. A variety of primary, secondary, and tertiary alcohols including complex structures were readily converted at 50 or 80 °C to give the corresponding alkyl aryl ethers in moderate to excellent yields. The phenol derivatives also performed well in this system, furnishing the target diaryl ethers in satisfactory yields. A deuterium labeling study, the control experiments, along with the absence of regioisomers of the O-arylated products using 4-substituted triarylsulfonium triflates, suggested a plausible mechanism via nucleophilic attack of alkoxide or phenoxide ions at the aromatic carbon atoms of the C–S bonds of triarylsulfonium cations for the base-mediated production of aromatic ethers. The mild and environmentally friendly conditions, wide range of substrates, good functional groups tolerance, excellent chemoselectivity, and good to high yields of the desired products promise applications of this new arylation protocol in future organic syntheses.

Experimental Section

General information

All reactions were carried out under a nitrogen atmosphere. Unless otherwise specified, NMR spectra were recorded in $CDCl_3$ on a 500 MHz (for ^1H), 471 MHz (for ^{19}F), and 126 MHz (for ^{13}C) spectrometer. All chemical shifts were reported in ppm relative to TMS (0 ppm for ^1H NMR) or $PhCF_3$ (−63.5 ppm for ^{19}F NMR) as an internal or external standard, respectively. The HPLC experiments were carried out on a Wufeng LC-100 II instrument (column: Shodex, C18, 5 μm, 4.6×250 mm), and the yields of products were determined by using the corresponding pure compounds as the external standards, respectively. The coupling constants were reported in Hertz (Hz). The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, brs = broad singlet. Melting points were measured and uncorrected. MS experiments were performed on a TOF-Q ESI or EI instrument. Arylsulfonium triflates (**2a–h**) used in this work were prepared according to the literatures.[8f,13] The solvents were dried before use according to the literature.[14] Other reagents in the reactions were all purchased from the commercial sources and used without further purification.

General procedures for O-arylation of alcohols and phenols (1) by triarylsulfonium triflates (2)

Procedure A: In a nitrogen filled glovebox, a sealed tube was charged with triarylsulfonium triflate (**2**, 0.3 mmol), CsOH (45.0 mg, 0.3 mmol), alcohol or phenol (**1**, 0.2 mmol), and 1,4-dioxane (2 mL) with vigorous stirring. The mixture was reacted at 50 °C for 24 h and cooled to room temperature. *m*-Chloroperoxybenzoic acid (216.0 mg, 1.05 mmol, 85%) was added. The mixture was kept at room temperature for 3.5 h, diluted with ethyl ether (20 mL), and washed with H_2O (3×25 mL). The aqueous solutions were extracted with ethyl ether (30 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and concentrated to dryness. The residue was purified by column chromatography on silica gel using petroleum ether or a mixture of petroleum ether and ethyl acetate as eluents to give the desired product (**3**).

Procedure B: In a nitrogen filled glovebox, a sealed tube was charged with triarylsulfonium triflate (**2**, 0.3 mmol), CsOH (45.0 mg, 0.3 mmol), alcohol or phenol (**1**, 0.2 mmol) and 1,4-dioxane (2 mL) with vigorous stirring. The mixture was reacted at 50 °C for 24 h, cooled to room temperature, diluted with ethyl ether (20 mL), and washed with H_2O (3×25 mL). The aqueous solutions were extracted with ether (30 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and concentrated to dryness. The residue was purified by column chromatography on silica gel using petroleum ether or a mixture of petroleum ether and ethyl acetate as eluents to give the desired product (**3**).

(3-Phenoxypropyl)benzene (**3a**).[15] Colorless oil (40.0 mg, 94%, **Procedure A**), petroleum ether as eluent for column chromatogra-

phy. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.28–7.25 (m, 4H), 7.21–7.17 (m, 3H), 6.94–6.88 (m, 3H), 3.95 (t, J = 6.2 Hz, 2H), 2.80 (t, J = 7.2 Hz, 2H), 2.10 ppm (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 159.1, 141.6, 129.5, 128.6, 128.5, 126.0, 120.6, 114.6, 66.8, 32.2, 30.9 ppm.

Dodecyloxybenzene (**3b**).[16] Yellowish oil (43.9 mg, 84%, **Procedure A**), petroleum ether as eluent for column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.29 (tm, J = 8.0 Hz, 2H), 6.96–6.91 (m, 3H), 3.97 (t, J = 6.6 Hz, 2H), 1.80 (m, 2H), 1.48 (m, 2H), 1.39–1.29 (m, 16H), 0.91 ppm (t, J = 6.90 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 159.2, 129.4, 120.5, 114.5, 67.9, 32.0, 29.7, 29.7, 29.7, 29.6, 29.5, 29.4, 29.4, 26.1, 22.7, 14.2 ppm.

(Benzyloxy)benzene (**3c**).[15] Colorless oil (33.5 mg, 91%, **Procedure A**), petroleum ether as eluent for column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.49 (d, J = 7.4 Hz, 2H), 7.44 (t, J = 7.6 Hz, 2H), 7.38 (d, J = 7.4 Hz, 1H), 7.35 (t, J = 7.7 Hz, 2H), 7.05–7.00 (m, 3H), 5.11 ppm (s, 2H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 158.9, 137.2, 129.6, 128.6, 128.0, 127.5, 121.0, 114.9, 70.0 ppm.

1-Methoxy-4-(phenoxymethyl)benzene (**3d**).[15] White solid (35.7 mg, 84%, **Procedure A**), a mixture of petroleum ether/ ethyl acetate = 40/ 1 (v/ v) as eluents for column chromatography. M.p.: 95.4–96.3 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.40 (d, J = 8.5 Hz, 2H), 7.32 (t, J = 8.0 Hz, 2H), 7.02–6.98 (m, 3H), 6.95 (d, J = 8.6 Hz, 2H), 5.02 (s, 2H), 3.84 ppm (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 159.5, 158.9, 129.5, 129.3, 129.2, 120.9, 114.9, 114.1, 69.7, 55.3 ppm.

1-Fluoro-4-(phenoxymethyl)benzene (**3e**).[15] White solid (36.2 mg, 89%, **Procedure A**), petroleum ether as eluent for column chromatography. M.p.: 43.1–44.2 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.42 (dd, J = 8.6, 5.6 Hz, 2H), 7.31 (t, J = 8.0 Hz, 2H), 7.09 (t, J = 8.7 Hz, 2H), 7.00–6.98 (m, 3H), 5.04 ppm (s, 2H). ^{19}F NMR (471 MHz, $CDCl_3$): δ = −114.3 ppm (m, 1F). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 162.5 (d, J = 246.3 Hz), 158.6, 132.9 (d, J = 3.2 Hz), 129.6, 129.4 (d, J = 8.2 Hz), 121.1, 115.5 (d, J = 21.5 Hz), 114.9, 69.3 ppm.

1-Chloro-4-(phenoxymethyl)benzene (**3f**).[15] White solid (39.0 mg, 89%, **Procedure A**), petroleum ether as eluent for column chromatography. M.p.: 85.1–87.0 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.40–7.36 (m, 4H), 7.31 (t, J = 8.0 Hz, 2H), 7.00–6.97 (m, 3H), 5.05 ppm (s, 2H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 158.6, 135.6, 133.7, 129.6, 128.8, 128.8, 121.2, 114.9, 69.2 ppm.

1-Bromo-4-(phenoxymethyl)benzene (**3g**).[15] White solid (51.3 mg, 97%, **Procedure A**), petroleum ether as eluent for column chromatography. M.p.: 95.6–97.2 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.52 (d, J = 8.3 Hz, 2H), 7.33–7.30 (m, 4H), 7.01–6.96 (m, 3H), 5.03 ppm (s, 2H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 158.6, 136.2, 131.7, 129.6, 129.1, 121.8, 121.2, 114.9, 60.8 ppm.

1-Iodo-4-(phenoxymethyl)benzene (**3h**).[17] White solid (32.9 mg, 53%, **Procedure A**; 5.0 mg from Cs_2CO_3 (97.8 mg, 0.3 mmol) as a base at 50 °C, 8%), petroleum ether as eluent for column chromatography. M.p.: 97.7–100.3 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.72 (d, J = 8.2 Hz, 2H), 7.30 (tm, J = 8.0 Hz, 2H), 7.19 (d, J = 8.2 Hz, 2H), 6.99–6.95 (m, 3H), 5.02 ppm (s, 2H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 158.5, 137.7, 136.8, 129.6, 129.3, 121.2, 114.9, 93.4, 69.2 ppm.

4-(Phenoxymethyl)benzonitrile (**3i**).[18] Light yellow solid (39.1 mg, 94%, **Procedure B**), a mixture of petroleum ether/ ethyl acetate = 80/ 1 (v/ v) as eluents for column chromatography. M.p.: 63.9–65.0 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.67 (d, J = 8.2 Hz, 2H), 7.55 (d, J = 8.2 Hz, 2H), 7.31 (tm, J = 8.0 Hz, 2H), 7.00 (t, J = 7.3 Hz, 1H), 6.97 (d, J = 7.9 Hz, 2H), 5.13 ppm (s, 2H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 158.2, 142.6, 132.4, 129.7, 127.6, 121.5, 118.7, 114.8, 111.7, 68.8 ppm.

Methyl 4-(phenoxymethyl)benzoate (**3j**).[19] White solid (22.0 mg, 45%, **Procedure B**; 26.3 mg from Cs_2CO_3 (97.8 mg, 0.3 mmol) as a base at 50 °C, 55%), a mixture of petroleum ether/ethyl acetate = 20/1 (v/ v) as eluents for column chromatography. M.p.: 93.0–93.8 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.06 (d, J = 8.3 Hz, 2H), 7.51 (d, J = 8.4 Hz, 2H), 7.30 (tm, J = 8.1 Hz, 2H), 6.99–6.96 (m, 3H), 5.13 (s, 2H), 3.92 ppm (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 166.9, 158.5, 142.3, 129.9, 129.7, 129.6, 127.0, 121.2, 114.9, 69.3, 52.2 ppm.

***N*-(4-(Phenoxymethyl)phenyl)acetamide** (**3k**). White solid (43.0 mg, 89%, **Procedure B**), a mixture of petroleum ether/ ethyl acetate = 2/1 (v/v) as eluents for column chromatography. M.p.: 133.7–134.2 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.53–7.51 (m, 3H), 7.38 (d, J = 8.3 Hz, 2H), 7.29 (t, J = 8.0 Hz, 2H), 6.98–6.95 (m, 3H), 5.02 (s, 2H), 2.17 ppm (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 168.6, 158.7, 137.7, 132.9, 129.5, 128.3, 121.0, 120.0, 114.9, 69.6, 24.6 ppm. IR (KBr): $\tilde{\nu}$ = 3317, 3191, 3121, 3069, 3060, 3042, 2943, 2923, 2872, 1664, 1612, 1598, 1586, 1525, 1496, 1468, 1458, 1409, 1382, 1365, 1339, 1314, 1291, 1266, 1236, 1179, 1159, 1113, 1080, 1031, 1015, 993, 968, 884, 876, 856, 819, 808, 794, 755, 727, 693 cm^{-1}. HRMS-ESI (m/z) calcd for $C_{15}H_{16}NO_2$ ([M+H]$^+$): 242.1176; found: 242.1179.

5-(Phenoxymethyl)benzo[*d*][1,3]dioxole (**3l**). White solid (42.9 mg, 94%, **Procedure B**), a mixture of petroleum ether/ ethyl acetate = 40/ 1 (v/ v) as eluents for column chromatography. M.p.: 57.4–58.7 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.32 (t, J = 8.0 Hz, 2H), 7.01–6.97 (m, 4H), 6.92 (dm, J = 7.9 Hz, 1H), 6.84 (d, J = 7.9 Hz, 1H), 5.98 (s, 2H), 4.98 ppm (s, 2H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 158.7, 147.9, 147.4, 130.9, 129.5, 121.3, 121.0, 114.9, 108.4, 108.3, 101.1, 69.9 ppm. IR (KBr): $\tilde{\nu}$ = 3105, 3059, 3040, 3011, 2923, 2870, 2840, 2799, 1845, 1597, 1583, 1501, 1465, 1447, 1389, 1293, 1264, 1244, 1172, 1100, 1080, 1028, 1006, 988, 926, 888, 870, 801, 777, 749, 695 cm^{-1}. HRMS-ESI (m/z) calcd for $C_{14}H_{13}O_3$ ([M+H]$^+$): 229.0859; found: 229.0852.

2-(Phenoxymethyl)thiophene (**3m**).[20] Yellow oil (32.3 mg, 85%, **Procedure B**), petroleum ether as eluent for column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.35–7.31 (m, 3H), 7.14 (d, J = 3.3 Hz, 1H), 7.04–6.99 (m, 4H), 5.25 ppm (s, 2H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 158.4, 139.4, 129.6, 126.8, 126.2, 121.3, 115.1, 65.0 ppm.

2-(Phenoxymethyl)furan (**3n**).[21] Yellow oil (26.1 mg, 75%, **Procedure B**), a mixture of petroleum ether/ ethyl acetate = 80/ 1 (v/ v) as eluents for column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.46 (dd, J = 1.8 Hz, J = 0.7 Hz, 1H), 7.31 (m, 2H), 7.01–6.98 (m, 3H), 6.45 (d, J = 3.2 Hz, 1H), 6.39 (dd, J = 3.2 Hz, J = 1.9 Hz, 1H), 5.02 ppm (s, 2H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 158.4, 150.4, 143.1, 129.5, 121.3, 115.0, 110.5, 109.9, 62.4 ppm.

(*E*)-((3,7-Dimethylocta-2,6-dien-1-yl)oxy)benzene (**3o**).[21] Light yellow oil (42.8 mg, 93%, **Procedure B**), petroleum ether as eluent for column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.30 (t, J = 8.0 Hz, 2H), 6.98–6.94 (m, 3H), 5.53 (t, J = 6.5 Hz, 1H), 5.13 (t, J = 6.4 Hz, 1H), 4.57 (d, J = 6.6 Hz, 2H), 2.19–2.10 (m, 4H), 1.77 (s, 3H), 1.71 (s, 3H), 1.64 ppm (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 158.9, 141.1, 131.8, 129.4, 123.9, 120.6, 119.7, 114.7, 64.8, 39.6, 26.4, 25.7, 17.7, 16.7 ppm.

(Cinnamyloxy)benzene (**3p**).[21] White solid (39.6 mg, 94%, **Procedure B**), petroleum ether as eluent for column chromatography. M.p.: 69.2–70.3 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.44 (d, J = 7.6 Hz, 2H), 7.37–7.26 (m, 5H), 7.01–6.98 (m, 3H), 6.76 (d, J = 16.0 Hz, 1H), 6.45 (dt, J = 16.0 Hz, J = 5.8 Hz, 1H), 4.73 ppm (dd, J = 5.8 Hz, J = 1.4 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 158.7, 136.5, 133.0, 129.6, 128.6, 127.9, 126.6, 124.6, 121.0, 114.9, 68.6 ppm.

(3-Phenoxyprop-1-yn-1-yl)benzene (**3q**).[22] Light yellow oil (34.8 mg, 84%, **Procedure B**), petroleum ether as eluent for column chromatography. M.p.: 44.7–46.1 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.48 (m, 2H), 7.37–7.31 (m, 5H), 7.08 (d, J = 7.9 Hz, 2H), 7.03 (t, J = 7.4 Hz, 1H), 4.94 ppm (s, 2H). ^{13}C NMR (126 MHz, $CDCl_3$):

δ = 157.9, 131.9, 129.5, 128.7, 128.3, 122.4, 121.5, 115.1, 87.2, 84.0, 56.7 ppm.

***N*-(4-(4-Fluorophenyl)-6-isopropyl-5-(phenoxymethyl)pyrimidin-2-yl)-*N*-methylmethanesulfonamide** (**3r**). White solid (86.2 mg, >99%, **Procedure B**; 1.48 g from a 3.5 mmol scale, 97%), a mixture of petroleum ether/ ethyl acetate = 40/ 1 (v/ v) as eluents for column chromatography. M.p.: 152.4–155.1 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.75 (m, 2H), 7.34 (m, 2H), 7.10 (tm, J = 8.7 Hz, 2H), 7.05 (t, J = 7.4 Hz, 1H), 6.96 (d, J = 7.9 Hz, 2H), 4.91 (s, 2H), 3.61 (s, 3H), 3.54 (s, 3H), 3.36 (m, 1H), 1.34 ppm (d, J = 6.7 Hz, 6H). ^{19}F NMR (471 MHz, $CDCl_3$): δ = −110.9 ppm (m, 1F). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 178.4, 166.6, 163.8 (d, J = 250.5 Hz), 158.4, 158.0, 133.8 (d, J = 3.2 Hz), 131.5 (d, J = 8.2 Hz), 129.7, 121.6, 117.5, 115.6 (d, J = 21.8 Hz), 114.7, 63.3, 42.5, 33.1, 31.8, 22.1 ppm. IR (KBr): $\tilde{\nu}$ = 3117, 3077, 3041, 3013, 2977, 2929, 2901, 2872, 1603, 1554, 1509, 1496, 1484, 1441, 1403, 1363, 1335, 1230, 1152, 1068, 1026, 994, 965, 948, 898, 872, 846, 821, 774, 763, 729, 711, 693, 630, 603 cm^{-1}. HRMS-ESI (*m*/*z*) calcd for $C_{22}H_{25}FN_3O_3S$ ($[M+H]^+$): 430.1595; found: 430.1595.

(3α*S*,5α*R*,8α*R*,8β*S*)-2,2,7,7-Tetramethyl-3α-(phenoxymethyl)tetrahydro-5*H*-bis([1,3]dioxolo)[4,5-*b*:4′,5′-*d*]pyran (**3s**). Light yellow oil (61. 6 mg, 96%, **Procedure B**), a mixture of petroleum ether/ ethyl acetate = 20/ 1 (v/ v) as eluents for column chromatography. $[\alpha]_D^{23.5}$ = −15.93 (*c* = 0.676 g/100 mL, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.39 (tm, J = 8.0 Hz, 2H), 6.98–6.93 (m, 3H), 4.65 (dd, J = 7.9 Hz, J = 2.6 Hz, 1H), 4.57 (d, J = 2.6 Hz, 1H), 4.27 (dd, J = 7.8 Hz, J = 1.2 Hz, 1H), 4.16 (d, J = 10.1 Hz, 1H), 4.05 (d, J = 10.1 Hz, 1H), 3.98 (dd, J = 13.0 Hz, J = 1.9 Hz, 1H), 3.80 (d, J = 13.0 Hz, 1H), 1.58 (s, 3H), 1.51 (s, 3H), 1.50 (s, 3H), 1.35 ppm (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 158.6, 129.4, 121.1, 114.7, 109.0, 108.9, 102.3, 71.1, 70.3, 70.1, 68.8, 61.2, 26.6, 26.0, 25.4, 24.1 ppm. IR (KBr): $\tilde{\nu}$ = 3064, 3041, 2990, 2936, 1601, 1589, 1497, 1456, 1381, 1373, 1337, 1301, 1250, 1215, 1184, 1165, 1105, 1071, 1050, 1015, 981, 914, 891, 868, 837, 816, 754, 715, 691, 673, 637 cm^{-1}. HRMS-ESI (*m*/*z*) calcd for $C_{18}H_{25}O_6$ ($[M+H]^+$): 337.1646; found: 337.1654.

(*R*)-2,2-Dimethyl-4-(phenoxymethyl)-1,3-dioxolane (**3t**). Light yellow oil (36.8 mg, 88%, **Procedure B**), a mixture of petroleum ether/ ethyl acetate = 40/ 1 (v/ v) as eluents for column chromatography. $[\alpha]_D^{25}$ = −27.20 (*c* = 0.500 g/100 mL, Et_2O). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.29 (t, J = 7.7 Hz, 2H), 6.97 (t, J = 7.3 Hz, 1H), 6.93 (d, J = 8.0 Hz, 2H), 4.49 (m, 1H), 4.18 (t, J = 6.8 Hz, 1H), 4.07 (m, 1H), 3.96–3.90 (m, 2H), 1.48 (s, 3H), 1.42 ppm (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 158.6, 129.5, 121.1, 114.6, 109.7, 74.1, 68.8, 66.9, 26.8, 25.4 ppm. HRMS-ESI (*m*/*z*) calcd for $C_{12}H_{17}O_3$ ($[M+H]^+$): 209.1172; found: 209.1173.

(Cyclohexyloxy)benzene (**3u**).[21] Colorless oil (7.0 mg, 20%, **Procedure A**; 38.8 mg from a 1.0 mmol scale of **1u** (100.2 mg), 22%; 13.0 mg from NaH (12.0 mg, 0.3 mmol, 60%) as a base, 37%). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.29 (t, J = 7.5 Hz, 2H), 6.96–6.93 (m, 3H), 4.27 (m, 1H), 2.01 (m, 2H), 1.83 (m, 2H), 1.62–1.52 (m, 3H), 1.44–1.33 ppm (m, 3H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 157.9, 129.4, 120.5, 116.2, 75.4, 31.9, 25.7, 23.8.

(3-Phenoxybutyl)benzene (**3v**).[23] Brown oil (28.5 mg, 63%, **Procedure B**), a mixture of petroleum ether/ ethyl acetate = 80/ 1 (v/ v) as eluents for column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.26–7.23 (m, 4H), 7.19–7.16 (m, 3H), 6.92 (t, J = 7.2 Hz, 1H), 6.86 (d, J = 8.0 Hz, 2H), 4.35 (m, 1H), 2.76 (m, 2H), 2.06 (m, 1H), 1.88 (m, 1H), 1.31 ppm (d, J = 6.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 158.2, 141.9, 129.5, 128.5, 128.4, 125.9, 120.6, 116.0, 72.8, 38.3, 31.9, 19.8 ppm.

(Phenoxymethylene)dibenzene (**3w**).[21] Light yellow oil (49.2 mg, 94%, **Procedure A**), petroleum ether as eluent for column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.49 (d, J = 7.5 Hz, 4H), 7.40 (t, J = 7.6 Hz, 4H), 7.32 (t, J = 7.3 Hz, 2H), 7.27 (t, J = 8.0 Hz, 2H), 7.03 (d, J = 8.0 Hz, 2H), 6.97 (t, J = 7.4 Hz, 1H), 6.28 ppm (s, 1H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 158.3, 141.4, 129.4, 128.7, 127.8, 127.0, 121.1, 116.3, 81.9 ppm.

(3α*S*,5*S*,6*R*,6α*S*)-5-((*S*)-2,2-Dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyl-6-phenoxytetrahydrofuro[2,3-*d*][1,3]dioxole (**3x**). White solid (61.2 mg, 91%, **Procedure B**), a mixture of petroleum ether/ ethyl acetate = 40/ 1 (v/ v) as eluents for column chromatography. M.p.: 101–103.2 °C. $[\alpha]_D^{23.5}$ = −44.44 (*c* = 0.676 g/100 mL, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.32 (t, J = 7.9 Hz, 2H), 7.02–6.98 (m, 3H), 5.94 (d, J = 3.8 Hz, 1H), 4.74 (d, J = 3.0 Hz, 1H), 4.61 (d, J = 3.8 Hz, 1H), 4.48 (dd, J = 13.0 Hz, J = 6.1 Hz, 1H), 4.35 (dd, J = 7.5 Hz, J = 3.1 Hz, 1H), 4.17–4.11 (m, 2H), 1.56 (s, 3H), 1.45 (s, 3H), 1.33 (s, 3H), 1.31 ppm (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 156.9, 129.7, 121.8, 115.5, 112.1, 109.1, 105.3, 82.2, 80.5, 79.8, 72.3, 67.0, 26.8, 26.8, 26.3, 25.3 ppm. IR (KBr): $\tilde{\nu}$ = 3063, 3041, 2987, 2935, 2879, 1601, 1588, 1498, 1470, 1456, 1381, 1371, 1291, 1246, 1173, 1157, 1078, 1054, 976, 884, 847, 815, 755, 692 cm^{-1}. HRMS-ESI (*m*/*z*) calcd for $C_{18}H_{25}O_6$ ($[M+H]^+$): 337.1646; found: 336.1636.

(3*s*,5*s*,7*s*)-1-Phenoxyadamantane (**3y**).[23] White solid (1.4 mg, 3%, **Procedure A**; 26.8 mg from NaH (12.0 mg, 0.3 mmol, 60%) as a base, 59%), a mixture of petroleum ether/ ethyl acetate = 80/ 1 (v/ v) as eluents for column chromatography. M.p.: 108.0–110.4 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.28 (t, J = 7.5 Hz, 2H), 7.12 (t, J = 7.3 Hz, 1H), 7.02 (d, J = 7.6 Hz, 2H), 2.20 (brs, 3H), 1.91 (d, J = 2.1 Hz, 6H), 1.64 ppm (m, 6H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 153.0, 127.6, 124.0, 122.6, 76.6, 41.8, 35.1, 29.8 ppm.

***N*-(2-Phenoxyethyl)aniline** (**3z**).[24] Yellow solid (9.3 mg, 22%, **Procedure B**; 23.7 mg from **2a** ($[Ph_3S][OTf]$, 247.2 mg, 0.6 mmol), **1z** (12.3 mg, 0.2 mmol) and CsOH (90.0 mg, 0.6 mmol), 56%), a mixture of petroleum ether/ ethyl acetate = 40/ 1 (v/ v) as eluents for column chromatography. M.p.: 48.8–49.3 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.32 (t, J = 7.7 Hz, 2H), 7.23 (t, J = 7.6 Hz, 2H), 7.00 (t, J = 7.4 Hz, 1H), 6.96 (d, J = 8.0 Hz, 2H), 6.77 (t, J = 7.3 Hz, 1H), 6.71 (d, J = 7.9 Hz, 2H), 4.19 (t, J = 5.3 Hz, 2H), 4.12 (brs, 1H), 3.56 ppm (t, J = 5.3 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 158.7, 147.9, 129.6, 129.4, 121.1, 117.9, 114.6, 113.2, 66.4, 43.4 ppm.

***N*-(2-Phenoxyethyl)-*N*-phenylaniline** (**3z′**). Trace, **Procedure B**; 11.1 mg from **2a** ($[Ph_3S][OTf]$, 247.2 mg, 0.6 mmol), **1z** (12.3 mg, 0.2 mmol) and CsOH (90.0 mg, 0.6 mmol), 19%), a mixture of petroleum ether/ ethyl acetate = 80/ 1 (v/ v) as eluents for column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.28–7.23 (m, 6H), 7.06 (d, J = 7.9 Hz, 4H), 6.98–6.91 (m, 3H), 6.85 (d, J = 8.0 Hz, 2H), 4.19–4.13 ppm (m, 4H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 158.7, 147.8, 129.5, 129.4, 121.6, 121.0, 120.9, 114.5, 64.8, 51.2 ppm. IR (KBr): $\tilde{\nu}$ = 3060, 3037, 2924, 2871, 2853, 1598, 1588, 1576, 1496, 1475, 1461, 1362, 1300, 1242, 1172, 1154, 1098, 1078, 1056, 1040, 993, 885, 865, 818, 800, 750, 692 cm^{-1}. HRMS-ESI (*m*/*z*) calcd for $C_{20}H_{19}NOK$ ($[M+K]^+$): 328.1098; found: 328.1099.

4-Phenoxybutan-2-ol (**3aa**).[25] Light yellow oil (58.4 mg from a 0.4 mmol scale of **1aa** (36.2 mg), 88%), a mixture of petroleum ether/ ethyl acetate = 80/ 1 (v/ v) as eluents for column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.29 (tm, J = 8.0 Hz, 2H), 6.96 (t, J = 7.3 Hz, 1H), 6.92 (d, J = 8.0 Hz, 2H), 4.17 (m, 1H), 4.10 (m, 2H), 2.31 (brs, 1H), 1.93 (m, 2H), 1.28 ppm (d, J = 6.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$): δ = 158.7, 129.5, 120.9, 114.6, 66.3, 65.8, 38.2, 23.7 ppm.

Oxydibenzene (**3ab**).[16] Colorless oil (27.2 mg, 80%, **Procedure A**), petroleum ether as eluent for column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.35 (t, J = 7.9 Hz, 4H), 7.12 (t, J = 7.4 Hz, 2H),

7.04 ppm (d, $J=7.8$ Hz, 4H). ^{13}C NMR (126 MHz, $CDCl_3$): $\delta=157.3$, 129.7, 123.2, 118.9 ppm.

4-Phenoxyaniline (**3 ac**).[26] Brown solid (33.5 mg, 90%, **Procedure B**), a mixture of petroleum ether/ ethyl acetate = 40/ 1 (v/ v) as eluents for column chromatography. M.p.: 85.1–87.4 °C. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta=7.30$ (tm, $J=8.0$ Hz, 2H), 7.03 (t, $J=7.4$ Hz, 1H), 6.95 (dm, $J=7.8$ Hz, 2H), 6.90 (dm, $J=8.7$ Hz, 2H), 6.69 (dm, $J=8.7$ Hz, 2H), 3.59 ppm (brs, 2H). ^{13}C NMR (126 MHz, $CDCl_3$): $\delta=$ 158.9, 148.6, 142.7, 129.6, 122.1, 121.2, 117.3, 116.3 ppm.

1-Bromo-4-phenoxybenzene (**3 ad**).[16] Light yellow oil (38.2 mg, 77%, **Procedure A**; 46.4 mg from **2 a** ([Ph_3S][OTf], 123.6 mg, 0.3 mmol), **1 ad** (34.6 mg, 0.2 mmol) and CsOH (90.0 mg, 0.6 mmol), 93%), petroleum ether as eluent for column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta=7.43$ (d, $J=8.8$ Hz, 2H), 7.35 (t, $J=$ 7.9 Hz, 2H), 7.13 (t, $J=7.4$ Hz, 1H), 7.01 (d, $J=8.0$ Hz, 2H), 6.89 ppm (dm, $J=8.8$ Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$): $\delta=156.8$, 156.6, 132.7, 129.9, 123.7, 120.5, 119.1, 115.6 ppm.

4-Phenoxybenzonitrile (**3 ae**).[21] Yellow oil (17.6 mg, 45%, **Procedure B**; 36.9 mg from **2 a** ([Ph_3S][OTf], 123.6 mg, 0.3 mmol), **1 ae** (24.6 mg, 0.2 mmol) and CsOH (90.0 mg, 0.6 mmol), 94%), a mixture of petroleum ether/ ethyl acetate = 80/ 1 (v/ v) as eluents for column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta=7.60$ (dm, $J=8.8$ Hz, 2H), 7.42 (tm, $J=8.0$ Hz, 2H), 7.23 (tm, $J=7.4$ Hz, 1H), 7.07 (dm, $J=8.0$ Hz, 2H), 7.01 ppm (dm, $J=8.9$ Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$): $\delta=161.7$, 154.8, 134.1, 130.2, 125.2, 120.4, 118.8, 117.9, 105.8 ppm.

The characterization data of **3 af**–**3 am** in Scheme 3 were described in the supporting information

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Conflict of interest

The authors declare no conflict of interest.

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