Palladium-catalyzed Decarbonylative Catellani Reaction

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The transition metal-catalyzed Catellani reaction of aryl halides has drawn significant attentions as an efficient and practical tool for the synthesis of substituted arenes. We describe herein the palladium-catalyzed, norbornene (NBE)-mediated synthesis of polysubstituted arenes from aromatic acids via decarbonylative Catellani reaction. A variety of alkenyl, alkyl, aryl and sulfur moieties could be conveniently introduced into the *ipso*-positions of aromatic thioesters. By merging carboxyl-directed C−H functionalization and the classical Catellani reactions, our protocol allowed the construction of 1,2,3-trisubstituted and 1,2,3,4-tetrasubstituted arenes from simple aromatic acids. Furthermore, the late-stage functionalization of a series of drug molecules highlights the potential utility of the reaction.

The Pd/norbornene (NBE) cooperative catalysis offers distinct access to multisubstituted arenes.[1] The high ring strain of the [2.2.1] bicyclic scaffold together with its structural rigidity enables the NBE to function as a transient mediator to complete intermolecular carbopalladation and final extrusion.[2] In 1997, Catellani and coworkers disclosed the first cooperative Pd/NBE-catalyzed reaction using aryl iodides, allowing for the expeditious synthesis of *ipso-ortho* bifunctionalized arenes (Figure 1a).[3] Following this initial breakthrough, Lautens, Dong and others have greatly enriched this chemistry in the past few years, making this methodology a reliable route for the synthesis of complex arenes.[4] It is noteworthy that the Dong group recently synthesized tetrasubstituted alkenes via a palladium/NBE catalyzed alkenyl Catellani reaction.[5] With NBE as a transient mediator, Bach first developed a Pd(II)-initiated 2-functionalization of indoles and pyrroles.[6] In 2015, Yu creatively combined the amide directed *ortho* C−H activation and Pd/NBE chemistry, which subtly achieved *meta*-selective inert C−H alkylation and arylation.[7] Subsequently, this norbornene relay approach

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has been extensively applied in *meta*- and *para*-C−H activations.[8] Recently, the Dong group reported a distal alkenyl C−H functionalization through this directed Pd/NBE cooperative catalysis.[9] Furthermore, Pd(II)-initiated Catellani-type reactions with arylboron species have been elegantly realized by Zhou, Zhang and Dong.[10]

The carboxylic acid functionality is ubiquitous in natural products, drugs, and agrochemicals. Their benign syntheses and structural diversities enable the carboxylic acids to play an important role in organic chemistry.[11] Over the past few years, transition metal-catalyzed transformations of aryl carboxylic acids into new C−C and C−heteroatom bonds have drawn significant attention as an alternative to traditional cross-coupling procedures. In 2002, Myers disclosed a Pd-catalyzed decarboxylative Heck-type olefination of aromatic acids.[12] Subsequently, Gooßen reported a Pd/Cu-cocatalyzed decarboxylative coupling between aromatic acids and aryl halides to form biaryl skeletons.[13] Since these seminal works, tremendous progress has been made in the area of decarboxylative cross-coupling reactions of aromatic acids.[14] Moreover, carboxyl groups are efficient directing groups in C−H functionalization.[15] Carboxyl-directed *ortho*-C−H functionalization and subsequent *ipso*-decarboxylation is a promising approach for the construction of polysubstituted arenes,[16] which are ubiquitous structural cores in medicinal and material science.[17] However, these reactions mainly focus on decarboxylative protonation. Thus, the development and validation of a general approach to achieve decarboxylative bis- and even poly-functionalization of broadly available aromatic acids is still a challenge.[18]

Thioesters are common intermediates in both organic synthesis and biochemical processes.[19] Compared with other carboxylic acid derivatives, thioesters are stable but reactive, and can facilitate the oxidative addition of C(O)−S to low-valent transition-metal species.[20] Further decarbonylation of the thioester affords the ArM species, which then undergoes the cross coupling reaction with the nucleophiles.[21] In connection with our interest in thioester and Catellani reaction, we envisage that a combination of Pd(0)/NBE and aromatic thioesters could afford the 1,2-disubstituted arenes via decarbonylation of the thioester and subsequent NBE-mediated *ortho*-C−H and *ipso*-C−Pd functionalizations. Herein, we report a cooperative palladium-catalyzed, NBE-mediated disubstitution of (hetero)aromatic thioesters via decarbonylative Catellani reaction (Figure 1b). The potential applications of this protocol were demonstrated by the late-stage bifunctionalization of some commercial drugs and construction of polysubstituted arenes from aromatic acids.

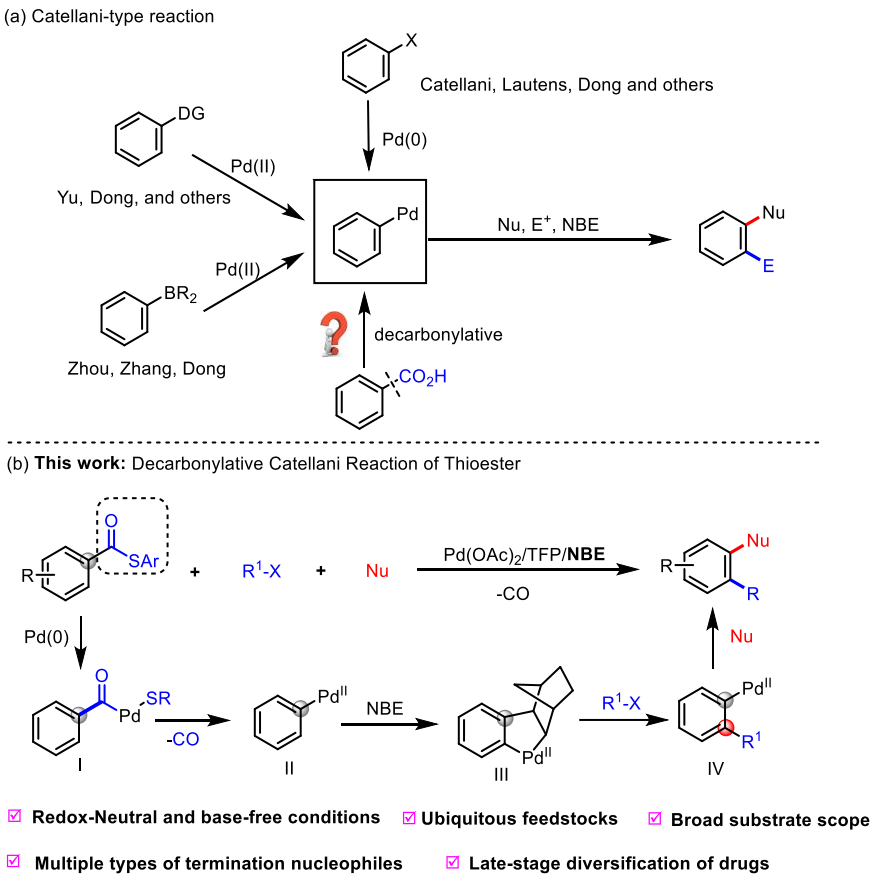
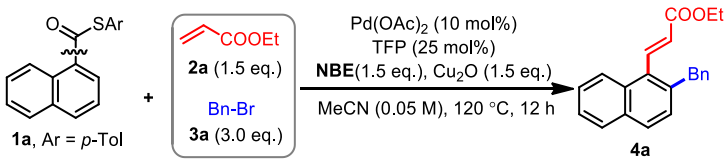


Fig. 1 | Catellani-type Reaction.

Results and discussion

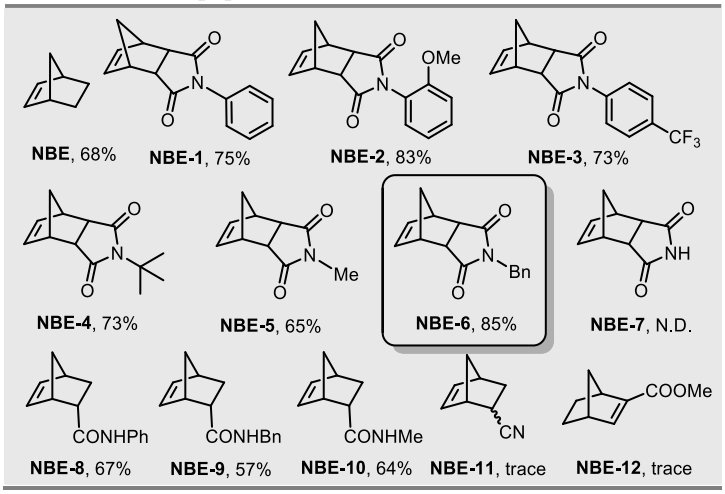
We began our investigation by choosing S-(*p*-tolyl) naphthalene-1-carbothioate **1a** as the model substrates, ethyl acrylate **2a** as the nucleophile, and benzyl bromide **3a** as the electrophile. After screening of various reaction parameters, the desired product **4a** was obtained in 68% yield in the presence of $Pd(OAc)_2$ (10 mol%), TFP (25 mol%), norbornene (NBE) (1.5 equiv.), and Cu_2O (1.5 equiv.) at 120 °C (Table 1, entry 1). Control experiments indicated that the palladium, phosphine ligand (TFP), NBE and Cu_2O were all essential for the reaction (Table 1, entries 2−5). Other palladium catalysts could also afford the desired product, albeit in lower yields (Table 1, entry 6). In Catellani-type reactions, the addition of an exogenous base is often required.[1f] To our delight, our protocol was able to proceed under

Table 1. | Reaction Optimization



Entry	Reaction condition variations	Yield(%)
1	none	68
2	no $Pd(OAc)_2$	0
3	no TFP	0
4	no **NBE**	0
5	no Cu_2O	0
6	$Pd_2(dba)_3$ instead of $Pd(OAc)_2$	49
7	Cs_2CO_3 was added as base	trace
8	NaOAc was added as base	66
9	P(*p*-MePh)$_3$ instead of TFP	trace
10	DPEPhos instead of TFP	42
11	CuTC instead of Cu_2O	28
12	CuOAc instead of Cu_2O	44
13	other NBEs instead of **NBE**	listed below

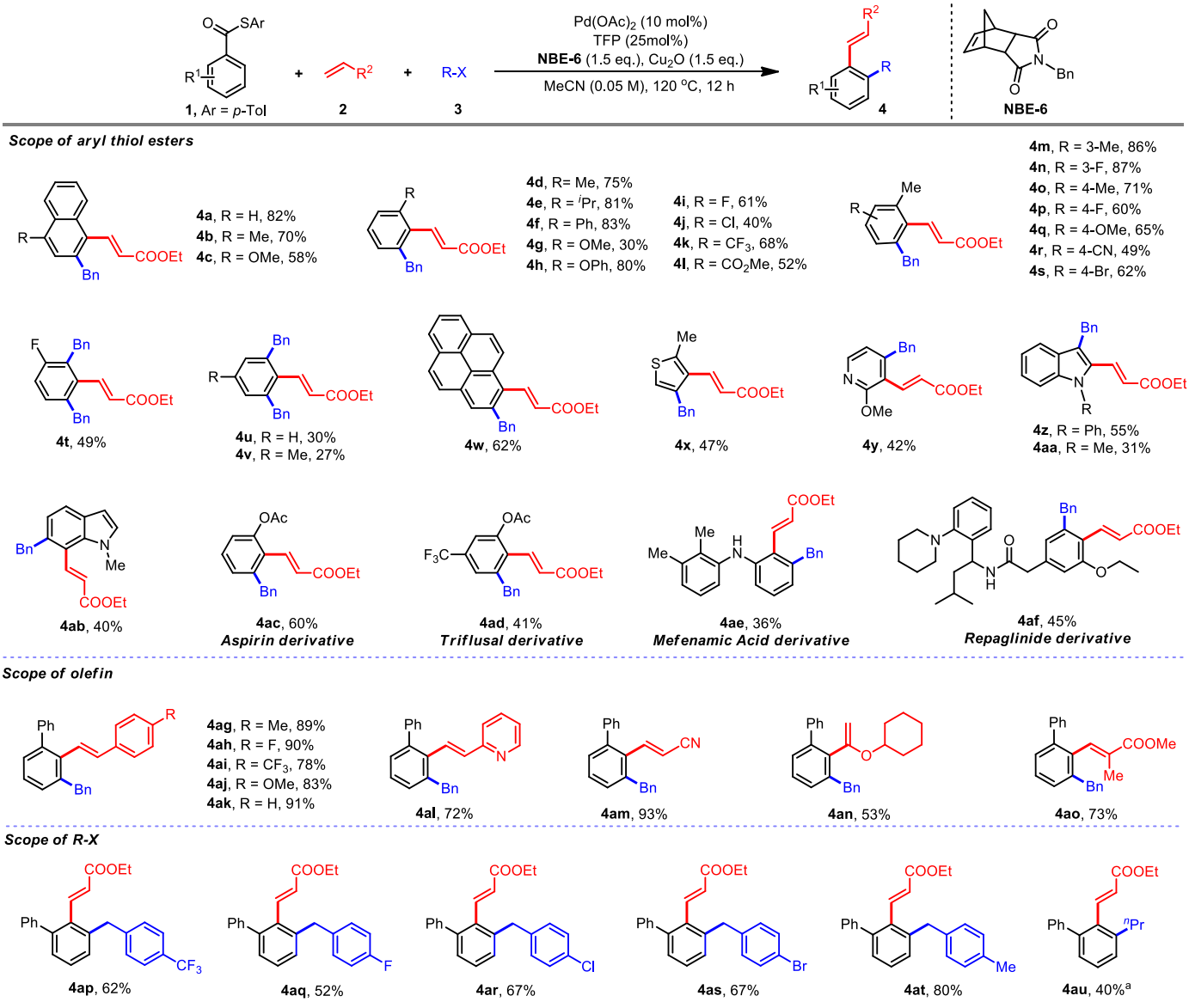
[a]Reactions were run on a 0.10 mmol scale. Yields were determined from the crude ^{1}H NMR spectra using CH_2Br_2 as an internal standard. TFP = tri(2-furyl)phosphine.



base-free condition and diminished yields were observed in the presence of bases (Table 1, entries 7, 8). Among the various phosphine ligands (Table S3), TFP delivered the best yields (Table 1, entries 9, 10). Copper salts are often required as activators to accelerate the C(O)−S cleavage.[22] After screening of various copper salts, we found Cu_2O was the best (Table 1, entries 11, 12). To further enhance the reactivity, a series of modified NBEs were prepared and examined. Compared to simple NBE, succinimide-containing NBEs with relatively bulkier substituents at the *N*-position were found to be more efficient (**NBE-1**−**4**). In contrast, the *N*-methyl substituted NBE (**NBE-5**) was inferior, and unprotected succinimide-derived NBE (**NBE-7**) was completely ineffective. To our delight, *N*-benzyl substituted NBE (**NBE-6**) delivered the best yield (85%). C5 amide-substituted NBEs (**NBE-8**−**10**) were less effective. Both 5-cyano substituted NBE (**NBE-11**) and 2-methyl ester substituted NBE (**NBE-12**) afforded trace amounts of desired products.

With the above optimal reaction conditions in hand, we first examined the thioester scope (Table 2). Polycyclic substrates containing a naphthyl or pyrenyl moiety afforded moderate to good yields (**4a**–**4c**, **4w**). *Ortho*-substituted thioesters (**1d**–**1s**) bearing both electron-donating and electron-withdrawing substituents were all compatible with the reaction conditions, and a variety of tri- and tetrasubstituted arenes were obtained in yields of 30-87% (**4d**–**4s**). Aryl bromides, which are commonly employed as aryl electrophiles in transition metal-catalyzed cross-couplings, were

Table 2. | Scope of aryl thioesters, olefins and benzyl bromides



Reaction conditions: **1** (0.1 mmol), **2** (0.15 mmol), **3** (0.3 mmol), $Pd(OAc)_2$ (0.01 mmol), TFP (0.025 mmol), **NBE-6** (0.15 mmol), Cu_2O (0.15 mmol), MeCN (2 mL), 120 °C, 12 h. Isolated yields are reported. [a]1-Iodopropane was used in the reaction.

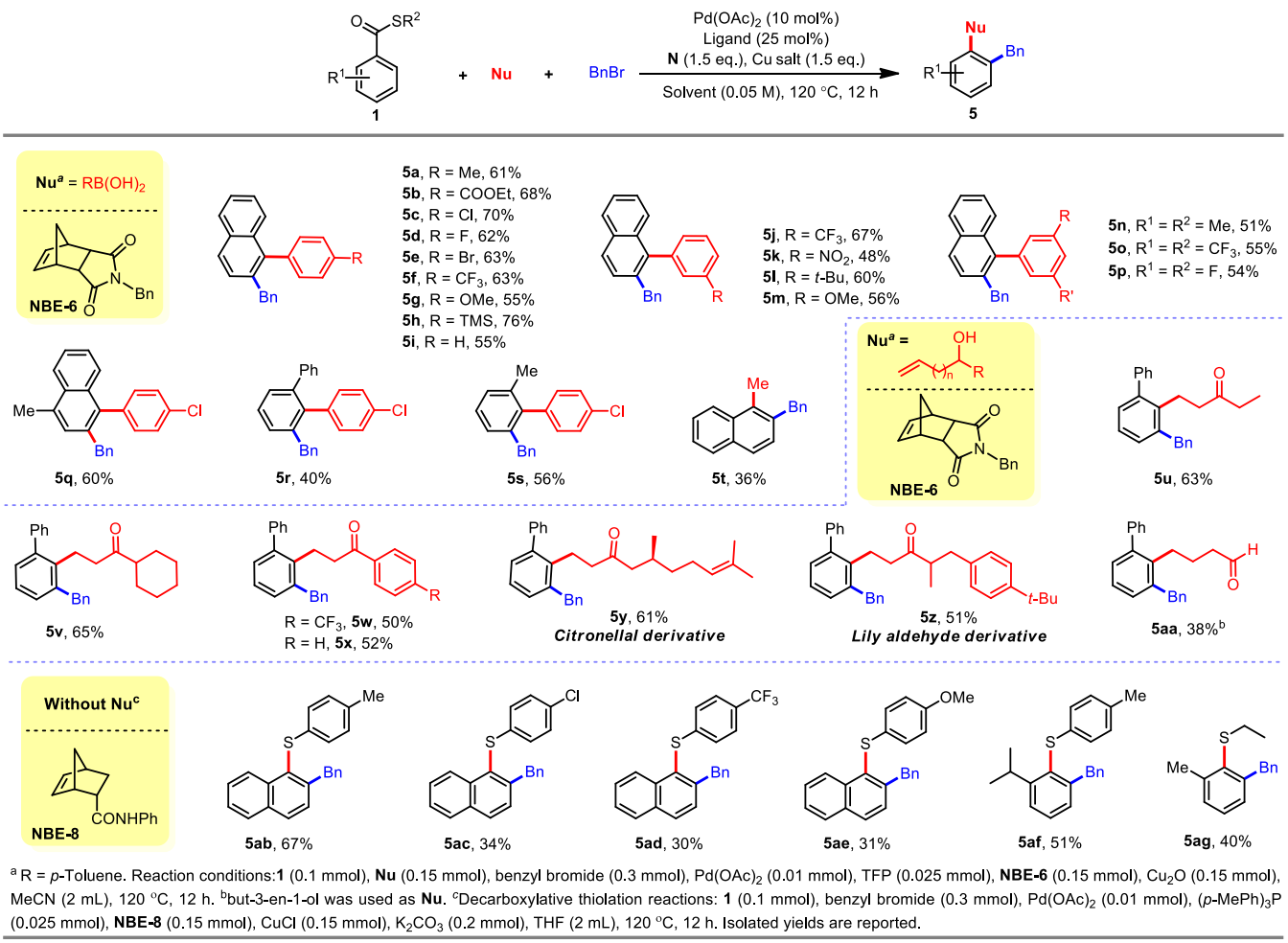
well tolerated, providing a handle for further functionalization. For *ortho*-unsubstituted aryl thioesters, dibenzylated products were obtained (**4t**–**4v**). Substrates bearing thiophene, pyridine and indole groups provided the desired products

in moderate yields (**4x**−**4ab**). The synthetic utility of our protocol was further showcased by the diversification of commercial pharmaceuticals, including aspirin, triflusal, mefenamic acid and repaglinide, furnishing the corresponding products (**4ac**−**4af**) in acceptable yields. The scope with respect to the olefin terminating reagents was next investigated. In addition to ethyl acrylate, other electron-deficient olefins, such as acrylonitrile (**4am**), 2-vinylpyridine (**4al**), and especially methyl methacrylate (**4ao**) could be smoothly introduced at the *ipso*-position. Gratifyingly, styrene and its derivatives, irrespective of bearing electron-donating or electron-withdrawing groups, could serve as effective terminating reagents (**4ak**−**4aj**). Intriguingly, when the electron rich and less reactive cyclohexyl vinyl ether was subjected to the reaction conditions, compound **4an** bearing branched alkenyl ethers was isolated in 53% yield. Regarding the scope of the electrophiles, benzyl bromides containing trifluoromethyl (**4ap**), fluorine (**4aq**), chlorine (**4ar**), bromine (**4as**) and methyl (**4at**) functionalities were all suitable substrates. *Ortho*-alkylated product **4au** was obtained in 40% yield when 1-iodopropane was employed.

To further illustrate the feasibility of this protocol, an *ortho*-benzylation/*ipso*-Suzuki cascade of aryl thioesters was carried out (Table 3). By employing thioester **1a,** benzyl bromide **3a** and arylboronic acid as the substrates, *ipso*-arylation occurred smoothly with moderate to good yields (**5a**−**5q**). Aryl boronic acids containing ester (**5b**), chloro (**5c**), bromo (**5e**), trimethylsilyl (**5h**) and nitro (**5k**) groups were well tolerated, offering additional opportunity for further structural elaboration. When *ortho*-substituted aryl thioesters were subjected to the reaction conditions, multi-substituted biaryl skeletons were obtained (**5r**, **5s**). *Ipso*-methylation product (**5t**) was synthesized in 36% yield using methylboronic acid as the terminating reagent. Subsequently, we employed allylic alcohol as the terminating nucleophile. A variety of ketone and aldehyde products were obtained *via* chain-walking strategies (**5u**−**5aa**).[23] It is worth noting that citronellal- and Lily aldehyde-derived allylic alcohols afforded the corresponding products **5y** and **5z** in 61% and 51% yields, respectively. Thioesters are versatile synthons for the synthesis of thioethers.[24] In the absence of terminating nucleophiles, *ortho*-C−H benzylation and *ipso*-decarbonylative thiolation of aryl thioester **1a** furnished the thioether product **5ab** in 67% yield after further optimization of the reaction conditions (see Supplementary Information). Aryl and alkyl thiols could be readily introduced to the *ipso*-positions, leading to polysubstituted thioethers in acceptable

yields (**5ab**–**5ae**).

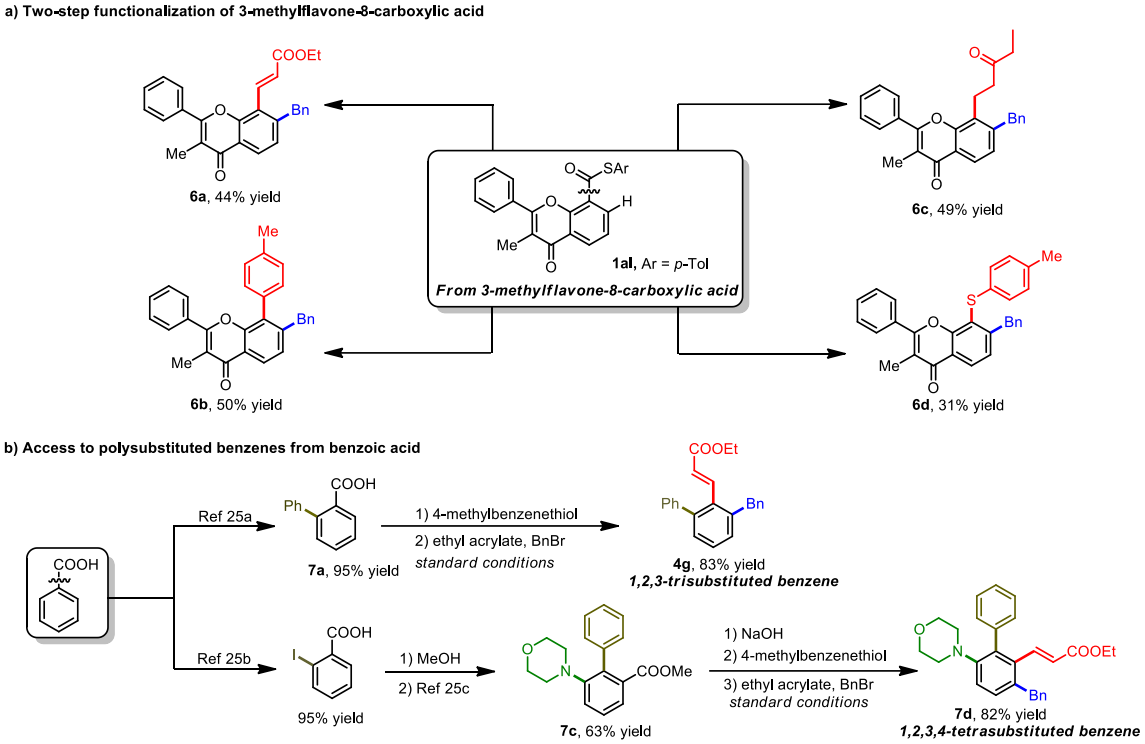
Table 3. | Scope of boronic acids, alkenyl alcohols and thiols



[a] R = *p*-Toluene. Reaction conditions:**1** (0.1 mmol), **Nu** (0.15 mmol), benzyl bromide (0.3 mmol), $Pd(OAc)_2$ (0.01 mmol), TFP (0.025 mmol), **NBE-6** (0.15 mmol), Cu_2O (0.15 mmol), MeCN (2 mL), 120 °C, 12 h. [b]but-3-en-1-ol was used as **Nu**. [c]Decarboxylative thiolation reactions: **1** (0.1 mmol), benzyl bromide (0.3 mmol), $Pd(OAc)_2$ (0.01 mmol), $(p\text{-MePh})_3P$ (0.025 mmol), **NBE-8** (0.15 mmol), CuCl (0.15 mmol), K_2CO_3 (0.2 mmol), THF (2 mL), 120 °C, 12 h. Isolated yields are reported.

The synthetic utility of this methodology was further demonstrated in the late-stage diversification of thioesters **1al** derived from 3-methylflavone-8-carboxylic acid, a drug used for the treatment of coronary heart disease (Figure 2a). *Ortho*-benzylation and a subsequent *ipso*-Heck- or Suzuki cascade of aryl thioester **1al** gave the product **6a** and **6b** in 44% and 50% yields, respectively. When pent-1-en-3-ol was used as a terminating nucleophile, an *ipso*-alkylated product **6c** was synthesized in 49% yield. In addition, thiolation terminated derivative **6d** was isolated in 31% yield. The carboxylate functionality is a practical directing group in transition metal-catalyzed C–H bond functionalization.[5] Thus, 1,2,3-trisubstituted arene **4g** and 1,2,3,4-tetrasubstituted arene **7d** were readily synthesized from benzoic acid by a combination of *ortho* C–H functionalization, the Catellani reaction, and Pd/NBE catalyzed *ipso*-alkenylation/*ortho*-benzylation (Figure 2b).[25]

Fig. 2 | Synthetic applications



Conclusion

In summary, we have developed a new protocol for the palladium-catalyzed, norbornene (NBE)-mediated decarbonylative Catellani reaction of aromatic thioesters. Our protocol utilizes widely available aryl carboxylic acids as feedstocks and featured redox-neutral and base-free reaction conditions. The termination step is flexible, which is demonstrated by Heck reaction, Suzuki coupling, alkylation and thiolation. Synthetic utility of this protocol is further highlighted by late-stage diversification of some pharmaceutical drugs. Carboxyl-directed *ortho* C−H iodination, Catellani reaction, and subsequent decarbonylative Catellani reaction allowed for the rapid construction of trisubstituted and tetrasubstituted arene from benzoic acid.

Data availability

The data supporting the findings of this study are available within the article and its Supplementary Information Files.

Acknowledgements We gratefully acknowledge the Shanghai Institute of Materia Medica, the Chinese Academy of Sciences, the NSFC (21772211), the Youth Innovation Promotion Association CAS (No. 2014229 and 2018293), the Science and Technology Commission of Shanghai Municipality (17JC1405000 and 18431907100), the Program of Shanghai Academic Research Leader (19XD1424600), the National Science & Technology Major Project "Key New

Drug Creation and Manufacturing Program", China (2018ZX09711002-006) for financial support.

Competing interests The authors declare no competing interests.

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