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Palladium-catalyzed ring-opening defluorinative cross-coupling of *gem*–difluorocyclopropanes with fluoromalonates or fluorobis(phenylsulfonyl)methane

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ABSTRACT

Fluorine locates a pivotal position in modern medicinal chemistry due to its distinctive impact on the properties of organic molecules. This work described an efficient divergent palladium/XPhos-catalyzed ring-opening defluorinative cross-coupling of *gem*–difluorocyclopropanes with less nucleophilic fluorinated malonates or fluorobis(phenylsulfonyl)methane. The corresponding difluoro malonates and 2,4-difluorobutadienyl sulfones were obtained in good yields, respectively. Besides, this protocol also enabled the modification of structurally diverse complex molecules.

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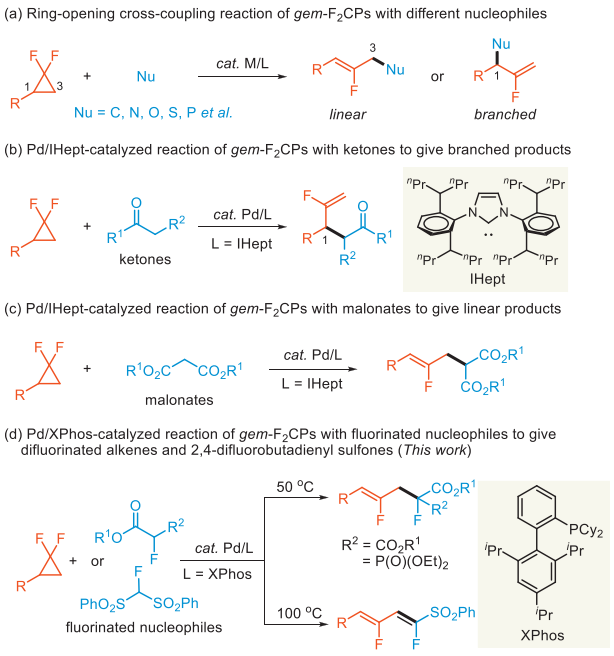
The C-F bond, recognised as the strongest single bond, has received considerable attention in the selective activation of the C-F bond, as it enhances the synthetic versatility of fluorinated compounds [1–5]. Significant progress has been achieved *via* different strategies, including Lewis acid activation [6], transition-metal catalysis [7–9], photocatalysis [10,11], and radical-mediated processes [12,13]. Despite these advances, the defluorinative functionalization of C(sp^3)-F bonds remains a formidable challenge, which is primarily attributed to the higher bond dissociation energy of C(sp^3)-F compared to that of C(sp^2)-F bonds [14–18]. *gem*–Difluorocyclopropanes (*gem*–F_2CPs) are recognized as valuable fluorinated synthons [19–21], playing a pivotal role in the synthesis of a range of valuable products *via* transition-metal-catalysed C-F bond activation [22–25]. Pioneering work in this field was performed by Fu and coworkers in 2015, in which β-F elimination was used as a critical step to facilitate C-F bond cleavage and subsequent cross-coupling reactions [26]. Inspired by this seminal work, a plethora of significant advancements have been made in this emerging field (Scheme 1a) [27–49]. Also noteworthy is the work of Li, Lv and coworkers, who achieved high branched regioselectivity by using ambident nucleophiles and bulky *N*-heterocyclic carbene (NHC) ligands [50–53].

It is well-known that carbonyls represent one of the most pivotal and versatile functional groups in organic synthesis. In 2021, our group demonstrated that simple ketones can function as ambident nucleophiles to react with *gem*–F_2CPs under the Pd/IHept catalytic system [54]. This transformation has been shown to be highly efficient and selective in the formation of branched products (Scheme 1b). Based on this result, our group has recently investigated the Pd/IHept-catalyzed ring-opening reactions of *gem*–F_2CPs with malonates and their derivatives, which has delivered a variety of linear 2-fluorinated products in good to excellent yields (Scheme 1c) [55].

The incorporation of a fluorine atom at the α-position of malonates could significantly enhance its acidity, which is also accompanied by an obvious decrease in nucleophilicity [56]. Our previously developed palladium/IHept catalytic system, which has been effective for the catalytic transformations of *gem*–F_2CPs with malonates, is not capable of facilitating the analogous transformations with fluoromalonates [54]. The development of alternative catalytic strategies to address this issue is necessary. We hypothesize that this problem may be addressed by utilizing phosphine as the auxiliary ligand on the assumption that the phosphine ligand's en-

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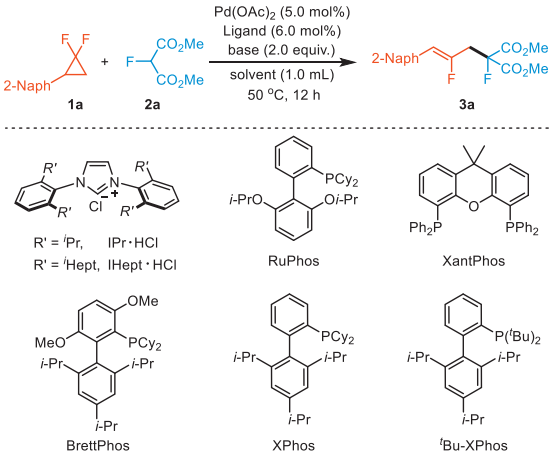
Scheme 1. Ring-opening defluorinative cross-coupling reactions of *gem*–F_2CPs with nucleophiles.

hanced π-acceptor capacity could reduce the electron density at the palladium center compared to observed with NHC ligand. Consequently, this reduction of electron richness at the palladium centre is expected to facilitate the ligand exchange process with the less nucleophilic fluoromalonates [57–65]. Herein, we report the Pd/XPhos-catalyzed defluorinative ring-opening cross-coupling of *gem*–F_2CPs with fluoromalonates (Scheme 1d). A range of difluorinated alkenes were obtained in moderate to good yields. Besides, 2,4-difluorobutadienyl sulfones were efficiently synthesized when 1-fluorobis(phenylsulfonyl)methane (FBSM) was used as the nucleophile. The utility of this protocol for the late-stage modification of complex molecules has also been demonstrated.

We began our study by investigating the coupling of *gem*–F_2CP **1a** with fluoromalonate **2a** using $Pd(OAc)_2$ as the catalyst and K_2CO_3 as the base in THF at 50 °C for 12 h (Table 1). As can be seen, the nature of ligands significantly influences the efficiency of this transformation. No desired product could be detected in the combination of $Pd(OAc)_2$ with *N*-heterocyclic carbene (NHC) ligand, leaving almost all the starting materials remaining unreacted (entries 1 and 2). Gratifyingly, the coupling product **3a** was obtained in 32 % yield when RuPhos was used as the ligand (entry 3). To improve the yield, a series of sterically-bulky phosphine ligands were evaluated for this reaction (entries 4–7), and XPhos behaved the best, delivering the desired product **3a** in 90 % yield (entry 6). A further evaluation of solvents such as toluene, EtOAc, 1,4-dioxane and MeCN were performed (entries 8–11), and MeCN turned out to be the optimal solvent, affording **3a** in 94 % yield (entry 11). Optimization of the base revealed that K_3PO_4 displayed the similar performance of K_2CO_3 (entry 12), while other bases including Cs_2CO_3, $KHCO_3$ or Na_2CO_3 were inferior (entries 13–15). Control experiments revealed that no target **3a** was detected in the absence of the palladium catalyst, ligand or base (entries 16–18).

With the optimal conditions established, we evaluated the scope of *gem*–F_2CPs using dimethyl 2-fluoromalonate **2a** as the nucleophile (Scheme 2a). Generally, a variety of *gem*–F_2CPs with phenyl rings bearing substituents at different positions (*para, ortho*, or *meta*) exhibited good reaction efficiency. For example,

Table 1
Optimization of the reaction conditions.[a]

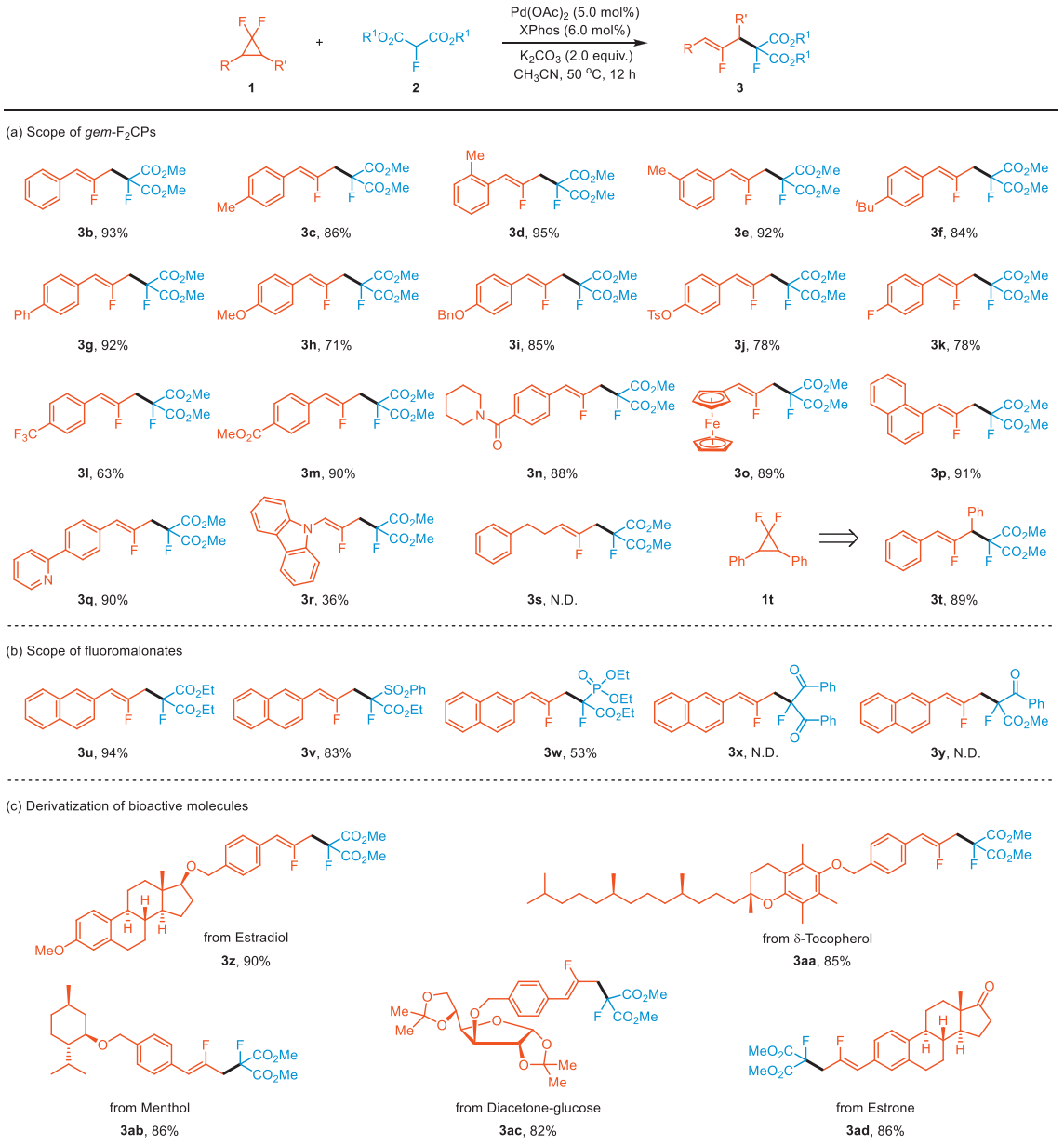


Entry	Ligand	Solvent	Base	Yield of **3a** (%)[b]
1	IPr·HCl	THF	K_2CO_3	0
2	IHept·HCl	THF	K_2CO_3	0
3	RuPhos	THF	K_2CO_3	32
4	XantPhos	THF	K_2CO_3	46
5	BrettPhos	THF	K_2CO_3	83
6	XPhos	THF	K_2CO_3	90
7	^{t}Bu-XPhos	THF	K_2CO_3	0
8	XPhos	Toluene	K_2CO_3	83
9	XPhos	EtOAc	K_2CO_3	85
10	XPhos	1,4-Dioxane	K_2CO_3	87
11	XPhos	MeCN	K_2CO_3	94 (91)
12	XPhos	MeCN	K_3PO_4	93
13	XPhos	THF	Cs_2CO_3	80
14	XPhos	MeCN	$KHCO_3$	26
15	XPhos	MeCN	Na_2CO_3	30
16	XPhos	MeCN	–	0
17	–	THF	K_2CO_3	0
18	XPhos	MeCN	–	0

[a] Reaction conditions: **1a** (0.1 mmol), **2a** (0.2 mmol), $Pd(OAc)_2$ (5.0 mol%), ligand (6.0 mol%), base (0.2 mmol), solvent (1.0 mL), 50 °C, 12 h under Ar unless otherwise noted.
[b] Reported yields were based on **1a** and determined by ^{1}H NMR using mesitylene as an internal standard.

the desired products **3b**-**3e** were obtained in 86 %, 95 % and 92 % yields, respectively, when *para*-, *ortho*- and *meta*-methyl-substituted *gem*–F_2CPs were tested under the standard conditions. *gem*–F_2CPs with electron-donating substituents (-^{t}Bu, -Ph, -OMe, -OBn) afforded products **3f**-**3i** in 71 %–92 % yields, and those with electron-withdrawing substituents (-OTs, -F, -CF_3, -CO_2Me, -amide) also proved effective, affording the corresponding products **3j**-**3n** in 63 %–90 % yields. Further attempts were made to expand the substrate scope. Reactions of *gem*–F_2CPs containing ferrocene, 1-naphthalene and pyridine moieties were also tolerated, resulting in the formation of the desired products **3o**-**3q** in 89 %–91 % yields. A carbazole group in the *gem*–F_2CP was feasible but resulted in a slightly lower yield (**3r**, 36 %). Alkyl-substituted *gem*–F_2CP (**3s**) was incompatible in this transformation, likely due to the thermodynamic unfavourability of the corresponding allyl metal intermediates compared to the aryl substitution. Fortunately, 1,2-disubstituted *gem*–F_2CP was suitable, as evidenced by the formation of product **3t** in 89 % yield.

We next explored the scope of fluorinated nucleophiles (Scheme 2b). Fluorinated diethyl malonate and phenylsulfonyl-acetate reacted efficiently with *gem*–F_2CP **1a**, giving the corresponding products **3u** and **3v** in 94 % and 83 % yields, respectively. Diethoxy-phosphoryl-fluoroacetate also proved to be a competent reaction partner under the optimal conditions, affording the desired product **3w** in 53 % yield. However, fluorinated 1,3-diketones



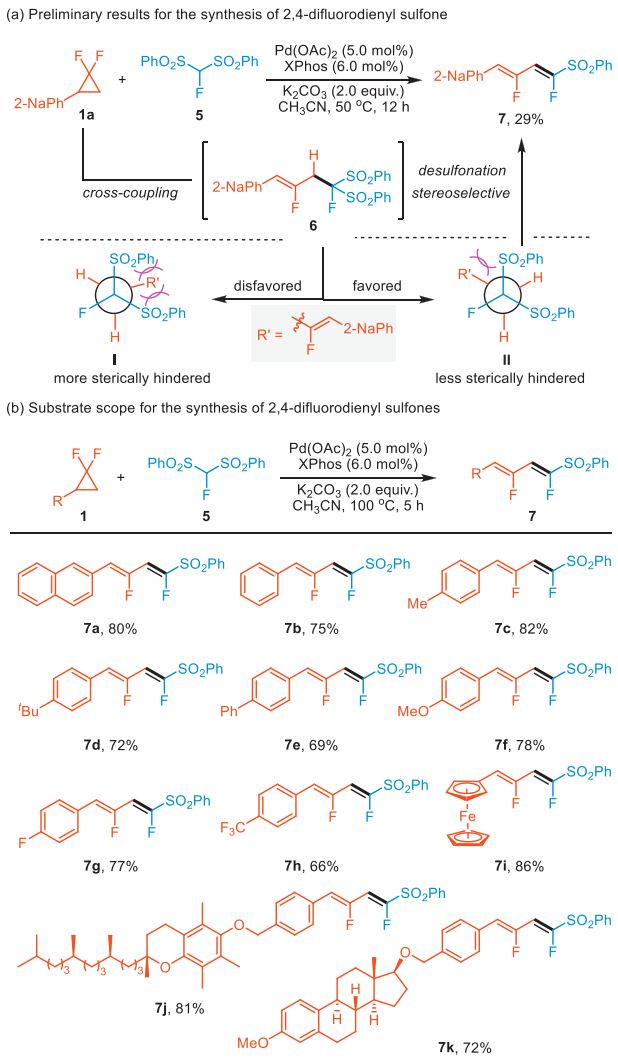
Scheme 2. Scope of substrates. Reaction conditions: **1** (0.1 mmol), **2** (0.2 mmol), $Pd(OAc)_2$ (5.0 mol%), XPhos (6.0 mol%), K_2CO_3 (0.2 mmol), CH_3CN (1.0 mL), under Ar at 50 °C for 12 h. Isolated yields based on **1**.

and keto-esters were not suitable for this transformation, resulting in almost the complete recovery of the starting materials. Notably, *gem*–F_2CPs derived from estradiol, δ-tocopherol, menthol, diacetone glucose and estrone all reacted smoothly with fluorinated dimethyl malonate **2a**, delivering the corresponding products **3z**-**3ad** in 82 %–90 % yields, as shown in Scheme 2c. These results highlight the potential of this protocol for the late-stage modification of bioactive and complex molecules.

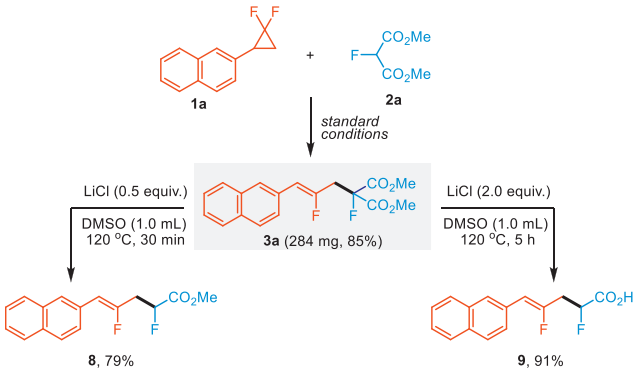
During the study of fluoro nucleophiles, an intriguing result was observed when fluorobis(phenylsulfonyl)methane was applied as the nucleophile [66–72]. The desired fluorobis(phenylsulfonyl)methylated product **6** was detected in only trace amount, while an unexpected product 2,4-difluorobutadienyl sulfone **7** was obtained in 29 % yield (Scheme 3a). When the reaction temperature was elevated to 100 °C, compound 6 was completely disappeared while 2,4-difluorobutadienyl sulfone **7** could be obtained in 80 % yield in a highly stereoselective manner. This result indicated that **7** should be derived from **6** *via* further desulfonation [73]. To elucidate the origin of the stereoselectivity of the desulfonation process, a possible configuration analysis is proposed. It is apparent that configuration **I** was disfavoured due to the more steric hinderance, whereas configuration **II** would undergo desulfonation with *trans*-configuration of the sulfone group and fluoroalkene moiety [74].

We then investigated the substrate scope for the synthesis of 2,4-difluorodienyl sulfones (Scheme 3b). *gem*–F_2CPs bearing either the electron-donating (-Me, -^{t}Bu, -Ph, -OMe) or electron-withdrawing (-F, -CF_3) substituents on the phenyl ring reacted smoothly with FBSM to afford the corresponding products **7a**-**7h** in 66 %–82 % yields. A ferrocene-substituted *gem*–F_2CP also reacted successfully under standard conditions. Besides, we were able to efficiently introduce the 2,4-difluorodienyl sulfone moiety onto δ-tocopherol and estradiol, and the desired products **7j** and **7k** were obtained in 81 % and 72 % yields, respectively.

We also conducted a 1.0 mmol-scale experiment with the model substrates under the optimal reaction conditions, providing **3a** in



Scheme 3. Reaction of *gem*–F_2CPs with FBSM. Reaction conditions: **1** (0.1 mmol), **5** (0.2 mmol), $Pd(OAc)_2$ (5.0 mol%), XPhos (6.0 mol%), K_2CO_3 (0.2 mmol), CH_3CN (1.0 mL), under Ar at 100 °C for 5 h. Isolated yields based on **1**.



Scheme 4. Scale-up preparation of **3a** and synthetic transformations.

85 % yield (Scheme 4). Subsequent synthetic transformations of the product were performed. For example, Krapcho decarboxylation [75] of **3a** using 0.5 equiv. of LiCl for 30 min afforded the difluorinated monoester **8** in 79 % yield, while treatment of **3a** with 2.0 equiv. of LiCl and extending the reaction time to 5 h resulted in the difluorinated carboxylic acid **9** in 91 % yield.

In summary, we have developed a divergent Pd/XPhos-catalyzed ring-opening defluorinative cross-coupling methodology involving *gem*–difluorocyclopropanes and fluorinated malonates or fluorobis(phenylsulfonyl)methane. A variety of difluoromalonates and 2,4-difluorobutadienyl sulfones were synthesized with excellent stereoselectivity and in good to excellent yields. Furthermore, the versatility of this protocol is exemplified by its successful application to the modification of structurally diverse bioactive molecules.

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

Junqi Su: Investigation, Data curation. **Wenhao Liu:** Investigation. **Jianjun Wang:** Writing – review & editing. **Weifen Luo:** Writing – review & editing. **Yangyang Ma:** Writing – review & editing. **Leiyang Lv:** Writing – review & editing, Writing – original draft, Project administration, Funding acquisition, Conceptualization. **Zhiping Li:** Writing – review & editing.

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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.2025.111288.

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