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P^{III}/P^{V}-catalytic platform enabling *N*-fluoro-thiocarbonylation of diaryl amines for modular synthesis of tertiary *N*-trifluoromethylamines

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ABSTRACT

Tertiary N–CF_3 compounds have attracted intensive attention due to their great significance in discovery of new lead compounds, however, the synthesis of tertiary diaryl N–CF_3 derivatives is still challenging. Herein, we successfully edit diaryl N–H into thiocarbamoyl fluorides with trifluoromethanesulfonyl chloride by use of a P^{III}/P^{V} redox catalyst, leading to the formation of series of diaryl N–CF_3 with silver fluoride. In addition, this process is also highly efficient to dialkyl and alkylaryl secondary amines. The mechanism investigation illustrated that the use of hydrosilane is crucial to the success of this transformation. It acts as both terminal reductants to cycle the P^{III}/P^{V} couple and fluoride acceptor to promote the reaction between less reactive amine and thiocarbonyl difluoride intermediate.

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Diaryl amine structures are key components in pharmaceuticals, biologically active compounds, and material science (Fig. 1A) [1–11]. Notably, due to their common occurrence in kinase inhibitors like imatinib (anti-cancer drug), diaryl amines are often referred to as "kinase-privileged fragments" [12–14]. The incorporation of fluorinated groups has proven to be an effective strategy for modifying molecular properties, including lipophilicity, solubility, electrophilicity, metabolic stability, and molecular conformation [15–19]. Therefore, transformation of the ubiquitous N–H bond in diaryl amines to an N–CF_3 is pivotal to unlock new properties and functionalities in complex molecules, allowing to extend the boundaries of pharmaceutical and material innovation [20–24]. However, the conversion of N–H motifs to R_2N–CF_3 compounds remains insufficiently investigated [25,26].

Existing direct N–H trifluoromethylation methods, typically employing Togni and Ruppert-Prakash reagents, face challenges related to generality and specific substrate types, such as azoles [27–30], imines [31–33], certain amides [34,35], and selected dialkyl/alkylaryl amines [36]. A promising alternative involves stepwise sequences in functional group interconversion, gaining attention over the past decade through different retrosynthetic approaches [37–43]. A notable advancement was introduced by Schoenebeck, who developed a one-pot method for synthesizing trifluoromethyl amines (R_2N–CF_3) from N–H motif. Their method involved the formation of *N*-thiocarbonyl fluoride (R_2N–CSF) intermediate with solid $Me_4N^+SCF_3^-$ and the subsequent desulfurfluorination (Fig. 1B, right) [44,45]. Later on, Xiao and Jiang, as well as Yi independently realized the one-pot syntheses of secondary R_2N–CF_3 from free amines by utilizing the intermediacy of R_2N–CSF [46–48]. Tlili's recent work demonstrated a new synthetic method of R_2N–CSF through the fluorinative desulfurization of CS_2 by developing versatile SF_5-based reagent [49,50]. However, these approaches usually benefit dialkyl and alkylaryl NH substrates with more nucleophilic and basic properties, such as cyclopentylamine (p*K*aH = 11.27), Me_2NH (p*K*aH = 10.71) and PhMeNH (p*K*aH = 4.85) [51–53]. In contrast, diaryl amine derivatives present a challenge in these developed systems due to their electron-deficient and weakly basic N–H sites, exemplified by Ph_2NH (p*K*aH = 0.79) [54]. Furthermore, transformations involving significantly less basic substrates like carbazoles (p*K*aH = −4.87) remain untapped [55]. Encouraged by Radosevich's recent works [56], we herein address the challenges on tertiary N–CF_3 formation through diaryl amines by developing a synergistic organophosphorus(III)-silane catalytic *N*-fluoro-thiocarbonylation (Fig. 1C). The mechanistic insight revealed that the reaction undergoes a P^{III}/P^{V} redox cycle. Hydrosilane serves two critical functions: (1) as a terminal reductant to cycle the P^{III}/P^{V} redox pair and (2) acting as an effective fluoride acceptor, thus enhancing the reaction between the thiophosgene intermediate and inert secondary amines. Notably, recently reported stability of R_2N–CSF compounds in the presence of different amino acids or serums in PBS, allows for their further applications under near-physiological conditions [57].

We initiated the synthesis of thiocarbamoyl fluorides *via* P^{III}/P^{V} catalysis by reacting the challenging substrate carbazole **1a** with

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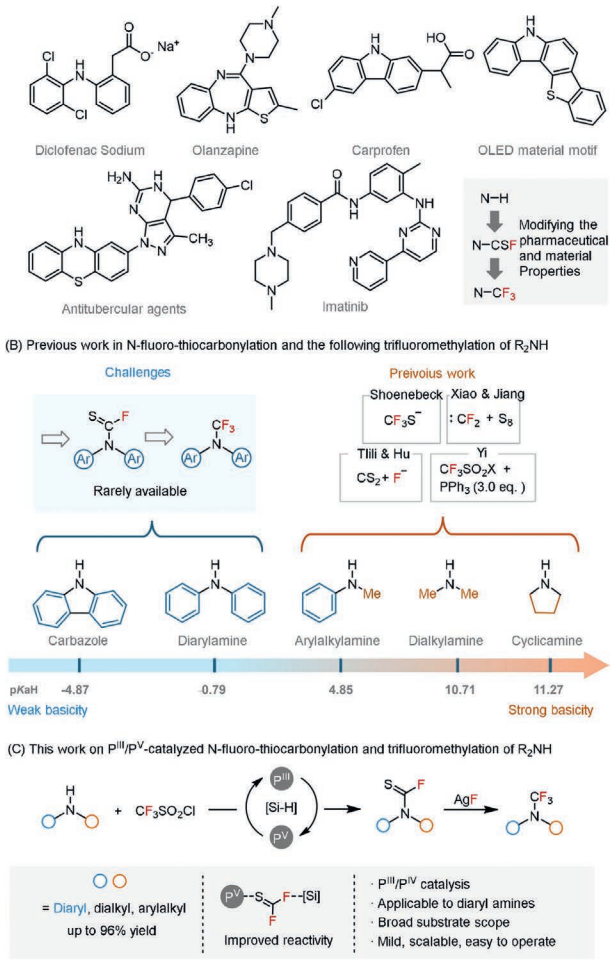
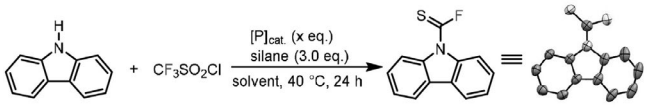


Fig. 1. The background and our study on *N*-fluoro-thiocarbonylation and the following *N*-trifluoromethylation of diaryl amines.

TfCl **2** and phenylsilane (Table 1). The reaction afforded desired product (carbazole-CSF) in only trace amount at 40 °C in the presence of 0.25 equiv. of triphenylphosphine or tri(*o*-tolyl)phosphine in DCE, while tri-*n*-butylphosphine gave 67 % yield (entries 1–3). Replacing the catalyst with a bulkier tricyclohexylphosphine (Cy_3P) enhanced the yield to 95 %, whereas reducing the catalyst loading to 0.1 equiv. decreased the yield to 78 % (entries 4 and 5). Alternative solvents such as THF, acetone, DCE, and toluene resulted in lower yields (entries 6–9). Further studies revealed that the corresponding tricyclohexylphosphine oxide ($Cy_3P{=}O$) also facilitated the formation of the desired product with a yield of 72 %, indicating that the reaction proceeds *via* a P^{III}/P^{V} redox cycle (entry 10). Control experiments in the absence of organophosphorus(III) catalyst or hydrosilane showed no reactivity, confirming the necessity of both components for this catalytic *N*-fluoro-thiocarbonylation of carbazole (entries 11 and 12). The structure of **3a** was verified through X-ray diffraction analysis (CCDC: 2377182).

Having established optimized reaction conditions, we proceeded to explore the scope of sequential *N*-fluoro-thiocarbonylation and desulfurization-fluorination reactions using silver fluoride (Scheme 1). This protocol was effectively applied to carbazoles bearing various electron-rich, -neutral, or -deficient functional groups, such as alkyl, alkoxy, phenyl, halogen, nitro, ester, and trifluoromethyl groups, affording carbazole N–CSF and $N–CF_3$ products (**3a**-**3p** and **4a**-**4p**). Substrates featuring reducible functionalities, such as ester and nitro groups, successfully formed the desired products (**3k**, **3m**, **4k**, **4m**). The tolerance of aryl

Table 1
Reaction conditions optimization.[a]

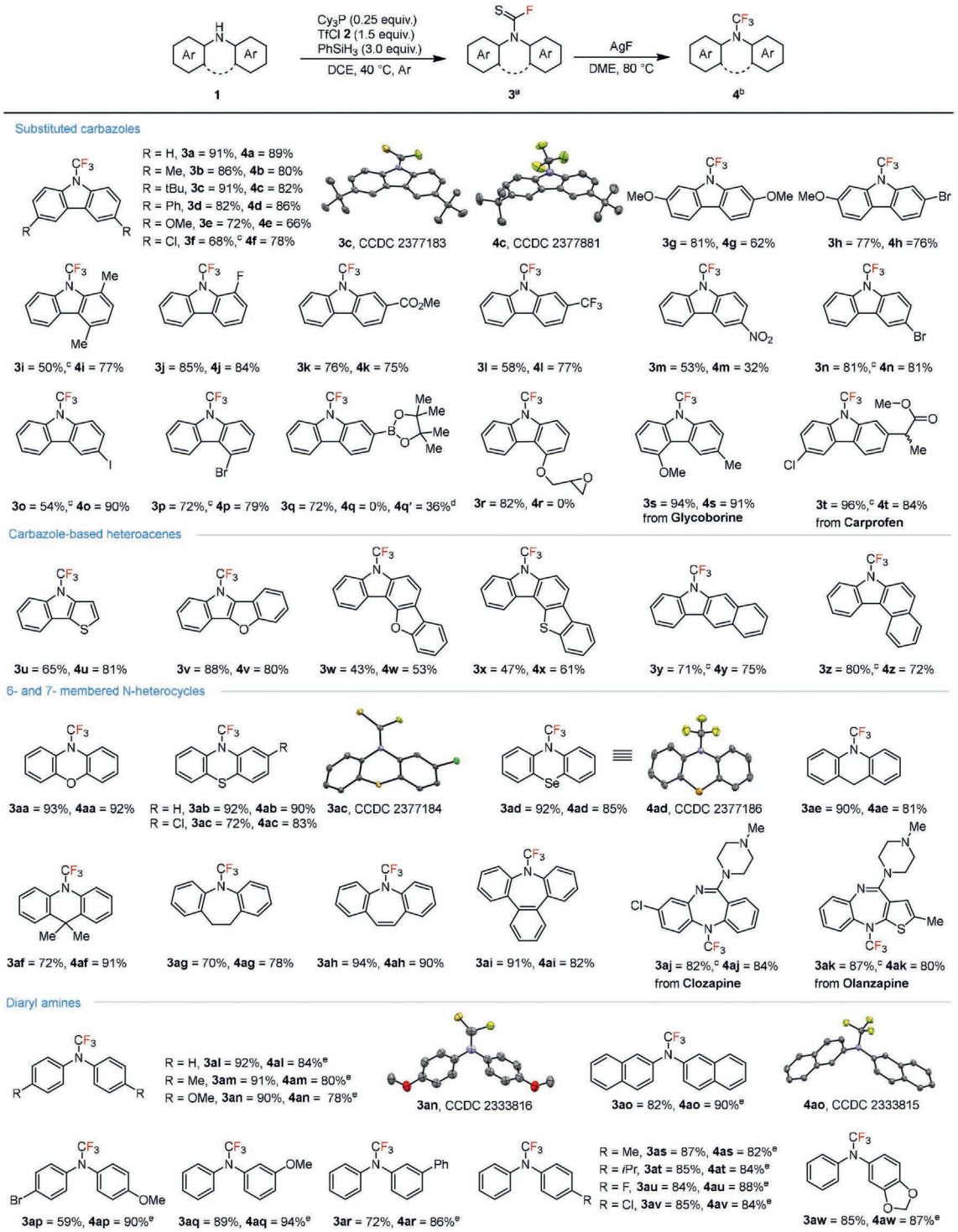


Entry	$[P]_{cat.}$ (eq.)	Silane	Solvent	Yield (%)[b]
1	Ph_3P (0.25)	$PhSiH_3$	DCE	2
2	$(o\text{-tolyl})_3P$ (0.25)	$PhSiH_3$	DCE	1
3	nBu_3P (0.25)	$PhSiH_3$	DCE	67
4	Cy_3P (0.25)	$PhSiH_3$	DCE	95
5[c]	Cy_3P (0.10)	$PhSiH_3$	DCE	78
6	Cy_3P (0.25)	$PhSiH_3$	THF	46
7	Cy_3P (0.25)	$PhSiH_3$	Acetone	56
8	Cy_3P (0.25)	$PhSiH_3$	MeCN	74
9	Cy_3P (0.25)	$PhSiH_3$	Toluene	22
10[d]	$Cy_3P{=}O$ (0.25)	$PhSiH_3$	DCE	72
11	Cy_3P (0.25)	None	DCE	0
12	None	$PhSiH_3$	DCE	0

[a] Reaction was carried out by using **1a** (0.25 mmol), **2** (0.375 mmol, 1.5 equiv.), $[P]_{cat.}$ (0–0.25 equiv.), phenylsilane (0–3.0 equiv.), in solvent at 40 °C for 24 h under Ar.
[b] Yields were determined by GC–MS using naphthalene as an internal standard.
[c] For 36 h.
[d] Phenylsilane (1.25 mmol, 5.0 equiv.) was used.

halide, especially bromide and iodide, allows further elaboration of the $N–CF_3$ products (**3n**-**3p** and **4n**-**4p**). Remarkably, sensitive groups like epoxy and Bpin could be tolerated in the *N*-fluoro-thiocarbonylation stage (**3q**-**3r**), although they did not proceed to form trifluoromethylated products. Specifically, for transformation of **3q**, a homocoupling of arylboronic ester occurred in the presence of silver salt, resulting in a trifluoromethyl carbazole dimer intermediate **4q′** (see Supporting information for details). These results encouraged us to examine carbazole-based heteroacenes, which are commonly utilized in organic light-emitting diodes (OLEDs) and organic field-effect transistors (OFETs) [58–60], were successfully converted into $R_2N–CSF$ and the following $R_2N–CF_3$ products (**3u**-**3z** and **4u**-**4z**) in moderate to good yields. The low solubility of both N–H substrate and the corresponding products were probably attributed to the diminished yields, caused by the planarity of these molecules. Additionally, this protocol proved compatible with other NH-containing heterocycles such as phenoxazine, phenothiazine, phenoselenazine, dihydroacridine and dibenzo[*b*,*f*]azepine (**3aa**-**3ai** and **4aa**-**4ai**). Furthermore, the more nucleophilic and basic structures based on *N*,*N*-diphenyl amines demonstrated robust reactivity as expected. Both symmetrical and asymmetrical diphenyl amines with either electron-rich or electron-poor substituents yielded the desired compounds efficiently (**3al**-**3aw** and **4al**-**4aw**). To illustrate the practical application of this method in late-stage functionalization of complex scaffolds, natural products and drug molecules were involved, such as Glycoborine (**3s** and **4s**), Carprofen methyl ester (**3t** and **4t**), Clozapine (**3ak** and **4ak**), and Olanzapine (Zyprexa). The reactions produced the desired $R_2N–CSF$ and $R_2N–CF_3$ compounds in good to excellent yields without altering the core structures of these molecules.

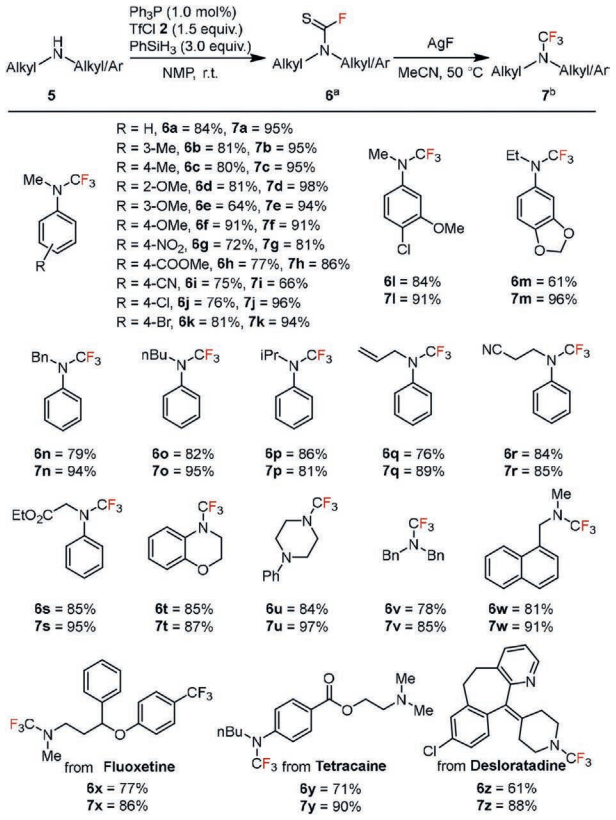
When employing more nucleophilic and basic dialkyl and arylalkyl amines, the P^{III} catalyst was effectively replaced with Ph_3P at a reduced loading of 1 mol% in NMP at room temperature (Scheme 2). This adjustment allowed various alkylaryl and dialkyl amines to undergo *N*-fluoro-thiocarbonylation, yielding the corresponding products in good to excellent quantities while maintaining compatibility with various functional groups (**6a**-**6w**). Subsequent desulfurization-fluorination steps using AgF were efficiently carried out in acetonitrile (**7a**-**7w**). This process was then applied to phar-



Scheme 1. Scope of diaryl amines for synthesizing thiocarbamoyl fluorides and the tertiary N–CF_3 products. [a] Reaction conditions: substrate **1** (0.25 mmol), Cy_3P (0.25 equiv.), TfCl **2** (1.5 equiv.), $PhSiH_3$ (3.0 equiv.), DCE (1.0 mL), 40 °C, 24 h, Ar, isolated yield. [b] Substrate **3** (0.25 mmol), AgF (5.0 equiv.), DME (1.0 mL), 80 °C, 12 h, Ar, isolated yield. [c] 48 h. [d] 9,9′-Bis(trifluoromethyl)-9*H*,9′*H*-2,2′-bicarbazole **4q′** was obtained. [e] Substrate **3** (0.2–0.5 mmol), AgF (4.5 equiv.), MeCN (1.5 mL), 50 °C, 5 h. Please see Supporting information for experimental details.

maceutical compounds such as Fluoxetine, Tetracaine, and Desloratadine, producing *N*-trifluoromethylated derivatives with satisfactory yields (**6x**-**6z** and **7x**-**7z**).

The unique structure of thiocarbamoyl fluorides not only facilitates the transformation to attractive N–CF_3 compounds, but also serves as a versatile linker to connect different bio-active scaffold, leveraging fluoride's role as a leaving group (Scheme 3). To demonstrate the synthetic application of thiocarbamoyl fluorides, we initially scaled up the synthesis of carbazole-CSF **3a** to 12.0 mmol, maintaining a high yield of 92 % (Scheme 3A). Furthermore, the fluoride on **3a** was effectively replaced with various phenols using Cs_2CO_3 in acetone. This substitution involved natural bioactive molecules such as Eugenol, endogenous hormones like Estrone, and other compounds including Coumarin and Mal-

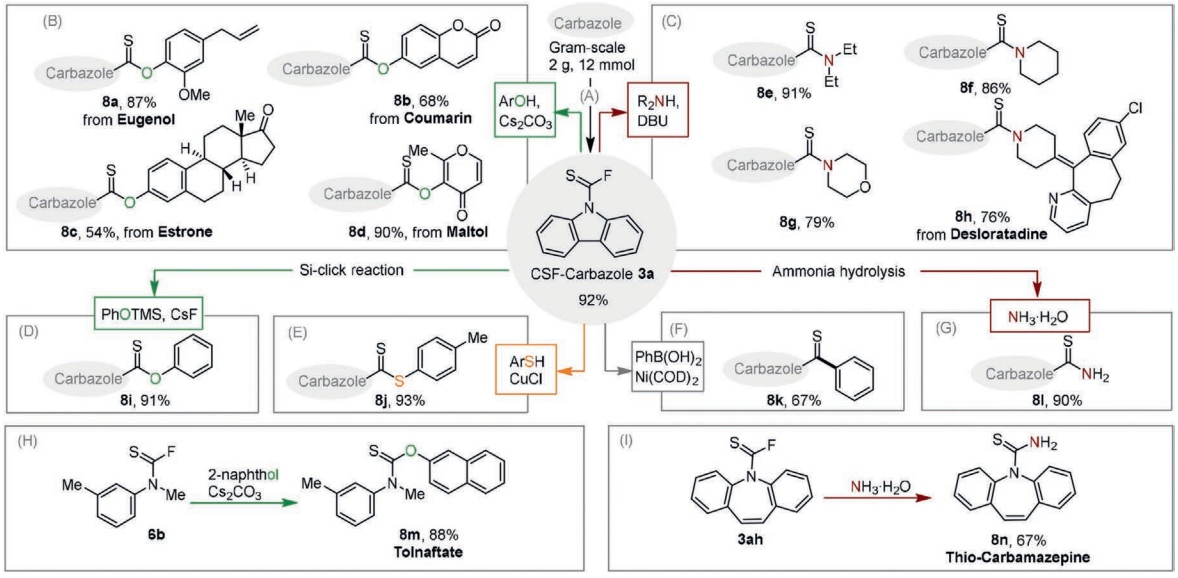


Scheme 2. Scope of other secondary amine derivatives. [a] Secondary amine **5** (0.5 mmol), Ph_3P (1.0 mol%), TfCl **2** (1.5 equiv.), $PhSiH_3$ (3.0 equiv.), in NMP, r.t, 5 h, under air, isolated yield. [b] R_2N–CSF **6** (0.3–0.5 mmol), AgF (4.5 equiv.), MeCN (1.5 mL), 50 °C, 5 h, Ar, isolated yield.

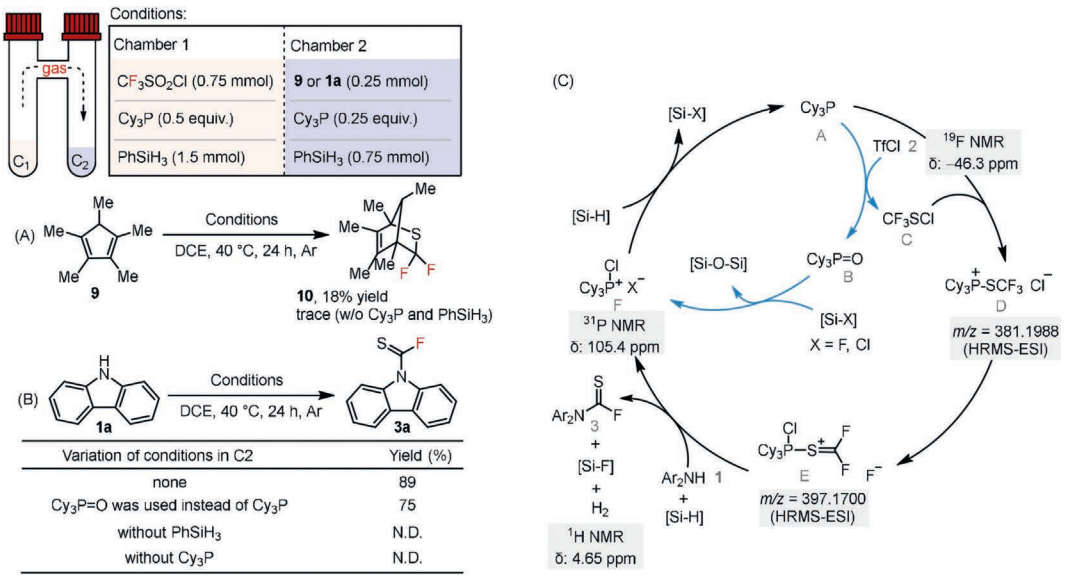
tol (Scheme 3B, **8a**-**8d**). Additionally, carbamothioate **8i** was synthesized through a Si-click reaction utilizing PhOTMS (Scheme 3D). Remarkably, the antifungal drug Tolnaftate **8m** was afforded with an 88 % yield starting from *N*-methyl-*m*-toludine (Scheme 3H). Employing aliphatic amines or ammonia as nucleophiles, including Desloratadine, led to the creation of several notable carbothioamides (**8e**-**8h** and **8l**), and thio-carbamazepine **8n** was synthesized from **3ah** through the same reaction conditions (Schemes 3C, G and I). The carbamoyl fluoride proved reactive towards thiophenol in the presence of a catalytic amount of CuCl, facilitating its conversion to carbamodithioate **8j** (Scheme 3E). Finally, an efficient nickel-catalyzed Suzuki cross-coupling reaction was utilized to produce thioamide **8k** (Scheme 3F).

During the reaction, we observed gas formation, which was suggested as molecular hydrogen ($\delta(^1H) = 4.65$) (Fig. S1B in Supporting information). To explore further insight of the roles of organophosphorus(III) and silicon species in the reaction pathway, a two-chamber reactor was employed. Initially, TfCl, Cy_3P, and $PhSiH_3$ were mixed in chamber 1, and trace amount of bridged product **10** was captured using pentamethylcyclopentadiene **9** in chamber 2. Meanwhile, the yield of **10** increased to 18 % in the presence of Cy_3P and $PhSiH_3$ in chamber 2, indicating that the generation of thiocarbonyl difluoride was promoted by organophosphorus(III) and hydrosilane (Scheme 4A). To further confirm the intermediacy of thiocarbonyl difluoride, we added carbazole in chamber 2 along with Cy_3P and $PhSiH_3$, resulting product **3a** in an 89 % yield (Scheme 4B). A slightly lower yield of 75 % was obtained when replacing Cy_3P with $Cy_3P{=}O$. Additional control experiments without Cy_3P or $PhSiH_3$ did not produce the target product, underscoring their critical roles in the reaction of thiophosgene and the less nucleophilic diaryl amines. Further NMR spectroscopy analysis of the catalytic reaction mixture revealed that trifluoromethylsulfenyl chloride **C** ($\delta(^{19}F) = -46.3$) and chlorophosphonium salt **F** ($\delta(^{31}P) = 105.4$) were present in the reaction (Figs. S4 and S5 in Supporting information); HRMS-ESI analysis revealed peaks at *m/z* 381.1988 and 397.1700, which were proposed to be the trifluoromethyl thiophosphonium cation **D** and an adduct **E** of the phosphonium salt with thiocarbonyl difluoride, respectively (Fig. S6 in Supporting information).

Based on the above results, we propose a plausible reaction pathway for the *N*-fluoro-thiocarbonylation step as depicted in Scheme 4C. The reaction initiates with sequential deoxygenation of TfCl **2** facilitated by P^{III} **A**. The reduced intermediate sulfenium ion **C** forms Lewis acid-base adduct with P^{III} [61], which then decomposes into **E**. The reaction of **E** with diarylamine leads the final product with the release of molecular hydrogen, supported by the high fluorophilicity of silicon species. This step is corroborated by the detection of a Si-F species [62], evidenced by ^{19}F NMR detection of the reaction mixture (Fig. S4 in Supporting information).



Scheme 3. The synthetic application of thiocarbamoyl fluoride compounds. Please see Supporting information for experimental details.



Scheme 4. (A) Detection of thiophosgene with **9** using two-chamber reactor. (B) Control experiments with **3a** using two-chamber reactor. (C) Proposed reaction pathway for the *N*-fluoro-thiocarbonylation stage in organophosphorus-silicon catalytic system. Chamber 1: TfCl **2** (0.75 mmol), Cy_3P (0.5 equiv.), $PhSiH_3$ (1.5 mmol), DCE (2.0 mL), 40 °C, 24 h, Ar. Chamber 2: **9** or **2a** (0.25 mmol), Cy_3P (0.25 equiv.), $PhSiH_3$ (0.75 mmol), DCE (1.0 mL), 40 °C, 24 h, Ar, isolated yield. Please see Supporting information for experimental details.

The fluorosilane species suggests an increase in the Lewis acidity at the silicon center, further enhancing its affinity for fluoride. In the other cycle, the resulting P^V=O **B** was speculated to convert into phosphonium cation **F**, facililated by halosilane due to its enhanced oxophilicity [63,64]. Finally, Si-H species reduce the released halophosphonium salt liberating P^{III} to complete the catalytic cycle.

In summary, we have disclosed catalytic *N*-fluoro-thiocarbonylation of secondary amines with trifluoromethanesulfonyl chloride and hydrosilane through a P^{III}/P^V couple, facilitating efficient conversion of N–H into R_2N–CSF and leading to R_2N–CF_3 with silver fluoride. This work addresses the gap of diaryl amine derivatives, which pose a challenge in previous works due to their electron-deficient and weakly basic characteristics. The mechanistic research confirms the essential roles of the P^{III}/P^V catalyst and silane species. This procedure opens new and efficient pathways for the synthesis of structurally diverse tertiary N–CSF and N–CF_3 compounds, allowing for various applications in chemistry and materials.

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

Dongke Zhang: Methodology, Investigation, Data curation. **Li-Ao Ding:** Validation, Investigation. **Qiuyu Xiang:** Methodology, Investigation. **Zhuojun Li:** Methodology, Investigation. **Qian Wu:** Writing – review & editing, Writing – original draft, Validation, Supervision, Project administration, Funding acquisition, Data curation, Conceptualization.

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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.2025.111461.

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