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Authors: Yafei Liu, Long Lu, and Qilong Shen

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Monofluoromethyl-Substituted Sulfonium Ylides: Electrophilic Monofluoromethylating Reagents with Broad Substrate Scopes

Yafei Liu,[a] Long Lu*[a] and Qilong Shen*[a]

Abstract: Two electrophilic monofluoromethylating reagents *S*-monofluoromethyl-*S*-phenyl-bis(carbomethoxy) methylide sulfonium ylide **3a** and *S*-monofluoromethyl-*S*-(4-nitrophenyl)-bis(carbo - methoxy) methylide sulfonium ylide **3b** and their reactions with a variety of nucleophiles such as alcohols and malonate derivatives, sulfonic and carboxylic acids, phenols, amides, and nitrogen of heteroarenes under mild conditions were described. Mechanistic studies by employing deuterated reagents **3a**-D_2/**3b**-D_2 suggest that these monofluoromethylating reactions proceed via an electrophilic substitution pathway.

Over the past 20 years, fluorine and its magic "fluorine effect", which was coined as "a small atom with a big ego",[1] have been well recognized in the fields of pharmaceutical and agrochemical industry.[2] Nowadays, strategic incorporation of a fluorine atom or a fluoroalkyl group at different stage of drug development has become a routine practice for new drug discovery.[2a-b,f] Thus, the high demands in these fields have urged organic chemists to develop general, efficient reagents and methods for the preparation of fluorinated compounds.[3] Consequently, in recent years, elegant methods for trifluoromethylation or difluoromethylation have emerged, which now allows for the effective late-stage installation of a trifluoromethyl (CF_3)[4] or a difluoromethyl (CF_2H)[5] on the target molecules. In contrast, methods that are capable of direct introduction of their analogous monofluoromethyl group (CH_2F) are far less developed.[6-7]

Direct electrophilic monofluoromethylation through nucleophilic attack of an appropriate nucleophile with an electrophilic monofluoromethylating reagent represents one of potentially applicable strategies for the introduction of the monofluoromethyl group. Early studies on direct electrophilic monofluoromethylation typically involved the use of fluoromethanol[8] and its derivative fluoromethyl triflate[9] or fluoromethyl halides ($ClCH_2F$[10] or ICH_2F[11]). However, preparation of fluoromethanol and fluoromethyl triflate typically required special equipments or harsh conditions and the scope for their reactions was rather limited. On the other hand, even though chlorofluoromethane reacted with a variety of nucleophiles, it is an ozone depleting gas, and its future use is thus questionable, while iodofluoromethane was mainly used to synthesize ^{18}[F]-labeled radiopharmaceuticals.[11]

To overcome the drawbacks of the electrophilic monofluoromethylating reagents mentioned above, Prakash and coworkers reported, in 2008, the first shelf-stable yet highly reactive electrophilic monofluoromethylating reagent *S*-monofluoromethyl-*S*-phenyl-2,3,4,5-tetramethylphenylsulfonium tetrafluoroborate **1**,[12] which allowed to efficiently transfer the monofluoromethyl group to various substrates such as amines, phosphines, sulfonic and carboxylic acids and phenols. The reactions of reagent **1** with alcohols or carbon nucleophiles, however, were less successful. Only the trifluoromethyl-substituted alcohols with low basicity and carbon-nucleophiles with three electron-withdrawing groups were able to react with reagent **1** to give the corresponding monofluoromethylated products in high yields. Shortly after, Shibata and coworkers discovered that a new electrophilic monofluoromethylating reagent *N*,*N*-dimethyl-*S*-monofluoromethyl-*S*-phenyl-sulfoximinium hexafluorophosphate **2**[13] was able to react with a variety of 1,3-dicarbonyl compounds, although *O*-selective monofluoromethyl enol ethers were obtained. Reagent **2** was also capable of monofluoromethylation of other oxygen-containing nucleophiles such as sulfonic and carboxylic acids and phenols. However, again, conventional alcohols did not react with reagent **2** under the same reaction conditions. Thus, clearly, new, readily accessible and easy-to-handle electrophilic monofluoromethylating reagents that are effective with a broad substrate scope under mild conditions are highly desirable.[14]

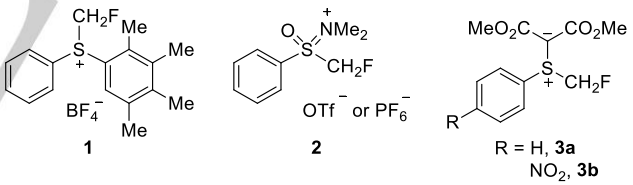

Figure 1. Electrophilic monofluoromethylating reagents.

Recently, we discovered that trifluoromethyl- or difluoromethyl-substituted sulfonium ylides can be served as electrophilic trifluoromethylating or difluoromethylating reagents,[15] we thus questioned ourselves whether monofluoromethyl-substituted sulfonium ylide is an electrophilic monofluoromethylating reagent. We now disclose herein the invention of two monofluoromethyl-substituted sulfonium ylides **3a**-**b**[16] that were able to electrophilically monofluoromethylate a variety of alcohols and malonate derivatives, for the first time. In addition, the superior reactivity of reagent **3a**-**b** was further demonstrated by electrophilic monofluoromethylating a variety of nucleophiles such as sulfonic and carboxylic acids, phenols, amides, and nitrogen of heteroarenes under mild conditions. Mechanistic studies by employing deuterated reagents **3a**-D_2/**3b**-D_2 suggest that these monofluoromethylating reactions proceed via an electrophilic substitution pathway.

S-monofluoromethyl-*S*-phenyl-bis(carbomethoxy) methylide sulfonium ylide **3a** could be easily synthesized as crystalline white solids in 69% yield on a 7.5 g scale by initial treatment of

[a] Y. Liu, Prof. Dr. L. Lu, Prof. Dr. Q. Shen
Key Laboratory of Organofluorine Chemistry
Shanghai Institute of Organic Chemistry
University of Chinese Academy of Sciences
Chinese Academy of Sciences
345 Lingling Road, Shanghai 200032
lulong@sioc.ac.cn; shenql@sioc.ac.cn

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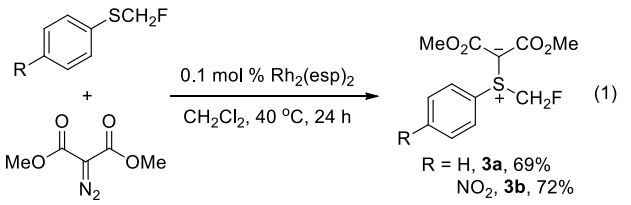
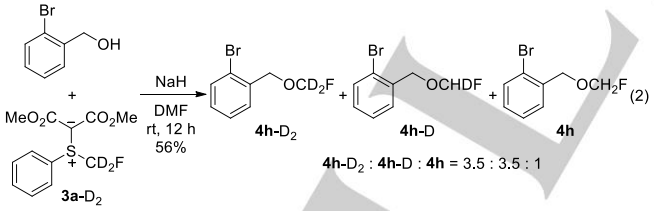
commercially available chloromethyl phenylthioether with CsF in a mixed PEG-200/CH_3CN (1:2) for 2.0 h to generate fluoromethyl phenylthioether,[17] followed by reaction of the crude fluoromethyl phenylthioether with dimethyl diazomalonate in the presence of 0.1 mol% $Rh_2(esp)_2$ (esp = α,α,α',α'-tetramethyl-1,3-benzenedipropionic acid) in dichloromethane after 24 h at 40 °C (Eq.1). Likewise, *S*-monofluoromethyl-*S*-(4-nitrophenyl)-bis(carbomethoxy)methylide sulfonium ylide **3b** was synthesized by reaction of dimethyl diazomalonate with fluoromethyl 4-nitrophenylthioether in 72% yield. Compounds **3a-b** were characterized by ^1H, ^{13}C, and ^{19}F NMR spectroscopies and both structures were unambiguously confirmed by X-ray analysis of their single crystals (See supporting information for details). No detectable decomposition was observed after one-month storage on shelf at ambient temperature.

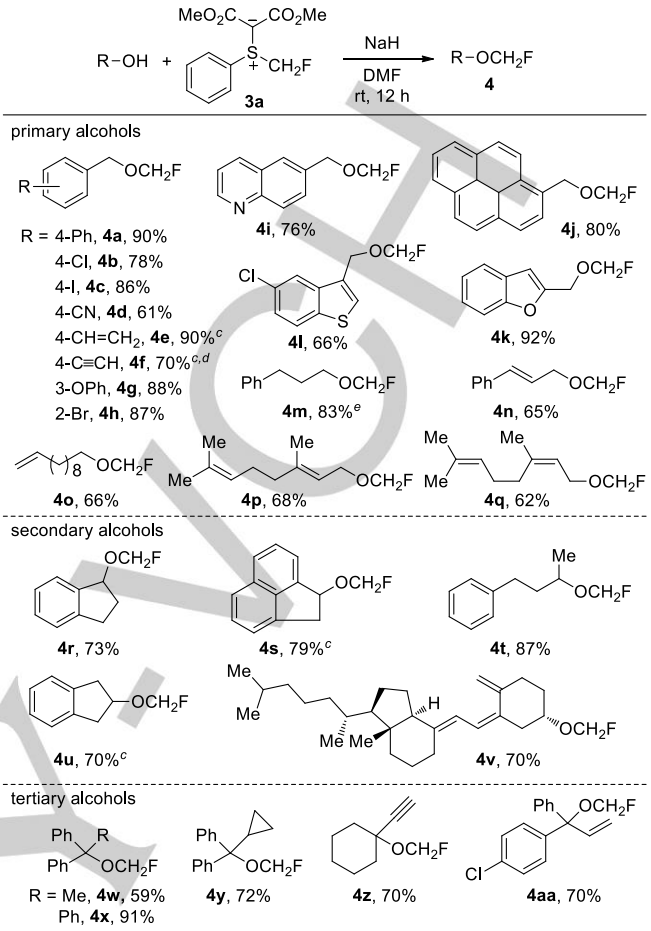


With these new reagents in hand, we first explored the reactivity of reagents **3a-b** with aliphatic alcohols. After extensive survey of the bases and the solvents, it was discovered that reactions of a variety of alcohols with reagent **3a** in DMF occurred to full conversion after 12 h at room temperature when NaH was used as the base, as summarized in Scheme 1. In general, not only primary and secondary alcohols but also tertiary alcohols reacted to give the corresponding monofluoromethyl ethers in good to excellent yields. Allylic or propargyl alcohols reacted smoothly to form the desired products in high yields (Scheme 1, **4n**, **4p**-**q**, **4z**, **4aa**). Likewise, alcohols with heteroaryl moiety such as quinoline, benzothiophene or benzofuran also reacted efficiently to give the corresponding monofluoromethyl ethers in high yields (Scheme, 1, **4i**, **4l**-**k**). Substrates with functional groups such as chloride, bromide, iodide, cyano or cyclopropyl group were compatible with the reaction conditions (Scheme 1, **4b**-**d**, **4h**, **4l**, **4y, 4aa**).



Mechanistically, monofluoromethylation of alcohols with reagent **3a** may process via a monofluorocarbene pathway or a pathway involving direct nucleophilic attack of the alkoxide to the electrophilic monofluoromethyl-substituted sulfonium ylide **3a**. To distinguish these two pathways, we synthesized a deuterated monofluoromethyl-substituted sulfonium ylide **3a**-D_2 and subjected it to the optimized reaction conditions. Experimentally, partial loss of deuterium in the products was observed (Eq. 2). Further studies indicated that the partial loss of deuterium is due to H/D exchange in compound **3a**-D_2 under the basic conditions (see supporting information for details). Thus, these results suggest that the monofluoromethylether formation reaction proceeds highly likely through a nucleophilic substitution pathway instead of a monofluorocarbene pathway.

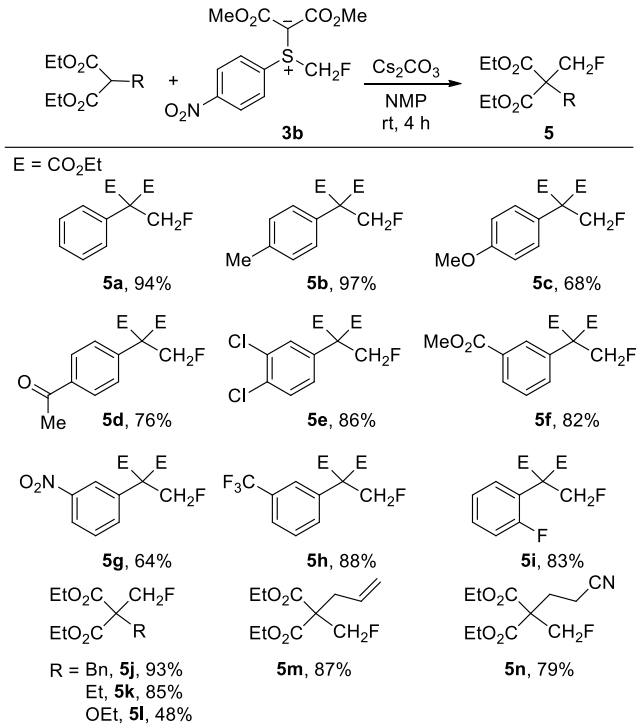
Scheme 1. Scope for the reaction of reagent **3a** with alcohols.[a,b]



[a] Reaction conditions: Alcohol (0.5 mmol), reagent **3a** (0.5 mmol), NaH (60% purity, 2.1 equiv.) in DMF (3.0 mL) at room temperature for 12 h; [b] Isolated yields; [c] 2.0 equiv of reagent was used; [d] 1.2 equiv of ^{t}BuONa was used; [e] 2.0 equiv of alcohol was used.

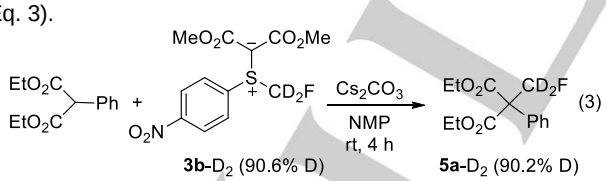
Having established that compound **3a** is a good electrophilic monofluoromethylating reagent, we next tried to address another challenging reaction-monofluoromethylation of carbon nucleophiles. Reaction of diethyl 2-phenylmalonate with reagent **3a** was initially chosen as a model reaction to optimize the reaction conditions. However, the yields for the formation of the desired monofluoromethyl-substituted product were less than 13% as determined by ^{19}F NMR spectroscopy whatever the reaction was conducted in the presence of common inorganic bases such as Li_2CO_3, Na_2CO_3, Cs_2CO_3, K_2CO_3, K_3PO_4, KOH, ^{t}BuOK or organic bases such as DMAP, DBU, DABCO in different solvents such as toluene, CH_2Cl_2, THF, diglyme, DMF, CH_3CN, acetone or NMP. Interestingly, the yield for the formation of the desired diethyl 2-phenyl-2-monofluoromethyl malonate was significantly improved to 87% yield when reagent **3b** was allowed to react with diethyl 2-phenylmalonate in NMP for 4 h at room temperature in the presence of 2.0 equivalents of Cs_2CO_3 (see Supporting Information for details). Under the optimized conditions, we then explored the applicability of these conditions to other malonate derivatives. As summarized in Scheme 2, a variety of malonate derivatives were monofluoromethylated in moderate to high yields. In general, both 2-aryl or 2-alkyl-substituted malonates reacted with reagent **3b** to generate the corresponding monofluoromethylated malonates in high yields. For example, reaction of diethyl 2-(4-

Scheme 2. Scope for the reaction of reagent **3b** with diethyl 2-substituted malonates.[a,b]



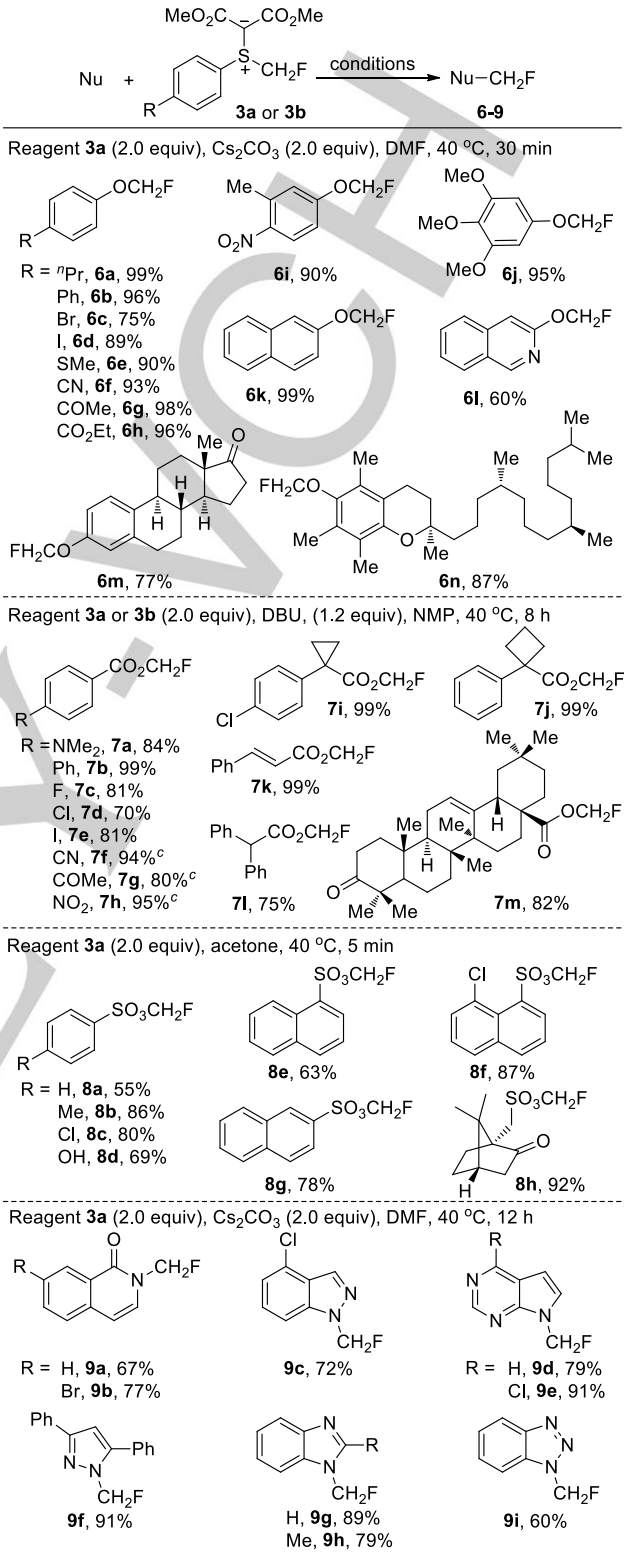
[a] Reaction conditions: diethyl 2-substituted malonate (0.5 mmol), reagent **3b** (0.6 mmol), Cs_2CO_3 (1.0 mmol) in NMP (3.0 mL) at room temperature for 4 h; [b] Isolated yields.

methylphenyl)malonate with reagent **3b** generated compound **5b** in 97% yield, while reaction of diethyl 2-allylmalonate with reagent **3b** occurred to full conversion to give **5m** in 87% yield (Scheme 2, **5b** and **5m**). Electron-donating or electron withdrawing substituted groups on the arene moiety of diethyl 2-arylmalonates have negligible effect on the yields of the reactions (Scheme 2, **5a**-**i**). Due to the mild conditions, common functional groups such as chloro, fluoro, cyano, nitro group or ester, enolizable ketone were compatible. Again, no H/D exchange was observed for the reaction of deuterated reagent **3b**-D_2 with diethyl 2-phenylmalonate under the optimized conditions, which indicates a nucleophilic substitution pathway (Eq. 3).



Encouraged by the excellent electrophilicity of reagents **3a**/**3b**, we next studied their reactions with a variety of oxygen-or nitrogen-nucleophiles. As summarized in Scheme 3, a variety of phenols with electron-donating or withdrawing groups reacted with reagent **3a** in good to excellent yields (Scheme 3, **6a**-**n**). Likewise, both aryl- or alkyl-substituted carboxylic acids also reacted with reagent **3a** to give the corresponding monofluoromethyl carboxylic esters in high yields when the reactions were conducted in NMP in the presence of 1.2 equivalents of DBU (Scheme 3, **7a**-**e**, **7i**-**m**). Benzoic acids with strong electron-withdrawing groups such as cyano, ketone or nitro group reacted with reagent **3a** much slower and in lower yields than other carboxylic acids. In these cases, switching the reagent from **3a** to **3b** greatly improved the conversion and the

Scheme 3. Scope for the reaction of reagent **3a**/**3b** with other nucleophiles.[a,b]



[a] Reaction conditions as indicated in the table; [b] Isolated yields; [c] Reagent **3b** was use and the reaction was conducted at room temperature for 2 h.

yields of the corresponding esters (Scheme 3, **7f**-**h**), which again suggests the higher electrophilicity of reagent **3b** than that of **3a**. Interestingly, reactions of sulfonic acids with reagent **3a** occurred smoothly in full conversion without the need of any additive or base. Mixing the sulfonic acid with reagent **3a** in

acetone at 40 °C for 5 min generated the corresponding monofluoromethyl sulfonate in good yields (Scheme 3, **8a**-**h**). Finally, nitrogen nucleophiles including amides or heteroarenes such as indazole, pyrazole, imidazole, pyrrolo[2,3-d]pyrimidine or benzotrizole can also be efficiently monofluoromethylated in good yields (Scheme 3, **9a**-**i**). Again, no H/D exchange was observed when **3a**-D_2 was used in these reactions, which indicates the nucleophilic substitution pathway (See Supporting Information for details).

In summary, we have successfully developed two electrophilic monofluoromethylating reagents *S*-monofluoromethyl-*S*-phenyl-bis(carbomethoxy) methylide sulfonium ylide **3a** and *S*-monofluoromethyl-*S*-(4-nitrophenyl)-bis(carbomethoxy)methylide sulfonium ylide **3b** that can be easily synthesized from easily available starting materials. Both reagents showed much higher electrophilicity than the reagents previously reported. A variety of nucleophiles such as alcohols and malonate derivatives, sulfonic and carboxylic acids, phenols, amides, and nitrogen of heteroarenes reacted with either reagent **3a** or **3b** to give the corresponding monofluoromethylated products in high yields under mild conditions. Mechanistic studies by employing deuterated reagents **3a**-D_2/**3b**-D_2 suggest that these monofluoromethylating reactions proceed via an electrophilic substitution pathway. Further expand the scope of these reagents are undergoing currently in our laboratory.

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Keywords: fluorine • monofluoromethyl • electrophilic • ylide • sulfonium ylide

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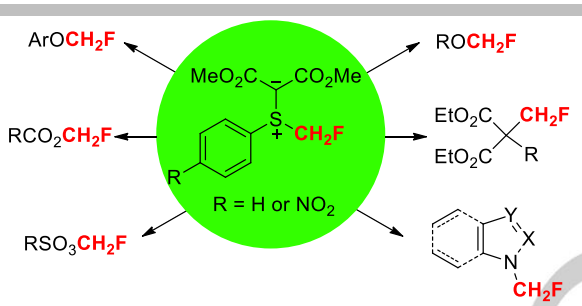
Two sulfonium ylide-based electrophilic monofluoromethyl-ating reagents **3a** and **3b** were invented. These reagents were capable of monofluoromethylating a variety of nucleophiles such as alcohols and malonate derivatives, sulfonic and carboxylic acids, phenols, amides, and nitrogen of heteroarenes under mild conditions.



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