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Reactions of novel mono- and difluoroacetyltrialkylsilanes with sulfur and phosphorus ylides

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Dedicated to Professor Lev M. Yagupolskii on the occasion of his 80th birthday.

Abstract

The reactions of difluoroacetyltrialkylsilanes with methylidene triphenylphosphorane and benzylidene triphenylphosphorane are affected by the nature of the silyl substituents giving either the enol silyl ether or normal Wittig product exclusively, or mixture of both. Reactions with Horner–Emmons type ylide gave only the alkene products. Reactions of mono- and difluoroacetyltrialkylsilanes with dimethylsulfoxonium methylide gave the enol silyl ether products exclusively. Conversion of an enol silyl ether to an epoxide was effected with *m*-CPBA.

Keywords: Fluorinated acetylsilanes; Wittig reaction; Horner–Emmons reaction; Ylides; Dimethylsulfoxonium methylide; Enol silyl ether

1. Introduction

Acylsilanes, known as the synthetic equivalent of aldehydes due to the easy removal of the silyl group by treatment with fluoride ion, have been used as versatile building blocks for the syntheses of complex organic molecules [1,2]. Furthermore, fluorinated acylsilanes are also useful building blocks for the formation of fluorinated enol silyl ethers [3,4]. Preparations of difluoroenol silyl ethers from fluorinated acylsilanes by treatment with organometallic reagents [3,4] or from non-fluorinated acylsilanes by reaction with trifluoromethyltrimethylsilane [5] are reported. However, the preparation of fluorinated acylsilanes [3] or the synthetic utility of these building blocks [6,7] has still not been fully explored.

As part of our research on the development of new fluorinated building blocks, we recently reported the synthesis of novel mono- and difluoroacetyltrialkylsilanes [8]. In this paper, we wish to present the results of our continuing studies on the synthetic utility of mono- and difluoroacetyltrialkylsilanes (**1**, **2a–c**), in particular, by reactions with sulfur and phosphorus ylides Scheme 1.

2. Results and discussion

Reactions of acylsilanes with sulfur and phosphorus ylides are profoundly influenced by silyl group migration forming the silyl enol ethers as one of the products [9–13]. This tendency is even more pronounced in the transformations of fluorinated acylsilanes, giving the enol silyl ethers as the major product [10,11]. However, no information on the influence of the nature of the silyl group on the reaction pathway is available.

Wittig reaction of difluoroacetyltriethylsilane (**1a**) with triphenylphosphonium methylide generated from methyltriphenylphosphonium bromide by deprotonation with *n*-butyllithium (Scheme 2), produced two fluorinated compounds: the expected alkene (**3a**) and the enol silyl ether (**4a**) (Table 1). However, it is clear that the nature of both the reactive ylide and silyl group substituents affect the out come of the reaction.

Brook has reported that reactions of aryl silyl ketones with methylidene triphenylphosphoranes as well as alkyl-substituted methylene-phosphoranes such as ethylidene and propylidene triphenylphosphoranes resulted in the formation of silyl enol ethers, the Brook rearrangement products (Scheme 3, path a), while the reactions of alkyl silyl ketones only gave normal Wittig products (path b) [9].

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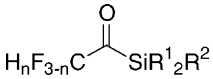
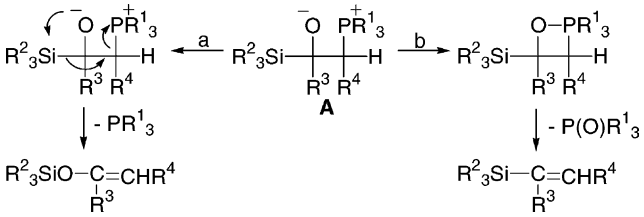


1, n = 1; **2**, n = 2
a: $R^1 = R^2 = Et$;
b: $R^1 = R^2 = i\text{-}Pr$
c: $R^1 = Ph$; $R^2 = t\text{-}Bu$

Scheme 1. Mono- and difluoroacetyltrialkylsilanes.



Scheme 3. Formation of enol silyl ether (a) and normal Wittig product (b).

The promotion of silyl group migration in the reaction of fluoroacetylsilanes is consistent with the premise that substituents (R^3) alpha to the carbonyl of the acylsilane capable of reducing the buildup of charge density resulting on silyl migration and will promote the rearrangement. Xu and coworker reported the formation of only enol silyl ethers from the Wittig reactions of trifluoroacetyltriphenylsilane with various alkylidenetriphenylphosphoranes, reasoning that the electron-withdrawing ability of the CF_3 enhances oxygen attack on silicon over that on phosphorus [11]. Similarly, in our hands, reactions of difluoroacetyl-*tert*-butyldiphenylsilane (**1c**) yielded only the silyl enol ether (**4c**). However, use of a less positive silyl group (as estimated by PM3 calculations) as in compound (**1a**) ($R^2 = Et$) resulted in formation of both the enol silyl ether (**4a**) and the alkene (**3a**). Since alkyl groups increase the electron density at the silicon center, paths a and b become competitive even on such a subtle perturbation. Furthermore, when compound (**1a**) was allowed to react with a resonance-stabilized ylide such as triphenylphosphonium benzylide ($R^4 = Ph$), the only product isolated was the normal Wittig product, the alkene (**3d**). Clearly, the nature of the substituents on the silicon affects the reaction pathway. Ab initio calculations of intermediate A (Scheme 3, $R^1 = H$, $R^2 = H$, $R^3 = CF_2H$, $R^4 = H$, atomic charges calculated using B3LYP and a 6-31G* basis set) showed an increase in the charge difference between Si and P when $R^4 = CO_2Me$, CN or Ph compared to when $R^4 = H$. A similar trend is observed when $R^2 = Et$. This suggests the electrophilicity of phosphorous increases relative to silicon with the result that olefination is increasingly favored over Brooke type rearrangement to form the enol ether. It is certain however that steric encumbrance to the assumption of the necessary synperiplanar conformation by oxygen and phosphorous can also influence the course of the reaction. Previously, no reports have been made on the influence of the silyl substituents on the outcome of ylide additions. We are continuing to explore these phenomena to rationalize the reactivity of mono- and difluoroacetyltrialkylsilanes with Wittig reagents, sulfur ylides and diazo compounds.

Table 1
Wittig reactions of difluoroacetyltrialkylsilanes

Entry	1	R^3	Time (h)	Yield (%)[a]	Yield (%)[a]
1	**a**	H	1	**3a** (21)	**4a** (25)
2	**c**	H	1	**3c** (0)	**4c** (63)
3	**a**	Phenyl	24	**3d** (75)	**4d** (0)

[a] Isolated yield.

Table 2
Horner–Emmons reactions of difluoroacetyltrialkylsilanes

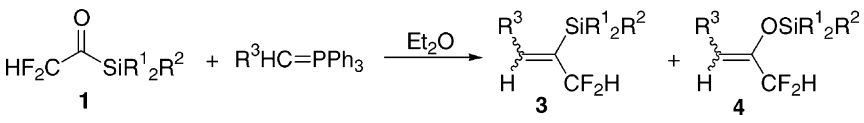
Product	1	Time (h)	Yield (%)[a]
5a	**a**	1	61
5b	**b**	4	52
5c	**c**	1	59

[a] Isolated yield.

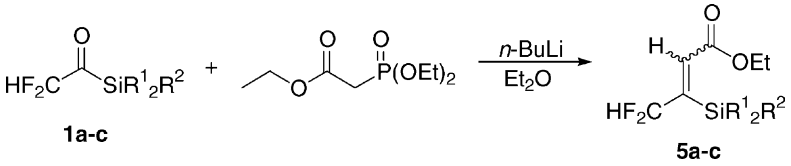
Treatment of difluoroacetyltrialkylsilanes with the Horner–Emmons type reagent [14], triethyl phosphonoacetate, was also studied (Scheme 4) and results are summarized in Table 2.

All three difluoroacetyltrialkylsilanes gave only the normal Horner–Emmons products with no rearrangement products found. Since the carboethoxy group stabilizes the ylide, these results are in agreement with those obtained with triphenylphosphonium benzylide (Table 1, entry 3). The reaction of difluoroacetyltriisopropylsilane (**1b**) required longer reaction times than compounds (**1a** and **1c**) as a consequence of steric obstruction of the carbonyl carbon by the alkyl groups on silicon. Only one vinyl proton resonance and a single fluorine peak was observed at around 6 ppm in the ^1H NMR spectrum and about −110 ppm in the ^{19}F NMR spectrum, respectively, indicating the high stereoselectivity of the reaction although additional experiments will be required to establish the exact stereochemistry of the products.

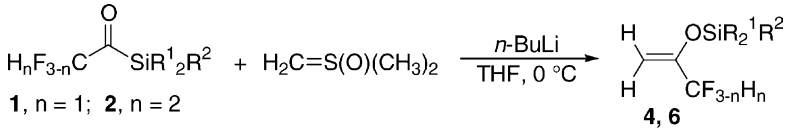
Reactions of mono- and difluoroacetyltrialkylsilanes with sulfur ylides were also attempted (Scheme 5). Reactions of



Scheme 2. Reactions of difluoroacetyltrialkylsilanes with phosphorus ylides.



Scheme 4. Reaction of difluoroacetyltrialkylsilanes with a Horner–Emmons reagent.

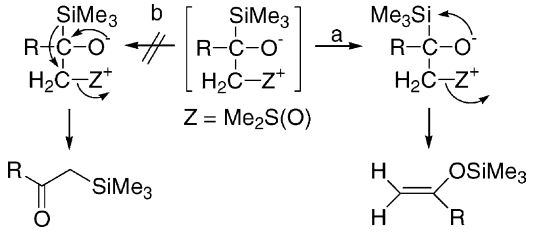


Scheme 5. Reactions of mono- and difluoroacetyltrialkylsilanes with sulfoxonium ylide.

the least bulky derivative, difluoroacetyltriethylsilane (**1a**), with dimethylsulfonium methylides, generated by the deprotonation of trimethylsulfonium iodide with *n*-butyllithium, in various solvents and at different reaction temperatures produced complex mixtures of fluorinated compounds. On the other hand, dimethylsulfoxonium methylide generated from trimethyloxosulfonium iodide and *n*-butyllithium in THF at 0 °C formed a single fluorinated product cleanly. It is well known that dimethylsulfonium methylide is more reactive with carbonyl groups and less stable than dimethylsulfoxonium methylide [15]. The pronounced reactivity of this ylide has been suggested to account for the lack of selectivity in the formation of fluorinated products observed.

Dimethylsulfoxonium methylide reacted with all fluoroacylsilanes examined to give the enol silyl ether products exclusively. Nakajima et al. has reported similar silyl migration results for the reaction of non-fluorinated acylsilanes with sulfur ylides [12]. Those authors obtained both the enol silyl ether products resulting from a cationotropic rearrangement (Scheme 6, path a), and β-ketosilanes (path b) resulting from anionotropic rearrangement. It appears that the electron-withdrawing fluorines at the α-position (R) hinder the second type of migration (path b).

Results of the reactions of various mono- and difluoroacetyltrialkylsilanes with dimethylsulfoxonium methylide are summarized in Table 3. The enol silyl ethers (**4a–c**, **6b**, **6c**) were formed in good yields. The rearrangement was seemingly insensitive to both the steric demand of trialkylsilanes and the number of fluorine substituents on the α-carbon. The low isolated yield of compound (**4a**) can be



Scheme 6. Cationotopic (a) and anionotopic (b) rearrangements.

Table 3
Formation of enol silyl ether with mono- and difluoroacetyltrialkylsilanes and dimethylsulfoxonium methylide

Product	Silyl ketone	Yield (%)[a]
4a	**1a**	31
4b	**1b**	70
4c	**1c**	84
6b	**2b**	69
6c	**2c**	67

[a] Isolated yield.

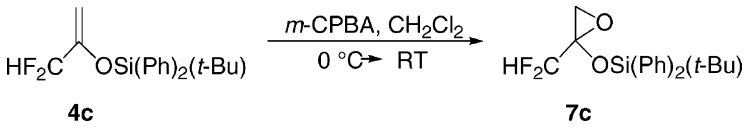
a consequence of the low boiling point of the compound. Unfortunately, the monofluoroacetyltriethylsilane (**2a**) did not undergo reaction in a manner that lead to facile isolation of the product.

In preliminary experiments, reactivity of the product enol silyl ethers was explored by treatment of the enol silyl ether (**4c**) with *m*-CPBA to form the desired epoxide (**7c**) (Scheme 7), a building block with wide potential utility.

Further reactions of the enol silyl ethers are underway. Preliminary results from the Mukaiyama aldol condensation with benzaldehyde show that the difluoro derivatives, especially those with more sterically demanding silyl substituents, (**4b**, **4c**), are remarkably unreactive under common reaction conditions. While the monofluoro compounds (**6b**, **6c**) react readily at room temperature, compound (**4a**) reacted sluggishly under the same conditions. All three derivatives furnished a dehydrated aldol product under the reaction conditions explored to date.

3. Experimental

All reactions were carried out under a dry argon atmosphere with oven-dried glasswares. Reagents were purchased from commercial sources and used without further purification. The mono- and difluoroacetyltrialkylsilanes were prepared following reported procedures [8]. Solvents were purified by standard methods and freshly distilled under argon from either calcium hydride (CH_2Cl_2), or sodium/benzophenone ketyl (THF and Et_2O). ^1H



Scheme 7. Epoxidation of (**4c**).

(300 MHz), ^{13}C (75 MHz), and ^{19}F (282 MHz) NMR spectra were measured on a Varian Gemini-300 NMR spectrometer. ^{1}H and ^{13}C chemical shifts are reported relative to the residual signals of the $CDCl_3$ solvents, taken as δ 7.24 for ^{1}H and 77.00 for ^{13}C. ^{19}F chemical shifts are referenced to $CFCl_3$ as external standard.

3.1. General procedure for the Wittig reaction

To a solution of alkyltriphenylphosphonium bromide (1.5 mmol) in 5 ml of Et_2O at 0 °C was added *n*-butyllithium (1.5 mmol, 0.6 ml of 2.5 M solution in hexanes). The resulting solution was stirred for 30 min and a solution of the difluoroacetyltrialkylsilane (**1**) (1 mmol) mixed with 5 ml of Et_2O was added. The mixture was stirred for a certain time (Table 2), quenched with water and then extracted with Et_2O. The organic layer was dried over anhydrous $MgSO_4$, filtered, and concentrated. Purification by silica gel column chromatography afforded the pure products.

3.1.1. (1-Difluoromethylvinyl)triethylsilane (*3a*)

Yield: 21%, clear oil. ^{1}H NMR: δ 6.09 (m, 1H), 6.05 (t, J_{HF} = 58.1 Hz, 1H), 5.69 (d, J = 1.9 Hz, 1H), 0.93 (t, J = 8.0 Hz, 9H), 0.68 (q, J = 8.0 Hz, 6H). ^{13}C NMR: δ 132.7 (t, $^{2}J_{CF}$ = 14.9 Hz), 119.6 (t, $^{1}J_{CF}$ = 236 Hz), 7.0, 2.9. ^{19}F NMR: δ −107.3 (dd, J = 58.0, 3.1 Hz).

3.1.2. (1-Difluoromethyl-2-phenylvinyl)triethylsilane (*3d*)

Yield: 75%, clear oil. ^{1}H NMR: δ 7.59 (t, J = 3.5 Hz, 1H, one isomer), 7.37–7.17 (m, 5H), 7.11 (s, 1H the other isomer), 6.38 (t, J_{HF} = 57.7 Hz, 1H the other isomer), 6.15 (t, J_{HF} = 58.1 Hz, 1H one isomer), 0.83 (t, J = 8.0 Hz, 9H), 0.51 (q, J = 8.0 Hz, 6H). ^{13}C NMR: δ 148.8 (t, $^{2}J_{CF}$ = 15.5 Hz, one isomer), 147.0 (t, $^{2}J_{CF}$ = 14.9 Hz, the other isomer), 128.4, 128.3, 128.0, 127.9, 120.8 (t, $^{1}J_{CF}$ = 238 Hz one isomer), 116.7 (t, $^{1}J_{CF}$ = 231 Hz the other isomer), 7.2, 3.8. ^{19}F NMR: δ −104.1 (d, J = 57.8 Hz, 2F, the other isomer), −104.3 (dd, J = 58.0, 3.1 Hz, 2F, one isomer).

3.2. General procedure for the preparation of enol silyl ethers (*4*, *6a–c*)

To a solution of trimethylsulfoxonium iodide (1.5 mmol, 0.33 g) in 5 ml of THF at 0 °C was added *n*-butyllithium (1.5 mmol, 0.6 ml of a 2.5 M solution in hexanes). The resulting solution was stirred for 30 min and a solution of (**1**) or (**2**) (1 mmol) mixed with 5 ml of THF was added. After stirring for a further 30 min at 0 °C, the reaction was quenched with water and then extracted with diethyl ether. The organic layer was dried over anhydrous $MgSO_4$, filtered, and concentrated. Purification by silica gel column chromatography afforded the pure products.

3.2.1. (1-Difluoromethylvinyloxy)triethylsilane (*4a*)

Yield: 31%, clear oil. ^{1}H NMR: δ 5.81 (t, J_{HF} = 55.2 Hz, 1H), 4.64 (d, J = 2.2 Hz, 1H), 4.46 (dd, J = 3.8, 1.8 Hz, 1H), 0.98 (t, J = 8.0 Hz, 9H), 0.72 (q, J = 8.0 Hz, 6H). ^{13}C NMR: δ 150.7 (t, $^{2}J_{CF}$ = 22.7 Hz), 111.7 (t, $^{1}J_{CF}$ = 240 Hz), 94.2 (t, $^{3}J_{CF}$ = 5.9 Hz), 6.3, 4.7. ^{19}F NMR: δ −122.1 (d, J = 55.2 Hz).

3.2.2. (1-Difluoromethylvinyloxy)triisopropylsilane (*4b*)

Yield: 70%, clear oil. ^{1}H NMR: δ 5.83 (t, J_{HF} = 55.2 Hz, 1H), 4.63 (d, J = 2.2 Hz, 1H), 4.45 (dd, J = 3.9, 1.8 Hz, 1H), 1.27–1.17 (m, 3H), 1.09 (d, J = 6.7 Hz, 18H). ^{13}C NMR: δ 150.9 (t, $^{2}J_{CF}$ = 22.8 Hz), 111.8 (t, $^{1}J_{CF}$ = 240 Hz), 93.3 (t, $^{3}J_{CF}$ = 5.8 Hz), 17.7, 12.5. ^{19}F NMR: δ −121.9 (d, J = 55.1 Hz).

3.2.3. (1-Difluoromethylvinyloxy)-tert-butyldiphenylsilane (*4c*)

Yield: 84%, white solid, mp 56–58 °C. ^{1}H NMR: δ 7.74–7.29 (m, 10H), 5.91 (t, J_{HF} = 54.9 Hz, 1H), 4.48 (d, J = 2.4 Hz, 1H), 4.04 (dd, J = 3.8, 1.6 Hz, 1H), 1.05 (s, 9H). ^{13}C NMR: δ 149.9 (t, $^{2}J_{CF}$ = 22.5 Hz), 135.4, 131.6, 130.1, 127.8, 111.9 (t, $^{1}J_{CF}$ = 240 Hz), 95.8 (t, $^{3}J_{CF}$ = 5.7 Hz), 26.3, 19.4. ^{19}F NMR: δ −122.1 (d, J = 55.1 Hz).

3.2.4. (1-Fluoromethylvinyloxy)triisopropylsilane (*6b*)

Yield: 69%, clear oil. ^{1}H NMR: δ 4.63 (d, J_{HF} = 48.2 Hz, 2H), 4.38 (bs, 1H), 4.31 (t, J = 1.7 Hz, 1H), 1.27–1.17 (m, 3H), 1.09 (d, J = 6.4 Hz, 18H). ^{13}C NMR: δ 153.9 (d, $^{2}J_{CF}$ = 17.5 Hz), 91.5 (d, $^{3}J_{CF}$ = 6.6 Hz), 82.6 (d, $^{1}J_{CF}$ = 172 Hz), 17.8, 12.6. ^{19}F NMR: δ −218.7 (t, J = 47.1 Hz).

3.2.5. (1-Fluoromethylvinyloxy)-tert-butyldiphenylsilane (*6c*)

Yield: 67%, white low melting solid. ^{1}H NMR: δ 7.84–7.74 (m, 4 H), 7.51–7.39 (m, 10H), 4.77 (d, J_{HF} = 47.3 Hz, 2H), 4.36 (bs, 1H), 4.08 (t, J = 1.4 Hz, 1H), 1.11 (s, 9H). ^{13}C NMR: δ 153.1 (d, $^{2}J_{CF}$ = 17.2 Hz), 135.5, 132.3, 130.0, 127.7, 94.5 (d, $^{3}J_{CF}$ = 6.7 Hz), 82.9 (d, $^{1}J_{CF}$ = 171 Hz), 26.4, 19.3. ^{19}F NMR: δ −218.3 (t, J = 47.4 Hz).

3.3. General procedure for the Horner–Emmons reactions with difluoroacetyltrialkylsilanes

To a solution of triethyl phosphonoacetate (1.5 mmol, 0.34 g) in 5 ml of an Et_2O at 0 °C was added *n*-butyllithium

(1.5 mmol, 0.6 ml of 2.5 M solution in hexanes). After stirring for 1 h, a solution of the difluoroacetyltrialkylsilane (**1**) (1 mmol) mixed with 5 ml of Et_2O was added. The reaction mixture was stirred for a certain time (Table 3) at 0 °C and then quenched with water and extracted with Et_2O. The organic layer was dried over anhydrous $MgSO_4$, filtered, and concentrated. Purification by silica gel column chromatography afforded the pure products.

*3.3.1. 4,4-Difluoro-3-triethylsilanyl-2-butenoic acid ethyl ester (**5a**)*

Yield: 61%, light yellow oil. ^{1}H NMR: δ 7.30 (t, $J_{HF} = 57.7$ Hz, 1H), 6.19 (s, 1H), 4.18 (q, $J = 7.2$ Hz, 2H), 1.28 (t, $J = 7.2$ Hz, 3H), 0.93 (t, $J = 8.0$ Hz, 9H), 0.73 (q, $J = 8.0$ Hz, 6H). ^{13}C NMR: δ 163.9, 152.9 (t, $^2J_{CF} = 32.2$ Hz), 134.3 (t, $^3J_{CF} = 13.1$ Hz), 113.4 (t, $^1J_{CF} = 232$ Hz), 60.9, 14.0, 7.0, 33.1. ^{19}F NMR: δ −110.1 (d, $J = 57.7$ Hz).

*3.3.2. 4,4-Difluoro-3-triisopropylsilanyl-2-butenoic acid ethyl ester (**5b**)*

Yield: 59%, clear oil. ^{1}H NMR: δ 7.30 (t, $J_{HF} = 57.3$ Hz, 1H), 6.26 (s, 1H), 4.19 (q, $J = 7.1$ Hz, 2H), 1.33–1.23 (t, $J = 7.1$ Hz, 3H and m, 3H), 1.07 (d, $J = 7.3$ Hz, 18H). ^{13}C NMR: δ 163.8, 151.6 (t, $^2J_{CF} = 31.9$ Hz), 135.8 (t, $^3J_{CF} = 13.4$ Hz), 113.3 (t, $^1J_{CF} = 234$ Hz), 61.0, 18.5, 14.1, 11.5. ^{19}F NMR: δ −109.0 (d, $J = 57.5$ Hz).

*3.3.3. 4,4-Difluoro-3-(tert-butyldiphenylsilanyl)-2-butenoic ethyl ester (**5c**)*

Yield: 52%, light yellow oil. ^{1}H NMR: δ 7.54–7.16 (m, 11H), 6.33 (s, 1H), 4.18 (q, $J = 7.2$ Hz, 2H), 1.26 (t, $J = 7.2$ Hz, 3H), 1.19 (s, 9H). ^{13}C NMR: δ 164.1, 150.6 (t, $^2J_{CF} = 31.4$ Hz), 139.5 (t, $^3J_{CF} = 12.3$ Hz), 136.2, 133.1, 129.6, 127.8, 113.2 (t, $^1J_{CF} = 235$ Hz), 61.1, 28.8, 18.8, 14.0. ^{19}F NMR: δ −106.7 (d, $J = 56.8$ Hz).

*3.4. (2-Difluoromethyl-oxiranyloxy)-tert-butyldiphenylsilane (**7c**)*

To a solution of *m*-CPBA (77%, 1.5 mmol, 0.34 g) in CH_2Cl_2 (2 ml) at 0 °C was added a solution of (**4c**) (0.5 mmol, 0.17 g) in 5 ml CH_2Cl_2. The mixture was stirred at 0 °C for 1 h, allowed to warm to RT and stirred for an additional 36 h. The reaction was quenched with saturated $NaHCO_3$, then extracted with CH_2Cl_2. The organic layer was dried over $MgSO_4$, filtered and concentrated. Purification by silica gel chromatography afforded the pure product. Yield: 92%, clear oil. ^{1}H NMR: δ 7.75–7.63 (m, 4H), 7.47–7.33 (m, 6H), 5.71 (t, $J_{HF} = 55.3$ Hz, 1H), 2.68 (d, $J = 3.6$ Hz, 1H), 2.66–2.62 (m, 1H), 1.06 (s, 9H). ^{13}C NMR: δ 135.5, 135.4, 132.4, 130.3, 130.2, 127.8, 127.8, 112.2 (t, $^1J_{CF} = 245$ Hz), 78.8 (t, $^2J_{CF} = 30.6$ Hz), 49.7, 26.5, 19.2. ^{19}F NMR: δ −130.2 (dd, $J = 57.2$, 7.8 Hz).

Acknowledgements

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