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Short communication

New approach to synthesize β,β-difluorohomoallylic alcohols

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ABSTRACT

Treatment of ethyl 4,4-difluoro-3-(triethylsilyl)but-3-enoate (**3**) with TBAF in the presence of MS4Å followed by addition of an aromatic aldehyde afforded ethyl (*E*)-4,4-difluoro-5-hydroxy-5-arylpent-2-enoate (**1**) that was a versatile intermediate for constructing a stable isostere of peptide or fluorinated sugar molecules. This strategy offers straightforward approach to synthesize such a versatile intermediate (**1**).

1. Introduction

Methods for *gem*-difluoroalkylation of organic compounds have been currently the focus of much synthetic interest due to potential utility of a difluoromethylene unit as a biological and medicinal material. 3-Bromo-3,3-difluoropropene and bromodifluoroacetic derivatives are markedly accepted as convenient building blocks for constructing difluoromethylene backbone [1]. Qing and co-workers reported syntheses of *gem*-difluorinated sugar nucleosides by way of *gem*-difluoroalkylation with 3-bromo-3,3-difluoropropene [2]. During roughly the same period, Percy and co-workers attained the syntheses of 4-deoxy-4,4-difluorosugars with (3-bromo-3,3-difluoroprop-1-ynyl)benzene as a reagent for *gem*-difluoroalkylation [3]. On the other hand, monofluoroalkene material such as **2** also constitutes an important issue in the field of peptide chemistry because **2** is perceived as a chemically stable isostere of dipeptide. Successful synthesis of **2** was demonstrated by Taguchi [4] and Otaka [5] through same intermediate **1** in the both cases. While there is an obvious fact that **1** is an important platform to a variety of monofluoroalkene, construction of **1** is not simple. Indeed, **1** has been synthesized from ethyl bromodifluoroacetate through many reaction steps in low chemical yield (Scheme 1).

In this view, we expected that the advent of convenient synthetic approach to **1** will offer fruitful advance to the area of sugar and peptide science. Considering these, we have developed a new building block (**3**) for difluoromethylene unit. Several experiments revealed that fluoride anion attacked to triethylsilyl (TES) group to generate the carbanion that could be isomerized to dienolate anion, and then an additional aldehyde could be trapped by the terminal carbanion to give **1**. This strategy would realize the fastest access to **1**. Herein, we wish to report the result on convenient synthesis of **1** from **3** (Scheme 2).

2. Results and discussion

Preparation of **3** was attained through three steps from 2,2,2-trifluoroethanol in moderate yield [6–8]. To explore feasibility of bond dissociation of the Si-Csp^2 bond, the reaction of **3** with benzaldehyde (**4a**) was conducted at 0 °C in a THF solvent in the presence of several classes of fluoride anion (Table 1). The reaction was sluggish in the condition with potassium fluoride and 18-crown-6, to give **1a** in 12% yield (entry 1). The condition with cesium fluoride afforded the product in a similar yield (entry 2). These results suggested that the use of alkali metals as counter cation were not suitable for the reaction. In contrast, a class of tetraalkylammonium fluoride such as tris(dimethylamino)sulfonium difluorotrimethylsilicate (TASF), tetrabutylammonium difluorotriphenylsilicate (TBAT) and tetrabutylammonium fluoride (TBAF) seemed to promote the bond dissociation of the Si-Csp^2 bond. Among them, TBAF in the presence of MS4Å afforded the product in 51% yield (entry 5). In the conditions using TASF, TABT and TBAF, fluoride anion attacked **3** completely so that **3** was not recovered. These results indicated that TBAF was a suitable fluoride source for this reaction.

Subsequently, we addressed optimization of the reaction conditions with a different solvent and temperature. The results are summarized in Table 2. Dimethylformamide worked well in the reaction to give the product in moderate yield (entries 1 and 2, Table 2). Other aprotic amide solvent such as DMA or NMP

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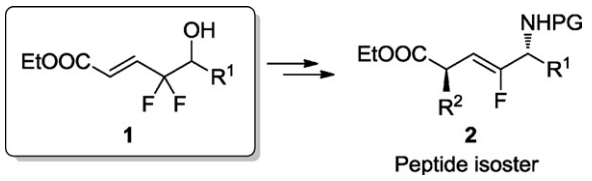
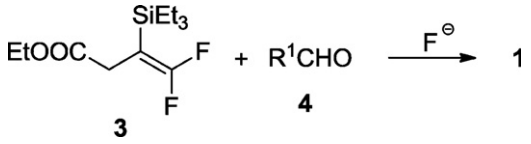
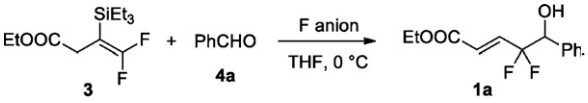


Scheme 1. Structure of key intermediate **1** to synthesize peptide isoster **2**.



Scheme 2. Synthetic strategy to synthesize **1** from **3**.

Table 1
Search for suitable fluoride anion.



Entry[a]	F anion	Time (h)	Yield (%) **1a**[b]
1	KF + 18-croun-6	10	12
2	CsF	10	18
3	TASF	2	42
4	TBAT	2	29
5	TBAF + MS4Å	2	51

[a] Reaction was carried out with **3** (1 mmol), **4a** (2 mmol) and F anion (1.5 mmol).
[b] Isolated yield.

decreased a yield of the product rather than that with DMF. On the other hand, diethyl ether was not effective due to considerable retardation of the reaction. THF was found to be the solvent of choice for the reaction and a 64% yield of **1a** was obtained under the condition at −20 °C (entry 7). Dropping a temperature off to −40 °C, the reaction was not improved. The lower temperature at −78 °C suppressed the reaction completely and **3** was recovered (entry 9).

Subsequently, we assessed the scope and limitation of the reaction with several aromatic and aliphatic aldehydes under the optimized condition (Table 3). For an aromatic aldehyde, *p*-anisaldehyde with electron-donating methoxy group was converted into adduct (**1b**) in 27% yield. On the other hand, aromatic aldehyde with electron-withdrawing trifluoromethyl group at para-position was also resulted in a low yield of **1c** (33%, entry 2). Conjugated cinnamaldehyde was reacted in the same manner to yield **1d** in 32% yield (entry 3). Heteroaromatic aldehyde,

Table 2
Solvent effect of the reaction of 3 with PhCHO in the presence of TBAF and MS4Å.

Entry[a]	Solvent	Temp.	Time (h)	Yield (%) **1a**[b]
1	DMF	0	1.5	57
2	DMF	−20	1.5	54
3	DMA	0	1.5	41
4	NMP	0	1.5	36
5	Et_2O	0	5	32
6	THF	0	2	51
7	THF	−20	2	64
8	THF	−40	2	58
9	THF	−78	12	n.r.

[a] Reaction was carried out with **3** (1 mmol), **4a** (2 mmol) and F anion (1.5 mmol).
[b] Isolated yield.

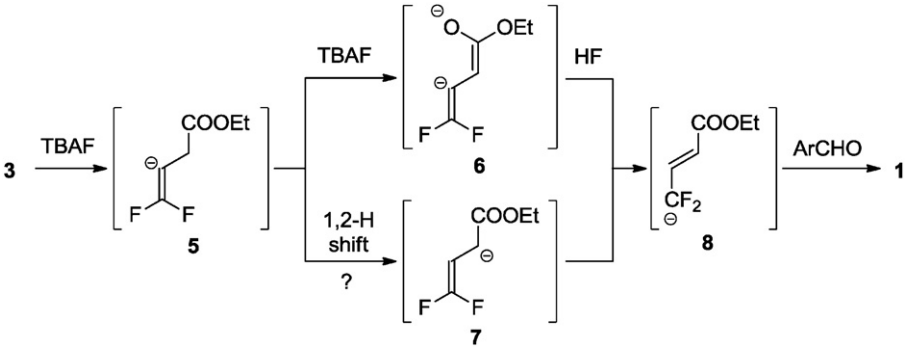
Table 3
Scope and limitations of the reaction with several aromatic and aliphatic aldehydes.

Entry[a]	R-CHO (**4**)		Yield (%) **1**[b]	
1	4-CH_3O–C_6H_4–	**4b**	**1b**	27
2	4-CF_3–C_6H_4–	**4c**	**1c**	33
3	C_6H_5–CH=CH–	**4d**	**1d**	32
4	2-Furyl–	**4e**	**1e**	61
5	2-F-C_6H_4–	**4f**	**1f**	65
6	2-Cl-C_6H_4–	**4g**	**1g**	44
7	*n*-C_7H_{15}–	**4h**	**1h**	n.d.

[a] Reaction was carried out with **3** (1 mmol), **4** (2 mmol), F anion (1.5 mmol) and THF (4 mL) at −20 °C for 2 h.
[b] Isolated yield.

2-furylaldehyde, was suitable for the reaction to afford **1e** in 61% yield. The reaction with 2-fluorobenzaldehyde also gave **1f** in moderate yield. However, 2-chlorobenzaldehyde was converted into **1g** in a lower yield. Aliphatic aldehyde was not a competent substrate in the reaction because basicity of fluoride anion promoted aldol reaction of such an aliphatic aldehyde leading to decomposition of it. The disorderly trend of the reaction was not clear yet.

Plausible reaction mechanism of the reaction is demonstrated in Scheme 3. The reaction would be triggered off by the dissociation of Si-Csp^2 bond with fluoride attack on the silicon atom. The carbanion **5** generated could be guided into divergent ways. One possibility was that another fluoride anion abstracted the α-proton of **5** to generate dianion **6** with slightly stabilized by formation of diene intermediate [9]. Another was that the carbanion of **5** abstracted the α-proton of **5** intermolecularly or intramolecularly, in which **5** had to undergo tentative 1,2-proton shift [10]. Both intermediates **6** and **7** could eventually give the same tautomer **8** that would be stabilized by tautomerization through carbonyl group. Subsequently, **8** could add to aldehyde to afford the product **1**.



Scheme 3. Plausible reaction mechanism of the reaction.

3. Conclusion

In summary, we have synthesized new *gem*-difluorocarbanion source (**3**) from easily available 2,2,2-trifluoroethanol. Treatment of **3** with TBAF in the presence of MS4Å and subsequent addition of aromatic aldehyde afforded the adduct **1**. Structural interest of **1** was exemplified by extensive pioneers work. Our method offers straightforward approach to the significant scaffold of **1**. Although the reaction proceeded in moderate yield, but rapid accessibility to the scaffold of **1** is fascinating. To get the detailed reaction character, mechanistic studies are now under investigation.

4. Experimental

4.1. General

^{1}H NMR and ^{13}C NMR spectra were recorded on JNM-GX400 and JEOL-ECA-600SN spectrometers. ^{19}F NMR spectrum was recorded on Hitachi FT-NMR R-90H spectrometer. Chemical shifts of ^{1}H NMR and ^{13}C NMR are reported in ppm from tetramethylsilane (TMS) as an internal standard. Chemical shifts of ^{19}F NMR are reported in ppm from α,α,α-trifluorotoluene as an internal standard. Mass spectra were obtained on JEOL JMS-700T spectrometer. Melting points were measured on Yanagimoto micro melting point apparatus MP-S3.

4.2. Typical procedure for the synthesis of **1**

Under an argon atmosphere, TBAF (1.5 mL, 1.0 M in THF, 1.5 mmol) was added into a suspension of frame-dried MS4Å (1.0 g) in THF (4.0 mL) and the mixture was stirred at room temperature for 2.0 h. Then the mixture was chilled at −20 °C and solution of **3** (265 mg, 1.0 mmol) and benzaldehyde (204 μL, 2.0 mmol) was added to the mixture over 1.0 h at −20 °C. After stirring the mixture for 1 h at the same temperature, the whole was poured into aqueous 10% HCl, extracted with Et_2O and organic layer was dried with anhydrous $MgSO_4$. After evaporation of the solvent, the residue was purified by column chromatography (SiO_2, EtOAc:hexane) to give **1**.

4.2.1. Ethyl (E)-4,4-difluoro-5-hydroxy-5-phenylpent-2-enoate (**1a**) [11]

A colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.27 (t, *J* = 7.6 Hz, 3H), 2.60 (d, *J* = 4.0 Hz, 1H), 4.21 (q, *J* = 7.6 Hz, 2H), 4.96 (td, *J* = 11.2, 4.0 Hz, 1H), 6.20 (dt, *J* = 15.6, 2.4 Hz, 1H), 6.81 (ddd, *J* = 15.6, 12.8, 11.2 Hz, 1H), 7.32–7.44 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 14.0, 61.1, 75.5 (t, *J* = 28.9 Hz), 119.0 (dd, *J* = 244.2, 243.4 Hz), 126.6 (t, *J* = 8.3 Hz), 127.5, 128.2, 128.8, 135.6 (d, *J* = 3.8 Hz), 136.5 (t, *J* = 25.5 Hz), 165.0; ^{19}F NMR (90 MHz, $CDCl_3$) δ: −44.1 (dt, *J* = 252.0, 10.0 Hz, 1F), −47.0 (dt, *J* = 252.0, 10.0 Hz, 1F); IR (neat) ν_{max} 3450, 3020, 1743 cm^{-1}; MS *m*/*z* = 256 (M^+); HRMS *m*/*z* M^+ calcd. for $C_{13}H_{14}F_2O_3$: 256.091; found: 256.092.

4.2.2. Ethyl (E)-4,4-difluoro-5-hydroxy-5-(4-methoxyphenyl)pent-2-enoate (**1b**)

A colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.29 (t, *J* = 7.2 Hz, 3H), 2.60 (bs, 1H), 3.82 (s, 3H), 4.21 (q, *J* = 7.2 Hz, 2H), 4.90 (t, *J* = 10.0 Hz, 1H), 6.20 (dt, *J* = 16.0, 2.0 Hz, 1H), 6.81 (ddd, *J* = 16.0, 12.8, 11.6 Hz, 1H), 6.89 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 14.1, 55.2, 61.1, 75.3 (t, *J* = 19.6 Hz), 113.8, 119.0 (t, *J* = 214.0 Hz), 126.8 (t, *J* = 8.5 Hz), 127.4 (d, *J* = 3.6 Hz), 128.7, 136.3 (t, *J* = 26.4 Hz), 160.0, 164.8; ^{19}F NMR (90 MHz, $CDCl_3$) δ: −44.3 (dt, *J* = 251.4, 9.6 Hz, 1F), −47.3 (dt, *J* = 251.4, 9.6 Hz, 1F); IR (neat) ν_{max} 3480, 2988, 1716 cm^{-1}; MS *m*/*z* = 286 (M^+); HRMS *m*/*z* M^+ calcd. for $C_{14}H_{16}F_2O_4$: 286.102; found: 286.102.

4.2.3. Ethyl (E)-4,4-difluoro-5-hydroxy-5-(4-(trifluoromethyl)phenyl)pent-2-enoate (**1c**)

A colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.28 (t, *J* = 7.6 Hz, 3H), 3.15 (bs, 1H), 4.21 (q, *J* = 7.6 Hz, 2H), 5.03 (t, *J* = 9.6 Hz, 1H), 6.20 (dt, *J* = 15.6, 2.0 Hz, 1H), 6.80 (ddd, *J* = 15.6, 12.4, 11.2 Hz, 1H), 7.54 (d, *J* = 8.4 Hz, 2H), 7.64 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 14.0, 61.4, 75.0 (t, *J* = 31.9 Hz), 118.8 (t, *J* = 244.9 Hz), 123.9 (q, *J* = 271 Hz), 125.2 (q, *J* = 3.7 Hz), 126.3, 127.2 (t, *J* = 8.1 Hz), 127.9, 131.0 (q, *J* = 32.5 Hz), 135.7 (t, *J* = 25.6 Hz), 164.8; ^{19}F NMR (90 MHz, $CDCl_3$) δ: 0.00 (s, 3F), −43.4 (dt, *J* = 255.1, 10.2 Hz, 1F), −47.3 (dt, *J* = 255.1, 10.2 Hz, 1F); IR (neat) ν_{max} 3472, 2992, 1726 cm^{-1}; MS *m*/*z* = 324 (M^+); HRMS *m*/*z* M^+ calcd. for $C_{14}H_{13}F_5O_3$: 324.078; found: 324.078.

4.2.4. Ethyl (2E,6E)-4,4-difluoro-5-hydroxy-7-phenylhepta-2,6-dienoate (**1d**)

A colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.31 (t, *J* = 7.2 Hz, 3H), 2.34 (bs, 1H), 4.24 (q, *J* = 7.2 Hz, 2H), 4.56 (m, 1H), 6.18 (m, 1H), 6.37 (dt, *J* = 16.0, 2.8 Hz, 1H), 6.80 (m, 1H), 6.92 (ddd, *J* = 16.0, 12.4, 11.6 Hz, 1H), 7.27–7.43 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 14.1, 61.2, 74.3 (t, *J* = 29.3 Hz), 118.9 (t, *J* = 244.5 Hz), 122.2 (t, *J* = 2.1 Hz), 126.7, 127.0 (t, *J* = 8.3 Hz), 128.4, 128.6, 135.2, 135.6, 136.3 (t, *J* = 25.8 Hz), 164.7; ^{19}F NMR (90 MHz, $CDCl_3$) δ: −43.6 (dt, *J* = 250.3, 10.7 Hz, 1F), −49.4 (dt, *J* = 250.3, 10.7 Hz, 1F); IR (neat) ν_{max} 3472, 2988, 1724 cm^{-1}; MS *m*/*z* = 282 (M^+); HRMS *m*/*z* M^+ calcd. for $C_{15}H_{16}F_2O_3$: 282.107; found: 282.107.

4.2.5. Ethyl (E)-4,4-difluoro-5-(furan-2-yl)-5-hydroxypent-2-enoate (**1e**)

A colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.30 (t, *J* = 7.2 Hz, 3H), 2.97 (bs, 1H), 4.23 (q, *J* = 7.2 Hz, 2H), 4.95 (t, *J* = 10.0 Hz, 1H), 6.32 (dt, *J* = 15.2, 2.4 Hz, 1H), 6.43 (m, 2H), 6.90 (dt, *J* = 15.2, 12.4 Hz, 1H), 7.44 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 14.1, 61.3, 70.0 (t, *J* = 31.0 Hz), 109.9, 110.6, 118.1 (t, *J* = 254.2 Hz), 127.0 (t, *J* = 8.3 Hz), 136.2 (t, *J* = 25.6 Hz), 143.2, 148.6, 164.8; ^{19}F NMR (90 MHz, $CDCl_3$) δ: −43.1 (dt, *J* = 252.0, 11.3 Hz, 1F), −46.7 (dt, *J* = 252.0, 11.3 Hz, 1F); IR (neat) ν_{max} 3476, 2988, 1728 cm^{-1}; MS *m*/*z* = 246 (M^+); HRMS *m*/*z* M^+ calcd. for $C_{11}H_{12}F_2O_4$: 246.070; found: 246.070.

4.2.6. Ethyl (E)-4,4-difluoro-5-(2-fluorophenyl)-5-hydroxypent-2-enoate (**1f**)

A colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.29 (t, *J* = 7.2 Hz, 3H), 2.92 (bs, 1H), 4.22 (q, *J* = 7.2 Hz, 2H), 5.32 (td, *J* = 10.0, 4.8 Hz, 1H), 6.23 (dt, *J* = 15.6, 2.4 Hz, 1H), 6.88 (dt, *J* = 15.6, 12.0 Hz, 1H), 7.03–7.55 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 14.1, 61.2, 69.1 (t, *J* = 31.1 Hz), 115.3 (d, *J* = 20.1 Hz), 118.8 (t, *J* = 243.5 Hz), 122.8 (d, *J* = 11.8 Hz), 124.2, 126.9 (t, *J* = 8.2 Hz), 128.9 (d, *J* = 3.3 Hz), 130.6 (d, *J* = 8.9 Hz), 136.2 (t, *J* = 24.8 Hz), 160.2 (d, *J* = 247 Hz), 164.8; ^{19}F NMR (90 MHz, $CDCl_3$) δ: −45.8 (ddt, *J* = 251.5, 9.3, 9.3 Hz, 1F), −48.4 (ddt, *J* = 251.5, 9.3, 9.3 Hz, 1F), −54.5 (m, 1F); IR (neat) ν_{max} 3468, 2992, 1718 cm^{-1}; MS *m*/*z* = 274 (M^+); HRMS *m*/*z* M^+ calcd. for $C_{13}H_{13}F_3O_3$: 274.082; found: 274.082.

4.2.7. Ethyl (E)-5-(2-chlorophenyl)-4,4-difluoro-5-hydroxypent-2-enoate (**1g**)

A colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.30 (t, *J* = 7.2 Hz, 3H), 2.76 (bs, 1H), 4.23 (q, *J* = 7.2 Hz, 2H), 5.54 (t, *J* = 10.0 Hz, 1H), 6.22 (dt, *J* = 15.6, 2.4 Hz, 1H), 6.88 (dt, *J* = 15.6, 11.2 Hz, 1H), 7.27–7.68 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 14.1, 61.2, 71.4 (t, *J* = 29.1 Hz), 119.0 (t, *J* = 244.9 Hz), 126.9 (t, *J* = 8.1 Hz), 127.0, 129.2, 129.5, 130.1, 133.2, 133.6, 136.3 (t, *J* = 25.9 Hz), 164.7; ^{19}F NMR (90 MHz, $CDCl_3$) δ: −45.4 (dt, *J* = 249.3, 10.8 Hz, 1F), −48.1 (dt, *J* = 249.3, 10.8 Hz, 1F); IR (neat) ν_{max} 3496, 2988, 1716 cm^{-1}; MS *m*/*z* = 290 (M^+); HRMS *m*/*z* M^+ calcd. for $C_{13}H_{13}ClF_2O_3$: 290.052; found: 290.059.

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