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Stereoselective synthesis of 4-fluoro-1,3-alkadienylboronates and their application in the stereoselective synthesis of fluoropolyenes

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Abstract (*1E, 3E*)- and (*1E, 3Z*)-4-Fluoro-1,3-alkadienylboronates were stereoselectively prepared by the Heck-reaction of a vinylboronate with (*E*)- or (*Z*)-2-fluoro-1-alkenyliodonium salts, and applied to the Suzuki-Miyaura coupling reaction for the synthesis of fluoropolyenes.

Keywords: Heck-reaction, Fluoroalkadienylboronates, Suzuki-Miyaura coupling, Fluoropolyene, Fluoroalkenyliodonium salts

1. Introduction

Polyene natural products that exhibit significant bioactivities are widely known in nature [1, 2]. The fluorinated analogs of the polyene are of great interest for studying their actions *in vivo* and the modification of their activities [3-8]. Fluorodienylmetals (**1**) are effective as building blocks for the synthesis of polyene analogs with fluorine attached to the double bonds [5, 8]. Although fluorodienylboronates (**1**, M = B in scheme 1) can be used as building blocks for the synthesis of various fluoropolyenes by the Suzuki-Miyaura coupling reaction [9], they have not yet been synthesized.

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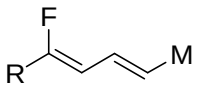
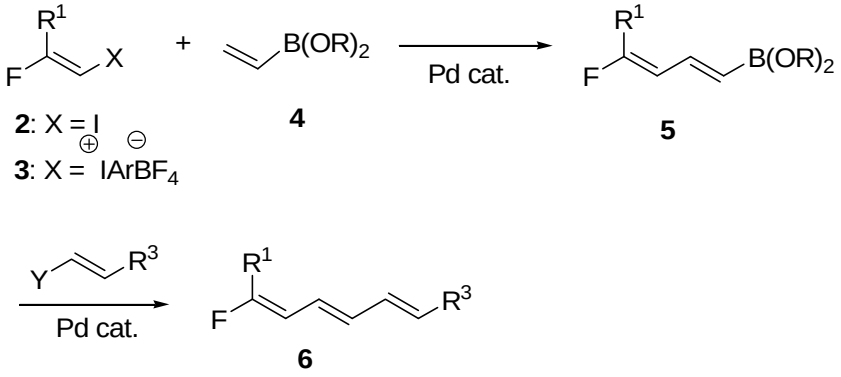


1

M = Sn, Si, B

Scheme 1

Alkadienylboronates are synthesized by the Heck reaction of vinylboronates (**4**) with haloalkenes and used for polyene synthesis [10, 11]. Recently, we succeeded in the stereoselective synthesis of (*E*)- and (*Z*)-2-fluoro-1-iodoalkenes (**2**) and 2-fluoroalkenyliodonium salts (**3**) [12-14]. For the synthesis of fluoroalkadienylboronates (**5**) and their application in the synthesis of fluoropolyenes (**6**), we examined the Heck reaction of vinylboronates (**4**) with 2-fluoro-1-iodoalkenes **2** and 2-fluoroalkenyliodonium salts **3** (Scheme 2).



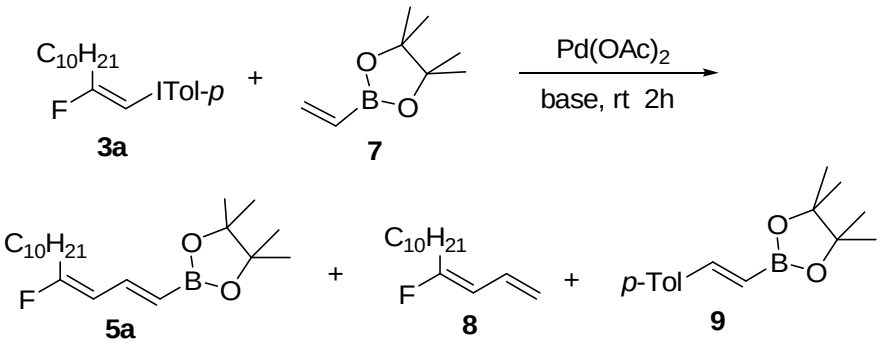
Scheme 2

2. Result and discussion

In the Heck-reaction of the vinylboronates with haloalkenes, the Suzuki-Miyaura coupling reaction competes to give dienes and the yield of the desired dienylboronates decreases [10]. Recently, Whiting et al. successfully depressed the undesired Suzuki-Miyaura coupling using 4,4,6-trimethyl-2-vinyl-1,3,2-dioxaborinane as the vinylboronate and performing the reaction at a high temperature [11]. However, under

the same conditions, the Heck reaction of (*E*)-2-fluoro-1-iodoalkenes (**2**) with vinylboronates such as 4,4,6-trimethyl-2-vinyl-1,3,2-dioxaborinane or 4,4,5,5-tetramethyl-2-vinyl-[1,3,2]-dioxaborolane (**7**) proceeded non-selectively to give a mixture of products, e.g. (*E*)- and (*Z*)-fluorodienylboronates, and the Suzuki-Miyaura coupling product. Alkenyliodonium salts are more reactive than the corresponding iodides in the cross-coupling reaction [15, 16], and therefore, the Heck reaction of (*E*)-2-fluoro-1-dodecenyl iodonium salt (**3a**) with 4,4,5,5-tetramethyl-2-vinyl-[1,3,2]-dioxaborolane (**7**) was examined (Table 1). As expected, the reaction proceeded at room temperature and the desired (1*E*, 3*E*)-4-fluoro-1,3-tetradecadienylboronate (**5a**) was obtained stereoselectively (>95%). Under those conditions, (2-tolylvinyl)boronate (**9**), the Heck reaction product with a tolyl group on **3a**, and 4-fluoro-1,3-tetradecadiene (**8**), the Suzuki-Miyaura coupling product, were also formed as by-products [17]. As reported previously, amine is superior to K_2CO_3 as a base to selectively obtain the Heck product (entries 2 and 4) [18]. When the reaction was carried out using Bu_3N as a base and 2.4 eq of **7** to **3a**, **5a** was obtained in 65% yield (entry 5). Under these conditions, **8** (5% yield) and **9 (**3% yield), which can be separated from **5a** by silica gel column chromatography, were also formed.

Table 1
Synthesis of fluoroalkadienylboronate by Heck reaction using fluoroalkenyliodonium salt[a]



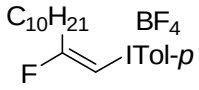
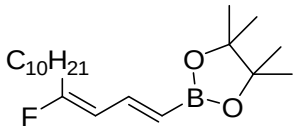
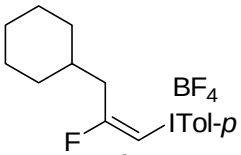
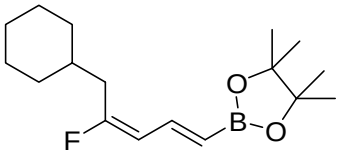
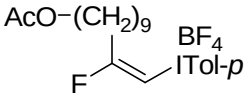
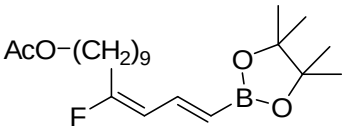
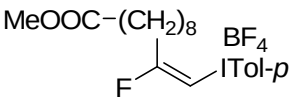
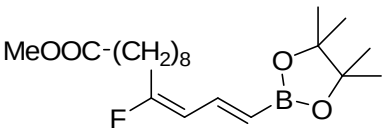
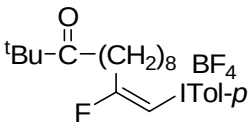
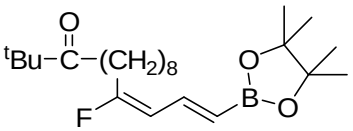
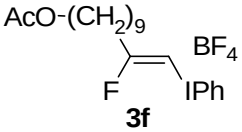
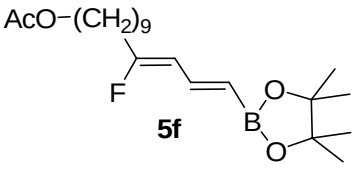
Entry	Solvent	Base	Yield of **5a** (%)[b]	Yield of **8** and **9** (%)[c] **8**	**9**
1	toluene	Bu_3N	40 (97)	2	3
2	DMF	Bu_3N	60 (96)	10	4
3	THF	Bu_3N	60 (97)	10	3
4	DMF	K_2CO_3	19 (96)	6	2
5	DMF	Bu_3N	65 (96)[d]	5	3

[a]If otherwise not mentioned, the reaction was carried out using 1.2 eq of **7**, 1.2 eq of base, and 5 mol% of $Pd(OAc)_2$.
[b] Isolated yield base on 3**a**. In parenthese, stereoselectivity, and a (1*E*, 3*Z*)-isomer was formed as by-product.
[c]Determined by GC.
[d]2.4 eq of **7** was used.

Various 2-fluoroalkenyliodonium salts (**3a-f**) were used in the synthesis of 4-fluoro-1,3-alkadienylboronates **5a-f** (Table 2). When (*E*)-fluoroalkenyliodonium salts (**3a-e**) were used, the corresponding (1*E*, 3*E*)-4-fluoro-1,3-alkadienylboronates (**5a-e**) were obtained in 55-65% yield with good stereoselectivity (94-98%). Functional groups such as ketone and ester can tolerate these reaction conditions, and functionalized fluoroalkadienylboronates (**5c-e**) were obtained (entries 3-5). On the other hand, the reaction with (*Z*)-fluoroalkenyliodonium salt (**3f)** proceeded less selectively, and the desired (1*E*, 3*Z*)-4-fluoro-1,3-alkadienylboronate (**5f**) was obtained

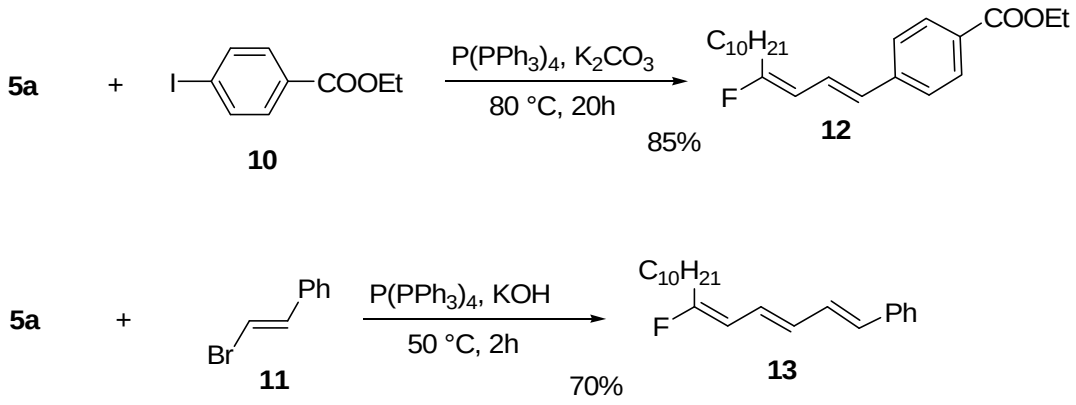
in lower yield (46%) with a significant amount of the Suzuki-Miyaura coupling product (20%) (Entry 6) [17].

Table 2
Synthesis of fluoroalkadienylboronates by Heck reaction using various fluoroalkenyliodonium salt[a]

Entry	Fluoroalkenyliodonium salt **3**	Product **5**	Yield of **5** (%)[b]
1	 **3a**	 **5a**	65 (96)
2	 **3b**	 **5b**	57(95)
3	 **3c**	 **5c**	60 (97)
4	 **3d**	 **5d**	60 (98)
5	 **3e**	 **5e**	55 (95)
6	 **3f**	 **5f**	46 (94)

[a]If otherwise not mentioned, the reaction was carried out using 2.4eq of **7**, 1.2 eq of Bu_3N, and 5 mol% of $Pd(OAc)_2$.
[b]Isolated yield base on **3**. In parenthese, stereoselectivity determined by ^{19}FNMR.

The resulting fluoroalkadienylboronates **5** can be used for fluoropolyene synthesis by the Suzuki-Miyaura coupling reaction. When **5a** was subjected to the coupling reaction with ethyl 4-iodobenzoate (**10**), arylated (*1E, 3E*)-4-fluorotetradecadiene (**12**) was obtained in good yield. Similarly, (*1E, 3E, 5E*)-1-phenyl-6-fluorohexadecatriene (**13**) was obtained by the coupling reaction with β-bromostyrene (**11**) (Scheme 3).



Scheme 3

3. Conclusion

In conclusion, various (*1E, 3E*)- and (*1E, 3Z*)-4-fluoro-1,3-alkadienylboronates were stereoselectively prepared by the Heck-reaction of 4,4,5,5-tetramethyl-2-vinyl-[1,3,2]-dioxaborolane with (*E*)- or (*Z*)-2-fluoro-1-alkenyliodonium salts. The reaction proceeded at room temperature and the pure fluoroalkadienylboronates were isolated by column chromatography. The resulting fluoroalkadienylboronates can be used for the synthesis of fluoropolyenes by the Suzuki-Miyaura coupling reaction with aryl or alkenyl halides.

4. Experimental

4.1. General methods

The IR spectra were recorded using a JASCO FT/IR-410 spectrophotometer. The ^{1}H NMR (400 MHz) spectra, ^{19}F NMR (376 MHz) spectra and ^{13}C NMR (100 MHz) spectra were recorded in $CDCl_3$ on a JEOL JNM-A400II FT NMR spectrometer and the chemical shift, δ, are referred to TMS (^{1}H, ^{13}C) and $CFCl_3$ (^{19}F), respectively. The EI-high-resolution mass spectra were measured on a JEOL JMS-700TZ spectrometer.

(*E*)-2-Fluoroalkenyliodonium salts **3a-e** were prepared from the corresponding alkynes and iodotoluene difluoride according to a literature [13]. (*Z*)-2-Fluoroalkenyliodonium salt **3f** was prepared from the corresponding 1-alkyne in two steps according to the literatures [14]. 4,4,5,5-Tetramethyl-2-vinyl-[1,3,2]-dioxaborolane **7** was purchased from Aldrich or prepared according to the literature [19].

4.2. Synthesis of fluoroalkadienylboronates

*4.2.1. (1E,3E)-2-(4-Fluoro-1,3-tetradecadienyl)-4,4,5,5-tetramethyl-[1,3,2]-dioxaborolane (**5a**)*

A mixture of $Pd(OAc)_2$ (11 mg, 0.05 mmol), Bu_3N (110 mg, 0. 6 mmol), **3a** (238 mg, 0.5 mmol), and **7** (185 mg, 1.2 mmol) in DMF (1.5 ml) was stirred under N_2 atmosphere at room temperature for 2h. Then, the mixture was poured into water and extracted with ether three times. The combined organic layers were dried over $MgSO_4$ and

concentrated under reduced pressure. Purification by column chromatography (silica gel/hexane-ether) gave **5a** (110 mg, 0.33 mmol) in 65% yield. IR (neat) 2927, 1665, 1145 cm^{-1}. ^{1}H NMR δ 0.88 (t, *J* = 6.8 Hz, 3H), 1.27 (s, 12H), 1.27 – 1.31 (m, 14H), 1.52 – 1.54 (m, 2H), 2.42 (dt, *J* = 23.4, 7.3 Hz, 2H), 5.48 (d, *J* = 17.5 Hz, 1H), 5,82 (dd, *J* = 19.8, 11.2 Hz, 1H), 6.97 (dd, *J* = 17.5, 11.3 Hz, 1H). ^{19}F NMR δ - 95.64 (dt, *J* = 19.6, 23.3 Hz, 1F). ^{13}C NMR δ 14.10, 22.67, 24.73 (4C), 26,32, 28.64 (d, *J* = 25.9 Hz), 28.98, 29.29, 29.31, 29.46, 29.55, 31.88, 83.15 (2C), 110.62 (d, *J* = 26.5 Hz), 142.99 (d, *J* = 12.4 Hz), 166.10 (d, *J* = 261.2 Hz). HRMS (EI): calc. for $C_{20}H_{36}O_2FB$: 338.2792, found 338.2794.

*4.2.2. (1E,3E)-2-(5-Cyclohexyl-4-fluoro-1,3-pentadienyl)-4,4,5,5-tetramethyl-[1,3,2]-dioxaborolane (**5b**).* IR (neat) 2926, 1663, 1608, 1331, 1146 cm^{-1}. ^{1}H NMR δ 1.16 – 1.26 (m, 10H), 1.28 (s, 12H), 1.64 – 1.72 (m, 1H), 2.31 (dd, *J* = 24.5, 7.1 Hz, 2H), 5.47 (d, *J* = 17.5 Hz, 1H), 5.87 (dd, *J* = 20.1, 11.2 Hz, 1H), 6.93 (dd, *J* = 17.5, 11.2 Hz, 1H). ^{19}F NMR δ - 93.15 (dt, *J* = 20.2, 24.4 Hz, 1F). ^{13}C NMR δ 24.73 (4C), 26.13 (2C), 26.25, 32.86 (2C), 35.40, 36.33 (d, *J* = 26.0 Hz), 83.13 (2C), 111.73 (d, *J* = 26.6 Hz), 143.00 (d, *J* = 12.1 Hz), 164.99 (d, *J* = 261.2 Hz). HRMS (EI): calc. for $C_{17}H_{28}O_2FB$: 294.2166, found 294.2149.

*4.2.3. (1E,3E)-2-(13-Acetoxy-4-fluoro-1,3-tridecadienyl)-4,4,5,5-tetramethyl-[1,3,2]-dioxaborolane (**5c**).* IR (neat) 2931, 1741, 1664, 1608, 1335, 1240, 1146 cm^{-1}. ^{1}H NMR δ 1.27 (s, 12H), 1.27 – 1.30 (m, 10H), 1.55 – 1.62 (m, 4H), 2.05 (s, 3H), 2.42 (dt, *J* = 23.4, 7.6 Hz, 2H), 4.05 (t, *J* = 6.7 Hz, 2H), 5.48 (d, *J* = 17.5 Hz, 1H), 5.82 (dd, *J* = 19.8, 11.3 Hz, 1H), 6.96 (dd, *J* = 17.6, 11.4 Hz, 1H). ^{19}F NMR δ - 95.70 (dt, *J* =19.5, 23.2 Hz, 1F). ^{13}C NMR δ 20.81, 24.59 (4C), 25.73, 26.14, 28.43. 28.50 (d, *J* = 26.2 Hz), 28.75, 29.03, 29.09, 29.17, 64.45, 83.01 (2C), 110.53 (d, *J* = 26.2 Hz), 142.79 (d, *J* = 12.4 Hz), 165.84 (d, *J* = 261.3 Hz), 171.04. HRMS (EI): calc. for $C_{21}H_{36}O_4FB$:

382.2690, found 382.2681.

*4.2.4. Methyl (10E,12E)-10-fluoro-13-(4,4,5,5-tetramethyl-[1,3,2]-dioxaborolan-2-yl)-10, 12-tridecadienoate (**5d**).* IR (neat) 2932, 1740, 1664, 1608, 1335 cm^{-1}. ^{1}H NMR δ 1.27 (s, 12H), 1.27 – 1.31 (m, 8H), 1.43 – 1.62 (m, 4H), 2.30 (t, *J* = 7.6 Hz, 2H), 2.42 (dt, *J* = 23.4, 7.6 Hz, 2H), 3.67 (s, 3H), 5.48 (d, *J* = 17.6 Hz, 1H), 5.82 (dd, *J* = 19.7, 11.2 Hz, 1H), 6.96 (dd, *J* = 17.6, 11.2 Hz, 1H). ^{19}F NMR δ - 95.71 (dt, *J* =20.2, 23.2 Hz, 1F). ^{13}C NMR δ 24.63 (4C), 24.77, 26.16, 28.54 (d, *J* = 26.5 Hz), 28.75, 28.94 (2C), 29.01, 33.92, 51.28, 83.03 (2C), 110.56 (d, *J* = 26.5 Hz), 142.80 (d, *J* = 12.4 Hz), 165.86 (d, *J* = 261.3 Hz), 174.10. HRMS (EI): calc. for $C_{20}H_{34}O_4FB$: 368.2534, found 368.2526.

4.2.5.

*(12E,14E)-12-Fluoro-2,2-dimethyl-15-(4,4,5,5-tetramethyl-[1,3,2]-dioxaborolan-2-yl)-12, 14-pentadecadien-3-one (**5e**).* IR (neat) 2931, 1705, 1664, 1608, 1334 cm^{-1}. ^{1}H NMR δ 1.13 (s, 9H), 1.27 (s, 12H), 1.27 – 1.30 (m, 8H), 1.54 – 1.55 (m, 4H), 2.37 – 2.48 (m, 4H), 5.48 (d, *J* = 17.5 Hz, 1H), 5.82 (dd, *J* = 19.8, 11.2 Hz, 1H), 6.96 (dd, *J* = 17.6, 11.4 Hz, 1H). ^{19}F NMR δ - 95.67 (dt, *J* =19.9, 23.5 Hz, 1F). ^{13}C NMR δ 23.85, 24.72 (4C), 26.28, 26.37 (3C), 28.66 (d, *J* = 26.5 Hz), 28.91, 29.20, 29.22, 29.31, 36.35, 44.05, 83.13 (2C), 110.62 (d, *J* = 26.6 Hz), 142.93 (d, *J* = 12.4 Hz), 166.02 (d, *J* = 261.5 Hz), 216.08. HRMS (EI) calc. for $C_{23}H_{40}O_3FB$: 394.3054, found 394.3056.

*4.2.6. (1E,3Z)-2-(13-Acetoxy-4-fluoro-1,3-tridecadienyl)-4,4,5,5-tetramethyl-[1,3,2]-dioxaborolane (**5f**).* IR (neat) 2930, 1740, 1360 cm^{-1}. ^{1}H NMR δ 1.24 – 1.32 (m, 10H), 1.27 (s, 12H), 1.51 – 1.63 (m, 4H), 2.05 (s, 3H), 2.21 (dt, *J* = 17.6, 7.4 Hz, 2H), 4.05 (t, *J* = 6.7 Hz, 2H), 5.35 (dd, *J* = 35.0, 10.9 Hz, 1H), 5.42 (d, *J* = 18.1 Hz, 1H), 7.29 (dd, *J* = 17.9, 10.9 Hz, 1H). ^{19}F NMR δ - 98.26 (dt, *J* = 36.4, 17.7 Hz, 1F). ^{13}C

NMR δ 20.98, 24.72 (4C), 25.85, 25.95, 28.55, 28.81, 29.15, 29.29 (2C), 32.21(d, *J* = 25.7 Hz), 64.58 (2C), 83.11, 108.72 (d, *J* = 11.4 Hz), 141.42 (d, *J* = 5.7 Hz), 163.14 (d, *J* = 269.9 Hz), 171.19.

*4.3. Suzuki-Miyaura reaction using **5a***

*4.3.1. Ethyl 4-{(1E,3E)-4-fluoro-1,3-tetradecadienyl}benzoate (**12**)*

A mixture of $Pd(PPh_3)_4$ (28.8 mg, 0.025 mmol), ethyl *p*-iodobenzoate (166 mg, 0.6 mmol), **5a** (169 mg, 0.5 mmol), aq K_2CO_3 (0.6 ml of 2M solution), and EtOH (0.6 ml) in toluene (5 ml) was stirred under N_2 atmosphere at 80 °C for 20h. The mixture was poured into water and extracted with ether three times. Combined organic layers were dried over $MgSO_4$ and concentrated under reduced pressure. Purification by column chromatography (silica gel/hexane-ether) gave **12** (153 mg, 0.43 mmol) in 85% yield. IR (neat) 2926, 2855, 1718, 1665, 1604, 1276 cm^{-1}. ^1H NMR δ 0.87 (t, *J* =7.1 Hz, 3H), 1.26 – 1.43 (m, 14H), 1.40 (t, *J* = 7.1 Hz, 3H), 1.55 – 1.61 (m, 2H), 2.46 (dt, *J* = 23.4, 7.4 Hz, 2H), 4.37 (q, *J* = 7.1 Hz, 2H), 5.93 (dd, *J* = 19.5, 11.3 Hz, 1H), 6.49 (d, *J* = 15.5 Hz, 1H), 6.78 (dd, *J* = 15.1, 11.5 Hz, 1H), 7.41 (d, *J* = 8.4 Hz, 2H), 7.98 (d, *J* = 8.3 Hz, 2H). ^{19}F NMR δ - 96.81 (dt, *J* = 19.9, 23.1 Hz, 1F). ^{13}C NMR δ 14.01, 14.22, 22.59, 26.30, 28.62 (d, *J* = 26.8 Hz), 28.87, 29.24, 29.26, 29.44, 29.52, 31.81, 60.72, 108.56 (d, *J* = 28.4 Hz), 124.53 (d, *J* = 11.2 Hz), 125.66 (2C), 128.79, 129.58(d, *J* = 10.1 Hz), 129.82 (2C), 141.72, 164.80 (d, *J* = 259.4 Hz), 166.20. HRMS(EI) calc. for $C_{23}H_{33}FO_2$: 360.2464, found: 360.2470.

*4.3.2. (1E, 3E, 5E)-6-Fluoro-1-phenyl-1,3,5-hexadecatriene (**13**)*

A mixture of $Pd(PPh_3)_4$ (28.8 mg, 0.025 mmol), (*E*)-β-bromostyrene (165 mg, 0.9

mmol), **5a** (169 mg, 0.5 mmol), aq KOH (0.6 ml of 2M solution), and EtOH (0.6 ml) in toluene (5 ml) was stirred under N_2 atmosphere at 50 °C for 2h. The mixture was poured into water and extracted with ether three times. Combined organic layers were dried over $MgSO_4$ and concentrated under reduced pressure. Purification by column chromatography (silica gel/hexane-ether) gave **13** (110 mg, 0.35 mmol) in 70% yield. IR (neat) 2926, 2854, 1663, 1604, 984 cm^{-1}. ^1H NMR δ 0.87 (t, *J* = 6.5 Hz, 3H), 1.26 (brs, 14H), 1.31 – 1.58 (m, 2H), 2.39 (dt, *J* = 23.4, 7.3 Hz, 2H), 5.84 (dd, *J* = 19.6, 10.1 Hz, 1H), 6.22 – 6.35 (m, 2H), 6.51 (d, *J* = 15.6 Hz, 1H), 6.83 (dd, *J* = 15.4, 9.5 Hz, 1H), 7.21 – 7.40 (m, 5H). ^{19}F NMR δ - 98.91 (dt, *J* = 19.9, 23.1 Hz, 1F). ^{13}C NMR δ 14.11, 22.68, 26.41, 28.65 (d, *J* = 27.1 Hz), 28.98, 29.32, 29.33, 29.51, 29.58, 31.89, 108.69 (d, *J* = 28.2 Hz), 126.21 (2C), 126.35 (d, *J* = 10.8 Hz), 127.34, 128.60 (2C), 129.06, 131.44, 131.50 (d, *J* = 7.2 Hz), 137.39, 163.81 (d, *J* = 258.0 Hz). HRMS (EI): calc. for $C_{22}H_{31}F$: 314.2410, found: 314.2415.

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