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Stille cross-coupling reaction of polyfluorovinylstannanes Stereospecific synthesis of polyfluoro-alkenes and -α,β-unsaturated ketones

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Abstract

The palladium/copper (I) iodide cocatalyzed coupling reaction of (*Z*)-α-fluoro-β-trifluoromethylvinylstannanes **1** with aryl iodides **2** and acid chloride **4** has been explored affording substituted (*E*)-α-fluoro-β-trifluoromethylalkenes **3** and (*E*)-α-fluoro-β-trifluoromethyl-α,β-unsaturated ketones **5**, respectively. The effect of cocatalyst, solvent as well as reaction temperature has been investigated in more detail. The geometric isomers were easily ascertained on the basis of their coupling constant ($^4J_{FF}$) across the double bond and the retention of the configuration of the products for this conversion was found.

Keywords: Polyfluorovinylstannane; Polyfluoroalkene; Polyfluoro-α,β-unsaturated ketone; Stille cross-coupling reaction; Stereospecific synthesis

1. Introduction

Much attention has been paid to the introduction of a fluorine atom or trifluoromethyl group into organic compounds with biological properties during the last several decades, since the resulting compounds often leads to pronounced activity enhancement, and organofluorine compounds are increasingly being applied in pharmaceuticals, agrochemicals and other field [1–9], as exemplified in 2′, 3′-dideoxy-3-(trifluoromethyl)-pentafuranosyl nucleosides which were used as antitumor and antiviral agents [10]. The synthetic utility of fluorine-containing organometallic reagents has been reviewed [11–13]. Among them, the synthetic application of fluorine-containing organostannanes in organic synthesis is still limited due to the limited methodology for their preparation [14]. The Stille cross-coupling reaction, which is a widely employed method for the carbon–carbon bond formation [15–18], has recently become a popular synthetic tool in organic synthesis, particularly for the synthesis of naturally occurring compounds [19–27]. Recently 1-fluorovinylstannanes [28], 1,2-difluorovinylstannanes [14,29], and trifluoromethylvinylstannanes [30] as coupling partners in Stille cross-coupling reaction have been reported. However, the use of polyfluorovinylstannanes as coupling partner in Stille cross-coupling reaction has not been reported previously.

2. Results and discussion

In our previous paper [31], a stereoselective synthesis of (*Z*)-α-fluoro-β-trifluoromethylvinylstannanes has been reported. Herein, we report its application in the Stille cross-coupling reaction of (*Z*)-α-fluoro-β-trifluoromethylvinylstannanes with organic electrophiles giving substituted polyfluoroalkenes and polyfluoro-α,β-unsaturated ketones stereospecifically.

The palladium/copper (I) iodide cocatalyzed coupling reaction of (*Z*)-α-fluoro-β-trifluoromethylvinylstannanes **1** with aryl iodides **2** afforded substituted (*E*)-α-fluoro-β-trifluoromethylethenes **3** stereospecifically. The reaction is shown in Scheme 1.

The results are summarized in Table 1.

In our initial studies, the effect of cocatalyst and solvent as well as reaction temperature has been investigated in detail with (*Z*)-3,3,3-trifluoro-2-(4-chlorophenyl)-1-fluoro-tributylstannylprop-1-ene (**1a**) and iodobenzene as reactants. The results are summarized in Table 2.

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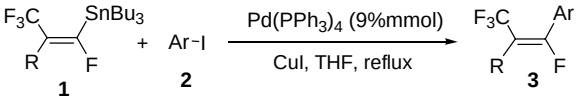
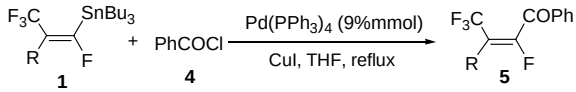


Scheme 1.



Scheme 2.

The reaction proceeds best in THF and with $Pd(PPh_3)_4$ (9 mol%) as catalyst, CuI as cocatalyst at refluxing temperature for 24 h. Blank experiments demonstrated that no reaction occurred in the absence of $Pd(PPh_3)_4$ (entries 3 and 4), while in the absence of CuI, the reaction was sluggish and only 28% yield of **3a** was obtained in THF as solvent after stirring for 24 h at 20 °C (entry 2). The lower reactivity of fluorinated vinylstannanes [28–30] as compared with the corresponding nonfluorinated vinylstannanes is obviously due to its strong electron-withdrawing effect of fluorine. Thus, the addition of CuI as cocatalyst [32] is necessary.

On the basis of coupling constant, across the double bond reported in the literature [33], if the trifluoromethyl group was *trans* with respect to the F group, the $^4J_{FFtrans}$ ranged from 7 to 13 Hz, while for those *cis* with respect to the F group, the $^4J_{FFcis}$ ranged from 21 to 31 Hz. In our cases, $^4J_{FF}$ is ranged from 10.7 to 11.3 Hz; hence the configuration of the products **3** is ascertained as *E*-isomer. The Stille cross coupling reaction of fluorinated vinylstannanes has been reported with retention of configuration [28–30], in our studies, the retentation of configuration was observed similarly on the basis of their $^4J_{FF}$ coupling constant. It can be seen from the Table 1 that the yield of the reaction of aryl iodide with an electron-donating group was lower (compound **3b**) being similarly as compared with the result reported in the literature [28]. However, there is no apparent effect on the yields of products when an electron-donating group is located in the R group of vinylstannanes **1** (compounds **3d** and **3e**).

Table 1
Preparation of substituted (*E*)-α-fluoro-β-trifluoromethylethenes (**3**)

Compound	R	Ar	Yield (%)[a]	*E*:*Z*[b]
3a	4-$CH_3OC_6H_4$	C_6H_5	90	100:0
3b	4-ClC_6H_4	4-$CH_3OC_6H_4$	50	100:0
3c	4-$CH_3OC_6H_4$	4-$CF_3C_6H_4$	81	100:0
3d	4-ClC_6H_4	4-$CH_3C_6H_4$	98	100:0
3e	2-thienyl	2-naphthyl	79	100:0
3f	2-thienyl	C_6H_5	80	100:0
3g	4-ClC_6H_4	4-BrC_6H_4	81	100:0

[a] Isolated yields.
[b] The ratios of *E*- to *Z*-isomers were estimated on the basis of NMR data. No *Z*-isomer was isolated or detectable.

Table 2
The effect of cocatalyst, temperature, and solvent on the yield of **3a**

Entry	Condition	Yield[a]
1	$Pd(PPh_3)_4$ (9 mol%)/DMF/20 °C/24 h	Trace
2	$Pd(PPh_3)_4$ (9 mol%)/THF/20 °C/24 h	28
3	CuI/DMF/20 °C/24 h	–
4	CuI/THF/20 °C/24 h	–
5	$Pd(PPh_3)_4$ (9 mol%)/CuI/DMF/20 °C/24 h	37
6	$Pd(PPh_3)_4$ (9 mol%)/CuI/DMF/80 °C/24 h	49
7	$Pd(PPh_3)_4$ (9 mol%)/CuI/THF/20 °C/24 h	61
8	$Pd(PPh_3)_4$ (9 mol%)/CuI/THF/reflux/24 h	90

[a] Isolated yields.

Table 3
Preparation of (*E*)-α-fluoro-β-trifluoromethyl-α,β-unsaturated ketones (**5**)

Compound	R	Yield (%)[a]	*E*:*Z*[b]
5a	4-$CH_3OC_6H_4$	83	100:0
5b	4-ClC_6H_4	87	100:0
5c	2-thienyl	68	100:0

[a] Isolated yields.
[b] The ratios of *E*- to *Z*-isomers were estimated on the basis of NMR data. No *Z*-isomer was isolated or detectable.

The palladium/copper (I) iodide cocatalyzed coupling reaction of (*Z*)-α-fluoro-β-trifluoromethylvinylstannanes **1** with acid chloride **4** proceeded smoothly under the same conditions giving substituted (*E*)-α-fluoro-β-trifluoromethyl-α,β-unsaturated ketones **5** stereospecifically. The reaction is shown in Scheme 2.

The results are summarized in Table 3. Similarly, the geometric isomers were easily ascertained on the basis of their coupling constant ($^4J_{FF}$) across the double bond and the retention of the configuration of the product for this conversion was also found.

In summary, the palladium/copper (I) iodide cocatalyzed coupling reaction of (*Z*)-α-fluoro-β-trifluoromethylvinylstannanes **1** with aryl iodides **2** and acid chloride **4** has been explored affording substituted (*E*)-α-fluoro-β-trifluoromethylalkenes **3** and (*E*)-α-fluoro-β-trifluoromethyl-α,β-unsaturated ketones **5**, respectively. The effect of cocatalyst, solvent as well as reaction temperature has been investigated in more detail. This general and stereospecific methodology provides a convenient route to the title compounds, which would potentially be employed as useful intermediates in the synthesis of fluorine-containing biologically active compounds.

3. Experimental

All boiling points are uncorrected. The IR spectra of liquid products were obtained as films on a Digilab FTS-20E spectrometer. ^1H NMR spectra were recorded on a Bruker AM-300 (300 MHz) spectrometer (δ values in ppm from tetramethylsilane, in $CDCl_3$, *J*-values are given in Hz). The published ^{19}F NMR spectra were taken on a Varian

EM-360 (60 MHz) spectrometer and re-calculated using the standard chemical shift of reference δ(F) (CF_3CO_2H) −76.5 ppm with respect to $\delta(CFCl_3)$ 0.00 ppm. Mass spectra were measured on a Finnigan GC-MS-4021 mass spectrometer.

(*Z*)-α-Fluoro-β-trifluoromethylvinylstannanes (**1**) were prepared according to the known procedure [31].

3.1. General procedure for the preparation of substituted (E)-α-fluoro-β-trifluoromethylethenes

A mixture of (*Z*)-α-fluoro-β-trifluoromethylvinylstannane (**1**) (0.21 mmol), ArI (0.21 mmol), tetrakis(triphenylphosphine)palladium(0) (19 mg, 0.018 mmol), and CuI (21 mg, 0.11 mmol) in THF (5 ml) was heated at refluxing temperature for 24 h. TLC showed that the starting material was disappeared. The reaction mixture was poured into diethyl ether (40 ml), washed with water (3 × 10 ml) and dried over $MgSO_4$. Evaporation of the solvent gave a residue which was purified by column chromatography eluting with petroleum ether (60–90 °C) to give the product **3**.

*3.1.1. (E)-3,3,3-Trifluoro-2-(4-methoxyphenyl)-1-phenyl-1-fluoro-prop-1-ene (**E-3a**)*

Yield: 90%; mp: 84–85 °C; IR (KBr) (cm^{-1}): 1690, 1510, 1330, 1290, 1250, 1180, 1170, 1110, 960. ^{1}H NMR ($CDCl_3$/TMS): δ 7.60–7.20 (m, 7H), 7.00–6.92 (m, 2H), 3.83 (s, 3H). ^{19}F NMR ($CDCl_3$/TFA): δ −55.7 (d, J = 11.3 Hz, 3F), −76.9 (q, J = 11.3 Hz, 1F). MS: *m/z* (rel. int.): 296 (M^+, 100), 281(4), 265(5), 245(6), 233(14), 226(21), 212(16). Anal. calc. for $C_{16}H_{12}F_4O_2$ (296.26): C, 64.87; H, 4.08. Found: C, 64.70; H, 4.22.

*3.1.2. (E)-3,3,3-Trifluoro-2-(4-chlorophenyl)-1-(4-methoxyphenyl)-1-fluoro-prop-1-ene (**E-3b**)*

Yield: 50%, oil; IR (film) (cm^{-1}): 1660, 1610, 1510, 1340, 1300, 1260, 1180, 1110, 1090, 1070, 960. ^{1}H NMR ($CDCl_3$/TMS): δ 7.53 (d, J = 8.5 Hz, 2H), 7.44–7.34 (m, 4H), 6.97 (d, J = 8.3 Hz, 2H), 3.84 (s, 3H). ^{19}F NMR ($CDCl_3$/TFA): δ −54.5 (d, J = 11.0 Hz, 3F), −74.5 (q, J = 11.0 Hz, 1F). MS: *m/z* (rel. int.): 332 (M^+ + 2, 39), 330 (M^+, 100), 311(4), 295(7), 275(3), 260(6), 226(32). Anal. calc. for $C_{16}H_{11}ClF_4O$ (330.70): C, 58.11; H, 3.35. Found: C, 58.21; H, 3.57.

*3.1.3. (E)-3,3,3-Trifluoro-2-(4-methoxyphenyl)-1-(4-trifluoromethylphenyl)-1-fluoro-prop-1-ene (**E-3c**)*

Yield: 81%, oil; IR (film) (cm^{-1}): 1690, 1610, 1520, 1320, 1290, 1250, 1180, 1130, 960. ^{1}H NMR ($CDCl_3$/TMS): δ 7.81–7.78 (m, 1H), 7.68–7.57 (m, 3H), 7.41–7.32 (m, 2H) 7.01–6.97 (m, 2H), 3.86 (s, 3H). ^{19}F NMR ($CDCl_3$/TFA): δ −57.5 (d, J = 10.7 Hz, 3F), −59.5 (s, 3F), −72.7 (q, J = 10.7 Hz, 1F). MS: *m/z* (rel. int): 364 (M^+, 100), 345(3), 314(1), 295(10), 275(7), 252(7), 226(20). Anal. calc. for $C_{17}H_{11}F_7O$ (364.26): C, 56.05; H, 3.04. Found: C, 56.04; H, 3.21.

*3.1.4. (E)-3,3,3-Trifluoro-2-(4-chlorophenyl)-1-(4-methylphenyl)-1-fluoro-prop-1-ene (**E-3d**)*

Yield: 98%, mp: 41–43 °C; IR (KBr) (cm^{-1}): 1670, 1490, 1330, 1220, 1180, 1120, 1090, 1070, 960. ^{1}H NMR ($CDCl_3$/TMS): δ 7.58–7.24 (m, 8H), 2.42 (s, 3H). ^{19}F NMR ($CDCl_3$/TFA): δ −55.0 (d, J = 11.0 Hz, 3F), −76.0 (q, J = 11.0 Hz, 1F). MS: *m/z* (rel. int.): 316 (M^+ + 2, 37), 314 (M^+, 100), 300(32), 279(30), 259(22), 245(21), 230(20), 210(59). Anal. calc. for $C_{16}H_{11}ClF_4$ (314.70): C, 61.06; H, 3.52. Found: C, 60.80; H, 3.53.

*3.1.5. (E)-3,3,3-Trifluoro-2-(2-thienyl)-1-naphthenyl-1-fluoro-prop-1-ene (**E-3e**)*

Yield: 79%, oil; IR (film) (cm^{-1}): 1670, 1330, 1180, 1120, 1000. ^{1}H NMR ($CDCl_3$/TMS): δ 8.01–7.88 (m, 3H), 7.65–7.49 (m, 5H), 7.42–7.40 (m, 1H), 7.18–7.10 (m, 1H). ^{19}F NMR ($CDCl_3$/TFA): δ −55.0 (d, J = 11.0 Hz, 3F), −66.1 (q, J = 11.0 Hz, 1F). MS: *m/z* (re. int.): 324 (M^+ + 2, 3), 322 (M^+, 100), 303(7), 289(14), 269(7), 253(38), 220(37). Anal. calc. for $C_{17}H_{10}F_4S$ (322.32): C, 63.35; H, 3.13. Found: C, 63.55; H, 3.26.

*3.1.6. (E)-3,3,3-Trifluoro-2-(2-thienyl)-1-phenyl-1-fluoro-prop-1-ene (**E-3f**)*

Yield: 80%, oil; IR (film) (cm^{-1}): 1650, 1320, 1170, 1130, 700. ^{1}H NMR ($CDCl_3$/TMS): δ 7.62–7.42 (m, 6H), 7.33–7.28 (m, 1H), 7.15–7.10 (m, 1H). ^{19}F NMR ($CDCl_3$/TFA): δ −54.5 (d, J = 11.0 Hz, 3F), −69.3 (q, J = 11.0 Hz, 1F). MS: *m/z* (rel. int.): 274 (M^+ + 2, 6), 272 (M^+, 100), 253(5), 251(18), 239(18), 227(16), 202(56). HRMS: *m/z* calc. for $C_{13}H_8F_4S$ (272. 26): 272.0283. Found 272.0296.

*3.1.7. (E)-3,3,3-Trifluoro-2-(4-chlorophenyl)-1-(4-bromophenyl)-1-fluoro-prop-1-ene (**E-3g**)*

Yield: 81%, mp: 49–51 °C; IR (film) (cm^{-1}): 1670, 1490, 1330, 1220, 1180, 1130, 960. ^{1}H NMR ($CDCl_3$/TMS): δ 7.64–7.58 (m, 2H), 7.47–7.39 (m, 4H), 7.38–7.30 (m, 2H). ^{19}F NMR ($CDCl_3$/TFA): δ −54.7 (d, J = 11.0 Hz, 3F), −71.6 (q, J = 11.0 Hz, 1F). MS: *m/z* (rel. int.): 382 (M^+ + 2, 23), 380 (M^+, 94), 378(72), 300(15), 279(17), 264(100), 230(53). HRMS: *m/z* calc. for $C_{15}H_8BrClF_4$ (379.57): 377.9434. Found 377.9404.

3.2. General procedure for the preparation of (E)-α-fluoro-β-trifluoromethyl-α,β-unsaturated ketones

The procedure is same with aforementioned method.

*3.2.1. (E)-3-Fluoro-3-benzoyl-2-(4-methoxyphenyl)-1,1,1-trifluoroprop-1-ene (**E-5a**)*

Yield: 83%, oil; IR (filn) (cm^{-1}): 1690, 1610, 1510, 1330, 1260, 1170, 1130, 990. ^{1}H NMR ($CDCl_3$/TMS): δ 8.03–7.95 (m, 2H), 7.21–7.38 (m, 5H), 7.08–6.92 (m, 2H), 3.85 (s, 3H). ^{19}F NMR ($CDCl_3$/TFA): δ −58.5 (d, J = 8.4 Hz, 3F), −99.7 (q, J = 8.4 Hz, 1F). MS: *m/z* (rel. int.): 324 (M^+, 22), 309(5), 305(2), 293(51), 255(9), 105(100). Anal. calc. for

$C_{17}H_{12}F_4O_2$ (324.27): C, 62.97; H, 3.73. Found: C, 62.90; H, 3.70.

3.2.2. (E)-3-Fluoro-3-benzoyl-2-(4-chlorophenyl)-1,1,1-trifluoroprop-1-ene (**E-5b**)

Yield: 87%, oil; IR (film) (cm^{-1}): 1690, 1600, 1490, 1330, 1280, 1140, 990. ^1H NMR ($CDCl_3$/TMS): δ 8.00–7.96 (m, 2H), 7.74–7.41 (m, 7H). ^{19}F NMR ($CDCl_3$/TFA): δ −57.1 (d, $J = 9.0$ Hz, 3F), −96.5 (q, $J = 9.0$ Hz, 1F). MS: *m/z* (rel. int): 330 ($M^+ + 2$, 8), 328 (M^+, 22), 309(3), 293(100), 277(2). Anal. calc. for $C_{16}H_9ClF_4O$ (328.69): C, 58.47; H, 2.76. Found: C, 58.70; H, 2.82.

3.2.3. (E)-3-Fluoro-3-benzoyl-2-(2-thienyl)-1,1,1-trifluoroprop-1-ene (**E-5c**)

Yield: 68%, oil; IR (film) (cm^{-1}): 1690, 1450, 1330, 1270, 1180, 1140, 990. ^1H NMR ($CDCl_3$/TMS): δ 8.00–7.96 (m, 1H), 7.71–7.51 (m, 5H), 7.42–7.40 (m, 1H), 7.16–7.13 (m, 1H). ^{19}F NMR ($CDCl_3$/TFA): δ −57.1 (d, $J = 9.4$ Hz, 3F), −94.1 (q, $J = 9.4$ Hz, 1F). MS: *m/z* (rel. int.): 302 ($M^+ + 2$, 2), 300 (M^+, 27), 281(8), 271(2), 231(100), 105(52). HRMS: *m/z* Anal. calc. for $C_{14}H_8F_4OS$ (300.27): 300.0232. Found 300.0234.

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