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An improved, efficient route to 2,2-difluoroethenylbenzenes

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Dedicated to Professor
Wei-Yuan Huang on the occasion
of his 90th birthday.

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ABSTRACT

Treatment of vinylidene fluoride with tert-BuLi at −115 °C gave a solution of [F_2C=CHLi]. Addition of Bu_3SnCl to this lithium reagent at −110 °C gave an 88% isolated yield of F_2C=CH$SnBu_3$. Reaction of F_2C=CH$SnBu_3$ with substituted aryl iodides under Stille-Liebeskind conditions [$Pd(PPh_3)_4$/Cu(I)I] at room temperature afforded the 2,2-difluoroethenylbenzines in good yield. In the absence of the Cu(I)I co-catalyst, no reaction occurred. This work provides the most efficient route for the conversion of aryl halides to 2,2-difluorostyrenes.

1. Introduction

Recently, the introduction of the 2,2-difluorovinyl and the 1,1,2-trifluorovinyl groups into organic compounds has been of interest for organic synthesis and biological studies. Munroe and co-workers have investigated coupling of unsaturated stannanes with 1-carbacephem-3-enol triflates. The vinyl stannanes consistently gave the highest yield of the coupled product and proceeded at the lowest temperature [1]. The 2,2-difluorovinylstannane gave a 78% yield of the coupled products at 25 °C. The experimental preparation of F_2C=CH$SnBu_3$ was not described. Subsequently, Farina et al. described general methodology for the synthesis of cephem side chains in cephalosporin chemistry [2]. The tables in this manuscript describe the use of F_2C=CF$SnBu_3$ (with Pd(0) catalysis) as an efficient route to coupling of vinyl triflates and 3-(halomethyl)cephems. Sauvetre and Normant reported the metallation of vinylidene fluoride with sec-butyllithium at low temperatures, as illustrated in Eq. (1) [3].

$$F_2C{=}CH_2 \xrightarrow[THF/Et_2O,\ -115\ ^\circ C]{\text{sec-BuLi}} [F_2C{=}CHLi] \quad (1)$$

Although this unstable lithium reagent was employed in the preparation of the 2,2-difluorovinylzinc reagent and added to aldehydes and ketones to provide allylic alcohols, the vinylstannane was not described [4,5]. The F_2C=CH$SnBu_3$ was prepared in our laboratory *via* the conversion of F_2C=CH$SiEt_3$ with KF and Bu_3SnCl, as illustrated in Eq. (2) [6]. Subsequently, Lentz and co-workers reported the preparation of [F_2C=CHLi] from

$$F_2C{=}CH_2 + Et_3SiCl \xrightarrow[^tBuLi,\ -110\ ^\circ C]{THF/Et_2O} F_2C{=}CHSiEt_3\ (60\%) \xrightarrow[RT\ overnight]{KF/DMF,\ Bu_3SnCl} F_2C{=}CHSnBu_3\ (92\%) \quad (2)$$

either 1,1-difluoroethene or 2-chloro-1,1-difluoroethane *via* Normant's method. Reaction of this vinyllithium with Bu_3SnCl provided the 2,2-difluoroethenylstannane, as shown in Eq. (3) [7].

$$F_2C{=}CH_2 + \text{sec-BuLi} \xrightarrow[-110\ ^\circ C]{THF/Et_2O} [F_2C{=}CHLi] \xrightarrow{Bu_3SnCl} F_2C{=}CHSnBu_3\ (78\%) \quad (3)$$

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2. Results and discussion

Recently, we reported a useful, convenient preparation of the 2,2-difluorovinylzinc reagent in good yield *via* treatment of $F_2C{=}CHI$ with acid-washed zinc in dry DMF, as shown in Eq. (4) [8]. The zinc reagent reacted smoothly with aryl iodides

$$F_2C{=}CHI + Zn \xrightarrow[60\ ^\circ C]{DMF} \underset{X=I \text{ or } F_2C=CH}{[F_2C{=}CHZnX]} \quad (4)$$

or bromides in DMF in the presence of 5 mol.% $Pd(PPh_3)_4$ at 60 °C to give 2,2-difluorostyrenes, Eq. (5) [8]. The one drawback to this approach is the use of $F_2C{=}CHI$

$$[F_2C{=}CHZnX] + \underset{R}{\bigcirc}\!\!-X \xrightarrow[\text{DMF, 60 °C}\ \text{1-4 h}]{Pd(PPh_3)_4} \underset{R}{\bigcirc}\!\!-CH{=}CF_2 \quad (5)$$

X = I, Br 69-92%

as the 2,2-difluorovinyl precursor. This vinyl iodide is not commercially available. It has been prepared by the addition of ICl to vinylidene fluoride followed by elimination of HCl with base, as illustrated in Eq. (6). In his initial report, Park employed KOH/mineral oil

$$F_2C{=}CH_2 \xrightarrow{ICl} \underset{80-90\%}{CF_2ClCH_2I} \xrightarrow{base} F_2C{=}CHI \quad (6)$$

as the base and obtained 65% of the vinyl iodide [9]. Later, Lentz utilized potassium tert-butylate as the base and obtained 62% of the vinyl iodide [7]. Thus, a two step preparation of the key precursor is employed and the overall yield of the vinyl iodide is modest at best. An alternative precursor is $F_2C{=}CHBr$. However, when this vinyl bromide was reacted with zinc to prepare the requisite vinylzinc reagent, less satisfactory results were obtained, and a novel acid–base reaction was observed and produced a mixture of vinylzinc reagents, as illustrated in Eq. (7) [8]. Thus, the vinyl iodide was the

$$F_2C{=}CHBr + Zn \xrightarrow[90\ ^\circ C/1\ h]{DMF} [F_2C{=}CHZnX] + [F_2C{=}CBrZnX] \quad (7)$$

only 2,2-difluorovinyl halide precursor that gave satisfactory results.

The 2,2-difluorostyrenes also provide a useful entry to the 2,2,2-trifluoroethylbenzenes, as shown in Eq. (8) [8]. Thus, an efficient preparation of 2,2-

$$F_2C{=}CH\!\!-\underset{R}{\bigcirc} \xrightarrow[\text{DMSO or DMF}]{KF,\ H_2O} CF_3CH_2\!\!-\underset{R}{\bigcirc} \quad (8)$$

83-95%

difluorostyrenes provides an entry to two classes of compounds.

Heitz and Knebelkamp have reported a possible simple one step approach to 2,2-difluorostyrenes *via* a Heck reaction. They reacted aryl iodides or bromides with vinylidene fluoride, utilizing palladium (II) acetate as the catalyst, as illustrated in Eq. (9) [10]. Unfortunately, the 2,2-difluorostyrenes were found only as a minor product; the

$$\underset{R}{\bigcirc}\!\!-I(Br) + F_2C{=}CH_2 \xrightarrow[\text{solvent, } Et_3N,\ 115\ ^\circ C]{Pd(OAc)_2} \underset{R}{\bigcirc}\!\!-\underset{F}{\overset{H}{C}}{=}\overset{H}{C} \quad (9)$$

32-72%

major product was 1-fluorostyrenes.

Later, Nenajdenko et al. reported the direct preparation of 2,2-difluorovinylbenzenes from aromatic aldehydes, as shown in Eq. (10) [11]. This type of

$$ArCHO \xrightarrow{NH_2NH_2\ H_2O} \left[\begin{matrix} Ar \\ H \end{matrix}\!\!>\!\!=NNH_2\right] \xrightarrow[Cu(I)Cl]{CF_2Br_2} \begin{matrix} Ar \\ H \end{matrix}\!\!>\!\!=CF_2 \quad (10)$$

20-36%

conversion, aldehydes to 2,2-difluorovinylbenzenes, is a variant of the Wittig approach to this class of compounds [12]. The approaches by Heitz and Nenajdenko simplify the approach to 2,2-difluorostyrenes and utilize readily available precursors, but they do not offer any advantage over the use of $F_2C{=}CHI$. The preparation of $F_2C{=}CHI$ utilizes vinylidene fluoride as the initial precursor. Thus, like the work of Heitz, if vinylidene fluoride could be utilized directly or in a one-step approach to a useful precursor, then perhaps a more efficient route to the 2,2-difluorostyrenes could be developed. Our choice of precursors was $F_2C{=}CHSnBu_3$. McCarthy and co-workers have demonstrated that 1-fluorovinylstannanes provides a mild, stereospecific entry to monofluoroolefins [13]. Also, recent work in our laboratory has demonstrated the utility of vinylstannanes in the preparation of functionalized vinyl fluoride derivatives [14–20].

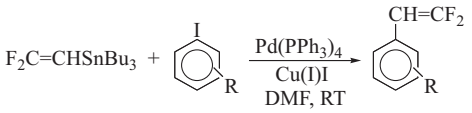
The $F_2C{=}CHSnBu_3$ can be prepared *via* the previously described route shown in Eq. (2). This route looks attractive, since all the starting materials are commercially available. However, the 60% yield of the vinylsilane has the same modest yield limitation of $F_2C{=}CHI$. Also, two steps and two isolations are required before conversion to the 2,2-difluorostyrenes. Thus, we decided to attempt a more direct route to $F_2C{=}CHSnBu_3$, and the direct conversion of this vinylstannane to the styrenes.

As noted in Eq. (1), Normant prepared the vinyllithium reagent directly from vinylidene fluoride *via* metallation with sec-BuLi. To accomplish the same conversion, we utilized tert-BuLi as the base for better handling at low temperatures (−115 °C). Initially, we attempted to accomplish this transformation in a manner similar to the preparation of $F_2C{=}CHSiEt_3$ [21]. As demonstrated by others, Et_3SiCl reacts slower in a metallation process than a vinyl hydrogen or halogen in a fluoroolefin. Thus, the olefin and Et_3SiCl can be treated at low temperature with an alkyllithium and only metallation of the olefin occurs, which then captures the Et_3SiCl to give the vinylsilane. Thus, the unstable vinyllithium does not need to be pregenerated. However, when we treated a mixture of vinylidene fluoride and Bu_3SnCl at −115 °C with tert-BuLi, a 1:1 mixture of $F_2C{=}CHSnBu_3$ and tert-$BuSnBu_3$ was observed. Bu_3SnCl is known to be a better electrophile than R_3SiCl and competes for the tert-BuLi. Consequently, we pregenerated the vinyllithium at low temperature. Addition of Bu_3SnCl to the vinyllithium provided an excellent isolated yield of $F_2C{=}CHSnBu_3$, as illustrated in Eq. (11) [22]. Subsequent Pd(0)

$$F_2C{=}CH_2 \underset{THF/Et_2O}{\xrightarrow[-115\ \text{to}\ -110\ ^\circ C]{t\text{-}BuLi}} [F_2C{=}CHLi] \xrightarrow[-105\ ^\circ C]{Bu_3SnCl} \underset{88\%}{F_2C{=}CHSnBu_3} \quad (11)$$

catalyzed coupling of $F_2C{=}CHSnBu_3$ with aromatic iodides, under Liebeskind conditions (Cu(I)I as a co-catalyst) [23,24], gave good yields of the substituted 2,2-difluorostyrenes (Table 1). Electron-withdrawing substituents on the ring promote a rapid RT reaction (entries 1–5). An electron-releasing substituent reacts at RT but at slower rate (entry 6). Note: when the co-catalyst, Cu(I)I is not utilized, the reaction does not proceed (entry 7), consistent with the formation of a vinylcopper intermediate in the Stille-Liebeskind reaction process [17].

Table 1
$Pd(PPh_3)_4/Cu(I)I$ catalyzed coupling reactions of $F_2C{=}CHSnBu_3$ with aryl iodides.



Entry	R	Rx. conditions	Yield (%)[a]
1	3-NO_2	RT/2 h	78
2	2-NO_2	RT/2 h	70
3	4-NO_2	RT/2 h	81
4	4-C(O)Me	RT/2 h	66
5	4-CN	RT/2 h	85
6	3-OMe	RT/48 h	53
7	4-CN	RT/24 h	No Rx[b]

[a] Isolated yields based on ArI.
[b] In the absence of Cu(I)I.

3. Conclusion

Vinylidene fluoride can be readily converted to $F_2C{=}CHSnBu_3$ in good yield *via* reaction of [$F_2C{=}CHLi$] and Bu_3SnCl at low temperature. The vinylstannane can be efficiently coupled with aromatic iodides using Stille-Liebeskind conditions, $Pd(PPh_3)_4$/Cu(I)I. This route provides a shorter, more efficient route to 2,2-difluorostyrenes.

4. Experimental

4.1. General

All boiling points are uncorrected. ^{19}F NMR (282.44 MHz), ^1H NMR (200.17 MHz) and ^{13}C NMR (75.48 MHz) spectra were recorded in $CDCl_3$ solvent. All chemical shifts are reported in parts per million downfield of the standards. ^{19}F NMR spectra are referenced against internal $CFCl_3$, and ^1H NMR and ^{13}C NMR spectra against TMS. FTIR spectra were recorded as CCl_4 solutions using a solution cell with a 0.1 cm path length and absorbance frequencies reported in cm^{-1}. GCMS spectra were obtained at 70 eV in the electron-impact mode. High resolution mass spectra determinations were made at the University of Iowa High Resolution Mass Spectrometry Facility.

4.2. Materials

Tert-BuLi (1.7 M in pentane) was obtained from the Aldrich Chemical Company. Vinylidene fluoride was obtained from PCR. Most aromatic iodides and n-tributyltin chloride were obtained from Aldrich and used directly. Tetrakis (triphenylphosphine) palladium was prepared by the literature procedure [25]. Cu(I)I was prepared *via* the reported procedure [26]. THF was distilled from sodium benzophenone at atmospheric pressure. DMF was dried by stirring overnight over CaH_2, then distilled at reduced pressure (bp ~65 °C/5 mm Hg) prior to use and stored under nitrogen. 4-iodobenzonitrile was obtained from Kodak and used directly. Silica gel was obtained from EM Scientific (silica gel 60, particle size 0.063–0200 μm, 70–30 Mesh, ASTM).

4.2.1. Preparation of 2,2-difluoroethenyltri-n-butylstannane

A 250 ml three-necked flask, equipped with a low temperature thermometer, a magnetic stir bar, a N_2 tee and a dry ice/acetone condenser, was charged with anhydrous ether (20 ml), anhydrous THF (40 ml), and vinylidene fluoride (5.0 g, 78 mmol) and then cooled to −115 °C with a pentane/liquid nitrogen bath. Then tert-BuLi (35 ml, 1.7 M in pentane, 59 mmol) was slowly added over 1 h (*via* syringe) with stirring. The internal temperature was maintained between −105 and −115 °C during the addition. After the addition of the tert-BuLi was completed, the solution was stirred at −100 °C for 1 h, then n-tributyltin chloride (16.28 g, 50 mmol) was added slowly over 2 h while the solution was controlled below −95 °C, Then, the reaction mixture was stirred at −95 °C for 1 h and then allowed to warm to RT over 3 h and stirred overnight at RT. The reaction mixture was treated with 0.1 N aqueous HCl (30 ml) and extracted with ether (2 × 100 ml). The combined organic layer was washed with brine and dried over anhydrous $MgSO_4$. After filtration, the ether was removed *via* rotary evaporation, and the residue was purified on a silica gel chromatography column using hexane as eluent to give 2,2-difluoroethenyltri-n-burtystannane (15.5 g, 88% yield) as a colorless liquid. The ^{19}F NMR, ^1H NMR, and ^{13}C NMR were in good agreement with the literature data [6].

4.2.2. Preparation of 3-(2,2-difluoroethenyl)nitrobenzene

General procedure: A 250 ml flask, equipped with a magnetic stir bar and septum, was charged with $Pd(PPh_3)_4$ (0.09 g, 0.078 mmol), Cu(I)I (0.19 g, 1.0 mmol), 3-iodonitrobenzene (0.50 g, 2.0 mmol), and dry DMF (10 ml). Then, $F_2C{=}CHSnBu_3$ (0.88 g, 2.5 mmol) was added at RT with stirring. The reaction was completed within 2 h at RT, as determined by ^{19}F NMR analysis for the disapperance of the vinylstannane. The reaction mixture was then diluted with Et_2O (100 ml) and washed with aqueous KF solution (15%, 50 ml). The ether layer was separated, dried over anhydrous $MgSO_4$ and concentrated. The residue was chromatographed on a silica gel column using a 1:20 mixture of ethylacetate and hexane as eluent to afford 0.29 g (78%) of 3-(2,2-difluoroethenyl) nitrobenzene as a white solid, mp 31–33 °C (lit. 32–33 °C [8]). The ^{19}F NMR, ^1H NMR, ^{13}C NMR were in good agreement with the literature data [8].

4.2.3. Preparation of 2-(2,2-difluoroethenyl)nitrobenzene

Similar to Section 4.2.2, reaction of $F_2C{=}CHSnBu_3$ (0.88 g, 2.5 mmol), 2-iodonitrobenzene (0.50 g, 200 mmol), $Pd(PPh_3)_4$ (0.09 g, 0.078 mmol), Cu(I)I (0.19 g, 1.0 mmol) and dry DMF (10 ml) at RT for 2 h yielded 2-(2,2-difluoroethenyl)nitrobenzene (0.26 g, 70%) as a yellow oil after chromatography using a mixture of ethyl acetate and hexane (1:20) as eluent. ^{19}F NMR: δ −80.56 (dd, $^2J_{FF}$ = 22.2 Hz, $^3J_{HF}$ = 3.5 Hz, 1F), −82.58 (dd, $^3J_{HF}$ = 24.5 Hz, $^2J_{FF}$ = 22.2 Hz, 1F); ^1H NMR: δ 7.98 (d, $^3J_{HH}$ = 8.0 Hz, 1H), 7.61 (d, $^3J_{HH}$ = 4.8 Hz, 2H), 7.42 (m, 1H), 5.93 (dd, $^3J_{HF}$ = 24.5 Hz, $^3J_{HF}$ = 3.5 Hz); ^{13}C NMR: δ 156.8 (dd, $^1J_{CF}$ = 299.0 Hz, $^1J_{CF}$ = 289.9 Hz), 147.8 (m), 133.1 (s), 130.4 (dd, $^4J_{CF}$ = 8.2 Hz, $^4J_{CF}$ = 1.2 Hz), 128.0 (s), 125.1 ($^3J_{CF}$ = 8.9 Hz, $^3J_{CF}$ = 5.5 Hz), 124.9 (s), 77.97 (dd, $^2J_{CF}$ = 34.7 Hz, $^2J_{CF}$ = 12.5 Hz). GC–MS, *m*/*z* (relative intensity): 185 (M^+, 25). FTIR (CCl_4, cm^{-1}): 1661.59 (C=C) cm^{-1}.

4.2.4. Preparation of 4-(2,2-difluoroethenyl)nitrobenzene

Similar to Section 4.2.2, reaction of $F_2C{=}CHSnBu_3$ (0.88 g, 2.5 mmol), 4-iodonitrobenzene (0.50 g, 2.0 mmol), $Pd(PPh_3)_4$ (0.19 g, 0.078 mmol), Cu(I)I (0.19 g, 1.0 mmol) in dry DMF (10 ml) at RT for 2 h afforded 4-(2,2-difluoroethenyl)nitrobenzene (0.30 g, 81%) as a white solid after chromatography using a mixture of ethylacetate and hexane (1:20) as eluent, mp 35–37 °C (lit. 35–37 °C [8]). The ^{19}F NMR, ^1H NMR, ^{13}C NMR were in good agreement with the literature data [8].

4.2.5. Preparation of 4-(2,2-difluoroethenyl)benzonitrile

Similar to Section 4.2.2, reaction of $F_2C{=}CHSnBu_3$ (0.88 g, 2.5 mmol), 4-iodobenzonitrile (0.46 g, 2.0 mmol), $Pd(PPh_3)_4$ (0.09 g, 0.078 mmol), Cu(I)I (0.19 g, 1.0 mmol) in dry DMF (10 ml) at RT for 2 h gave 0.28 g (85%) of 4-(2,2-difluoroethenyl) benzonitrile as colorless crystals after chromatography using a mixture of ethylacetate and hexane (1:20) as eluent, mp 64–65 °C.

The ^{19}F NMR: δ −78.36 (dd, $^{3}J_{HF}$ = 25.7 Hz, $^{2}J_{FF}$ = 20.4 Hz, 1F), −80.00 (d, $^{2}J_{FF}$ = 20.4 Hz, 1F); ^{1}H NMR: δ 7.62 (d, $^{3}J_{HH}$ = 8.3 Hz, 2H), 7.43 (d, $^{3}J_{HH}$ = 8.3 Hz, 2H), 5.35 (dd, $^{3}J_{HF}$ = 25.7 Hz, $^{3}J_{HF}$ = 3.2 Hz, 1H); ^{13}C NMR: δ 156.9 (dd, $^{1}J_{CF}$ = 301.2 Hz, $^{1}J_{CF}$ = 292.1 Hz), 135.2 (dd, $^{4}J_{CF}$ = 7.7 Hz, $^{4}J_{CF}$ = 6.2 Hz), 132.3 (s), 127.9 (dd, $^{5}J_{CF}$ = 7.0 Hz, $^{5}J_{CF}$ = 3.7 Hz), 118.5 (s), 110.4 (t, $^{6}J_{CF}$ = 2.3 Hz), 81.71 (dd, $^{2}J_{CF}$ = 30.5 Hz, $^{2}J_{CF}$ = 12.8 Hz). GC–MS, *m*/*z* (relative intensity): 165 (M^+, 100). FTIR (CCl_4, cm^{-1}): 2230.29, 1926.09, 1610.70 cm^{-1}. HRMS: calc'd. for $C_9H_5NF_2$, 165.0390; obsvd. 165.0378.

4.2.6. Preparation of 4-(2,2-difluoroethenyl)acetophenone

Similar to Section 4.2.2, reaction of $F_2C{=}CHSnBu_3$ (0.88 g, 2.5 mmol), 4-iodoacetophenone (0.49 g, 2.0 mmol), $Pd(PPh_3)_4$ (0.09 g, 0.078 mmol), Cu(I)I (0.19 g, 1.0 mmol) in dry DMF (10 ml) for 2 h yielded 0.24 g (66%) of 4-(2,2-difluoroethenyl)acetophenone as colorless crystals after chromatography using a mixture of ethylacetate and hexane (1:20) as eluent, mp 38–40 °C (lit. 38–39 °C [8]). The ^{19}F NMR, ^{1}H NMR, ^{13}C NMR and FTIR were consistent with the literature data [8]. GC–MS, *m*/*z* (relative intensity): 182 (M^+, 44), 167 (M^+–CH_3, 100), 139 (M^+–$COCH_3$, 43), 119 (M^+–$CH{=}CF_2$, 41).

4.2.7. Preparation of 3-(2,2-difluoroethenyl)anisole

Similar to Section 4.2.2, $F_2C{=}CHSnBu_3$ (0.88 g, 2.5 mmol), 3-iodoanisole (0.47 g, 2.0 mmol), $Pd(PPh_3)_4$ (0.19 g, 0.078 mmol), Cu(I)I (0.19 g, 1.0 mmol) in dry DMF (10 ml) at RT for 48 h afforded 3-(2,2-difluoroethenyl)anisole (0.18 g, 53%) as an oil after chromatography using a mixture of ethyl acetate and hexane (1:20) as eluent. The ^{19}F NMR, ^{1}H NMR, ^{13}C NMR were in good agreement with the reported literature data [8]. GC–MS, *m*/*z* (relative intensity): 170 (M^+, 100), 140 (38), 127 (47).

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