

The indium-mediated Reformatsky reactions of 2,2-difluoro-2-halo-1-furan-2-yl ethanones in aqueous media

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Abstract

New 2,2-difluoro 2-halo-1-furan-2-yl ethanones (**1a–c**) were synthesized by two different methods. The Reformatsky reactions of 2,2-difluoro-2-halo-1-furan-2-yl ethanones with various aldehydes using indium metal in aqueous media gave α,α -difluoro- β -hydroxy ketones (**4a–j**). © 2001 Elsevier Science B.V. All rights reserved.

Keywords: 2,2-Difluoro-2-halo-1-furan-2-yl ethanone; Reformatsky reaction; α,α -Difluoro- β -hydroxy ketone

1. Introduction

The difluoromethylene functionality has found widespread utility as a substituent in compounds with applications such as pharmaceutical agents and agrochemical [1–3]. For example, the difluoromethylene functional group not only facilitates the formation of stable hydrates and hemiacetals capable of inhibiting proteases and esterases [4], but can also serve as an isopolar–isosteric replacement for oxygen in phosphate analogues [5].

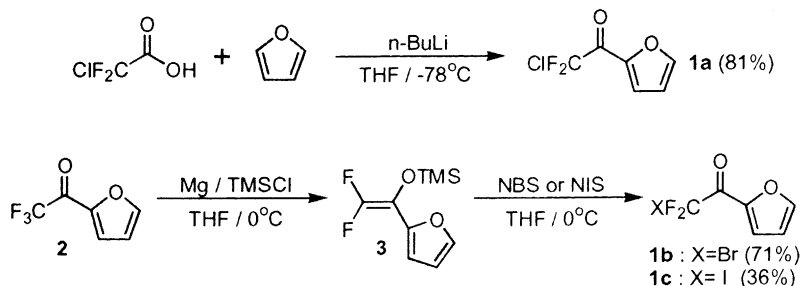
The most widely employed method for the introduction of the difluoromethylene functionality is the Reformatsky reaction of difluorohaloacetates [6–8], difluorohaloketones [9] (bromodifluoromethyl)acetylenes [10] or difluoro enol silyl ethers [11] employing zinc, zinc–copper couple, or samarium. In this work, the utility of the indium-mediated Reformatsky reaction of difluorinated compounds is reported [12].

In the 1990s, indium, uniquely, has been utilized frequently to promote organic reactions in aqueous media [13,14]. Water is an ideal solvent for organic reactions

[15–17], avoiding the need for flammable or anhydrous solvents while mitigating current environmental and economic concerns. Moreover, under these conditions, reactive functional groups, such as hydroxyl and carboxylic acid react directly without the need for time consuming protection–deprotection schemes.

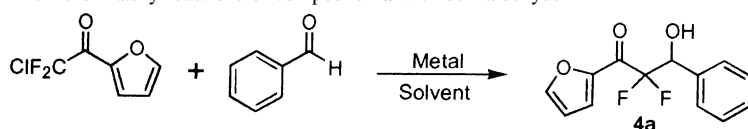
2. Results and discussion

2-Chloro-2,2-difluoro-1-furan-2-yl ethanone **1a** was synthesized in good yield by the reaction of chlorodifluoroacetic acid with lithiated furan in THF after a modest modification of a literature procedure [18,19]. 2-Bromo-2,2-difluoro-1-furan-2-yl ethanone **1b** and 2,2-difluoro-2-iodo-1-furan-2-yl ethanone **1c** were obtained by the halogenation of difluoro enol silyl ether **3** with *N*-bromosuccinimide or *N*-iodosuccinimide, respectively. The enol silyl ether was formed from 2,2-trifluoro-1-furan-2-yl ethanone **2** [20] by reductive treatment with magnesium in the presence of chlorotrimethylsilane [21].



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Table 1
The Reformatsky reactions of compound **1a** with benzaldehyde



Entry	1a (eq.)	Benzaldehyde (eq.)	Metal	Solvent	Temperature	Yield (%) ^a
1a	1.5	1	Zn(Cu) (4.5 eq.)	DMF	Ultrasound	0
1b	1.5	1	Zn(Cu) (4.5 eq.)	DMF	70 °C	68
1c	1.5	1	Zn (4.5 eq.)/CuCl (cat)	Ether:THF (4:1)	Reflux	75
1d	1	1	In (1.2 eq.)	H ₂ O:THF (4:1)	Room temperature	80

^a Isolated yield and compound is identified by ¹H, ¹³C, ¹⁹F NMR and GC–MS.

Chlorodifluoroketone **1a** was the starting material of choice as it could be synthesized easily in over a 0.1 mol scale from inexpensive chlorodifluoroacetic acid.

A series of the Reformatsky reactions of compound **1a** with benzaldehyde was studied using different metals and in diverse solvents. The results, as summarized in Table 1, indicated the indium-mediated reaction to be the method of choice as the addition to benzaldehyde can be effected stoichiometrically with compound **1a** and the aldehyde.

The influence of the water concentration of the solvent system on the indium-mediated Reformatsky reaction of **1a** and benzaldehyde was explored and is summarized in Table 2. In pure H₂O (entry **2a**), reduction of compound **1a** became a serious side reaction, lowering the yield of condensation. Curiously, no reduced product was found in undried THF (entry **2g**) but a longer reaction time was required for completing the reaction. Only trace amounts of the desired product was found in anhydrous THF freshly distilled from sodium benzophenone ketyl (entry **2h**). Even though the reaction was carried out for 4 days, unreacted starting material was recovered.

The indium-mediated Reformatsky reaction in mixtures of THF and H₂O is a three-phase reaction even though THF is miscible with water. Therefore, other water–solvent systems

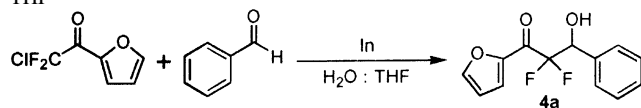
were explored including both water-miscible solvents such as dimethylformamide (DMF), 1,2-dimethoxyethane, dioxane and tetraglyme and water-immiscible solvents such as ethyl ether, dichloromethane and hexane. As summarized in Table 3, the reaction was seemingly insensitive to the organic solvent (entries **3a–g**), suggesting that the reaction occurred in the aqueous phase, where the organic phase played little or no role in the outcome of the reaction.

In a further effort to determine the influence of organic solvent on this reaction, reactions in the various solvents containing as little water as possible were examined. It had been determined that as little as 2% water was required for the formation of **4a** in reasonable yield (Table 2 entry **2f**).

The results, as summarized in Table 4, illustrate curious differences between water-miscible and water-immiscible solvents. Yields of product **4a** were moderately improved in water-miscible solvents (Table 4, entries **4b–d**) in contrast to the results found in aqueous systems containing organic solvents (Table 3, entries **3b–d**). Lower yields of product formation were found with water-immiscible solvents (Table 4, entries **4e–f**) than in aqueous systems (Table 3, entries **3e–f**).

The generality of the reaction of compound **1a** was examined with various aldehydes in the water:THF (4:1)

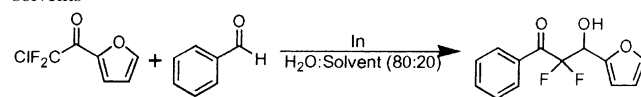
Table 2
The indium-mediated Reformatsky reaction in different ratio of H₂O and THF



Entry	Solvent	Yield (%) ^a
2a	100% H ₂ O	53
2b	80% H ₂ O:20% THF	80
2c	20% H ₂ O:80% THF	62
2d	10% H ₂ O:90% THF	60
2e	5% H ₂ O:95% THF	57
2f	2% H ₂ O:98% THF	53
2g	100% Wet THF	14
2h	100% Anhydrous THF	Trace

^a Isolated yield and compound is identified by ¹H, ¹³C, ¹⁹F NMR and GC–MS.

Table 3
The indium-mediated Reformatsky reactions in H₂O and different organic solvents

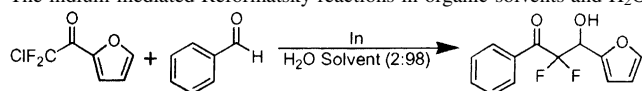


Entry	Solvent ^a	Yield (%) ^b
3a	DMF	53
3b	Dioxane	50
3c	DME	60
3d	Tetraglyme	51
3e	Hexane	60
3f	Ethyl ether	57
3g	Dichloromethane	50

^a Water (80%) and indicated solvent (20%).

^b Isolated yield and compound is identified by ¹H, ¹³C, ¹⁹F NMR and GC–MS.

Table 4
The indium-mediated Reformatsky reactions in organic solvents and H₂O



Entry	Solvent ^a	Yield (%) ^b
4a	DMF	27
4b	Dioxane	63
4c	DME	65
4d	Tetraglyme	60
4e	Hexane	47
4f	Ethyl ether	24
4g	Dichloromethane	37

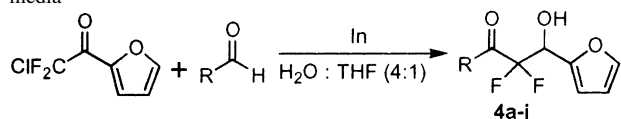
^a Water (2%) and indicated solvent (98%).

^b Isolated yield and compound is identified by ¹H, ¹³C, ¹⁹F NMR and GC–MS.

solvent system as the best previous yields were obtained under these conditions (Table 5). Aromatic aldehydes (entries **5a–c**) generally reacted in higher yields in contrast to reactions with aliphatic aldehydes. In the case of aliphatic aldehydes, linear aldehydes (entries **5d–f**) gave better results than branched aldehydes. Reactions of **1a** with branched aldehydes occurred only in low yields (entries **5h–j**). In the case of **4h** formation (entry **5h**), 3 eq. of the aldehyde and 2,2-difluoro-2-iodo-1-furan-2-yl ethanone **1c** was employed to suppress the homocoupling of the difluoroketone, which prominently took place with the chlorodifluoroketone **1a**. The reaction of *trans*-cinnamaldehyde gave the 1,2-addition product exclusively (entry **5g**).

2,2-Difluoro-2-halo-1-furan-2-yl ethanones (**1a–c**) were prepared for use in indium-mediated Reformatsky reaction. The indium-mediated addition of **1a** to benzaldehyde in aqueous solvent systems containing as little as 20% organic

Table 5
The indium-mediated Reformatsky reactions with aldehydes in aqueous media



Entry	Aldehyde	Product	Yield (%) ^a
5a	Benzaldehyde	4a	80
5b	4-Chlorobenzaldehyde	4b	73
5c	2-Furaldehyde	4c	50
5d	Butyraldehyde	4d	42
5e	Heptaldehyde	4e	60
5f	Dihydrocinnamaldehyde	4f	40
5g	<i>trans</i> -Cinnamaldehyde	4g	67
5h	Isobutyraldehyde	4h	42 ^b
5i	2-Ethylbutyraldehyde	4i	<20 ^c
5j	Cyclohexanecarboxaldehyde	4j	<20 ^c

^a Isolated yield and compound is identified by ¹H, ¹³C, ¹⁹F NMR and GC–MS.

^b Compound **1c** was used.

^c Cannot be separated in pure.

solvents by volume resulted in the formation of α,α -difluoro- β -hydroxy ketone **4a** independent of whether water-miscible solvents or water-immiscible organic solvents were employed. In contrast, the formation of **4a** in organic solvent systems containing as little as 2% water gave different results. Moderately improved yields for the formation **4a** were observed in water-miscible solvents contrasting with diminished yields obtained in water-immiscible solvents. The generality of the indium-mediated Reformatsky reactions of compound **1a** with various aldehydes was shown with the formation of diverse α,α -difluoro- β -hydroxy ketone derivatives (**4a–j**) in moderate yields.

3. Experimental

¹H NMR (300 MHz), ¹³C NMR (75 MHz) and ¹⁹F NMR (282 MHz) spectra were measured in CDCl₃ using CHCl₃ and CFC₃ as internal standards. Mass spectra were measured on Hewlett-Packard 5989A mass spectrometer (70 eV).

3.1. 2-Chloro-2,2-difluoro-1-furan-2-yl ethanone (**1a**)

n-BuLi (60 mmol, 24 ml of 2.5 M solution in hexane) was added to a solution of furan (60 mmol) in 50 ml of THF cooled to 0 °C and then stirred for 1 h. The mixture was cooled to –78 °C and then 2-chloro-2,2-difluoroacetic acid (20 mmol) was added. The resulting solution was stirred for 4 h at –78 °C and then poured into 100 ml of 10% HCl solution cooled to 0 °C. The mixture was stirred for 1 h at 0 °C and then extracted with ethyl ether (50 ml) three times. The organic layer was dried over anhydrous MgSO₄, filtered, and concentrated. The product was purified by vacuum distillation.

Yield 81%: bp 64 °C/5 mmHg. ¹H NMR δ = 7.79 (d, *J* = 1.4 Hz, 1H), 7.50 (m, 1H), 6.65 (dd, *J* = 3.8, 1.7 Hz, 1H); ¹⁹F NMR δ = –63.5 (s, 2F); ¹³C NMR δ = 170 (t, *J* = 32 Hz), 150, 146, 124 (t, *J* = 3.5 Hz), 120 (t, *J* = 303 Hz), 113.

3.2. General procedure for preparation of 2,2-difluoro-2-halo-1-furan-2-yl ethanones (**1b–c**)

To a suspension of magnesium (8 mmol, turnings) and chlorotrimethylsilane (4 mmol) in 5 ml of THF cooled to 0 °C was added 2,2,2-trifluoro-1-furan-2-yl ethanone **2** (1 mmol). After stirring for 1 h, the resulting solution was filtered and poured into a solution of NBS or NIS (1.5 mmol) in 5 ml of THF cooled to 0 °C. The resultant mixture was stirred for an additional 1 h, then was quenched with 5 ml of H₂O and extracted with ethyl ether (10 ml) three times. The organic layer was dried over anhydrous MgSO₄, filtered, and concentrated. The difluorohaloketones were purified by silica gel column chromatography eluting with a mixture of hexane and ethyl acetate (20:1).

3.2.1. 2-Bromo-2,2-difluoro-1-furan-2-yl ethanone (**1b**)

Yield: 71%. $^1\text{H NMR}$ δ = 7.78 (d, J = 2.3 Hz, 1H), 7.51 (m, 1H), 6.65 (dd, J = 3.7, 1.6 Hz, 1H); $^{19}\text{F NMR}$ δ = -60.3 (s, 2F); $^{13}\text{C NMR}$ δ = 170 (t, J = 29 Hz), 150, 145, 124 (t, J = 3.9 Hz), 113, 112 (t, J = 317 Hz).

3.2.2. 2,2-Difluoro-2-iodo-1-furan-2-yl ethanone (**1c**)

Yield: 36%. $^1\text{H NMR}$ δ = 7.77 (d, J = 1.1 Hz, 1H), 7.50 (m, 1H), 6.65 (dd, J = 3.8, 1.7 Hz, 1H); $^{19}\text{F NMR}$ δ = -56.7 (s, 2F); $^{13}\text{C NMR}$ δ = 171 (t, J = 25 Hz), 150, 145, 124 (t, J = 4.2 Hz), 113, 93.8 (t, J = 325 Hz).

3.3. 2,2,2-Trifluoro-1-furan-2-yl ethanone (**2**)

It was prepared according to the method reported in the literature [20].

3.4. (2,2-Difluoro-1-furan-2-yl-vinyloxy)-trimethylsilane (**3**)

It was prepared according to the method reported in the literature [21].

3.5. Reformatsky reaction of **1a** and benzaldehyde with zinc-copper couple

To a suspension of **1a** (136 mg, 0.75 mmol) and zinc-copper couple [22] (147 mg, 2.25 mmol) in 5 ml of DMF was added benzaldehyde (53 mg, 0.5 mmol) and mixture was heated at 70 °C for 20 h. The reaction was quenched with 10% HCl solution and extracted with ethyl ether (20 ml) three times. The organic layer was dried over anhydrous MgSO_4 filtered and concentrated. Purification by silica gel column chromatography afforded the product **4a** in 68% yield.

3.6. Reformatsky reaction of **1a** and benzaldehyde with acid washed zinc and copper(I) chloride

A suspension of acid washed zinc (147 mg, 2.25 mmol) and copper(I) chloride (23 mg, 0.25 mmol) in 4 ml of anhydrous ethyl ether and 1 ml of anhydrous THF was stirred for 30 min at room temperature. Benzaldehyde (53 mg, 0.5 mmol) and **1a** (136 mg, 0.75 mmol) were added to the suspension and mixture was refluxed for 16 h. The reaction was quenched with 10% HCl solution and was extracted with ethyl ether (20 ml) three times. Organic layer was dried over anhydrous MgSO_4 , filtered, and concentrated. Purification by silica gel column chromatography afforded the product **4a** in 75% yield.

3.7. General procedure for the preparation of α,α -difluoro- β -hydroxy ketones (**4a-h**)

To a solution of compound **1a** (1 mmol) and an aldehyde (1 mmol) stirred with H_2O (4 ml) and THF (1 ml) was added indium (1.2 mmol, -100 mesh powder) and the mixture was

vigorously stirred for 24 h. The reaction was quenched with saturated NH_4Cl solution and was then extracted with ethyl acetate (10 ml) three times. The organic layer was dried over anhydrous MgSO_4 , filtered, and concentrated. Purification by silica gel column chromatography afforded the pure products.

3.7.1. 2,2-Difluoro-1-furan-2-yl-3-hydroxy-3-phenylepropan-1-one (**4a**)

Yield: 80%. $^1\text{H NMR}$ δ = 7.8 (d, J = 1.3 Hz, 1H), 7.7, 7.0 (m, 6H), 6.6 (dd, J = 3.7, 1.6 Hz, 1H), 5.4 (dd, J = 17.5, 7 Hz, 1H), 3.7, 3.2 (br, 1H); $^{19}\text{F NMR}$ δ = -109 (dd, J = 273 Hz, 6.3, 1F), -120 (dd, J = 273, 17 Hz, 1F); $^{13}\text{C NMR}$ δ = 178 (dd, J = 32, 29 Hz), 149, 135, 129, 128, 127, 124 (t, J = 4.1 Hz), 115 (dd, J = 262, 255 Hz), 112, 73 (dd, J = 28, 24 Hz); MS (EI/70 eV) m/z (%) 252 $[\text{M}]^+$ (0.3), 232 (19), 146 (100), 79 (67), 77 (55), 51 (28).

3.7.2. 3-(4-Chloro-phenyl)-2,2-difluoro-1-furan-2-yl-3-hydroxypropan-1-one (**4b**)

Yield: 73%. $^1\text{H NMR}$ δ = 7.7 (d, J = 1 Hz, 1H), 7.5, 7.2 (m, 5H), 6.5 (dd, J = 3.5, 1.4 Hz, 1H), 5.3 (dd, J = 17.8, 6.1 Hz, 1H), 3.2, 2.9 (br, 1H); $^{19}\text{F NMR}$ δ = -108 (dd, J = 280, 5.8 Hz, 1F), -120 (dd, J = 280, 18 Hz, 1F); $^{13}\text{C NMR}$ δ = 178 (dd, J = 32, 29 Hz), 149, 135, 133, 129, 128, 124 (dd, J = 8.0, 4.1 Hz), 115 (dd, J = 263, 256 Hz), 113, 72 (dd, J = 28, 24 Hz); MS (EI/70 eV) m/z (%) 285 $[\text{M}-1]^+$ (0.2), 265(11), 146 (100), 139(37), 95(58), 77 (37), 51 (14).

3.7.3. 2,2-Difluoro-1, 3-difuran-2-yl-3-hydroxypropan-1-one (**4c**)

Yield: 50%. $^1\text{H NMR}$ δ = 7.7 (d, J = 1.3 Hz, 1H), 7.5, 7.4 (m, 1H), 7.4 (d, J = 2.3 Hz, 1H), 6.6 (dd, J = 3.8, 1.7 Hz, 1H), 6.5 (d, J = 3.3 Hz, 1H), 6.4 (dd, J = 3.3, 1.8 Hz, 1H), 5.4 (dd, J = 16, 7.5 Hz, 1H), 2.9, 2.7 (br, 1H); $^{19}\text{F NMR}$ δ = -110 (dd, J = 276, 7.5 Hz, 1F), -118 (dd, J = 276, 15.5 Hz, 1F); $^{13}\text{C NMR}$ δ = 177 (t, J = 31 Hz), 149, 143, 124 (dd, J = 7.5, 4.3 Hz), 115 (dd, J = 262, 257 Hz), 112, 111, 110, 68 (dd, J = 29, 25 Hz); MS (EI/70 eV) m/z (%) 242 $[\text{M}]^+$ (0.9), 222 (36), 146 (65), 97 (100), 69 (20), 51 (13).

3.7.4. 2,2-Difluoro-1-furan-2-yl-3-hydroxyhexan-1-one (**4d**)

Yield: 42%. $^1\text{H NMR}$ δ = 7.7 (t, J = 1, 1 Hz, 1H), 7.5, 7.4 (m, 1H), 6.6 (dd, J = 3.6, 1.7 Hz, 1H), 4.3, 4.0 (m, 1H), 2.4, 2.1 (br, 1H), 1.8, 1.3 (m, 4H), 0.95 (t, J = 7 Hz, 3H); $^{19}\text{F NMR}$ δ = -112 (dd, J = 280, 7 Hz, 1F), -121 (dd, J = 280, 17 Hz, 1F); $^{13}\text{C NMR}$ δ = 178 (t, J = 31 Hz), 148, 123 (t, J = 5.1 Hz), 117 (dd, J = 259, 256 Hz), 112, 71 (dd, J = 27, 24 Hz), 31, 18, 13; MS (EI/70 eV) m/z (%) 218 $[\text{M}]^+$ (0.7), 146 (33), 95 (100), 68 (10), 55 (14).

3.7.5. 2,2-Difluoro-1-furan-2-yl-3-hydroxynonan-1-one (**4e**)

Yield: 60%. $^1\text{H NMR}$ δ = 7.7 (d, J = 1.4 Hz, 1H), 7.5, 7.4 (m, 1H), 6.6 (d, J = 3.7, 1.7 Hz, 1H), 4.2, 4.0 (m, 1H),

2.8, 2.2 (br, 1H), 1.8, 1.5 (m, 2H), 1.4, 1.2 (m, 8H), 0.85 (t, $J = 6.7$ Hz, 3H); ^{19}F NMR $\delta = -115$ (dd, $J = 279.7$ Hz, 1F), -121 (dd, $J = 279$, 16 Hz, 1F); ^{13}C NMR $\delta = 178$ (t, $J = 31$ Hz), 149, 124 (t, $J = 5.4$ Hz), 116 (t, $J = 257$ Hz), 112, 71 (t, $J = 26$ Hz), 32, 29, 25, 22, 13; MS (EI/70 eV) m/z (%) 260 $[M]^+$ (0.6), 146 (13), 95 (100), 55 (21).

3.7.6. 2,2-Difluoro-1-furan-2-yl-3-hydroxy-5-phenyl pentan-1-one (4f)

Yield: 40%. ^1H NMR $\delta = 7.7$ (t, $J = 1$ Hz, 1H), 7.6, 7.4 (m, 1H), 7.3, 7.2 (m, 5H), 6.6 (dd, $J = 3.6$, 1.6 Hz, 1H), 4.3, 4.1 (m, 1H), 3.0, 2.8 (m, 1H), 2.7–2.6 (m, 1H), 2.4 (d, $J = 5.2$ Hz, 1H), 2.1, 1.8 (m, 2H); ^{19}F NMR $\delta = -112$ (dd, $J = 281$, 6 Hz, 1F), -120 (dd, $J = 281$, 17 Hz, 1F); ^{13}C NMR $\delta = 178$ (t, $J = 30$ Hz), 149, 141, 128.5, 128.4, 126, 124 (t, $J = 5.7$ Hz), 116 (dd, $J = 260$, 256 Hz), 113, 70 (t, $J = 28$, 24 Hz), 31, 30; MS (EI/70 eV) m/z (%) 280 $[M]^+$ (1.6), 242 (6), 146 (15), 95 (100), 91 (65), 77 (12), 65 (16).

3.7.7. trans-2,2-Difluoro-1-furan-2-yl-3-hydroxy-5-phenyl pent-4-en-1-one (4g)

Yield: 67%. ^1H NMR $\delta = 7.7$ (d, $J = 1.4$ Hz, 1H), 7.6, 7.5 (m, 1H), 7.4, 7.2 (m, 5H), 6.8 (d, $J = 16$ Hz, 1H), 6.6 (dd, $J = 3.7$, 1.6 Hz, 1H), 6.3 (dd, $J = 16$, 6.5 Hz, 1H), 5.0, 4.9 (m, 1H), 2.7 (d, $J = 6.1$ Hz, 1H); ^{19}F NMR $\delta = -110$ (dd, $J = 279$, 6.7 Hz, 1F), -119 (dd, $J = 279$, 15 Hz, 1F); ^{13}C NMR $\delta = 178$ (t, $J = 31$ Hz), 149, 136, 135, 129, 128, 127, 124, 122, 116 (dd, $J = 260$, 256 Hz), 113, 72 (dd, $J = 28$, 25 Hz); MS (EI/70 eV) m/z (%) 278 $[M]^+$, 0.2, 222 (4), 46 (26), 131 (77), 103 (56), 95 (100), 77 (64), 51 (94).

3.7.8. 2,2-Difluoro-1-furan-2-yl-3-hydroxy-4-methyl pentan-1-one (4h)

Yield: 67%. ^1H NMR $\delta = 7.7$ (d, $J = 2.2$ Hz, 1H), 7.4 (m, 1H), 6.6 (dd, $J = 3.8$, 1.5 Hz, 1H), 3.9 (ddd, $J = 19.6$, 7.0,

5.2 Hz, 1H), 2.6 (br, 1H), 2.1 (oct, $J = 6.7$ Hz, 1H), 1.0 (t, $J = 6.2$ Hz, 6H); ^{19}F NMR $\delta = 108$ (dd, $J = 276$, 6.4 Hz, 1F), -119 (dd, $J = 276$, 20 Hz, 1F); ^{13}C NMR $\delta = 178$ (t, $J = 32$ Hz), 149, 124 (dd, $J = 7$, 4 Hz), 117 (dd, $J = 261$, 257 Hz), 113, 75 (dd, $J = 26$, 23 Hz), 28 (d, $J = 2.2$ Hz), 20, 17; MS (EI/70 eV) m/z (%) 217 $[M - 1]^+$ (0.03), 98 (18), 89 (18), 83 (27), 71(100), 56 (44).

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