# Supporting Information

# Deoxygenative Nucleophilic Difluoromethylselenylation of Carboxylic Acids and Alcohols with BT-SeCF<sub>2</sub>H

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## 1 General Information

Unless otherwise stated, all reactions were performed under argon as inert gas. The glass apparatus used was heated in an oil pump vacuum and purged with argon. Screw-cap reaction vessels were rinsed with argon. All purchased chemicals were used without further treatment. Used THF was dried and distilled over sodium in the presence of benzophenone and DCM was taken from the solvent purification system MB-SPS-800 (Braun). All dry solvents were stored over molecular sieves (3 or 4 Å). The solvents pentane, DCM and EtOAc used for column chromatography were distilled prior to use. 2-Bis(benzo[*d*]thiazol-2-yl)diselane was prepared according to the procedure of Mitsunobu.<sup>[1]</sup> All other starting materials were purchased from commercial suppliers and used as received.

Thin-layer chromatography was performed on silica gel coated aluminium plates ALUGRAM<sup>®</sup> Xtra SIL G/UV254 (Macherey-Nagel). The product spots were detected by UV light (254 nm) or as permanganate stains. Flash column chromatography was performed with silica gel 60 M (0.040–0.063 mm, 230–400 mesh, Macherey-Nagel).

<sup>1</sup>H, <sup>19</sup>F and <sup>13</sup>C NMR spectra were acquired on a JEOL ECS 400 (400 MHz), JEOL ECZ 400 (400 MHz), JEOL ECX 400 (400 MHz), JEOL ECP 500/ Bruker Avance 500 (500 MHz), Varian INOVA 600 (600 MHz) or a Bruker Avance 700 (700 MHz) and analysed on MestReNova. Chemical shifts ( $\delta$ ) are reported in parts per million (ppm) relative to tetramethyl silane (TMS) and coupling constants (*J*) are presented in hertz (Hz). CD<sub>3</sub>CN or CDCl<sub>3</sub> are used as deuterated solvent and the residual solvent signals are used as reference in the <sup>1</sup>H and <sup>13</sup>C NMR spectra. <sup>19</sup>F NMR spectra are not calibrated by an internal reference, but with CFCl<sub>3</sub> as reference. <sup>1</sup>H NMR yields were measured using CH<sub>2</sub>Br<sub>2</sub> as an internal standard. The multiplicities have been explained using the following abbreviations : s = singlet, d = doublet, t = triplet, q = quartet, and m = multiplet.

CHN Analysis was obtained on an ELEMENTAR Vario El elemental analyzer and High-resolution mass spectra were measured with an Agilent (6210 ESI-TOF; 4  $\mu$ L/min, 1.0 bar, 4 kV) instrument. Infrared spectra were measured with a NICOLET spectrometer (iS10) equipped with an ATR unit (NICOLET SMART DuraSampl*IR*) and only diagnostic absorption bands are reported.

**Safety Notice**: As recommended for all organoselenium compounds, the synthesis of BT-SeCF<sub>2</sub>H and all deoxygenative difluoromethylselenylation reactions (including work-up, purification) should be conducted in well-ventilated fumehoods using appropriate personal protective equipment.

## 2 Synthesis of BT-SeCF<sub>2</sub>H

#### 2.1 Synthesis of difluoromethyl trifluoromethanesulfonate



Difluoromethyl trifluoromethanesulfonate was prepared according to a literature procedure:<sup>[2]</sup> TiCl<sub>4</sub> (1 mol%, 2.21 mmol, 243 µL) was added dropwise to TfOH (1.2 equiv., 288 mmol, 25.5 mL) under vigorous stirring at room temperature for 5 mins. The homogeneous yellow solution was evacuated at 13–20 mbar until gas evolution ceased. The mixture was cooled to -20 °C, TMSCF<sub>3</sub> (1.0 equiv., 221.5 mmol, 32.8 mL) was added, and the mixture was kept for 2 min. The cooling bath was replaced by an ice bath, which was kept for 2 min, and the mixture was stirred at room temperature for 1 hour. Volatile materials were distilled off under a vacuum of 133 mbar in a cold trap. The collected liquid was purified via vacuum distillation to provide HCF<sub>2</sub>OTf as a colorless liquid (34.6 g, 172.8 mmol, 78 %).

<sup>1</sup>H NMR (400 MHz, Chloroform-d)  $\delta$  = 6.85 (t, *J* = 68.0 Hz, 1H). <sup>19</sup>F NMR (376 MHz, Chloroform-d)  $\delta$  = -74.60 (s), -82.17 (d, *J* = 68.2 Hz).

The characterization data agrees with literature values.<sup>[2]</sup>

#### 2.2 Synthesis of 2-((trifluoromethyl)thio)benzo[d]thiazole



2-Bis(benzo[d]thiazol-2-yl)diselane<sup>[1]</sup> (**1**, 1.1 eq, 26.5 mmol, 11.3 g) was suspended in degassed MeOH/THF (4:1, 350 mL) and NaBH<sub>4</sub> (2.4 eq, 57.8 mmol, 2.2 g) was added portionwise under vigorous stirring at 0 °C. After 10 min, degassed 1M HCl (80 mL) was added, and the precipitate was washed with degassed H<sub>2</sub>O (3 x 50 mL). The solid was added to a degassed solution of 6M KOH (55 mL) and MeCN (55 mL) before difluoromethyl trifluoromethanesulfonate (2 eq, 48.2 mmol, 9.64 g) was added to the mixture at 0 °C. The mixture was stirred for 15 min at 0 °C and was diluted with H<sub>2</sub>O (100 mL) and extracted with diethyl ether (200 mL). The organic phase was dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated *in vacuo*. The crude product was purified by column chromatography (SiO<sub>2</sub>, Pentane/DCM 10:1). Yellow solid (**2**, 9.1 g, 34.45 mmol, 71 %).

<sup>1</sup>H NMR (400 MHz, Chloroform-d) δ = 8.03 (d, J = 8.2 Hz, 1H), 7.84 (t, J = 54.3 Hz, 1H), 7.83 (d, J = 8.0 Hz, 1H), 7.47 (t, J = 8.0 Hz, 1H), 7.38 (t, J = 7.8 Hz, 1H). <sup>19</sup>F NMR (376 MHz, Chloroform-d) δ = -89.93 (d, J = 54.3 Hz). <sup>13</sup>C NMR (101 MHz, Chloroform-d<sub>3</sub>) δ = 153.18, 151.24, 136.56, 126.14, 125.13, 122.40, 120.75, 117.65 (t, J = 290.6 Hz). IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] =1460, 1392, 1296, 1252, 1105, 1050, 952, 749, 565. HRMS (ESI) m/z: [M+Na]\*Calcd for C<sub>8</sub>H<sub>5</sub>F<sub>2</sub>NSSe 264.9276; Found 264.9296. Melting point = 77-79 °C

## 2.3 Synthesis of 2-((difluoromethyl)selanyl)-3-methylbenzo[d]thiazol-3-ium trifluoromethanesulfonate (BT-SeCF<sub>2</sub>H)



2-((Trifluoromethyl)thio)benzo[d]thiazole (1.0 equiv., 34.45 mmol, 9.1 g) was dissolved in dry  $CH_2Cl_2$  (1.0 M) and methyl trifluoromethanesulfonate (3 equiv., 103.4 mmol, 11.4 mL) was added dropwise. The reaction mixture was stirred overnight at rt and the product was precipitated with diethyl ether. The crude product was redissolved with  $CH_2Cl_2$ , precipitated with diethyl ether a second time, and dried *in vacuo*. BT-SeCF<sub>2</sub>H was obtained as an off-white solid (14.2 g, 33.2 mmol, 96 %, 68 % over two steps from **1**).

<sup>1</sup>H NMR (600 MHz, Acetonitrile-d<sub>3</sub>)  $\delta$  = 8.31 (d, *J* = 8.3 Hz, 1H), 8.18 (d, *J* = 8.6 Hz, 1H), 7.95 (t, *J* = 7.7 Hz, 1H), 7.86 (t, *J* = 7.7 Hz, 1H), 7.77 (d, *J* = 52.4 Hz, 1H), 4.33 (s, 3H).<sup>19</sup>F NMR (565 MHz, Acetonitrile-d<sub>3</sub>)  $\delta$  = -79.23, -87.86 (d, *J* = 52.5 Hz). <sup>13</sup>C NMR (151 MHz, Acetonitrile-d<sub>3</sub>)  $\delta$  = 163.91, 143.60, 133.74, 131.22, 130.03, 124.61, 121.80 (q, *J* = 320.8 Hz), 118.45 (t, *J* = 295.4 Hz), 40.71. IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] = 1581, 1440, 1390, 1265, 1245, 1223, 1144, 1067, 1028, 756, 674, 637, 572. HRMS (ESI) m/z: [M]\*Calcd for C<sub>9</sub>H<sub>8</sub>F<sub>2</sub>NSSe 279,9505; Found 279,9497. Melting point = 110-112°C

## 3 Optimization of the Deoxydifluoromethyselenylation of carboxylic acids and alcohols with BT-SeCF<sub>2</sub>H



## 3.1 Optimization table with aliphatic acid 3a

## 3.2 Optimization table with aromatic acid 3m





Equiv. of BT-SeCF <sub>2</sub> H	NMR Yield of 4m
1.3	23%
1.6	59%
2	91%

## 3.3 Optimization tables with alcohols

## 3.3.1 Optimization table with electron deficient substrate 5a





6a

Equiv. of BT-SeCF <sub>2</sub> H	Base	Equiv. of	Temp.	Time	Notes	NMR Yield	
		Base				of 6a	
1.25	DIPEA	2	r.t.	2 h		42%	
1.25	DIPEA	2	r.t.	4 h		35%	
1.5	DIPEA	2	r.t.	2 h		40%	
2	DIPEA	2	r.t.	2 h		33%	
2	DIPEA	2	r.t	4 h	Portion-wise addition of	39%	
					BTSeCF <sub>2</sub> H (0.75 after 2 h)		
1.25	DIPEA	2	-40°C	2 h		45%	
1.25	DIPEA	2	-40°C	4 h		46%	
1.25	NaH	2	-40°C	2 h		29%	
1.25	Proton	2	-40°C	2 h		33%	
	sponge						
1.25	DIPEA	2	-40°C	2 h	degassed	42%	
1.5	DIPEA	4	-40°C	4 h	0.25 equiv. of BTSeCF <sub>2</sub> H and 2 equiv. of DIPEA were added after 2 h	65%	

BT-SeCF<sub>2</sub>H (x equiv.) Base (x equiv.)

MeCN, temperature, time

## 3.3.2 Optimization table with electron rich substrate 5f



i) BT-SeCF<sub>2</sub>H (1.25 equiv.) DIPEA (2 equiv.), Ag<sup>I</sup> salt (x equiv.) solvent, temperature, 2h

ii) BT-SeCF<sub>2</sub>H (0.25 equiv.)

DIPEA (2 equiv.) temperature, 2h



Silver(I) salt	Equiv. of Silver(I) salt	Solvent	Temperature	NMR Yield of 6f
		MeCN	-40°C	31%
		DCM	-78°C	12%
Ag <sub>2</sub> O	0.5 (1 equiv. of Ag⁺)	DCM	-78°C	41%
		THF	-78°C	traces
Ag <sub>2</sub> O	0.5 (1 equiv. of Ag⁺)	THF	-78°C	29%
Ag <sub>2</sub> O	0.15 (0.3 equiv. of Ag <sup>+</sup> )	MeCN	-40°C	51%
Ag <sub>2</sub> O	0.2 (0.4 equiv. of Ag⁺)	MeCN	-40°C	58%
Ag <sub>2</sub> O	0.25 (0.5 equiv. of Ag <sup>+</sup> )	MeCN	-40°C	63%
Ag <sub>2</sub> O	0.5 (1 equiv. of Ag⁺)	MeCN	-40°C	50%
Ag <sub>2</sub> O	1 (2 equiv. of Ag⁺)	MeCN	-40°C	50%
Ag <sub>2</sub> O	1.5 (3 equiv. of Ag⁺)	MeCN	-40°C	48%
Ag <sub>2</sub> O	2 (4 equiv. of Ag⁺)	MeCN	-40°C	44%
AgNO <sub>3</sub>	0.5	MeCN	-40°C	70%
AgOTf	0.5	MeCN	-40°C	81%

## 4 Scope and Limitations of the Deoxydifluoromethylselenylation of carboxylic acids and alcohols

## 4.1 General Procedures for the Deoxydifluoromethylselenylesterification of Carboxylic acids

Sodium hydride (2.0 equiv.) and the acid (1.0 equiv., 0.3 mmol) were dissolved in dry THF (at 45°C for aliphatic acids, at rt for aromatic acids) and were allowed to react for 0.5 h. BT-SeCF<sub>2</sub>H (2.0 equiv,) was added and the mixture was stirred for 2 h at rt. The reaction mixture was filtered through a silica pad to remove solids and the crude difluororomethylselenoesters were isolated using column chromatography (SiO<sub>2</sub>).

## 4.2 Characterization Data for Deoxydifluoromethylselenylester Products 4

## Se-(difluoromethyl) dodecaneselenoate (4a)



According to the general procedure, selenoester **4a** (73 mg, 78%) was obtained from dodecanoic acid **3a** on a 0.3 mmol scale after flash column chromatography (pentane) as a colorless oil.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\boldsymbol{\sigma}$  = 7.44 (t, *J* = 53.5 Hz, 1H), 2.68 (t, *J* = 7.5 Hz, 2H), 1.68 (p, *J* = 7.5 Hz, 2H), 1.37 – 1.22 (m, 16H), 0.88 (t, *J* = 7.0 Hz, 3H).<sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\boldsymbol{\sigma}$  = -96.16 (d, *J* = 53.4 Hz).<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\boldsymbol{\sigma}$  = 198.20, 120.15 (t, *J* = 283.4 Hz), 49.19, 32.04, 29.70, 29.67, 29.46, 29.46, 29.28, 28.82, 24.96, 22.82, 14.25.

The characterization data agree with the literature values.<sup>[3]</sup>

## Se-(difluoromethyl) tetradecaneselenoate (4b)



According to the general procedure, selenoester **4b** (89 mg, 87%) was obtained from tetradecanoic acid **4b** on a 0.3 mmol scale after flash column chromatography (pentane) as a colorless oil.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.44 (t, J = 53.5 Hz, 1H), 2.68 (t, J = 7.5 Hz, 2H), 1.68 (p, J = 7.5 Hz, 2H), 1.38 – 1.19 (m, 20H), 0.88 (t, J = 7.0 Hz, 3H). <sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -96.15 (d, J = 53.6 Hz). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 198.18, 120.15 (t, J = 283.3 Hz), 49.19, 32.07, 29.80, 29.78, 29.75, 29.68, 29.50, 29.47, 29.29, 28.83, 24.96, 22.84, 14.25.

## Se-(difluoromethyl) octadecaneselenoate (4c)



According to the general procedure, selenoester **4c** (103 mg, 86%) was obtained from octadecanoic acid **3c** on a 0.3 mmol scale after flash column chromatography (pentane) as a colorless oil.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.44 (t, *J* = 53.5 Hz, 1H), 2.68 (t, *J* = 7.5 Hz, 2H), 1.68 (p, *J* = 7.5 Hz, 2H), 1.42 – 1.16 (m, 28H), 0.88 (t, *J* = 6.9 Hz, 3H). <sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -96.15 (d, *J* = 53.0 Hz). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 198.15, 120.15 (t, *J* = 283.3 Hz), 49.18, 32.09, 29.85, 29.83, 29.83, 29.82, 29.80, 29.79, 29.76, 29.68, 29.52, 29.47, 29.29, 28.83, 24.96, 22.85, 14.25.

The characterization data agree with the literature values.<sup>[3]</sup>

## Se-(difluoromethyl) 2,2-diphenylethaneselenoate (4d)



According to the general procedure, selenoester **4d** (66 mg, 67%) was obtained from 2,2-diphenylacetic acid **3d** on a 0.3 mmol scale after flash column chromatography (pentane/DCM 20:1) as a yellow oil.

<sup>1</sup>H NMR (601 MHz, CDCI<sub>3</sub>)  $\delta$  = 7.43 (t, *J* = 53.3 Hz, 1H), 7.41 – 7.30 (m, 10H), 5.17 (s, 1H).<sup>19</sup>F NMR (565 MHz, CDCI<sub>3</sub>)  $\delta$  = -96.59 (d, *J* = 53.3 Hz).<sup>13</sup>C NMR (151 MHz, CDCI<sub>3</sub>)  $\delta$  = 199.37, 136.39, 129.49, 129.10, 128.44, 120.30 (t, *J* = 283.9 Hz), 69.44. IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] = 3029, 1715, 1494, 1452, 1269, 1056, 977, 728, 686, 643, 589. HRMS (ESI) m/z: [M+Na]<sup>+</sup> Calcd for C<sub>15</sub>H<sub>12</sub>F<sub>2</sub>NaOSe 348.9919; Found 348.9913.

## Se-(difluoromethyl)-adamantane-1-carboselenoate (4e)



According to the general procedure, selenoester **4e** (78 mg, 89%) was obtained from adamantane 1-carboxylic acid **3e** on a 0.3 mmol scale after flash column chromatography (pentane) as a white solid.

<sup>1</sup>H NMR (601 MHz, CDCI<sub>3</sub>)  $\delta$  = 7.37 (t, *J* = 53.8 Hz, 1H), 2.09 (s, 3H), 1.88 (d, *J* = 3.0 Hz, 6H), 1.80 – 1.66 (m, 6H).<sup>19</sup>F NMR (565 MHz, CDCI<sub>3</sub>)  $\delta$  = -96.57 (d, *J* = 53.5 Hz).<sup>13</sup>C NMR (151 MHz, CDCI<sub>3</sub>)  $\delta$  = 206.04, 120.17 (t, *J* = 282.4 Hz), 52.55, 38.83, 36.35, 28.12. IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] = 2928, 2850, 1708, 1452, 1273, 1059, 983, 910, 784, 755, 664. HRMS (ESI) m/z: [M+Na]\*Calcd for C<sub>12</sub>H<sub>16</sub>F<sub>2</sub>NaOSe 317.0232; Found 317.0240. Melting Point = 35-36°C.

## Se-(difluoromethyl) 3-(4-trifluoromethylphenyl)propaneselenoate (4f)



According to the general procedure, selenoester **4f** (68 mg, 68%) was obtained from 3-(4-trifluoromethylphenyl)propanoic acid **3f** on a 0.3 mmol scale after flash column chromatography (pentane/DCM 40:1) as a yellow oil.

<sup>1</sup>H NMR (601 MHz, CDCI<sub>3</sub>)  $\delta$  = 7.57 (d, *J* = 8.1 Hz, 2H), 7.45 (t, *J* = 53.4 Hz, 1H), 7.31 (d, *J* = 8.0 Hz, 2H), 3.09 – 3.02 (m, 4H).<sup>19</sup>F NMR (565 MHz, CDCI<sub>3</sub>)  $\delta$  = -62.39(s, 3F), -95.88 (d, *J* = 53.0 Hz, 2F).<sup>13</sup>C NMR (151 MHz, CDCI<sub>3</sub>)  $\delta$  = 196.77, 143.16, 129.29 (q, *J* = 32.6 Hz), 128.83, 125.81 (q, *J* = 3.8 Hz), 124.26 (q, *J* = 271.9 Hz), 119.82 (t, *J* = 284.3 Hz), 49.98, 30.33. IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] = 2932, 1724, 1619, 1332, 1271, 1163, 1121, 1065, 944, 826, 687. HRMS (EI) m/z: [M]<sup>+</sup> Calcd for C<sub>11</sub>H<sub>9</sub>F<sub>5</sub>OSe 331.9739; Found 331.9740.

#### Se-(difluoromethyl) 3-(4-methoxyphenyl)propaneselenoate (4g)



According to the general procedure, selenoester **4g** (77 mg, 88%) was obtained from 3-(4-methoxyphenyl)propanoic acid **3g** on a 0.3 mmol scale after flash column chromatography (pentane/DCM 20:1) as a yellow oil.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.45 (t, *J* = 53.5 Hz, 1H), 7.10 (d, *J* = 8.7 Hz, 2H), 6.85 (d, *J* = 8.7 Hz, 2H), 3.79 (s, 3H), 3.01 - 2.92 (m, 4H). <sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -96.00 (d, *J* = 53.0 Hz).<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 197.28, 158.53, 131.02, 129.40, 119.99 (t, *J* = 283.7 Hz), 114.24, 55.38, 50.88, 29.87. IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] = 2936, 2837, 1721, 1612, 1245, 1178, 1029, 822, 780, 685. HRMS (ESI) m/z: [M+H]<sup>+</sup> Calcd for C<sub>11</sub>H<sub>13</sub>F<sub>2</sub>O<sub>2</sub>Se 295.0049; Found 295.0052.

Se-(difluoromethyl) (E)-3-(4-bromophenyl)prop-2-eneselenoate (4h)



According to the general procedure, selenoester **4h** (67 mg, 57%) was obtained from (*E*)-3-(4-bromophenyl)acrylic acid **3h** on a 0.3 mmol scale after flash column chromatography (pentane/DCM,50:1) as a yellow solid.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.56 (t, *J* = 53.5 Hz, 1H), 7.55 (d, *J* = 8.5 Hz, 2H), 7.48 (d, *J* = 15.6 Hz, 1H), 7.41 (d, *J* = 8.6 Hz, 2H), 6.63 (d, *J* = 15.8 Hz, 1H).<sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -95.63 (d, *J* = 53.8 Hz). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 188.06, 142.44, 132.61, 132.18, 130.20, 126.89 – 126.65 (m), 126.24, 120.08 (t, *J* = 283.9 Hz). IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] = 2923, 1688, 1605, 1583, 1487, 1400, 1266, 1076, 1007, 974, 881, 806, 757, 685. HRMS (EI) m/z: [M]\*Calcd for C<sub>10</sub>H<sub>7</sub>BrF<sub>2</sub>OSe 339.8814; Found 339.8825. Melting Point = 103-105°C.

#### Se-(difluoromethyl) 4-chlorobenzoselenoate (4i)



According to the general procedure, selenoester **4i** (53 mg, 66%) was obtained from 4-chlorobenzoic acid **3i** on a 0.3 mmol scale after flash column chromatography (pentane) as a yellow solid.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.75 (d, J = 8.6 Hz, 2H), 7.61 (t, J = 53.5 Hz, 1H), 7.49 (d, J = 8.5 Hz, 2H). <sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -95.66 (d, J = 54.0 Hz).<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 190.00, 141.61, 136.18, 129.72, 128.97, 120.12 (t, J = 284.3 Hz).

The characterization data agree with the literature values.<sup>[3]</sup>

#### Se-(difluoromethyl) 4-bromobenzoselenoate (4j)



According to the general procedure, selenoester **4j** (66 mg, 70%) was obtained from 4-bromobenzoic acid **3j** on a 0.3 mmol scale after flash column chromatography (pentane) as a yellow solid.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.68 – 7.63 (m, 4H), 7.60 (d, J = 53.5 Hz, 1H). <sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -95.68 (d, J = 53.7 Hz). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 190.27, 136.60, 132.71, 130.34, 128.98, 120.07 (t, J = 284.4 Hz).

The characterization data agree with the literature values.<sup>[3]</sup>

#### Se-(difluoromethyl) 4-iodobenzoselenoate (4k)



According to the general procedure, selenoester **4k** (60 mg, 55%) was obtained from 4-iodobenzoic acid **3k** on a 0.3 mmol scale after flash column chromatography (pentane) as a yellow solid.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.88 (d, J = 8.5 Hz, 2H), 7.60 (t, J = 53.5 Hz, 1H), 7.51 (d, J = 8.6 Hz, 2H).<sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -95.67 (d, J = 53.9 Hz).<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 190.65, 138.70, 137.18, 128.77, 120.03 (t, J = 284.4 Hz), 103.24.

### Se-(difluoromethyl) 4-methoxybenzoselenoate (4I)



According to the general procedure, selenoester **4I** (16 mg, 20%) was obtained from 4-methoxybenzoic acid **3I** on a 0.3 mmol scale after flash column chromatography (pentane/AcOEt 30:1) as a yellow solid.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.78 (d, *J* = 8.9 Hz, 1H), 7.61 (t, *J* = 53.6 Hz, 0H), 6.96 (d, *J* = 9.1 Hz, 1H), 3.89 (s, 2H). <sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -95.84 (d, *J* = 54.0 Hz). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 188.91, 165.08, 130.61, 130.21, 120.55 (t, *J* = 283.1 Hz), 114.54, 55.85.

The characterization data agree with the literature values.<sup>[3]</sup>

#### Se-(difluoromethyl) 4-nitrobenzoselenoate (4m)



According to the general procedure, selenoester **4m** (47 mg, 56%) was obtained from 4-nitrobenzoic acid **3m** on a 0.3 mmol scale after flash column chromatography (pentane/AcOEt, 30:1) as a yellow solid.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 8.35 (d, *J* = 8.9 Hz, 2H), 8.00 (d, *J* = 8.6 Hz, 2H), 7.63 (t, *J* = 53.3 Hz, 1H).<sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -95.37 (d, *J* = 52.9 Hz). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 190.38, 151.33, 142.13, 128.61, 124.60, 119.74 (t, *J* = 285.8 Hz). IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] = 2926, 2855, 1693, 1606, 1529, 1347, 1266, 1195, 1068, 891, 840, 733, 686. HRMS (ESI) m/z: [M]<sup>+</sup> Calcd for C<sub>8</sub>H<sub>5</sub>F<sub>2</sub>NNaO<sub>3</sub>Se 303.9300; Found 303.9311.

#### Se-(difluoromethyl) [1,1'-biphenyl]-4-carboselenoate (4n)



According to the general procedure, selenoester **4n** (34 mg, 45%) was obtained from [1,1'-biphenyl]-4-carboxylic acid **3n** on a 0.3 mmol scale after flash column chromatography (pentane/DCM 30:1) as a white solid.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.89 (d, J = 8.6 Hz, 2H), 7.72 (d, J = 8.5 Hz, 2H), 7.65 (t, J = 53.7 Hz, 1H), 7.63 (d, J = 8.6 Hz, 2H), 7.49 (t, J = 7.8 Hz, 2H), 7.44 (t, J = 7.3 Hz, 1H). <sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -95.83 (d, J = 52.7 Hz).<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 190.55, 147.84, 139.40, 136.46, 129.25, 128.87, 128.33, 127.92, 127.45, 120.34 (t, J = 283.6 Hz).

## Se-(difluoromethyl) 4-methylbenzoselenoate (40)



According to the general procedure, selenoester **4o** (34 mg, 45%) was obtained from 4-methyl benzoic acid **3o** on a 0.3 mmol scale after flash column chromatography (pentane) as a yellow solid.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.71 (d, *J* = 8.4 Hz, 2H), 7.61 (t, *J* = 53.4 Hz, 1H), 7.30 (d, *J* = 8.0 Hz, 2H), 2.43 (s, 3H). <sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -95.97 (d, *J* = 53.9 Hz).<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 190.48, 146.34, 135.38, 130.01, 127.87, 120.41 (t, *J* = 283.3 Hz), 21.97.

The characterization data agree with the literature values.<sup>[3]</sup>

#### Se-(difluoromethyl) 3-methylbenzoselenoate (4p)



According to the general procedure, selenoester **4p** (40 mg, 54%) was obtained from 3-methylbenzoic acid **3p** on a 0.3 mmol scale after flash column chromatography (pentane) as a yellow solid.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.61 (t, *J* = 53.5 Hz, 1H), 7.61 (d, *J* = 7.6 Hz, 2H), 7.47 (d, *J* = 7.6 Hz, 1H), 7.39 (t, *J* = 7.8 Hz, 1H), 2.43 (s, 3H).<sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -96.03 (d, *J* = 52.7 Hz).<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 191.25, 139.45, 137.90, 135.81, 129.21, 128.07, 125.04, 120.35 (t, *J* = 283.4 Hz), 21.38.

The characterization data agree with the literature values.<sup>[3]</sup>

#### Se-(difluoromethyl) 2-methylbenzoselenoate (4q)



According to the general procedure, selenoester **4q** (37 mg, 49%) was obtained from 2-methyl benzoic acid **3q** on a 0.3 mmol scale after flash column chromatography (pentane) as a yellow oil.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.70 (d, J = 7.9 Hz, 1H), 7.55 (t, J = 53.6 Hz, 1H), 7.48 (t, J = 7.6, 7.1 Hz, 1H), 7.33 (t, J = 7.7 Hz, 1H), 7.29 (d, J = 7.7 Hz, 1H), 2.52 (s, 3H).<sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -96.59 (d, J = 53.5 Hz). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 192.32, 137.64, 137.39, 133.39, 132.34, 129.49, 126.58, 120.82 (t, J = 283.2 Hz), 21.10.

## Se-(difluoromethyl) naphthalene-2-carboselenoate (4r)



According to the general procedure, selenoester **4r** (45 mg, 53%) was obtained from 2-naphthoic acid **3r** on a 0.3 mmol scale after flash column chromatography (pentane) as a colourless oil.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 8.34 (s, 1H), 7.98 (d, *J* = 8.1 Hz, 1H), 7.94 – 7.87 (m, 2H), 7.82 (dd, *J* = 8.6, 1.9 Hz, 1H), 7.69 (t, *J* = 53.5 Hz, 1H), 7.68-7.65 (m, 1H), 7.63 – 7.57 (m, 1H).<sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -95.78 (d, *J* = 54.0 Hz).<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 190.91, 136.47, 135.07, 132.49, 130.02, 129.86, 129.54, 129.32, 128.07, 127.60, 122.64, 120.38 (t, *J* = 283.7 Hz).

The characterization data agree with the literature values.<sup>[3]</sup>

#### Se-(difluoromethyl) 6-chloropyridine-3-carboselenoate (4s)



According to the general procedure, selenoester **4s** (48 mg, 59%) was obtained from 6-chloronicotinic acid **3s** on a 0.3 mmol scale after flash column chromatography (pentane:ethyl acetate, 70:1) as a yellow solid.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 8.80 (d, *J* = 2.6 Hz, 1H), 8.01 (dd, *J* = 8.4, 2.6 Hz, 1H), 7.62 (t, *J* = 53.3 Hz, 1H), 7.49 (d, *J* = 8.4 Hz, 1H).<sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -95.23 (d, *J* = 53.7 Hz).<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  =  $\delta$  188.92, 157.47, 148.94, 137.15, 132.48, 125.16, 119.56 (t, *J* = 285.9 Hz). IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] = 2923, 1675, 1572, 1462, 1365, 1206, 1064, 1018, 880, 842, 735, 683, 633. HRMS (ESI) m/z: [M+Na]+Calcd for C<sub>7</sub>H<sub>4</sub>ClF<sub>2</sub>NNaOSe 293.9013; Found 293.9024. Melting Point = 87-88°C.

Se-(difluoromethyl) 2-(6-methoxynaphthalen-2-yl)propaneselenoate (4t)



According to the general procedure, selenoester **4t** (86 mg, 90%) was obtained from Ibuprofen **3t** on a 0.3 mmol scale after flash column chromatography (pentane) as a colourless oil.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.32 (t, *J* = 53.7 Hz, 1H), 7.21 – 7.15 (m, 4H), 3.84 (q, *J* = 7.1 Hz, 1H), 2.50 (d, *J* = 7.2 Hz, 2H), 1.88 (hept, *J* = 6.7 Hz, 1H), 1.56 (d, *J* = 7.2 Hz, 3H), 0.92 (d, *J* = 6.6 Hz, 6H).<sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -96.68 (dd, *J* = 252.3, 53.2 Hz).<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 201.39, 142.53, 134.37, 129.91, 128.76, 120.29 (t, *J* = 282.9 Hz), 58.31, 45.21, 30.29, 22.48, 17.30. IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] = 2956, 2869, 1716, 1465, 1367, 1272, 1060, 915, 846, 686, 548. HRMS (EI) m/z: [M]<sup>+</sup>Calcd for C<sub>14</sub>H<sub>18</sub>F<sub>2</sub>OSe 320.0491; Found 320.0483.

#### Se-(difluoromethyl) 2-(3-benzoylphenyl)propaneselenoate (4u)



According to the general procedure, selenoester **4u** (74 mg, 67%) was obtained from Ketoprofen **3u** on a 0.3 mmol scale after flash column chromatography (pentane/AcOEt 35:1) as a colourless oil.

<sup>1</sup>H NMR (601 MHz, CDCI<sub>3</sub>)  $\delta$  = 7.81 (d, *J* = 8.0 Hz, 2H), 7.78 (d, *J* = 6.7 Hz, 1H), 7.73 (s, 1H), 7.62 (t, *J* = 7.4 Hz, 1H), 7.54 – 7.48 (m, 4H), 7.36 (t, *J* = 53.5 Hz, 1H), 3.96 (q, *J* = 7.1 Hz, 1H), 1.60 (d, *J* = 7.1 Hz, 3H).<sup>19</sup>F NMR (565 MHz, CDCI<sub>3</sub>)  $\delta$  = -96.20 (dd, *J* = 266.4, 55.2 Hz).<sup>13</sup>C NMR (151 MHz, CDCI<sub>3</sub>)  $\delta$  = 200.27, 196.18, 138.50, 137.74, 137.33, 132.88, 132.57, 130.35, 130.34, 130.22, 129.21, 128.55, 120.01 (t, *J* = 284.0 Hz), 58.53, 17.51. IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] = 2982, 1715, 1657, 1597, 1448, 1271, 1055, 910, 820, 786, 687. HRMS (ESI) m/z: [M+Na]<sup>+</sup>Calcd for C<sub>17</sub>H<sub>14</sub>F<sub>2</sub>NaO<sub>2</sub>Se 391.0025; Found 391.0025.

#### Se-(difluoromethyl) 2-(10-oxo-10,11-dihydrodibenzo[b,f]thiepin-2-yl)propaneselenoate (4v)



According to the general procedure, selenoester 4v (99 mg, 80%) was obtained from Zaltoprofen 3v on a 0.3 mmol scale after flash column chromatography (pentane/ethyl acetate, 60:1) as a yellow oil.

<sup>1</sup>H NMR (601 MHz, CDCI<sub>3</sub>)  $\delta$  = 8.20 (d, *J* = 7.9 Hz, 1H), 7.65 (d, *J* = 7.9 Hz, 1H), 7.60 (d, *J* = 8.0 Hz, 1H), 7.43 (t, *J* = 7.5 Hz, 1H), 7.37 (s, 1H), 7.34 (t, *J* = 53.5 Hz, 1H), 7.32 (t, *J* = 7.9 Hz, 1H), 7.13 (d, *J* = 7.9, 1H), 4.38 (s, 2H), 3.88 (q, *J* = 7.1 Hz, 1H), 1.54 (d, *J* = 7.1 Hz, 3H). <sup>19</sup>F NMR (565 MHz, CDCI<sub>3</sub>)  $\delta$  = -95.57 (dd, *J* = 251.5, 53.1 Hz), -96.78 (dd, *J* = 251.2, 52.5 Hz).<sup>13</sup>C NMR (151 MHz, CDCI<sub>3</sub>)  $\delta$  = 199.96, 191.05, 139.88, 139.42, 138.59, 136.16, 135.05, 132.77, 131.98, 131.68, 130.98, 129.60, 127.44, 127.10, 119.93 (t, *J* = 284.0 Hz), 58.29, 51.12, 17.52. IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] =2982, 1715, 1670, 1587, 1429, 1283, 1056, 930, 897, 729, 681. HRMS (ESI) m/z: [M+Na]<sup>+</sup> Calcd for C<sub>18</sub>H<sub>14</sub>F<sub>2</sub>NaO<sub>2</sub>SSe 434.9745; Found 434.9761.

#### Se-(difluoromethyl) 2-(6-methoxynaphthalen-2-yl)propaneselenoate (4w)



According to the general procedure, selenoester **4w** (94 mg, 91%) was obtained from Naproxen **3w** on a 0.3 mmol scale after flash column chromatography (pentane/DCM 15:1) as an off-white solid.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.76 (t, J = 9.1 Hz, 2H), 7.70 (d, J = 1.9 Hz, 1H), 7.36 (t, J = 53.5 Hz, 1H), 7.33 (dd, J = 8.4, 1.9 Hz, 1H), 7.21 (dd, J = 8.9, 2.5 Hz, 1H), 7.16 (d, J = 2.7 Hz, 1H), 4.00 (q, J = 7.1 Hz, 1H), 3.94 (s, 3H), 1.65 (d, J = 7.1 Hz, 3H).<sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -96.29 (dd, J = 252.8, 53.2 Hz).<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 201.25, 158.39, 134.61, 132.19, 129.58, 128.93, 128.26, 127.90, 126.69, 120.25 (t, J = 283.2, 282.1 Hz), 119.67, 105.82, 58.63,

55.47, 17.36. **IR (ATR)**:  $\tilde{v}$  [cm<sup>-1</sup>] =2984, 2938, 1714, 1601, 1454, 1270, 1231, 1176, 1161, 1062, 1037, 933, 907, 855, 819, 683. **HRMS (ESI) m/z**: [M+Na]⁺Calcd for C<sub>15</sub>H<sub>14</sub>F<sub>2</sub>NaO<sub>2</sub>Se 367.0025; Found 367.0020. Melting Point = 96-98°C.

Se-(difluoromethyl) 4-(*N*,*N*-dipropylsulfamoyl)benzoselenoate (4x)



According to the general procedure, selenoester **4x** (78 mg, 65%) was obtained from Probenecid **3x** on a 0.3 mmol scale after flash column chromatography (pentane/AcOEt 30:1) as a yellow solid.

<sup>1</sup>**H NMR (601 MHz, CDCI<sub>3</sub>)**  $\delta$  = 7.95 – 7.89 (m, 4H), 7.61 (t, *J* = 53.4 Hz, 1H), 3.11 (t, *J* = 7.7 Hz, 4H), 1.55 (h, *J* = 7.7 Hz, 4H), 0.86 (t, *J* = 7.4 Hz, 6H). <sup>19</sup>**F NMR (565 MHz, CDCI<sub>3</sub>)**  $\delta$  = -95.59 (d, *J* = 53.6 Hz). <sup>13</sup>**C NMR (151 MHz, CDCI<sub>3</sub>)**  $\delta$  = 190.57, 146.10, 140.31, 128.18, 127.90, 119.87 (t, *J* = 285.0 Hz), 50.07, 22.06, 11.24. **IR (ATR)**:  $\tilde{v}$  [cm<sup>-1</sup>] =2970, 2937, 2877, 1693, 1461, 1397, 1338, 1266, 1181, 1061, 977, 880, 799, 730, 684, 564. **HRMS (ESI) m/z:** [M+H]<sup>+</sup> Calcd for C<sub>14</sub>H<sub>20</sub>F<sub>2</sub>NO<sub>3</sub>SSe 400.0297; Found 400.0301. Melting Point = 54-56°C

Se-(difluoromethyl) (9Z,12Z)-octadeca-9,12-dieneselenoate (4y)



According to the general procedure, selenoester **4y** (109 mg, 92%) was obtained from linoleic acid on a 0.3 mmol scale after flash column chromatography (pentane) as a yellow oil.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.44 (t, *J* = 53.5 Hz, 1H), 5.42 – 5.30 (m, 4H), 2.77 (t, *J* = 6.9 Hz, 2H), 2.68 (t, *J* = 7.5 Hz, 2H), 2.05 (q, *J* = 7.0 Hz, 4H), 1.68 (p, *J* = 7.4 Hz, 2H), 1.40 – 1.25 (m, 14H), 0.89 (t, *J* = 7.0 Hz, 3H).<sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -96.13 (d, *J* = 53.5 Hz).<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 198.13, 130.39, 130.05, 128.31, 128.01, 120.13 (t, *J* = 283.4 Hz), 49.16, 31.67, 29.65, 29.49, 29.18, 29.08, 28.78, 27.35, 27.27, 25.77, 24.93, 22.72, 14.21. IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] = 2929, 2857, 2288, 2205, 1646, 1516, 1457, 1380, 1284, 1097, 972, 873, 799, 773, 677, 575. HRMS (ESI) m/z: [M+Na]\*Calcd for C<sub>19</sub>H<sub>32</sub>F<sub>2</sub>NaOSe 417.1484; Found 417.1490.

## 4.3 General Procedure for the Deoxydifluoromethylselenylation of Alcohols

## 4.3 General Procedure for the Deoxydifluoromethylselenylation of Alcohols

**Method A:** BT-SeCF<sub>2</sub>H (1.25 equiv.) and the alcohol (1.0 equiv., 0.5 mmol) were added to dry MeCN (0.5M) at -40 °C in a Schlenk tube under argon. DIPEA (2 equiv.) was added dropwise and the reaction mixture was stirred for 2 h at -40 °C, after which an additional 2 equiv. of DIPEA and 0.25 equiv. of BT-SeCF<sub>2</sub>H were added. The reaction was stirred for a further 2 hours, after which the crude mixture was filtered through a silica pad and the solvent was removed *in vacuo*. The crude difluoromethylselenoethers were isolated using column chromatography (SiO<sub>2</sub>).

**Method B:** BT-SeCF<sub>2</sub>H (1.25 equiv.), AgOTf (0.5 equiv) and the alcohol (1.0 equiv., 0.5 mmol) were added to dry MeCN (0.5M) at -40 °C in a Schlenk tube under argon. DIPEA (2 equiv.) was added dropwise and the reaction mixture was stirred for 2 h at -40 °C, after which an additional 2 equiv. of DIPEA and 0.25 equiv. of BT-SeCF<sub>2</sub>H were added. The reaction was stirred for a further 2 hours, after which the crude mixture was filtered through a silica pad and the solvent was removed *in vacuo*. The crude difluoromethylselenoethers were isolated using column chromatography (SiO<sub>2</sub>).

## 4.4 Characterization Data for Deoxydifluoromethylselenoether Products 6

## (Difluoromethyl)(4-nitrobenzyl)selane (6a)



According to Method A, selenoether **6a** (82 mg, 62%) was obtained from (4-nitrophenyl)methanol **5a** on a 0.5 mmol scale after flash column chromatography (pentane/ethyl acetate, 50:1) as a yellow oil.

<sup>1</sup>H NMR (601 MHz, CDCI<sub>3</sub>)  $\boldsymbol{\delta}$  = 8.17 (d, *J* = 8.1,2H), 7.50 (d, *J* = 8.5 Hz, 2H), 7.14 (t, *J* = 54.6 Hz, 1H), 4.16 (s, 2H). <sup>19</sup>F NMR (565 MHz, CDCI<sub>3</sub>)  $\boldsymbol{\delta}$  = -92.03 (d, *J* = 54.2 Hz).<sup>13</sup>C NMR (151 MHz, CDCI<sub>3</sub>)  $\boldsymbol{\delta}$  = 147.20, 145.73, 129.97, 124.15, 115.18 (t, *J* = 288.4 Hz), 24.99 (t, *J* = 3.3 Hz). IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] = 1598, 1519, 1192, 1042, 908, 860, 799, 698, 599. HRMS (EI) m/z: [M]<sup>+</sup> Calcd for C<sub>8</sub>H<sub>7</sub>F<sub>2</sub>NO<sub>2</sub>Se 266.9610; Found 266.9618.

## 4-(((Difluoromethyl)selanyl)methyl)benzonitrile (6b)



According to Method A, selenoether **6b** (68 mg, 52%) was obtained from 4-(hydroxymethyl)benzonitrile **5b** on a 0.5 mmol scale after flash column chromatography (pentane/ethyl acetate, 30:1) as a yellow oil.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.61 (d, *J* = 8.0, 2H), 7.44 (d, *J* = 8.2 Hz, 2H), 7.12 (t, *J* = 54.7 Hz, 1H), 4.11 (s, 2H). <sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -92.12 (d, *J* = 54.6 Hz).<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 143.55, 132.68, 129.84, 118.68, 115.20 (t, *J* = 288.3 Hz), 111.33, 25.39 (t, *J* = 3.1 Hz). IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] = 2228, 1606, 1508, 1413, 1279, 1029, 847, 689, 605, 544. HRMS (EI) m/z: [M]<sup>+</sup> Calcd for C<sub>9</sub>H<sub>7</sub>F<sub>2</sub>NSe 246.9712; Found 246.9726.

## Methyl 4-(((difluoromethyl)selanyl)methyl)benzoate (6c)



According to Method A, selenoether **6c** (78 mg, 56%) was obtained from methyl 4-(hydroxymethyl)benzoate **5c** on a 0.5 mmol scale after flash column chromatography (pentane/ethyl acetate, 30:1) as a yellow oil.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.99 (d, *J* = 8.4 Hz, 2H), 7.40 (d, *J* = 8.4 Hz, 2H), 7.09 (t, *J* = 55.0 Hz, 1H), 4.12 (s, 2H), 3.91 (s, 3H). <sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -92.52 (d, *J* = 55.5 Hz).<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 166.75, 142.94, 130.19, 129.32, 129.08, 115.39 (t, *J* = 287.8 Hz), 52.24, 25.73 (t, *J* = 2.9 Hz). IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] = 2953, 1713, 1610, 1455, 1275, 1179, 1032, 863, 769, 690. HRMS (EI) m/z: [M]<sup>+</sup> Calcd for C<sub>10</sub>H<sub>10</sub>F<sub>2</sub>O<sub>2</sub>Se 279.9814; Found 279.9827.

## (Difluoromethyl)(4-(trifluoromethyl)benzyl)selane (6d)



According to Method A, selenoether **6d** (71 mg, 49%) was obtained from methyl (4-(trifluoromethyl)phenyl)methanol **5d** on a 0.5 mmol scale after flash column chromatography (pentane/CH<sub>2</sub>Cl<sub>2</sub>, 30:1) as a yellow oil.

<sup>1</sup>H NMR (601 MHz, CDCI<sub>3</sub>)  $\delta$  = 7.58 (d, J = 8.1 Hz, 2H), 7.46 (d, J = 8.0 Hz, 2H), 7.11 (t, J = 54.9 Hz, 1H), 4.13 (s, 2H).<sup>19</sup>F NMR (565 MHz, CDCI<sub>3</sub>)  $\delta$  = -62.50 (s, 3F), -92.46 (d, J = 55.9 Hz, 2F). <sup>13</sup>C NMR (151 MHz, CDCI<sub>3</sub>)  $\delta$  = 141.94, 129.78 (q, J = 32.3 Hz), 129.45, 125.91 (q, J = 3.8 Hz), 124.14 (q, J = 271.6 Hz), 115.35 (t, J = 287.9 Hz), 25.42(t, J = 2.7 Hz). IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] = 1617, 1418, 1322, 1165, 1120, 1063, 848, 678, 613. HRMS (EI) m/z: [M]<sup>+</sup> Calcd for C<sub>9</sub>H<sub>7</sub>F<sub>5</sub>Se 289.9633; Found 289.9648.

## (Difluoromethyl)(2,4,6-trichlorobenzyl)selane (6e)



According to Method A, selenoether **6e** (108 mg, 67%) was obtained from (2,4,6-trichlorophenyl)methanol **5e** on a 0.5 mmol scale after flash column chromatography (pentane) as a yellow oil.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.35 (t, *J* = 55.1 Hz, 1H), 7.35 (s, 2H), 4.30 (s, 2H).<sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -90.86 (d, *J* = 54.8 Hz).<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 135.67, 134.00, 133.66, 128.61, 115.41 (t, *J* = 287.9 Hz), 20.78 (t, *J* = 2.9 Hz). IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] =1579, 1547, 1437, 1374, 1275, 1038, 907, 855, 780, 735, 688, 608. HRMS (EI) m/z: [M]<sup>+</sup> Calcd for C<sub>8</sub>H<sub>5</sub>Cl<sub>3</sub>F<sub>2</sub>Se 323.8590; Found 323.8580.

## (Difluoromethyl)(4-bromobenzyl)selane (6f)



According to Method B, selenoether **6f** (101 mg, 67%) was obtained from (4-bromophenyl)methanol **5f** on a 0.5 mmol scale after flash column chromatography (pentane) as a yellow oil.

<sup>1</sup>H NMR (601 MHz, CDCI<sub>3</sub>)  $\boldsymbol{\delta}$  = 7.44 (d, *J* = 8.4 Hz, 2H), 7.21 (d, *J* = 8.4 Hz, 2H), 7.08 (t, *J* = 55.0 Hz, 1H), 4.05 (s, 2H).<sup>19</sup>F NMR (565 MHz, CDCI<sub>3</sub>)  $\boldsymbol{\delta}$  = -92.58 (d, *J* = 55.4 Hz).<sup>13</sup>C NMR (151 MHz, CDCI<sub>3</sub>)  $\boldsymbol{\delta}$  = 136.67, 132.06, 130.77, 121.42, 115.51 (t, *J* = 287.5 Hz), 25.52 (t, *J* = 3.0 Hz). IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] =1486, 1401, 1275, 1190, 1030, 1005, 825, 676, 596. HRMS (EI) m/z: [M]<sup>+</sup> Calcd C<sub>8</sub>H<sub>7</sub>F<sub>2</sub>BrSe for 299.8864; Found 299.8857.

#### (Difluoromethyl)(4-iodobenzyl)selane (6g)



According to Method B, selenoether **6g** (96 mg, 55%) was obtained from (4-iodophenyl)methanol **5g** on a 0.5 mmol scale after flash column chromatography (pentane) as a yellow oil.

<sup>1</sup>H NMR (601 MHz, CDCI<sub>3</sub>)  $\delta$  = 7.64 (d, *J* = 8.4 Hz, 2H), 7.09 (d, *J* = 9.0 Hz, 2H), 7.08 (d, *J* = 55.0 Hz, 1H), 4.03 (s, 2H).<sup>19</sup>F NMR (565 MHz, CDCI<sub>3</sub>)  $\delta$  = -92.57 (d, *J* = 55.0 Hz).<sup>13</sup>C NMR (151 MHz, CDCI<sub>3</sub>)  $\delta$  = 138.04, 137.34, 131.01, 115.50 (t, *J* = 287.5 Hz), 92.87, 25.63 (t, *J* = 2.8 Hz). IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] =1483, 1397, 1276, 1189, 1030, 1005, 796, 689, 590. HRMS (EI) m/z: [M]<sup>+</sup> Calcd C<sub>8</sub>H<sub>7</sub>F<sub>2</sub>ISe for 347.8726; Found 347.8709.

## ([1,1'-Biphenyl]-4-ylmethyl)(difluoromethyl)selane (6h)



According to the general procedure, selenoether **6h** (86 mg, 58%) was obtained from [1,1'-biphenyl]-4-ylmethanol **5h** on a 0.5 mmol scale after flash column chromatography (pentane/CH2Cl2, 50:1) as a yellow solid.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.59 (d, *J* = 7.4 Hz, 2H), 7.56 (d, *J* = 8.3 Hz, 2H), 7.46 (t, *J* = 7.6 Hz, 2H), 7.42 (d, *J* = 8.2 Hz, 2H), 7.36 (t, *J* = 7.2, 1H), 7.12 (t, *J* = 55.2 Hz, 1H), 4.16 (s, 2H).<sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -92.71 (d, *J* = 55.6 Hz). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 140.67, 140.50, 136.49, 129.53, 128.95, 127.67, 127.57, 127.18, 115.77 (t, *J* = 287.1 Hz), 26.10 (t, *J* = 2.8 Hz). IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] = 3031, 2970, 1486, 1406, 1274, 1036, 833, 762, 689, 603. HRMS (EI) m/z: [M]\* Calcd C<sub>14</sub>H<sub>12</sub>F<sub>2</sub>Se for 298.0072; Found 298.0072. Melting Point = 40-42 °C.

## (4-(((Difluoromethyl)selanyl)methyl)phenyl)(methyl)sulfane (6i)



According to Method B, selenoether **6i** (92 mg, 69%) was obtained from (4-(methylthio)phenyl)methanol **5i** on a 0.5 mmol scale after flash column chromatography (pentane/CH<sub>2</sub>Cl<sub>2</sub>, 50:1) as a yellow oil.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.25 (d, *J* = 8.3 Hz, 2H), 7.20 (d, *J* = 8.3, 2H), 7.07 (t, *J* = 55.5, 1H), 4.07 (s, 2H), 2.48 (s, 3H).<sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -92.74 (d, *J* = 55.7 Hz).<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 137.88, 134.15, 129.55, 126.97, 115.74 (t, *J* = 287.2 Hz), 26.01 (t, *J* = 3.1 Hz), 15.90. IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] = 2921, 1558, 1492, 1437, 1271, 1191, 1029, 968, 811, 681, 599. HRMS (EI) m/z: [M]<sup>+</sup> Calcd for C<sub>9</sub>H<sub>10</sub>F<sub>2</sub>SSe 267.9636; Found 267.9623.

## (4-(tert-Butyl)benzyl)(difluoromethyl)selane (6j)



According to Method B, selenoether **6j** (132 mg, 95%) was obtained from (4-(tert-butyl)phenyl)methanol **5j** on a 0.5 mmol scale after flash column chromatography (pentane) as a yellow oil.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.36 (d, *J* = 8.6 Hz, 2H), 7.28 (d, *J* = 8.4 Hz, 2H), 7.09 (t, *J* = 55.2 Hz, 1H), 4.10 (s, 2H), 1.33 (s, 9H).<sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -92.90 (d, *J* = 55.8 Hz).<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 150.54, 134.19, 128.75, 125.85, 115.84 (t, *J* = 286.8 Hz), 34.64, 31.39, 26.05 (t, *J* = 2.9 Hz). IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] =2963, 1514, 1364, 1294, 1269, 1109, 1035, 909, 833, 734, 691, 609. HRMS (EI) m/z: [M]<sup>+</sup> Calcd for C<sub>12</sub>H<sub>16</sub>F<sub>2</sub>Se 278.0385; Found 278.0371.

(Difluoromethyl)(4-ethynylbenzyl)selane (6k)



According to Method B, selenoether **6k** (84 mg, 69%) was obtained from (4-ethynylphenyl)methanol **5k** on a 0.5 mmol scale after flash column chromatography (pentane) as a yellow oil.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  =7.45 (d, *J* = 8.2 Hz, 2H), 7.29 (d, *J* = 8.2 Hz, 2H), 7.08 (t, *J* = 55.0 Hz, 1H), 4.08 (s, 2H), 3.09 (s, 1H).<sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -92.61 (d, *J* = 55.9 Hz).<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 138.44, 132.66, 129.09, 121.32, 115.54 (t, *J* = 287.4 Hz), 83.34, 77.78, 25.98 (t, *J* = 2.4 Hz). IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] =3294, 2107, 1506, 1293, 1276, 1191, 1032, 908, 843, 734, 657, 603. HRMS (EI) m/z: [M]<sup>+</sup> Calcd for C<sub>10</sub>H<sub>8</sub>F<sub>2</sub>Se 245.9759 ; Found 245.9748.

## (Difluoromethyl)(3-phenylprop-2-yn-1-yl)selane (6l)



According to Method B, selenoether **6I** (52 mg, 42%) was obtained from 3-phenylprop-2-yn-1-ol **5I** on a 0.5 mmol scale after flash column chromatography (pentane) as a yellow oil.

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.44 – 7.41 (m, 2H), 7.37 (t, *J* = 55.2 Hz, 1H), 7.33 – 7.30 (m, 3H), 3.76 (s, 2H).<sup>19</sup>F NMR (565 MHz, CDCl<sub>3</sub>)  $\delta$  = -93.14 (d, *J* = 55.4 Hz).<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  = 131.86, 128.64, 128.47, 122.72, 116.04 (t, *J* = 287.5 Hz), 84.48, 84.47, 8.70 (t, *J* = 3.5 Hz). IR (ATR):  $\tilde{v}$  [cm<sup>-1</sup>] = 1597, 1490, 1442, 1272, 1190, 1037, 754, 689, 606. HRMS (EI) m/z: [M]<sup>+</sup> Calcd for C<sub>10</sub>H<sub>8</sub>F<sub>2</sub>Se 245.9759; Found 245.9765.

## Benzyl(difluoromethyl)selane (A)



According to Method B, selenoether A (256 mg, 58%) was obtained from benzylic alcohol on a 2 mmol scale after flash column chromatography (pentane) as a colourless oil.

<sup>1</sup>H NMR (401 MHz, CDCl<sub>3</sub>)  $\delta$  7.42 – 7.36 (m, 4H), 7.32 (m, 1H), 7.11 (t, *J* = 55.1 Hz, 1H), 4.15 (s, 2H). <sup>19</sup>F NMR (377 MHz, CDCl<sub>3</sub>)  $\delta$  = -92.68 (d, *J* = 55.4 Hz).<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  = 137.42, 129.02, 128.86, 127.45, 115.77 (t, *J* = 286.7 Hz), 26.32 (t, *J* = 2.9 Hz).

The characterization data agree with the literature values.<sup>[4]</sup>

## 5 Literature

[1] K. Shibata, O. Mitsunobu, Bull. Chem. Soc. Jpn. 1992, 65, 3163-3173.

[2] V. V. Levin, A. D. Dilman, P. A. Belyakov, M. I. Struchkova, V. A. Tartakovsky, *J. Fluorine Chem.* **2009**, *130*, 667-670.

- [3] R.-L. Guo, X.-Q. Zhu, X.-L. Zhang, Y.-Q. Wang, Chem. Commun. 2020, 56, 8976-8979.
- [4] T. Dong, J. Nie, C.-P. Zhang, *Tetrahedron* **2018**, *74*, 5642-5649.

# 6 NMR Spectra



0 -20 -40 -1 -30 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 f1 (ppm)





lo 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1 f1 (ppm)

# Se-(difluoromethyl) dodecaneselenoate 4a



-70 0 -20 -60 -30 -40 -50 -80 -90 -100 -110 -120 -130 -140 -150 -160 -1 f1 (ppm)





l0 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1 f1 (ppm)

# Se-(difluoromethyl) octadecaneselenoate ${\bf 4c}$



-90 f1 (ppm) 0 -20 -30 -60 -70 -80 -120 -40 -50 -100 -110 -130 -140 -150 -160 -1





l0 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 f1 (ppm)



f1 (ppm)





f1 (ppm) 180 170 160 150 140 130 120 ò -1



# Se-(difluoromethyl) 3-(4-methoxyphenyl)propaneselenoate 4g





lo 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 f1 (ppm)

# Se-(difluoromethyl) 4-chlorobenzoselenoate 4i



.0	-20	-30	-40	-50	-60	-70	-80	-90	-100	-110	-120	-130	-140	-150	-160	-1
f1 (ppm)																




200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1 f1 (ppm)



0 -20 -30 -40 -50 -60 -70 -80 -90 f1 (ppm) -100 -120 -130 -140 -150 -160 -1 -110





lo 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1 f1 (ppm)

# Se-(difluoromethyl) 4-nitrobenzoselenoate 4m

<sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>)







lo 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1 f1 (ppm)





-90 f1 (ppm) 0 -20 -30 -40 -50 -60 -70 -80 -100 -110 -120 -130 -140 -150 -160 -1





200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1 f1 (ppm)

# Se-(difluoromethyl) 2-methylbenzoselenoate 4q



-90 f1 (ppm) 0 -20 -30 -40 -50 -60 -70 -80 -100 -110 -120 -130 -140 -150 -160 -1





### Se-(difluoromethyl) naphthalene-2-carboselenoate 4r





lo 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1 f1 (ppm)

# Se-(difluoromethyl) 6-chloropyridine-3-carboselenoate 4s

8.80 8.80 8.02 8.00 8.00 7.71 7.54 7.54 7.54 7.54 7.54

<sup>1</sup>H NMR (601 MHz, CDCI<sub>3</sub>)



-90 f1 (ppm) 0 -20 -30 -40 -50 -60 -70 -80 -100 -110 -140 -150 -160 -1 -120 -130





LO 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1 f1 (ppm)



### 0 -20 -30 -50 -60 -70 -80 -140 -160 -1 -40 -100 -110 -120 -130 -150





f1 (ppm) 180 170 160 150 ò



### Se-(difluoromethyl) 2-(6-methoxynaphthalen-2-yl)propaneselenoate 4w





lo 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1 f1 (ppm)



10 -20 -30 -40 -50 -100 -140 -1 -60 -70 -80 -90 -110 -120 -130 -150 -160 f1 (ppm)





lo 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 f1 (ppm)

# 4-(((Difluoromethyl)selanyl)methyl)benzonitrile 6b



-90 f1 (ppm) 0 -20 -30 -40 -50 -60 -70 -80 -100 -110 -120 -130 -140 -150 -160 -1





(Difluoromethyl)(4-(trifluoromethyl)benzyl)selane 6d







f1 (ppm) ò 0 200 190 180 170 160 150 140 130 120 -1

# (Difluoromethyl)(4-bromobenzyl)selane 6f



.0	-20	-30	-40	-50	-60	-70	-80	-90	-100	-110	-120	-130	-140	-150	-160	-1
								f1 (ppm)								





lo 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1 f1 (ppm)





-90 f1 (ppm) 0 -20 -30 -40 -50 -60 -70 -80 -110 -120 -130 -140 -150 -160 -1 -100




lo 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1 f1 (ppm)

## (4-(tert-Butyl)benzyl)(difluoromethyl)selane 6j



0	-20	-30	-40	-50	-60	-70	-80	-90	-100	-110	-120	-130	-140	-150	-160	-1
								f1 (ppm)								





lo 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1 f1 (ppm)

## (Difluoromethyl)(3-phenylprop-2-yn-1-yl)selane 6l



0 -20 -30 -40 -50 -60 -70 -90 f1 (ppm) -140 -150 -160 -1 -80 -100 -110 -120 -130





## Benzyl(difluoromethyl)selane A





lo 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1 f1 (ppm)