Stereoselective Synthesis of Fluoroalkylated Butanolides

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Supporting Information
**General Procedures:**

All syntheses involving air- and moisture-sensitive compounds were carried out using standard Schlenk-type glassware (or in a glove box) under an atmosphere of argon. Solvents were dried with the solvent purification system MB-SPS 800 from *M. Braun* or predistilled according to standard laboratory methods. Unless stated otherwise all chemicals were purchased from *Acros* or *Aldrich* and used without further purification.

The following instruments were used for characterization of the compounds: $^1$H, $^{13}$C, and $^{19}$F NMR spectra were recorded at room temperature using a Bruker AC 250, JEOL ECX 400, JEOL Eclipse 500, or Bruker Advance 3 (700 MHz). $^1$H NMR and $^{13}$C NMR: chemical shift $\delta$ are referenced against the residual solvent signal (CDCl$_3$, $\delta$ $^1$H = 7.26 ppm, $^{13}$C = 77.0 ppm, (CD$_3$)$_2$CO, $\delta$ $^1$H = 2.05 ppm, $^{13}$C = 29.84 ppm; MeOD, $\delta$ $^1$H = 3.31 ppm, $^{13}$C = 49.0 ppm). $^{19}$F NMR: chemical shift $\delta$ is given relative to CFCl$_3$ (external reference). The order of citation in parentheses is a) multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), b) coupling constants, c) number of protons and d) assignment. Coupling constants ($J$) are reported in Hertz (Hz). The attributions of the chemical shifts were determined by means of COSY and HMQC. Melting points (mp) were determined using a Büchi 510 apparatus and are uncorrected. High resolution mass spectra (HRMS) were determined on an Agilent 6210 ESI-TOF MS instrument with a flow rate of 4 $\mu$L/min, spray voltage 4 kV, and the desolvation gas set to 15 psi. All other parameters were optimized for maximal abundance of [M+H]$^+$ or [M+Na]$^+$. IR spectra were recorded using a Jasco 6200 FTIR spectrometer. The specific rotation of optically active compounds was determined with a 241 *Perkin-Elmer* polarimeter using a cell with 10.0 cm length and a capacity of 1 mL. Measurements were performed at room temperature using a Na-lamp with a wavelength of $\lambda$ = 589.3 nm. All reactions were monitored by thin-layer chromatography (TLC) using Merck silica gel 60 F254 (0.25 mm thickness). Flash chromatography was carried out using Merck silica gel 60 (0.040–0.063 mm).
Synthesis of Fluoroalkylated γ-Butyrolactones

(S)-4-Benzyl-3-[(S)-2-[[R]-1,1,1-trifluorobutan-2-yl]pent-4-enoyl]oxazolidin-2-one (4)

A solution of NaHMDS (2 M in THF, 0.761 mL, 1.52 mmol) was diluted with THF (5.0 mL) and a solution of 3[1] (334 mg, 1.01 mmol) in THF (5.0 mL) was slowly added at -78 °C. The resulting mixture was stirred for 1 h at that temperature. A solution of allyl iodide (464 μL, 5.08 mmol) in THF (3.0 mL) was added dropwise. The reaction mixture was stirred at -40 °C for 5 h and then allowed to warm to rt overnight. Water (7 mL) was added and the aqueous layer was extracted with diethyl ether (4 x 7 mL). The combined organic phases were washed with brine, dried over sodium sulfate, filtered, and concentrated in vacuo. The crude product (dr = 82:18, 19F NMR) was purified by flash column chromatography (SiO2, ethyl acetate/hexane 1:9) to afford 161 mg (43%, 0.436 mmol) of 4 as colorless oil and 18.0 mg (4.8%, 0.049 mmol) of a mixture of 4/2-epi-4 as a light yellow oil.

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\begin{align*}
\text{[α]}_D^{22} &= +44.2 \ (c = 1.00, \ CHCl_3); \ \text{1H NMR (500 MHz, CDCl}_3): \ \delta = 1.05 \ (t, \ J = 7.6 \ Hz, \ 3 \ H, \ CH_2CH_3), \ 1.69-1.76 \ (m, \ 2 \ H \ CH_2CH_3), \ 2.42-2.47 \ (m, \ 1 \ H, \ CH_2CH=CH_2), \ 2.51-2.58 \ (m, \ 1 \ H, \ CHCF_3), \ 2.60-2.66^* \ (m, \ 1 \ H, \ CH_2CH=CH_2), \ 2.67^* \ (dd, \ J = 10.1, \ 13.3 \ Hz, \ 1 \ H, \ CH_3Ph), \ 3.31 \ (dd, \ J = 3.3, \ 13.3 \ Hz, \ 1 \ H, \ CH_3Ph), \ 4.12-4.19 \ (m, \ 2 \ H, \ OCH_2), \ 4.34 \ (ddd, \ J = 4.7, \ 6.2, \ 9.7 \ Hz, \ 1 \ H, \ COCH), \ 4.63-4.68 \ (m, \ 1 \ H, \ NCH), \ 5.09-5.16 \ (m, \ 2 \ H, \ CH_2CH=CH_2), \ 5.78-5.87 \ (m, \ 1 \ H, \ CH_2CH=CH_2), \ 7.22-7.35 \ (m, \ 5 \ H, \ Ph) \ ppm; \ \text{13C NMR (126 MHz, CDCl}_3): \ \delta = 12.0 \ (CH_2CH_3), \ 19.9 \ (q, \ J_{CF} = 2.4 \ Hz, \ CH_2CH_3), \ 33.4 \ (CH_2CH=CH_2), \ 37.8 \ (CH_2Ph), \ 41.0 \ (m_c, \ COCH), \ 45.6 \ (q, \ J_{CF} = 24.7 \ Hz, \ CHCF_3), \ 55.9 \ (NCH), \ 66.0 \ (OCH_2), \ 118.0 \ (CH_2CH=CH_2) \ 127.3 \ (para-Ar-C), \ 127.9 \ (q, \ J_{CF} = 282 \ Hz, \ CF_3), \ 128.9 \ (ortho-Ar-C), \ 129.4 \ (meta-Ar-C), \ 134.4 \ (Ar-C), \ 135.2 \ (CH_2CH=CH_2), \ 152.9 \ (OCON), \ 172.9 \ (NCOCH) \ ppm; \ \text{19F NMR (376 MHz, CDCl}_3): \ \delta = -65.6 \ (d, \ J = 9.4 \ Hz, \ 3 \ F, \ CF_3) \ ppm; \ \text{IR (Film): v} = 702, \ 746, \ 923, \ 1050, \ 1107, \ 1170, \ 1252, \ 1350, \ 1366, \ 1387, \ 1701, \ 1781, \ 2887, \ 2944, \ 2978 \ cm^{-1}; \ \text{HRMS (ESI): m/z [M+Na]^+ calcd. for [C_19H_22F_3NNaO_3]^+: 392.1444; found: 392.1445.}
\end{align*}
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(S)-4-Benzyl-3-[(S)-2-[[R]-1,1,1-trifluoropropan-2-yl]pent-4-enoyl]oxazolidin-2-one (7)

A solution of NaHMDS (2 M in THF, 0.987 mL, 1.97 mmol) was diluted with THF (7.0 mL) and a solution of 6[1] (415 mg, 1.32 mmol) in THF (7.0 mL) was slowly added at -78 °C. The
resulting mixture was stirred for 1 h at that temperature. A solution of allyl bromide (558 μL, 6.58 mmol) in THF (4.0 mL) was added dropwise. The reaction mixture was stirred at -40 °C for 4 h and then allowed to warm to rt overnight. Water (7 mL) was added and the aqueous layer was extracted with diethyl ether (4 x 10 mL). The combined organic phases were washed with brine, dried over sodium sulfate, filtered, and concentrated in vacuo. The crude product (dr = 81:19, 1H NMR) was purified by flash column chromatography (SiO₂, ethyl acetate/hexane 2:11) to afford 236 mg (50%, 0.664 mmol) of 7 as colorless oil.

\[ \alpha \]D = +37.8 (c = 1.01, CHCl₃); ¹H NMR (500 MHz, CDCl₃): δ = 1.24 (d, J = 7.1 Hz, 3 H, CH₃), 2.44-2.51 (m, 1 H, CH₂CH=CH₂), 2.57-2.65' (m, 2 H, CH₂CH=CH₂, CH₂CH₃), 2.65'' (dd, J = 10.2, 13.3 Hz, 1 H, CH₂Ph), 3.31 (dd, J = 3.3, 13.3 Hz, 1 H, CH₂Ph), 4.14-4.15 (m, 2 H, OCH₂), 4.43 (m, 1 H, COCH), 4.63-4.68 (m, 1 H, NCH), 5.09-5.17 (m, 2 H, CH₂CH=CH₂), 5.81 (m, 1 H, CH₂CH=CH₂), 7.22-7.35 (m, 5 H, Ph) ppm; *signal overlap; ¹³C NMR (126 MHz, CDCl₃): δ = 10.8 (q, J_CF = 3.0 Hz, CH₃), 34.7 (CH₂CH=CH₂), 37.8 (CH₂Ph), 39.9 (q, J_CF = 26.4 Hz, CHCH₃), 40.8 (m, COCH), 55.9 (NCH), 65.9 (OCH₂), 118.4 (CH₂CH=CH₂), 127.3 (para-Ar-C), 127.5 (q, J_CF ~ 279 Hz, CF₃), 128.9 (ortho-Ar-C), 129.4 (meta-Ar-C), 133.9 (Ar-C), 135.3 (CH₂CH=CH₂), 153.0 (OCON), 173.0 (NCOCH) ppm; ¹⁹F NMR (376 MHz, CDCl₃): δ = -69.1 (d, J = 9.3 Hz, 3 F, CF₃) ppm; IR (Film): ν = 702, 747, 923, 1105, 1132, 1173, 1210, 1257, 1351, 1387, 1699, 1783, 2925, 2981 cm⁻¹; HRMS (ESI): m/z [M+Na]+ calcd. for [C₁₈H₂₀F₃NNaO₃]+: 378.1288; found: 378.1293.

(S)-3-[(R)-1,1,1-Trifluorobutan-2-yl]dihydrofuran-2(3H)-one (5)

A solution of 4 (125 mg, 0.338 mmol) in dichloromethane (6.0 mL) was treated with ozone at -78 °C for 20 min (laboratory ozonator using commercial grade oxygen as source [6.00 V, 0.130 mA, 15 cc/min]). The intense blue solution was then flushed with nitrogen for 15 min at -78 °C and a solution of triphenylphosphane (97.0 mg, 0.370 mmol) in dichloromethane (1.0 mL) was added. The mixture was stirred at -78 °C for 30 min. The solution was allowed to warm to rt and stirring was continued for 2 h. The mixture was concentrated and diluted with dichloromethane (1.0 mL). The solution was triturated with hexane and the phosphine oxide was filtered off. The solution was concentrated on the rotary evaporator (40 °C, ~250 mbar) to afford 150 mg (quant.) of the aldehyde that was used directly in the next step. The aldehyde obtained from ozonolysis of 4 was dissolved in diethylether (2.0 mL) and was
added to a stirred suspension of NaBH₄ on alumina[2] (326 mg, ~0.816-0.978 mmol) in Et₂O (2.0 mL) at rt. Stirring was continued for 15 min, the solid was then filtered off and washed with diethylether. The solution was concentrated on the rotary evaporator (40 °C, ~250 mbar). The crude product was purified by flash column chromatography (SiO₂, diethylether/pentane 1:2) to afford 27.3 mg (41%, 0.139 mmol) of 5 as a colorless oil.

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\text{[\alpha]D}^{22} = +7.08 \quad (c = 0.50, \text{CHCl}_3)
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{^1}\text{H NMR (500 MHz, CDCl}_3\text{):} \quad \delta = 1.04 \quad (t, \text{ J} = 7.5 \text{ Hz, 3 H, CH}_2\text{CH}_3), \quad 1.37-1.43 \quad (m, \text{ 1 H, CH}_2\text{CH}_3), \quad 1.69-1.78 \quad (m, \text{ 1 H, CH}_2\text{CH}_3) \quad 2.18-2.33 \quad (m, \text{ 2 H, CH}_2\text{CH}_2\text{O}), \quad 2.75-2.83 \quad (m, \text{ 1 H, CHCF}_3), \quad 2.99 \quad (ddd, \text{ J} = 11.8, 9.1, 2.4 \text{ Hz, 1 H, COCH}), \quad 4.22 \quad (ddd, \text{ J} = 6.6, 8.9, 10.8 \text{ Hz, 1 H, CH}_2\text{CH}_2\text{O}), \quad 4.43 \quad (m, \text{ 1 H, CH}_2\text{CH}_2\text{O}) \quad \text{ppm;} \quad {^{13}}\text{C NMR (126 MHz, CDCl}_3\text{):} \quad \delta = 12.5 \quad (\text{CH}_2\text{CH}_3), \quad 18.3 \quad (q, \text{ J}_{\text{CF}} = 1.9 \text{ Hz, CH}_2\text{CH}_3), \quad 23.4 \quad (\text{CH}_2\text{CH}_2\text{O}), \quad 39.0 \quad (q, \text{ J}_{\text{CF}} = 2.7 \text{Hz, COCH}), \quad 43.3 \quad (q, \text{ J}_{\text{CF}} = 26.0 \text{ Hz, CHCF}_3), \quad 66.6 \quad (\text{CH}_2\text{CH}_2\text{O}) \quad \text{ppm; signals for CF}_3 \quad \text{and OCCH were not found;} \quad {^{19}}\text{F NMR (376 MHz, CDCl}_3\text{):} \quad \delta = -69.4 \quad (d, \text{ J} = 9.2 \text{ Hz, 3 F, CF}_3) \quad \text{ppm; IR (Film):} \quad \nu = 952, 1024, 1058, 1108, 1175, 1256, 1378, 1770, 2328, 2363, 2923 \quad \text{cm}^{-1}; \quad \text{HRMS (ESI):} \quad \text{m/z [M+Na]}^+ \quad \text{calcd. for} \quad [\text{C}_8\text{H}_{11}\text{F}_3\text{NaO}_2]^+: 219.0603; \quad \text{found: 219.0598.}
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(3S,5S)-5-(Hydroxymethyl)-3-[(R)-1,1,1-trifluoropropan-2-yl]dihydrofuran-2(3H)-one (8a) and (3S,5R)-5-(Hydroxymethyl)-3-[(R)-1,1,1-trifluoropropan-2-yl]dihydrofuran-2(3H)-one (8b)

Following the typical procedure, a solution of 7 (125 mg, 0.352 mmol, dissolved in 0.7 mL acetone/water 8:1), K₂OsO₄·2H₂O (10.3 mg, 28.1 μmol, 8 mol%), and N-methylmorpholine-N-oxide (82.2 mg, 0.702 mmol) in acetone/water 8:1 (1.8 mL) was stirred at rt for 17 h. After work-up, the crude product (dr = 60:40, \(^1\text{H NMR}) was purified by flash column chromatography (SiO₂, dichloromethane/methanol 97:3) to afford 46.0 mg (61%, 0.217 mmol) of 8 as a colorless oil.
The diastereoisomers were separated by HPLC (7.0% iPrOH/hexane; flow: 2.0 mL/min, 32 x 250 mm, Nucleosil 50-5, Machery-Nagel) and obtained as colorless oils.

(8a) \([\alpha]_D^{22} = +22.3 \) (c = 1.00, CHCl\(_3\)); \(^1\)H NMR (500 MHz, CDCl\(_3\)): \(\delta = 1.33 \) (d, \(J = 7.1\) Hz, 3 H, CH\(_3\)), 2.27-2.42 (m, 3 H, CH\(_2\)CH\(_2\)OH, CH\(_2\)CHCH\(_2\)OH), 2.70-2.79 (m, 1 H, CHCH\(_3\)), 2.88-2.93 (m, 1 H, COCH), 3.67 (dd, \(J = 3.7\), 12.4 Hz, 1 H, CH\(_2\)CH\(_2\)OH), 3.94 (dd, \(J = 2.7\), 12.4 Hz, 1 H, CH\(_2\)CH\(_2\)OH), 4.60-4.64 (m, 1 H, CH\(_2\)CH\(_2\)OH) ppm; \(^{13}\)C NMR (126 MHz, CDCl\(_3\)): \(\delta = 12.5\) (q, \(J_{CF} = 2.9\) Hz, CH\(_3\)), 26.5 (CH\(_2\)CH\(_2\)OH), 38.7 (q, \(J_{CF} = 26.6\) Hz, CHCH\(_3\)), 40.8 (br. m, COCH), 64.3 (CH\(_2\)CH\(_2\)OH), 78.2 (CH\(_2\)CH\(_2\)OH), 127.3 (q, \(J_{CF} = 281\) Hz, CF\(_3\)), 176.8 (OCCH) ppm; \(^{19}\)F NMR (376 MHz, CDCl\(_3\)): \(\delta = -68.6\) (d, \(J = 8.7\) Hz, 3 F, CF\(_3\)) ppm; IR (Film): \(\nu = 631, 701, 966, 1020, 1062, 1139, 1171, 1199, 1264, 1359, 1466, 1768, 2953, 2989, 3427\) cm\(^{-1}\); HRMS (ESI): \(m/z\) [M+Na]\(^+\) calcd. for [C\(_8\)H\(_{11}\)F\(_3\)NaO\(_3\)]\(^+\): 235.0553; found: 235.0554.

(8b) \([\alpha]_D^{22} = -22.9 \) (c = 1.00, CHCl\(_3\)); \(^1\)H NMR (500 MHz, CDCl\(_3\)): \(\delta = 1.39 \) (d, \(J = 6.7\) Hz, 3 H, CH\(_3\)), 2.03 (br. m, 1 H, OH), 2.11-2.18 (m, 1 H, CH\(_2\)CH\(_2\)OH), 2.35-2.40 (m, 1 H, CH\(_2\)CH\(_2\)OH), 2.68-2.76 (m, 2 H, CHCH\(_3\), COCH), 3.66 (dd, \(J = 4.8\), 12.7 Hz, 1 H, CH\(_2\)CH\(_2\)OH), 3.94 (dd, \(J = 2.8\), 12.7 Hz, 1 H, CH\(_2\)CH\(_2\)OH), 4.46-4.51 (m, 1 H, CH\(_2\)CH\(_2\)OH) ppm; \(^{13}\)C NMR (126 MHz, CDCl\(_3\)): \(\delta = 12.3\) (q, \(J_{CF} = 3.1\) Hz, CH\(_3\)), 27.5 (CH\(_2\)CH\(_2\)OH), 38.8 (q, \(J_{CF} = 26.8\) Hz, CHCH\(_3\)), 41.9 (br. m, COCH), 63.3 (CH\(_2\)CH\(_2\)OH), 78.5 (CH\(_2\)CH\(_2\)OH), 127.2 (q, \(J_{CF} = 280\) Hz, CF\(_3\)), 175.4 (OCCH) ppm; \(^{19}\)F NMR (376 MHz, CDCl\(_3\)): \(\delta = -69.1\) (d, \(J = 8.0\) Hz, 3 F, CF\(_3\)) ppm; IR (Film): \(\nu = 744, 913, 970, 1046, 1141, 1170, 1201, 1266, 1353, 1396, 1465, 1768, 2928, 2992, 3420\) cm\(^{-1}\); HRMS (ESI): \(m/z\) [M+Na]\(^+\) calcd. for [C\(_8\)H\(_{11}\)F\(_3\)NaO\(_3\)]\(^+\): 235.0553; found: 235.0569.

**Synthesis of Fluoroalkylated Hydroxy γ-Butyrolactones**

(S)-4-Benzyl-3-[(2S,3R,E)-3-hydroxy-2-(2,2,2-trifluoroethyl)hex-4-enoyl]oxazolidin-2-one (10b)

Following the typical procedure, a solution of 9\(^a\) (500 mg, 1.66 mmol), TiCl\(_4\) (191 \(\mu\)L, 1.74 mmol), (-)-sparteine (953 \(\mu\)L, 4.15 mmol), and (E)-crotonaldehyde (176 \(\mu\)L, 2.16 mmol) in CH\(_2\)Cl\(_2\) (11.8 mL) was stirred at 0 °C for 3 h. After work-up, the crude product (dr > 95:5, \(^{19}\)F NMR) was purified by flash column chromatography (SiO\(_2\), ethyl acetate/hexane 1:2) to afford 475 mg (77%, 1.28 mmol) of syn-aldol derivate 10\(^b\) as colorless oil.
[α]D22 = +43.5 (c = 2.00, CHCl3); 1H NMR (500 MHz, CDCl3): δ = 1.71 (d, J = 7.2 Hz, 3 H, CHCH=CHCH3), 2.39-2.49 (m, 1 H, CH2CF3), 2.61 (br. s, 1 H, OH), 2.67 (dd, J = 13.4, 10.0 Hz, 1 H, CH2Ph), 2.83 (m, 1 H, CH2CF3), 3.31 (dd, J = 13.4, 3.2 Hz, 1 H, CH2Ph), 4.14-4.17 (m, 2 H, OCH2), 4.31 (m, 1 H, CHCH=CHCH3), 4.51 (ddd, J = 10.9, 5.3, 1.7 Hz, 1 H, COCH), 4.68 (m, 1 H, NCH), 5.51 (m, 1 H, CHCH=CHCH3), 5.77 (m, 1 H, CHCH=CHCH3), 7.20-7.34 (5 H, Ph) ppm; 13C NMR (126 MHz, CDCl3): δ = 17.6 (CHCH=CHCH3), 31.7 (q, JCF = 29.2 Hz, CH2CF3), 37.4 (CH2Ph), 42.4 (m, COCH), 55.5 (NCH), 66.1 (OCH2), 73.0 (CHCH=CHCH3), 126.7 (q, JCF = 277 Hz, CH2CF3), 127.3 (para-Ar-C), 128.9 (ortho-Ar-C), 129.2 (CHCH=CHCH3), 129.3 (meta-Ar-C), 129.8 (CHCH=CHCH3), 135.1 (Ar-C), 153.4 (OCON), 172.5 (NCOC) ppm; 19F NMR (376 MHz, CDCl3): δ = -64.7 (t, J = 11.0 Hz, 3 F, CF3) ppm; IR (Film): ν = 653, 704, 762, 839, 972, 1030, 1101, 1148, 1260, 1360, 1454, 1481, 1604, 1696, 2860, 2921, 2968, 3031, 3479 cm−1; HRMS (ESI): m/z [M+Na]+ calcd. for [C18H20F3NNaO4]+: 394.1237; found: 394.1250.

(S)-4-Benzyl-3-[(2S,3R,E)-3-hydroxy-5-phenyl-2-(2,2,2-trifluoroethyl)pent-4-enoyl]oxazolidin-2-one (10c) and (S)-4-Benzyl-3-[(2R,3S,E)-3-hydroxy-5-phenyl-2-(2,2,2-trifluoroethyl)pent-4-enoyl]oxazolidin-2-one (10c')

Following the typical procedure, a solution of 9a[3] (650 mg, 2.16 mmol), TiCl4 (248 μL, 2.27 mmol), (-)-sparteine (1.24 mL, 5.40 mmol), and (E)-cinnamaldehyde (353 μL, 2.81 mmol) in CH2Cl2 (15.3 mL) was stirred at 0 °C for 2 h. After work-up, the crude product (dr = 90:10, 19F NMR) was purified by flash column chromatography (SiO2, ethyl acetate/hexane 2:7 → 2:5) to afford 603 mg (64%, 1.39 mmol) of syn-aldol derivate 10c as colorless, foamy oil and 38.0 mg (4.0%, 0.088 mmol) of syn-aldol[4] 10c’ as colorless oil.

[α]D22 = +129 (c = 2.00, CHCl3); 1H NMR (500 MHz, CDCl3): δ = 2.53-2.63 (m, 1 H, CH2CF3), 2.68 (dd, J = 13.5, 10.0 Hz, 1 H, CH2Ph), 2.77 (br. s, 1 H, OH), 2.94 (m, 1 H, CH2CF3), 3.31 (dd, J = 13.5, 3.2 Hz, 1 H, CH2Ph), 3.92 (m, 1 H, OCH2), 4.09 (dd, J = 9.1, 2.6 Hz, 1 H,
OCH₂), 4.53 (m, 1 H, CHCH=CHPh), 4.63 (m, 1 H, NCH), 4.68 (m, 1 H, COCH), 6.25 (d, J = 15.9, 6.9 Hz, 1 H, CHCH=CHPh), 6.64 (d, J = 15.9 Hz, 1 H, CHCH=CHPh), 7.20-7.40 (10 H, Ph, CHCH=CHPh) ppm; *signal overlap; ¹³C NMR (126 MHz, CDCl₃): δ = 31.8 (q, J CF = 29.3 Hz, CH₂CF₃), 37.4 (CH₂Ph), 42.8 (m, COCH), 55.5 (NCH), 66.1 (OCH₂), 73.3 (CHCH=CHPh), 126.7 (q, J CF = 277 Hz, CH₂CF₃), 126.5 (Ar), 127.2 (Ar), 127.3 (CHCH=CHPh), 128.3 (Ar), 128.7 (Ar), 128.9 (Ar), 129.3 (Ar), 132.8 (CHCH=CHPh), 135.0 (Ar-C), 135.7 (Ar-C), 153.5 (OCON), 172.4 (NCOC) ppm; ¹⁹F NMR (376 MHz, CDCl₃): δ = -64.6 (t, J = 11.0 Hz, 3 F, CF₃) ppm; IR (Film): ν = 699, 736, 911, 972, 1016, 1100, 1147, 1210, 1261, 1353, 1393, 1453, 1496, 1697, 1779, 2921, 3029, 3485 cm⁻¹; HRMS (ESI): m/z [M+Na⁺] calcd. for [C₂₃H₂₂F₃NNaO₄⁺]: 456.1393; found: 456.1408.

(S)-4-Benzyl-3-[(S)-4,4,5,5,6,6,6-heptafluoro-2-[(R)-1-hydroxyallyl]hexanoyl]oxazolidin-2-one (10d)

Following the typical procedure, a solution of 9b[¹] (105 mg, 0.262 mmol), TiCl₄ (30.2 μL, 0.275 mmol), (-)-sparteine (150 μL, 0.654 mmol), and acroleine (22.7 μL, 0.340 mmol) in CH₂Cl₂ (2.2 mL) was stirred at 0 °C for 2 h. After work-up, the crude product (dr > 95:5, ¹⁹F NMR) was purified by flash column chromatography (SiO₂, ethyl acetate/hexane 1:2) to afford 86.0 mg (71%, 0.188 mmol) of syn-aldol derivate 10d as light yellow oil.

[¹]"The typical procedure"
(S)-4-Benzyl-3-{(2S,3R)-3-hydroxy-2-[((R)-1,1,1-trifluorobutan-2-yl]pent-4-enoyl}oxazolidin-2-one (10e)

Acyl-oxazolidinone 3[1] (390 mg, 1.18 mmol) was dissolved in dichloromethane (12.0 mL) and TiCl₄ (138 μL, 1.25 mmol) was added dropwise at -78 °C. The solution was warmed to 0 °C and stirred for 10 min before N,N-diisopropylethylamine (0.489 mL, 2.96 mmol) was added dropwise. The resulting dark brown solution was stirred at 0 °C for 1 h. The mixture was cooled to -78 °C, and freshly distilled acroleine (119 μL, 1.78 mmol) was added. After 1 h, the solution was warmed to 0 °C and stirred at that temperature for 12 h. The reaction was then allowed to warm to rt and aqueous saturated ammonium chloride solution (3.5 mL) was added. The mixture was filtered over celite and the aqueous phase was extracted with dichloromethane (4 x 5.0 mL). The combined organic phases were washed with aqueous saturated sodium bicarbonate solution and brine, dried over sodium sulfate, filtered, and concentrated in vacuo. The crude product (dr = 76:24, ¹⁹F NMR) was purified flash column chromatography (SiO₂, ethyl acetate/hexane 1:4 → 1:3) to afford 170 mg (37%, 0.441 mmol) of syn-aldol derivative 10e as light yellow oil and 65.0 mg (14%, 0.169 mmol) of a mixture of the minor diastereoisomers as light yellow oil.
(S)-4-Benzyl-3-[(2S,3R)-3-[(tert-butyldimethylsilyl)oxy]-2-[(R)-1,1,1-trifluorobutan-2-yl]pent-4-enoyl]oxazolidin-2-one (10f)

TBSOTf (50.8 μL, 0.221 mmol) was added dropwise to a solution of syn-aldol derivative 10e (50.0 mg, 0.130 mmol) and 2,6-lutidine (45.2 μL, 0.390 mmol) in dichloromethane (0.5 mL) at 0 °C. The reaction mixture was stirred at 0 °C for 6 h and was then allowed to warm to rt overnight. The reaction was quenched by the addition of aqueous saturated ammonium chloride solution (1.5 mL). The aqueous layer was extracted with diethyl ether (4 x 2 mL). The combined organic phases were washed with cold aqueous 0.1 N HCl solution and with aqueous saturated NaHCO3 solution, dried over sodium sulfate, filtered, and concentrated in vacuo. The crude product was purified by flash column chromatography (SiO2, ethyl acetate/hexane 1:9) to afford 49.0 mg (75%, 0.098 mmol) of 10f as colorless oil.
(S)-4-Benzyl-3-{{(2S,3R)-3-{{(tert-butyldimethylsilyl)oxy}-2-(2,2,2-trifluoroethyl)pent-4-enoyl}oxazolidin-2-one (10g)

TBSOTf (317 μL, 1.38 mmol) was added dropwise to a solution of syn-aldol derivative 10a (290 mg, 0.812 mmol) and 2,6-lutidine (282 μL, 2.44 mmol) in dichloromethane (2.9 mL) at 0 °C. The reaction mixture was stirred at 0 °C for 5 h and was then allowed to warm to rt overnight. The reaction was quenched by the addition of aqueous saturated ammonium chloride solution (3.5 mL). The aqueous layer was extracted with dichloromethane (4 x 4 mL). The combined organic phases were washed with cold aqueous 0.1 N HCl solution and with aqueous saturated NaHCO₃ solution, dried over sodium sulfate, filtered, and concentrated in vacuo. The crude product was purified by flash column chromatography (SiO₂, ethyl acetate/hexane 1:11) to afford 322 mg (84%, 0.683 mmol) of 10g as light yellow oil.

[α]D²² = +50.9 (c = 2.00, CHCl₃); ¹H NMR (500 MHz, CDCl₃): δ = 0.00 (s, 6 H, Si(CH₃)₂), 0.90 (s, 9 H, SiC(CH₃)₃), 2.34-2.44 (m, 1 H, CH₂CF₃), 2.75 (dd, J = 13.5, 9.7 Hz, 1 H, CH₂Ph), 2.89 (m, 1 H, CH₂CF₃), 3.28 (dd, J = 13.5, 3.1 Hz, 1 H, CH₂Ph), 4.12-4.20 (m, 2 H, OCH₂), 4.36-4.41 (m, 2 H, COCH, CHCH=CH₂), 4.64 (m, 1 H, NCH), 5.21-5.29 (m, 2 H, CHCH=CH₂), 5.88 (ddd, J = 17.0, 10.4, 5.5 Hz, 1 H, CHCH=CH₂), 7.21-7.35 (5 H, Ph) ppm;
$^{13}$C NMR (126 MHz, CDCl$_3$): $\delta$ = -5.40 (Si(CH$_3$)$_2$), -4.60 (Si(CH$_3$)$_2$), 18.0 (SiC(CH$_3$)$_3$), 25.6 (SiC(CH$_3$)$_3$), 30.6 (q, $J_{CF}$ = 29.1 Hz, CH$_2$CF$_3$), 37.3 (CH$_2$Ph), 43.3 (m, COCH), 55.6 (NCH), 65.9 (OCH$_2$), 73.9 (CHCH=CH$_2$), 116.8 (CHCH=CH$_2$), 126.9 (q, $J_{CF}$ = 277 Hz, CH$_2$CF$_3$), 127.3 (para-Ar-C), 128.9 (ortho-Ar-C), 129.4 (meta-Ar-C), 135.1 (Ar-C), 137.9 (CHCH=CH$_2$), 153.1 (OCON), 171.4 (NCOC) ppm; $^{19}$F NMR (376 MHz, CDCl$_3$): $\delta$ = -64.8 (t, $J = 11.1$ Hz, 3 F, CF$_3$) ppm; IR (Film): $\nu$ = 649, 703, 739, 779, 836, 939, 1031, 1102, 1135, 1211, 1264, 1362, 1391, 1472, 1705, 1790, 2859, 2931, 2956, 2956 cm$^{-1}$; HRMS (ESI): $m/z$ [M+Na]$^+$ calcd. for [C$_{23}$H$_{32}$F$_3$NNaO$_4$Si]: 494.1945; found: 494.1964.

(3S,4S,5R)-4-Hydroxy-5-[[(S)-1-hydroxyethyl]-3-(2,2,2-trifluoroethyl)dihydrofuran-2(3H)-one (11b)

Following the typical procedure, a solution of 10b (183 mg, 0.492 mmol, dissolved in 1.4 mL acetone/water 8:1), K$_2$OsO$_4$·2H$_2$O (18.1 mg, 49.2 $\mu$mol, 10 mol%), and $N$-methylmorpholine-$N$-oxide (115 mg, 0.984 mmol) in acetone/water 8:1 (1.0 mL) was stirred for 17 h. After work-up, the crude product (dr = 93:7, $^{19}$F NMR) was purified by flash column chromatography (SiO$_2$, dichloromethane/methanol 30:1.7) to afford 60.0 mg (53%, 0.263 mmol) of 11b as a colorless solid.

mp 102-105 °C; [\(\alpha\)]$_D^{22}$ = +31.7 (c = 1.00, MeOH); $^1$H NMR (500 MHz, MeOD): $\delta$ = 1.30 (d, 3 H, $J = 6.6$ Hz, CHCHOHCH$_3$), 2.48-2.59 (m, 1 H, CH$_2$CF$_3$), 2.71 (m, 1 H, CH$_2$CF$_3$), 2.90 (m, 1 H, COCH), 3.91 (qd, $J = 6.6$, 3.2 Hz, 1 H, CHCHOHCH$_3$), 4.05 (dd, $J = 6.3$, 3.2 Hz, 1 H, CHCHOHCH$_3$), 4.27 (m, 1 H, CHCHOH) ppm; signal for OH was not found; $^{13}$C NMR (126 MHz, MeOD): $\delta$ = 19.8 (CHCHOHCH$_3$), 33.8 (q, $J_{CF}$ = 30.3 Hz, CH$_2$CF$_3$), 45.1 (q, $J_{CF}$ = 2.4 Hz, COCH), 67.0 (CHCHOHCH$_3$), 73.9 (CHCHOH), 89.6 (CHCHOHCH$_3$), 127.9 (q, $J_{CF}$ = 276 Hz, CF$_3$), 176.5 (OCOC) ppm; $^{19}$F NMR (376 MHz, MeOD): $\delta$ = -65.8 (t, $J = 11.0$ Hz, CF$_3$) ppm; IR (Film): $\nu$ = 622, 837, 993, 1054, 1083, 1146, 1234, 1264, 1368, 1439, 1770, 2937, 2983, 3401 cm$^{-1}$. HRMS (ESI): $m/z$ [M+Na]$^+$ calcd. for [C$_{8}$H$_{11}$F$_{3}$NaO$_4$]: 251.0502; found: 251.0516.

(3S,4S,5S)-4-Hydroxy-5-[(S)-hydroxy(phenyl)methyl]-3-(2,2,2-trifluoroethyl)-dihydrofuran-2(3H)-one (11c)
Following the typical procedure, a solution of 10c (263 mg, 0.607 mmol, dissolved in 1.7 mL acetone/water 8:1), K$_2$OsO$_4$·2H$_2$O (22.4 mg, 60.7 μmol, 10 mol%), and N-methylmorpholine-N-oxide (142 mg, 1.22 mmol) in acetone/water 8:1 (1.2 mL) was stirred for 17 h. After work-up, the crude product (dr > 95:5, $^{19}$F NMR) was purified by flash column chromatography (SiO$_2$, dichloromethane/methanol 30:0.8) to afford 84.0 mg (47%, 0.289 mmol) of 11c as a colorless solid.

![Chemical structure of 11c](image)

mp 67-70 °C; [α]$_D^{22}$ = +45.2 (c = 0.60, MeOH); $^1$H NMR (500 MHz, MeOD): δ = 2.37-2.48 (m, 1 H, CH$_2$CF$_3$), 2.66 (m, 1 H, CH$_2$CF$_3$), 2.88 (ddd, J = 9.6, 6.9, 3.8 Hz, 1 H, COCH), 4.38 (dd, J = 5.6, 2.5 Hz, 1 H, CHCHOHPh), 4.43-4.46 (m, 1 H, CHCHOH), 4.89 (d, J = 6.2 Hz, 1 H, CHCHOHPh), 7.27-7.30 (m, 1 H, Ph), 7.34-7.38 (m, 2 H, Ph), 7.40-7.44 (m, 2 H, Ph) ppm; signal for OH was not found; $^{13}$C NMR (126 MHz, MeOD): δ = 33.8 (q, J$_{CF}$ = 30.2 Hz, CH$_2$CF$_3$), 45.2 (q, J$_{CF}$ = 2.5 Hz, COCH), 72.7 (CHCHOHPh), 73.8 (CHCHOH), 89.8 (CHCHOHPh), 127.8 (ortho-Ar-C), 127.9 (q, J$_{CF}$ = 276 Hz, CF$_3$), 128.8 (para-Ar-C), 129.3 (meta-Ar-C), 142.2 (Ar-C), 176.7 (OCOC) ppm; $^{19}$F NMR (376 MHz, MeOD): δ = -64.0 (t, J = 10.7 Hz, CF$_3$) ppm; IR (Film): ν = 623, 648, 702, 741, 843, 913, 950, 1022, 1148, 1230, 1264, 1439, 1454, 1776, 2926, 3424 cm$^{-1}$. HRMS (ESI): m/z [M+Na]$^+$ calcd. for [C$_{13}$H$_{13}$F$_3$NaO$_4$]$^+$: 313.0658; found: 313.0664.

(3S,4S,5R)-3-(2,2,3,3,4,4,4-Heptafluorobutyl)-4-hydroxy-5-(hydroxymethyl)-dihydrofuran-2(3H)-one (11d)

Following the typical procedure, a solution of 10d (50 mg, 0.109 mmol, dissolved in 0.5 mL acetone/water 8:1), K$_2$OsO$_4$·2H$_2$O (4.00 mg, 10.9 μmol, 10 mol%), and N-methylmorpholine-N-oxide (25.6 mg, 0.219 mmol) in acetone/water 8:1 (0.8 mL) was stirred for 17 h. After work-up, the crude product (dr = 91:9, $^{19}$F NMR) was purified by flash column chromatography (SiO$_2$, dichloromethane/methanol 20:1) to afford 16.5 mg (48%, 52.5 μmol) of 11d as a colorless solid.
1H NMR (500 MHz, C3D6O): \( \delta = 2.56-2.75 \) (m, 2 H, CH2C3F7), 3.07 (m, 1 H, COCH), 3.72-3.76 (m, 1 H, CHCH2OH), 3.89-3.94 (m, 1 H, CHCH2OH), 4.28 (m, 1 H, CHCH2OH), 4.32 (m, 1 H, CHCH2OH), 4.40-4.44 (m, 1 H, CHCHOH), 5.04 (d, \( J = 5.9 \) Hz, 1 H, CHCHOH) ppm; 13C NMR (126 MHz, C3D6O): \( \delta = 29.8 \) (m, C3H2C3F7), 43.4 (m, COCH), 61.2 (CHCH2OH), 73.2 (CHCH2OH), 86.3 (CHCH2OH), 175.2 (OOC) ppm; signals for CF2CF2CF3, CF2CF2CF3, CF2CF2CF3 could not be detected; 19F NMR (376 MHz, C3D6O): \( \delta = -81.3 \) (t, \( J = 9.9 \) Hz, 3 F, CF2CF2CF3), -113.7 (m, 2 F, CF2CF2CF3), -128.4 (m, 2 F, CF2CF2CF3) ppm; IR (Film): \( \nu = 666, 731, 993, 1046, 1146, 1176, 1224, 1307, 1337, 1361, 1756, 2326 \) cm\(^{-1}\); HRMS (ESI): m/z [M+Na]\(^+\) calcd. for [C9H9F7NaO4]: 337.0281; found: 337.0288.

Following the typical procedure, a solution of 10f (126 mg, 0.252 mmol, dissolved in 1.3 mL acetone/water 8:1), K2OsO4·2H2O (9.28 mg, 25.2 \( \mu \)mol, 10 mol%), and N-methylmorpholine-N-oxide (59.0 mg, 0.504 mmol) in acetone/water 8:1 (1.2 mL) was stirred for 16 h. After work-up, the crude product (dr = 93:7, 19F-NMR) was purified by flash column chromatography (SiO2, ethylacetate/hexane 1:3) to afford 37.8 mg (42%, 0.106 mmol) of 11f as a colorless oil.

\[ \alpha \]D\(^{22} \) = +2.40 (c = 1.00, CHCl3); 1H NMR (500 MHz, CDC13): \( \delta = 0.10 \) (s, 6 H, Si(CH3)2), 0.87 (s, 9 H, SiC(CH3)3), 1.01 (t, \( J = 7.5 \) Hz, 3 H, CH2CH3), 1.53 (m, 1 H, CH2CH3), 1.76-1.87 (m, 1 H, CH2CH3), 2.11 (br. s, 1 H, OH), 2.68-2.77 (m, 1 H, CHCF3), 3.11 (dd, \( J = 7.8 \), 2.4 Hz, 1 H, COCH), 3.77 (dd, \( J = 12.9 \), 3.1 Hz, 1 H, CHCH2OH), 4.02 (dd, \( J = 12.9 \), 2.3 Hz, 1 H, CHCH2OH), 4.21 (m, 1 H, CHCH2OH), 4.66 (m, 1 H, CHCHOTBS) ppm; 13C NMR (126 MHz, CDC13): \( \delta = -5.03 \) (Si(CH3)2), -3.97 (Si(CH3)2), 12.3 (CH2CH3), 17.8 (SiC(CH3)3), 19.0
15

(m,c, CH₂CH₃), 25.6 (SiC(CH₃)₃), 43.4 (q, J_CF = 26.4 Hz, CHCF₃), 49.2 (m,c, COCH), 60.6 (CHCH₂OH), 68.6 (CHCHOTBS), 85.2 (CHCH₂OH), 127.4 (q, J_CF = 280 Hz, CF₃), 174.2 (OCOC) ppm; ¹⁹F NMR (376 MHz, CDCl₃): δ = -66.9 (d, J = 10.3 Hz, 3 F, CF₃) ppm; IR (Film): ν = 779, 839, 1040, 1146, 1177, 1258, 1390, 1465, 1777, 2860, 2933, 2953 cm⁻¹. HRMS (ESI): m/z [M+Na]⁺ calcd. for [C₁₅H₂₇F₃NaO₄Si]⁺: 379.1523; found: 379.1515.

(3S,4S,5R)-4-[(tert-Butyldimethylsilyl)oxy]-5-(hydroxymethyl)-3-(2,2,2-trifluoroethyl)dihydrofuran-2(3H)-one (11g)

Following the typical procedure, a solution of 10g (164 mg, 0.348 mmol, dissolved in 1.0 mL acetone/water 8:1), K₂OsO₄·2H₂O (12.8 mg, 34.8 μmol, 10 mol%), and N-methylmorpholine-N-oxide (81.5 mg, 0.696 mmol) in acetone/water 8:1 (0.8 mL) was stirred for 15 h. After work-up, the crude product (dr = 95:5, ¹⁹F NMR) was purified by flash column chromatography (SiO₂, ethyl acetate/hexane 1:6 → 1:4) to afford 63.0 mg (55%, 0.192 mmol) of 11g as a colorless solid.

mp 53-56 °C; [α]D²² = +3.08 (c = 0.12, CHCl₃); ¹H NMR (500 MHz, CDCl₃): δ = 0.09 (s, 6 H, Si(CH₃)₂), 0.88 (s, 9 H, SiC(CH₃)₃), 2.49-2.67 (m, 3 H, CH₂CF₃, CHCH₂OH), 2.86-2.90 (m, 1 H, COCH), 3.71 (dd, J = 12.8, 2.4 Hz, 1 H, CHCH₂OH), 3.99 (dd, J = 12.8, 2.2 Hz, 1 H, CHCH₂OH), 4.29 (m,c, 1 H, CHCH₂OH), 4.46 (m,c, 1 H, CHCHOTBS) ppm; ¹³C NMR (126 MHz, CDCl₃): δ = -4.96 (Si(CH₃)₂), -4.56 (Si(CH₃)₃), 17.7 (SiC(CH₃)₃), 25.5 (SiC(CH₃)₃), 32.4 (q, J_CF = 30.2 Hz, CH₂CF₃), 45.3 (q, J_CF = 2.5 Hz, COCH), 60.6 (CHCH₂OH), 72.9 (CHCHOTBS), 86.5 (CHCH₂OH), 126.1 (q, J_CF = 277 Hz, CF₃), 175.6 (OCOC) ppm; ¹⁹F-NMR (376 MHz, CDCl₃): δ = -64.2 (t, J = 10.9 Hz, CF₃) ppm; IR (Film): ν = 608, 782, 838, 898, 1008, 1081, 1150, 1227, 1263, 1378, 1766, 2860, 2932, 2958, 3294 cm⁻¹. HRMS (ESI): m/z [M+Na]⁺ calcd. for [C₁₃H₂₇F₃NaO₄Si]⁺: 351.1210; found: 351.1220.

(3S,4S,5R)-4-Hydroxy-5-(hydroxymethyl)-3-(2,2,2-trifluoroethyl)dihydrofuran-2(3H)-one (11a) from (11g)

Protected syn-aldol derivative 11g (40.0 mg, 0.122 mmol) was dissolved in dichloromethane (1.1 ml) and treated with triethylamine trishydrofluoride (37% in Et₃N, 134 μL, 0.305 mmol) at
rt for 17 h. The mixture was diluted with dichloromethane (10 mL) and brine (6 mL) was added. The aqueous layer was extracted with ethyl acetate (3 x 6 mL). The combined organic phases were dried over sodium sulfate, filtered and concentrated in vacuo. The crude product was purified by flash column chromatography (SiO₂, dichloromethane/methanol 20:1) to afford 21.0 mg (80%, 98.0 μmol) of 11a as a colorless solid.
Compound 7

\begin{align*}
\text{O} & \quad \text{N} \\
\text{CF}_3 & \quad \text{Me} \\
\text{Bn} & \quad \text{Me}
\end{align*}

\textbf{\textsuperscript{1}H NMR (500 MHz, CDCl}_3)\n
\textbf{\textsuperscript{13}C NMR (126 MHz, CDCl}_3)
$^{19}\text{F NMR} \ (376 \text{ MHz, CDCl}_3)$

Compound 4

$^{1}\text{H NMR} \ (500 \text{ MHz, CDCl}_3)$
$^{13}$C NMR (126 MHz, CDCl$_3$)

$^{19}$F NMR (376 MHz, CDCl$_3$)
Compound 5

\[ \text{\textsuperscript{1}H NMR (500 MHz, CDCl}_3\text{)} \]

\[ \text{\textsuperscript{13}C NMR (126 MHz, CDCl}_3\text{)} \]
Compound 8a

$^{19}$F NMR (376 MHz, CDCl$_3$)

$^1$H NMR (500 MHz, CDCl$_3$)
$^{13}$C NMR (126 MHz, CDCl$_3$)

$^{19}$F NMR (376 MHz, CDCl$_3$)
Compound 8b

\[ \text{\(^1H NMR\ (500 MHz, CDCl}_3\)} \]

\[ \text{\(^{13}C NMR\ (126 MHz, CDCl}_3\)} \]

23
\[ 1^9\text{F NMR} \ (376 \text{ MHz, CDCl}_3) \]

**Compound 10a**

\[ \text{\begin{tikzpicture}[scale=0.8,transform shape]
    % NMR spectrum
    % Chemical structure
\end{tikzpicture}} \]

\[ 1^1\text{H NMR} \ (500 \text{ MHz, CDCl}_3) \]
$^{13}\text{C NMR} \ (126 \text{ MHz, CDCl}_3)$

$^{19}\text{F NMR} \ (376 \text{ MHz, CDCl}_3)$
Compound 10b

\[
\begin{align*}
&\text{O} \\
&\text{N} \\
&\text{Bn} \\
&\text{OH} \\
&\text{\text{CF}_3} \\
&\text{Me}
\end{align*}
\]

\[\text{1}^1\text{H NMR (500 MHz, CDCl}_3)\]

\[\text{1}^3\text{C NMR (126 MHz, CDCl}_3)\]
Compound 10c

$^{19}\text{F NMR (376 MHz, CDCl}_3\text{)}$

$^{1}\text{H NMR (500 MHz, CDCl}_3\text{)}$
$^{13}\text{C NMR (126 MHz, CDCl}_3\text{)}$

$^{19}\text{F NMR (376 MHz, CDCl}_3\text{)}$
Compound 10c'

$\text{\textsuperscript{1}H NMR} \ (500 \text{ MHz, CDCl}_3)$

$\text{\textsuperscript{13}C NMR} \ (101 \text{ MHz, CDCl}_3)$
\[ \text{\textsuperscript{19}F NMR (376 MHz, CDCl}_3) \]

Compound 10d

\[ \text{\textsuperscript{1}H NMR (500 MHz, CDCl}_3) \]
$^{13}$C NMR (126 MHz, CDCl$_3$)

$^{19}$F NMR (376 MHz, CDCl$_3$)
Compound 10e

\[ \text{O} \quad \text{O} \quad \text{H} \quad \text{OH} \]
\[ \text{O} \quad \text{N} \quad \text{O} \]
\[ \text{Br} \quad \text{El} \quad \cdots \quad \text{CF}_3 \]

\[^1H\text{ NMR (500 MHz, CDCl}_3\text{)}\]

\[^{13}\text{C NMR (126 MHz, CDCl}_3\text{)}\]
$^{19}$F NMR (376 MHz, CDCl$_3$)
Compound 10f

\[ \text{\(1^H\) NMR (500 MHz, CDCl\textsubscript{3})} \]

\[ \text{\(13^C\) NMR (126 MHz, CDCl\textsubscript{3})} \]
$^{19}$F NMR (376 MHz, CDCl$_3$)

Compound 10g

$^1$H NMR (500 MHz, CDCl$_3$)
$^{13}$C NMR (126 MHz, CDCl$_3$)

$^{19}$F NMR (376 MHz, CDCl$_3$)
Compound 11a

$^1$H NMR (500 MHz, C$_3$D$_6$O)

$^{13}$C NMR (126 MHz, C$_3$D$_6$O)
Compound 11b

$^{19}$F NMR (376 MHz, C$_3$D$_6$O)

$^1$H NMR (500 MHz, MeOD)
$^{13}$C NMR (126 MHz, MeOD)

$^{19}$F NMR (376 MHz, MeOD)
Compound 11c

\[
\text{HO} - \text{CF}_3
\]

\[\text{Ph}\]

\[
\text{H} \quad \text{H} \quad \text{OH}
\]

\[\text{1H NMR (500 MHz, MeOD)}\]

\[\text{13C NMR (126 MHz, MeOD)}\]

\[\text{FT (ppm)}\]

\[\text{FT (ppm)}\]
$^{19}\text{F NMR}$ (376 MHz, MeOD)

**Compound 11d**

$^1\text{H NMR}$ (500 MHz, C$_3$D$_6$O)
$^{13}$C NMR (126 MHz, C$_3$D$_6$O)

$^{19}$F NMR (376 MHz, C$_3$D$_6$O)
Compound 11f

$\begin{align*}
\text{HO} & \quad \text{CF}_3 \\
\text{O} & \quad \text{Et} \\
\text{O} & \quad \text{OTBS}
\end{align*}$

$^1$H NMR (500 MHz, CDCl$_3$)

$^13$C NMR (126 MHz, CDCl$_3$)
\[^{19}F\] NMR (376 MHz, CDCl\(_3\))

Compound 11g

\[^{1}H\] NMR (500 MHz, CDCl\(_3\))
$^{13}$C NMR (126 MHz, CDCl$_3$)

$^{19}$F NMR (376 MHz, CDCl$_3$)
Compound 11b

$^{1} \text{H NMR} \ (700 \text{ MHz, MeOD})$

$^{1} \text{H GOESY} \ (700 \text{ MHz, MeOD})$
Compound 11c

$\text{H NMR (700 MHz, MeOD)}$
$^1\text{H GOESY} (700 \text{ MHz, MeOD})$
Compound 11g

$^1$H NMR (700 MHz, CDCl$_3$)

$^1$H GOESY (700 MHz, CDCl$_3$)
'\textsuperscript{1}H GOESY (700 MHz, CDCl\textsubscript{3})

References


