Supporting Information

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Materials and Methods

General: $^1$H-NMR spectra: Bruker Avance II 300 (300 MHz) and Bruker Avance II+ 600 (600 MHz). $^1$H chemical shifts are reported in ppm relative to residual peaks of deuterated solvents. Higher-order NMR spectra were approximately interpreted as first-order spectra, where possible. The observed signal multiplicities are characterized as follows: s = singlet, d = doublet, t = triplet, q = quartet, quin = quintet, m = multiplet, and br = broad. Coupling constants ($J$) were reported in Hertz (Hz). $^{13}$C-NMR spectra [additional APT (Attached Proton Test)]: Bruker Avance II 300 (75.5 MHz) and Bruker Avance II+ 600 (125.9 MHz). $^{13}$C chemical shifts are reported relative to residual peaks of deuterated solvents. Low resolution ESI-MS: Finnigan LCQ. High resolution ESI-MS: Bruker APEX IV 7T FTICR MS. TLC: Merck precoated sheets, 0.25 mm Sil G/UV$_{254}$. The chromatograms were viewed under UV light and/or by treatment with phosphomolybdic acid (10% in ethanol). Column chromatography: Merck silica gel, grade 60, 230–400 mesh. Solvent proportions are indicated in a volume:volume ratio. All reactions were carried out with magnetic
stirring unless otherwise stated and, in the case of air- or moisture-sensitive substrates and/or reagents, were handled in flame-dried glassware under argon or nitrogen. Organic extracts were dried with anhydrous MgSO₄. 2-Formyl-\(N,N,N\)-trimethylanilinium triflate,¹ 4-formyl-\(N,N,N\)-trimethylanilinium triflate,¹ 4-formyl-\(N,N,N\)-trimethylanilinium iodide,² 3-formyl-\(N,N,N\)-trimethylanilinium iodide,² HCl·H-βAla-Phe-OMe,³ TFA·H-βAla-Phe-OMe,³ (4-carboxyphenyl)(4-methoxyphenyl)iodonium tosylate,⁴ 1-\(O\)-methyl-2,3-\(O\)-isopropylidene-\(D\)-ribofuranoside (9),⁵ and 1-\(O\)-methyl-2,3-\(O\)-isopropylidene-5-\(O\)-toluenesulfonyl-\(D\)-ribofuranoside (12)⁶ and Ag(PPh₃)₂HCO₃⁷ were prepared according to the literature.

HPLC analyses and purifications were carried out on Dionex Ultimate 3000 System with Ultimate 3000 Diode Array Detector coupled in series with Berthold NaI detector. Unless otherwise stated, a Chromolith® SpeedROD RP-18e column (Merck, Darmstadt Germany), 50×4.6 mm, was used for analyses and purifications of radiofluorinated products. Aqueous MeCN solutions were used as a mobile phase. StrataX (RP polymeric phase) cartridges were obtained from Phenomenex (Aschaffenburg, Germany),* Sep-Pak Accell Plus QMA carbonate plus light cartridges,** 40 mg sorbent per cartridge from Waters GmbH (Eschborn, Germany) and Chromafix® 30-PS-HCO₃ cartridges from Macherey-Nagel (Düren, Germany).** ¹⁸F-labeled compounds were identified by spiking of the reaction mixture with ¹⁹F-reference substances.

[¹⁸F]Fluoride was produced via the \(^{18}\text{O}(p,n)\text{¹⁸F}\) reaction by bombardment of enriched [¹⁸O]water with 16.5 MeV protons using a MC16 cyclotron (Scanditronix, Uppsala, Sweden). All isolated radiochemical yields are not decay-corrected. Unless otherwise indicated, all radiochemical experiments were carried out at least in triplicates.

* Preconditioned with 1 mL EtOH followed by 10 mL H₂O.
** Preconditioned with 1 mL H₂O


**Chemistry**

**Preparation of (formylphenyl)(phenyl)iodonium bromides. General procedure 1 – (GP1):**

BF$_3$·Et$_2$O (4.5 mmol) was added dropwise to an ice-cold solution of the appropriate formylphenylboronic acid (3.0 mmol) in anhydrous CH$_2$Cl$_2$ (15 mL) under Ar and the resulting mixture was stirred for 10 min. Thereafter, bis(acetoxy)iodobenzene (3.0 mmol) was added, the cooling bath was removed and the mixture was stirred for a further hour. The solvent was removed under reduced pressure. The rest was taken in CH$_2$Cl$_2$ (25 mL) and stirred vigorously with saturated NaBr (15 mL) for 15 min. Thereafter, CH$_2$Cl$_2$-insoluble (3- and 4-formylphenyl)(phenyl)iodonium bromides were filtered off, washed with H$_2$O (20 mL), MeCN (2×50 mL), Et$_2$O (2×50 mL) and dried. In the case of the more soluble (2-formylphenyl)(phenyl)iodonium bromide, the organic layer was repeatedly treated with saturated NaBr (4×15 mL), dried, filtered and concentrated under reduced pressure. The residue was recrystallized from CH$_2$Cl$_2$/Et$_2$O to give the desired iodonium salt.

**Preparation of diaryliodonium and N,N,N-trimethylanilinium triflates, perchlorates and hydrogen carbonates using reaction of the corresponding bromides or iodides with the appropriate silver salts. General procedure 2 – (GP2):** To prepare the corresponding triflate the appropriate bromide or iodide (0.5 mmol) was dissolved in MeOH (12 mL) and treated with AgOTf, AgClO$_4$ or Ag(PPh$_3$)$_2$HCO$_3$ (0.5 mmol). After vigorous shaking of the mixture for 2 min [or 1 h in the case of Ag(PPh$_3$)$_2$HCO$_3$] while shielded from light, the precipitate of silver halide was centrifuged off (4000 rpm, 10 min). The supernatant was concentrated under reduced pressure and the residue was recrystallized from CH$_2$Cl$_2$/Et$_2$O or acetone/Et$_2$O to give the desired salt. Diaryliodonium bicarbonate salts could not be prepared. In the case of N,N,N-trimethylanilinium bicarbonate salts incomplete anionic exchange was observed owing to the low solubility of
Ag(PPh₃)₂HCO₃ in MeOH. In this case the supernatant was used or radiochemical experiments. Solutions of 3- and 4-substituted 𝑁,𝑁,𝑁-trimethylanilinium bicarbonates could be stored at −80 °C for at least 4 weeks. 2-Formyl-𝑁,𝑁,𝑁-trimethylanilinium bicarbonate was prepared directly before each experiment.

**(2-Formylphenyl)(phenyl)iodonium bromide:** The title compound (0.53 g, 54%) was obtained as an off-white solid according to GP1 from 2-formylphenyl boronic acid (0.38 g, 2.53 mmol), BF₃·Et₂O (0.46 mL, 0.52 g, 3.66 mmol) and bis(acetoxy)iodobenzene (0.86 g, 2.53 mmol). ¹H-NMR (CDCl₃, 300 MHz): δ = 7.58 (t, 𝐽 = 7.6 Hz, 3 H), 7.55–7.75 (m, 2 H), 7.80–7.88 (m, 1 H), 8.20–8.32 (m, 3 H), 10.25 (s, 1 H); ¹³C-NMR (CDCl₃, 75.5 MHz): δ = 115.9, 118.8, 131.5, 131.8, 132.2, 132.6, 133.6, 136.2, 136.4, 137.5, 194.4. MS (ESI): positive mode 𝑚/𝑧 = 341.0 ([M + MeOH]⁺), 309.0 ([M + H]⁺); ESI HRMS: calcd for C₁₃H₁₀O⁻I⁺: 308.9771; found: 308.9779.

**(2-Formylphenyl)(phenyl)iodonium triflate:** The title compound (0.26 g, 74%) was obtained as a colorless solid according to GP2 from (2-formylphenyl)(phenyl)iodonium bromide (0.3 g, 0.77 mmol) and AgOTf (198 mg, 0.77 mmol). ¹H-NMR (CDCl₃, 300 MHz): δ = 7.39 (d, 𝐽 = 7.9 Hz, 1 H), 7.55–7.75 (m, 2 H), 7.75–7.96 (m, 3 H), 8.31 (d, 𝐽 = 7.5 Hz, 2 H), 8.38 (dd, 𝐽 = 7.5, 1.6 Hz, 1 H), 10.29 (s, 1 H); ¹³C-NMR (CDCl₃, 75.5 MHz): δ = 111.1, 111.9, 113.0, 128.7 (q, 𝐽 = 128.1 Hz), 131.9, 132.28, 132.30, 133.2, 137.1, 137.6, 138.1, 194.8. MS (ESI): positive mode 𝑚/𝑧 = 341.0 ([M + MeOH]⁺), 309.0 ([M + H]⁺); MS (ESI): negative mode 𝑚/𝑧 = 149.0 ([CF₃SO₂]⁻); ESI HRMS: calcd for C₁₄H₁₄O₂I⁺: 341.0033; found: 341.0024; calcd for C₁₃H₁₀O⁻I⁺: 308.9771; found: 308.9772

**(3-Formylphenyl)(phenyl)iodonium bromide:**⁸ The title compound (1.23 g, 63%) was obtained as a colorless solid according to GP1 from 3-formylphenyl boronic acid (0.75 g, 5 mmol), BF₃·Et₂O (1 mL, 1.07 g, 7.55 mmol) and bis(acetoxy)iodobenzene
(1.61 g, 5 mmol). $^1$H-NMR [(CD$_3$)$_2$SO, 300 MHz]: $\delta$ = 7.42–7.50 (m, 2 H), 7.55–7.62 (m, 1 H), 7.69 (t, $J = 7.8$ Hz, 1 H), 8.10 (dt, $J = 7.7$, 1.2 Hz, 2 H), 8.23 (dd, $J = 8.3$, 1.1 Hz, 1 H), 8.46–8.50 (m, 1 H), 8.67 (t, $J = 1.4$ Hz, 1 H), 9.98 (s, 1 H).

(4-Formylphenyl)(phenyl)iodonium bromide: The title compound (0.89 g, 71%) was obtained as an off-white solid according to GP1 from 4-formylphenyl boronic acid (0.48 g, 3.2 mmol), BF$_3$·Et$_2$O (0.8 mL, 0.89 g, 6.32 mmol) and bis(acetoxy)iodobenzene (1.03 g, 3.2 mmol). $^1$H-NMR [(CD$_3$)$_2$SO, 300 MHz]: $\delta$ = 7.35–7.52 (m, 2 H), 7.55–7.65 (m, 1 H), 7.93 (d, $J = 8.3$ Hz, 2 H), 8.21 (d, $J = 7.4$ Hz, 2 H), 8.39 (d, $J = 8.3$ Hz, 2 H), 10.00 (s, 1 H); $^{13}$C-NMR [(CD$_3$)$_2$SO, 75.5 MHz]: $\delta$ = 120.77, 126.88, 131.99 (×2), 132.0, 135.5, 136.0, 137.9, 193.0. MS (ESI): positive mode m/z = 341.0 ([M + MeOH]+$^+$), 309.0 ([M + H]+$^+$); ESI HRMS: calcd for C$_{14}$H$_{14}$O$_2$I$: 341.0033; found: 341.0035; calcd for C$_{13}$H$_{10}$OI$: 308.9771; found: 308.9773.

(4-Formylphenyl)(phenyl)iodonium triflate: The title compound (0.58 g, 74%) was obtained as a beige solid according to GP2 from (4-formylphenyl)(phenyl)iodonium bromide (0.814 g, 2.09 mmol) and AgOTf (0.538 g, 2.09 mmol). $^1$H-NMR [(CD$_3$)$_2$SO, 300 MHz]: $\delta$ = 7.49–7.60 (m, 2 H), 7.63–7.75 (m, 1 H), 8.00 (d, $J = 8.3$ Hz, 2 H), 8.29 (d, $J = 7.4$ Hz, 2 H), 8.45 (d, $J = 8.3$ Hz, 2 H), 10.02 (s, 1 H); $^{13}$C-NMR [(CD$_3$)$_2$SO, 75.5 MHz]: $\delta$ = 116.6, 122.2, 131.9 (×2), 132.5, 135.5, 135.8, 137.9, 192.5. MS (ESI): positive mode m/z = 341.0 ([M + MeOH]+$^+$), 309.0 ([M + H]+$^+$); MS (ESI): negative mode m/z = 149.0 ([CF$_3$SO$_3$]$^-$); ESI HRMS: calcd for C$_{14}$H$_{14}$O$_2$I$: 341.0033; found: 341.0041; calcd for C$_{13}$H$_{10}$OI$: 308.9771; found: 308.9777.

4-Formyl-N,N,N-anilinium bicarbonate: The title compound was prepared according to GP2 from 4-formyl-N,N,N-anilinium iodide (0.24 g, 0.82 mmol) and Ag(PPh$_3$)$_2$HCO$_3$ (0.572 g, 0.82 mmol) in MeOH (12 mL). The supernatant was used directly for radiolabeling experiments.
4-Formyl-\(N,N,N\)-anilinium perchlorate: The title compound was prepared according to GP2 from 4-formyl-\(N,N,N\)-anilinium iodide (0.24 g, 0.82 mmol) and AgClO\(_4\) (0.170 g, 0.82 mmol) in MeOH (12 mL). The supernatant was directly used for radiolabeling experiments.

2-Formyl-\(N,N,N\)-anilinium iodide: \(^2\)NaI (0.23 g, 1.53 mol) was added to a stirred solution of 2-formyl-\(N,N,N\)-anilinium triflate\(^1\) (0.48 g, 1.53 mmol) in acetone (10 mL). After 10 min stirring the precipitate was filtered off, washed with acetone and dried to give the title compound (0.35 g, 78%) as a colorless solid. The spectral data were in accordance with those reported in the literature.\(^2\)

2-Formyl-\(N,N,N\)-anilinium perchlorate: LiClO\(_4\) (0.13 g, 1.22 mmol) was added to a stirred solution of 2-formyl-\(N,N,N\)-anilinium triflate\(^1\) (0.38 g, 1.22 mmol) in acetone (20 mL). After 15 min stirring Et\(_2\)O (5 mL) was added and the precipitate was filtered off, washed with acetone/Et\(_2\)O and dried to give the title compound (0.23 g, 72%) as a colorless solid. \(^1\)H-NMR [(CD\(_3\))\(_2\)SO, 300 MHz]: \(\delta = 3.76\) (s, 9 H), 7.87–8.03 (m, 2 H), 8.05–8.12 (m, 1 H), 8.27–8.40 (m, 1 H), 10.2 (s, 1 H); \(^{13}\)C-NMR [(CD\(_3\))\(_2\)SO, 75.5 MHz]: \(\delta = 56.7, 122.4, 129.5, 131.2, 135.8, 140.1, 144.2, 193.1\). MS (ESI): positive mode \(m/z = 196.1\) ([M + MeOH]\(^+\)), 164.1 ([M + H]\(^+\)); MS (ESI): negative mode \(m/z = 98.9\) ([ClO\(_4\)]\(^-\)); ESI HRMS: calcd for C\(_{11}\)H\(_{18}\)O\(_2\)N\(^+\): 196.1332.

2-Formyl-\(N,N,N\)-anilinium bicarbonate: The title compound was prepared according to GP2 from 2-formyl-\(N,N,N\)-anilinium iodide (0.12 g, 0.41 mmol) and Ag(PPh\(_3\))\(_2\)HCO\(_3\) (0.29 g, 0.42 mmol) in MeOH (6 mL). The supernatant was used directly for radiolabeling experiments.

2-Formyl-\(N,N,N\)-anilinium thiocyanate: NaCNS (52 mg, 0.64 mmol) was added to a stirred solution of 2-formyl-\(N,N,N\)-anilinium triflate\(^1\) (0.2 g, 0.64 mmol) in acetone (10 mL). After 15 min stirring the reaction mixture was concentrated under
reduced pressure. The residue was recrystallized from acetone/Et₂O to give the title compound (0.23 g, 72%) as a colorless solid. ¹H-NMR [(CD₃)₂SO, 300 MHz]: δ = 3.77 (s, 9 H), 7.81–7.95 (m, 2 H), 8.05–8.17 (m, 1 H), 8.32 (dd, J = 6.8, 1.5 Hz, 1 H), 10.2 (s, 1 H); ¹³C-NMR [(CD₃)₂SO, 75.5 MHz]: δ = 56.7, 122.5, 129.5, 131.2, 135.8, 140.2, 144.2, 193.1 (the signal of the thiocyanate carbon could not be observed). MS (ESI): positive mode m/z = 196.1 ([M + MeOH]+), 164.1 ([M + H]+); ESI HRMS: calcd for C₁₁H₁₈O₂N⁺: 196.1332; found: 196.1335; calcd for C₁₃H₁₀O₁⁻: 164.1070; found: 164.1076.

**(3-Formylphenyl)(4-methoxyphenyl)iodonium iodide:** Tos·H₂O (1.43 g, 7.50 mmol) was added to a solution of 3-iodobenzaldehyde (1.16 g, 5.0 mmol), 3-chloroperoxybenzoic acid (1.29 g, 7.5 mmol, 89% purity) and anisol (0.85 g, 0.86 mL, 7.91 mmol) in CH₂Cl₂/2,2,2-trifluoroethanol 1:1 (25 mL) and the mixture was stirred for 3 days. The resulting suspension was concentrated under reduced pressure and the residue was triturated with Et₂O. The crude product was recrystallized from CH₂Cl₂/Et₂O to give known (3-formylphenyl)(4-methoxyphenyl)iodonium tosylate⁹ (1.75 g, 69%) as a colorless solid. The latter was taken up in CH₂Cl₂ (40 mL) and the resulting solution was stirred vigorously with saturated NaI (4×20 mL). The precipitate was filtered off, washed with CH₂Cl₂ (30 mL), water (15 mL), acetone (15 mL), acetone/Et₂O 1:1 (100 mL) and dried to give the title compound as a yellow solid. The CH₂Cl₂ washings were dried and concentrated under reduced pressure. The residue was recrystallized from acetone/Et₂O to give a second crop of product (total yield 1.07 g, 46% in two steps). ¹H-NMR [(CD₃)₂SO, 300 MHz]: δ = 3.79 (s, 3 H), 7.02–7.08 (m, 2 H), 7.71 (t, J = 7.9 Hz, 1 H), 8.10–8.21 (m, 3 H), 8.44 (dt, J = 7.9, 1.2 Hz, 1 H), 8.65 (t, J = 1.2 Hz, 1 H), 9.99 (s, 1 H); ¹³C-NMR [(CD₃)₂SO, 75.5 MHz]: δ = 55.6, 106.6, 117.3, 118.8, 132.0, 132.5, 134.4, 137.1, 138.1, 139.7, 161.8, 191.4. MS (ESI): positive mode m/z = 371.0 ([M + MeOH]+), 339.0 ([M + H]+); MS (ESI): negative mode m/z = 126.9 ([I]⁻); ESI HRMS: calcd for C₁₄H₁₂O₂N⁺: 338.9876; found: 338.9878.
(3-Formylphenyl)(4-methoxyphenyl)iodonium perchlorate: The title compound was prepared according to GP2 from (3-formylphenyl)(4-methoxyphenyl)iodonium iodide (0.12 g, 0.257 mmol) and AgClO₄ (50 mg, 0.245 mmol) in MeOH (12 mL). The supernatant was used directly for radiolabeling experiments.

3-Formyl-N,N,N-anilinium perchlorate: The title compound was prepared according to GP2 from 3-formyl-N,N,N-anilinium iodide² (0.11 g, 0.38 mmol) and AgClO₄ (74 mg, 0.36 mmol) in MeOH (9 mL). The supernatant was used directly for radiolabeling experiments.

4-Fluorobenzoyl-βAla-Phe-OMe (1): DIEA (0.27 mL, 0.198 g, 1.53 mmol) was added to a stirred solution of HCl·H-βAla-Phe-OMe (0.22 g, 0.77 mmol) and 4-fluorobenzoyl chloride (90 μL, 0.12 g, 0.77 mmol) in CH₂Cl₂ (10 mL) and stirring was continued for 1 h. The mixture was concentrated under reduced pressure, the residue was taken up in EtOAc/H₂O (50 mL each), the organic layer was separated and washed with 1 N HCl (3×15 mL), H₂O (2×15 mL), 5% NaHCO₃ (3×15 mL), brine (2×15 mL), dried and concentrated under reduced pressure. The residue was recrystallized from EtOAc/hexane to give the title compound (0.26 g, 91%) as a colorless solid. Rᵣ= 0.55, acetone:hexane = 1:1. ¹H-NMR (CDCl₃, 600 MHz): δ = 2.49 (t, J = 5.6 Hz, 2 H), 3.05 (dd, J = 13.9, 6.6 Hz, 1 H), 3.15 (dd, J = 13.9, 5.6 Hz, 1 H) 3.59–3.67 (m, 1 H), 3.69–3.76 (m, 1 H), 3.72 (s, 3 H), 4.83–4.88 (m, 1 H), 6.29 (d, J = 7.7 Hz, 1 H), 7.06–7.11 (m, 4 H), 7.16–7.25 (m, 4 H), 7.75–7.80 (m, 2 H); ¹³C-NMR (CDCl₃, 150.9 MHz): δ = 35.3, 36.0, 37.7, 52.5, 53.3, 115.5 (d, J = 22.6 Hz), 127.3, 128.7, 129.1, 129.4 (d, J = 7.5 Hz), 130.5 (d, J = 3.0 Hz), 135.7, 165.1 (d, J = 365.2 Hz), 165.6, 171.7, 172.0. MS (ESI): positive mode \( m/z = 1057.4 (2 \text{ M} + \text{Na}^+) \), 1035.4 ([2 M + H]⁺), 540.2 ([M + Na]⁺), 518.2 ([M + H]⁺); MS (ESI): negative mode \( m/z = 630.2 ([M + 2 \text{HCOOH} - \text{H}]^-) \), 516.2 ([M − H]⁻); ESI HRMS: calcd for
C$_{29}$H$_{28}$FN$_{3}$O$_{5}$Na$^+$: 540.1905; found: 540.1907; calcd for C$_{29}$H$_{29}$FN$_{3}$O$_{5}$Na$^+$: 518.2086; found: 518.2085; calcd for C$_{29}$H$_{27}$FN$_{3}$O$_{5}$: 516.1940; found: 516.1933.

4-N,N-Dimethylaminobenzoyl-$\beta$-Ala-Phe-OMe: 4-N,N-Dimethylaminobenzoyl chloride (0.44 g, 2.42 mmol) was added to a stirred solution of HCl·H-$\beta$Ala-Phe-OMe (0.69 g, 2.42 mmol) and DIEA (0.843 mL, 0.63 g, 4.84 mmol) in CH$_2$Cl$_2$ (10 mL) and stirring was continued for a further hour. Thereafter, 4-N,N-dimethylaminobenzoyl chloride (0.15 g, 2.42 mmol) was added and the reaction mixture was stirred for a further 1 h. The mixture was concentrated under reduced pressure, the residue was taken up in EtOAc/5% NaHCO$_3$ (50 mL each), the organic layer was separated and washed with H$_2$O (2×15 mL), 5% NaHCO$_3$ (3×15 mL), brine (2×15 mL), dried and concentrated under reduced pressure. The residue was recrystallized from Et$_2$O/hexane to give the title compound (0.64 g, 67%) as a colorless solid. $R_f = 0.33$, acetone:hexane = 1:1. $^1$H-NMR (CDCl$_3$, 300 MHz): $\delta = 2.48$ (t, $J = 5.8$ Hz, 2 H), 3.03 (s, 6 H), 3.05 (dd, $J = 14.0$, 6.7 Hz, 1 H), 3.15 (dd, $J = 14.0$, 5.8 Hz, 1 H), 3.62–3.73 (m, 2 H), 3.71 (s, 3 H), 4.86 (q, $J = 7.3$ Hz, 1 H), 6.30 (d, $J = 7.7$ Hz, 1 H), 6.67 (d, $J = 8.9$ Hz, 2 H), 6.86–6.91 (br, 1 H), 7.05–7.10 (m, 2 H), 7.16–7.23 (m, 3 H), 7.66–7.70 (m, 2 H); $^{13}$C-NMR (CDCl$_3$, 75.5 MHz): $\delta = 35.7$, 36.8, 37.7 40.1, 52.3, 53.2, 111.1, 121.8, 127.1, 128.5, 128.6, 129.1; 135.7, 152.4, 167.4, 171.7, 171.9. MS (ESI): positive mode $m/z = 817.4$ ([2 M + Na$^+$]), 795.4 ([2 M + H$^+$]), 420.2 ([M + Na$^+$]), 398.2 ([M + H$^+$]); MS (ESI): negative mode $m/z = 396.2$ ([M – H$^-$]); ESI HRMS: calcd for C$_{22}$H$_{27}$N$_3$O$_4$Na$^+$: 420.1894; found: 420.1887; calcd for C$_{22}$H$_{28}$N$_3$O$_4$: 398.2074; found: 398.2072; calcd for C$_{22}$H$_{26}$N$_3$O$_4$–: 396.1929; found: 396.1919.

4-N,N,N-Trimethylammoniumbenzoyl-$\beta$-Ala-Phe-OMe iodide: Methyl iodide (0.28 mL, 0.64 g, 4.53 mmol) was added to a solution of 4-N,N-dimethylaminobenzoyl-$\beta$-Ala-Phe-OMe (0.6 g, 1.51 mmol) in acetone (10 mL) and the mixture was stirred under reflux for 16 h. Thereafter, the precipitate was
filtered off, washed with acetone (30 mL) and dried to give the title compound (0.63 g, 81%) as a colorless solid. $^1$H-NMR [(CD$_3$)$_2$SO, 300 MHz]: $\delta = 2.35–2.47$ (m, 2 H), 2.88 (dd, $J = 13.6$, 9.1 Hz, 1 H), 3.01 (dd, $J = 8.3$, 5.5 Hz, 1 H), 3.41 (q, $J = 6.7$ Hz, 2 H), 3.58 (s, 3 H), 3.64 (s, 9 H), 4.42–4.54 (m, 1 H), 7.12–7.27 (m, 5 H), 8.05 (q, $J = 10.2$ Hz, 4 H), 8.43 (d, $J = 7.9$ Hz, 1 H), 8.67 (d, $J = 5.5$ Hz, 1 H); $^{13}$C-NMR [(CD$_3$)$_2$SO, 75.5 MHz]: $\delta = 34.7$, 35.9, 36.6, 51.8, 53.5, 56.4, 120.7, 126.5, 128.2, 128.7, 128.9, 135.6, 137.2, 148.9, 164.4, 170.4, 172.0. MS (ESI): positive mode $m/z = 412.2$ ([M]$^+$); MS (ESI): negative mode $m/z = 538.1$ ([M + I–H]$^-$), 126.9 ([I]$^-^-$); ESI HRMS: calcd for C$_{23}$H$_{30}$N$_3$O$_4$: 412.2231; found: 412.2234; calcd for C$_{23}$H$_{30}$N$_3$O$_4$I: 538.1208; found: 538.1215.

4-$N$,$N$,$N$-Trimethylammoniumbenzoyl-$\beta$Ala-Phe-OMe triflate: The title compound (0.19 g, 95%) was obtained as a colorless solid according to GP2 from 4-$N$,$N$,$N$-trimethylammoniumbenzoyl-$\beta$Ala-Phe-OMe iodide (0.2 g, 0.37 mmol) and AgOTf (95 mg, 0.37 mmol). $^1$H-NMR [(CD$_3$)$_2$SO, 300 MHz]: $\delta = 2.31–2.47$ (m, 2 H), 2.82–2.94 (m, 1 H), 3.01–3.08 (m, 1 H), 3.35–3.47 (m, 2 H), 3.58 (s, 3 H), 3.63 (s, 9 H), 4.47–4.53 (m, 1 H), 7.16–7.27 (m, 5 H), 7.99–8.10 (m, 4 H), 8.43 (d, $J = 7.7$ Hz, 1 H), 8.67 (t, $J = 5.2$ Hz, 1 H); $^{19}$F-NMR [(CD$_3$)$_2$SO, 282.4 MHz]: $\delta = -77.74$; $^{13}$C-NMR [(CD$_3$)$_2$SO, 75.5 MHz]: $\delta = 34.7$, 36.0, 36.7, 51.8, 53.5, 56.4, 120.6, 126.5, 128.2, 128.7, 129.0, 135.7, 137.2, 149.0, 164.4, 170.4, 172.0.

4-$N$,$N$,$N$-Trimethylammoniumbenzoyl-$\beta$Ala-Phe-OMe bicarbonate: The title compound was prepared according to GP2 from 4-$N$,$N$,$N$-trimethylammoniumbenzoyl-$\beta$Ala-Phe-OMe iodide (0.12 g, 0.22 mmol) and Ag(PPh$_3$)$_2$HCO$_3$ (0.154 g, 0.22 mmol) in MeOH (10 mL). The supernatant was used directly for radiolabeling experiments.
**4-N,N,N-Trimethylammoniumbenzoyl-βAla-Phe-OMe perchlorate:** The title compound was prepared according to GP2 from 4-N,N,N-trimethylammoniumbenzoyl-βAla-Phe-OMe iodide (0.105 g, 0.19 mmol) and AgClO₄ (40 mg, 0.22 mmol) in MeOH (8.8 mL). The supernatant was used directly for radiolabeling experiments.

**4-(4-Methoxyphenyliodonium)benzoyl-βAla-Phe-OMe iodide:** TFA·H-βAla-Phe-OMe³ prepared from Boc-βAla-Phe-OMe (0.3 g, 0.86 mmol) was taken up in toluene (25 mL) and the mixture was concentrated under reduced pressure to remove the traces of TFA (×3). The residue was dissolved in DMF (10 mL) under Ar, HOBt (12 mg, 0.089 mmol) and (4-carboxyphenyl)(4-methoxyphenyl)iodonium tosylate (0.45 g, 0.85 mmol) were added and the heterogeneous mixture was cooled in an ice-water bath. DCC (0.176 g, 0.86 mmol) and thereafter 2,4,6-trimethylpyridine (0.23 mL, 0.21 g, 1.74 mmol) were added to the stirred reaction mixture. The cooling bath was removed and the mixture was stirred for a further 6 h. Afterwards, the reaction mixture was incubated at 4 °C for 16 h, DCU was filtered off and DMF was removed under reduced pressure. The residue was dissolved in CH₂Cl₂ (10 mL) and filtered. CH₂Cl₂ (20 mL) followed by saturated NaI (10 mL) were added and the mixture was stirred vigorously for 10 min. The resulting gel was centrifuged (4000 rpm, 15 min, 10 °C), the aqueous fraction was discarded, the pellet was resuspended in CH₂Cl₂ fraction, saturated NaI (10 mL) was added and the mixture was shaken vigorously for 10 min. The reaction mixture was centrifuged (4000 rpm, 15 min, 10 °C), the pellet was washed with CH₂Cl₂ (4×10 mL), H₂O (3×10 mL) and Et₂O (3×10 mL) (each time the pellet was suspended in the corresponding solvent, the suspension was centrifuged, the supernatant was discarded and the pellet was resuspended). Thereafter, the precipitate was triturated with acetone, filtered off and washed with acetone (30 mL) to give the title compound (0.31 g, 51%) as a colorless solid. ¹H-NMR [(CD₃)₂SO, 600 MHz]: δ=
2.36 (dq, $J = 18.0$, 7.4 Hz, 2 H), 2.87 (dd, $J = 13.8$, 9.3 Hz, 1 H), 3.00 (dd, $J = 13.8$, 5.6 Hz, 1 H), 3.36–3.47 (m, 2 H), 3.55 (s, 3 H), 3.78 (dd, $J = 13.8$, 9.3 Hz, 1 H), 3.00 (dd, $J = 13.8$, 5.6 Hz, 1 H), 3.36–3.47 (m, 2 H), 7.08–7.13 (m, 1 H), 7.15–7.20 (m, 4 H), 7.81 (d, $J = 8.4$ Hz, 2 H), 8.15 (d, $J = 9.2$ Hz, 2 H), 8.26 (d, $J = 8.4$ Hz, 2 H), 8.38 (d, $J = 7.7$ Hz, 1 H), 8.56 (t, $J = 5.4$ Hz, 1 H); $^{13}$C-NMR [(CD$_3$)$_2$SO, 150.9 MHz]: $\delta$ = 34.6, 35.9, 36.6, 51.8, 53.4, 55.7, 117.3, 121.0, 126.4, 128.1, 128.9, 129.8, 134.6, 137.1, 137.2, 150.9, 161.8, 164.9, 169.7, 170.3, 172.0. MS (ESI): positive mode $m/z = 587.1$ ([M]$^+$); MS (ESI): negative mode $m/z = 126.9$ ([I]$^-$); ESI HRMS: calcd for C$_{27}$H$_{28}$N$_2$O$_5$I$: 587.1037; found: 587.1038.

4-(4-Methoxyphenyliodonium)benzoyl-$\beta$Ala-Phe-OMe perchlorate: The title compound was prepared according to GP2 from 4-(4-methoxyphenyliodonium)benzoyl-$\beta$Ala-Phe-OMe iodide (0.126 g, 0.18 mmol) and AgClO$_4$ (36.6 mg, 0.18 mmol) in MeOH (10.5 mL). The supernatant was used directly for radiolabeling experiments.

2,3,5,6-Tetraphenyl 4-fluorobenzoate (2): Et$_3$N (0.54 mL, 0.39 g, 3.87 mmol) was added dropwise to a vigorously stirred solution of 4-fluorobenzoyl chloride (0.46 mL, 0.61 g, 3.87 mmol) and 2,3,5,6-tetrafluorophenol (0.64 g, 3.87 mmol) in Et$_2$O (30 mL) and the stirring was continued for a further 10 min. The reaction mixture was filtered, washed with H$_2$O (15 mL) and brine (2×10 mL), dried and concentrated under reduced pressure. The residue was recrystallized from hexane to give 4 (0.41 g) as a colorless solid. The mother liquor was concentrated under reduced pressure and the residue was recrystallized from hexane to give a second crop of 4 (0.45 g, total 77%). $R_f = 0.62$, EtOAc:hexane = 1:10. $^1$H-NMR (CDCl$_3$, 300 MHz): $\delta = 7.07$ (tt, $J = 9.9$, 7.1 Hz, 1 H), 7.21–7.30 (m, 2 H), 8.21–8.37 (m, 2 H); $^{19}$F-NMR (CDCl$_3$, 282.4 MHz): $\delta = -152.75$, −138.93, −102.24; $^{13}$C-NMR(CDCl$_3$, 75.5 MHz): $\delta = 103.4$ (t, $J = 21.9$ Hz), 116.2 (d, $J = 21.9$ Hz), 123.5 (d, $J = 1.5$ Hz), 512
133.5 (d, $J = 2.3$ Hz), 139.0–142.7 (m), 144.5–145.1 (m), 147.3–148.2 (m), 163.3 (d, $J = 259.7$ Hz), 168.5. MS (ESI): positive mode $m/z = 288.3$ ([M]$^+$). MS (EI, 70 eV): m/z (%): 165.0 [C$_6$HF$_4$O$^-$] (10), 123.0 [C$_6$H$_4$O$^-$] (100), 95.0 [C$_6$H$_4$F$^-$] (10).

2,3,5,6-Tetraphenyl 4-iodobenzoate: Et$_3$N (1.504 mL, 0.76 g, 7.51 mmol) was added dropwise to a vigorously stirred solution of 4-iodobenzoyl chloride (2 g, 7.51 mmol) and 2,3,5,6-tetrafluorophenol (1.25 g, 7.51 mmol) in Et$_2$O (60 mL) and the stirring was continued for a further 10 min. The reaction mixture was filtered, the filter cake was washed with Et$_2$O (30 mL) and the filtrate was concentrated under reduced pressure. The residue was dissolved Et$_2$O (10 mL) and filtered. The filtrate was concentrated under reduced pressure. The residue was recrystallized from Et$_2$O/hexane to give the title compound (1.38 g, 48%) as a colorless solid. $R_f = 0.46$, EtOAc:hexane = 1:10. $^1$H-NMR (CDCl$_3$, 300 MHz): $\delta = 7.07$ (tt, $J = 9.9$, 7.1 Hz, 1 H), 7.82–7.98 (m, 4 H); $^{19}$F-NMR (CDCl$_3$, 282.4 MHz): $\delta = -152.70, -138.80$; $^{13}$C-NMR (CDCl$_3$, 75.5 MHz): $\delta = 103.0, 103.4$ (t, $J = 23.0$ Hz), 126.6, 131.9, 138.3, 138.9–142.7 (m), 144.4 (dt, $J = 3.8$, 12.1 Hz), 147.7 (dt, $J = 4.5$, 12.1 Hz), 162.2. MS (ESI): positive mode $m/z = 397.3$ ([M + H]$^+$). MS (EI, 70 eV): m/z (%): 395.9 [M$^+$] (3), 230.9 [C$_7$H$_4$OI$^-$] (100), 202.9 [C$_6$H$_3$I$^-$] (100), 104.0 [C$_7$H$_4$O$^-$] (10).

(4-Methoxyphenyl)[4-(2,3,5,6-tetrafluorophenoxy carbonyl)phenyl]iodonium tosylate: Tos·H$_2$O (0.72 g, 3.79 mmol) was added to a solution of 2,3,5,6-tetraphenyl 4-iodobenzoate (1 g, 2.52 mmol), mCPBA [1.44 g, 85% purity, 7.09 mmol; commercially available 77% mCPBA (Aldrich) was dried at 2 mbar and 40 °C for 3 h before use] and anisole (0.51 mL, 0.51 g, 4.72 mmol) in 50% CF$_3$CH$_2$OH in CH$_2$Cl$_2$ (20 mL) and the mixture was stirred for 3 days. The reaction mixture was added to vigorously stirred Et$_2$O (450 mL) and stirring was continued for a further 45 min. The precipitate was filtered off and washed with Et$_2$O (100 mL),
redissolved in CH\textsubscript{2}Cl\textsubscript{2} (20 mL) and filtered through Celite\textsuperscript{®}. The filtrate was concentrated under reduced pressure. The residue was recrystallized from CH\textsubscript{2}Cl\textsubscript{2}/Et\textsubscript{2}O to give the title compound (1.53 g, 90\%) as a colorless solid. \textsuperscript{1}H-NMR (CDCl\textsubscript{3}, 300 MHz): \(\delta = 2.28 \text{ (s, 3 H)}, 3.77 \text{ (s, 3 H)}, 6.80 \text{ (d, } J = 9.0 \text{ Hz, 2 H}), 6.97 \text{ (d, } J = 6.0 \text{ Hz, 2 H}), 7.01–7.14 \text{ (m, 1 H)}, 7.36 \text{ (d, } J = 9.0 \text{ Hz, 2 H}), 7.98–8.03 \text{ (m, 4 H)}, 8.18 \text{ (d, } J = 9.0 \text{ Hz, 2 H}); \textsuperscript{19}F-NMR (CDCl\textsubscript{3}, 282.4 MHz): \(\delta = -152.70, -138.55\); \textsuperscript{13}C-NMR (CDCl\textsubscript{3}, 75.5 MHz): \(\delta = 21.2, 55.5, 103.7 \text{ (t, } J = 23.0 \text{ Hz)}, 104.7, 117.4, 123.0, 125.9, 128.5, 129.3, 132.7, 135.3, 137.9, 138.8–142.3 \text{ (m)}, 139.6, 142.2, 144.4 \text{ (dt, } J = 3.8, 15.9 \text{ Hz)}, 147.7 \text{ (dt, } J = 4.5, 16.6 \text{ Hz}), 161.3, 162.4. MS (ESI): positive mode \(m/z = 503.0 ([M]^+)\); MS (ESI): negative mode \(m/z = 171.0 ([C\textsubscript{7}H\textsubscript{7}SO\textsubscript{3}]^-)\); ESI HRMS: calcd for C\textsubscript{20}H\textsubscript{12}F\textsubscript{4}O\textsubscript{3}I\textsuperscript{+}: 502.9762; found: 502.9769.

(4-Methoxyphenyl)[4-(2,3,5,6-tetrafluorophenoxy carbonyl)phenyl]iodonium iodide:

(4-Methoxyphenyl)[4-(2,3,5,6-tetrafluorophenoxy carbonyl)phenyl]iodonium tosylate (1.19 g, 1.76 mmol) was dissolved in CH\textsubscript{2}Cl\textsubscript{2} (20 mL). After addition of saturated NaI (10 mL), the mixture was vigorously stirred for 15 min and centrifuged (4000 rpm, 15 °C, 10 min). The aqueous solution and precipitate were separated off, saturated NaI (10 mL) was added and the mixture was vigorously stirred for 15 min and centrifuged (×3). The organic fraction was filtered, dried and concentrated under reduced pressure. The residue was recrystallized from CH\textsubscript{2}Cl\textsubscript{2}/Et\textsubscript{2}O, the precipitate was filtered off, washed with acetone (10 mL) and Et\textsubscript{2}O (80 mL) to give the title compound (0.29 g, 26\%) as an off-white solid. The substance could be stored at 4 °C under Ar at least for 4 months. However, it was unstable in solution especially at elevated temperatures (dissolved in DMF or DMSO it was unstable already at ambient temperature). \textsuperscript{1}H-NMR (CDCl\textsubscript{3}, 300 MHz): \(\delta = 3.79 \text{ (s, 3 H)}, 6.65–6.73 \text{ (m, 2 H)}, 6.95–7.10 \text{ (m, 1 H)}, 7.56 \text{ (d, } J = 8.9 \text{ Hz, 2 H}), 7.81–8.05 \text{ (m, 3 H)}, 8.21–8.35 \text{ (m, 1 H}); \textsuperscript{19}F-NMR (CDCl\textsubscript{3}, 282.4 MHz): \(\delta = -152.63, -138.72\); \textsuperscript{13}C-NMR (CDCl\textsubscript{3}, 75.5 MHz): \(\delta = 55.5, 82.3, 103.0, 103.5 \text{ (t, } J = 22.6 \text{ Hz)}, 116.4, 126.6, 128.8, 131.9,
138.3, 138.6–142.5 (m), 144.2–144.7 (m), 144.5–144.9 (m), 159.5, 162.2. MS (ESI): positive mode 
\[m/z = 502.9 \ ([M]^+); \] MS (ESI): negative mode \[m/z = 126.9 \ ([I]^-); \] ESI HRMS: calcd for C_{20}H_{12}F_{4}O_{3}I^+: 502.9762; found: 502.9741.

(4 - Methoxyphenyl)[4 - (2,3,5,6 - tetrafluorophenoxyacarbonyl)phenyl]iodonium perchlorate:

The title compound (168 mg, 88%) was obtained as a colorless solid according to GP2 from (4-methoxyphenyl)[4-(2,3,5,6-tetrafluorophenoxyacarbonyl)phenyl]iodonium iodide (0.2 g, 0.32 mmol) and AgClO₄ (66 mg, 0.32 mmol) in acetone (10 mL). ^1H-NMR (CDCl₃, 300 MHz): \(\delta = 3.80 \) (s, 3 H), 6.88–6.98 (m, 2 H), 7.06 (tt, \(J = 9.9, 7.1 \) Hz, 1 H), 8.04–8.13 (m, 2 H), 8.14–8.18 (m, 2 H), 8.21–8.26 (m, 2 H); ^19F-NMR (CDCl₃, 282.4 MHz): \(\delta = -152.52, -138.48; \) ^13C-NMR (CDCl₃, 75.5 MHz): \(\delta = 55.5, 101.1, 103.8 \) (t, \(J = 21.1 \) Hz), 118.4, 120.1, 130.6, 133.5, 135.1, 138.3, 138.7–139.3 (m), 142.1–144.4 (m), 147.5–147.7 (m), 161.0, 163.4. MS (ESI): positive mode 
\[m/z = 503.0 \ ([M]^+); \] ESI HRMS: calcd for C_{20}H_{12}F_{4}O_{3}I^+: 502.9762; found: 502.9749. MS (ESI): positive mode \[m/z = 503.0 \ ([M]^+); \] MS (ESI): negative mode \[m/z = 171.0 \ ([C_{7}H_{7}SO_{3}]^-); \] ESI HRMS: calcd for C_{20}H_{12}F_{4}O_{3}I^+: 502.9762; found: 502.9749.

1-O-Methyl-2,3-O-isopropylidene-5-O-[5-(N,N-dimethylamino)naphthalene-1-sulfonyl]-D-ribofuranoside (8): Dansyl chloride (2.9 g, 10.75 mmol) was added to a stirred ice-cold solution of 1-O-methyl-2,3-O-isopropylidene-D-ribofuranoside (9) (1.7 g, 8.32 mmol) and Et₃N (1.6 g, 15.81 mmol) in CH₂Cl₂ (25 mL), the cooling bath was removed and the reaction mixture was stirred for further 16 h. The mixture was concentrated under reduced pressure. Et₂O and H₂O (100 mL each) were added to the residue, the organic fraction was separated and washed with H₂O (3×20 mL), 5% NaHCO₃ (3×20 mL) and brine (2×20 mL), then dried and concentrated under reduced pressure. The residue was purified by column chromatography [EtOAc/hexane = 1:4, silica gel (0.1% CaO)] and
recrystallization from Et₂O/pentane gave 10 (2.85 g, 78%) as a yellow solid. \( R_f = 0.11 \), EtOAc:hexane = 1:8. \(^1\)H-NMR (CDCl₃, 300 MHz): \( \delta = 1.24 \) (s, 3 H), 1.42 (s, 3 H), 2.90 (s, 6 H), 3.14 (s, 3 H), 3.93–4.0 (m, 2 H), 4.32 (t, \( J = 7.2 \) Hz, 1 H), 4.43–4.56 (m, 2 H), 4.88 (s, 1 H), 7.18–7.27 (m, 1 H), 7.53–7.65 (m, 2 H), 8.25–8.33 (m, 2 H), 8.63 (d, \( J = 8.5 \) Hz, 1 H); \(^{13}\)C-NMR (CDCl₃, 75.5 MHz): \( \delta = 24.8, 26.3, 45.4, 54.9, 69.5, 81.4, 83.5, 84.8, 109.4, 112.6, 115.7, 119.4, 123.0, 128.8, 129.8, 129.9, 130.6, 130.9, 131.7 \). MS (ESI): positive mode \( m/z = 897.3 \) ([2 M + Na]⁺), 875.3 ([2 M + H]⁺), 460.2 ([M + Na]⁺), 438.2 ([M + H]⁺); ESI HRMS: calcd for C_{22}H_{27}N_{3}O_{4}Na⁺: 420.1894; found: 420.1887; calcd for C_{21}H_{26}NO_{7}Na⁺: 460.1400; found: 460.1394; calcd for C_{21}H_{26}NO_{7}⁺: 438.1581; found: 438.1579.

1-\( O\)-Methyl-2,3-\( O\)-isopropylidene-5-\( O\)-[5-(N,N,N-trimethylammonium)naphthalene-1- sulfonyl]-\( \beta\)-ribofuranoside triflate (9): MeOTf (0.251 mL, 0.376 g, 2.29 mmol) was added to a solution 10 (1 g, 2.29 mmol) in CH₂Cl₂ (5 mL) in a dry box under Ar. The reaction flask was then removed from the dry box and the reaction mixture was incubated at ambient temperature for 3 days. Thereafter, Et₂O (50 mL) was added and the precipitated oil was separated, recrystallized twice from CH₂Cl₂/Et₂O, sonicated with Et₂O (3×20 mL) and thoroughly dried under reduced pressure to give 11 (1.05 g, 69%) as a colorless foam. \(^1\)H-NMR (CDCl₃, 300 MHz): \( \delta = 1.26 \) (s, 3 H), 1.41 (s, 3 H), 3.17 (s, 3 H), 3.95–4.15 (m, 2 H), 4.08 (s, 9 H), 4.30 (dt, \( J = 0.6, 14.1 \) Hz, 1 H), 4.50–4.54 (m, 1 H), 4.58–4.62 (m, 1 H), 4.90 (s, 1 H), 7.83 (t, \( J = 8.3 \) Hz, 1 H), 8.0 (dd, \( J = 8.9, 7.6 \) Hz, 1 H), 8.21 (d, \( J = 8.3 \) Hz, 1 H), 8.47 (d, \( J = 8.9 \) Hz, 1 H), 8.89–8.97 (m, 2 H); \(^{13}\)C-NMR (CDCl₃, 75.5 MHz): \( \delta = 24.8, 26.3, 55.1, 58.7, 70.5, 81.2, 83.5, 84.7, 109.5, 112.8, 120.6 \) (q, \( J = 320.1 \) Hz), 122.7, 125.2, 127.2, 127.6, 129.4, 131.16, 131.22, 133.9, 142.1 \). MS (ESI): positive mode \( m/z = 452.3 \) ([M⁺]); MS (ESI): negative mode \( m/z = 149.0 \) ([CF₃SO₃]⁻); ESI HRMS: calcd for C_{22}H_{30}NO_{7}S⁻: 452.1737; found: 452.1739.
Radiochemistry

Synthesis of $^{18}$F-fluorobenzaldehydes from iodonium precursors alone without any evaporation steps. General procedure 3 – (GP3): $^{18}$F-Fluoride (100–330 MBq) was fixed on a QMA cartridge. The cartridge was washed with the appropriate solvent (1 mL) and the radiofluoride was eluted with a solution of the corresponding iodonium salt (5 mg, unless not otherwise stated) in the appropriate solvent (0.5 mL). The elution yield was determined by comparing the radioactivity eluted into the reaction vial with the total radioactivity trapped on the anion exchange resin. The reaction mixture was stirred at the temperature and for the indicated time in Table S1. Subsequently the mixture was cooled down to room temperature, water (4 mL) was added and the reaction mixture was shaken vigorously for 30 s. Thereafter, RCC was determined by radio-HPLC.

Unless stated, HPLC conditions: Eluent: 30% MeCN, flow rate: 1.5 mL/min.
Figure S1. False high vs. correct radiochemical conversions obtained by the direct HPLC analysis of reaction mixtures and the HPLC analysis of reaction mixtures diluted with an excess of water: A – 4-[\(^{18}\)F]FBA (from 4-formyl-N,N,N-trimethylanilinium triflate, K2.2.2/K\(_2\)CO\(_3\) protocol, GP5), B – 2-[\(^{18}\)F]FBA (from only 2-formyl-N,N,N-trimethylanilinium perchlorate, GP4), C – 3-[\(^{18}\)F]FBA (from (3-formylphenyl)(phenyl)iodonium bromide, CsHCO\(_3\)/TEMPO protocol, GP6).
Table S1. Synthesis of $[^{18}\text{F}]$fluorobenzaldehydes from iodonium precursors alone without any evaporation steps\textsuperscript{[a]}

\[ \text{O} \begin{array}{c} X^- \text{Ph} \end{array} \xrightarrow{1)} \text{elution of} \ 18\text{F}^- \xrightarrow{2)} \text{solvent, } \Delta, t \ \text{H} \begin{array}{c} X^- = \text{Br}^- , \text{TfO}^- \end{array} \xrightarrow{18\text{F}} \text{Ph} \]

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\textsuperscript{[a]} All experiments were carried out as described in GP3; \textsuperscript{[b]} with 20 mg precursor

Elution of $^{18}\text{F}^-$ from a QMA cartridge with onium salts in anhydrous alcohols (Table S2):

$[^{18}\text{F}]$Fluoride (50–200 MBq) was fixed on a QMA cartridge. The cartridge was washed with the appropriate alcohol (1 mL), and the radiofluoride was eluted with a solution of the corresponding onium salt (5 mg) in the appropriate alcohol (0.5 mL). The elution yield was determined by comparing the radioactivity eluted into the reaction vial with the total radioactivity trapped on the anion exchange resin.
Table S2. Recovery yield for the elution of $^{18}\text{F}^-$ from a QMA cartridge with onium salts in anhydrous alcohols.

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**Elution of $^{18}\text{F}^-$ from a QMA cartridge with different amounts of onium salts (Table S3):**

$^{[18}\text{F}]$Fluoride (50–500 MBq) was fixed on a QMA cartridge. The cartridge was washed with the appropriate alcohol (1 mL) and the radiofluoride was eluted with a solution of the corresponding onium salt (see Table S3) in MeOH (0.5 mL). The elution yield was determined by comparing the radioactivity eluted into the reaction vial with the total radioactivity trapped on the anion exchange resin.
Synthesis of $^{18}$F-labeled compounds from only onium precursors. General procedure 4 – (GP4): Aqueous $[^{18}$F$]$fluoride (0.05–50 GBq) was trapped on an anion-exchange resin (QMA or Chromafix® 30-PS-HCO$_3$ cartridge). It should be noted, that in the case of QMA cartridges, the aqueous $[^{18}$F$]$fluoride was loaded onto the cartridge from the male side, whereas MeOH flushing and $^{18}$F$^-$ elution were done from the female side of the cartridge. If the QMA has been loaded, flushed and eluted from the female side only, sometimes a significant amount of $[^{18}$F$]$fluoride remained on the resin (this is probably because QMA-light (46 mg) cartridges have a single frit on the male side but four on the female side).

Unless otherwise stated the cartridge was washed with MeOH (1 mL) and $[^{18}$F$]$fluoride was eluted into a reaction vial with a solution of the appropriate precursor (5 mg; unless otherwise stated) in MeOH (0.5 mL). Methanol was evaporated under reduced pressure at 70 °C within 2–3 min. After cooling to room temperature the appropriate solvent (500 µL) was added. The reaction mixture was

Table S3. Elution of $^{18}$F$^-$ from a QMA cartridge with different amounts of onium salts.

<table>
<thead>
<tr>
<th>entry</th>
<th>precursor amount [mg]</th>
<th>$\text{Me}_3\text{N}^+$ recovery [%]</th>
<th>$\text{ClO}_4^-$ recovery [%]</th>
<th>$\text{Ph}^+$ recovery [%]</th>
<th>$\text{Ph}^+$ recovery [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>10</td>
<td>99</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>8</td>
<td>-</td>
<td>98</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>5</td>
<td>99</td>
<td>-</td>
<td>99</td>
<td>98</td>
</tr>
<tr>
<td>4</td>
<td>2.5</td>
<td>99</td>
<td>-</td>
<td>97</td>
<td>92</td>
</tr>
<tr>
<td>5</td>
<td>1</td>
<td>-</td>
<td>96</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>6</td>
<td>0.7</td>
<td>-</td>
<td>89</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>7</td>
<td>0.5</td>
<td>97</td>
<td>78</td>
<td>80</td>
<td>84</td>
</tr>
<tr>
<td>8</td>
<td>0.3</td>
<td>-</td>
<td>-</td>
<td>68</td>
<td>82</td>
</tr>
<tr>
<td>9</td>
<td>0.1</td>
<td>85</td>
<td>44</td>
<td>65</td>
<td>76</td>
</tr>
</tbody>
</table>
stirred under the conditions given in Tables S4–S10. Subsequently the mixture was cooled down to room temperature, water (4 mL) was added and the reaction mixture was shaken vigorously for 30 s. Thereafter, RCC was determined by radio-HPLC.

Unless stated, HPLC conditions: Eluent: 30% MeCN, flow rate: 1.5 mL/min.

**Preparation of ¹⁸F-labeled compounds using K2.2.2/K₂CO₃ protocol. General procedure 5 – (GP5):** [¹⁸F]Fluoride (0.05–50 GBq) was fixed on a QMA cartridge and eluted with 0.066 M K₂CO₃ (360 μL) into a solution of K2.2.2 (20 mg, 53.1 μmol) in MeCN (700 μL). After evaporation, the corresponding onium precursor (5 mg) dissolved in DMSO (500 μL) was added to the dry cryptate ([K⊂2.2.2]⁺/[¹⁸F]⁻) and the reaction mixture was stirred at the temperature and for the indicated time (Tables S4–5, 8). The mixture was cooled down to room temperature, water (4 mL) was added and the reaction mixture was shaken vigorously for 30 s. Thereafter, RCC was determined by radio-HPLC.

Unless otherwise stated, HPLC conditions: Eluent: 30% MeCN, flow rate: 1.5 mL/min.

**Synthesis of ¹⁸F-labeled compounds using CsHCO₃/TEMPO protocol. General procedure 6 – (GP6):** [¹⁸F]Fluoride (0.05–50 GBq) was fixed on an anion-exchange resin and eluted with an aqueous solution of CsHCO₃ (400 μL; 2.5 mg/mL) into the reaction vial containing MeCN (1 mL). After azeotropic drying under helium at 110 °C (×3), a solution of the appropriate iodonium salt (1–2 mg) and 2,2,6,6-tetramethylpiperidine-N-oxyl (TEMPO) (0.5–1 mg) in DMF (1 mL) was added and the reaction mixture was stirred at the temperature and for the indicated time (Tables S6). The mixture was cooled down to room temperature, water (4 mL) was added and the reaction mixture was shaken vigorously for 30 s. Thereafter, RCC was determined by radio-HPLC.

Unless otherwise stated, HPLC conditions: Eluent: 30% MeCN, flow rate: 1.5 mL/min.

**Preparative synthesis of 2-/4-[¹⁸F]fluorobenzaldehydes (2-/4-[¹⁸F]FBA):** The ¹⁸F-labeled compounds were prepared according to GP4. After cooling to ambient temperature, the reaction
mixture was diluted with water (9 mL) and loaded onto a Strata X cartridge. The cartridge was washed with 0.1 M HCl (10 mL) and H2O (5 mL) and the corresponding [18F]fluorobenzaldehyde (up to 35 GBq, 65–75% EOB; not decay corrected) was eluted with EtOH (0.3 mL). The radiochemical and chemical purities after solid phase extraction (SPE) purification were > 99%.
Table S4. Synthesis of 2-[\(^{18}\text{F}\)]FBA.\(^{[a]}\)

![Diagram of synthesis process]

\[ \text{LG}^+ = \text{Me}_3\text{N}^+, \text{PhI}^+ \\
\text{X}^- = \text{I}^-, \text{Br}^-, \text{ClO}_4^-, \text{TFO}^-, \text{SCN}^-, \text{HCO}_3^- \]

<table>
<thead>
<tr>
<th>entry</th>
<th>LG(^+)X(^-)</th>
<th>solvent</th>
<th>temperature [^{[\circ\text{C}}], time [min]</th>
<th>RCC [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Me(_3)N(^+)HCO(_3^-)</td>
<td>DMF</td>
<td>80, 10</td>
<td>58</td>
</tr>
<tr>
<td>2</td>
<td>Me(_3)N(^+)HCO(_3^-)</td>
<td>MeCN</td>
<td>80, 10</td>
<td>68</td>
</tr>
<tr>
<td>3</td>
<td>Me(_3)N(^+)I(^-)</td>
<td>DMSO</td>
<td>80, 10</td>
<td>62(62)(^c)</td>
</tr>
<tr>
<td>4</td>
<td>Me(_3)N(^+)I(^-)</td>
<td>DMSO</td>
<td>130, 10</td>
<td>63</td>
</tr>
<tr>
<td>5</td>
<td>Me(_3)N(^+)I(^-)</td>
<td>MeCN</td>
<td>130, 10</td>
<td>24</td>
</tr>
<tr>
<td>6</td>
<td>Me(_3)N(^+)SCN(^-)</td>
<td>DMSO</td>
<td>80, 10</td>
<td>26</td>
</tr>
<tr>
<td>7</td>
<td>Me(_3)N(^+)SCN(^-)</td>
<td>DMSO</td>
<td>130, 10</td>
<td>56</td>
</tr>
<tr>
<td>8</td>
<td>Me(_3)N(^+)SCN(^-)</td>
<td>MeCN</td>
<td>130, 10</td>
<td>46</td>
</tr>
<tr>
<td>9</td>
<td>PhI(^+)TFO(^-)</td>
<td>DMF</td>
<td>160, 15</td>
<td>35</td>
</tr>
<tr>
<td>10</td>
<td>Me(_3)N(^+)TFO(^-)</td>
<td>DMSO</td>
<td>130, 15</td>
<td>64(46)(^c)</td>
</tr>
<tr>
<td>11</td>
<td>PhI(^+)Br(^-)</td>
<td>DMSO</td>
<td>80, 2</td>
<td>10</td>
</tr>
<tr>
<td>12</td>
<td>PhI(^+)Br(^-)</td>
<td>DMSO</td>
<td>160, 15</td>
<td>61</td>
</tr>
<tr>
<td>13</td>
<td>PhI(^+)Br(^-)</td>
<td>DMF</td>
<td>160, 10</td>
<td>5</td>
</tr>
<tr>
<td>14</td>
<td>Me(_3)N(^+)ClO(_4^-)</td>
<td>DMSO</td>
<td>80, 15</td>
<td>63</td>
</tr>
<tr>
<td>15</td>
<td>Me(_3)N(^+)ClO(_4^-)</td>
<td>DMSO</td>
<td>150, 10</td>
<td>80(30)(^c)</td>
</tr>
<tr>
<td>16</td>
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<td>DMSO</td>
<td>200, 10</td>
<td>65</td>
</tr>
<tr>
<td>17</td>
<td>Me(_3)N(^+)ClO(_4^-)</td>
<td>MeCN</td>
<td>80, 10</td>
<td>37</td>
</tr>
<tr>
<td>18(^{[b]})</td>
<td>Me(_3)N(^+)TFO(^-)</td>
<td>DMSO</td>
<td>80, 2</td>
<td>46</td>
</tr>
</tbody>
</table>

\(^{[a]}\) Unless otherwise stated, all experiments were carried out as described in GP4; \(^{[b]}\) carried out according to the K2.2.2/K\(_2\)CO\(_3\) protocol (GP5); \(^{[c]}\) RCC with 2 mg precursor in 100 µL solvent.
Table S5. Preparation of 4-[^18F]FBA[^a]

![Chemical diagram](https://example.com/chemical_diagram)

LG\(^+\) = Me\(_3\)N\(^+\), PhI\(^+\)
X\(^-\) = I\(^-\), Br\(^-\), ClO\(_4\)^-, TfO\(^-\), HCO\(_3\)^-

<table>
<thead>
<tr>
<th>entry</th>
<th>LG(^+)X(^-)</th>
<th>solvent</th>
<th>temperature [°C], time [min]</th>
<th>RCC [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Me(_3)N(^+)HCO(_3)^-</td>
<td>DMF</td>
<td>80, 10</td>
<td>28</td>
</tr>
<tr>
<td>2</td>
<td>Me(_3)N(^+)HCO(_3)^-</td>
<td>MeCN</td>
<td>130, 10</td>
<td>45</td>
</tr>
<tr>
<td>3</td>
<td>Me(_3)N(^+)HCO(_3)^-</td>
<td>DMSO</td>
<td>80, 10</td>
<td>87(40)c</td>
</tr>
<tr>
<td>4</td>
<td>Me(_3)N(^+)HCO(_3)^-</td>
<td>sulfolane</td>
<td>200, 10</td>
<td>80</td>
</tr>
<tr>
<td>5</td>
<td>Me(_3)N(^+)I(^-)</td>
<td>DMSO</td>
<td>80, 10</td>
<td>75(65)c</td>
</tr>
<tr>
<td>6</td>
<td>PhI(^+)TfO(^-)</td>
<td>DMF</td>
<td>160, 15</td>
<td>50</td>
</tr>
<tr>
<td>7</td>
<td>PhI(^+)Br(^-)</td>
<td>DMF</td>
<td>115, 5</td>
<td>61</td>
</tr>
<tr>
<td>8</td>
<td>Me(_3)N(^+)ClO(_4)^-</td>
<td>DMSO</td>
<td>150, 10</td>
<td>90(33)c</td>
</tr>
<tr>
<td>9</td>
<td>Me(_3)N(^+)ClO(_4)^-</td>
<td>sulfolane</td>
<td>200, 10</td>
<td>89</td>
</tr>
<tr>
<td>10</td>
<td>Me(_3)N(^+)TfO(^-)</td>
<td>DMF</td>
<td>115, 10</td>
<td>63</td>
</tr>
<tr>
<td>11[c]</td>
<td>Me(_3)N(^+)TfO(^-)</td>
<td>DMSO</td>
<td>80, 10</td>
<td>65</td>
</tr>
</tbody>
</table>

[^a]: Unless otherwise stated, all experiments were carried out as described in GP4; [b] carried out according to the K2.2.2/K\(_2\)CO\(_3\) protocol (GP5); [c] RCC with 2 mg precursor in 100 µL solvent.
Table S6. Preparation of 3-[^18F]FBA[^a].

![Diagram of the reaction](image)

**LG** = Me₃N⁺, PhI⁺, (4-OMePh)I⁺

X⁻ = I⁻, Br⁻, ClO₄⁻, TfO⁻, HCO₃⁻

<table>
<thead>
<tr>
<th>entry</th>
<th>LG⁺X⁻</th>
<th>solvent</th>
<th>temperature [°C], time [min]</th>
<th>RCC [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>PhI⁺I⁻</td>
<td>95% DMSO</td>
<td>130, 10</td>
<td>14</td>
</tr>
<tr>
<td>2</td>
<td>PhI⁺I⁻</td>
<td>95% DMF</td>
<td>130, 10</td>
<td>12</td>
</tr>
<tr>
<td>3</td>
<td>PhI⁺I⁻</td>
<td>95% MeCN</td>
<td>130, 10</td>
<td>5</td>
</tr>
<tr>
<td>4</td>
<td>(4-OMePh)I⁺I⁻</td>
<td>DMSO</td>
<td>80, 10</td>
<td>12</td>
</tr>
<tr>
<td>5</td>
<td>(4-OMePh)I⁺I⁻</td>
<td>DMSO</td>
<td>130, 10</td>
<td>31</td>
</tr>
<tr>
<td>6</td>
<td>PhI⁺Br⁻</td>
<td>DMSO</td>
<td>80, 10</td>
<td>0</td>
</tr>
<tr>
<td>7</td>
<td>Me₃N⁺HCO₃⁻</td>
<td>DMSO</td>
<td>80, 10</td>
<td>0</td>
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<tr>
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<td>Me₃N⁺I⁻</td>
<td>DMSO</td>
<td>130, 10</td>
<td>0</td>
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<tr>
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<td>200, 10</td>
<td>6</td>
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<tr>
<td>10</td>
<td>PhI⁺TfO⁻</td>
<td>DMF</td>
<td>160, 15</td>
<td>10</td>
</tr>
<tr>
<td>11</td>
<td>(4-OMePh)I⁺ClO₄⁻</td>
<td>DMSO</td>
<td>80, 10</td>
<td>22</td>
</tr>
<tr>
<td>12</td>
<td>(4-OMePh)I⁺ClO₄⁻</td>
<td>DMSO</td>
<td>150, 10</td>
<td>40</td>
</tr>
<tr>
<td>13</td>
<td>(4-OMePh)I⁺ClO₄⁻</td>
<td>95% MeCN</td>
<td>80, 10</td>
<td>2</td>
</tr>
<tr>
<td>14</td>
<td>(4-OMePh)I⁺ClO₄⁻</td>
<td>95% DMSO</td>
<td>80, 10</td>
<td>6</td>
</tr>
<tr>
<td>15[^b]</td>
<td>PhI⁺Br⁻</td>
<td>DMF</td>
<td>130, 10</td>
<td>45</td>
</tr>
</tbody>
</table>

[^a] Unless otherwise stated, all experiments were carried out as described in GP4;[^b] carried out according to the TEMPO/CsHCO₃ protocol (GP6).
Table S7. Synthesis of dipeptide $[^{18}\text{F}]\text{I}$.\footnote{a}

\[ \text{LG}^+ = \text{Me}_3\text{N}^+, (4\text{-OMePh})\text{I}^+ \]
\[ X^- = \Gamma, \text{ClO}_4^-, \text{HCO}_3^- \]

<table>
<thead>
<tr>
<th>entry</th>
<th>LG$^+$X$^-$</th>
<th>solvent</th>
<th>temperature [$^\circ\text{C}$], time [min]</th>
<th>RCC [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Me$_3$N$^+$HCO$_3^-$</td>
<td>sulfolane</td>
<td>130, 15</td>
<td>30</td>
</tr>
<tr>
<td>2</td>
<td>Me$_3$N$^+$HCO$_3^-$</td>
<td>sulfolane</td>
<td>200, 20</td>
<td>31</td>
</tr>
<tr>
<td>3</td>
<td>Me$_3$N$^+$HCO$_3^-$</td>
<td>diglyme</td>
<td>200, 15</td>
<td>4</td>
</tr>
<tr>
<td>4</td>
<td>Me$_3$N$^+$HCO$_3^-$</td>
<td>DMSO</td>
<td>130, 15</td>
<td>29</td>
</tr>
<tr>
<td>5</td>
<td>Me$_3$N$^+$ClO$_4^-$</td>
<td>DMSO</td>
<td>140, 15</td>
<td>12</td>
</tr>
<tr>
<td>6</td>
<td>(4-OMePh)I$^+$I$^-$</td>
<td>DMSO</td>
<td>130, 10</td>
<td>56</td>
</tr>
<tr>
<td>7</td>
<td>(4-OMePh)I$^+$ClO$_4^-$</td>
<td>DMSO</td>
<td>140, 15</td>
<td>12</td>
</tr>
<tr>
<td>8[b]</td>
<td>(4-OMePh)I$^+$I$^-$</td>
<td>DMF</td>
<td>120, 10</td>
<td>56</td>
</tr>
<tr>
<td>9[b]</td>
<td>(4-OMePh)I$^+$I$^-$</td>
<td>diglyme</td>
<td>120, 10</td>
<td>16</td>
</tr>
<tr>
<td>10[c]</td>
<td>Me$_3$N$^+$I$^-$</td>
<td>DMSO</td>
<td>90, 10</td>
<td>0</td>
</tr>
<tr>
<td>11[c]</td>
<td>Me$_3$N$^+$I$^-$</td>
<td>sulfolane</td>
<td>150, 10</td>
<td>0</td>
</tr>
<tr>
<td>12[c]</td>
<td>Me$_3$N$^+$TfO$^-$</td>
<td>DMSO</td>
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<tr>
<td>13[c]</td>
<td>Me$_3$N$^+$TfO$^-$</td>
<td>sulfolane</td>
<td>150, 10</td>
<td>0</td>
</tr>
</tbody>
</table>

\footnote{a} Unless otherwise stated, all experiments were carried out as described in GP4; \footnote{b} carried out according to the K2.2.2/K$_2$CO$_3$ protocol (GP5); \footnote{c} carried out according to the TEMPO/CsHCO$_3$ protocol (GP6).
Table S8. Synthesis of 2,3,4,6-tetrafluoro 4-[\(^{18}\text{F}\)]fluorobenzoate (4-[\(^{18}\text{F}\)]TFP, \[^{18}\text{F}\]2).\[^{[a]}\]

\[
\begin{array}{cccc}
\text{entry} & \text{X}^- & \text{solvent} & \text{temperature [°C], time [min]} & \text{RCC [%]} \\
1^{[b]} & \text{I}^- & \text{DMSO} & 130, 10 & 10–25 \\
2 & \text{ClO}_4^- & \text{DMSO} & 130, 10 & 23–25 \\
3 & \text{TosO}^- & \text{DMSO} & 130, 10 & 11 \\
\end{array}
\]

[a] All experiments were carried out as described in GP4; [b] large fluctuations of RCCs were observed. HPLC conditions: Eluent: 50% MeCN, flow rate: 3 mL/min
Table S10. Synthesis of 1-[\(^{18}\)F]fluoro-4-iodobenzene ([\(^{18}\)F]FIB, [\(^{18}\)F]4).[\(^{a}\)]

1) elution of \(^{18}\)F-
2) evaporation of MeOH
3) solvent, \(\Delta\), t

<table>
<thead>
<tr>
<th>entry</th>
<th>solvent</th>
<th>temperature (^{\circ})C, time ([\text{min}])</th>
<th>RCC [%]</th>
</tr>
</thead>
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<td>2</td>
<td>MeCN</td>
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<td>DMSO</td>
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<td>16</td>
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<tr>
<td>4</td>
<td>sulfolane</td>
<td>85, 15</td>
<td>41</td>
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<td>5</td>
<td>DMF</td>
<td>85, 15</td>
<td>29</td>
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<tr>
<td>6(^{[b]})</td>
<td>MeCN</td>
<td>85, 15</td>
<td>50</td>
</tr>
<tr>
<td>7(^{[b]})</td>
<td>diglyme</td>
<td>85, 15</td>
<td>42</td>
</tr>
</tbody>
</table>

\(^{[a]}\) All experiments were carried out as described in GP4. \(^{[b]}\) carried out according to the K2.2.2/K\(_2\)CO\(_3\) protocol (GP5). HPLC conditions: Eluent: 50% MeCN, flow rate: 1.5 mL/min.

**Preparation of 5-[\(^{18}\)F]fluoro-d-ribose ([\(^{18}\)F]FDR, [\(^{18}\)F]4):** The protected intermediate of [\(^{18}\)F]FDR was obtained from precursor 11 according to GP4 using MeCN as solvent. The reaction was carried out at 80 °C for 10 min. Removal of the protective groups was performed by incubation of the crude reaction mixture with 1 M HCl (400 µL) for 5 min at 105 °C. Thereafter, the reaction mixture was neutralized with 1 M NaOH (400 µL) to give [\(^{18}\)F]8 in 74% RCC (determined by radio-HPLC). [\(^{18}\)F]8 was also prepared using the conventional K2.2.2/K\(_2\)CO\(_3\) protocol according to GP5. The subsequent deprotection of the radiolabeled intermediate gave [\(^{18}\)F]8 in 58% RCC (determined by radio-HPLC). HPLC conditions: Column: Luna C-18 (2) 5µm (250×4.6 mm, Phenomenex), eluent: 55% MeCN, flow rate: 1.5 mL/min.
\textbf{"\textsuperscript{1}H- and APT-NMR Spectra}
$\text{N}^+\text{O}^-$

$\text{F}_3\text{C-S=O}$

$\text{H-N=O-N-CO}_2\text{Me}$

$\text{Ph}$

Chemical Shift (ppm)

-77.74
HPLC Traces

Radio HPLC trace

UV trace of $^{19}$F-reference substance ($\lambda=254$ nm)

Radio HPLC trace

UV trace of $^{18}$F-reference substance ($\lambda=254$ nm)
References