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Journal of Fluorine Chemistry 107 (2001) 329–335

JOURNAL OF FLUORINE CHEMISTRY

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Fluorination of aromatic compounds from 1-aryl-3,3-dimethyltriazenes and fluoride anions in acidic medium 2. Synthesis of (*S*)-[^{18}F]-3-fluoro-α-methylphenylalanine

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Received 15 May 2000; accepted 3 August 2000

Abstract

We propose here a new methodology to prepare 3-fluoro-α-methylphenylalanine in which fluorine is introduced at the end of the synthesis, on an elaborated substrate bearing a triazeno substituent which is decomposed by triflic acid in the presence of fluoride anions (yield = 31% versus $^{19}F^-$). As $^{18}F^-$ cannot be available free of other basic anions, this technique has been modified and adapted with some success to the radiosynthesis of [^{18}F]-3-fluoro-α-methylphenylalanine.

Keywords: 3-Fluoro-α-methylphenylalanine; [^{18}F]-3-fluoro-α-methylphenylalanine; Triazene; Diazonium triflate; Fluorine 18

1. Introduction

As described in our preceding paper, positron emission tomography (PET) is now commonly used for the medical exploration of human body, in combination with NMR imaging. At the CERMEP of Lyon, for example, (*S*)-6-[^{18}F]-fluoroDOPA is routinely produced and used for the diagnostic of neurotransmission disorders in brain such as epilepsy and Parkinson's disease. However, the present synthesis of this short-lived [^{18}F]-fluoroaromatic aminoacid is not completely satisfactory since radioactive fluorine is introduced in the early stages by a nucleophilic substitution with $^{18}F^-$ fluoride anion (Scheme 1). This constraint is connected to the type of technology used at the CERMEP to produce ^{18}F which is only available as $^{18}F^-$ anions.

To circumvent this problem, we propose to introduce fluorine on the aromatic nucleus through the acidic decomposition of 1-aryl-3,3-dialkyltriazenes in the presence of fluoride anions, according to the general technique we described in our preceding paper. The required triazeno-substituted substrate must be close to the final target and contain, at least, the other aromatic substituents and an equivalent of the chiral α-aminoacid moiety. To simplify the problem, we first examined the synthesis of (*S*)-[^{18}F]-3-fluoro-α-methylphenylalanine **1**, a less-substituted aromatic aminoacid. Moreover, because of its α-methyl substituent, this non-proteogenic compound binds specifically to neurotransmission sites and could give interesting indications on tyrosine and DOPA biosyntheses. Our general strategy is summarised in Scheme 2.

2. Results and discussion

Different triazeno-substituted precursors of **1** have been prepared as depicted in Scheme 3.

Commercially available 3-aminobenzyl alcohol **2** was easily transformed into the corresponding triazene **3** in a conventional way [1–3] (yield: 88%). The hydroxyl function was then replaced either by bromine, after treatment with Me_2S and *N*-bromosuccinimide [4,5] (compound **4**, yield: 86%), or by chlorine after treatment with sodium hydride and thionyl chloride (compound **5**, yield: 73%). Compound **4** is unstable at room temperature and was stored at −18°C; in contrast, compound **5** is stable at room temperature but was stored at 4°C. Then, **4** and **5** were substituted by enolates of different equivalents of chiral alanine (**6**–**8**) which were either commercially available (**8**, the two diastereomers are available) or readily prepared: **6** resulted from methylation of commercial imidazolidinone **13** (yield: 92%, *trans*/

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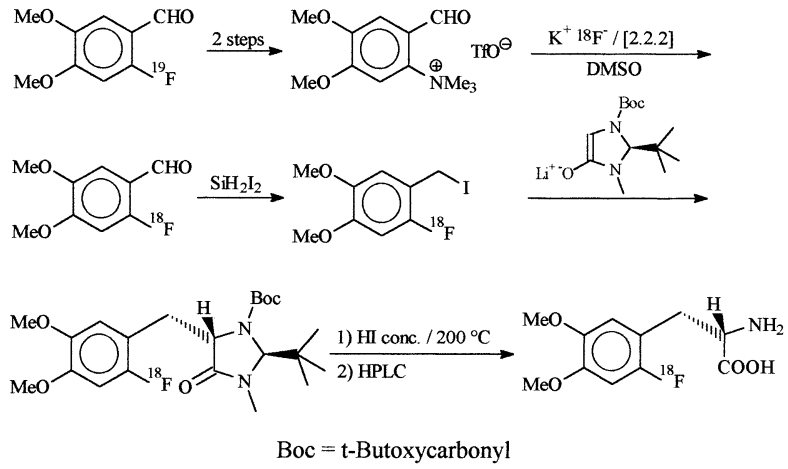
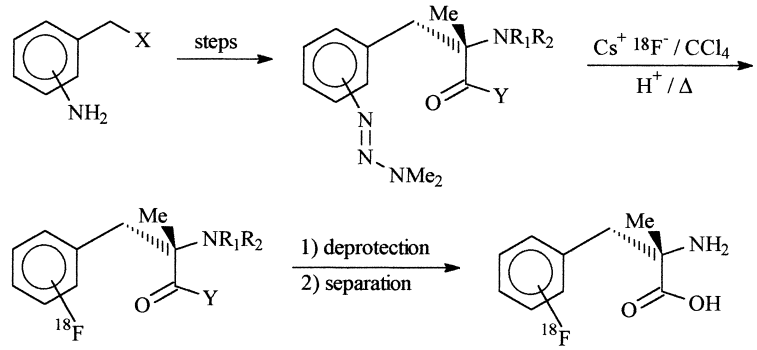
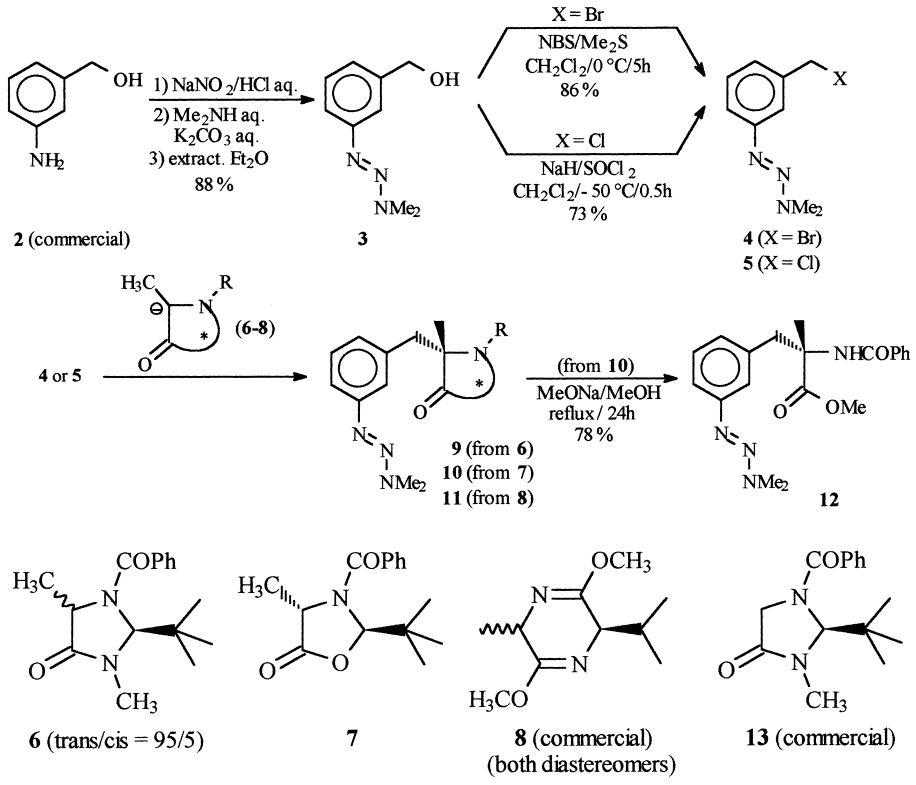
0022-1139/01/$ – see front matter
PII: S0022-1139(00)00377-8



Scheme 1. Present synthesis of (*S*)-[^{18}F]-6-fluoroDOPA.



Scheme 2. Synthetic strategy for (*S*)-[^{18}F]-3-fluorophenylalanine.



Scheme 3. Preparation of triazeno-substituted precursors.

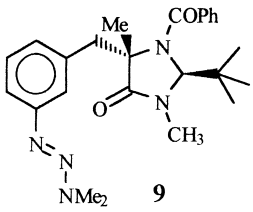
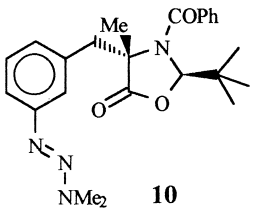
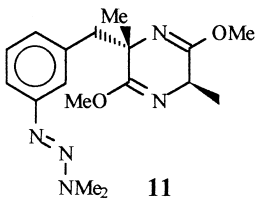
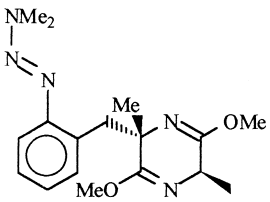
cis = 95/5) and **7** from condensation of (*S*)-alanine, pivalic aldehyde and benzyl chloride (*trans*-diastereomer, yield: 10%) [6]. The corresponding protected forms of (*S*)-3-(3′,3′-dimethyltriazeno)-α-methylphenylalanine (**9–11**) were thus obtained with an excellent diastereoselectivity (>95%); only one diastereomer was detected by NMR whatever the optical purity of **6–8**. Two rotamers, corresponding to the possible configurations of the benzamido moiety, were detected by NMR for products **9** (ratio: 70/30) and **10** (ratio: 80/20), as expected from the literature [7]. The results are reported in Table 1. In the same way, 2-aminobenzyl alcohol **14** was transformed into the corresponding 2-(3′,3′-dimethyltriazeno)benzyl chloride **15** (bromination failed) which was condensed with the dihydropyridazine **8** to form the *ortho*-isomer (**16**) of **11**. It must be noted that the condensation of **15** with **6** gave a very poor yield (10–14%), probably because of steric hindrance (Table 1).

In addition, compound **10** has been transformed into a methyl ester of a *N*-benzoyl aminoacid **12** (yield: 78%).

Compounds **9–12** and **16** were then submitted to fluorodetriazenation according to the technique reported in our preceding paper (procedure A). In order to verify our model with such substrates, non-radioactive caesium fluoride ($Cs^{19}F$) was first employed. The results are reported in Table 2.

Table 2 clearly indicates that **9** was the most suitable precursor of **1**. However, in contrast with our results from simple aryltriazenes (cf. preceding paper), fluorodetriazenation of **9** was far more efficient with triflic acid (31% versus CsF) than with sulfuric acid (7% versus CsF, whatever the acid amount). Moreover, it can be noted that no hydrodetriazenation product which would be impossible to separate from the fluorinated compound, was formed from **9–12** or **16** in triflic acid.

Table 1
Preparation of **9–11** and **16**

Product	Reactants[a]	Yield (%)	d.e. (%)[b]
 **9** 2 rotamers : 70/30	**4** (X = Br)/**6**/LDA	86	>95
	5 (X = Cl)/**6**/LDA	72	>95
 **10** 2 rotamers : 80/20	**4** (X = Br)/**7**/LDA	60	>95
	4 (X = Br)/**7**/LDEA	10	>95
 **11**	**4** (X = Br)/**8**/BuLi + DMEU	90	>95
 **16**	**15**/**8**/BuLi	62	>95

[a] LDA: lithium diisopropylamide; LDEA: lithium diethylamide; DMEU: dimethylethyleneurea.
[b] Only one diastereomer detected by NMR.

Table 2
Fluorotetriazenation of **9–12** and **16**[a]

Substrate	AH (eq)	ArF (%)		ArA (%)[b,c]	ArH[b]	ArCl[b]	Total (%)[b]
		versus CsF	versus substrate				
9	Oleum[d] (1.5)	7	1.5	N.D.	N.D.	N.D.	–
	Oleum[d] (2.1)	7	1.5	14	1	5	21.5
	CF_3SO_3H (2.1)	31	6	92	0	1	99
10	CF_3SO_3H (2.1)	18	3.6	86	0	N.D.	>90
11	CF_3SO_3H (2.1)	15	3	80	0	3	86
12	CF_3SO_3H (2.1)	20	4	53	0	N.D.	>57
16	CF_3SO_3H (2.1)	3	0.6	20	0	N.D.	>50

[a] $Ar{-}N{=}N{-}NMe_2 \xrightarrow[(3)\text{triazene}/CCl_4\ (4)90^\circ C/30\,\text{min}]{(1)CsF\,(0.2\,eq)/CCl_4/-10^\circ C\ (2)Acid(AH)(x\,eq)/-10^\circ C} Ar{-}F + Ar{-}A + Ar{-}H + Ar{-}Cl$

[b] Yields vs. triazene.

[c] Included ArOH when $AH = R_2SO_4$.

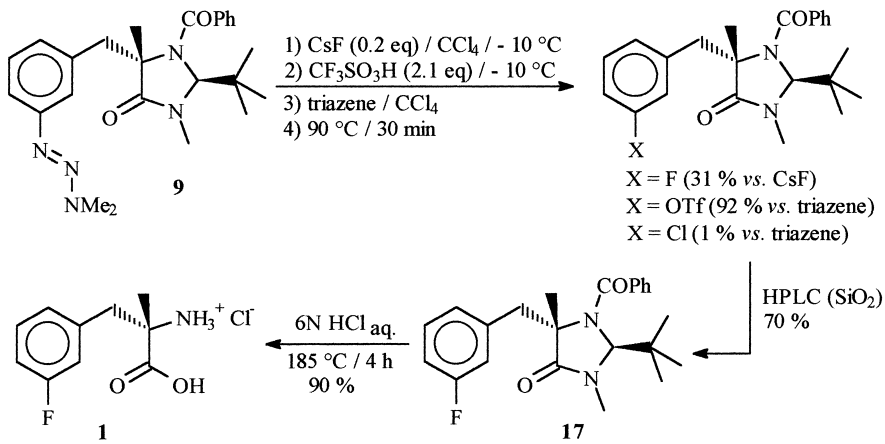
[d] Oleum: 6 wt.% SO_3.

Table 2 also shows that such a strategy was not adapted to the synthesis of the *ortho*-isomer of **1** since fluorodetriazenation of **16** delivered negligible yields of aryl fluoride but an extensive decomposition of organic materials (mass balance from identified compounds ≈50%). This was partly due to the dihydropyrazine moiety: indeed, we verified that, under fluorodetriazenation conditions (CsF/TfOH/90°C/0.5 h), **8** was converted (up to 90%) into alanine, valine and some undetermined products. Another reason of this failure was the vicinity of the triazeno group and the heterocyclic moiety which favoured the formation of tricyclic nitrogen-containing compounds that we effectively detected by GC–MS ($M^{\bullet+} = 286$, yield = 4%, $M^{\bullet+} = 272$, yield = 26%).

Finally, **17**, resulting from fluorodetriazenation of **9**, was separated and hydrolysed to **1** (Scheme 4).

As the conversion of **9** to **17** was achieved under conditions and in a yield suitable, in principle, for radiofluorination, we planned to adapt this step in order to replace $^{19}F^-$ by radioactive $^{18}F^-$. This adaptation was not a simple transposition for two main reasons. First, for safety reasons, $^{18}F^-$ must be used on the nanomolar scale (its amount being easily determined by radioactivity measurement) whereas the other reactants must be used on the 10^{-5} mole scale to be weighed with an acceptable accuracy. Thus, the F^-/triazene ratio drops down from 0.2 (in the case of $^{19}F^-$) to $\approx 10^{-4}$ (in the case of $^{18}F^-$). Secondly, $^{18}F^-$ is delivered by a cyclotron as a solution in $H_2{}^{18}O$ along with an unknown cation. It must first be loaded on a resin, to recover $H_2{}^{18}O$, then eluted with aqueous sodium carbonate in excess to provide a solution of a defined fluoride ($Na^{18}F$) which is evaporated. However, the so-formed $Na^{18}F$ is contaminated by a large amount of sodium carbonate which precludes the use of this salt mixture to carry out reactions in strongly acidic medium. Indeed, no ^{18}F-labelled **17** was obtained from **9**, under our selected conditions, with such a contaminated $Na^{18}F$.

Thus, the technique was modified. Two separate suspensions were prepared: the first was a suspension, in carbon tetrachloride, of the diazonium triflate resulting from the reaction, at −10°C, of equimolar amounts of **9** and triflic acid (suspension **A**). The second was a suspension, in carbon tetrachloride, of a thoroughly dried mixture of $Na^{18}F$ and



Scheme 4. Synthesis of **1**.

Na_2CO_3 complexed by crown-ether 15C5 (suspension **B**). Then, suspension **B** was poured on suspension **A**. Unfortunately, our apparatus was not well adapted and only a very small part of suspension **B** was effectively transferred; nevertheless, the transferred fraction was precisely known from radioactivity measurements. The mixture of these two suspensions was then heated at 90°C for 30 min and the crude mixture was analysed by radiodensitometry of TLC plates. This preliminary experiment delivered a 15% yield (corrected for radioactive decay) of ^{18}F-labelled **17**. This result was encouraging and work is now in progress to improve it, mainly by modifying the apparatus to allow a better transfer of suspensions.

In conclusion, we propose here a new methodology to prepare 3-fluoro-α-methylphenylalanine in which fluorine is introduced at the end of the synthesis, on an elaborated substrate bearing a triazeno substituent which is decomposed by triflic acid in the presence of fluoride anions (yield = 31% versus $^{19}F^-$). As $^{18}F^-$ cannot be available free of basic other anions, this technique has been modified and adapted with some success to the radiosynthesis of [^{18}F]-3-fluoro-α-methylphenylalanine.

3. Experimental

Prior to use, THF and diethyl ether were distilled over sodium-benzophenone and other solvents over calcium hydride. They were stored over 3 Å molecular sieves under N_2. CsF was ground and dried at 250°C for 24 h. Other reagents were used as received. TLC analyses were carried out on Kieselgel 60F 254 (Merck) deposited on aluminum plates, detection being done by UV (254 nm) or phosphomolybdic acid (10% in ethanol) or ninhydrin (1% in ethanol). Flash-chromatographies were performed on silica gel Geduran SI 60 (Merck). Uncorrected melting points were determined in capillary tubes (Büchi). Unless stated otherwise, NMR spectra were recorded in $CDCl_3$. ^{1}H NMR were recorded at 300 MHz on a Bruker AM 300 apparatus. ^{13}C NMR spectra were recorded on the same spectrometer at 75 MHz. The substitution pattern of the different carbons were determined by a "DEPT 135" sequence. ^{19}F NMR spectra were recorded on a Brucker AC 200 apparatus at 188 MHz. Chemical shifts (δ) are given in ppm versus TMS (^{1}H, ^{13}C) or $CFCl_3$ (^{19}F) used as internal references. Coupling constants are given in hertz. Crude yields were determined by ^{19}F NMR versus $PhOCF_3$ used as standard. GC was carried out on a Varian 3300 apparatus, fitted with a semi-capillary column (length: 15 m, Φ: 0.53 mm, film thickness (DB1): 1 μm) and a catharometric detector. Mass spectrometry was performed on a Nermag R10-10H spectrometer, using electron impact at 70 eV, and coupled with gas chromatography. When stereoisomers were supposed to be formed, the difference of their mass spectra was systematically carried out. Preparative HPLC was carried out on a Gilson equipment fitted with a reverse-phase type column (length: 250 mm, i.d.: 4.6 mm) filled with Inertsil ODS2 (Interchrom) and a UV detector (λ = 254 nm). The eluent flow was 1 ml·min^{-1}. Specific rotatory powers were measured on a Perkin-Elmer 141 polarimeter (cell: 1 cm).

3.1. 3-(3′,3′-Dimethyltriazeno)benzyl alcohol **3**

$NaNO_2$ (3.62 g, 52 mmol), dissolved in water (100 ml), was added at 0–5°C, over 30 min, to a solution of **2** (6.15 g, 50 mmol) in 1 M hydrochloric acid (130 ml). After 15 min stirring, a solution of dimethylamine (3.38 g, 75 mmol, as 10% aqueous solution) and K_2CO_3 (14.0 g, 100 mmol) in water (80 ml) was added over 30 min. The resulting mixture was stirred 15 min more then diethyl ether (150 ml) was added. After decantation, the aqueous phase was extracted with ether (3 × 100 ml) and the collected organic phases were dried over sodium sulfate and evaporated. The residue was purified by chromatography over silica gel with petroleum ether/AcOEt (55:45). Product **3** was obtained as a yellow oil (yield: 88–91%).

^{1}H NMR ($CDCl_3$, 300 MHz): δ 3.09 (broad s, 1H), 3.26 (broad s, 6H), 4.57 (s, 2H), 7.05–7.36 (m, 4H).

^{13}C NMR ($CDCl_3$, 75 MHz): δ 39.2, 64.8, 118.8, 119.0, 123.8, 128.8, 141.7, 150.9.

MS: *m/z* 179 ($M^{\bullet+}$), 135, 107, 89, 77.

3.2. 3-(3′,3′-Dimethyltriazeno)benzyl bromide **4**

A solution of dimethylsulfide (1.20 ml, 16 mmol) in CH_2Cl_2 (7 ml) was added, at 0°C over 15 min, to a solution of *N*-bromosuccinimide (2.67 g, 15 mmol) in CH_2Cl_2 (55 ml), kept under nitrogen. After 15 min stirring, the mixture was cooled to −25°C, then **3** (1.79 g, 10 mmol), dissolved in CH_2Cl_2 (15 ml), was added over 30 min. The resulting mixture was then stirred at 0°C for 6 h. After reaction, the medium was diluted with cold hexane (30 ml) then hydrolysed at 0°C with water (40 ml). The organic phase was washed with a cold saturated solution of potassium bromide (3 × 30 ml), dried over Na_2SO_4 and evaporated at 0°C in vacuo. The residue was rapidly purified over silica gel to deliver **4** as a yellow oil (yield: 86%) which was stored at −18°C under nitrogen.

^{1}H NMR ($CDCl_3$, 300 MHz): δ 3.33 (broad s, 6H), 4.50 (s, 2H), 7.13–7.52 (m, 4H).

^{13}C NMR ($CDCl_3$, 75 MHz): δ 33.7, 36.0, 42.8, 120.4, 120.6, 125.7, 129.1, 138.2, 151.1.

MS: *m/z* 243 ($M^{\bullet+}$), 241 ($M^{\bullet+}$), 199, 197, 171, 169, 162, 90, 89.

3.3. (2S,5R)-1-Benzoyl-2-tert-butyl-5-[3′-(3,3-dimethyltriazeno)benzyl]-3,5-dimethyl-imidazolidin-4-one **9**

2.35 ml of a 0.57 M solution of LDA in hexane-THF (3:1) were added, at −78°C within 15 min, to a solution of **6** (1.0 mmol), prepared from commercial **13** [7], in THF (6 ml). The resulting solution was stirred at −78°C for

1 h, then a solution of **4** (1.2 mmol) in THF (3 ml) was added dropwise at −78°C over 3 h, by means of a syringe–pump. The reaction mixture was stirred at this temperature for 2 h then at room temperature for 12 h. A saturated aqueous solution of NH_4Cl (10 ml) was added to the final mixture which was then extracted with diethyl ether (3 × 5 ml). The organic phase was dried over sodium sulfate, filtered and evaporated in vacuo. The residue was purified by chromatography over silica gel with petroleum ether/AcOEt (60:40) to give **9** as a pale yellow solid (yield: 86%).

mp: 138–139°C.

$[\alpha]_D^{25}$: +180.5° ($c = 1$, CH_2Cl_2).

^{1}H NMR ($CDCl_3$, 300 MHz, −20°C): δ 0.65 (s, 9H)*, 0.97 (s, 9H)**, 1.93 (s, 3H)*, 1.66 (s, 3H)**, 2.97 (s, 3H)*, 2.68 (s, 3H)**, 3.10 (broad s, 3H)*, 3.19 (broad s, 3H)**, 3.27 (d, $J = 13.7$, 1H)*, 2.61 (d, $J = 14.5$, 1H)**, 3.42 (broad s, 3H)*, 3.55 (broad s, 3H)**, 3.81 (d, $J = 13.7$, 1H)*, 2.78 (d, $J = 14.5$, 1H)**, 4.85 (s, 1H)*, 5.25 (s, 1H)**, 6.73–7.73 (m, 9H).

*: Major rotamer (70%); **: minor rotamer (30%).

^{13}C NMR ($CDCl_3$, 75 MHz, −20°C, major rotamer): δ 24.5, 27.5, 32.1, 39.3, 41.3, 67.6, 82.2, 119.6, 121.5, 127.9, 128.1, 128.6, 128.7, 130.0, 137.0, 137.1, 150.9, 170.7, 174.0.

MS: *m/z* 435 ($M^{\bullet+}$), 379, 378, 306, 273, 215, 201, 145, 144, 105, 77.

3.4. (2S,5R)-1-Benzoyl-2-tert-butyl-5-(3′-fluorobenzyl)-3,5-dimethyl-imidazolidin-4-one **17**

CCl_4 (4 ml) was added, through a septum, on CsF (0.18 mmol) conditioned as previously mentioned and kept under nitrogen. The suspension was cooled at −10°C then triflic acid (1.8 mmol) was added through the septum. The resulting mixture was stirred at −10°C for 5 min then the triazene **9** (0.9 mmol), dissolved in CCl_4 (4 ml), was added dropwise. After this final addition, the vessel was tightly closed, kept 5 min more at −10°C then immersed for the required time (see text and tables) in an oil bath preheated at 90°C. After cooling at room temperature, a 1 M aqueous solution of KOH was added then the crude mixture was extracted with diethyl ether. The organic phase was dried over sodium sulfate, filtered and evaporated in vacuo. The residue was separated by preparative HPLC (reverse phase Inertsil ODS2) with acetonitrile–water (2:1) as eluent.

mp: 139–140°C (white solid).

$[\alpha]_D^{25}$: +53.3° ($c = 1$, CH_2Cl_2).

^{1}H NMR ($CDCl_3$, 300 MHz): δ 0.68 (s, 9H)*, 0.99 (s, 9H)**, 1.90 (s, 3H)*, 1.63 (s, 3H)**, 2.97 (s, 3H)*, 2.68 (s, 3H)**, 3.52 (d, $J = 13.7$, 1H)*, 2.61 (d, $J = 14.7$, 1H)**, 3.56 (d, $J = 13.7$, 1H)*, 2.73 (d, $J = 14.7$, 1H)**, 4.88 (s, 1H)*, 5.28 (s, 1H)**, 6.73–7.64 (m, 9H).

*: Major rotamer (70%); **: minor rotamer (30%).

^{13}C NMR ($CDCl_3$, 75 MHz, major rotamer): δ 24.5, 27.6, 31.9, 39.3, 41.0, 67.4, 82.2, 113.8 ($J = 20.8$), 117.0 ($J = 21.0$), 126.4 ($J = 2.8$), 128.1, 128.4, 129.7 ($J = 8.1$), 130.4, 136.9, 139.1 ($J = 7.4$), 162.7 ($J = 245.8$), 170.6, 173.8.

^{19}F NMR ($CDCl_3$, $CFCl_3$, 188.2 MHz): δ −113.68 (m)**, −114.07 (m)*.

MS: *m/z* 435 ($M^{\bullet+}$), 325, 105, 77.

Calc. for $C_{22}H_{24}FNO_3$: C 72.23%, H 7.11%, N 7.32%, F 4.97%. Found: C 72.40%, H 7.18%, N 7.42%, F 5.09%.

3.5. (S)-3-Fluoro-α-methylphenylalanine **1**

A 6N solution of hydrochloric acid (2 ml) was added to **17** (74 mg, 0.17 mmol). The vessel was tightly closed and heated at 185°C for 4 h. The cooled reaction medium was washed with CH_2Cl_2 (3 × 2 ml) then evaporated in vacuo at 40°C to deliver **1** as a white solid (36 mg, yield: 90%).

^{1}H NMR (D_2O, 300 MHz): δ 1.58 (s, 3H), 2.88 (d, $J = 14.2$, 1H), 3.47 (d, $J = 14.2$, 1H), 7.02–7.17 (m, 3H), 7.42 (dd, $J = 13.8$, $J = 6.4$, 1H).

^{13}C NMR (D_2O, 75 MHz): δ 24.9, 44.9, 64.6, 117.2 ($J = 21.0$), 119.2 ($J = 21.5$), 128.4 ($J = 2.7$), 133.2 ($J = 8.6$), 139.2 ($J = 7.5$), 165.2 ($J = 243.8$), 178.5.

^{19}F NMR (D_2O, $CFCl_3$, 188.2 MHz): δ −112.3 (m).

3.6. Preparation of radioactive $^{18}F^-$

The solution of $^{18}F^-$ anions in $H_2{}^{18}O$, delivered by the cyclotron, was passed over a Cl^- resin (Biorad AG1-X8) previously conditioned by washing with 2 M aqueous sodium hydroxide then water. Then, the radioactive species were eluted with aqueous sodium carbonate (800 μl, 50.6 μmol ml^{-1}). About 80 μl of the resulting solution were placed in a PTFE flask and evaporated at 120°C under a nitrogen stream. The remaining water was withdrawn by azeotropic evaporation with acetonitrile (3 × 200 μl) and the solid residue was dried at 160°C under a nitrogen flow over 20 min.

3.7. Fluorination of **9** with $^{18}F^-$

A solution of **9** (25.0 μg, 57.4 μmol) in CCl_4 (200 μl) were placed in a glass flask and cooled to −10°C before adding triflic acid (10.1 μl, 2.0 eq). The resulting suspension was stirred at −10°C for 5 min. Then, an heterogeneous solution of $Na^{18}F/Na_2CO_3$ (23.0 μmol) and 15C5 crown-ether (27.6 μmol) in CCl_4 (200 ml) was transferred into the previous suspension at −10°C. The reaction flask was tightly closed and stirred at −10°C for 3 min before heating at 90°C over 30 min. Then, the reaction medium was cooled at 0°C and acetone (100 μl) was added to obtain an homogeneous solution. A sample of this solution was deposited on a TLC plate and eluted with a mixture of petroleum ether and ethyl acetate (60:40). The spots were analysed by radiodensitometry (Berthold LB 285 apparatus).

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