SUPPORTING INFORMATION

(S)-2-((R)-Fluoro(phenyl)methyl) oxirane: a general reagent for determining the ee of α -chiral amines.

Sergi Rodríguez-Esrich^{a, b}, Dana Popa^a, Ciril Jimeno^a, Anton Vidal-Ferran^{a, c, *}, Miquel A. Pericàs^{a, b, *}

^a*Institute of Chemical Research of Catalonia (ICIQ), Av. Països Catalans s/n, 43007 Tarragona (Spain).*

^b*Departament de Química Orgànica, Universitat de Barcelona, c/ Martí i Franquès 1-11, 08008 Barcelona (Spain).*

^c *ICREA*

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- xi) (2*S*,3*R*)-1*N*-((*DL*)-phenylalaninate methyl ester)-3-fluoro-
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I) Preparation of (S)-2-((R)-fluoro(phenyl)methyl)oxirane 1:

Monotosylated fluorodiol **3** (10.0 mmol, 3.24 g), which was prepared according to the procedure described in ref. 7 in the Letter, was dissolved in 10 mL of anhydrous THF in a flame-dried flask, and treated with 10 mL of 1 M lithium *tert*-butoxide in THF. After 30 minutes, the reaction was quenched with 20 mL of water and extracted with CH₂Cl₂ (3x15 mL). The organic extracts were dried (Na₂SO₄), and solvents removed off *in vacuo* to afford **1** in quantitative yield.

¹H NMR (CDCl₃, 400 MHz): δ 7.34-7.45: (5H, m, ArH), 5.13 (1H, dd, ³J_{HH} = 6,3 Hz, ²J_{HF} = 47,2 Hz, CHF), 3.35 (1H, m, CHO), 2.89 (1H, ddd, ⁴J_{HF} = 4.2 Hz, ³J_{HH} = 4.2 Hz, ²J_{HH} = 4.7 Hz, CHHO), 2.88 (1H, ddd, ⁴J_{HF} = 1.6 Hz, ³J_{HH} = 2.6 Hz, ²J_{HH} = 4.8 Hz, CHHO) ppm.

¹³C {¹H} NMR (CDCl₃, 100 MHz): δ 136,04 (d, ²J_{CF} = 21.2 Hz), 129.07 (d, ⁴J_{CF} = 2.2 Hz), 128.66, 126.00 (d, ³J_{CF} = 6.6 Hz), 94.62 (d, ¹J_{CF} = 175.7 Hz), 53.62 (d, ²J_{CF} = 27.9 Hz), 44.18 (d, ³J_{CF} = 8.1 Hz) ppm.

¹⁹F NMR (CDCl₃, 470.5 MHz): δ -180,96 (d, ²J_{FH} = 46.8 Hz) ppm.

[α]_D²⁷ -20.9 (c = 1.01, CHCl₃).

MS (EI⁺): m/z 152 (35%, M⁺), 109 (100%, C₇H₆F).

II) General procedure for the preparation of compounds 5a-5i:

82 mg (0.530 mmol) of fluoroepoxide **1**, and 568 mg (5.30 mmol) of LiClO₄ were weighed in a microwave reactor vessel, dissolved in 5 mL of dry THF, and purged with argon. Then 0.371 mmol of amine were added via syringe. The mixture was heated at 75 °C in the microwave reactor (CEM Discovery) for 1.5-3 hours. After cooling down to r.t., water was added to the vessel, and the mixture was extracted with CH₂Cl₂ (x3), dried (Na₂SO₄), and the solvents removed *in vacuo*. The resulting crude was purified by flash chromatography using silica gel and eluting with hexanes: ethyl acetate mixtures of increasing polarity. Partial resolution can be achieved when a mixture of diastereomers is obtained.

III) General analytical procedure for the determination of the ee of α -chiral amines:

4.1 mg (0.0265 mmol) of fluoroepoxide **1**, and 28.4 mg (0.265 mmol) of LiClO₄ were weighed in the microwave reactor vessel, which was purged with argon, and dissolved in 1.5 mL of dry THF. Then, 490 μ L of a 0.55 M solution (0.270 mmol) of amine in THF were added. The mixture was heated at 75 °C using microwaves for 20 minutes. After cooling down to r.t., water was added to the vessel, and the mixture was extracted with CH₂Cl₂ (x3), dried (Na₂SO₄), and the solvents removed *in vacuo*. The resulting crude was analyzed by ¹⁹F-NMR using CDCl₃ as solvent.

IV) Characterization of compounds:

* Indicates a signal corresponding to the other diastereomer.

+* Indicates a signal that corresponds to both diastereomers (signal not separated)

i) (1*R*,2*S*)-1-fluoro-1-phenyl-3-((*R*&*S*)-1'-phenylethylamino)propan-2-ol, **5a**:

¹H NMR (CDCl₃, 500MHz): δ 7.2-7.4(+*) (20H, m, ArH), 5.38 (1H, dd, ³J_{HH} = 5.6 Hz, ²J_{HF} = 47.2 Hz, CHF), 5.33* (1H, dd, ³J_{HH} = 6.2 Hz, ²J_{HF} = 47.5 Hz, CHF), 3.96* (1H, m, CHOH), 3.85 (1H, m, CHOH), 3.68 (1H, q, ³J_{HH} = 6.6 Hz, CHCH₃), 3.66* (1H, q, ³J_{HH} = 6.7 Hz, CHCH₃), 2.55 (1H, dd, ²J_{HH} = 12.3 Hz, ³J_{HH} = 6.7 Hz, CHH), 2.50 (1H, dd, ²J_{HH} = 12.3 Hz, ³J_{HH} = 4.0 Hz, CHH), 2.49* (1H, dd, ²J_{HH} = 12.3 Hz, ³J_{HH} = 4.0 Hz, CHH), 2.40* (1H, dd, ²J_{HH} = 12.3 Hz, ³J_{HH} = 7.1 Hz, CHH), 2.18 (2H, s, NH, OH), 1.33* (3H, d, ³J_{HH} = 6.7 Hz, CH₃), 1.31 (3H, d, ³J_{HH} = 6.6 Hz, CH₃).

^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 145.06, 144.84*, 136.71 (d, $^2J_{\text{CF}} = 20.3$ Hz), 136.55* (d, $^2J_{\text{CF}} = 20.3$ Hz), 128.81 (CH, $^4J_{\text{CF}} = 1.6$ Hz); 128.74* (CH, $^4J_{\text{CF}} = 1.5$ Hz), 128.49* (CH, $^3J_{\text{CF}} = 5.1$ Hz), 128.44 (CH, $^3J_{\text{CF}} = 4.9$ Hz), 127.08, 126.55*, 126.50, 126.46*, 126.40, 126.33, 126.27*, 95.82 (CHF, $^1J_{\text{CF}} = 173.0$ Hz); 95.68* (d, $^1J_{\text{CF}} = 173.6$ Hz), 72.78 (d, $^2J_{\text{CF}} = 22.6$ Hz), 72.60* (d, $^2J_{\text{CF}} = 21.4$ Hz), 58.44, 58.13*, 48.63 (d, $^3J_{\text{CF}} = 5.2$ Hz), 48.26* (d, $^3J_{\text{CF}} = 5.5$ Hz), 24.20*, 23.88.

^{19}F NMR (CDCl_3 , 470.5 MHz): δ -186.84* (dd, $^2J_{\text{FH}} = 47.3$ Hz, $^3J_{\text{FH}} = 17.2$ Hz), -188.80 (dd, $^2J_{\text{FH}} = 47.0$ Hz, $^3J_{\text{FH}} = 18.3$ Hz).

HRMS (EI⁺): m/z found: 273.1524 (M^+), calculated for $\text{C}_{17}\text{H}_{20}\text{NOF}$: 273.1529.

HPLC (column Zorbax RX-SIL, eluent hexane: 2-propanol (0.1% Et_2NH) 99:1, flow 1.0 ml/min), t_{R} (diastereomer "3R")= 14.6 min; t_{R} (diastereomer "3S")= 16.6 min. (Sample partially resolved after flash chromatography)

zorbax ; H:IPA* 99:1; 1mL/min; 10 µL

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Injection Date : 4/7/2005 3:02:50 PM      Seq. Line : 7
Sample Name    : SRE320Z                  Location  : Vial 14
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Acq. Instrument : HPLC1100                Inj Volume: 5 µl
Different Inj Volume from Sequence !      Actual Inj Volume: 10 µl
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Last changed   : 5/19/2005 8:08:56 PM by Felix
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                        Area Percent Report
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Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs

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Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	14.569	BV	0.6437	145.30632	3.70653	34.7256
2	16.581	VB	0.6819	273.13513	6.43067	65.2744

Totals : 418.44145 10.13719

ii) (1R,2S)-1-fluoro-1-phenyl-3-((S)-1'-phenylethylamino)propan-2-ol, (S)-5a:

¹H NMR (CDCl₃, 400MHz): δ 7.20-7.34 (10H, m, ArH), 5.33 (1H, dd, ³J_{HH} = 6.2 Hz, ²J_{HF} = 47.5 Hz, CHF), 3.97 (1H, m, CHOH), 3.66 (1H, q, ³J_{HH} = 6.6 Hz, CHCH₃), 2.50 (1H, dd, ²J_{HH} = 12.3 Hz, ³J_{HH} = 4.0 Hz, CHH), 2.40 (1H, dd, ²J_{HH} = 12.3 Hz, ³J_{HH} = 7.1 Hz, CHH), 2.27 (2H, bs, NH, OH), 1.33 (3H, d, ³J_{HH} = 6.6 Hz, CH₃).

^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz): δ 144.66, 136.48 (d, $^2J_{\text{CF}} = 20.4$ Hz), 128.77 (d, $^4J_{\text{CF}} = 2.0$ Hz), 128.44 (d, $^3J_{\text{CF}} = 5.1$ Hz), 127.08, 126.52, 126.41, 126.34, 95.74 (d, $^1J_{\text{CF}} = 173.5$ Hz), 72.52 (d, $^2J_{\text{CF}} = 22.7$ Hz), 58.12, 48.21 (d, $^3J_{\text{CF}} = 5.3$ Hz), 24.12.

^{19}F NMR (CDCl_3 , 470.5 MHz): δ -186.84 (dd, $^2J_{\text{FH}} = 47.4$ Hz, $^3J_{\text{FH}} = 17.0$ Hz).

IR (neat, ATR): 3299, 3148, 2915, 2863 (cm^{-1}).

HRMS (EI⁺): m/z found: 273.1519 (M⁺), calculated for C₁₇H₂₀NOF: 273.1529

[α]_D²⁷ -56.5 (c = 0.39, CHCl₃).

HPLC (column Zorbax RX-SIL, eluent hexane: 2-propanol (0.1% Et₂NH) 99:1, flow 1.0 ml/min), t_R = 17.0 min.

Data File C:\HPCHEM\1\DATA\SERGI\SRE321_3.D

Sample Name: SRE321_3

zorbax ; H:IPA* 99:1; 1mL/min; 10 μ L

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Sample Name     : SRE321_3                 Location  : Vial 15
Acq. Operator   : sergi                    Inj      :    1
Acq. Instrument : HPLC1100                 Inj Volume: 5  $\mu$ L
Different Inj Volume from Sequence !      Actual Inj Volume: 10  $\mu$ L
Acq. Method     : C:\HPCHEM\1\METHODS\PERICAS\SERGI\SRE320_4.M
Last changed    : 4/6/2005 4:49:07 PM by sergi
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Last changed    : 5/19/2005 8:08:56 PM by Felix
(modified after loading)
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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	1.943	PB	0.5022	216.99396	8.3149	6.98497
2	17.024	PB	0.5781	2392.69092	91.6851	63.68588

Totals : 2609.68488 70.67085

iii) (1*R*,2*S*)-3-((*R*&*S*)-1'-(*p*-bromophenyl)ethylamino)-1-fluoro-1-phenylpropan-2-ol, 5b:

^1H NMR (CDCl_3 , 400MHz): δ 7.25-7.41 (14H, m, ArH), 7.08-7.13 (4H, m, ArH), 5.35* (1H, dd, $^3J_{\text{HH}} = 5.6$ Hz, $^2J_{\text{HF}} = 47.2$ Hz, CHF), 5.32 (1H, dd, $^3J_{\text{HH}} = 6.4$ Hz, $^2J_{\text{HF}} = 47.6$ Hz, CHF), 3.98 (1H, m, CHOH), 3.90* (1H, m, CHOH), 3.66* (1H, q, $^3J_{\text{HH}} = 6.6$ Hz, CHCH₃), 3.63 (1H, q, $^3J_{\text{HH}} = 6.6$ Hz, CHCH₃), 2.98(+*) (4H, s, NH, OH), 2.55 (1H, dd, $^2J_{\text{HH}} = 12.2$ Hz, $^3J_{\text{HH}} = 7.3$ Hz, CHH), 2.46 (1H, dd, $^2J_{\text{HH}} = 12.2$ Hz, $^3J_{\text{HH}} = 4.7$ Hz, CHH), 2.45* (1H, dd, $^2J_{\text{HH}} = 12.2$ Hz, $^3J_{\text{HH}} = 4.9$ Hz, CHH), 2.35* (1H, dd, $^2J_{\text{HH}} = 12.3$ Hz, $^3J_{\text{HH}} = 7.2$ Hz, CHH), 1.29 (3H, d, $^3J_{\text{HH}} = 6.1$ Hz, CH₃), 1.28* (3H, d, $^3J_{\text{HH}} = 6.3$ Hz, CH₃).

^{13}C { ^1H } NMR (CDCl_3 , 125MHz): δ 144.13*, 143.89, 136.53* (d, $^2J_{\text{CF}} = 20.0$ Hz), 136.34 (d, $^2J_{\text{CF}} = 20.2$ Hz), 131.54*, 131.51, 128.88 (d, $^4J_{\text{CF}} = 1.3$ Hz); 128.78* (d, $^4J_{\text{CF}} = 1.3$ Hz), 128.47, 128.45*, 128.31, 128.23*, 126.39 (d, $^3J_{\text{CF}} = 6.6$ Hz), 126.21* (d, $^3J_{\text{CF}} = 7.2$ Hz), 120.65, 120.63*, 95.78 (d, $^1J_{\text{CF}} = 172.7$ Hz); 95.62* (d, $^1J_{\text{CF}} = 173.7$ Hz), 72.90* (d, $^2J_{\text{CF}} = 22.8$ Hz), 72.69 (d, $^2J_{\text{CF}} = 22.9$ Hz), 57.85*, 57.57, 48.54* (d, $^3J_{\text{CF}} = 5.0$ Hz), 48.16 (d, $^3J_{\text{CF}} = 5.6$ Hz), 24.12*, 23.89.

^{19}F NMR (CDCl_3 , 470.5 MHz): δ -185.84* (dd, $^2J_{\text{FH}} = 47.2$ Hz, $^3J_{\text{FH}} = 16.0$ Hz), -188.24 (dd, $^2J_{\text{FH}} = 48.1$ Hz, $^3J_{\text{FH}} = 17.2$ Hz).

HRMS (ES $^+$): m/z found: 352.0695 (M+H), calculated for $\text{C}_{17}\text{H}_{20}\text{NOFBr}$: 352.0712

HPLC (column Zorbax RX-SIL, eluent hexane:2-propanol (0.1% Et_2NH) 99:1, flow 1.0 ml/min), t_{R} (diastereomers)= 18.1 min, 21.1 min.

zorbax ; H:IPA* 99:1; 1mL/min; 10 µL; 5mg/ml

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Acq. Instrument  : HPLC1100                 Inj Volume: 5 µl
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Last changed     : 4/6/2005 4:49:07 PM by serqi
Analysis Method  : C:\HPCHEM\1\METHODS\PERICAS\FELIX\BN-EP01.M
Last changed     : 5/19/2005 8:08:56 PM by Felix
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                        Area Percent Report
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Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
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Signal 1: VWD1 A, Wavelength=254 nm

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1	18.126	BB	0.5798	1118.81921	29.66504	48.3785
2	21.094	BB	0.7028	1193.81982	26.96879	51.6215
Totals :				2312.63904	56.63383	

iv) (1*R*,2*S*)-1-fluoro-1-phenyl-3-((*R*&*S*)-1',2',3',4'-tetrahydronaphthyl-1'-amino)propan-2-ol, 5c:

^1H NMR (CDCl_3 , 500 MHz): δ 7.31-7.44: (12H, m, ArH); 7.05-7.20 (6H, m, ArH), 5.49 (1H, dd, CHF, $^3J_{\text{HH}} = 5.8$ Hz, $^2J_{\text{HF}} = 47.2$ Hz), 5.45* (1H, dd, CHF, $^3J_{\text{HH}} = 5.8$ Hz, $^2J_{\text{HF}} = 47.2$ Hz), 4.01* (1H, m, CHOH), 4.00 (1H, m, CHOH), 3.71 (1H, m, CHNH), 3.70* (1H, m, CHNH), 2.55-2.85(+*) (8H, m, CH_2NH , CH_2C), 2.41(+*) (4H, bs, NH, OH), 1.65-1.95(+*) (8H, m, $\text{CH}_2\text{CH}_2\text{CHNH}$).

^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz): δ 138.53*, 138.52, 137.25*, 137.22, 136.73* (d, $^2J_{\text{CF}} = 20.3$ Hz); 136.71 (d, $^2J_{\text{CF}} = 19.7$ Hz), 128.98*, 128.95, 128.82(+*), 128.66 (d, $^3J_{\text{CF}} = 1.6$ Hz); 128.60 (d, $^3J_{\text{CF}} = 1.9$ Hz) 128.37(+*), 126.74*, 126.27, 126.34*, 126.20, 125.73*, 125.65, 95.77* (d, $^1J_{\text{CF}} = 173.6$ Hz), 95.63 (d, $^1J_{\text{CF}} = 173.6$ Hz), 72.79 (d, $^2J_{\text{CF}} = 22.8$ Hz), 72.31* (d, $^2J_{\text{CF}} = 22.7$ Hz), 55.67, 55.03*, 48.04 (d, $^3J_{\text{CF}} = 5.4$ Hz), 47.82* (d, $^3J_{\text{CF}} = 5.7$ Hz), 29.24*, 29.15, 28.59, 27.96*, 18.81, 18.73*.

^{19}F NMR (CDCl_3 , 470.5 MHz): δ -187.07 (dd, $^2J_{\text{FH}} = 47.4$ Hz, $^3J_{\text{FH}} = 16.2$ Hz), -188.55* (dd, $^2J_{\text{FH}} = 47.9$ Hz, $^3J_{\text{FH}} = 16.6$ Hz).

HRMS (ES⁺): m/z found: 300.1758 (M+H), calculated for $\text{C}_{19}\text{H}_{23}\text{NOF}$: 300.1764.

HPLC (column Zorbax RX-SIL, eluent hexane: 2-propanol (0.1% Et_2NH) 99.8:0.2 to 99:1 and 20 min isocratic, flow 1.0 ml/min, $T=40$ °C), t_{R} (diastereomers)= 17.5 min; 19.0 min.

0.2%IPA* to 1% IPA* in 15 min (40°C)

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Acq. Instrument : HPLC1100                      Inj Volume : 2 µl
Acq. Method     : C:\HPCHEM\1\METHODS\SRE343 2.M
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Analysis Method : C:\HPCHEM\1\METHODS\NGG12C.M
Last changed    : 4/19/2005 11:27:27 AM by ecm
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Area Percent Report
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Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs

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Signal 1: DAD1 D, Sig=264,8 Ref=360,50

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.466	VB	0.3110	317.87903	14.77583	57.8464
2	19.048	PV	0.3178	231.64313	10.40058	42.1536

Totals : 549.52216 25.17642

v) (1R,2S)-3-((R&S)-indan-1'-amino)-1-fluoro-1-phenylpropan-2-ol, 5d:

¹H NMR (CDCl₃, 500MHz): δ 7.27-7.41 (12H, m, ArH); 7.13-7.23 (6H, m, ArH), 5.45* (1H, dd, ³J_{HH} = 5.9 Hz, ²J_{HF} = 47.2 Hz, CHF), 5.40 (1H, dd, ³J_{HH} = 5.8 Hz, ²J_{HF} = 47.2 Hz, CHF), 4.17 (1H, t, ³J_{HH} = 6.6 Hz, CHCH₂), 4.14* (1H, t, ³J_{HH} = 6.6 Hz, CHCH₂), 3.99 (2H, m, CHOH+*), 2.96(+*) (2H, m, CHHCH₂CH), 2.80(+*) (2H, m, CHHCH₂CH), 2.75(+*) (4H, bs, NH, OH), 2.73(+*) (2H, m, CHHNH), 2.66(+*) (2H, m, CHHNH), 2.34(+*) (2H, m, CHHCHNH), 1.76(+*) (2H, m, CHHCHNH).

^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz): δ 144.54*, 144.49, 143.53, 143.51*, 136.62* (d, $^2J_{\text{CF}} = 19.8$ Hz); 136.60 (CCHF, $^2J_{\text{CF}} = 20.5$ Hz), 128.80 (d, $^4J_{\text{CF}} = 2.1$ Hz), 128.75* (d, $^4J_{\text{CF}} = 1.9$ Hz), 128.48(+*), 127.60, 127.58*, 126.35 (d, $^3J_{\text{CF}} = 4.5$ Hz), 126.33* (d, $^3J_{\text{CF}} = 4.4$ Hz), 126.30, 126.27*, 124.79, 124.76*, 124.10, 124.07*, 95.85 (d, $^1J_{\text{CF}} = 174.1$ Hz), 95.72* (d, $^1J_{\text{CF}} = 173.6$ Hz), 72.78* (d, $^2J_{\text{CF}} = 22.8$ Hz), 72.33 (d, $^2J_{\text{CF}} = 22.7$ Hz), 63.35*, 63.00, 48.01* (d, $^3J_{\text{CF}} = 5.8$ Hz), 47.88 (d, $^3J_{\text{CF}} = 5.7$ Hz), 33.62*, 33.24, 30.27*, 30.21.

^{19}F NMR (CDCl_3 , 470.5 MHz): δ -187.27 (dd, $^2J_{\text{FH}} = 47.1$ Hz, $^3J_{\text{FH}} = 17.9$ Hz), -187.91* (dd, $^2J_{\text{FH}} = 47.1$ Hz, $^3J_{\text{FH}} = 17.3$ Hz).

HRMS (ES $^+$): m/z found: 286.1609 (M+H), calculated for $\text{C}_{18}\text{H}_{21}\text{NOF}$: 286.1607.

HPLC (column Zorbax RX-SIL, eluent hexane: 2-propanol (0.1% Et_2NH) 99:1, flow 1.0 ml/min), t_{R} (diastereomer "3S")= 10.7 min; t_{R} (diastereomer "3R")= 14.4 min.

zorbax ; H:IPA* 99:1; 1mL/min; 10 µL

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Injection Date : 4/7/2005 12:44:22 PM      Seq. Line :    4
Sample Name    : SRE324                    Location  : Vial 11
Acq. Operator  : sergi                     Inj       :    1
Acq. Instrument: HPLC1100                  Inj Volume: 5 µl
Different Inj Volume from Sequence !      Actual Inj Volume: 10 µl
Acq. Method    : C:\HPCHEM\1\METHODS\PERICAS\SERGI\SRE320_4.M
Last changed   : 4/6/2005 4:49:07 PM by sergi
Analysis Method: C:\HPCHEM\1\METHODS\PERICAS\FELIX\BN-EP01.M
Last changed   : 5/19/2005 8:24:41 PM by Felix
                (modified after loading)
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                        Area Percent Report
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Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
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Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.732	MM	0.8216	45.58485	9.24767e-1	3.7325
2	14.393	PB	0.8091	1175.70593	23.80808	96.2675

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Totals :                      1221.29079  24.73285
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vi) (1*R*,2*S*)-3-((*S*)-indan-1'-amino)-1-fluoro-1-phenylpropan-2-ol, (*S*)-5d:

^1H NMR (CDCl_3 , 400 MHz): δ 7.32-7.42 (5H, m, ArH), 7.13-7.29 (4H, m, ArH), 5.45 (1H, dd, $^3J_{\text{HH}} = 5.9$ Hz, $^2J_{\text{HF}} = 47.3$ Hz, CHF), 4.13 (1H, t, $^3J_{\text{HH}} = 6.6$ Hz, CHCH₂), 3.97 (1H, m, CHOH), 2.95 (1H, ddd, $^2J_{\text{HH}} = 15.9$ Hz, $^3J_{\text{HH}} = 8.5$ Hz, $^3J_{\text{HH}} = 4.9$ Hz, CHHCH₂CH), 2.79 (1H, dd, $^2J_{\text{HH}} = 12.2$ Hz, $^3J_{\text{HH}} = 3.9$ Hz, CHHNNH), 2.77 (1H, m, CHHCH₂CH), 2.64 (1H, dd, $^2J_{\text{HH}} = 12.2$ Hz, $^3J_{\text{HH}} = 7.2$ Hz, CHHNNH), 2.40 (2H, bs, NH, OH), 2.33 (1H, m, CHHCHNNH), 1.76 (1H, m, CHHCHNNH).

^{13}C { ^1H } NMR (CDCl_3 , 100 MHz): δ 144.41, 143.55, 136.65 (d, $^2J_{\text{CF}} = 19.8$ Hz), 128.74 (d, $^4J_{\text{CF}} = 2.1$ Hz), 128.47, 127.62, 126.30 (d, $^3J_{\text{CF}} = 5.0$ Hz), 126.25, 124.79, 124.11, 95.68 (d, $^1J_{\text{CF}} = 173.5$ Hz), 72.75 (d, $^2J_{\text{CF}} = 22.7$ Hz), 63.35, 48.00 (d, $^3J_{\text{CF}} = 5.3$ Hz), 33.51, 30.26.

^{19}F NMR (CDCl_3 , 470.5 MHz): δ -187.93 (dd, $^2J_{\text{FH}} = 47.2$ Hz, $^3J_{\text{FH}} = 17.4$ Hz).

IR (neat, ATR): 3248, 3067, 2916, 2849 (cm^{-1}).

HRMS (ES⁺): m/z found: 286.1621 (M+H); calculated for $\text{C}_{18}\text{H}_{21}\text{NOF}$: 286.1607.

$[\alpha]_{\text{D}}^{27}$ -3.2 ($c = 1.05$, CHCl_3).

HPLC (column Zorbax RX-SIL, eluent hexane: 2-propanol (0.1% Et_2NH) 99:1, flow 1.0 ml/min), $t_{\text{R}} = 11.1$ min.

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Injection Date   : 4/19/2005 11:40:47 AM      Seq. Line :    2
Sample Name     : SRE379 2                   Location  : Vial 22
Acq. Operator   : serqi                      Inj       :    1
Acq. Instrument : HPLC1100                   Inj Volume: 5 µl
Different Inj Volume from Sequence !      Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\PERICAS\SERGI\SRE320_4.M
Last changed    : 4/6/2005 4:49:07 PM by serqi
Analysis Method : C:\HPCHEM\1\METHODS\PERICAS\FELIX\BN-EP01.M
Last changed    : 5/19/2005 8:24:41 PM by Felix
                (modified after loading)

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                        Area Percent Report
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Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs

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Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	11.095	PB	0.3587	370.06372		19.71940	100.0000

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Totals :                      370.06372  19.71940
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vii) (1*R*, 2*S*)-1-fluoro-3-(2'-octylamino)-1-phenylpropan-2-ol, 5e:

^1H NMR (CDCl_3 , 400 MHz): 7.34-7.18(+*) (10H, m, ArH), 5.30(+*) (2H, dd, $^2J_{\text{H-F}} = 47.2$ Hz, $^3J_{\text{HH}} = 5.84$ Hz CHF), 3.89(+*) (2H, m, CHOH), 2.51(+*) (10H, m, CH_2NHCH , OH), 1.17(+*) (20H, m (CH_2)₅), 0.9* (3H, d, $^3J_{\text{HH}} = 6.43$ Hz, CH_3CH), 0.89 (3H, d, $^3J_{\text{HH}} = 6.43$ Hz, CH_3CH), 0.83(+*) (6H, m, CH_3CH_2).

^{13}C { ^1H } NMR (CDCl_3 , 125 MHz): 136.74, 136.57*, 128.77, 128.47(+*), 126.36, 126.31*, 95.80* (d, $^1J = 174.1$ Hz), 95.78 (d, $^1J_{\text{C-F}} = 174$ Hz), 72.4* (d, $^2J_{\text{C-F}} = 23.5$ Hz), 72.03 (d, $^2J_{\text{C-F}} = 22.7$ Hz), 53.50, 53.08*, 47.75* (d, $^3J_{\text{C-F}} = 5.5$ Hz), 47.46 (d, $^3J_{\text{C-F}} = 5.5$ Hz), 37.03*, 36.80, 31.80(+*), 29.38(+*), 25.95*, 25.85, 22.58(+*), 20.33*, 20.06, 14.05(+*).

^{19}F NMR (CDCl_3 , 470.5 MHz): δ -187.63* (dd, $^2J_{\text{FH}} = 46.3$ Hz, $J^3_{\text{FH}} = 17.7$ Hz), -187.72 (dd, $^2J_{\text{FH}} = 46.3$ Hz, $^3J_{\text{FH}} = 17.7$ Hz).

IR_{max} : 3500, 3176, 3033, 2925, 2854, 1454, 1085, 1073 cm^{-1}

HRMS (CI+): m/z found: 281.2148(M+), calculated for $\text{C}_{17}\text{H}_{28}\text{FNO}$: 281,2155

HPLC (column Zorbax RX-SIL, eluent hexane: 2-propanol (0.1% Et_2NH) 99:1, flow 1,0 ml/min), $t_{\text{R}}(\text{rac}) = 15$ min.

viii) 3-(3'-benzyloxy-2'-hydroxy-1'-phenylpropyl-1'-amino)-(1*R*, 2*S*)-1-fluoro-1-phenylpropan-2-ol, 5f:

^1H NMR (CDCl_3 , 500 MHz): 7.35-7.19(+*) (30H, m, ArH), 5.28* (1H, dd, $^2J_{\text{H-F}} = 47.37$ Hz, $^3J_{\text{HH}} = 6.11$ Hz CHF), 5.42 (1H, dd, $^2J_{\text{H-F}} = 47$ Hz, $^3J_{\text{HH}} = 6$ Hz CHF), 4.43* (2H, s, OCH_2Ph), 4.45 (2H, s, OCH_2Ph), 4.0* (1H, m, $\text{CHOH}(\text{CF})$), 3.97(+*) (2H, q, $^3J_{\text{HH}} = 5.13$ Hz $\text{CHOH}(\text{CHPh})$), 3.9 (1H, m, $\text{CHOH}(\text{CF})$), 3.75(+*) (2H, m, CHNHPh), 3.35(+*) (4H, m, CH_2NH), 2.5(+*) (4H, m, CH_2 O CH_2Ph).

^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): 139.24, 139*, 137.8(+*), 136.42(+*)(d, $^2J_{\text{CF}}=20.21$ Hz), 128.9*, 128.80, 128.51(+*), 128.46(+*), 128.41(+*), 127.74(+*), 127.68(+*), 127.51(+*), 126.44*, 126.39*, 126.35, 126.3, 95.8* (d, $^1J_{\text{C-F}} = 172.75$ Hz), 95.4 (d, $^1J_{\text{C-F}} = 173.64$ Hz), 73.5(+*), 73.39 (d, $^2J_{\text{CF}}=22.58$ Hz), 72.80* (d, $^2J_{\text{CF}}=22.58$ Hz), 72.58(+*), 71.42(+*), 65.38(+*), 64.81(+*), 48.52 (d, $^3J = 5.4$ Hz), 48.27* (d, $^3J = 5.5$ Hz).

^{19}F NMR (CDCl_3 , 470.5 MHz): δ -185.9* (dd, $^2J_{\text{FH}} = 48.5$ Hz, $^3J_{\text{FH}} = 17.8$ Hz), -183.51 (dd, $^2J_{\text{FH}} = 47.5$ Hz, $^3J_{\text{FH}} = 17$ Hz).

HRMS (ES⁺): m/z found (M+H): 410.2126, calculated for $\text{C}_{25}\text{H}_{29}\text{FNO}_3$: 410.2131.

HPLC (column Zorbax RX-SIL, eluent hexane: 2-propanol (0,1% Et_2NH) 90:10 to 99:1 and 20 min isocratic, flow 1,0 ml/min,) t_{R} (diastereomer R)=12.6, t_{R} (diastereomer S)= 16.0 min.

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Injection Date   : 4/18/2005 7:17:05 PM      Seq. Line :    3
Sample Name     : dp94                      Location  : Vial 22
Acq. Operator   : serqi                     Inj       :    1
Acq. Instrument : HPLC1100                  Inj Volume: 5 µl
Different Inj Volume from Sequence !      Actual Inj Volume : 10 µl
Acq. Method     : C:\HPCHEM\1\METHODS\PERICAS\SERGI\SRE320_9.M
Last changed    : 4/18/2005 5:32:02 PM by serqi
Analysis Method : C:\HPCHEM\1\METHODS\PERICAS\SERGI\DP94.M
Last changed    : 4/19/2005 10:07:17 AM by DANI
                  (modified after loading)
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                        Area Percent Report
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Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs

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Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	12.655	MM	0.7016	851.72375	56.4964	20.23347
2	16.021	BBA	0.4941	655.84839	43.5036	20.03124

Totals : 1507.57214 40.26470

ix) 3-((1'*R*, 2'*R*)-3'-benzyloxy-2'-hydroxy-1'-phenylpropyl-1'-amino)-(1*R*, 2*S*)-1-fluoro-1-phenylpropan-2-ol, (1*R*, 2*R*)-5f:

^1H NMR (CDCl_3 , 500 MHz): 7.34-7.19 (15H, m, ArH), 5.28 (1H, dd, $^2J_{\text{H-F}} = 47.4$ Hz, $^3J_{\text{HH}} = 6.11$ Hz CHF), 4.43 (2H, dd, $^2J_{\text{HH}} = 11.5$ Hz, OCH_2Ph), 4.01 (1H, m, $\text{CHOH}(\text{CF})$), 3.975 (1H, q, $^3J_{\text{HH}} = 5.13$ Hz $\text{CHOH}(\text{CHPh})$), 3.75 (1H, d, $^3J = 4.89$ Hz, CHNH), 3.34 (2H, m, CH_2NH), 2.5 (1H, dd, $^2J_{\text{HH}} = 12.3$ Hz, $^3J_{\text{HH}} = 7.5$ Hz, CHHOCH_2Ph), 2.38 (1H, dd, $^2J_{\text{HH}} = 12.2$ Hz, $^3J = 3.6$ Hz, CHHOCH_2Ph).

^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): 139, 137.8, 136.4 (d, $^2J_{\text{C-F}} = 20.21$ Hz), 128.93, 128.53, 128.46, 128.4, 127.80, 127.74, 127.61, 126.5, 126.4, 95.80 (d, $^1J_{\text{C-F}} = 173.64$ Hz), 73.52, 72.8 (d, $^2J_{\text{C-F}} = 22.58$ Hz), 72.51, 71.4, 64.81, 48.27 (d, $^3J_{\text{C-F}} = 5.51$ Hz).

^{19}F NMR (CDCl_3 , 470,5 MHz): δ -185.51 (dd, $^2J_{\text{FH}} = 48.5$ Hz, $J^3_{\text{FH}} = 17.7$ Hz).

IR (neat, ATR) ν_{max} : 3375, 3305, 3030, 1452, 1260 cm^{-1}

HRMS (ES+): m/z found: 410.2135(M+H), calculated for $\text{C}_{25}\text{H}_{29}\text{FNO}_3$: 410.2131.

$[\alpha]_{\text{D}} = -31.43$ ($c = 0.51$, CH_2Cl_2).

x) (2*S*,3*R*)-1-((2'(*R*&*S*),3'(*R*&*S*))-*N*-methyl-3'-hydroxy-3'-phenyl-2'-propylamino)-3-fluoro-3-phenylpropan-2-ol, 5g:

^1H NMR (CDCl_3 , 400MHz): δ 7.26-7.43 (20H, m, ArH), 5.19 (1H, dd, $^3J_{\text{HH}} = 5.4$ Hz, $^2J_{\text{HF}} = 46.8$ Hz, CHF), 5.17* (1H, dd, $^3J_{\text{HH}} = 5.5$ Hz, $^2J_{\text{HF}} = 47.1$ Hz, CHF), 4.64 (+*) (2H, d, $^3J_{\text{HH}} = 6.1$ Hz, CHOHPh), 3.83 (+*) (2H, m, CHOHCHF), 2.85 (1H, m, CHCH₃), 2.81* (1H, m, CHCH₃), 2.46 (4H, bs, OH, OH), 2.45 (1H, dd, $^2J_{\text{HH}} = 10.2$ Hz, $^3J_{\text{HH}} = 6.6$ Hz, CHH), 2.42* (1H, dd, $^2J_{\text{HH}} = 10.2$ Hz, $^3J_{\text{HH}} = 7.1$ Hz, CHH), 2.28 (1H, dd, $^2J_{\text{HH}} = 12.7$ Hz, $^3J_{\text{HH}} = 3.8$ Hz, CHH), 2.21* (1H, dd, $^2J_{\text{HH}} = 12.8$ Hz, $^3J_{\text{HH}} = 3.8$ Hz, CHH), 2.22 (3H, s,

HRMS (ES⁺): m/z found: 318.1685 (M+H), calculated for C₁₉H₇NO₂F: 318.1689.

HPLC (column Zorbax RX-SIL, eluent hexane:2-propanol (0.1% Et₂NH) 99:1, flow 1.0 ml/min): t_R(diastereomers)= 10.7 min, 13.5 min (Sample partially resolved by flash chromatography).

Data File C:\HPCHEM\1\DATA\SERGI\SRE389.D

Sample Name: sre389

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Injection Date : 7/11/2005 9:50:58 PM      Seq. Line : 4
Sample Name    : sre389                    Location  : Vial 74
Acq. Operator  : sergi                     Inj      : 1
Acq. Instrument: HPLC1100                  Inj Volume: 5 µl
Acq. Method    : C:\HPCHEM\1\METHODS\PERICAS\SERGI\SREDP.M
Last changed   : 7/11/2005 6:51:09 PM by dana
Analysis Method: C:\HPCHEM\1\METHODS\PERICAS\SERGI\GRADP3.M
Last changed   : 7/15/2005 4:04:10 PM by pineda
                  (modified after loading)
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                          Area Percent Report
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Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
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Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %s	Height [mAU]	Area %
1	10.706	MM	0.6082	952.93726		26.11514	71.6884
2	13.548	MM	0.7281	376.34033		8.61474	28.3116

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Totals :                      1329.27759  34.72989
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xi) (2*S*,3*R*)-1*N*-((*DL*)-phenylalaninate methyl ester)-3-fluoro-3-phenylpropan-2-ol, 5h:

^1H NMR (CDCl_3 , 500 MHz): 7.3-7.05 (+*) (20H, m, ArH), 5.31* (1H, dd, $^2J_{\text{H-F}} = 47.6$ Hz, $^3J_{\text{HH}} = 6.1$ Hz CHF), 5.22 (1H, dd, $^2J_{\text{H-F}} = 47.1$ Hz, $^3J_{\text{HH}} = 5.3$ Hz CHF), 3.79 (+*) (2H, m, CHOH), 3.59* (3H, s, CH_3), 3.57 (3H, s, CH_3), 3.38* (1H, t, $^3J_{\text{HH}} = 7.3$ Hz, CHNH), 3.29 (1H, t, $^3J = 7.9$ Hz CHNH) 2.94 (+*) (m, 8H, CH_2).

^{13}C { ^1H } NMR (CDCl_3 , 125 MHz): 174.7*, 174.53 (C=O), 137.42, 137.05* (C, Ph (CH_2Ph)), 136.54* (d, C, Ph, $^2J_{\text{C-F}} = 22.0$ Hz), 136.5 (d, C, Ph, $^2J_{\text{C-F}} = 19.8$ Hz), 129.1*, 129.25, 128.78*, 128.74, 128.49*, 128.43, 128.86*, 126.82, (+*), 126.32, 126.26, 126.25, 95.36* (d, $^1J_{\text{C-F}} = 174.8$ Hz), 95.25 (d, $^1J_{\text{C-F}} = 174.1$ Hz), 73.28 (d, $^2J_{\text{C-F}} = 22.8$ Hz), 72.07* (d, $^2J_{\text{C-F}} = 24.2$ Hz), 63.39, 62.69*, 51.83, 51.79*, 49.04 (d, $^3J_{\text{C-F}} = 6.6$ Hz), 48.68 (d, $^3J_{\text{C-F}} = 6.0$ Hz), 39.74*, 39.7.

^{19}F NMR (CDCl_3 , 470.73 MHz): δ -188.9* (dd, $^2J_{\text{FH}} = 47.1$ Hz, $J^3_{\text{FH}} = 16.3$ Hz), -189.08 (dd, $^2J_{\text{FH}} = 47.8$ Hz, $^3J_{\text{FH}} = 19.2$ Hz).

IR (neat, ATR) ν_{max} : 3400, 2875, 2750, 1495 cm^{-1}

HRMS (ES $^{+}$): m/z found: 332.165 (M+H); calculated for $\text{C}_{19}\text{H}_{23}\text{NO}_3\text{F}$: 332.166

HPLC/MASS (column Waters Atlantis dC18, 3 μm , 2.1*150mm, eluent MeOH/ H_2O ; 5% to 100% in 30 min hold for 15 min, flow 0.2ml/min, injection volume 10ul, MS detection +ve ESI, extracted ion : (M+Na) t_{R} (diastereomer R.)= 25.38 min, t_{R} (diastereomer S.)= 25.98 min.

xii) (2*S*,3*R*)-1-N-(ethyl 3'-aminobutanoate)-3-fluoro-3-phenylpropan-2-ol,5i:

^1H NMR (CDCl_3 , 400 MHz): 7.4-7.26 (+*) (10H, m, ArH), 5.38* (1H, dd, $^2J_{\text{H-F}} = 46.9$ Hz, $^3J_{\text{HH}} = 5.9$ Hz CHF), 5.37 (1H, dd, $^2J_{\text{H-F}} = 47.7$ Hz, $^3J_{\text{HH}} = 5.9$ Hz CHF), 4.12* (2H, q, $^3J_{\text{HH}} = 7.1$ Hz, CH_2CH_3), 4.11 (2H, q, $^3J_{\text{HH}} = 7.1$ Hz, CH_2CH_3), 3.94 (+*) (m, 2H, CHOH), 3.03 (+*) (m, 2H, $^3J_{\text{HH}} = 6.4$ Hz CHNH), 2.57 (+*) (m, 4H, CH_2OH), 2.34 (+*) (m, 8H, CH_2NH , NH, OH), 1.25* (t, 3H, $^3J_{\text{HH}} = 7.0$ Hz CH_3CH_2), 1.24 (t, 3H, $^3J_{\text{HH}} = 7.2$ Hz CH_3CH_2), 1.07* (d, 3H, $^3J_{\text{HH}} = 6.3$ Hz CH_3CHNH), 1.05 (d, 3H, $^3J_{\text{HH}} = 5.7$ Hz CH_3CHNH).

^{13}C { ^1H } NMR (CDCl_3 , 100 MHz): 172.19, 172.16*, 136.87, 136.67*, 128.76 (+*), 128.47 (+*), 126.42, 126.35*, 95.85 (d, $^1J_{\text{C-F}} = 174.1$ Hz), 95.75* (d, $^1J_{\text{C-F}} = 173.5$ Hz), 72.76* (d, $^2J_{\text{C-F}} = 22.8$ Hz), 72.33 (d, $^2J_{\text{C-F}} = 22.8$ Hz), 60.4 (+*), 50.56*, 50.04, 47.86* (d, $^3J_{\text{C-F}} = 5.6$ Hz), 47.61 (d, $^3J_{\text{C-F}} = 5.6$ Hz), 41.68, 41.51*, 20.54*, 20.27, 14.19*, 14.18.

^{19}F NMR (CDCl_3 , 376.47 MHz): δ -186.95 (dd, $^2J_{\text{FH}} = 47.3$ Hz, $J^3_{\text{FH}} = 16.7$ Hz), -187.1* (dd, $^2J_{\text{FH}} = 47.3$ Hz, $^3J_{\text{FH}} = 16.8$ Hz).

HRMS (ES^+): m/z found: 284.165 ($\text{M}+\text{H}$); calculated for $\text{C}_{15}\text{H}_{23}\text{NO}_3\text{F}$: 284.166

HPLC (column Zorbax RX-SIL, eluent hexane: 2-propanol (0.1% Et₂NH) 99:1, flow 1.0.

Data File C:\HPCHEM\1\DATA\SERGI\DP-152.D

Sample Name: 152

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Injection Date : 7/11/2005 8:59:49 PM      Seq. Line :   3
Sample Name    : 152                      Location  : Vial 73
Acq. Operator  : serqi                    Inj       :   1
Acq. Instrument: HPLC1100                 Inj Volume: 5 µl
Acq. Method    : C:\HPCHEM\1\METHODS\PERICAS\SERGI\SREDP.M
Last changed   : 7/11/2005 6:51:09 PM by dana
Analysis Method: C:\HPCHEM\1\METHODS\PERICAS\SERGI\GRADP3.M
Last changed   : 7/15/2005 3:32:29 PM by pineda
                  (modified after loading)
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Area Percent Report

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Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
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Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	1.823	BV	0.0585	69.57333	17.86835	8.1144
2	1.925	VV	0.0684	18.91005	3.78902	2.2055
3	3.510	VB	0.1265	182.57698	22.55573	21.2941
4	22.811	PV	0.4851	184.64272	5.43640	21.5351
5	23.633	VB	0.8993	401.70142	5.84552	46.8509

Totals : 857.40450 55.49503