## Supporting Information

# Syntheses of (S,S)-Reboxetine via a Catalytic Stereospecific Rearrangement of $\beta$-Amino Alcohols 

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## General

TLC was performed on pre-coated silica gel plates $60 \mathrm{~F}_{254}$ and visualized either with a UV lamp ( 254 nm ), or by using a solution of $\mathrm{KMnO}_{4} / \mathrm{K}_{2} \mathrm{CO}_{3} / \mathrm{NaOH}$ in water followed by heating. Flash chromatography was performed with Si60 silica gel (40-63 $\mu \mathrm{m}$ ). Infrared (IR) spectra were recorded on a IRFT spectrometer and wave-numbers are indicated in $\mathrm{cm}^{-1} \cdot{ }^{1} \mathrm{H}$ NMR spectra were obtained using a 400 MHz spectrometer and data are reported as follows: chemical shift in ppm from tetramethylsilane as an internal standard, multiplicity ( $\mathrm{s}=$ singlet, $\mathrm{d}=$ doublet, $\mathrm{t}=$ triplet, $\mathrm{q}=$ quartet, $\mathrm{m}=$ multiplet or overlap of non equivalent resonances), integration. ${ }^{13} \mathrm{C}$ NMR spectra were obtained using a 100 MHz spectrometer and data are reported as follows: chemical shift in ppm with the solvent as an internal indicator $\left(\mathrm{CDCl}_{3} \delta 77.0 \mathrm{ppm}\right)$, multiplicity with respect to proton (deduced from DEPT experiments, $\mathrm{s}=$ quaternary $\mathrm{C}, \mathrm{d}=\mathrm{CH}, \mathrm{t}=\mathrm{CH}_{2}, \mathrm{q}=$ $\mathrm{CH}_{3}$ ). Mass spectra were obtained by GC/MS with electron impact ionization at 70 eV .

Microwave irradiation experiments were performed using a single-mode Initiator ${ }^{\text {TM }}$ EXP (0$300 \mathrm{~W}, 2.45 \mathrm{GHz}$ ) from Biotage. ${ }^{1}$

## Experimental Procedures

(-)-(1R,2R)-2-N,N-Dibenzylamino-1-phenylpropan-1,3-diol (5). ${ }^{2,3}$ To a mixture of the (-)$(1 R, 2 R)$-2-amino-1-phenyl-1,3-propanediol $4\left(2.04 \mathrm{~g}, 12.2 \mathrm{mmol}, 1.0\right.$ equiv) and $\mathrm{K}_{2} \mathrm{CO}_{3}(5.05 \mathrm{~g}$, 36.6 mmol , 3.0 equiv) in $\mathrm{MeCN}(50 \mathrm{~mL}$ ) was added benzyl bromide ( $2.9 \mathrm{~mL}, 24.4 \mathrm{mmol}, 2.0$ equiv) and the mixture was stirred at reflux for 8 h . The reaction medium was concentrated under reduced pressure and the residue was dissolved in water ( 50 mL ) and EtOAc ( 50 mL ). The aqueous phase was extracted with EtOAc $(3 \times 80 \mathrm{~mL})$ and the combined organic extract was dried over $\mathrm{MgSO}_{4}$, filtered and concentrated in vacuo. Purification of the residue by flash chromatography (silica gel, hexane/AcOEt : 75/25) afforded $4.15 \mathrm{~g}(11.9 \mathrm{mmol}, 98 \%)$ of $\mathbf{5}$ as a white solid ; $\mathrm{C}_{23} \mathrm{H}_{25} \mathrm{NO}_{2} ; \mathrm{MW}=347.45 \mathrm{~g} . \mathrm{mol}^{-1}$; M.p. $103-104{ }^{\circ} \mathrm{C}$; [lit. ${ }^{3} \mathrm{M}$. p. $\left.102.5-104{ }^{\circ} \mathrm{C}\right]$; $[\alpha]_{\mathrm{D}}{ }^{20}=-89.8(c 1$, acetone $)\left[\right.$ lit. ${ }^{3}[\alpha]_{\mathrm{D}}=+92.3(c 1$, acetone $)$ for $(1 S, 2 S)$ isomer $]$; IR (neat) $3362,3028,2923,2855,2360,1494,1452,1229,1198,1117,1063,1052,983,749,731,698$ $\mathrm{cm}^{-1} ;{ }^{1} \mathrm{H} \operatorname{NMR}\left(400 \mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta 7.38-7.15(15 \mathrm{H}), 4.60(\mathrm{~d}, J=9.7 \mathrm{~Hz}, 1 \mathrm{H}), 4.12(\mathrm{bs}, 1 \mathrm{H})$, $4.08(\mathrm{~d}, J=13.0 \mathrm{~Hz}, 2 \mathrm{H}), 3.79(\mathrm{~d}, J=13.0 \mathrm{~Hz}, 2 \mathrm{H}), 3.66(\mathrm{~m}, 1 \mathrm{H}), 3.48(\mathrm{~m}, 1 \mathrm{H}), 2.95(\mathrm{~m}, 1 \mathrm{H})$, 2.36 (bs, 1H) ; ${ }^{13} \mathrm{C}$ NMR ( $100 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 142.0$ ( s ), 139.0 (s), 129.4 (d), 129.1 (d), 128.6 (d), 128.2 (d), 128.1 (d), 127.4 (d), 127.0 (d), 125.3 (d), 71.9 (d), 65.2 (d), 59.2 (t), 54.5 (t).
(-)-(1R,2S)-3-Amino-1-phenylpropan-1,2-diol (6). To a solution of $\mathbf{3}$ (3.3 g, $9.5 \mathrm{mmol}, 1.0$ equiv) in $\mathrm{MeOH}(200 \mathrm{~mL})$ was added $\mathrm{Pd}(\mathrm{OH})_{2} 20 \%(1.3 \mathrm{~g})$ and the mixture was vigorously stirred under an atmosphere of $\mathrm{H}_{2}$ during 26 h . The suspension was filtered through Celite and the filtrate was concentrated in vacuo. Purification of the residue by flash chromatography (silica gel, $\mathrm{CH}_{2} \mathrm{Cl}_{2} / \mathrm{MeOH} / \mathrm{NH}_{4} \mathrm{OH}: 90 / 10 / 1$ to $75 / 25 / 1$ ) afforded $1.12 \mathrm{~g}(6.7 \mathrm{mmol}, 71 \%)$ of $\mathbf{6}$

[^0]as a colorless oil ; $\mathrm{C}_{9} \mathrm{H}_{13} \mathrm{NO}_{2} ; \mathrm{MW}=167.21 \mathrm{~g} \cdot \mathrm{~mol}^{-1} ;[\alpha]^{20}{ }_{\mathrm{D}}=-25.0(c 2.6, \mathrm{MeOH}) ;$ IR (neat) 3600-2200, 1573, 1492, 1451, 1326, 1024, 760, $699 \mathrm{~cm}^{-1} ;{ }^{1} \mathrm{H}$ NMR ( $400 \mathrm{MHz}, \mathrm{CD}_{3} \mathrm{OD}$ ) $\delta$ $7.46-7.27(5 \mathrm{H}), 4.59(\mathrm{~d}, J=6.2 \mathrm{~Hz}, 1 \mathrm{H}), 3.70(\mathrm{~m}, 1 \mathrm{H}), 2.87(\mathrm{dd}, J=13.2,3.4 \mathrm{~Hz}, 1 \mathrm{H}), 2.70$ (dd, $J=13.2,7.9 \mathrm{~Hz}, 1 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR ( $\left.100 \mathrm{MHz}, \mathrm{CD}_{3} \mathrm{OD}\right) \delta 143.6$ (s), 129.1 (d), 128.4 (d), 128.1 (d), 77.3 (d), 76.9 (d), 44.5 (t) ; HRMS (ESI) Calcd for $\mathrm{C}_{9} \mathrm{H}_{14} \mathrm{NO}_{2}\left(\mathrm{M}+\mathrm{H}^{+}\right)$: 168.1019, found: 168.1021 .
(-)-2-Chloro- $N$-((2S,3R)-2,3-dihydroxy-3-phenylpropyl)-acetamide (7). To a solution of $\mathbf{6}$ ( $1.04 \mathrm{~g}, 6.23 \mathrm{mmol}, 1.0$ equiv) and $\mathrm{Et}_{3} \mathrm{~N}\left(1.3 \mathrm{~mL}, 9.3 \mathrm{mmol}, 1.5\right.$ equiv) in $\mathrm{CH}_{2} \mathrm{Cl}_{2} / \mathrm{MeCN}$ (9:1, 100 mL ) at $0{ }^{\circ} \mathrm{C}$ was added dropwise chloroacetyl chloride ( $546 \mu \mathrm{~L}, 6.85 \mathrm{mmol}, 1.1$ equiv). After being stirred at room temperature for 45 min , the reaction was quenched with a solution of $1 \mathrm{~N} \mathrm{HCl}(20 \mathrm{~mL})$. The organic phase was separated, dried over $\mathrm{MgSO}_{4}$, filtered and concentrated in vacuo. Purification of the residue by flash chromatography (silica gel, $\mathrm{CH}_{2} \mathrm{Cl}_{2} / \mathrm{MeOH}$ : 99/1) afforded $1.35 \mathrm{~g}(5.5 \mathrm{mmol}, 89 \%)$ of 7 as a yellow oil $; \mathrm{C}_{11} \mathrm{H}_{14} \mathrm{ClNO}_{3} ; \mathrm{MW}=243.69 \mathrm{~g} \cdot \mathrm{~mol}^{-1}$; $[\alpha]^{20}{ }_{\mathrm{D}}=-13.6\left(c 2.5, \mathrm{CHCl}_{3}\right) ; \mathrm{IR}$ (neat) 3550-3100, 2900-2600, 1647, 1539, 1407, 1260, 1082, 1024, 758, $700 \mathrm{~cm}^{-1} ;{ }^{1} \mathrm{H}$ NMR ( $400 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.36-7.26(5 \mathrm{H}), 7.17$ (broad s, 1H), $4.58(\mathrm{~d}, J=5.7 \mathrm{~Hz}, 1 \mathrm{H}), 4.02($ broad $\mathrm{s}, 1 \mathrm{H}), 3.92(\mathrm{~s}, 2 \mathrm{H}), 3.80(\mathrm{~m}, 1 \mathrm{H}), 3.45-3.34(3 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR (100 MHz, $\mathrm{CDCl}_{3}$ ) $\delta 167.8$ (s), 140.1 ( s ), 128.6 (d), 128.1 (d), 126.6 (d), 75.1 (d), 74.0 (d), 42.5 (t), 41.6 ( t$)$; HRMS (ESI) Calcd for $\mathrm{C}_{11} \mathrm{H}_{14} \mathrm{Cl}^{35} \mathrm{NNaO}_{3}\left(\mathrm{M}+\mathrm{Na}^{+}\right): 266.0554$, found: 266.0555. Calcd for $\mathrm{C}_{11} \mathrm{H}_{14} \mathrm{Cl}^{37} \mathrm{NNaO}_{3}\left(\mathrm{M}+\mathrm{Na}^{+}\right):$268.0528, found: 268.0532 .
(-)-(S)-6-((R)-Hydroxyphenylmethyl)-morpholin-3-one (2). To a solution of $\mathbf{7 ( 1 . 2 1 \mathrm { g } , 4 . 9 7}$ mmol, 1.0 equiv) in $i \operatorname{PrOH}(200 \mathrm{~mL})$ at $0^{\circ} \mathrm{C}$ was added dropwise a $t \mathrm{BuOK}(1.4 \mathrm{~g}, 12.5 \mathrm{mmol}$, 2.5 equiv) solution in $i \operatorname{PrOH}(50 \mathrm{~mL})$. After being stirred at room temperature for 2 h , the reaction was quenched with a 1 N HCl solution $(20 \mathrm{~mL})$ and concentrated in vacuo. The residue was diluted with water $(50 \mathrm{~mL})$ and $\operatorname{AcOEt}(50 \mathrm{~mL})$. The aqueous phase was extracted with AcOEt $(1 \times 50 \mathrm{~mL})$ and the combined organic extract was dried over $\mathrm{MgSO}_{4}$, filtered and
concentrated in vacuo. Purification of the residue by flash chromatography (silica gel, $\left.\mathrm{CH}_{2} \mathrm{Cl}_{2} / \mathrm{MeOH}: 96 / 4\right)$ afforded $639 \mathrm{mg}(3.08 \mathrm{mmol}, 62 \%)$ of 2 as a slightly pink solid ; $\mathrm{C}_{11} \mathrm{H}_{13} \mathrm{NO}_{3} ; \mathrm{MW}=207.23$ g. $\mathrm{mol}^{-1} ;$ M.p. $=126-128{ }^{\circ} \mathrm{C} ;[\alpha]^{20}{ }_{\mathrm{D}}=-62.1\left(c 0.58, \mathrm{CHCl}_{3}\right) ;$ IR (neat) $3500-3100,1659,1624,1492,1347,1132,1085,1015,899,760,694 \mathrm{~cm}^{-1} ;{ }^{1} \mathrm{H}$ NMR ( $400 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.51$ (broad s, 1H), 7.32-7.19 (5H), $4.69(\mathrm{~s}, 1 \mathrm{H}), 4.20(\operatorname{broad} \mathrm{~s}, 1 \mathrm{H}), 4.06$ $(\mathrm{d}, J=16.8 \mathrm{~Hz}, 1 \mathrm{H}), 3.88(\mathrm{~m}, 1 \mathrm{H}), 3.63(\mathrm{~m}, 1 \mathrm{H}), 3.35(\mathrm{~m}, 1 \mathrm{H}), 3.01(\mathrm{~d}, J=11.8 \mathrm{~Hz}, 1 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR ( $100 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 169.6$ (s), 140.2 ( s ), 128.5 (d), 128.0 (d), 126.3 (d), 76.2 (d), 73.5 (d), 67.2 (t), 41.4 (t) ; MS-EI $m / z(\%): 207\left(\mathrm{M}^{+\bullet}, 2\right), 130(5), 107$ (49), 101 (97), 91 (16), 79 (67), 77, (51), 73 (100), 72 (43), 51 (12) ; HRMS (ESI) Calcd for $\mathrm{C}_{11} \mathrm{H}_{13} \mathrm{NNaO}_{3}\left(\mathrm{M}+\mathrm{Na}^{+}\right)$: 230.0788, found: 230.0792 .
(+)-(2S,3R)-2-( $\alpha$-Hydroxyphenylmethyl)morpholine-4-carboxylic acid $t$-butyl ester (8). ${ }^{4}$ To a solution of $\mathbf{2}(50 \mathrm{mg}, 0.24 \mathrm{mmol}, 1.0$ equiv $)$ in THF $(10 \mathrm{~mL})$ at $0^{\circ} \mathrm{C}$ was added dropwise a solution of Red-Al ${ }^{\circledR}$ ( $65 \% \mathrm{w} / \mathrm{w}$ in toluene, $294 \mu \mathrm{~L}, 0.97 \mathrm{mmol}, 4.0$ equiv). After being stirred under reflux for 3 h , the mixture was cooled to $0{ }^{\circ} \mathrm{C}$ and water ( $50 \mu \mathrm{~L}$ ) was added dropwise followed by the addition of an aqueous 4 N KOH solution ( $100 \mu \mathrm{~L}$ ). After being stirred at room temperature for 16 h , the mixture was filtered over Celite and concentrated in vacuo. Purification of the residue by flash chromatography (silica gel, $\mathrm{CH}_{2} \mathrm{Cl}_{2} / \mathrm{MeOH} / \mathrm{NH}_{4} \mathrm{OH}$ : $95 / 5$ to $75 / 25 / 1$ ) afforded $33 \mathrm{mg}(0.17 \mathrm{mmol}, 71 \%)$ of the corresponding amine as a white solid. To a solution of this amine ( $33 \mathrm{mg}, 0.17 \mathrm{mmol}, 1.0$ equiv) in $\mathrm{CH}_{2} \mathrm{Cl}_{2}$ was added ( Boc$)_{2} \mathrm{O}(38 \mathrm{mg}$, $0.17 \mathrm{mmol}, 1.0$ equiv) and the mixture was stirred 4 h at room temperature. After concentration of the mixture under reduced pressure, the residue was purified by flash chromatography (silica gel, $\mathrm{CH}_{2} \mathrm{Cl}_{2} / \mathrm{MeOH}$ : $99 / 1$ ) to give 47 mg ( $0.16 \mathrm{mmol}, 94 \%, 67 \%$ over 2 steps) of $\mathbf{8}$ as a white solid ; $\mathrm{C}_{16} \mathrm{H}_{23} \mathrm{NO}_{4} ; \mathrm{MW}=293.36$ g.mol ${ }^{-1}$; M.p. $=150-152{ }^{\circ} \mathrm{C}\left[\right.$ lit..$^{4}$ M.p. $\left.141-142{ }^{\circ} \mathrm{C}\right] ;[\alpha]^{20}{ }_{\mathrm{D}}$ $=+3.45\left(c 2.35, \mathrm{CHCl}_{3}\right)\left[\right.$ lit. $\left.^{4}[\alpha]^{20}{ }_{\mathrm{D}}=+2.3\left(c 1.27, \mathrm{CHCl}_{3}\right)\right] ;$ IR (neat) 3687, 3440, 2975,

[^1]2902, 1675, 1425, 1366, 1266, 1253, 1221, 1162, 1122, 1058, 1023, 882, 764, $701 \mathrm{~cm}^{-1} ;{ }^{1} \mathrm{H}$ NMR ( $400 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.37-7.24(5 \mathrm{H}), 4.80(\mathrm{~s}, 1 \mathrm{H}), 3.90-3.70(3 \mathrm{H}), 3.60-3.45(2 \mathrm{H}), 2.93-$ $2.77(3 \mathrm{H}), 1.39(\mathrm{~s}, 9 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR ( $\left.100 \mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta 154.9(\mathrm{~s}), 139.7$ (s), 128.4 (d), 127.9 (d), 126.4 (d), 80.0 ( s$), 78.6$ (d), 74.4 (d), 66.7 (t), 43.9 ( t$), 43.1$ (t), 28.3 (q) ; MS-EI $m / z(\%)$ : $293\left(\mathrm{M}^{+\bullet}, 0.6\right), 234$ (1), 187 (41), 176 (6), 131 (79), 130 (74), 116 (52), 107 (26), 86 (63), 57 (100).
(+)-(2S,3S)-2-[ $\alpha$-(2-Ethoxyphenoxy)phenylmethyl]morpholine-4-carboxylic acid $t$-butyl ester (9). ${ }^{4}$ To a solution of $\mathbf{8}\left(47 \mathrm{mg}, 0.16 \mathrm{mmol}, 1.0\right.$ equiv), $\mathrm{PPh}_{3}(84 \mathrm{mg}, 0.32 \mathrm{mmol}, 2.0$ equiv) and 2-ethoxyphenol ( $41 \mu \mathrm{~L}, 0.32 \mathrm{mmol}, 2.0$ equiv) in THF $(5 \mathrm{~mL})$ at $0^{\circ} \mathrm{C}$ was added dropwise DIAD ( $63 \mu \mathrm{~L}, 0.32 \mathrm{mmol}, 2.0$ equiv) and the mixture was stirred 22 h at room temperature. After concentration of the mixture under reduced pressure, the residue was purified by flash chromatography (silica gel, $n$-pentane/AcOEt : 90/10 to $70 / 30$ ) to give $23 \mathrm{mg}(56 \mu \mathrm{~mol}$, $35 \%)$ of $\mathbf{9}$ as a yellow oil ; $\mathrm{C}_{24} \mathrm{H}_{31} \mathrm{NO}_{5} ; \mathrm{MW}=413.51 \mathrm{~g} \cdot \mathrm{~mol}^{-1} ;[\alpha]^{20}{ }_{\mathrm{D}}=+50.0\left(c 1.1, \mathrm{CHCl}_{3}\right)$ $\left[\right.$ lit. $\left.{ }^{4}[\alpha]^{20}{ }_{\mathrm{D}}=+51.0\left(c 1.01, \mathrm{CHCl}_{3}\right)\right]$; IR (neat) 2975, 2853, 1693, 1592, 1498, 1453, 1415, $1365,1250,1215,1167,1109,1043,742,700 \mathrm{~cm}^{-1} ;{ }^{1} \mathrm{H} \operatorname{NMR}\left(400 \mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta 7.40(\mathrm{~d}, J=$ $7.7 \mathrm{~Hz}, 2 \mathrm{H}), 7.34-7.24(3 \mathrm{H}), 6.88-6.82(2 \mathrm{H}), 6.78(\mathrm{~d}, J=7.9 \mathrm{~Hz}, 1 \mathrm{H}), 6.70(\mathrm{~m}, 1 \mathrm{H}), 5.16(\mathrm{~d}, J$ $=5.0 \mathrm{~Hz}, 1 \mathrm{H}), 4.06(\mathrm{q}, J=6.7 \mathrm{~Hz}, 2 \mathrm{H}), 3.96(\mathrm{~d}, J=11.1 \mathrm{~Hz}, 1 \mathrm{H}), 3.90-3.70(3 \mathrm{H}), 3.55(\mathrm{ddd}, J$ $=11.6,11.6,2.2 \mathrm{~Hz}, 1 \mathrm{H}), 3.05-2.75(2 \mathrm{H}), 1.46-1.40(12 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR ( $100 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta$ 154.8 (s), 150.1 (s), 147.7 (s), 137.6 (s), 128.3 (d), 128.1 (d), 127.4 (d), 122.5 (d), 120.8 (d), 118.7 (d), 114.2 (d), 79.9 (s), 78.1 (d), 66.7 (t), 64.6 (t), 28.4 (q), 15.0 (q) ; MS-EI $m / z(\%): 413$ $\left(\mathrm{M}^{+\bullet}, 0.4\right), 340(1), 276(5), 220(52), 176$ (100), 175 (63), 138 (31), 110 (22), 91 (50), 56 (55). (+)-(2S,3S)-2-[ $\alpha$-(2-Ethoxyphenoxy)phenylmethyl]morpholine or (S,S)-reboxetine (1). ${ }^{4}$ To a solution of $9\left(21 \mathrm{mg}, 51 \mu \mathrm{~mol}, 1.0\right.$ equiv) in $\mathrm{CH}_{2} \mathrm{Cl}_{2}(5 \mathrm{~mL})$ was added TFA ( $58 \mu \mathrm{~L}, 0.76$ mmol, 15 equiv). After being stirred at room temperature for 16 h , the reaction was quenched by an aqueous solution of $3.75 \mathrm{M} \mathrm{NaOH}(5 \mathrm{~mL})$. The aqueous phase was extracted with $\mathrm{AcOEt}(3 \times$

10 mL ), and the combined organic extract was dried over $\mathrm{Na}_{2} \mathrm{SO}_{4}$, filtered and concentrated in vacuo. Purification of the residue by flash chromatography (silica gel, $\mathrm{CH}_{2} \mathrm{Cl}_{2} / \mathrm{MeOH}$ : $90 / 10$ ) afforded $14 \mathrm{mg}(45 \mu \mathrm{~mol}, 88 \%)$ of $\mathbf{1}$ as a colorless oil ; $\mathrm{C}_{19} \mathrm{H}_{23} \mathrm{NO}_{3} ; \mathrm{MW}=313.39 \mathrm{~g} \cdot \mathrm{~mol}^{-1}$; $[\alpha]^{20}{ }_{\mathrm{D}}=+11.5\left(c \quad 0.6, \mathrm{CHCl}_{3}\right)\left[\right.$ lit. $\left.{ }^{4}[\alpha]^{20}{ }_{\mathrm{D}}=+13.0\left(c 1.03, \mathrm{CHCl}_{3}\right)\right]$; IR (neat) 3000-2800, $1711,1591,1500,1453,1252,1215,1092,1041,803,777,742,702 \mathrm{~cm}^{-1} ;{ }^{1} \mathrm{H}$ NMR ( 400 MHz , $\left.\mathrm{CD}_{3} \mathrm{OD}\right) \delta 7.41-7.37(2 \mathrm{H}), 7.33-7.23(3 \mathrm{H}), 6.89(\mathrm{dd}, J=8.0,1.6 \mathrm{~Hz}, 1 \mathrm{H}), 6.84-6.79(2 \mathrm{H}), 6.70$ (ddd, $J=8.4,7.2,1.6 \mathrm{~Hz}, 1 \mathrm{H}), 5.18(\mathrm{~d}, J=6.0 \mathrm{~Hz}, 1 \mathrm{H}), 4.10-4.02(2 \mathrm{H}), 3.94-3.88(2 \mathrm{H}), 3.63$ $(\mathrm{m}, 1 \mathrm{H}), 2.78-2.75(2 \mathrm{H}), 2.66-2.56(2 \mathrm{H}), 1.42(\mathrm{t}, J=7.0 \mathrm{~Hz}, 3 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR ( 100 MHz , $\left.\mathrm{CD}_{3} \mathrm{OD}\right) \delta 151.1$ (s), 149.1 (s), 139.1 ( s$), 129.3$ (d), 129.2 (d), 128.7 (d), 123.3 (d), 122.1 (d), 118.8 (d), 115.6 (d), 83.8 (d), 80.0 (d), 68.4 (t), 65.9 (t), 47.8 (t), 46.0 ( $t$ ), 15.4 (q) ; MS-EI $m / z$ (\%) : $313\left(\mathrm{M}^{+\bullet}, 2\right), 175$ (100), 138 (10), 104 (18), 91 (43), 56 (57).
(+)-Benzoic acid (2R,3R)-2-benzoylamino-3-hydroxy-3-phenylpropyl ester (14). To a solution of $(-)-(1 R, 2 R)$-2-amino-1-phenyl-1,3-propanediol $4(1.0 \mathrm{~g}, 6.0 \mathrm{mmol}, 1.0$ equiv) and $\mathrm{Et}_{3} \mathrm{~N}$ ( $2.1 \mathrm{~mL}, 15.0 \mathrm{mmol}, 2.5$ equiv) in THF ( 55 mL ) at $0{ }^{\circ} \mathrm{C}$ was added dropwise benzoyl chloride ( $1.46 \mathrm{~mL}, 12.6 \mathrm{mmol}, 2.1$ equiv). After being stirred at room temperature for 24 h , the mixture was cooled to $0^{\circ} \mathrm{C}$ and the reaction was quenched by a 2 N HCl solution ( 25 mL ). The aqueous phase was extracted with $\operatorname{AcOEt}(2 \times 20 \mathrm{~mL})$ and the combined organic extract was dried over $\mathrm{Na}_{2} \mathrm{SO}_{4}$, filtered and concentrated in vacuo. Purification of the residue by flash chromatography (silica gel, $n$-pentane/AcOEt : 60/40) afforded $2.16 \mathrm{~g}(5.7 \mathrm{mmol}, 96 \%)$ of $\mathbf{1 4}$ as a white solid ; $\mathrm{C}_{23} \mathrm{H}_{21} \mathrm{NO}_{4} ; \mathrm{MW}=375.42$ g.mol ${ }^{-1} ;$ M.p. $=170-172{ }^{\circ} \mathrm{C} ;[\alpha]^{20}{ }_{\mathrm{D}}=+8.6(c 0.54$, THF) ; IR (neat) $3355,1723,1635,1530,1270,1123,1027,709 \mathrm{~cm}^{-1} ;{ }^{1} \mathrm{H}$ NMR ( 400 MHz , DMSO-D6) $\delta 8.24(\mathrm{~d}, J=8.7 \mathrm{~Hz}, 1 \mathrm{H}), 7.93(\mathrm{~m}, 2 \mathrm{H}), 7.76(\mathrm{~m}, 2 \mathrm{H}), 7.64(\mathrm{~m}, 1 \mathrm{H}), 7.50(\mathrm{t}, J=$ $8.2 \mathrm{~Hz}, 3 \mathrm{H}), 7.45-7.40(4 \mathrm{H}), 7.32(\mathrm{t}, J=7.5 \mathrm{~Hz}, 2 \mathrm{H}), 7.23(\mathrm{~m}, 1 \mathrm{H}), 5.75(\mathrm{~d}, J=5.3 \mathrm{~Hz}, 1 \mathrm{H})$, $4.98(\mathrm{dd}, J=5.0,5.0 \mathrm{~Hz}, 1 \mathrm{H}), 4.63(\mathrm{~m}, 1 \mathrm{H}), 4.45(\mathrm{dd}, J=11.0,4.9 \mathrm{~Hz}, 1 \mathrm{H}), 4.24(\mathrm{dd}, J=11.0$, $8.0 \mathrm{~Hz}, 1 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR (100 MHz, DMSO-D6) $\delta 166.7$ (s), 165.7 (s), 142.5 (s), 134.6 (s), 133.3
(d), 131.1 (d), 129.7 (s), 129.2 (d), 128.7 (d), 128.2 (d), 127.9 (d), 127.3 (d), 127.1 (d), 126.4 (d), 71.3 (d), 64.3 (t), 54.1 (d) ; HRMS (ESI) Calcd for $\mathrm{C}_{23} \mathrm{H}_{21} \mathrm{NNaO}_{4}\left(\mathrm{M}+\mathrm{Na}^{+}\right): 398.1363$, found: 398.1357.
(+)-Benzoic acid (2R,3R)-1-benzoyl-3-phenylaziridin-2-ylmethyl ester (15). To a solution of 14 ( $200 \mathrm{mg}, 0.53 \mathrm{mmol}, 1.0$ equiv), $\mathrm{PPh}_{3}(280 \mathrm{mg}, 1.07 \mathrm{mmol}, 2.0$ equiv), and $2-$ ethoxyphenol ( $101 \mu \mathrm{~L}, 0.8 \mathrm{mmol}, 1.5$ equiv) in THF $(5 \mathrm{~mL})$ at $0^{\circ} \mathrm{C}$ was added DIAD $(210 \mu \mathrm{~L}$, $1.07 \mathrm{mmol}, 2.0$ equiv). After being stirred at room temperature for 1 h , the mixture was concentrated in vacuo. Purification of the residue by flash chromatography (silica gel, $n$ pentane/AcOEt : 85/15) afforded $145 \mathrm{mg}(0.41 \mathrm{mmol}, 76 \%)$ of $\mathbf{1 5}$ as white solid; $\mathrm{C}_{23} \mathrm{H}_{19} \mathrm{NO}_{3}$; MW $=357.40 \mathrm{~g} . \mathrm{mol}^{-1} ;$ M.p. $=109-110^{\circ} \mathrm{C} ;[\alpha]^{20}{ }_{\mathrm{D}}=+120\left(c 0.49, \mathrm{CHCl}_{3}\right) ;$ IR (neat) 2925, $1710,1670,1449,1290,1266,1247,1100,1069,734,698 \mathrm{~cm}^{-1} ;{ }^{1} \mathrm{H}$ NMR ( $400 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta$ 8.11-8.08 (2H), 8.01-7.98 (2H), 7.58-7.45 (4H), 7.45-7.33 (7H), 4.34 (dd, $J=12.0,7.0 \mathrm{~Hz}$, $1 \mathrm{H}), 4.28(\mathrm{dd}, J=12.0,5.5 \mathrm{~Hz}, 1 \mathrm{H}), 3.95(\mathrm{~d}, J=6.4 \mathrm{~Hz}, 1 \mathrm{H}), 3.35$ (apparent $\mathrm{q}, J=6.6 \mathrm{~Hz}, 1 \mathrm{H})$; ${ }^{13} \mathrm{C}$ NMR (100 MHz, $\mathrm{CDCl}_{3}$ ) $\delta 179.0$ (s), 166.3 (s), 133.8 (s), 133.3 (d), 133.2 (d), 132.4 (s), 129.9 (d), 129.8 ( s ), 129.4 (d), 128.8 (d), 128.6 (d), 128.5 (d), 128.3 (d), 127.6 (d), 62.8 (t), 43.3 (d), 41.7 (d) ; MS-EI $m / z(\%): 357\left(\mathrm{M}^{+\bullet}, 1\right), 252(5), 235(59), 222(18), 193$ (40), 130 (29), 105 (100), 91 (13), 77 (32) ; HRMS (ESI) Calcd for $\mathrm{C}_{23} \mathrm{H}_{20} \mathrm{NO}_{3}\left(\mathrm{M}+\mathrm{H}^{+}\right)$: 358.1438, found : 358.1440.
(+)-Benzoic acid (2R,3S)-2-benzoylamino-3-(2-ethoxyphenoxy)-3-phenylpropyl ester (13). To a solution of $\mathrm{PPh}_{3}$ ( $281 \mathrm{mg}, 1.07 \mathrm{mmol}$, 2.0 equiv), freshly distilled 2-ethoxyphenol ( 1.35 $\mathrm{mL}, 10.7 \mathrm{mmol}, 20$ equiv) in THF $(4 \mathrm{~mL})$ at $0^{\circ} \mathrm{C}$ was added DIAD $(211 \mu \mathrm{~L}, 1.07 \mathrm{mmol}, 2.0$ equiv). After being stirred at room temperature for $30 \mathrm{~min}, 14(200 \mathrm{mg}, 0.53 \mathrm{mmol}, 1.0$ equiv) was added in one portion and the mixture was stirred for 2 h at room temperature. After concentration of the mixture under reduced pressure, the residue was purified by flash chromatography (silica gel, $n$-pentane/AcOEt : 95/5) to give $202 \mathrm{mg}(0.41 \mathrm{mmol}, 76 \%)$ of $\mathbf{1 3}$ as
a white solid ; $\mathrm{C}_{31} \mathrm{H}_{29} \mathrm{NO}_{5} ;$ MW $=495.57$ g.mol ${ }^{-1} ;$ M.p. $=149-151{ }^{\circ} \mathrm{C} ;[\alpha]^{20}{ }_{\mathrm{D}}=+35.1(c 1.0$, $\mathrm{CHCl}_{3}$ ) ; IR (neat) $3256,3064,2927,1718,1639,1497,1272,1252,1201,1116,702,689 \mathrm{~cm}^{-1}$ ; ${ }^{1} \mathrm{H}$ NMR ( $400 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.95(\mathrm{dd}, J=7.9,0.8 \mathrm{~Hz}, 2 \mathrm{H}), 7.71(\mathrm{~m}, 2 \mathrm{H}), 7.56-7.29$ ( 12 H ), 6.95-6.89 (2H), 6.77-6.71 (2H), $5.46(\mathrm{~d}, J=3.5 \mathrm{~Hz}, 1 \mathrm{H}), 5.02(\mathrm{~m}, 1 \mathrm{H}), 4.77(\mathrm{dd}, J=11.6,8.0$ $\mathrm{Hz}, 1 \mathrm{H}), 4.42(\mathrm{dd}, J=11.6,4.1 \mathrm{~Hz}, 1 \mathrm{H}), 4.09(\mathrm{qm}, J=6.9 \mathrm{~Hz}, 2 \mathrm{H}), 1.36(\mathrm{t}, J=6.9 \mathrm{~Hz}, 3 \mathrm{H})$; ${ }^{13} \mathrm{C}$ NMR ( $100 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 167.5$ (s), 166.8 (s), 149.4 (s), 148.8 ( s ), 138.0 ( s ), 134.6 (s), 133.0 (d), 131.5 (d), 129.9 (s), 129.8 (d), 128.9 (d), 128.5 (d), 128.3 (d), 128.2 (d), 127.1 (d), 126.0 (d), 122.8 (d), 121.3 (d), 117.7 (d), 113.8 (d), 84.1 (d), 68.0 (t), 64.7 (t), 54.2 (d), 14.8 (q) ; MS-EI $m / z(\%): 373\left(\mathrm{M}^{+\bullet}-\mathrm{C}_{6} \mathrm{H}_{5} \mathrm{CO}_{2} \mathrm{H}, 1\right), 281$ (1), 267 (3), 236 (44), 227 (64), 133 (23), 105 (32), 91 (100), 77 (24) ; Anal. Calcd for $\mathrm{C}_{31} \mathrm{H}_{29} \mathrm{NO}_{5}: \mathrm{C}, 75.13$; H, 5.90 ; N, 2.83, found : C, 74.87 ; H, 6.19 ; N, 2.54.
(+)-(2R,3S)-2-Benzylamino-3-(2-ethoxyphenoxy)-3-phenylpropan-1-ol (16). To a solution of $\mathbf{1 3}\left(898 \mathrm{mg}, 1.8 \mathrm{mmol}, 1.0\right.$ equiv) in THF $(40 \mathrm{~mL})$ at $0^{\circ} \mathrm{C}$ was added $\mathrm{BH}_{3} . \mathrm{THF}(1 \mathrm{M}$ in THF ; $10.8 \mathrm{~mL}, 10.8 \mathrm{mmol}, 6.0$ equiv). After being stirred at reflux for 3 h , the reaction was quenched by addition of $\mathrm{MeOH}(10 \mathrm{~mL})$ at room temperature and concentrated in vacuo. The residue was then dissolved in a 1 M HCl solution $(40 \mathrm{~mL})$ and stirred at reflux for 2 h . The mixture was made basic $(\mathrm{pH}>10)$ by addition of a 3.75 M NaOH solution, and extracted with AcOEt $(2 \times 30 \mathrm{~mL})$. The combined organic extract was dried over $\mathrm{Na}_{2} \mathrm{SO}_{4}$, filtered and concentrated in vacuo. Purification of the residue by flash chromatography (silica gel, $\mathrm{CH}_{2} \mathrm{Cl}_{2}$ then $\mathrm{CH}_{2} \mathrm{Cl}_{2} / \mathrm{MeOH}$ : 99/1) afforded $632 \mathrm{mg}(1.67 \mathrm{mmol}, 92 \%)$ of $\mathbf{1 6}$ as a yellow oil ; $\mathrm{C}_{24} \mathrm{H}_{27} \mathrm{NO}_{3} ; \mathrm{MW}=377.48$ g. $\mathrm{mol}^{-1} ;[\alpha]^{20}{ }_{\mathrm{D}}=+5.79\left(c 1.9, \mathrm{CHCl}_{3}\right) ;$ IR (neat) 3550-3150, 3050-2700, 1592, 1499, 1452, $1249,1215,1122,1040,735,698 \mathrm{~cm}^{-1} ;{ }^{1} \mathrm{H}$ NMR ( $400 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.36-7.19(10 \mathrm{H}), 6.87-$ $6.84(2 \mathrm{H}), 6.68(\mathrm{~m}, 1 \mathrm{H}), 6.58(\mathrm{~d}, J=7.7 \mathrm{~Hz}, 1 \mathrm{H}), 5.29(\mathrm{~d}, J=4.5 \mathrm{~Hz}, 1 \mathrm{H}), 4.11-4.00(2 \mathrm{H}), 3.90$ $(\mathrm{d}, J=13.2 \mathrm{~Hz}, 1 \mathrm{H}), 3.85(\mathrm{dd}, J=11.4,4.1 \mathrm{~Hz}, 1 \mathrm{H}), 3.80(\mathrm{~d}, J=13.2 \mathrm{~Hz}, 1 \mathrm{H}), 3.65(\mathrm{dd}, J=$ $11.4,4.7 \mathrm{~Hz}, 1 \mathrm{H}), 2.97(\mathrm{ddd}, J=4.3,4.3,4.3 \mathrm{~Hz}, 1 \mathrm{H}), 1.95(\operatorname{broad~s}, 1 \mathrm{H}), 1.44(\mathrm{t}, J=7.0 \mathrm{~Hz}$,
$3 \mathrm{H}) ;{ }^{13} \mathrm{C} \operatorname{NMR}\left(100 \mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta 149.1$ (s), 147.5 (s), 140.1 (s), 139.1 (s), 128.7 (d), 128.4 (d), 128.1 (d), 127.9 (d), 127.0 (d), 126.4 (d), 121.9 (d), 120.6 (d), 115.7 (d), 112.7 (d), 83.8 (d), 64.2 (t), 63.0 (d), 60.2 (t), 51.2 (t), 14.8 (q) ; Anal. Calcd for $\mathrm{C}_{24} \mathrm{H}_{27} \mathrm{NO}_{3}: \mathrm{C}, 76.36 ; \mathrm{H}, 7.21$; N, 3.71, found : C, $76.44 ; \mathrm{H}, 7.31 ; \mathrm{N}, 3.87$.
(+)-(2R,3S)-2-[Benzyl-(2-hydroxyethyl)-amino]-3-(2-ethoxyphenoxy)-3-phenylpropan-1ol (12). To a solution of $\mathbf{1 6}\left(178 \mathrm{mg}, 0.47 \mathrm{mmol}, 1.0\right.$ equiv) and $\mathrm{K}_{2} \mathrm{CO}_{3}(398 \mathrm{mg}, 2.9 \mathrm{mmol}, 6.1$ equiv) in DMF ( 5 mL ) was added dropwise methyl bromoacetate ( $205 \mu \mathrm{~L}, 2.17 \mathrm{mmol}, 4.6$ equiv). After being stirred at room temperature for 7 days, the mixture was diluted with water ( 100 mL ) and extracted with $\mathrm{Et}_{2} \mathrm{O}(3 \times 25 \mathrm{~mL})$. The combined organic extract was dried over $\mathrm{MgSO}_{4}$ and concentrated in vacuo. A solution of the residue in dry THF ( 15 mL ) was added dropwise to a suspension of $\mathrm{LiAlH}_{4}\left(56 \mathrm{mg}, 1.47 \mathrm{mmol}, 3.1\right.$ equiv) in dry $\mathrm{THF}(15 \mathrm{~mL})$ at $0{ }^{\circ} \mathrm{C}$. After being stirred at room temperature for 2 h , the mixture was cooled to $0^{\circ} \mathrm{C}$ and water ( 65 $\mu \mathrm{L}$ ) was added dropwise followed by the addition of a 3.75 M NaOH solution ( $65 \mu \mathrm{~L}$ ) and finally water ( $165 \mu \mathrm{~L}$ ). After being stirred at room temperature for 16 h , the mixture was filtered over Celite and concentrated in vacuo. Purification of the residue by flash chromatography (silica gel, $n$-pentane/AcOEt : 80/20) afforded $138 \mathrm{mg}(0.33 \mathrm{mmol}, 70 \%)$ of $\mathbf{1 2}$ as a colorless oil $; \mathrm{C}_{26} \mathrm{H}_{31} \mathrm{NO}_{4} ; \mathrm{MW}=421.53 \mathrm{~g} . \mathrm{mol}^{-1} ;[\alpha]^{20}{ }_{\mathrm{D}}=+37.5\left(c \quad 1.5, \mathrm{CHCl}_{3}\right) ;$ IR (neat) 3600-3100, 3050-2650, 1639, 1592, 1500, 1453, 1249, 1216, 1123, 1040, 909, 731, $699 \mathrm{~cm}^{-1} ;{ }^{1} \mathrm{H}$ NMR ( $400 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.35-7.29(5 \mathrm{H}), 7.23-7.18(3 \mathrm{H}), 7.06-7.03(2 \mathrm{H}), 6.87-6.84(2 \mathrm{H}), 6.65(\mathrm{~m}$, $1 \mathrm{H}), 6.56(\mathrm{~m}, 1 \mathrm{H}), 5.18(\mathrm{~d}, J=8.1 \mathrm{~Hz}, 1 \mathrm{H}), 4.14-4.05(3 \mathrm{H}), 4.00(\mathrm{dd}, J=11.8,6.1 \mathrm{~Hz}, 1 \mathrm{H})$, $3.84(\mathrm{~d}, J=14.0 \mathrm{~Hz}, 1 \mathrm{H}), 3.69(\mathrm{~d}, J=14.0 \mathrm{~Hz}, 1 \mathrm{H}), 3.46(\mathrm{~m}, 1 \mathrm{H}), 3.35(\mathrm{~m}, 1 \mathrm{H}), 3.28(\mathrm{~m}, 1 \mathrm{H})$, $2.85-2.81(2 \mathrm{H}), 1.48(\mathrm{t}, J=7.0 \mathrm{~Hz}, 3 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR $\left(100 \mathrm{MHz}, \mathrm{CDCl}_{3}\right) \delta 149.1(\mathrm{~s}), 146.9(\mathrm{~s})$, 140.2 (s), 138.9 (s), 128.7 (d), 128.6 (d), 128.4 (d), 128.2 (d), 127.2 (d), 127.1 (d), 122.1 (d), 120.6 (d), 116.4 (d), 112.4 (d), 82.5 (d), 66.5 (d), 64.2 (t), 60.3 ( $t), 59.1$ (t), 55.0 ( $t$ ), 51.9 ( $t)$, 14.8 (q) ; HRMS (ESI) Calcd for $\mathrm{C}_{26} \mathrm{H}_{32} \mathrm{NO}_{4}\left(\mathrm{M}+\mathrm{H}^{+}\right): 422.2326$, found : 422.2326.
(+)-(S)-4-Benzyl-2-[(S)-(2-ethoxyphenoxy)phenylmethyl]morpholine or (+)-(S,S)-N-
Benzyl-reboxetine (10). ${ }^{5,6}$ To a solution of $\mathbf{1 1}\left(11 \mathrm{mg}, 26 \mu \mathrm{~mol}, 1.0\right.$ equiv), $\mathrm{Et}_{3} \mathrm{~N}(7 \mu \mathrm{~L}, 52$ $\mu \mathrm{mol}, 2.0$ equiv) and DMAP ( $1 \mathrm{mg}, 8 \mu \mathrm{~mol}, 0.3$ equiv) in $\mathrm{CH}_{2} \mathrm{Cl}_{2}(1 \mathrm{~mL})$ at $0{ }^{\circ} \mathrm{C}$ was added $\mathrm{TsCl}(5.0 \mathrm{mg}, 26 \mu \mathrm{~mol}, 1.0$ equiv). After being stirred at room temperature for $24 \mathrm{~h}, \mathrm{TsCl}(10$ $\mathrm{mg}, 52 \mu \mathrm{~mol}, 2.0$ equiv) and DMAP ( $6 \mathrm{mg}, 52 \mu \mathrm{~mol}, 2.0$ equiv) were added by portions untill complete disappearence of the starting material. After being stirred at room temperature for 24 h, the reaction was quenched by a 3.75 M NaOH solution ( 2 mL ) and vigorously stirred at room temperature for 24 h . The mixture was diluted with water ( 10 mL ) and extracted with AcOEt (2 $\times 10 \mathrm{~mL}$ ). The combined organic extract was dried over $\mathrm{MgSO}_{4}$, filtered and concentrated in vacuo. Purification of the residue by flash chromatography (silica gel, $n$-pentane/AcOEt : 90/10) afforded $6 \mathrm{mg}(15 \mu \mathrm{~mol}, 57 \%)$ of $\mathbf{1 0}$ as a colorless oil $; \mathrm{C}_{26} \mathrm{H}_{29} \mathrm{NO}_{3} ; \mathrm{MW}=403.51 \mathrm{~g} \cdot \mathrm{~mol}^{-1}$; $[\alpha]^{20}{ }_{\mathrm{D}}=+14.1\left(c 0.3, \mathrm{CHCl}_{3}\right) ;$ IR (neat) 3050-2750, 1723, 1593, 1500, 1454, 1381, 1254, $1215,1166,1120,1043,749,702 \mathrm{~cm}^{-1} ;{ }^{1} \mathrm{H}$ NMR ( $400 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 7.39-7.36(2 \mathrm{H}), 7.30-$ $7.21(8 \mathrm{H}), 6.83-6.81(2 \mathrm{H}), 6.77(\mathrm{~m}, 1 \mathrm{H}), 6.70(\mathrm{~m}, 1 \mathrm{H}), 5.17(\mathrm{~d}, J=5.7 \mathrm{~Hz}, 1 \mathrm{H}), 4.06-4.01$ (3H), 3.92 (ddd, $J=11.4,2.3,2.3 \mathrm{~Hz}, 1 \mathrm{H}), 3.67$ (ddd, $J=10.9,10.9,2.4 \mathrm{~Hz}, 1 \mathrm{H}), 3.51(\mathrm{~d}, J=$ $13.0 \mathrm{~Hz}, 1 \mathrm{H}), 3.34(\mathrm{~d}, J=13.0 \mathrm{~Hz}, 1 \mathrm{H}), 2.61-2.58(2 \mathrm{H}), 2.17-2.05(2 \mathrm{H}), 1.38(\mathrm{t}, J=7.0 \mathrm{~Hz}$, 3H) ; ${ }^{13} \mathrm{C}$ NMR ( $100 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) $\delta 150.1$ (s), 148.2 (s), 138.2 (s), 137.8 (s), 129.1 (d), 128.2 (d), 128.1 (d), 127.9 (d), 127.4 (d), 127.1 (d), 122.2 (d), 121.0 (d), 118.4 (d), 114.6 (d), 82.8 (d), 78.4 (d), 66.8 (t), 64.8 (t), 63.4 (t), 55.0 (t), 52.7 (t), $15.0(\mathrm{q})$; MS-EI $m / z(\%)$ : $403\left(\mathrm{M}^{+\bullet}, 2\right), 266$ (100), 181 (7), 176 (9), 175 (8), 91 (66), 56 (9).

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[^0]:    ${ }^{1}$ For more information see: www.biotage.com
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