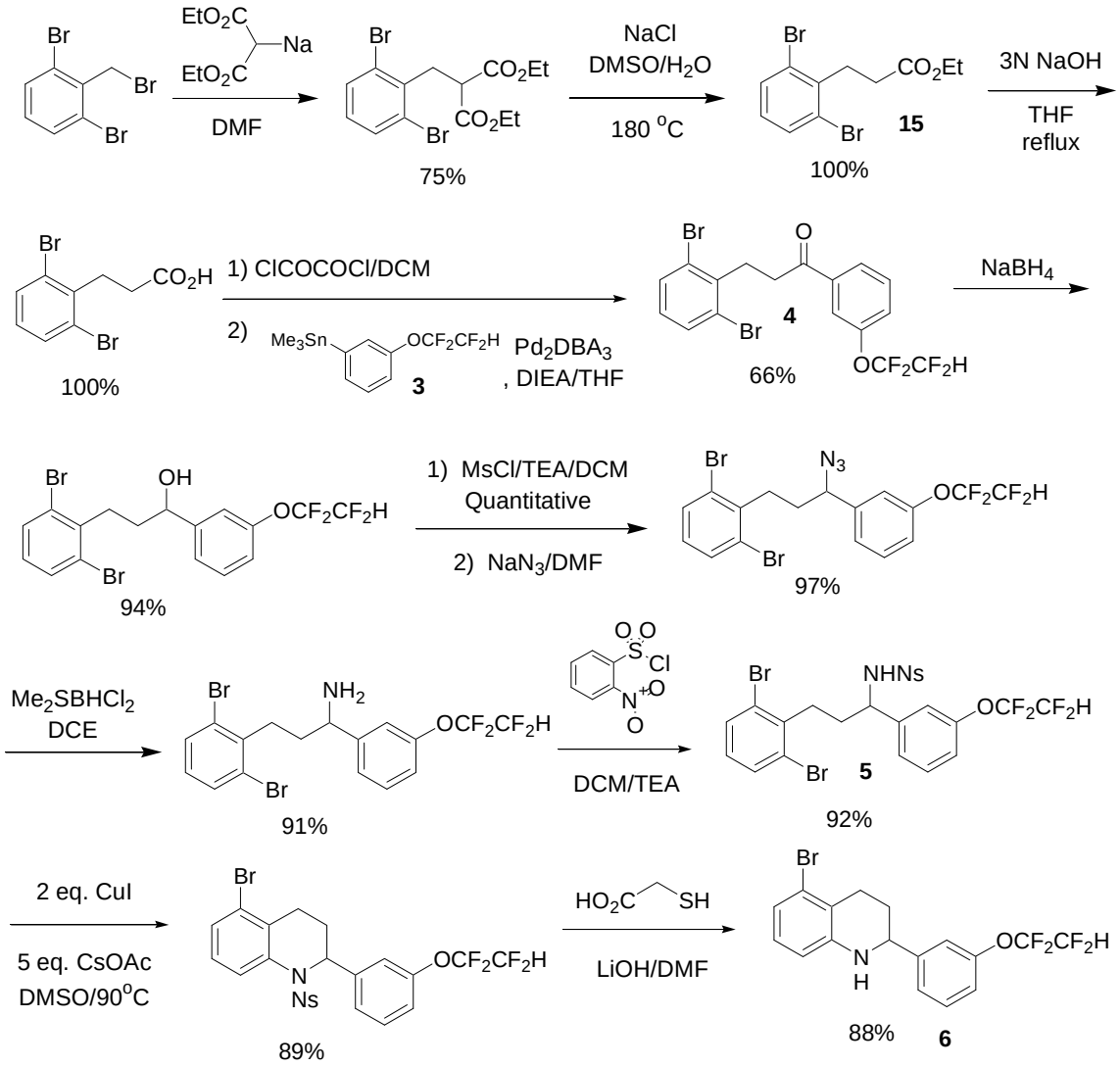
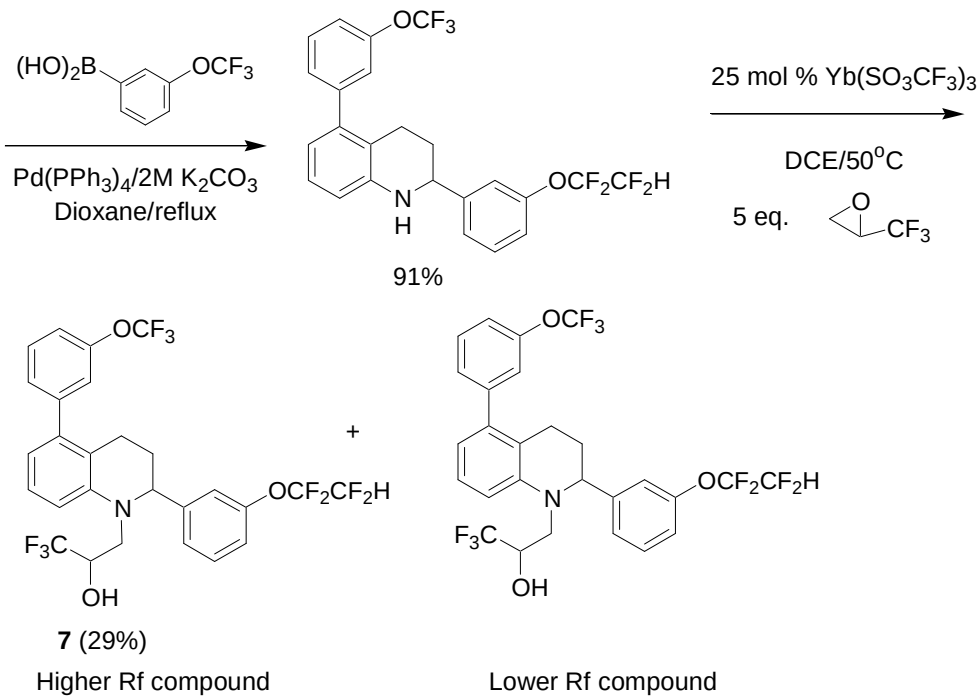
An Improved Asymmetric Synthesis of 4-dihydro-2-[3-(1,1,2,2-tetrafluoroethoxy)phenyl]-5-[3-(trifluoromethoxy)-phenyl]-α-(trifluoromethyl)-1(2*H*)-quinolineethanol, a Potent Cholesteryl Ester Transfer Protein (CETP) Inhibitor

Thomas A. Rano*, Gee-Hong Kuo

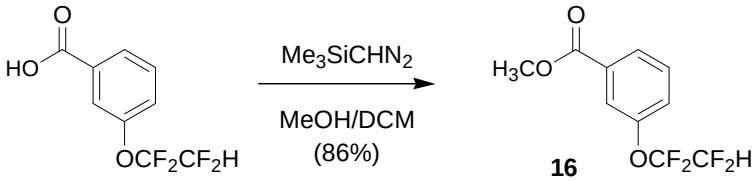
Supporting Information

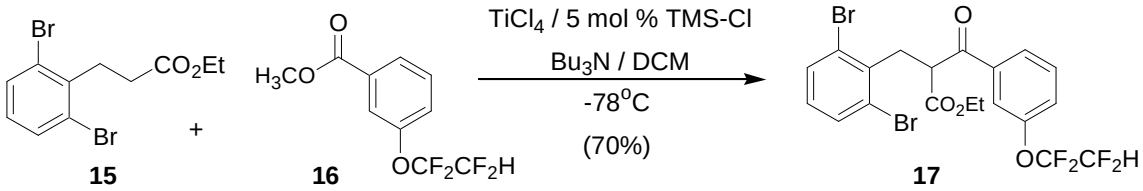
Scheme 1

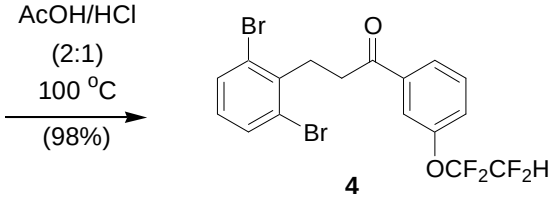

Br
Br
Br
EtO2C
Na
EtO2C
DMF
CO2Et
CO2Et
75%
NaCl
DMSO/H2O
180 °C
CO2Et
100%
15
3N NaOH
THF
reflux
CO2H
100%
1) ClCOCOCl/DCM
2)
Me3Sn
OCF2CF2H
3
Pd2DBA3
, DIEA/THF
O
4
66%
NaBH4
OH
94%
1) MsCl/TEA/DCM
Quantitative
2) NaN3/DMF
N3
97%
Me2SBHCl2
DCE
NH2
91%
Cl
N
O
DCM/TEA
NHNs
5
92%
2 eq. CuI
5 eq. CsOAc
DMSO/90°C
N
Ns
89%
HO2C
SH
LiOH/DMF
N
H
88%
6

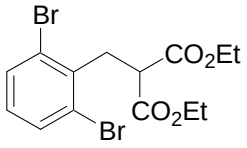


Alternate Route to 4



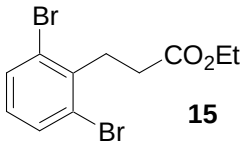






2-(2,6-Dibromo-benzyl)-malonic acid diethyl ester

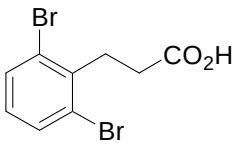
To a solution of sodium diethyl malonate (2.40 g, 13.2 mmol) in DMF (15 mL) under a N_2 atmosphere was added 1,3-dibromo-2-bromomethyl-benzene (4.56 g, 13.9 mmol). After stirring at room temperature for 2 h, ether was added and the solution was washed with H_2O and brine, dried ($MgSO_4$), concentrated and purified by column chromatography to afford 4.28 g (75%) the bis-ester as an oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51 (d, *J* = 8.0 Hz, 2 H), 6.95 (t, *J* = 8.0 Hz, 1 H), 4.18 (q, *J* = 7.1 Hz, 4 H), 3.84 (t, *J* = 7.7 Hz, 1 H), 3.63 (d, *J* = 7.7 Hz, 2 H), 1.21 (t, *J* = 7.1 Hz, 6 H); MS (ES) m/z: 409 ($M+H^+$).



15

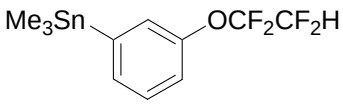
3-(2,6-Dibromo-phenyl)-propionic acid ethyl ester

A mixture of the bis-ester (4.2 g, 10.3 mmol), sodium chloride (602 mg, 10.3 mmol) and H_2O (371 mg, 20.6 mmol) in DMSO (75 mL) was heated at 180 ^{o}C for 1 h. After cooling to room temperature, the reaction mixture was poured into EtOAc (500 mL) and washed with H_2O and brine, dried ($MgSO_4$) and concentrated to afford 3.49 g (100%) of **15** as an oil : ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51 (d, *J* = 8.0 Hz, 2 H), 6.93 (t, *J* = 8.0 Hz, 1 H), 4.18 (q, *J* = 7.1 Hz, 2 H), 3.33 (m, 2 H), 2.57 (m, 2 H), 1.28 (t, *J* = 7.1 Hz, 3 H); MS (ES) m/z: 337 ($M+H^+$).



3-(2,6-Dibromo-phenyl)-propionic acid

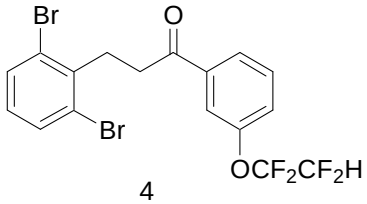
A mixture of **15** (3.46 g, 10.3 mmol) and 3 M sodium hydroxide (25 mL, 75 mmol) in THF (25 mL) was heated at reflux for 5 h. Upon cooling to 0 ^{o}C, the reaction mixture was acidified with concentrated HCl followed by extraction with EtOAc. The combined organic phases were then washed with brine, dried ($MgSO_4$) and concentrated to afford 3.29 g (100%) the acid as a white solid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.52 (d, *J* = 8.0 Hz, 2 H), 6.95 (t, *J* = 8.0 Hz, 1 H), 3.35 (m, 2 H), 2.65 (m, 2 H); MS (ES) m/z: 307 ($M\text{-}H^{+}$).



3

Trimethyl-[3-(1,1,2,2-tetrafluoro-ethoxy)-phenyl]-stannane

A mixture of 1-bromo-3-(1,1,2,2-tetrafluoro-ethoxy)-benzene (3.87 g, 14.1 mmol), hexamethylditin (5.11 g, 15.6 mmol), PPh_3 (110 mg, 0.423 mmol) in toluene (70 mL) under a N_2 atmosphere was degassed by bubbling N_2 through the solution for 15 min. $Pd(PPh_3)_4$ (814 mg, 0.7 mmol) was added and the reaction mixture was heated at 80 ^{o}C for 2 h. After cooling to room temperature, the reaction mixture was poured into EtOAc (500 mL). The solution was then washed with H_2O and brine, dried ($MgSO_4$), concentrated and purified by column chromatography (2% EtOAc/Hex) to afford 3.76 g (67%) **3** as an oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37 – 7.29 (m, 3 H), 7.16 – 7.13 (m, 1 H), 5.91 (tt, *J* = 53.2, 2.9 Hz, 1 H), 0.31 (s, 9 H).



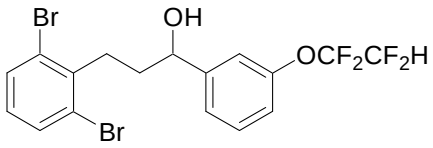
4

3-(2,6-Dibromo-phenyl)-1-[3-(1,1,2,2-tetrafluoro-ethoxy)-phenyl]-propan-1-one

To a solution of the acid (3.27 g, 10.6 mmol) in CH_2Cl_2 (45 mL) under a N_2 atmosphere was added 2M oxalyl chloride in CH_2Cl_2 (7.95 mL, 15.9 mmol). After

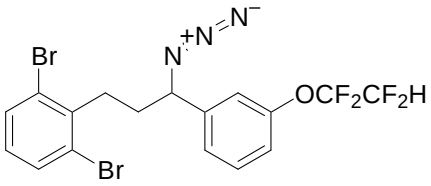
stirring at room temperature for 18 h, the solution was concentrated to give 3.26 g of acid chloride **2** which was used without further purification.

To a solution of the above intermediate (3.26 g, 9.98 mmol) in dry THF (50 mL) at 0 ^{o}C under a N_2 atmosphere was added N,N-diisopropylethylamine (2.6 mL, 15.0 mmol), **3** (4.27 g, 12.0 mmol) and $Pd_2(dba)_3$ (456 mg, 0.499 mmol). After heating at 50 ^{o}C for about 30 min, the reaction mixture was cooled and poured into EtOAc (300 mL) and washed with saturated $NaHCO_3$, H_2O and brine, dried ($MgSO_4$), concentrated and purified by column chromatography (5% EtOAc/Hex) to afford 3.20 g (66%) of **4** as an oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.94 – 7.89 (m, 1 H), 7.83 (s, 1 H), 7.55 – 7.40 (m, 4 H), 6.96 (t, *J* = 8.0 Hz, 1 H), 5.93 (tt, *J* = 53.0, 2.8 Hz, 1 H), 3.47 – 3.40 (m, 2 H), 3.25 – 3.20 (m, 2 H); MS (ES) m/z: 485 ($M+H^+$).



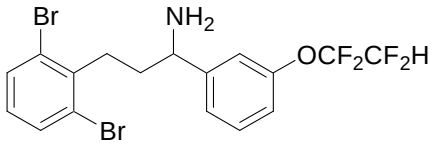
3-(2,6-Dibromo-phenyl)-1-[3-(1,1,2,2-tetrafluoro-ethoxy)-phenyl]-propan-1-ol

To a solution of **4** (3.04 g, 6.27 mmol) in EtOH (50 mL) under a N_2 atmosphere was added $NaBH_4$ (118 mg, 12.5 mmol). After 1 h the reaction was cooled to 0 ^{o}C and quenched with several drops of glacial AcOH. The EtOH was evaporated and the residue was dissolved in EtOAc. The organic phase was washed with saturated $NaHCO_3$, water and brine, dried ($MgSO_4$), concentrated and purified by column chromatography (10%-15%-20% EtOAc/Hex) to provide 2.89 g (95%) of the alcohol as an oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 (d, *J* = 8.0 Hz, 2 H), 7.41 – 7.28 (m, 3 H), 7.14 (d, *J* = 7.7 Hz, 1 H), 6.89 (t, *J* = 8.0 Hz, 1 H), 5.91 (tt, *J* = 53.1, 2.8 Hz, 1 H), 4.86 (dd, *J* = 10.1, 6.2 Hz, 1 H), 3.19 – 3.05 (m, 1 H), 3.03 – 2.92 (m, 1 H), 2.09 – 1.95 (m, 3 H); MS (ES) m/z: 509 ($M+Na^+$).



To a solution of the alcohol (2.89 g, 5.94 mmol) in CH_2Cl_2 (30 mL) under a N_2 atmosphere at 0 ^oC was added triethylamine (1.66 mL, 11.9 mmol) and methanesulfonyl chloride (0.690 mL, 8.9 mmol). The cooling bath was removed and the solution was stirred at room temperature for 2 h. The reaction mixture was poured into EtOAc and washed with 1 N HCl, water, saturated $NaHCO_3$ and brine. The organic layer was dried ($MgSO_4$) and concentrated to give the mesylate as a crude intermediate.

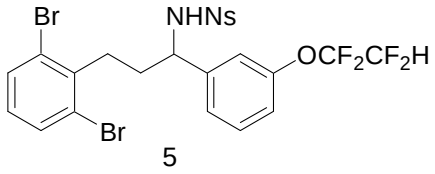
A mixture of the above crude mesylate and sodium azide (1.93 g, 29.7 mmol) in DMF (45 mL) under a N_2 atmosphere was heated at 50 ^oC for ~ 1 h. After cooling to room temperature, the reaction mixture was poured into EtOAc (500 mL), the solution was then washed with H_2O, saturated $NaHCO_3$ solution and brine, dried ($MgSO_4$) and concentrated to afford 2.90 g (95% for two steps) the azide as an oil: ^1H NMR (300 MHz, $CDCl_3$) δ 7.48 (d, *J* = 8.0 Hz, 2 H), 7.42 (t, *J* = 7.9 Hz, 1 H), 7.31 – 7.19 (m, 3 H), 6.90 (t, *J* = 8.0 Hz, 1 H), 5.92 (tt, *J* = 53.1, 2.8 Hz, 1 H), 4.60 (t, *J* = 6.9 Hz, 1 H), 3.15 – 3.05 (m, 1 H), 2.99 – 2.82 (m, 1 H), 2.09 – 1.97 (m, 2 H); MS (ES) m/z: 484 ($M-N_2+H^+$).



3-(2,6-Dibromo-phenyl)-1-[3-(1,1,2,2-tetrafluoro-ethoxy)-phenyl]-propylamine

To a solution of the azide (2.90 g, 5.67 mmol) in 1,2-dichloroethane (38 mL) under a N_2 atmosphere was added $Me_2S{\cdot}BHCl_2$ (1.64 mL, 14.2 mmol) dropwise. The solution was stirred at room temperature for 0.5 h and then heated at 50 ^oC for 1.5 h. The reaction was cooled to 0 ^oC, then 6 N HCl (10 mL) was added. The reaction mixture was then heated at reflux for 1 h. Upon cooling to 0 ^oC, the solution was basified with 3 N NaOH and extracted several times with

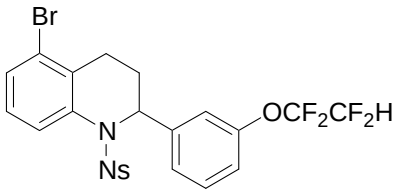
$CHCl_3$. The combined organic phases were dried ($MgSO_4$), concentrated and purified by column chromatography (100% EtOAc) to provide 2.69 g (98%) the amine as an oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.46 (d, *J* = 8.2 Hz, 2 H), 7.39 – 7.26 (m, 3 H), 7.14 – 7.10 (m, 1 H), 6.88 (t, *J* = 8.0 Hz, 1 H), 5.91 (tt, *J* = 53.1, 2.9 Hz, 1 H), 4.08 (t, *J* = 6.6 Hz, 1 H), 3.09 – 3.00 (m, 1 H), 2.90 – 2.80 (m, 1 H), 1.98 – 1.88 (m, 2 H), 1.57 (brs, 2 H); MS (ES) m/z: 486 ($M+H^+$).



5

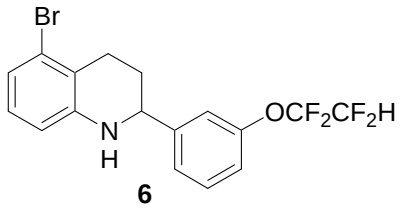
N-{3-(2,6-Dibromo-phenyl)-1-[3-(1,1,2,2-tetrafluoro-ethoxy)-phenyl]-propyl}-2-nitro-benzenesulfonamide

To a solution of the amine (2.67 g, 5.50 mmol) and triethylamine (1.53 mL, 11.0 mmol) in dichloromethane (27 mL) under a N_2 atmosphere was added NsCl (1.34 g, 6.05 mmol) under N_2. The reaction mixture was stirred at room temperature for 1 h and then poured into EtOAc / Et_2O. The solution was washed with saturated $NaHCO_3$, H_2O and brine, dried ($MgSO_4$), concentrated and purified by column chromatography (5%-10%-15%-20% EtOAc/Hex) to afford 3.54 g (95%) **5** as an oil: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.0 Hz, 1 H), 7.66 (d, *J* = 7.9 Hz, 1 H), 7.55 – 7.33 (m, 4 H), 7.13 – 7.08 (m, 2 H), 7.01 (s, 1 H), 6.95 – 6.88 (m, 2 H), 5.96 (d, *J* = 8.9 Hz, 1 H), 5.86 (tt, *J* = 53.1, 2.8 Hz, 1 H), 4.69 (dd, *J* = 16.0, 7.8 Hz, 1 H), 3.19 – 3.11 (m, 1 H), 2.88 – 2.80 (m, 1 H), 2.14 – 1.94 (m, 2 H); MS (ES) m/z: 693 ($M+Na^+$).



5-Bromo-1-(2-nitro-benzenesulfonyl)-2-[3-(1,1,2,2-tetrafluoro-ethoxy)-phenyl]-1,2,3,4-tetrahydro-quinoline

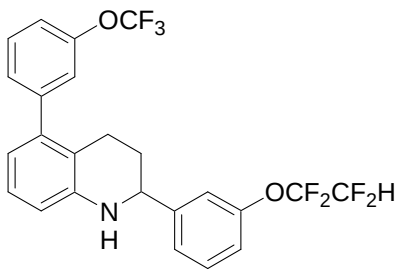
A mixture of **5** (3.54 g, 5.26 mmol), CuI (2.00 g, 10.5 mmol) and CsOAc (5.04 g, 26.3 mmol) in DMSO (52 mL) under a N_2 atmosphere was heated at 95 ^oC for 24 h. After cooling to room temperature, the reaction mixture was poured into EtOAc (400 mL), washed with saturated NH_4Cl (3x), water, $Na_2S_2O_3$ solution and brine, dried ($MgSO_4$) concentrated and purified by column chromatography (25% EtOAc/Hex) to afford 2.99 g (96%) Ns-THQ as an oil: ^1H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.1 Hz, 1 H), 7.73 – 7.69 (m, 1 H), 7.63 – 7.50 (m, 3 H), 7.43 (d, *J* = 8.0 Hz, 1 H), 7.39 – 7.09 (m, 5 H), 5.88 (tt, *J* = 53.1, 2.9 Hz, 1 H), 5.62 (t, *J* = 6.9 Hz, 1 H), 2.74 – 2.66 (m, 1 H), 2.47 – 2.39 (m, 1 H), 2.35 – 2.27 (m, 1 H), 2.05 – 1.96 (m, 1 H); MS (ES) m/z: 589 (M).



6

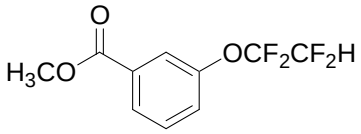
5-Bromo-2-[3-(1,1,2,2-tetrafluoro-ethoxy)-phenyl]-1,2,3,4-tetrahydro-quinoline

To a solution of the Ns-THQ (2.99 g, 5.06 mmol) in DMF (25 mL) under a N_2 atmosphere was added thioacetic acid (0.707 mL, 10.1 mmol) and powdered LiOH (485 mg, 20.2 mmol). The reaction mixture was stirred at room temperature for ~ 6 h and then poured into EtOAc, washed with saturated $NaHCO_3$, H_2O and brine, dried ($MgSO_4$), concentrated and purified by column chromatography (25% EtOAc/Hex) to afford 1.80 g (88%) racemic THQ **6** as an oil: ^1H NMR (300 MHz, $CDCl_3$) δ 7.37 (t, *J* = 7.8 Hz, 1 H), 7.30 – 7.21 (m, 2 H), 7.15 (d, *J* = 7.9 Hz, 1 H), 6.95 – 6.71 (m, 2 H), 6.51 (d, *J* = 7.8 Hz, 1 H), 5.90 (tt, *J* = 53.1, 2.8 Hz, 1 H), 4.40 (dd, *J* = 9.3, 3.1 Hz, 1 H), 4.13 (brs, 1 H), 2.88 – 2.79 (m, 2 H), 2.21 – 2.11 (m, 1 H), 2.05 – 1.90 (m, 1 H); MS (ES) m/z: 406 (M+2).



2-[3-(1,1,2,2-Tetrafluoro-ethoxy)-phenyl]-5-(3-trifluoromethoxy-phenyl)-1,2,3,4-tetrahydro-quinoline

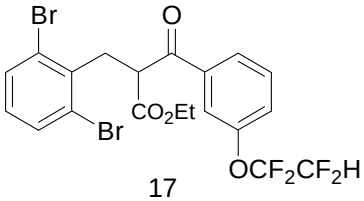
Under a N_2 atmosphere, a mixture of the **6** (30 mg, 0.074 mmol), 3-trifluoromethoxy-phenyl-boronic acid (30 mg, 0.148 mmol), $Pd(PPh_3)_4$ (9 mg, 0.0074 mmol) and 2 N K_2CO_3 (0.11 mL, 0.22 mmol) in 1,4-dioxane (0.75 mL) was heated at reflux for 2 h. After cooling to room temperature, EtOAc was added and the solution was washed with Na_2HCO_3, H_2O and brine. The organic layer was dried ($MgSO_4$), concentrated and purified by column chromatography to give 33 mg (91%) of the biphenyl-THQ as a clear oil: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.30 (m, 4 H), 7.25 (m, 1 H), 7.21 – 7.08 (m, 4 H), 6.62 (s, 1 H), 6.60 (s, 1 H), 5.90 (tt, *J* = 53.1, 2.8 Hz, 1 H), 4.51 (dd, *J* = 8.9, 3.3 Hz, 1 H), 4.20 (brs, 1 H), 2.81 – 2.71 (m, 1 H), 2.53 (dt, *J* = 16.6, 4.8 Hz, 1 H), 2.10 – 2.02 (m, 1 H), 1.92 – 1.82 (m, 1 H); MS (ES) m/z: 486 ($M+H^+$).



16

3-(1,1,2,2-Tetrafluoro-ethoxy)-benzoic acid methyl ester

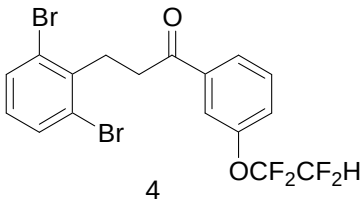
To 3-(1,1,2,2-tetrafluoro-ethoxy)-benzoic acid (10 g; 41.9 mmol) in 20 mL of DCM and 30 mL of MeOH cooled to 0^{o}C was added TMS-diazomethane (2M; 35 mL). The reaction was stirred for 10 minutes, followed by removal of the solvent *in vacuo*. Purification by column chromatography provided 9.1 g (86%) of **16**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.94 (s, 3H), 5.93 (tt, *J* = 53.1, 2.8 Hz, 1 H),7.40-7.50 (m. 2H), 7.88 (s, 1H), 7.97 (d, *J*=8.9 Hz, 1H).



17

2-(2,6-Dibromo-benzyl)-3-oxo-3-[3-(1,1,2,2-tetrafluoro-ethoxy)-phenyl]-propionic acid ethyl ester

To the mono ester (308 mg; 0.95 mmol) and **16** (725 mg; 2.87 mmol) in anhydrous PhMe (2.5 mL) under an atmosphere of N_2 cooled to 0^{o}C was added $TiCl_4$ (2.87 mL; 2.87 mmol), TMSOTf (8.6 uL; 0.0475 mmol) and Bu_3N (1.01 mL; 4.27 mmol). The ice bath was removed. After 10 minutes, A2 was completely consumed. The ice bath was replaced and the reaction was quenched with water. EtOAc was added and the mixture separated. The organic layer was washed with water (2X), saturated sodium bicarbonate solution (2X), water and brine. The organic layer was dried ($MgSO_4$), concentrated and purified by column chromatography (0-5% EtOAc/Hexanes) to provide **17** (364 mg) in 70% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.09(t, 3H), 3.69 ½ ABX (J_{ab}=14.4 Hz, J_{ax}=5.9 Hz, 1H), 3.80 ½ ABX (J_{ab}=14.4 Hz, J_{ax}=8.6 Hz, 1H), 4.08-4.15 (m, 2H), 4.70 (dd, J=8.5, 6.1 Hz, 1H), 5.93 (tt, J = 53.1, 2.8 Hz, 1 H), 6.91 (t, J=8.0 Hz, 1H), 7.37-7.50 (m, 4H), 7.75 (s, 1H), 7.78 (d, J=7.46 Hz 1H).

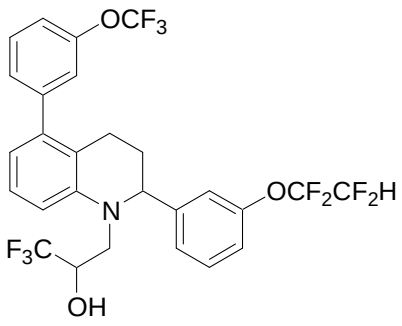


4

3-(2,6-Dibromo-phenyl)-1-[3-(1,1,2,2-tetrafluoro-ethoxy)-phenyl]-propan-1-one

17 (369 mg; 0.663 mmol) was heated in a 2:1 mixture of glacial AcOH and concentrated HCl (5 mL) for 1 hour under an atmosphere of N_2. After cooling, water was added and extraction with EtOAc followed. The organic layer was washed with water (3X), 1N NaOH (1X), water (1X) and brine. The organic layer

was dried ($MgSO_4$) and concentrated to provide **4** (~305 mg) in 95% yield. The compound was identical in all respects to **4** which was prepared employing the original method.

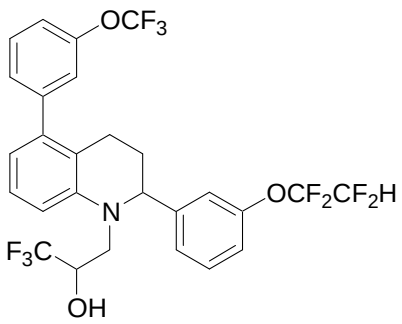


7

Higher Rf compound

1,1,1-Trifluoro-3-[2-[3-(1,1,2,2-tetrafluoro-ethoxy)-phenyl]-5-(3-trifluoromethoxy-phenyl)-3,4-dihydro-2*H*-quinolin-1-yl]-propan-2-ol

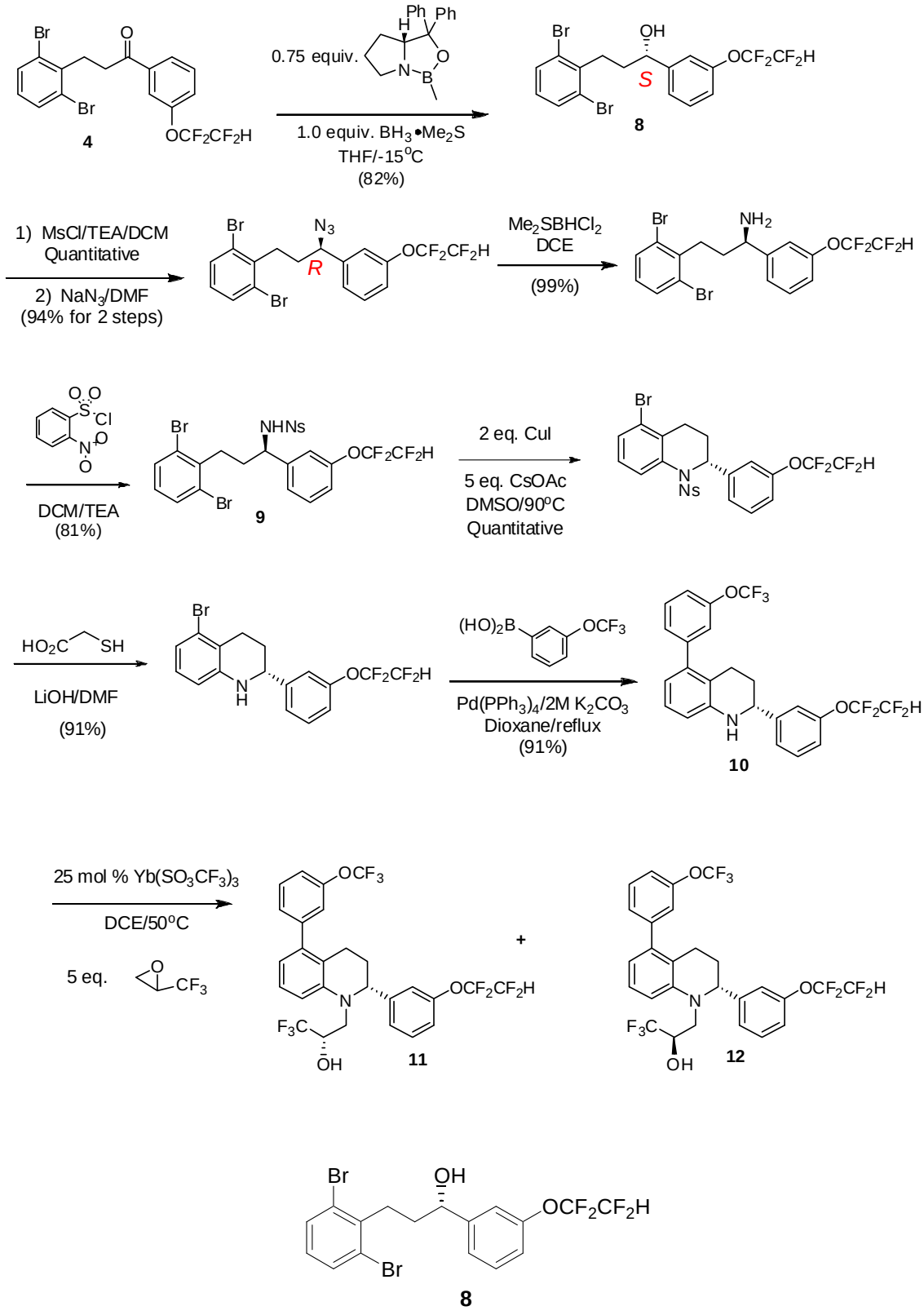
To a solution of the biphenyl-THQ (33 mg, 0.068 mmol) and 1,1,1-trifluoro-2,3-epoxy-propane (38 mg, 0.34 mmol) in DCE (0.45 mL) under a N_2 atmosphere was added $Yb(OTf)_3$ (10.5 mg, 0.0169 mmol). The reaction mixture was heated at 50 ^oC for 48 h and then cooled to ambient temperature. EtOAc was added and the solution was washed with saturated $NaHCO_3$, H_2O and brine, dried ($MgSO_4$), concentrated and purified by column chromatography to afford 12 mg (29%) of **7** as an oil: ^1H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.20 (m, 2 H), 7.28 – 7.10 (m, 6 H), 7.04 (s, 1 H), 6.73 (d, *J* = 8.3 Hz, 1 H), 6.67 (d, *J* = 7.4 Hz, 1 H), 5.89 (tt, *J* = 53.1, 2.8 Hz, 1 H), 4.89 (t, *J* = 4.4 Hz, 1 H), 4.42 (m, 1 H), 3.91 (d, *J* = 15.5 Hz, 1 H), 3.30 (dd, *J* = 15.6, 9.7 Hz, 1 H), 2.48 (dt, *J* = 16.3, 4.4 Hz, 1 H), 2.42 – 2.31 (m, 2 H), 2.19 – 2.09 (m, 1 H), 2.00 – 1.92 (m, 1 H); MS (ES) m/z: 598 ($M+H^+$).



Lower Rf compound
1,1,1-Trifluoro-3-[2-[3-(1,1,2,2-tetrafluoro-ethoxy)-phenyl]-5-(3-trifluoromethoxy-phenyl)-3,4-dihydro-2*H*-quinolin-1-yl]-propan-2-ol

The lower Rf compound was isolated as the other diasteriomer (27%) in the synthesis of **7**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.32 (m, 2 H), 7.28 – 7.09 (m, 6 H), 7.02 (s, 1 H), 6.89 (d, *J* = 8.3 Hz, 1 H), 6.68 (d, *J* = 7.4 Hz, 1 H), 5.89 (tt, *J* = 53.1, 2.8 Hz, 1 H), 4.61 (t, *J* = 4.3 Hz, 1 H), 4.34 (m, 1 H), 3.80 (dd, *J* = 15.7, 6.5 Hz, 1 H), 3.51 (dd, *J* = 15.7, 5.4 Hz, 1 H), 2.48 – 2.33 (m, 2 H), 2.24 (d, *J* = 5.0 Hz, 1 H), 2.17 – 2.08 (m, 1 H), 1.99 – 1.91 (m, 1 H); MS (ES) m/z: 598 ($M+H^+$).

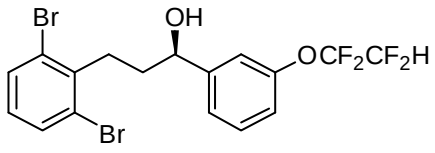
Scheme 2



8

(αS)-2,6-Dibromo-α-[3-(1,1,2,2-tetrafluoroethoxy)phenyl]benzenepropanol

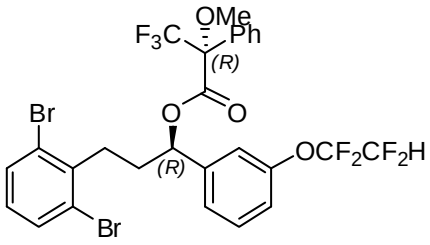
To a stirred solution of **4** (511 mg; 1.05 mmol) in anhydrous THF under nitrogen was added (*R*)-2-Methyl-CBS-oxazaborolidine (792 uL; 0.792 mmol). The reaction vessel was cooled to –15^{o}C followed by the addition of borane-dimethyl sulfide complex (528 uL; 1.05 mmol) slowly dropwise. The reaction was aged for 50 minutes before being quenched with MeOH at –20^{o}C. The contents of the reaction vessel were poured into EtOAc and washed with water/2N HCl (2:1), water, saturated sodium bicarbonate solution, water and brine. The organic layer was dried over $MgSO_4$, filtered and the solvent removed *in vacuo*. Purification employing SiO_2 flash column chromatography (15%EtOAc/Hex) provided 420 mg (82%) of alcohol **8** of the *S* absolute configuration as an oil. Analysis by chiral HPLC (Chiralcel AS; Isocratic elution 90/10 Hexane/IPA) by area integration at 210 nm indicated the enantiomeric excess > 95%. This alcohol was identical in all respects to the racemic alcohol, except for the optical rotation. $[\alpha]_D^{20}$ = -12.1^{o} (c1; $CHCl_3$); The reaction was later repeated on a larger scale, employing 15.36 grams (31.7 mmol) of **4**, 23.7 mL (0.75 equiv.) of (*R*)-2-Methyl-CBS-oxazaborolidine, and 15.85 mL (1 equiv.; 31.7 mmol) of borane-dimethyl sulfide complex in 130 mL of anhydrous THF under the conditions described above to provide 14.33 grams (92%) of alcohol **8**. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 (d, *J* = 8.0 Hz, 2 H), 7.41 – 7.28 (m, 3 H), 7.14 (d, *J* = 7.7 Hz, 1 H), 6.89 (t, *J* = 8.0 Hz, 1 H), 5.91 (tt, *J* = 53.1, 2.8 Hz, 1 H), 4.86 (dd, *J* = 10.1, 6.2 Hz, 1 H), 3.19 – 3.05 (m, 1 H), 3.03 – 2.92 (m, 1 H), 2.09 – 1.95 (m, 3 H); MS (ES) m/z: 509 (M+Na$^+$).



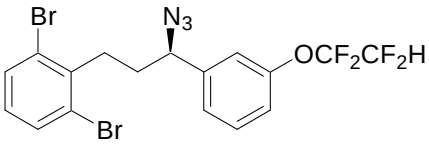
(αR)-2,6-Dibromo-α-[3-(1,1,2,2-tetrafluoroethoxy)phenyl]benzenepropanol

The ***R*** alcohol **8** was prepared exactly as ***S*** alcohol **8**, substituting (*S*)-2-Methyl-CBS-oxazaborolidine for (*R*)-2-Methyl-CBS-oxazaborolidine. Analysis by chiral HPLC (Chiralcel AS; Isocratic elution 90/10 Hexane/IPA) by area integration at

210 nm indicated the enantiomeric excess > 95%. This alcohol was identical in all respects to the racemic alcohol A6, except for the optical rotation. $[\alpha]_D^{20}$ = +11.4^o (c1; CHCl3); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 (d, *J* = 8.0 Hz, 2 H), 7.41 – 7.28 (m, 3 H), 7.14 (d, *J* = 7.7 Hz, 1 H), 6.89 (t, *J* = 8.0 Hz, 1 H), 5.91 (tt, *J* = 53.1, 2.8 Hz, 1 H), 4.86 (dd, *J* = 10.1, 6.2 Hz, 1 H), 3.19 – 3.05 (m, 1 H), 3.03 – 2.92 (m, 1 H), 2.09 – 1.95 (m, 3 H); MS (ES) m/z: 509 ($M+Na^+$).



To a stirred solution of ***S*** alcohol **8** (7.5 mg; 0.0154 mmol) in anhydrous DCM under nitrogen was added DIEA (8 uL; 0.046 mmol) followed by ***S-(+)-***MTPA (5.76 uL; 0.031 mmol). The reaction was aged for 30 minutes before the solvent removed *in vacuo*. Purification employing SiO_2 flash column chromatography (5%EtOAc/Hex) provided quantitative yield of the ***R,R*** Mosher Ester. The ***R,S*** Mosher Ester was prepared in exactly the same manner. The NMR spectra of these Mosher Esters are pictured in the NMR section.

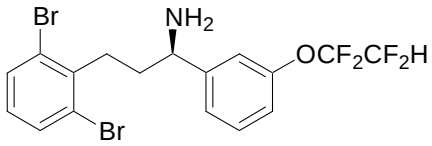


2-[(3R)-3-Azido-3-[3-(1,1,2,2-tetrafluoroethoxy)phenyl]propyl]-1,3-dibromobenzene

To a solution of the ***S*** alcohol **8** (14.33 g, 29.48 mmol) in CH_2Cl_2 (200 mL) under a N_2 atmosphere at 0 ^{o}C was added DIEA (10.27 mL, 58.9 mmol) and methanesulfonyl chloride (3.42 mL, 44.22 mmol). The cooling bath was removed and the solution was stirred at room temperature for 2 h. The reaction mixture was poured into EtOAc and washed with 1 N HCl, water, saturated $NaHCO_3$ and

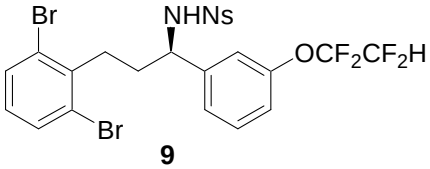
brine. The organic layer was dried ($MgSO_4$) and concentrated to give the mesylate as a crude intermediate.

A mixture of the above crude mesylate and sodium azide (9.5 g, 65.01 mmol) in DMF (150 mL) under a N_2 atmosphere was heated at 50 ^oC for ~ 1 h. After cooling to room temperature, the reaction mixture was poured into EtOAc (1.5 L), the solution was then washed with H_2O, saturated $NaHCO_3$ solution and brine, dried ($MgSO_4$) and concentrated to afford 13.91 g (92% for two steps). This azide was identical in all respects to the racemic azide, except for the optical rotation. $[\alpha]_D^{20}$ = +39.3^o (c1; $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.48 (d, *J* = 8.0 Hz, 2 H), 7.42 (t, *J* = 7.9 Hz, 1 H), 7.31 – 7.19 (m, 3 H), 6.90 (t, *J* = 8.0 Hz, 1 H), 5.92 (tt, *J* = 53.1, 2.8 Hz, 1 H), 4.60 (t, *J* = 6.9 Hz, 1 H), 3.15 – 3.05 (m, 1 H), 2.99 – 2.82 (m, 1 H), 2.09 – 1.97 (m, 2 H); MS (ES) m/z: 484 ($M-N_2+H^+$).



(αR)-2,6-Dibromo-α-[3-(1,1,2,2-tetrafluoroethoxy)phenyl]benzenepropanamine

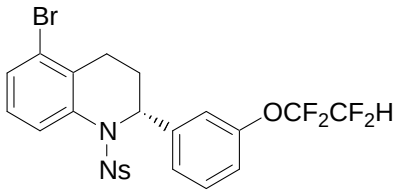
To a solution of the azide (13.91 g, 27.2 mmol) in 1,2-dichloroethane (180 mL) under a N_2 atmosphere was added $Me_2S{\cdot}BHCl_2$ (7.85 mL, 68 mmol) dropwise. The solution was stirred at room temperature for 0.5 h and then heated at 50 ^oC for 1.5 h. The reaction was cooled to 0 ^oC, then 6 N HCl (50 mL) was added. The reaction mixture was then heated at reflux for 1 h. Upon cooling to 0 ^oC, the solution was basified with 3 N NaOH and extracted several times with $CHCl_3$. The combined organic phases were dried ($MgSO_4$), concentrated and purified by column chromatography (100% EtOAc) to provide 13.1 g (99%) of the amine as an oil. ^1H NMR (300 MHz, $CDCl_3$) δ 7.46 (d, *J* = 8.2 Hz, 2 H), 7.39 – 7.26 (m, 3 H), 7.14 – 7.10 (m, 1 H), 6.88 (t, *J* = 8.0 Hz, 1 H), 5.91 (tt, *J* = 53.1, 2.9 Hz, 1 H), 4.08 (t, *J* = 6.6 Hz, 1 H), 3.09 – 3.00 (m, 1 H), 2.90 – 2.80 (m, 1 H), 1.98 – 1.88 (m, 2 H), 1.57 (brs, 2 H); MS (ES) m/z: 486 ($M+H^+$).



9

N-[(1R)-3-(2,6-Dibromophenyl)-1-[3-(1,1,2,2-tetrafluoroethoxy)phenyl]-propyl]
-2-nitrobenzenesulfonamide

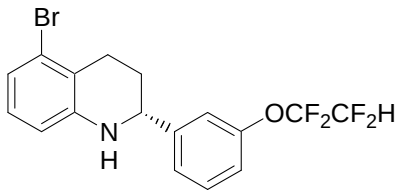
To a solution of the amine (13.1 g, 27.0 mmol) and triethylamine (9.4 mL, 54.0 mmol) in dichloromethane (135 mL) under a N_2 atmosphere was added NsCl (6.58 g, 29.7 mmol) under N_2. The reaction mixture was stirred at room temperature for 1 h and then poured into EtOAc / Et_2O. The solution was washed with saturated $NaHCO_3$, H_2O and brine, dried ($MgSO_4$), concentrated and purified by column chromatography (5%-10%-15%-20% EtOAc/Hex) to afford 15.2 g (84%) **9** as an oil. Compound **9** was obtained of the *R* absolute configuration. $[\alpha]_D^{20}$ = +100.0^o (c1; $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.0 Hz, 1 H), 7.66 (d, *J* = 7.9 Hz, 1 H), 7.55 – 7.33 (m, 4 H), 7.13 – 7.08 (m, 2 H), 7.01 (s, 1 H), 6.95 – 6.88 (m, 2 H), 5.96 (d, *J* = 8.9 Hz, 1 H), 5.86 (tt, *J* = 53.1, 2.8 Hz, 1 H), 4.69 (dd, *J* = 16.0, 7.8 Hz, 1 H), 3.19 – 3.11 (m, 1 H), 2.88 – 2.80 (m, 1 H), 2.14 – 1.94 (m, 2 H); MS (ES) m/z: 693 ($M+Na^+$).



(2R)-5-Bromo-1,2,3,4-tetrahydro-1-[(2-nitrophenyl)sulfonyl]
-2-[3-(1,1,2,2-tetrafluoroethoxy)phenyl]quinoline

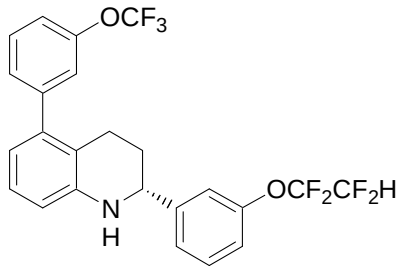
A mixture of **9** (15.2 g, 22.68 mmol), CuI (8.63 g, 45.36 mmol) and CsOAc (21.7 g, 113.4 mmol) in DMSO (200 mL) under a N_2 atmosphere was heated at 95 ^{o}C for 24 h. After cooling to room temperature, the reaction mixture was poured into EtOAc, washed with saturated NH_4Cl (3x), water, $Na_2S_2O_3$ solution and brine, dried ($MgSO_4$) concentrated to provide the Ns-THQ as an oil. This reaction was so clean it was carried on to the next step as a crude reaction mixture. As small portion was purified by column chromatography (25%

EtOAc/Hex) for characterization purposes. This compound was identical in all respects to the racemic Ns-THQ, except for the optical rotation. $[\alpha]_D^{20}$ = +55.0° (c1; $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.1 Hz, 1 H), 7.73 – 7.69 (m, 1 H), 7.63 – 7.50 (m, 3 H), 7.43 (d, *J* = 8.0 Hz, 1 H), 7.39 – 7.09 (m, 5 H), 5.88 (tt, *J* = 53.1, 2.9 Hz, 1 H), 5.62 (t, *J* = 6.9 Hz, 1 H), 2.74 – 2.66 (m, 1 H), 2.47 – 2.39 (m, 1 H), 2.35 – 2.27 (m, 1 H), 2.05 – 1.96 (m, 1 H); MS (ES) m/z: 589 (M).



(2R)-5-Bromo-1,2,3,4-tetrahydro-2-
[3-(1,1,2,2-tetrafluoroethoxy)phenyl]quinoline

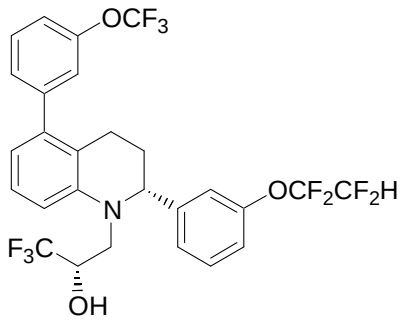
To a solution of the Ns-THQ (assuming quantitative yield from previous reaction, 22.6 mmol) in DMF (100 mL) under a N_2 atmosphere was added thioacetic acid (3.16 mL, 45.36 mmol) and powdered LiOH (2.17 g, 90.72 mmol). The reaction mixture was stirred at room temperature for ~ 6 h and then poured into EtOAc, washed with saturated $NaHCO_3$, H_2O and brine, dried ($MgSO_4$), concentrated and purified by column chromatography (25% EtOAc/Hex) to afford 8.25 g (90% yield for the two steps) the THQ as an oil, which was identical in all in all respects to racemic **6**, except for the optical rotation. $[\alpha]_D^{20}$ = +17.6° (c1; $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37 (t, *J* = 7.8 Hz, 1 H), 7.30 – 7.21 (m, 2 H), 7.15 (d, *J* = 7.9 Hz, 1 H), 6.95 – 6.71 (m, 2 H), 6.51 (d, *J* = 7.8 Hz, 1 H), 5.90 (tt, *J* = 53.1, 2.8 Hz, 1 H), 4.40 (dd, *J* = 9.3, 3.1 Hz, 1 H), 4.13 (brs, 1 H), 2.88 – 2.79 (m, 2 H), 2.21 – 2.11 (m, 1 H), 2.05 – 1.90 (m, 1 H); MS (ES) m/z: 406 (M+2).



10

(2R)-1,2,3,4-Tetrahydro-2-[3-(1,1,2,2-tetrafluoroethoxy)phenyl]-5-[3-(trifluoromethoxy)phenyl]quinoline

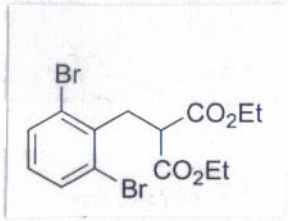
Under a N_2 atmosphere, a mixture of the THQ (8.25 g, 20.4 mmol), 3-trifluoromethoxy-phenyl-boronic acid (8.4 g, 40.8 mmol), $Pd(PPh_3)_4$ (2.3 g, 10 mol %) and 2 N K_2CO_3 (30.6 mL, 61.2 mmol) in 1,4-dioxane (200 mL) was heated at reflux for 2 h. After cooling to room temperature, EtOAc was added and the solution was washed with Na_2HCO_3, H_2O and brine. The organic layer was dried ($MgSO_4$), concentrated and purified by gradient column chromatography (4-10% EA/Hex) to give 8.78 g (88%) of the biphenyl-THQ **10** as a clear oil of the ***R*** configuration: $[\alpha]_D^{20}$ = -13.1^o (c1; $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.30 (m, 4 H), 7.25 (m, 1 H), 7.21 – 7.08 (m, 4 H), 6.62 (s, 1 H), 6.60 (s, 1 H), 5.90 (tt, *J* = 53.1, 2.8 Hz, 1 H), 4.51 (dd, *J* = 8.9, 3.3 Hz, 1 H), 4.20 (brs, 1 H), 2.81 – 2.71 (m, 1 H), 2.53 (dt, *J* = 16.6, 4.8 Hz, 1 H), 2.10 – 2.02 (m, 1 H), 1.92 – 1.82 (m, 1 H); MS (ES) m/z: 486 ($M+H^+$).

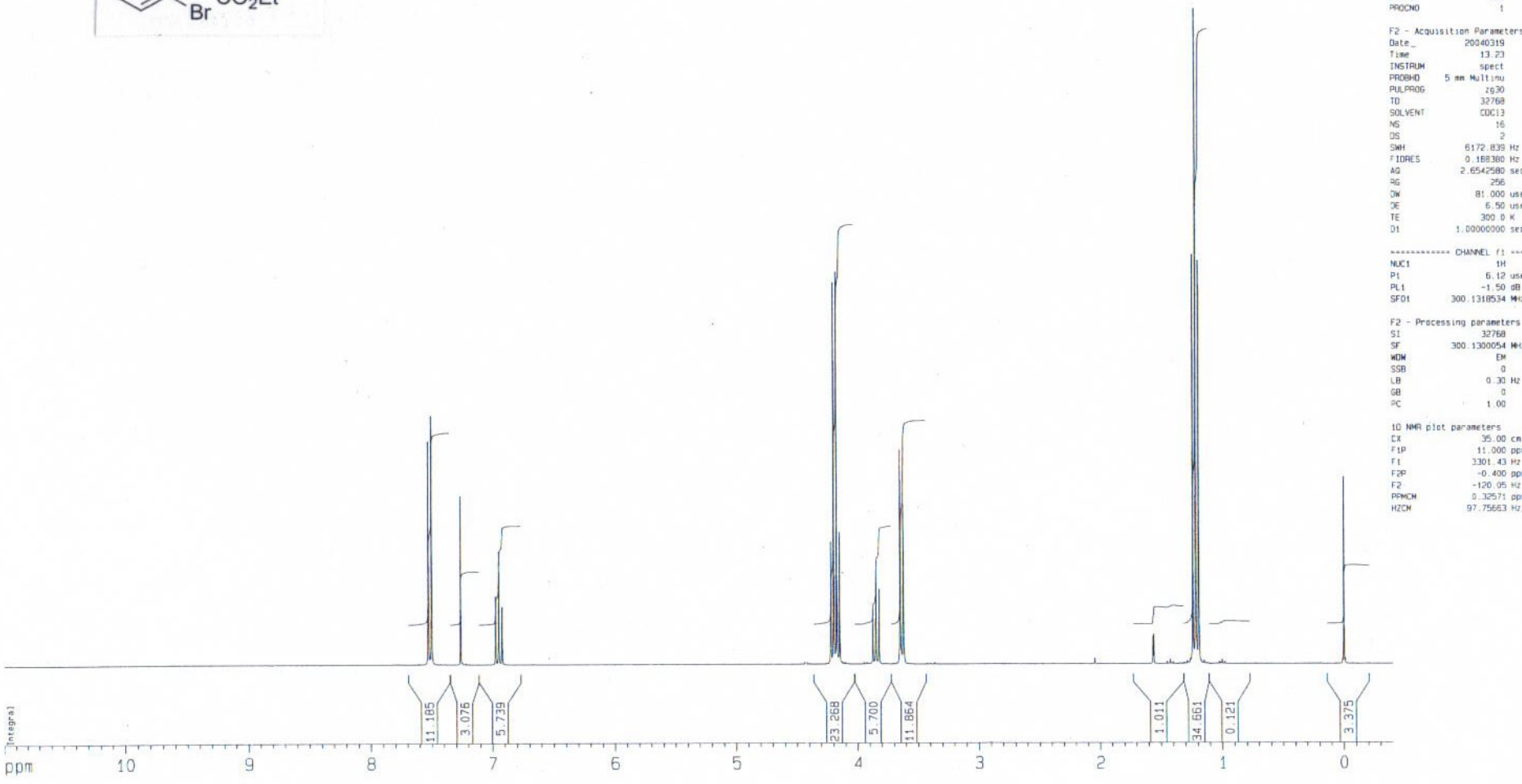


11

(2R, αS)-3,4-Dihydro-2-[3-(1,1,2,2-tetrafluoroethoxy)phenyl]-5-[3-(trifluoromethoxy)phenyl]-α-(trifluoromethyl)-1(2H)-quinolineethanol

To a solution of the biphenyl-THQ **10** (8.78 g, 18.08 mmol) and commercially available ***S*** isomer enriched 1,1,1-trifluoro-2,3-epoxy-propane (10.1 g, 90.4 mmol) in DCE (90 mL) under a N_2 atmosphere was added $Yb(OTf)_3$ (2.8 g, 10.5 mmol). The reaction mixture was heated at 50 ^oC for 19 h and then cooled to ambient temperature. EtOAc was added and the solution was washed with saturated $NaHCO_3$, H_2O and brine, dried ($MgSO_4$), concentrated and purified by Isco CombiFlash Companion column chromatography (330 g RediSep Flash Column, 2-10% EA/Hex gradient) to afford 7.91 g (73%) of higher R_f compound **11** as an oil. 1.95 g (18%) of lower R_f compound **12** was also isolated as an oil. Several overlap fractions were discarded. The optical rotation of **11** was determined to be $[\alpha]_D^{20} = -117.3^o$ (c1.13; $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.20 (m, 2 H), 7.28 – 7.10 (m, 6 H), 7.04 (s, 1 H), 6.73 (d, *J* = 8.3 Hz, 1 H), 6.67 (d, *J* = 7.4 Hz, 1 H), 5.89 (tt, *J* = 53.1, 2.8 Hz, 1 H), 4.89 (t, *J* = 4.4 Hz, 1 H), 4.42 (m, 1 H), 3.91 (d, *J* = 15.5 Hz, 1 H), 3.30 (dd, *J* = 15.6, 9.7 Hz, 1 H), 2.48 (dt, *J* = 16.3, 4.4 Hz, 1 H), 2.42 – 2.31 (m, 2 H), 2.19 – 2.09 (m, 1 H), 2.00 – 1.92 (m, 1 H); MS (ES) m/z: 598 ($M+H^+$).





Current Data Parameters
NAME P1_Mar19-2004
EXPNO 250
PROCNO 1

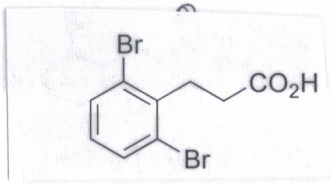
F2 - Acquisition Parameters
Date_ 20040319
Time 13.23
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 se
RG 256
DW 81.000 us
DE 6.50 us
TE 300.0 K
D1 1.00000000 se

CHANNEL f1
NUC1 1H
P1 6.12 us
PL1 -1.50 dB
SFO1 300.1318534 MH

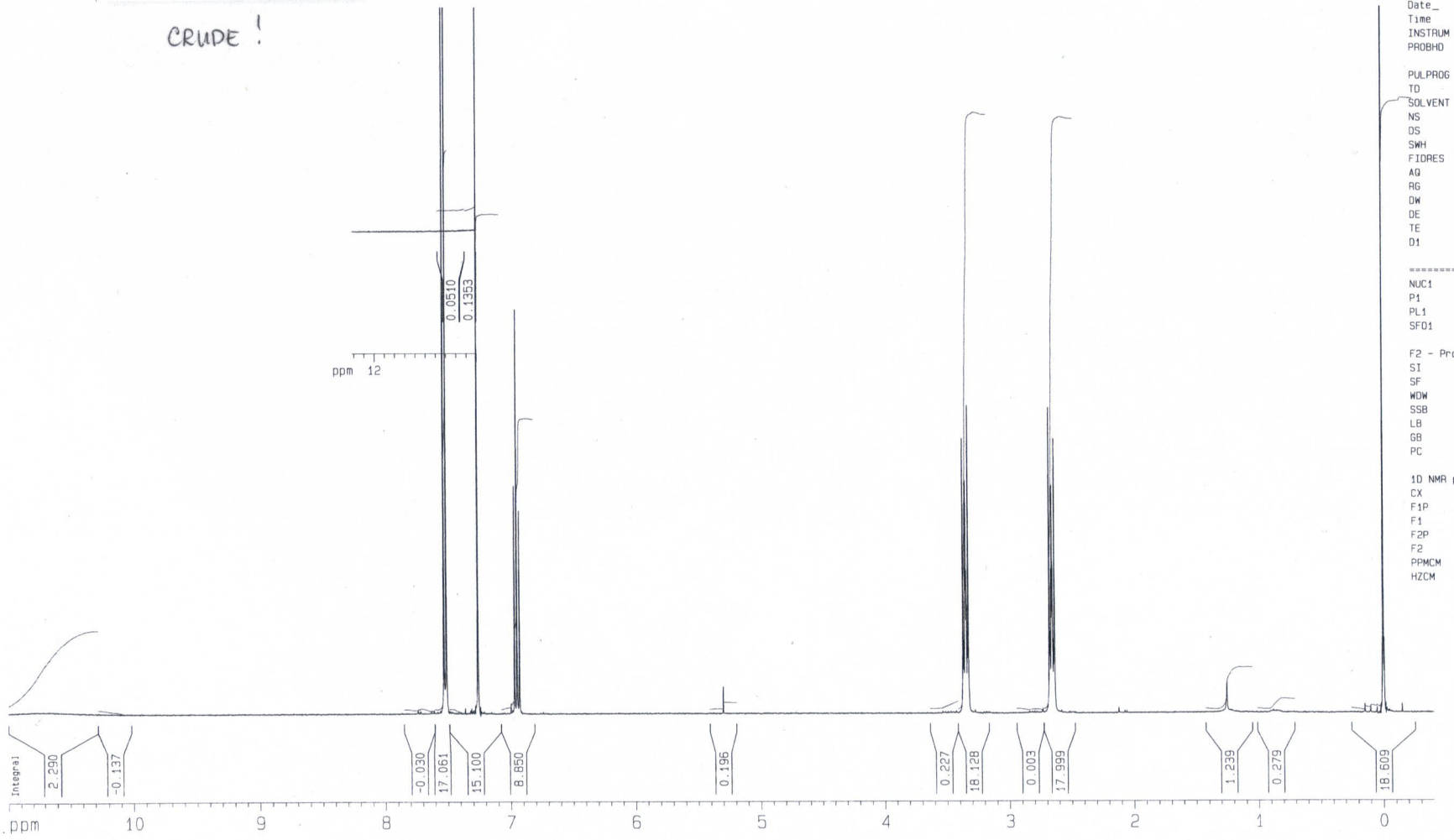
F2 - Processing parameters
SI 32768
SF 300.1300054 MH
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 35.00 cm
F1P 11.000 pp
F1 3301.43 Hz
F2P -0.400 pp
F2 -120.05 Hz
PPMCM 0.32571 pp
HZCM 97.75663 Hz

Rano-Mono-Acid



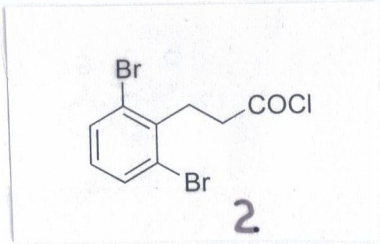
CRUDE !

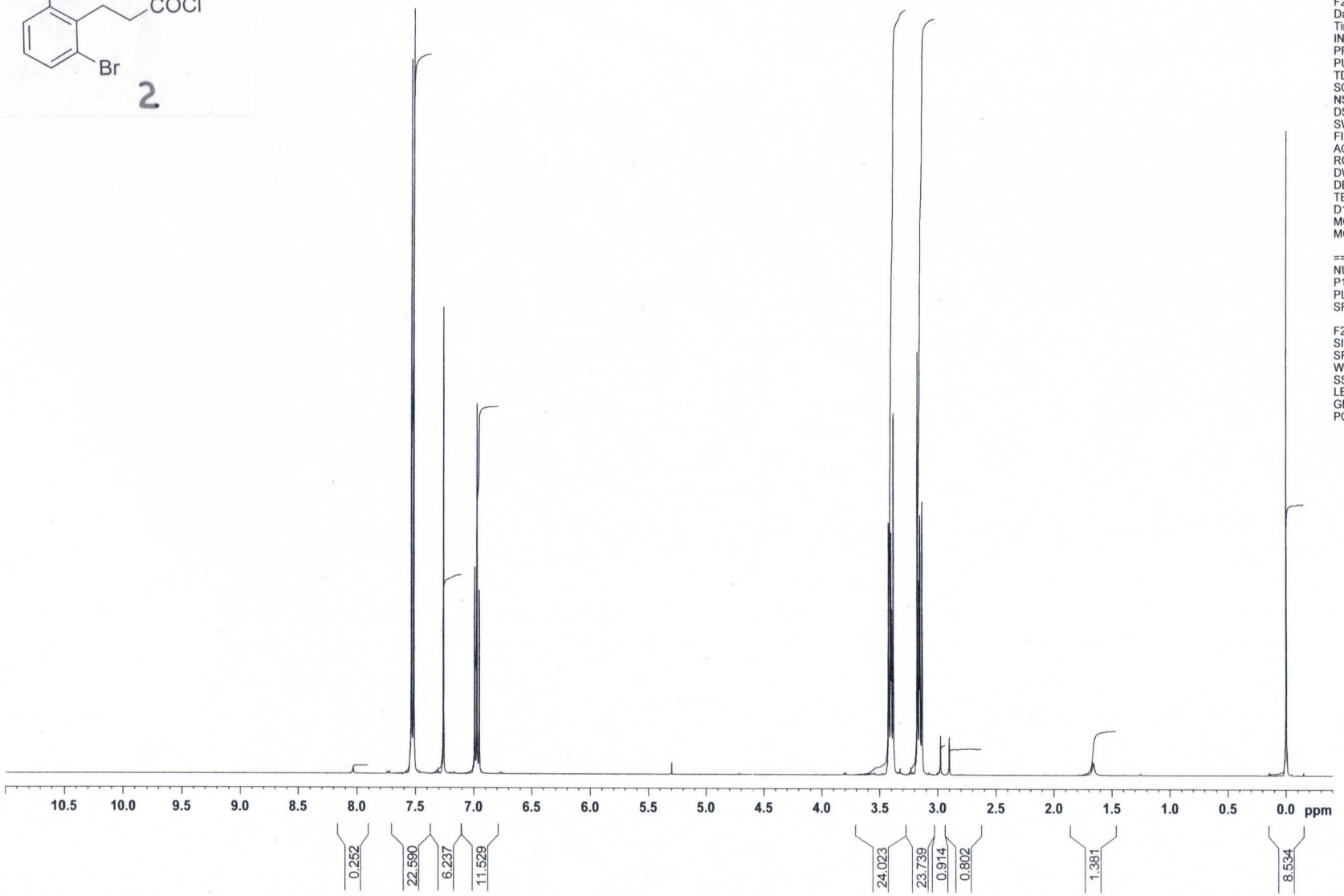



```
Current Data Parameters
NAME        P2_Oct17-2003
EXPNO                  40
PROCNO                  1

F2 - Acquisition Parameters
Date_            20031017
Time                9.56
INSTRUM            spect
PROBHD      5 mm Dual

PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           5995.204 Hz
FIDRES        0.182959 Hz
AQ            2.7329011 sec
RG                287.4
DW               83.400 use
DE                6.00 use
TE               300.0 K
D1            1.00000000 sec

========== CHANNEL f1 ===
NUC1                  1H
P1                8.00 use
PL1              -1.00 dB
SFO1         400.2069007 MHz

F2 - Processing parameters
SI                 32768
SF          400.2050095 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00

1D NMR plot parameters
CX                35.00 cm
F1P              11.000 ppm
F1              4402.26 Hz
F2P              -0.400 ppm
F2              -160.08 Hz
PPMCM            0.32571 ppm
HZCM           130.35248 Hz/
```

Title

COCl good



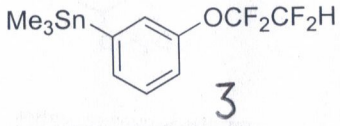


Current Data Parameters
NAME P2_Jun29-2005
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050629
Time 9.38
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 287.4
DW 60.400 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 400.2074714 MHz

F2 - Processing parameters
SI 32768
SF 400.2050100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

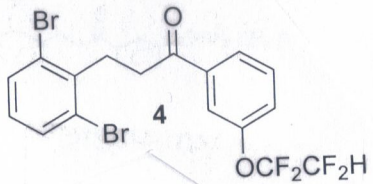


3


```
NAME          P1_Jan19-2004
EXPNO                  400
PROCNO                   1

F2 - Acquisition Parame
Date_             20040119
Time                 16.12
INSTRUM              spect
PROBHD     5 mm Multinu
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      2
SWH               6172.839
FIDRES            0.188380
AQ                2.6542580
RG                    362
DW                   81.000
DE                    6.50
TE                  300.0
D1                1.00000000

============ CHANNEL f1
NUC1                   1H
P1                   6.12
PL1                 -1.50
SFO1          300.1318534

F2 - Processing paramet
SI                  32768
SF            300.1300073
WDW                    EM
SSB                     0
LB                   0.30
GB                      0
PC                   1.00

1D NMR plot parameters
CX                   35.00
F1P                  11.000
F1                 3301.43
F2P                 -0.400
F2                 -120.05
PPMCM              0.32571
HZCM               97.75663
```

Title

tr

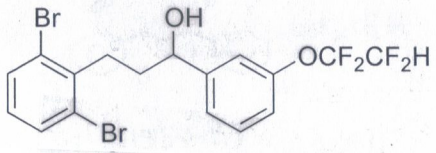


Current Data Parameters
NAME P2_Oct13-2005
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters
Date_ 20051013
Time 9.01
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 50.8
DW 60.400 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ====
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 400.2074714 MHz

F2 - Processing parameters
SI 32768
SF 400.2050103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

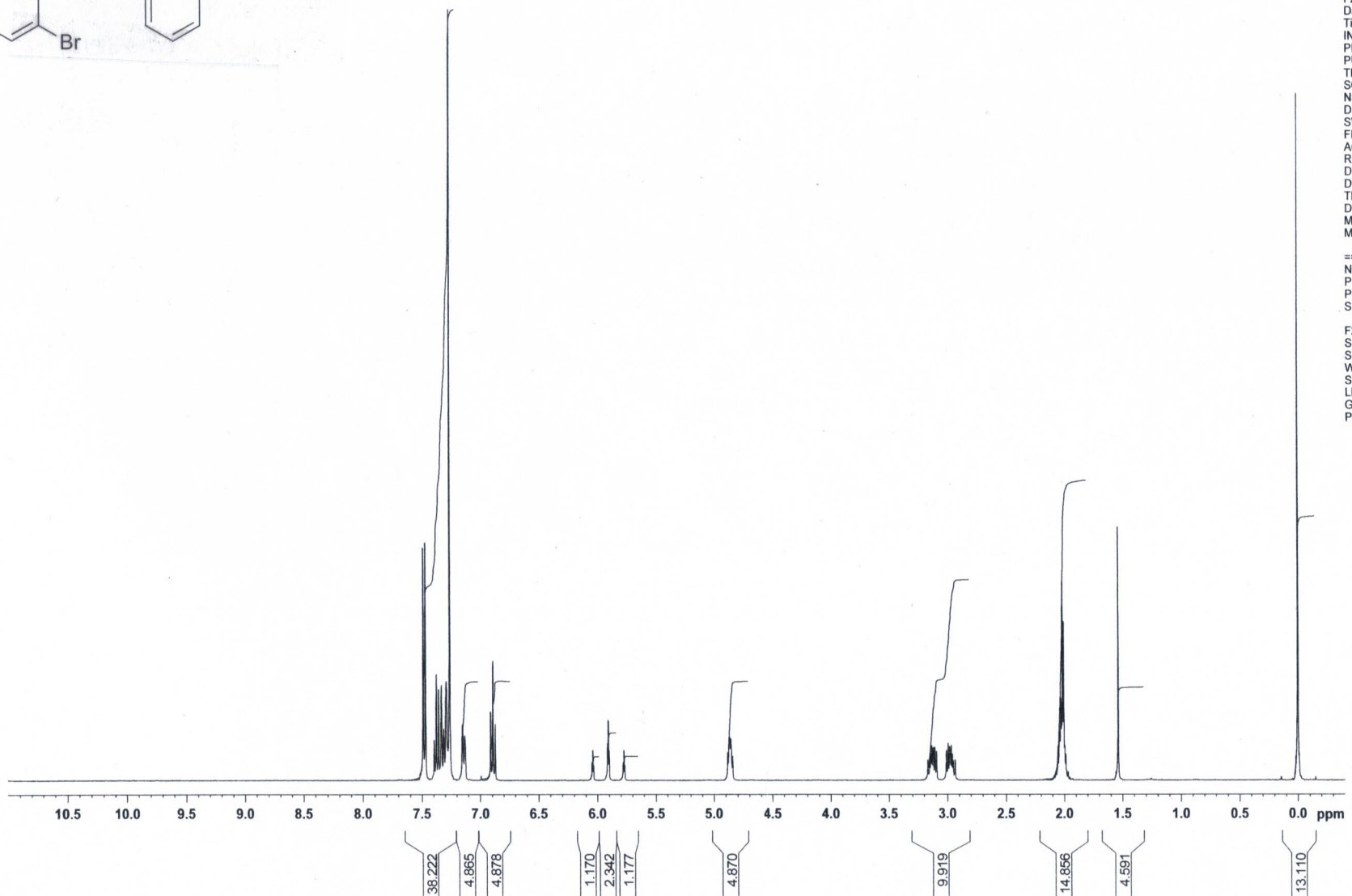


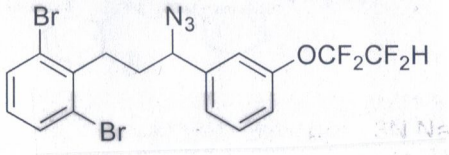
Current Data Parameters
NAME P2_Aug04-2005
EXPNO 220
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050804
Time 14.04
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 574.7
DW 60.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

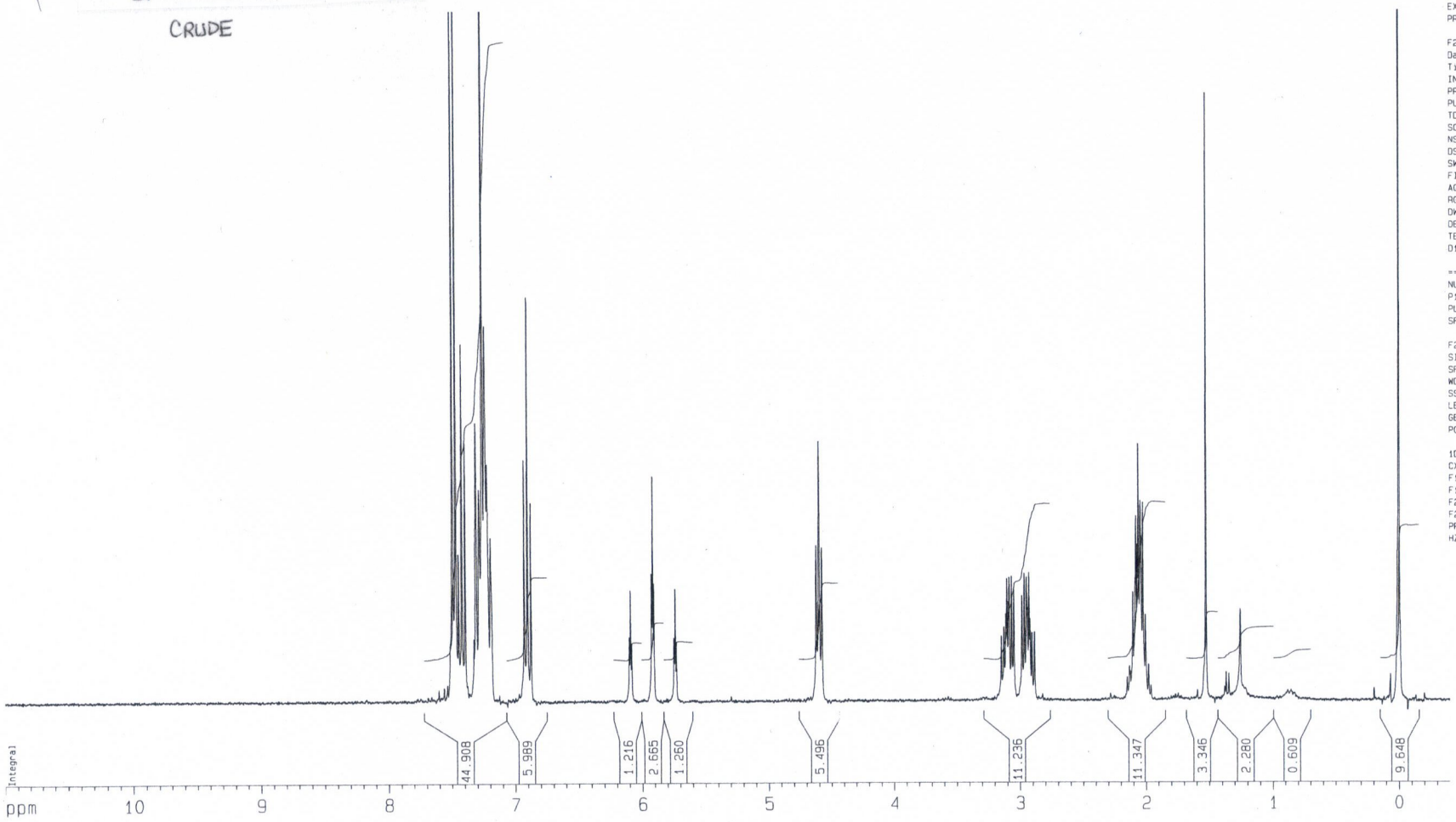
======== CHANNEL f1 ===
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 400.2074714 MHz

F2 - Processing parameters
SI 32768
SF 400.2050097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

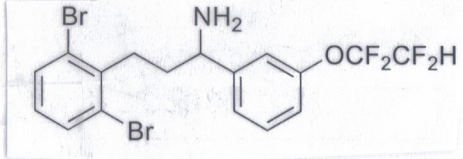




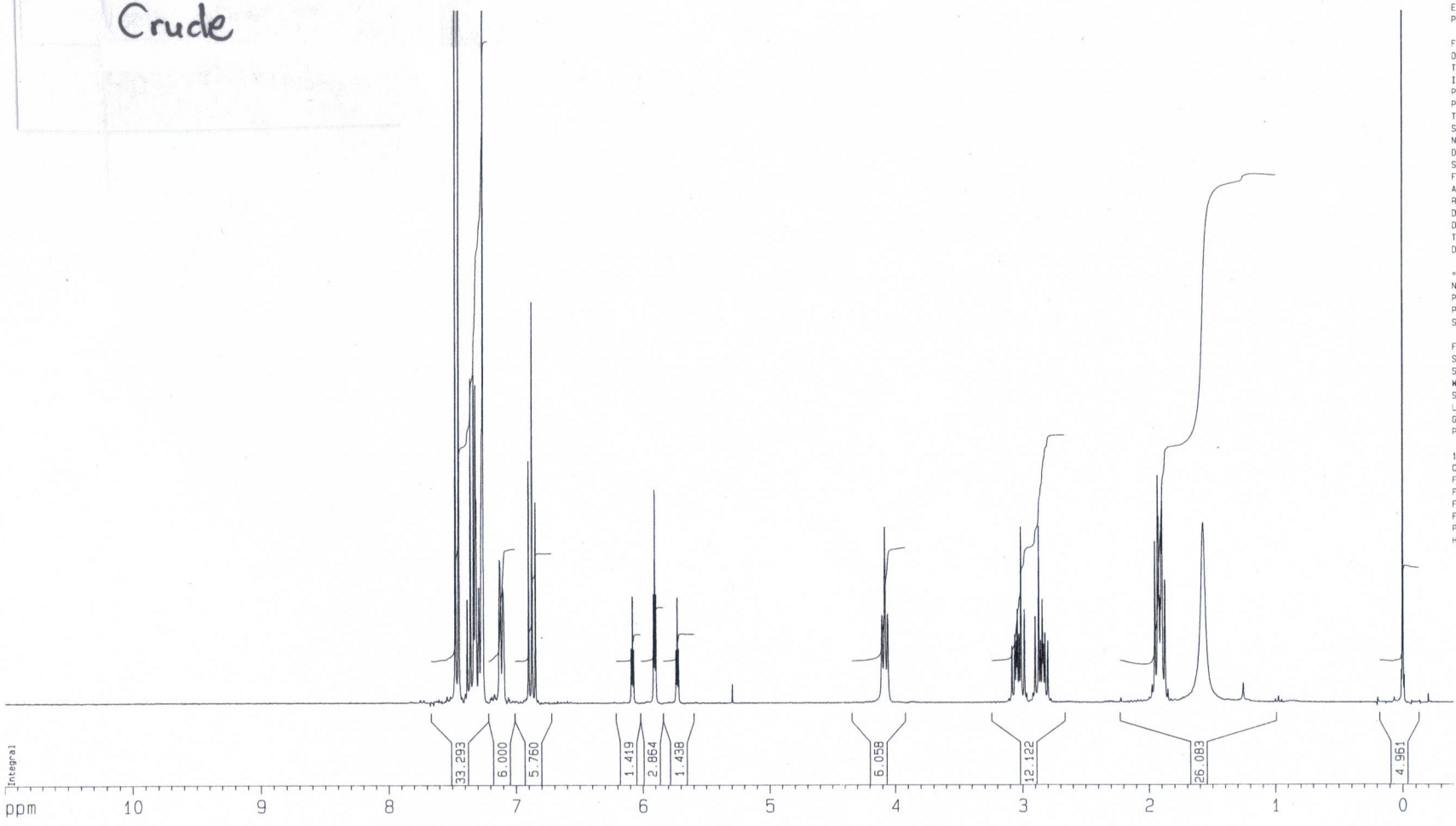
CRUDE



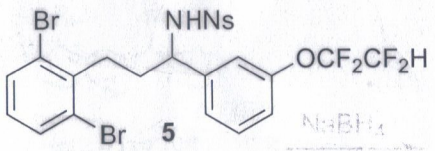

```
Current Data Parameters
NAME        P1_Jan27-2004
EXPNO                270
PROCNO                 1

F2 - Acquisition Paramet
Date_           20040127
Time               17.23
INSTRUM            spect
PROBHD     5 mm Multinu
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           6172.839
FIDRES        0.188380
AQ            2.6542580
RG                812.7
DW                81.000
DE                  6.50
TE                300.0
D1             1.00000000

============ CHANNEL f1
NUC1                 1H
P1                 6.12
PL1               -1.50
SFO1          300.1318534

F2 - Processing paramete
SI                32768
SF          300.1300067
WDW                  EM
SSB                   0
LB                 0.30
GB                    0
PC                 1.00

1D NMR plot parameters
CX                35.00
F1P               11.000
F1              3301.43
F2P              -0.400
F2              -120.05
PPMCM           0.32571
HZCM            97.75663
```



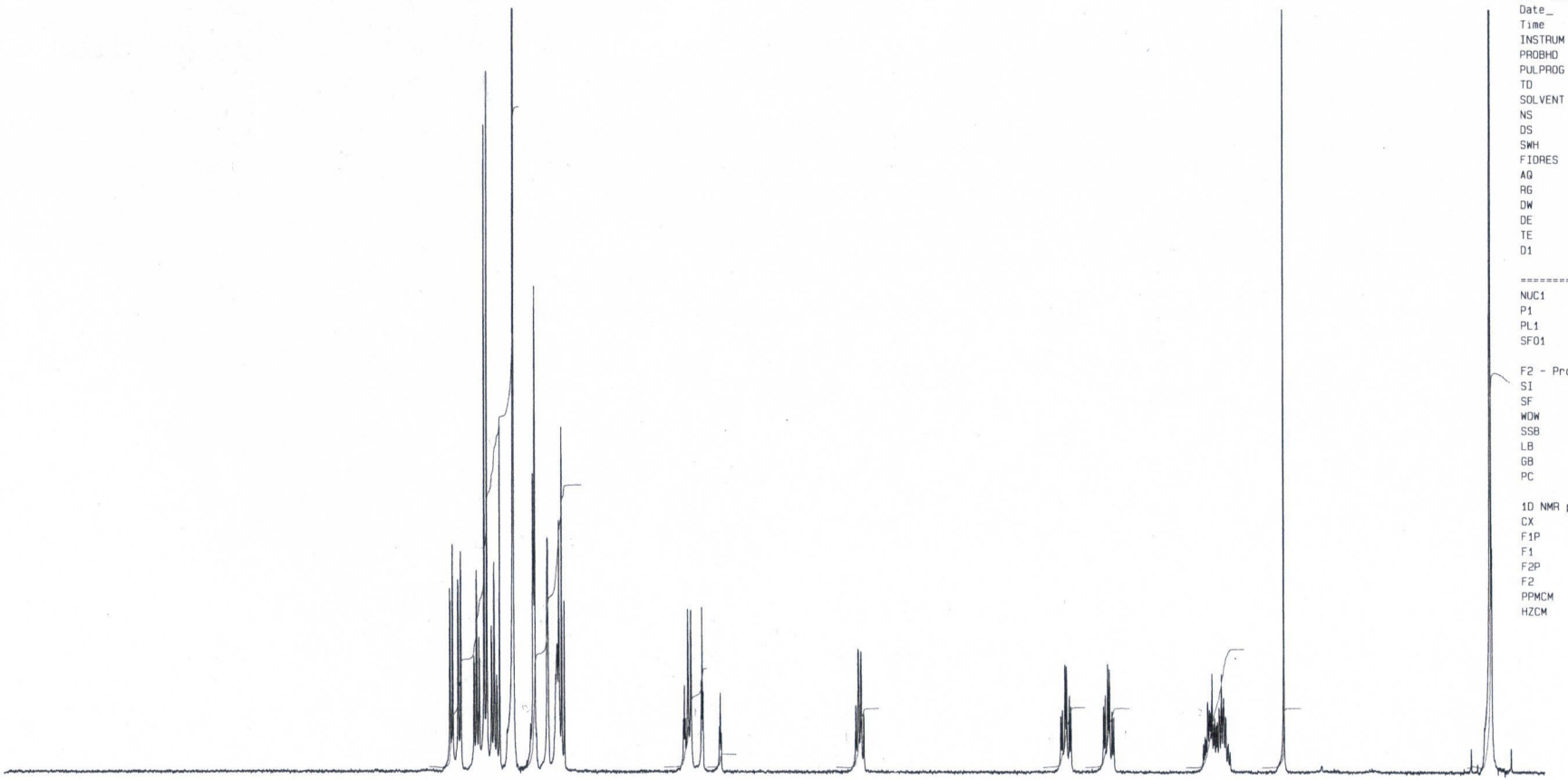
Crude




```
Current Data Parameters
NAME        P1_Feb17-2004
EXPNO                 160
PROCNO                  1

F2 - Acquisition Parameters
Date_           20040217
Time               11.52
INSTRUM            spect
PROBHD     5 mm Multinu
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           6172.839 Hz
FIDRES        0.188380 Hz
AQ            2.6542580 sec
RG                  362
DW             81.000 use
DE              6.50 use
TE             300.0 K
D1          1.00000000 sec

============ CHANNEL f1 ===
NUC1                1H
P1              6.12 use
PL1            -1.50 dB
SFO1      300.1318534 MHz

F2 - Processing parameters
SI                32768
SF        300.1300064 MHz
WDW                EM
SSB                 0
LB             0.30 Hz
GB                  0
PC             1.00

1D NMR plot parameters
CX             35.00 cm
F1P            11.000 ppm
F1           3301.43 Hz
F2P            -0.400 ppm
F2           -120.05 Hz
PPMCM          0.32571 ppm
HZCM          97.75663 Hz/
```



Ns'd Amine



Current Data Parameters
NAME P2_Mar02-2004
EXPNO 220
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040302
Time 14.01
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8802.817 Hz
FIDRES 0.268641 Hz
AQ 1.8612725 sec
RG 574.7
DW 56.800 use
DE 6.00 use
TE 300.0 K
D1 1.00000000 sec

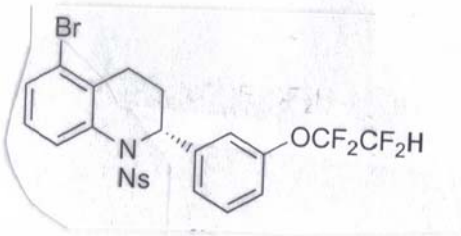
========== CHANNEL f1 ===
NUC1 1H
P1 7.50 use
PL1 0.00 dB
SFO1 400.2086018 MHz

F2 - Processing parameters
SI 32768
SF 400.2050092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 35.00 cm
F1P 11.000 ppm
F1 4402.26 Hz
F2P -0.400 ppm
F2 -160.08 Hz
PPMCM 0.32571 ppm
HZCM 130.35248 Hz/

Title

Rano



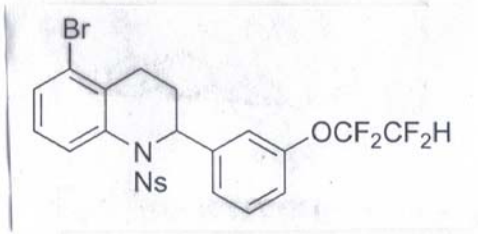
Current Data Parameters
NAME P1_Sep08-2004
EXPNO 210
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040908
Time 11.23
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 456.1
DW 81.000 usec
DE 6.50 usec
TE 299.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 6.00 usec
PL1 -1.50 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300066 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Title



rano

Current Data Parameters
NAME P2_Apr05-2004
EXPNO 260
PROCNO 1

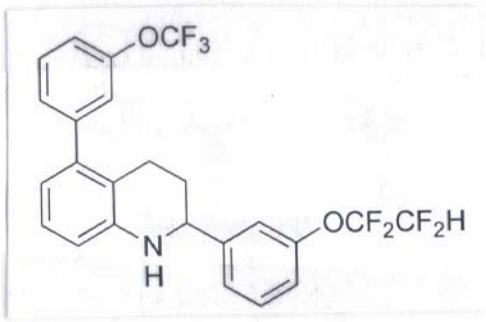
F2 - Acquisition Parameters
Date_ 20040405
Time 16.53
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 512
DW 60.400 usec
DE 6.50 usec
TE 296.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 400.2074714 MHz

F2 - Processing parameters
SI 32768
SF 400.2050091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Title

Rano



Current Data Parameters
NAME P2_May20-2004
EXPNO 240
PROCNO 1

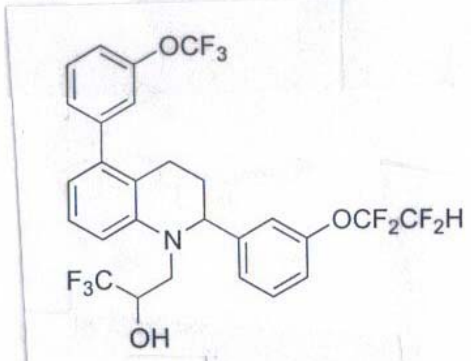
F2 - Acquisition Parameters
Date_ 20040520
Time 16.01
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 usec
DE 6.50 usec
TE 297.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 400.2074714 MHz

F2 - Processing parameters
SI 32768
SF 400.2050121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Title

Higher Rf spot



7

Higher Rf compound

Current Data Parameters
NAME P2_May24-2004
EXPNO 100
PROCNO 1

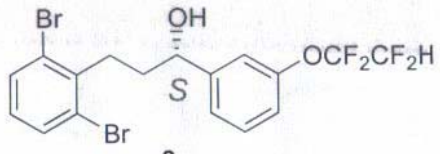
F2 - Acquisition Parameters
Date_ 20040524
Time 10.43
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
MCREST 0.00000000 se
MCWRK 0.01500000 se

======== CHANNEL f1 ===
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 400.2074714 MHz

F2 - Processing parameters
SI 32768
SF 400.2050110 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Title

Rano
35



8

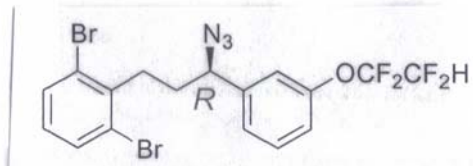
$[\alpha]_D^{20}$ = -12.1° (C=1; $CHCl_3$)

Current Data Parameters
NAME P2_Aug30-2004
EXPNO 20
PROCNO 1

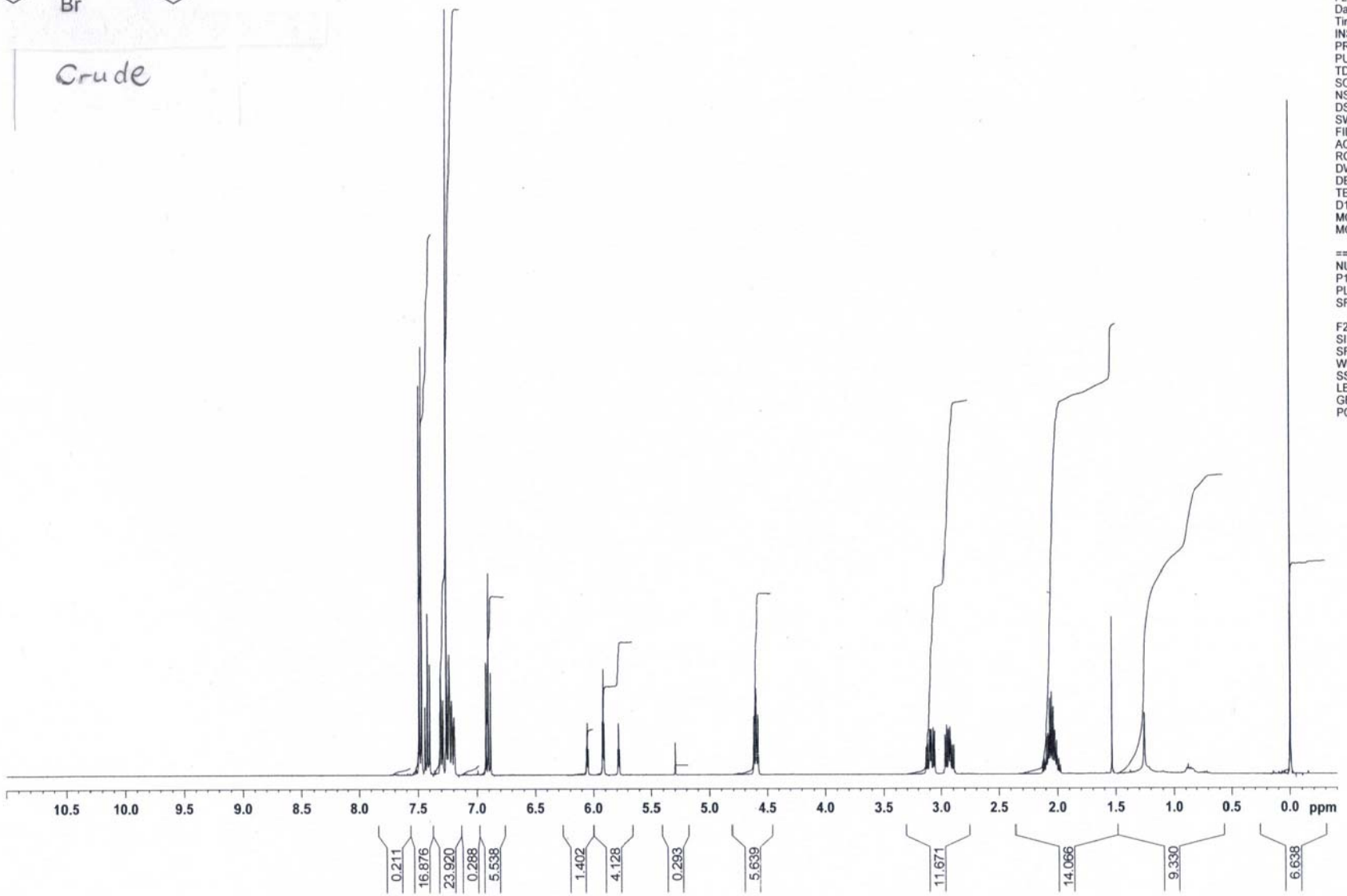
F2 - Acquisition Parameters
Date_ 20040830
Time 9.24
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
MCREST 0.00000000 se
MCWRK 0.01500000 se

======== CHANNEL f1 ===
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 400.2074714 MHz

F2 - Processing parameters
SI 32768
SF 400.2050100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Crude

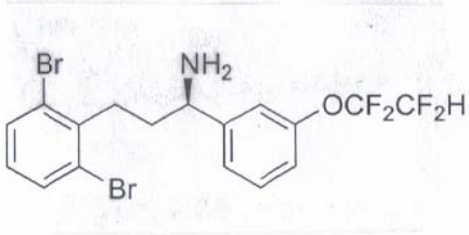


Current Data Parameters
NAME P2_Aug31-2004
EXPNO 90
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040831
Time 10.22
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

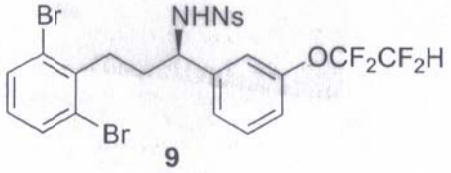
======== CHANNEL f1 ===
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 400.2074714 MHz

F2 - Processing parameters
SI 32768
SF 400.2050103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


Br
NH2
OCF2CF2H
Br

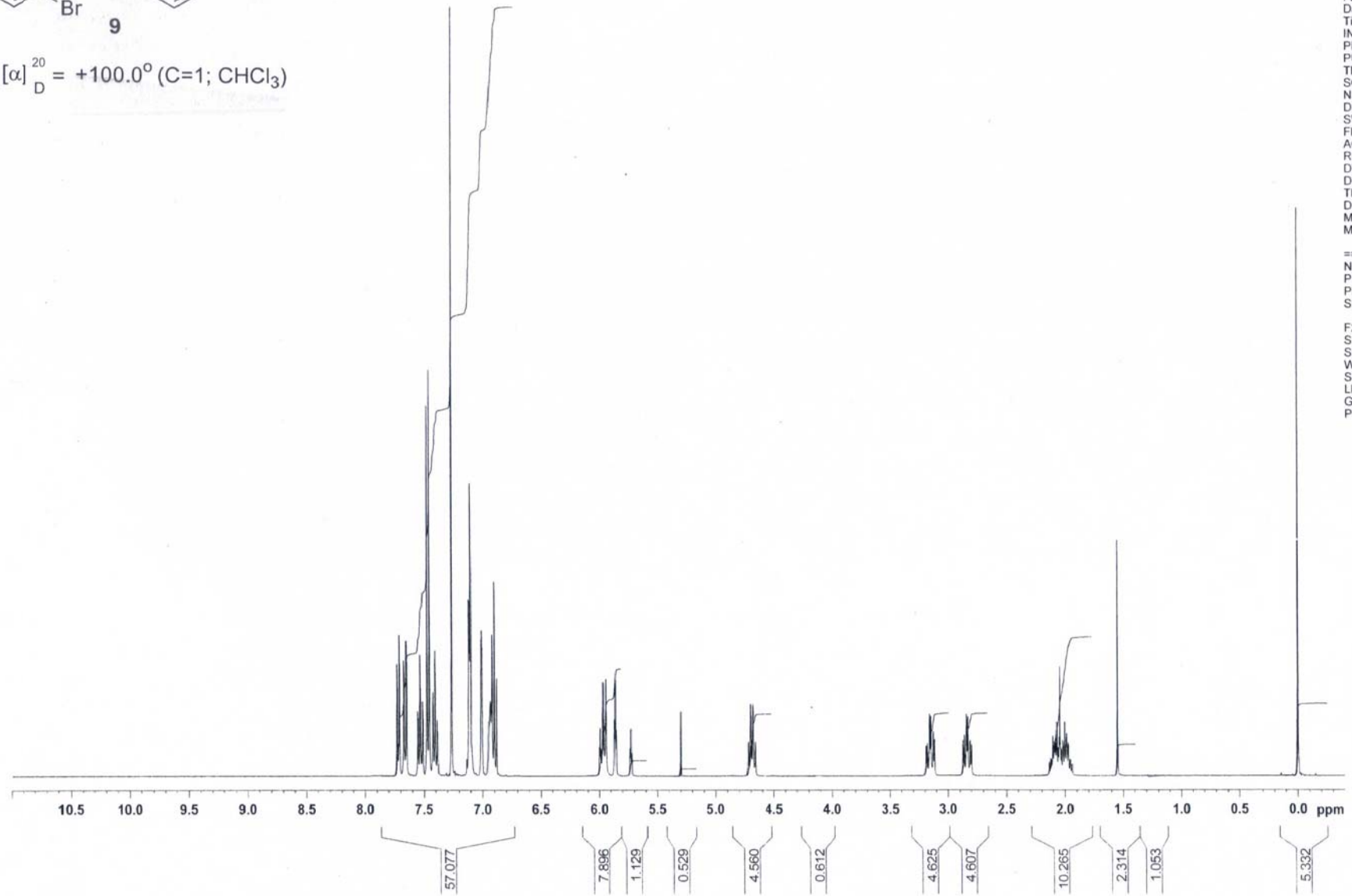
CRUDE

Amine
Current Data Parameters
NAME P2_Aug31-2004
EXPNO 230
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040831
Time 15.31
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 35.9
DW 60.400 usec
DE 6.50 usec
TE 296.7 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ===
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 400.2074714 MHz
F2 - Processing parameters
SI 32768
SF 400.2050114 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
41.611
1.402
4.259
5.644
46.338
0.746



9

$[\alpha]_D^{20} = +100.0^{o}$ (C=1; $CHCl_3$)



Current Data Parameters
NAME P2_Aug10-2005
EXPNO 40
PROCNO 1

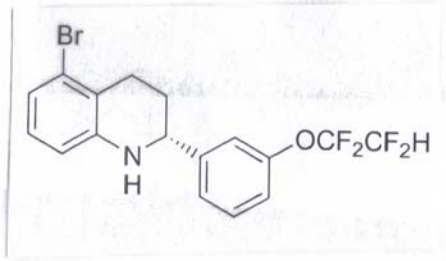
F2 - Acquisition Parameters
Date_ 20050810
Time 9.50
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
MCREST 0.00000000 se
MCWRK 0.01500000 se

======== CHANNEL f1 ===
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 400.2074714 MHz

F2 - Processing parameters
SI 32768
SF 400.2050094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Title

Rano

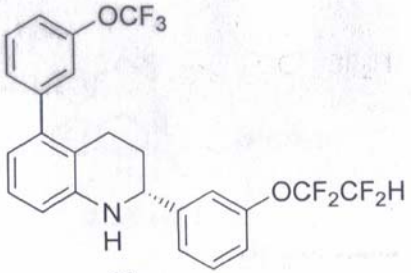


Current Data Parameters
NAME P2_Sep10-2004
EXPNO 60
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040910
Time 10.42
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 1625.5
DW 60.400 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
MCREST 0.00000000 se
MCWRK 0.01500000 se

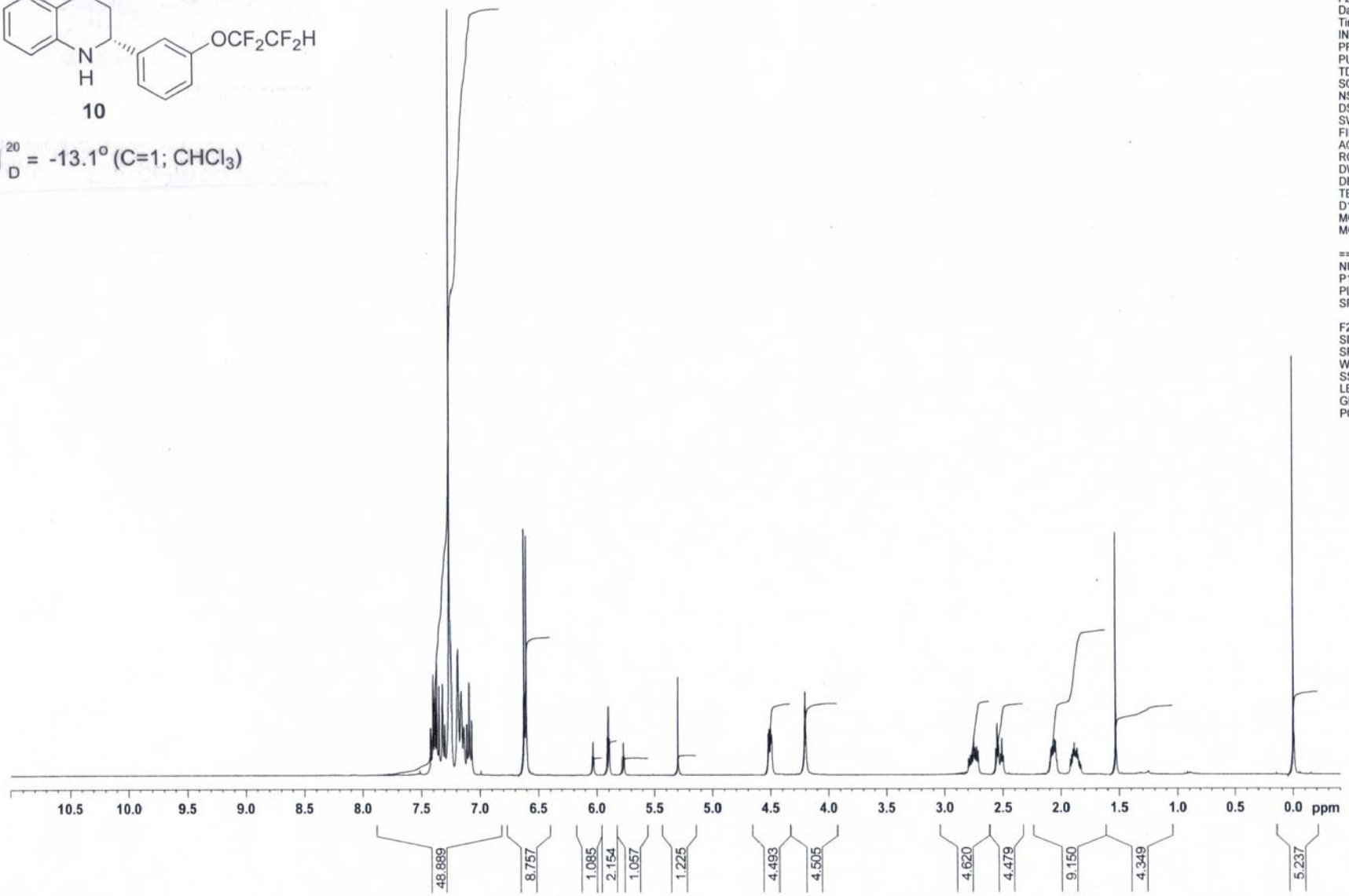
======== CHANNEL f1 ===
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 400.2074714 MHz

F2 - Processing parameters
SI 32768
SF 400.2050102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



10

$[\alpha]_D^{20}$ = -13.1° (C=1; $CHCl_3$)

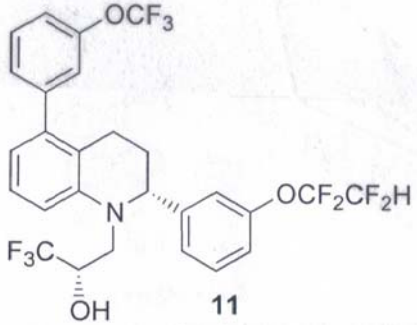


Current Data Parameters
NAME P2_Aug17-2005
EXPNO 460
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050817
Time 17.03
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.50 usec
TE 297.9 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 400.2074714 MHz

F2 - Processing parameters
SI 32768
SF 400.2050110 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



11

$[\alpha]_D^{20}$ = -117.3° (C1.1; $CHCl_3$)

Current Data Parameters
NAME P2_Jul29-2005
EXPNO 40
PROCNO 1

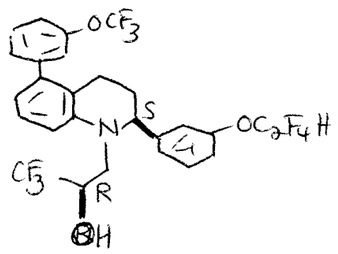
F2 - Acquisition Parameters
Date_ 20050729
Time 9.25
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.50 usec
TE 297.7 K
D1 1.00000000 sec
MCREST 0.00000000 se
MCWRK 0.01500000 se

======== CHANNEL f1 ===
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 400.2074714 MHz

F2 - Processing parameters
SI 32768
SF 400.2050104 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Title

Rano



20764-21

Higher Rf (S,R)

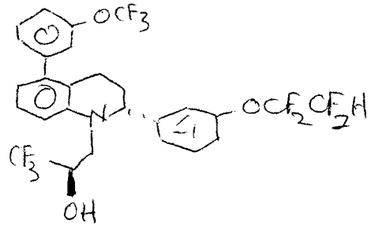
Current Data Parameters
NAME P2_Aug30-2004
EXPNO 60
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040830
Time 13.36
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 203.2
DW 60.400 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 400.2074714 MHz

F2 - Processing parameters
SI 32768
SF 400.2050113 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Title



21554-48
Lower Rf Spot

tr

Current Data Parameters
NAME P2_Aug10-2005
EXPNO 70
PROCNO 1

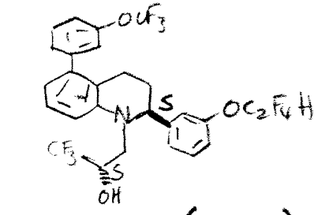
F2 - Acquisition Parameters
Date_ 20050810
Time 10.37
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 287.4
DW 60.400 usec
DE 6.50 usec
TE 297.7 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 400.2074714 MHz

F2 - Processing parameters
SI 32768
SF 400.2050106 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Title

20764-2 Lower Rf Spot



Lower Rf (S,S)

Current Data Parameters
NAME P2_Aug16-2004
EXPNO 220
PROCNO 1

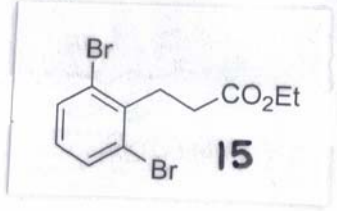
F2 - Acquisition Parameters
Date_ 20040816
Time 15.42
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 161.3
DW 60.400 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

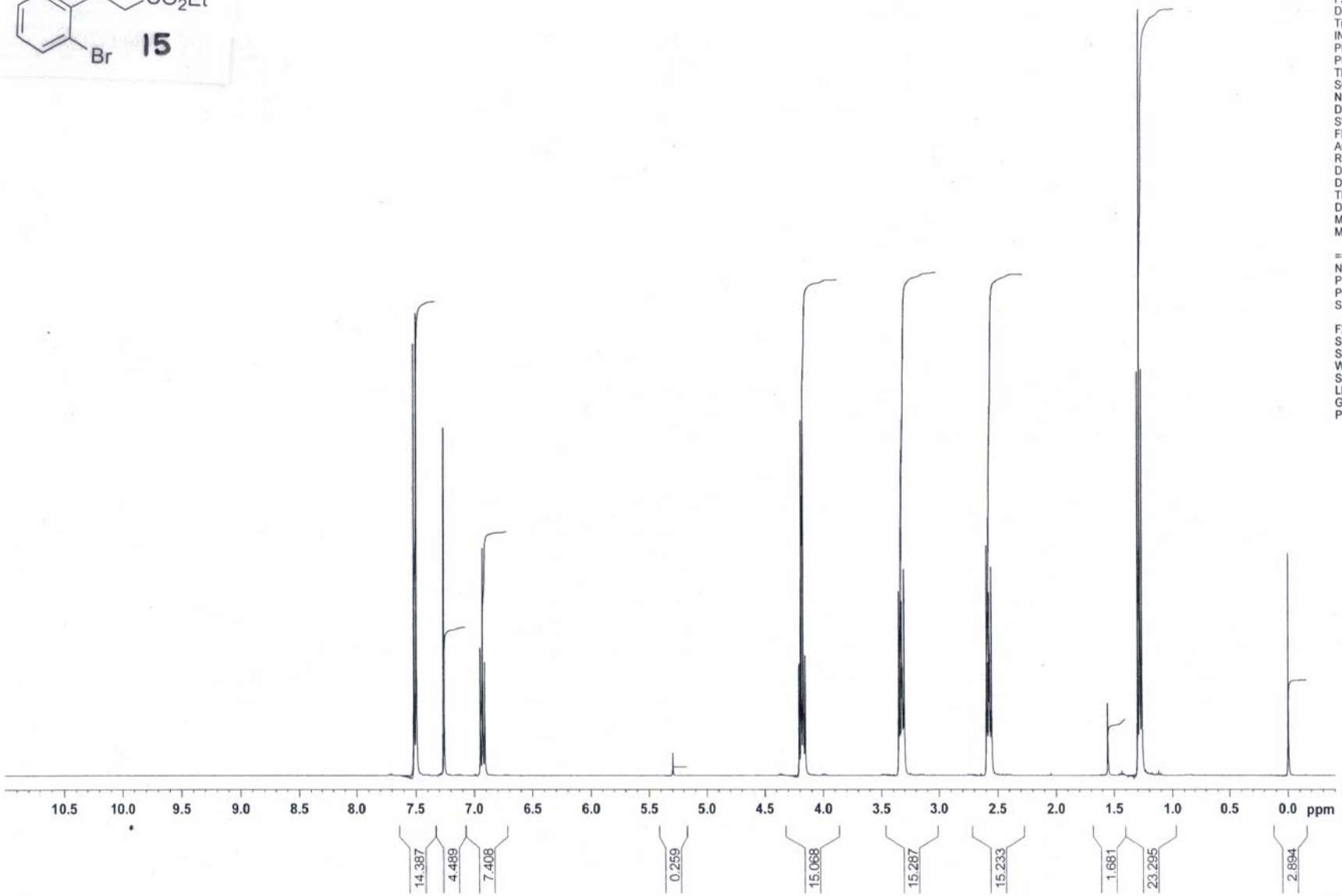
======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 400.2074714 MHz

F2 - Processing parameters
SI 32768
SF 400.2050113 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Title

tr





Current Data Parameters
NAME P2_Oct13-2005
EXPNO 190
PROCNO 1

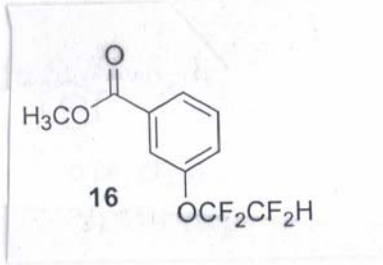
F2 - Acquisition Parameters
Date_ 20051013
Time 12.03
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 228.1
DW 60.400 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 400.2074714 MHz

F2 - Processing parameters
SI 32768
SF 400.2050095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Title

tr

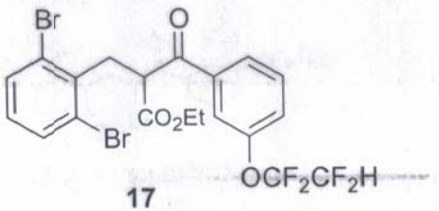


Current Data Parameters
NAME P2_Oct13-2005
EXPNO 290
PROCNO 1

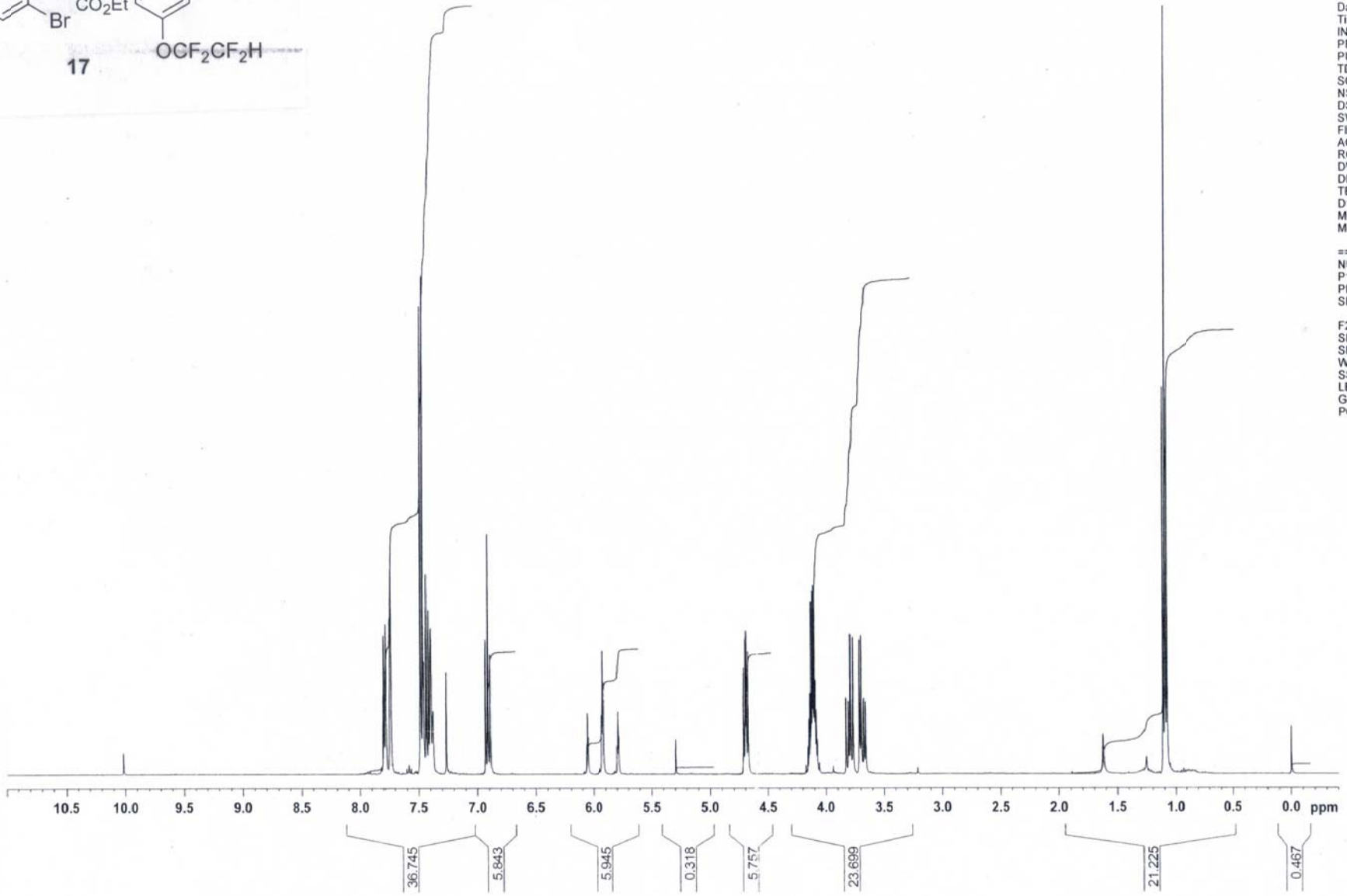
F2 - Acquisition Parameters
Date_ 20051013
Time 15.37
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
MCREST 0.00000000 se
MCWRK 0.01500000 se

======== CHANNEL f1 ===
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 400.2074714 MHz

F2 - Processing parameters
SI 32768
SF 400.2050095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



17



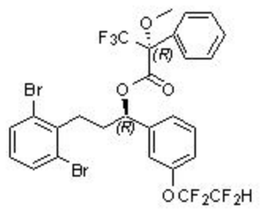
Current Data Parameters
NAME P2_Oct14-2005
EXPNO 260
PROCNO 1

F2 - Acquisition Parameters
Date_ 20051014
Time 14.38
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 45.3
DW 60.400 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ===
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 400.2074714 MHz

F2 - Processing parameters
SI 32768
SF 400.2050076 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

R, R Mosher Ester



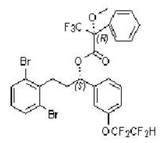
Current Data Parameters
NAME P2_Aug09-2004
EXPNO 140
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040809
Time 16.13
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 287.4
DW 60.400 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ====
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 400.2074714 MHz

F2 - Processing parameters
SI 32768
SF 400.2050100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

R, S Mosher Ester



Current Data Parameters
NAME P2_Aug09-2004
EXPNO 150
PROCNO 1

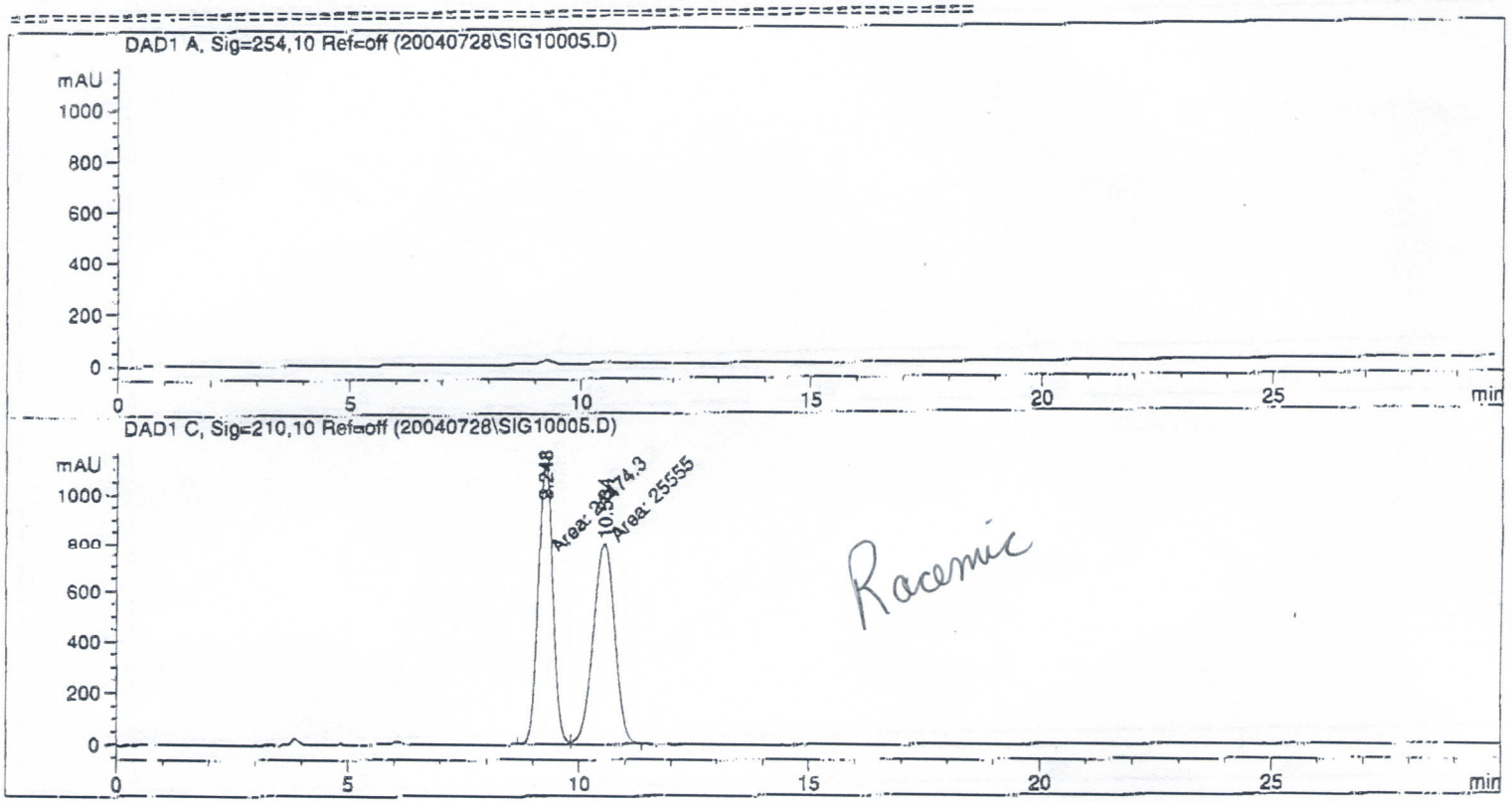
F2 - Acquisition Parameters
Date_ 20040809
Time 16.29
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 228.1
DW 60.400 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
MCREST 0.00000000 se
MCWRK 0.01500000 se

======== CHANNEL f1 ===
NUC1 1H
P1 7.50 usec
PL1 0.00 dB
SFO1 400.2074714 MHz

F2 - Processing parameters
SI 32768
SF 400.2050103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Run Logbook

Method		Method started: line# 4 vial# 3 inj# 1	16:05:53 07/28/04
Method		Instrument running sample Vial 3	16:05:54 07/28/04
1100 PMP	1	Pressure = 18.8 bar	16:06:45 07/28/04
1100 PMP	2	Pressure = 0.1 bar	16:06:45 07/28/04
1100 PMP	2	Flow = 0.000 ml/min	16:06:45 07/28/04
1100 ALS	1	Air temperature (tray) = 19.9 °C	16:06:45 07/28/04
1100 PMP	2	Pressure = 0.2 bar	16:36:45 07/28/04
1100 PMP	1	Pressure = 19.3 bar	16:36:45 07/28/04
Method		Instrument run completed	16:36:47 07/28/04
Method		Saving Method RUN.M	16:36:49 07/28/04
Method		Method completed	16:36:49 07/28/04



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Signal 1: DAD1 A, Sig=254,10 Ref=off

Signal 2: DAD1 C, Sig=210,10 Ref=off

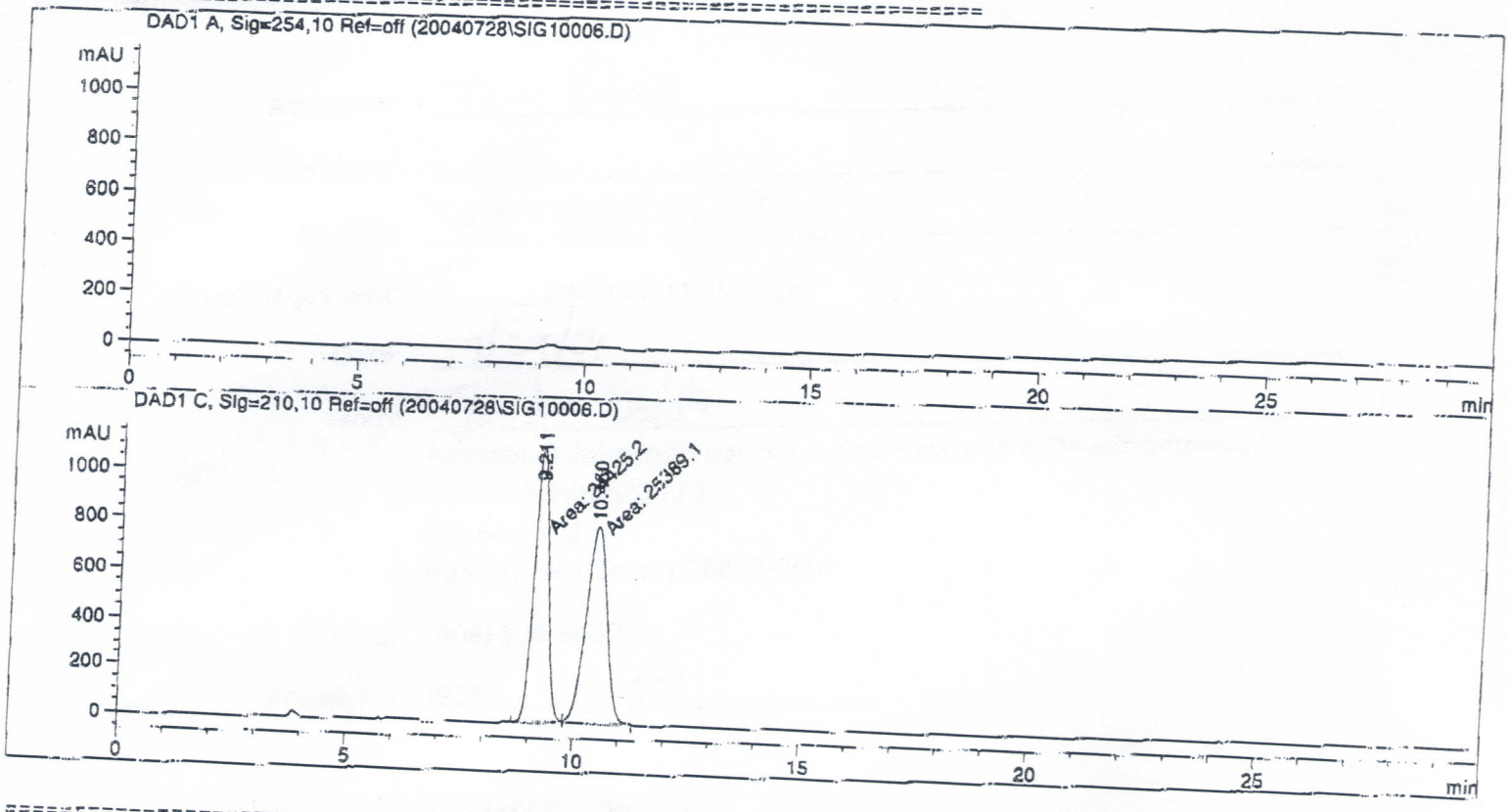
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.248	MF	0.3687	2.44743e4	1106.24939	48.9200
2	10.534	FM	0.5382	2.55550e4	791.30408	51.0800
Totals :				5.00293e4	1897.55347	

Results obtained with enhanced integrator!

*** End of Report ***

Run Logbook

Method		Method started: line# 4 vial# 3 inj# 2	16:36:51 07/28/04
Method		Instrument running sample Vial 3	16:36:51 07/28/04
1100 PMP	1	Pressure = 19.1 bar	16:37:42 07/28/04
1100 PMP	2	Pressure = 0.1 bar	16:37:42 07/28/04
1100 PMP	2	Flow = 0.000 ml/min	16:37:42 07/28/04
1100 ALS	1	Air temperature (tray) = 20.0 °C	16:37:42 07/28/04
1100 PMP	1	Pressure = 19.2 bar	17:07:41 07/28/04
1100 PMP	2	Pressure = 0.2 bar	17:07:42 07/28/04
Method		Instrument run completed	17:07:44 07/28/04
Method		Saving Method RUN.M	17:07:46 07/28/04
Method		Method completed	17:07:46 07/28/04



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 A, Sig=254,10 Ref=off

Signal 2: DAD1 C, Sig=210,10 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.211	MF	0.3659	2.44252e4	1112.68213	49.0325
2	10.460	FM	0.5324	2.53891e4	794.79333	50.9675

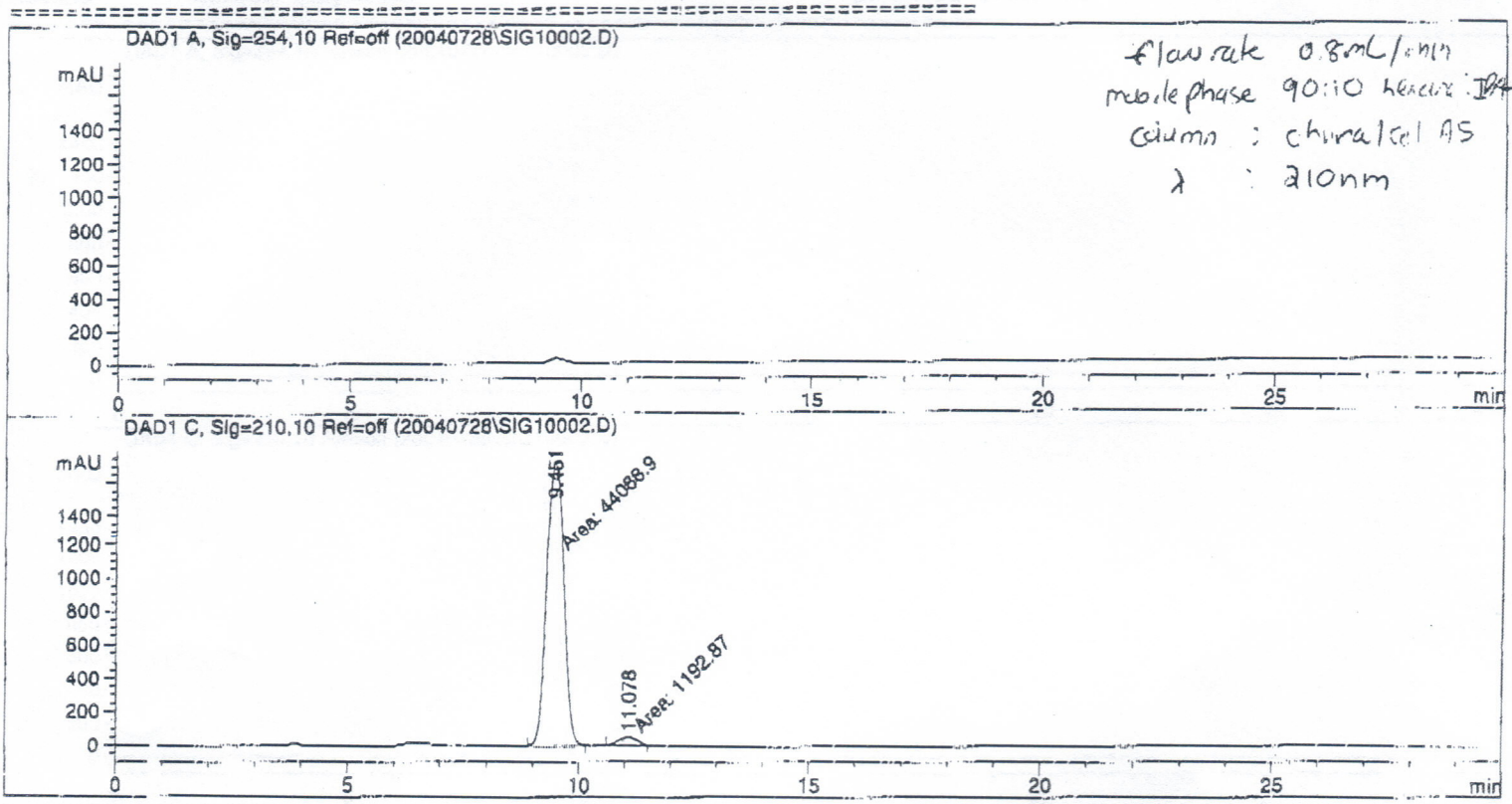
Totals : 4.98143e4 1907.47546

Results obtained with enhanced integrator!

*** End of Report ***

Run Logbook

Method	Method started: line# 2 vial# 1 inj# 1		14:32:59 07/28/04
Method	Instrument running sample Vial 1		14:32:59 07/28/04
1100 PMP	1	Pressure = 19.0 bar	14:33:51 07/28/04
1100 PMP	2	Pressure = 0.2 bar	14:33:51 07/28/04
1100 PMP	2	Flow = 0.000 ml/min	14:33:51 07/28/04
1100 ALS	1	Air temperature (tray) = 20.0 °C	14:33:51 07/28/04
1100 PMP	1	Pressure = 19.3 bar	15:03:51 07/28/04
1100 PMP	2	Pressure = 0.1 bar	15:03:51 07/28/04
Method	Instrument run completed		15:03:53 07/28/04
Method	Saving Method RUN.M		15:03:55 07/28/04
Method	Method completed		15:03:55 07/28/04



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Sample TR-19738-182
"R"

Signal 1: DAD1 A, Sig=254,10 Ref=off

Signal 2: DAD1 C, Sig=210,10 Ref=off

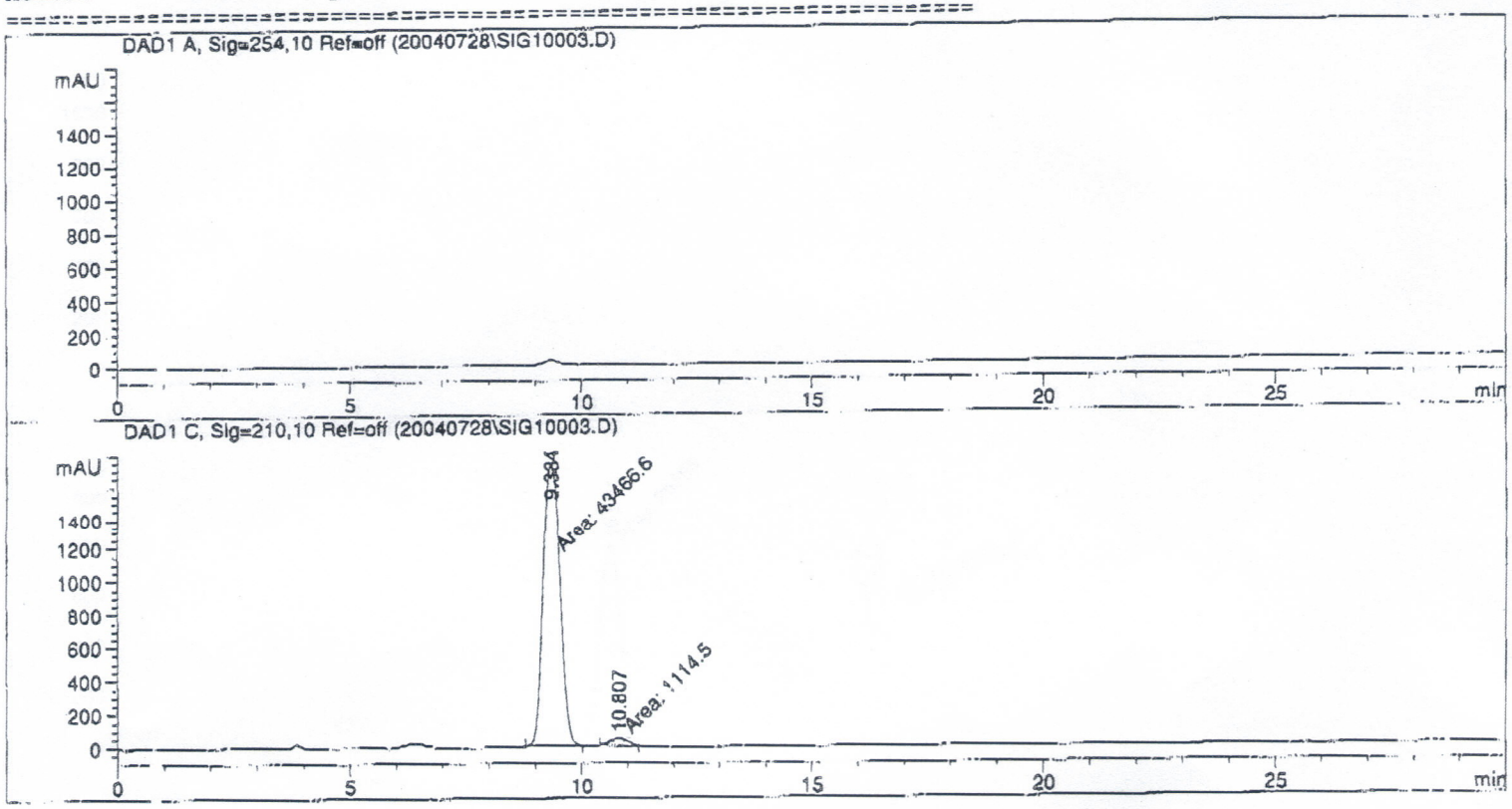
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.451	MM	0.4305	4.40889e4	1706.72131	97.3657
2	11.078	MM	0.4660	1192.87073	42.66501	2.6343
Totals :				4.52818e4	1749.38633	

Results obtained with enhanced integrator!

*** End of Report ***

Run Logbook

Method		Method started: line# 2 vial# 1 inj# 2	15:03:57 07/28/04
Method		Instrument running sample Vial 1	15:03:57 07/28/04
1100 PMP	1	Pressure = 19.1 bar	15:04:47 07/28/04
1100 PMP	2	Pressure = 0.1 bar	15:04:47 07/28/04
1100 PMP	2	Flow = 0.000 ml/min	15:04:47 07/28/04
1100 ALS	1	Air temperature (tray) = 20.0 °C	15:04:47 07/28/04
1100 PMP	1	Pressure = 19.4 bar	15:34:47 07/28/04
1100 PMP	2	Pressure = 0.1 bar	15:34:47 07/28/04
Method		Instrument run completed	15:34:49 07/28/04
Method		Saving Method RUN.M	15:34:51 07/28/04
Method		Method completed	15:34:52 07/28/04



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 A, Sig=254,10 Ref=off

Signal 2: DAD1 C, Sig=210,10 Ref=off

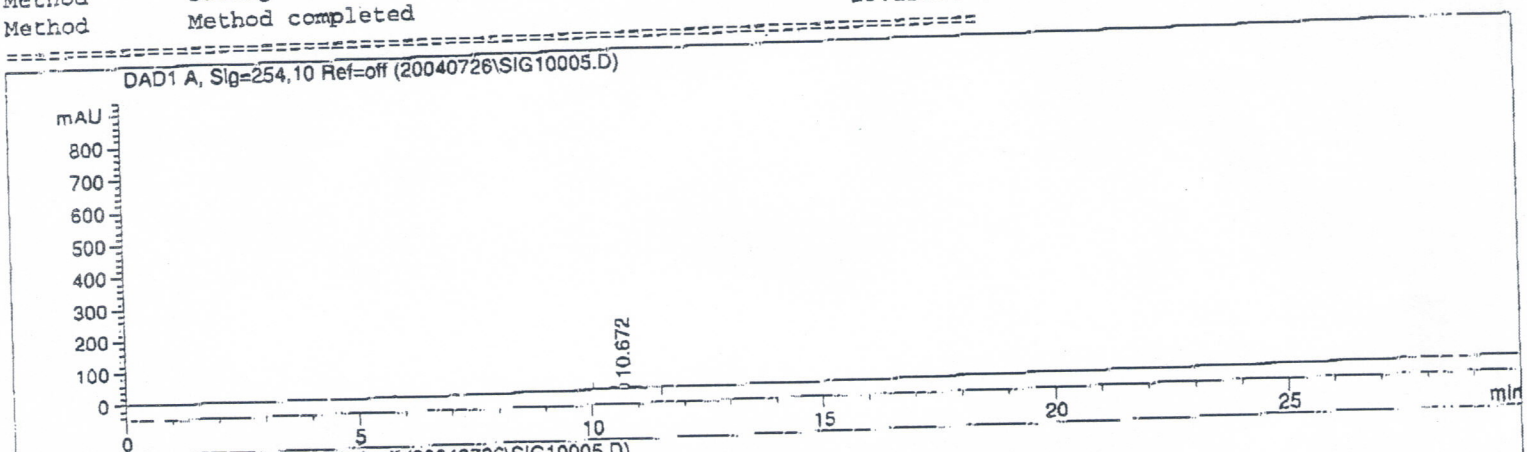
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.334	MM	0.4227	4.34666e4	1713.78467	97.5001
2	10.807	MM	0.4418	1114.49854	42.04097	2.4999
Totals :				4.45811e4	1755.82564	

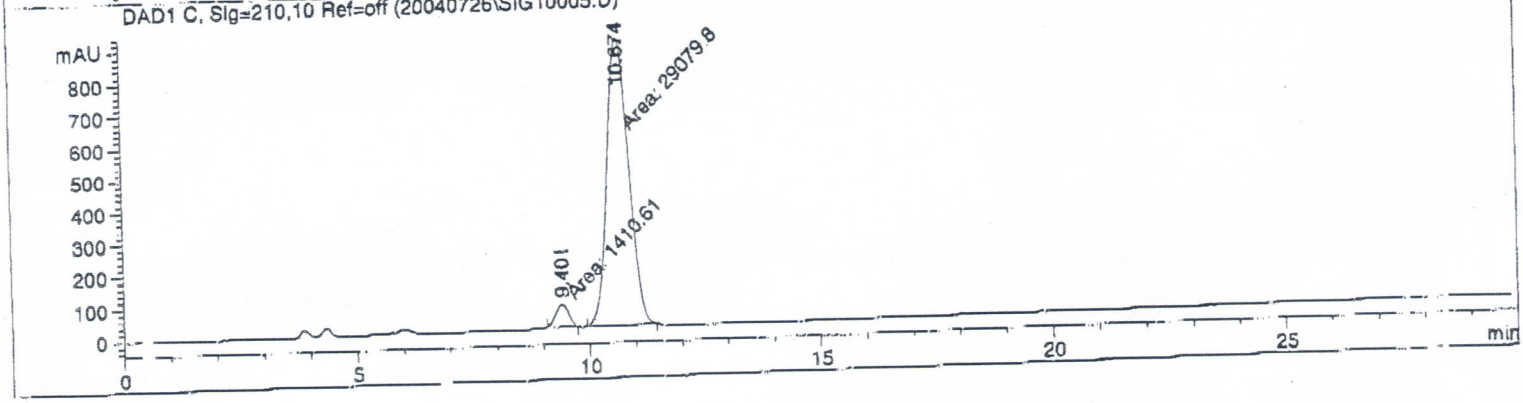
Results obtained with enhanced integrator!

*** End of Report ***

Run Logbook

Method		Method started: line# 4 vial# 2 inj# 1	14:42:00 07/26/04
Method		Instrument running sample Vial 2	14:42:01 07/26/04
1100 PMP	1	Pressure = 19.1 bar	14:42:52 07/26/04
1100 PMP	2	Pressure = 0.2 bar	14:42:52 07/26/04
1100 PMP	2	Flow = 0.000 ml/min	14:42:52 07/26/04
1100 ALS	1	Air temperature (tray) = 20.0 °C	14:42:52 07/26/04
1100 PMP	2	Pressure = 0.0 bar	15:12:52 07/26/04
1100 PMP	1	Pressure = 19.3 bar	15:12:52 07/26/04
Method		Instrument run completed	15:12:54 07/26/04
Method		Saving Method RUN.M	15:12:56 07/26/04
Method		Method completed	15:12:56 07/26/04





Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

19738-177
"S"

Signal 1: DAD1 A, Sig=254,10 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.672	BB	0.4907	359.71466	11.26048	100.0000

Signal 2: DAD1 C, Sig=210,10 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.401	MM	0.3297	1410.60803	71.31191	4.6264
2	10.674	MM	0.5444	2.90798e4	890.27887	95.3736
Totals :				3.04904e4	961.59078	

Results obtained with enhanced integrator!

*** End of Report ***