Supporting information

Catalytic enantioselective conjugate addition of fluorobis(phenylsulfonyl)methane to enals: Synthesis of chiral monofluoromethyl compounds

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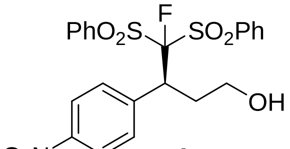
General Information: Commercial reagents and solvents from VWR or Aldrich were used as received, unless otherwise stated. Merck 60 silica gel was used for chromatography, and Sorbent Technologies silica gel plates with fluorescence F_{254} were used for thin-layer chromatography (TLC) analysis. Analytical high performance liquid chromatography (HPLC) was performed using a dual-wavelength UV detector on a Shimadzu SCL-10A or SPD-20A with HPLC-grade isopropanol and hexanes as the eluting solvents. ^1H and ^{13}C NMR spectra were recorded on Bruker Avance 500, and tetramethylsilane (TMS) was used as a reference. Data for ^1H are reported as follows: chemical shift (ppm), and multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet). Data for ^{13}C NMR are reported as ppm. Mass spectra were obtained from University of New Mexico mass spectral facility using electrospray positive mode (ES^+).

Typical Procedure for Conjuagte Addition Reaction

A mixture of α, β-unsaturated aldehyde **1** (0.15-0.50 mmol, as shown in Table 2), Fluorobis(phenylsulfonyl)methane (FBSM) **2** (31.4 mg, 0.10 mmol), catalyst **III** (0.02 mmol) and PhCOOH (2.4 mg, 0.02 mmol) in 0.8 ml of toluene was stirred at 0 °C for shown time. The mixture was then directly purified by silica gel chromatography, eluted by hexane/EtOAc = 3/1 to give the desired product aldehyde **3**.

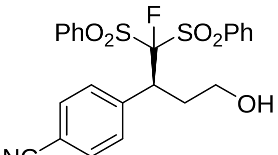
The ontained aldehyde **3** was dissolved in 1.0 mL MeOH, the solution was cooled to 0 °C. $NaBH_4$ (11 mg, 0.3 mmol) was added in three portions and the resulting mixture was stirred at 0℃ for 20 min. The reaction mixture was poured into water and extracted with CH_2Cl_2 for three times. The combined organic layers were then washed with water and brine, dried (Na_2SO_4), and concentrated. The residue was then purified by silica gel chromatography, eluted by pentane/EtOAc = 2/1 to give the desired product alcohol **4**.



4a

(*S*)-4-Fluoro-3-(4-nitrophenyl)-4,4-bis(phenylsulfonyl)butan-1-ol (4a) (Table 2, entry 1)

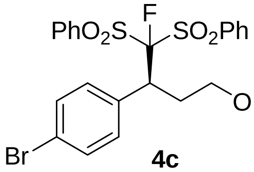
The title compound was prepared according to the general procedure, as described above in 83% yield. ^1H NMR (500 MHz, $CDCl_3$): δ 8.05 (d, *J* = 8.5 Hz, 2H), 7.86 (d, *J* = 8.5 Hz, 2H), 7.77 (d, *J* = 8.0 Hz, 2H), 7.75 (t, *J* = 7.5 Hz, 1H), 7.65 (t, *J* = 7.5 Hz, 1H), 7.56 (t, *J* = 7.5 Hz, 2H), 7.45 (t, *J* = 7.5 Hz, 2H), 7.28 (d, *J* = 8.5 Hz, 2H), 4.36 (d, *J* = 12 Hz, 1H), 3.83 (m, 1H), 3.21 (m, 1H), 3.10 (m, 1H), 2.82 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 147.5, 140.9 (*J* = 5.5 Hz), 136.3, 135.5, 135.1, 134.0, 132.0, 131.1, 131.0, 129.2, 128.6, 123.0, 114.8 (*J* = 266 Hz), 59.1, 44.8 (*J* = 17 Hz), 31.0; HPLC (Chiralcel OD-H, *i*PrOH/hexane = 30/70, flow rate = 0.6 mL min^{-1}, λ = 254 nm): t_{major} = 16.65 min, t_{minor} = 29.97 min, ee = 99%; $[\alpha]_D^{25}$ = -210.7 (*c* = 1.0 in $CHCl_3$).



4b

(*S*)-4-(1-Fluoro-4-hydroxy-1,1-bis(phenylsulfonyl)butan-2-yl)benzonitrile (4b) (Table 2, entry 2)

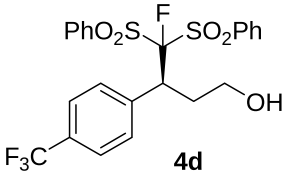
The title compound was prepared according to the general procedure, as described above in 72% yield. ^1H NMR (500 MHz, $CDCl_3$): δ 7.86 (d, *J* = 8.0 Hz, 2H), 7.77 (d, *J* = 8.5 Hz, 2H), 7.75 (t, *J* = 7.5 Hz, 1H), 7.66 (t, *J* = 7.5 Hz, 1H), 7.56 (t, *J* = 8.0 Hz, 2H), 7.50 (d, *J* = 8.5 Hz, 2H), 7.46 (t, *J* = 8.0 Hz, 2H), 7.22 (d, *J* = 8.0 Hz, 2H), 4.29 (d, *J* = 11.5 Hz, 1H), 3.82 (m, 1H), 3.20 (m, 1H), 3.07 (m, 1H), 2.79 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 138.9 (*J* = 5.2 Hz), 136.4, 135.4, 135.0, 134.1, 131.8, 131.7, 131.2, 131.0, 129.2, 128.5, 118.4, 114.9 (J = 266 Hz), 111.9, 59.2, 45.0 (J = 17 Hz), 30.9; HPLC (Chiralcel AD, *i*PrOH/hexane = 30/70, flow rate = 0.6 mL min^{-1}, λ = 210 nm): t_{major} = 17.88 min, t_{minor} = 34.85 min, ee = 99%; $[\alpha]_D^{25}$ = -209.6 (c = 1.07 in $CHCl_3$).



4c

(*S*)-3-(4-Bromophenyl)-4-fluoro-4,4-bis(phenylsulfonyl)butan-1-ol (4c) (Table 2, entry 3)

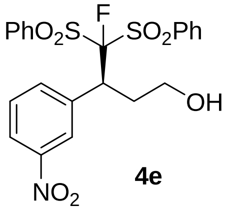
The title compound was prepared according to the general procedure, as described above in 81% yield. ^1H NMR (500 MHz, $CDCl_3$): δ 7.86 (d, J = 8.0 Hz, 2H), 7.78 (d, J = 8.5 Hz, 2H), 7.73 (t, J = 7.5 Hz, 1H), 7.65 (t, J = 7.5 Hz, 1H), 7.55 (t, J = 8.0 Hz, 2H), 7.45 (t, J = 8.0 Hz, 2H), 7.31 (d, J = 8.5 Hz, 2H), 6.94 (d, J = 8.5 Hz, 2H), 4.18 (m, 1H), 3.81 (m, 1H), 3.26 (m, 1H), 3.04 (m, 1H), 2.75 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 136.6, 135.2, 134.8, 134.3, 132.6, 132.2 (J = 5.0 Hz), 131.2, 131.1, 130.9, 129.1, 128.4, 122.2, 115.3 (J = 266 Hz), 59.4, 44.6 (J = 18 Hz), 31.1; HPLC (Chiralcel AD, *i*PrOH/hexane = 30/70, flow rate = 0.6 mL min^{-1}, λ = 210 nm): t_{major} = 13.73 min, t_{minor} = 20.90 min, ee > 99%; $[\alpha]_D^{25}$ = -187.3 (c = 1.42 in $CHCl_3$).



4d

(*S*)-4-Fluoro-4,4-bis(phenylsulfonyl)-3-(4-(trifluoromethyl)phenyl)butan-1-ol (4d) (Table 2, entry 4)

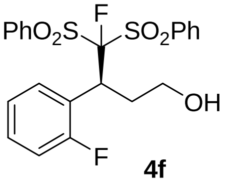
The title compound was prepared according to the general procedure, as described above in 80% yield. ^1H NMR (500 MHz, $CDCl_3$): δ 7.88 (d, J = 8.0 Hz, 2H), 7.78 (d, J = 8.0 Hz, 2H), 7.74 (t, J = 7.5 Hz, 1H), 7.64 (t, J = 7.5 Hz, 1H), 7.55 (t, J = 7.5 Hz, 2H), 7.44 (m, 4H), 7.20 (d, J = 7.5 Hz, 2H), 4.30 (d, J = 12 Hz, 1H), 3.81 (m, 1H), 3.22 (m, 1H), 3.10 (m, 1H), 2.80 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 137.4, 136.6, 135.4, 134.9, 134.3, 131.4, 131.2, 131.0, 130.2, 130.0, 129.1, 128.5, 125.0, 122.9, 115.2 (J = 267 Hz), 59.3, 44.8 (J = 17.5 Hz), 31.0; HPLC (Chiralcel AD, *i*PrOH/hexane = 30/70, flow rate = 0.6 mL min^{-1}, λ = 210 nm): t_{major} = 10.55 min, t_{minor} = 14.85 min, ee > 99%; $[\alpha]_D^{25}$ = -163.3 (c = 1.16 in $CHCl_3$).



4e

(*S*)-4-Fluoro-3-(3-nitrophenyl)-4,4-bis(phenylsulfonyl)butan-1-ol (4e) (Table 2, entry 5)

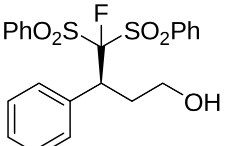
The title compound was prepared according to the general procedure, as described above in 75% yield. ^1H NMR (500 MHz, $CDCl_3$): δ 8.08 (d, J = 7.5 Hz, 1H), 7.88 (d, J = 7.0 Hz, 2H), 7.77 (m, 4H), 7.64 (m, 1H), 7.57 (m, 3H), 7.44 (m, 3H), 4.36 (d, J = 11.5 Hz, 1H), 3.84 (m, 1H), 3.23 (m, 1H), 3.11 (m, 1H), 2.83 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 147.9, 136.9, 136.4, 135.6, 135.5 (J = 5.6 Hz), 135.0, 134.0, 131.1, 131.0, 129.2, 129.0, 128.8, 128.5, 126.0, 123.0, 114.7 (J = 266 Hz), 59.1, 44.7 (J = 17 Hz), 30.9; HPLC (Chiralcel OD-H, *i*PrOH/hexane = 30/70, flow rate = 0.6 mL min^{-1}, λ = 210 nm): t_{major} = 15.84 min, t_{minor} = 19.75 min, ee = 97%; $[\alpha]_D^{25}$ = -182.1 (c = 0.92 in $CHCl_3$).



4f

(*S*)-4-Fluoro-3-(2-fluorophenyl)-4,4-bis(phenylsulfonyl)butan-1-ol (4f) (Table 2, entry 6)

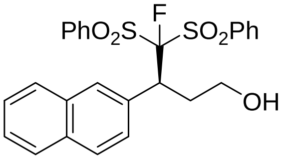
The title compound was prepared according to the general procedure, as described above in 74% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.89 (m, 4H), 7.78 (t, *J* = 7.0 Hz, 1H), 7.71 (t, *J* = 7.5 Hz, 1H), 7.66 (t, *J* = 7.0 Hz, 1H), 7.54 (t, *J* = 8.0 Hz, 2H), 7.48 (t, *J* = 8.0 Hz, 2H), 7.21 (m, 1H), 7.14 (t, *J* = 7.0 Hz, 1H), 6.79 (t, *J* = 9.0 Hz, 1H), 4.52 (d, *J* = 12 Hz, 1H), 3.80 (m, 1H), 3.26 (m, 1H), 3.14 (m, 1H), 2.85 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ162.4 (*J* = 246 Hz), 136.8, 135.2, 134.8, 134.2, 131.5, 131.2, 129.6 (*J* = 8Hz), 129.0, 128.4, 123.9, 120.4, 115.0 (*J* = 265 Hz), 114.8 (*J* = 23 Hz), 59.6, 36.9 (*J* = 17 Hz), 30.2; HPLC (Chiralcel AD, *i*PrOH/hexane = 30/70, flow rate = 0.6 mL min^{-1}, λ = 210 nm): t_{major} = 11.642 min, t_{minor} = 17.575 min, ee > 99%; $[\alpha]_D^{25}$ = -207.4 (*c* = 1.09 in $CHCl_3$).



4g

(*S*)-4-Fluoro-3-phenyl-4,4-bis(phenylsulfonyl)butan-1-ol (4g) (Table 2, entry 7)

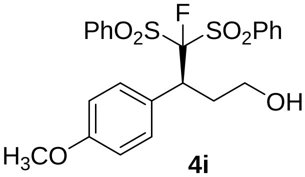
The title compound was prepared according to the general procedure, as described above in 71% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.88 (d, *J* = 8.0 Hz, 2H), 7.79 (d, *J* = 8.5 Hz, 2H), 7.73 (t, *J* = 7.5 Hz, 1H), 7.62 (t, *J* = 7.5 Hz, 1H), 7.54 (t, *J* = 8.0 Hz, 2H), 7.43 (t, *J* = 8.0 Hz, 2H), 7.19 (m, 3H), 7.07 (d, *J* = 7.0 Hz, 2H), 4.20 (d, *J* = 12 Hz, 1H), 3.79 (m, 1H), 3.27 (m, 1H), 3.05 (m, 1H), 2.81 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 136.9, 135.2, 134.7, 134.5, 133.1, 131.2, 131.0, 130.9, 129.0, 128.4, 128.1, 128.0, 115.6 (*J* = 264 Hz), 59.7, 45.2 (*J* = 16 Hz), 31.2; HPLC (Chiralcel AD, *i*PrOH/hexane = 30/70, flow rate = 0.6 mL min^{-1}, λ = 210 nm): t_{major} = 12.40 min, t_{minor} = 16.82 min, ee > 99%; $[\alpha]_D^{25}$ = -181.6 (*c* = 0.82 in $CHCl_3$).



4h

(*S*)-4-Fluoro-3-(naphthalen-2-yl)-4,4-bis(phenylsulfonyl)butan-1-ol (4h) (Table 2, entry 8)

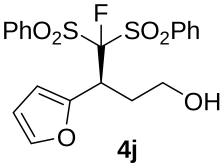
The title compound was prepared according to the general procedure, as described above in 42% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.90 (d, *J* = 7.5 Hz, 2H), 7.77-7.71 (m, 4H), 7.65 (d, *J* = 8.0 Hz, 2H), 7.57-7.52 (m, 3H), 7.45-7.42 (m, 3H), 7.35 (t, *J* = 8.0 Hz, 2H), 7.24 (t, *J* = 7.0 Hz, 1H), 4.41 (m, 1H), 3.81 (m, 1H), 3.30 (m, 1H), 3.14 (m, 1H), 2.93 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 136.8, 135.1, 134.7, 134.6, 132.9, 131.2, 130.9, 130.5, 129.0, 128.3, 128.2, 128.0, 127.7, 127.4, 126.3, 126.0, 115.9 (*J* = 266 Hz), 59.8, 45.2 (*J* = 18 Hz), 31.3; HPLC (Chiralcel AD, *i*PrOH/hexane = 20/80, flow rate = 0.6 mL min^{-1}, λ = 210 nm): t_{major} = 21.72 min, t_{minor} = 31.53 min, ee = 96%; $[\alpha]_D^{25}$ = -211.0 (*c* = 1.23 in $CHCl_3$).



4i

(*S*)-4-Fluoro-3-(4-methoxyphenyl)-4,4-bis(phenylsulfonyl)butan-1-ol (4i) (Table 2, entry 9)

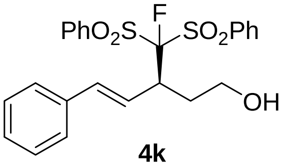
The title compound was prepared according to the general procedure, as described above in 71% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.87 (d, *J* = 7.5 Hz, 2H), 7.78 (d, *J* = 7.5 Hz, 2H), 7.71 (t, *J* = 7.5 Hz, 1H), 7.62 (t, *J* = 7.5 Hz, 1H), 7.53 (t, *J* = 7.5 Hz, 2H), 7.43 (t, *J* = 7.5 Hz, 2H), 6.99 (d, *J* = 8.5 Hz, 2H), 6.71 (d, *J* = 8.5 Hz, 2H), 4.14 (d, *J* = 13.5 Hz, 1H), 3.75 (m, 4H), 3.29 (m, 1H), 3.02 (m, 1H), 2.75 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 159.2, 136.9, 135.1, 134.6, 132.0, 131.1, 130.9, 129.0, 128.4, 124.9, 115.7 (*J* = 260 Hz), 113.5, 59.7, 55.1, 44.5 (*J* = 18 Hz), 31.4; HPLC (Chiralcel AD, *i*PrOH/hexane = 30/70, flow rate = 0.6 mL min^{-1}, λ = 210 nm): t_{major} = 14.89 min, t_{minor} = 23.48 min, ee = 92%; $[\alpha]_D^{25}$ = -165.0 (*c* = 0.89 in $CHCl_3$).



4j

(*S*)-4-Fluoro-3-(furan-2-yl)-4,4-bis(phenylsulfonyl)butan-1-ol (4j) (Table 2, entry 10)

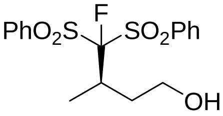
The title compound was prepared according to the general procedure, as described above in 79% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.94 (d, *J* = 8.5 Hz, 2H), 7.73-7.70 (m, 3H), 7.60 (t, *J* = 7.5 Hz, 1H), 7.55 (t, *J* = 8.0 Hz, 2H), 7.42 (t, *J* = 7.5 Hz, 2H), 7.18 (t, *J* = 1.0 Hz, 1H), 6.31 (d, *J* = 3.5 Hz, 1H), 6.22 (m, 1H), 4.34 (m, 1H), 3.83 (m, 1H), 3.43 (m, 1H), 2.90 (m, 1H), 2.71 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 147.2, 142.3, 136.3, 135.1, 134.6, 131.2, 130.7, 128.9, 128.5, 115.0 (*J* = 265 Hz), 111.5, 110.7, 59.9, 38.9 (*J* = 18 Hz), 30.5; HPLC (Chiralpak AS-H, *i*PrOH/hexane = 30/70, flow rate = 0.6 mL min^{-1}, λ = 210 nm): t_{major} = 26.17 min, t_{minor} = 43.57 min, ee = 86%; $[\alpha]_D^{25}$ = -54.0 (*c* = 1.41 in $CHCl_3$).



4k

(*S*, *E*)-3-(Fluorobis(phenylsulfonyl)methyl)-5-phenylpent-4-en-1-ol (4k) (Table 2, entry 11)

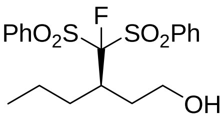
The title compound was prepared according to the general procedure, as described above in 77% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.84-7.80 (m, 4H), 7.62 (t, *J* = 7.5 Hz, 1H), 7.56 (t, *J* = 7.5 Hz, 1H), 7.45 (t, *J* = 8.0 Hz, 2H), 7.39 (t, *J* = 8.0 Hz, 2H), 7.20 (m, 4H), 7.16 (m, 1H), 6.20-6.09 (m, 2H), 3.75 (m, 1H), 3.49 (m, 2H), 2.71 (m, 1H), 2.16 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 136.5, 136.1, 135.1, 134.9, 131.1, 130.9, 128.9, 128.6, 128.5, 127.9, 126.6, 123.6 (*J* = 5.0 Hz), 115.6 (*J* = 264 Hz), 59.5, 44.4 (*J* = 17 Hz), 31.6; HPLC (Chiralpak AS-H, *i*PrOH/hexane = 30/70, flow rate = 0.6 mL min^{-1}, λ = 254 nm): t_{major} = 22.15 min, t_{minor} = 28.79 min, ee = 93%; $[\alpha]_D^{25}$ = -158.7 (*c* = 1.04 in $CHCl_3$).



4l

(*R*)-4-Fluoro-3-methyl-4,4-bis(phenylsulfonyl)butan-1-ol (4l) (Table 2, entry 12)

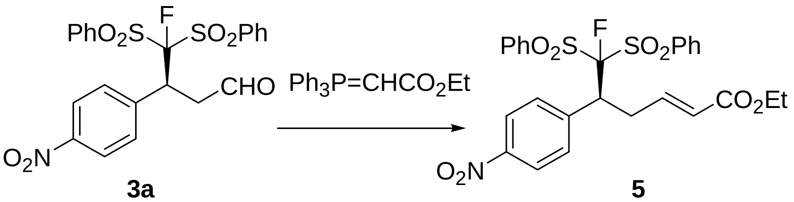
The title compound was prepared according to the general procedure, as described above in 66% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.86 (dd, *J* = 8.0, 16.5 Hz, 4H), 7.68 (m, 2H), 7.50 (m, 4H), 3.78 (m, 1H), 3.59 (m, 1H), 2.84 (m, 1H), 2.54 (m, 1H), 1.93 (m, 1H), 1.39 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 136.2, 135.7, 135.0, 134.9, 130.9, 130.9, 128.8, 128.7, 117.2 (*J* = 266 Hz), 60.1, 34.4 (*J* = 17 Hz), 33.2, 13.7 (*J* = 8 Hz); HPLC (Chiralcel OD-H, *i*PrOH/hexane = 30/70, flow rate = 0.6 mL min^{-1}, λ = 210 nm): t_{major} = 12.50 min, t_{minor} = 15.55 min, ee = 94%; $[\alpha]_D^{25}$ = -12.4 (*c* = 0.68 in $CHCl_3$).



4m

(*R*)-3-(Fluorobis(phenylsulfonyl)methyl)hexan-1-ol (4m) (Table 2, entry 14)

The title compound was prepared according to the general procedure, as described above in 81% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.89 (m, 4H), 7.68 (m, 2H), 7.52 (m, 4H), 3.68 (m, 1H), 3.56 (m, 1H), 2.66-2.60 (m, 2H), 2.15 (m, 1H), 1.91 (m, 1H), 1.57 (m, 1H), 1.21-1.14 (m, 2H), 0.78 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 136.0, 135.3, 135.0, 135.0, 131.1, 131.0, 128.8, 128.7, 117.6 (*J* = 265 Hz), 61.1, 38.9 (*J* = 15 Hz), 33.1 (*J* = 5 Hz), 32.0 (*J* = 6 Hz), 22.6, 13.7; HPLC (Chiralcel OD-H, *i*PrOH/hexane = 30/70, flow rate = 0.6 mL min^{-1}, λ = 210 nm): t_{major} = 10.18 min, t_{minor} = 11.50 min, ee = 92%; $[\alpha]_D^{25}$ = -26.9 (*c* = 0.74 in $CHCl_3$).



3a **5**

(*S, E*)-ethyl 6-fluoro-5-(4-nitrophenyl)-6,6-bis(phenylsulfonyl)hex-2-enoate (5)

A mixture of aldehyde **3a** (13 mg, 0.026 mmol) and Wittig reagent $Ph_3P=CHCO_2Et$ (27.6 mg, 0.079 mmol) in 0.5 mL CH_2Cl_2 was stirred at room temperature for 12 hours. The mixture was then directly purified by silica gel chromatography, eluted by hexane/EtOAc = 2/1 to give 13 mg the desired product **5**, yield: 88%. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.07 (d, *J* = 9.0 Hz, 2H), 7.80 (d, *J* = 8.5 Hz, 2H), 7.75 (t, *J* = 7.5 Hz, 1H), 7.67-7.61 (m, 3H), 7.55 (t, *J* = 7.5 Hz, 2H), 7.40 (t, *J* = 7.5 Hz, 2H), 7.32 (d, *J* = 8.5 Hz, 2H), 6.60 (m, 1H), 5.83 (d, *J* = 15.5 Hz, 1H), 4.11-4.05 (m, 3H), 3.72-3.68 (m, 1H), 3.63-3.56 (m, 1H), 1.23 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 165.8, 147.7, 143.5, 139.7, 136.0, 135.6, 135.1, 134.2, 132.0, 130.9, 130.8, 129.3, 128.6, 124.5, 123.1, 113.4, 60.4, 47.8 (*J* = 17 Hz), 30.5, 14.1.

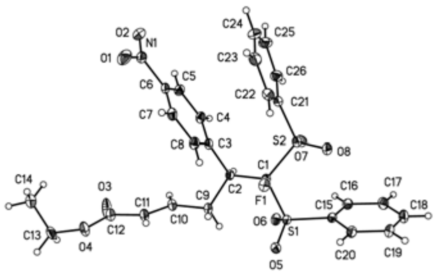
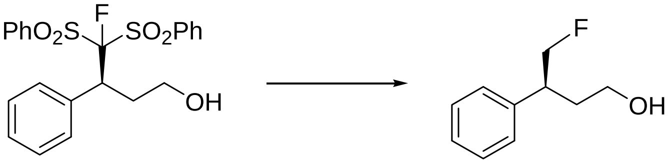


Figure S1. Single X-ray crystal structure **5**.



4g **6**

(*S*)-4-Fluoro-3-phenylbutan-1-ol (6)

Compound **4g** (15 mg, 0.033 mmol) was dissolved in 1 mL MeOH. Activated Mg (prepared from Mg and $BrCH_2CH_2Br$ in Et_2O) (16 mg, 0.66 mmol) was added to this solution and the mixture was stirred at room temperature for 2 h. HCl (5 mL, 1 mol/L) was added and extracted with Et_2O for three times. The combined organic layers were then washed with water and brine, dried (Na_2SO_4), and concentrated. The residue was then purified by silica gel chromatography, eluted by hexane/EtOAc = 3/1 to give 4.5 mg the desired product **6**, yield: 79%. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.33 (t, *J* = 7.5 Hz, 2H), 7.27-7.23 (m, 3H), 4.63-4.56 (m, 1H), 4.54-4.47 (m, 1H), 3.69-3.64 (m, 1H), 3.58-3.53 (m, 1H), 3.21-3.13 (m, 1H), 2.14-2.07 (m, 1H), 1.96-1.94 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 140.4 (*J* = 6 Hz), 128.7, 128.0, 127.1, 87.1 (*J* = 172 Hz), 60.6, 43.1 (*J* = 19 Hz), 34.5; HPLC (Chiralpak AS-H, *i*PrOH/hexane = 20/80, flow rate = 0.6 mL min^{-1}, λ = 210 nm): t_{major} = 9.31 min, t_{minor} = 11.53 min, ee = 98%; $[\alpha]_D^{25}$ = +54.0 (*c* = 0.35 in $CHCl_3$).

Crystallography Report

*Experimental for compound **5***

A few large colorless prisms, largest being about 2mm on an edge, were submitted. Optical examination showed them to be a bit cloudy. These were cut to size. The best was used for data collection. It diffracted xrays well and it measured 0.179 x 0.239 x 0.643 mm. It was mounted on a standard Bruker X8 Apex2 CCD-based X-ray diffractometer equipped with an Oxford Cryostream 700 low temperature device and normal focus Mo-target X-ray tube (λ = 0.71073 Å) operated at 1500 W power (50 kV, 30 mA). The X-ray intensities were measured at 228(2) K; the detector was placed at a distance 5.00 cm from the crystal. A full sphere of data consisting of 3872 frames was collected with a scan width of 0.5°in ω and phi with an exposure time of 12s/frame. Data collection took 18 hours. The frames were integrated with the Bruker SAINT software package with a narrow frame algorithm. The integration of the data yielded a total of 75139 reflections to a maximum 2θ value of 64.31°. Of these 9341 were independent. The final cell constants for data collection follow; these were based on the xyz centroids of 9911 reflections above 10σ(I).

```
CELL    13.3126  15.0787  14.0844  90.0000 114.0210  90.0000  2582.387
CELLSD   0.0008   0.0008   0.0008   0.0000   0.0027   0.0000     0.294
```

Analysis of the data showed negligible decay during data collection; the data were processed with SADABS and corrected for absorption. The structure was solved and refined with the Bruker SHELXTL (version 6.12) software package, using the chiral monoclinic space group P2(1). Refinement proceeds smoothly in this cell with one unique molecule and a total of four molecules in the unit cell.

All non-hydrogen atoms were refined anisotropically. The carbon hydrogen atoms were included in ideal positions. Their isotropic U's were fixed, U=1.5Uequiv of parent atom for methyl hydrogens and U=1.2Uequiv of parent atom for others. There is disorder in the molecules. Molecule 1 [S1-H26] is disordered at O3; oxygen at O3 occupies 2 positions: O3 with occupancy at 55.8% and O3A with occupancy at 44.2%. Molecule 2 [S2-H52] is disordered at the terminal methyl group C40; C40 occupies 2 positions: C40 at 64% occupancy and C40A at 36% occupancy. In the later cycles of refinement, the occupancies were fixed and the isotropic U's allowed to vary. When allowed to vary freely, the C39-C40 and C39-C40A bond lengths are 1.27-1.37Å with relatively large anisotropic U's. This is indicative of further disorder which is very smeared out about the primary disorder sites and would necessitate many more disordered positions for C40 and C40A. Rather than include more disordered positions for C40 and C40A, the model was refined with the C39-C40, C39-C40A distance fixed to 1.468Å [the same as the C13-C14 distance at the time] while allowing the anisotropic U's to vary. By fixing the position and occupancy of C39-C40/C40A we could calculate the positions of all the hydrogen atoms. The C-S bonds on C1 and C27 are dissimilar: C1-S1 and C27-S4 are about 1.908(2)Å, and C1-S2 and C27-S3 are about 1.854(2)Å. A cursory search through the Cambridge Crystallographic Data does not show any other structures with R-CF[S(O)2R'] unit. The final model represents the absolute geometry of the molecule as determined by anomalous dispersion effects of S. The geometries of C2 and C27 are S. There are some close interatomic approaches which may be hydrogen bonds. In general, there is no interaction between A and D if the interatomic distance between D and A is greater than the sum of their van der Waals radii. Interaction is likely to exist if this distance D—A is less than the sum of their van der Waals radii. If D and A are electronegative, and there is a hydrogen atom bonded to one, then the interaction is possibly the hydrogen bond. The hydrogen bond is a weak electrostatic dipole-dipole interaction with some features of covalent bonding: it is directional, strong, produces interatomic distances shorter than sum of van der Waals radii, and usually involves a limited number of interaction partners. Sum of van der Waals radii for C,F=3.44 Å, for C,O=3.22 Å. Also linear angles [DHA=180°] indicate strong hydrogen bonds. Generally speaking C-H—O,F type hydrogen bonds are not considered strong interactions. See Possible Hydrogen Bonds.

CheckCIF: alert levels PLAT413, PLAT301, PLAT161-163,PLAT752-757 are due to disorder and the fixing of positions[C39-C40 and associated hydrogen atoms] and are not serious.

Sheldrick, G.M. SHELXTL, v. 6.12; Bruker Analytical X-ray, Madison, WI, 2001.
Sheldrick, G.M. SADABS, v. 2.10. Program for Empirical Absorption Correction of Area Detector Data, University of Gottingen: Gottingen, Germany, 2003.
Saint Plus, v. 7.01, Bruker Analytical X-ray, Madison, WI, 2003.

Refinement Summary

Model	p--	wR2%*	R1(F)*	R1(F)	GOOF
1.all nonhydrogen atoms isotropic, Flack parameter –0.0251(919).	305	33.02	14.31	11.92	2.26
2.all nonhydrogen atoms anisotropic, no hydrogen atoms. Disorder on O3 and C40 included. Flack parameter -0.0142(920).	313	31.05	13.82	11.43	1.94
3.all nonhydrogen atoms anisotropic, no disorder. Flack parameter= -.029(511). C39-C40/C40A vary from 1.26-1.37A. Fix C39-C40/C40A to be equal to C12-C13.	703	17.55	7.93	5.82	1.08
4.final, all nonhydrogen atoms anisotropic. all disordered atoms with fixed occupancies;C39 C40,C40A positions. All hydrogens included; carbon hydrogens in ideal positions with fixed Uiso=1.5Uequiv of parent atoms [methyl] or Uiso=1.2Uequiv of parent atom[other carbon atoms]; Flack=0.0312(360). Low angle reflections omitted: 011, 0-11,110,-101, -1-11,-111,100,111,1-11,001, -102.	695	13.18	6.55	4.54	1.00

SPECIAL NOTES :

wR2 is based on F*F and with R1(F)* uses 17970 unique reflections. R1(F) and R1(F)* is based on F and R1(F) uses only the 13818 observed reflections [F>4 σ(Fo)]. Omitted reflections accounted for.

Fo=observed F	Fc=calculated F
n= observed reflections	p=parameters refined

$R(int)=\Sigma|Fo^2-Fo^2(mean)|/\Sigma|Fo^2|$
$R(sigma)=\Sigma[\sigma(Fo^2)]/\Sigma[Fo^2]$
$R_1=R1=\Sigma||Fo|-|Fc||/\Sigma|Fo|$
$wR_2=wR2=[\Sigma w(Fo^2-Fc^2)^2/\Sigma w(Fo)^2]^{1/2}$
$GOF=GOOF=S=[\Sigma w(Fo^2-Fc^2)^2/(n-p)]^{1/2}$

Flack x parameter = -0.0312 with esd 0.0360➔Flack parameter =-0.03(4)

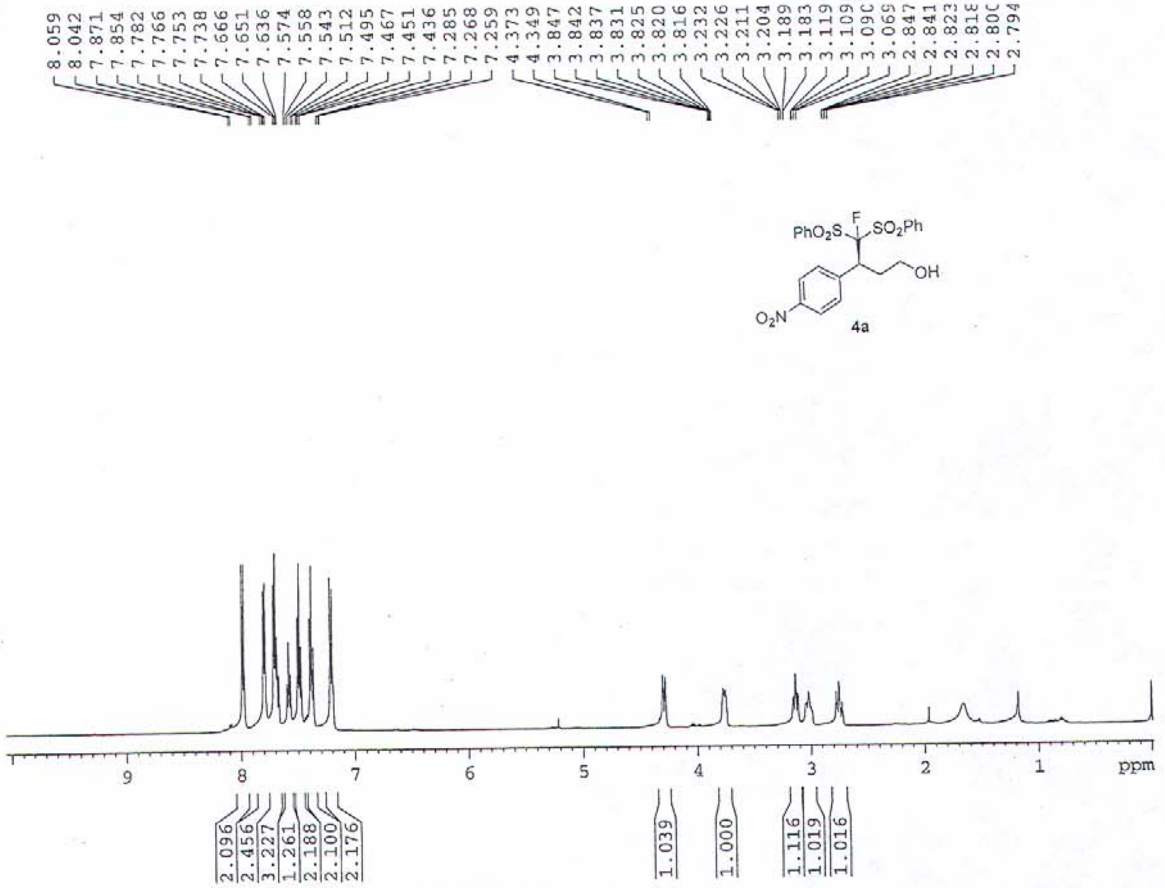
Expected values are 0 (within 3 esd's) for correct and +1 for inverted absolute structure. Note that this rough estimate ignores correlation with other parameters; if the above value differs significantly from zero, it is ESSENTIAL to test the nverted structure or refine x as a full-matrix parameter using TWIN and BASF

C26 H24 F N O8 S2 Shilei Zhang/W.Wang 25 November 2008 ww88

Formula weight =561.58 Temperature =228(2) K Wavelength =0.71073 Å
Crystal system =Monoclinic Space group =P2(1)Unit cell dimensions=a = 13.3126(8) Å
α= 90°. b = 15.0787(8) Å β= 114.021(3)°. c = 14.0844(8) Å γ = 90°.Volume=2582.4(3) Å^3
Z=4 Density (calculated)=1.444 Mg/m^{3}Absorption coefficient=0.265 mm^{-1}F(000)=1168
Crystal size=0.64 x 0.24 x 0.17 mm^{3}θ range for data collection=3.07 to 32.14°.
Index ranges=-19<=h<=19, -22<=k<=22, -20<=l<=20Reflections collected=75048
Independent reflections=17970 [R(int) = 0.0333]Completeness to theta = 32.14°=99.6 %
Absorption correction=Semi-empirical from equivalents
Max. and min. transmission=0.9551 and 0.8482
Refinement method=Full-matrix least-squares on F^2
Data / restraints / parameters=17970 / 0 / 695 Goodness-of-fit on F^2=1.003
Final R indices [I>2sigma(I)]=R1 = 0.0454, wR2 = 0.1189
R indices (all data)=R1 = 0.0655, wR2 = 0.1318 Absolute structure parameter=-0.03(4)
Largest diff. peak and hole=1.090 and -0.387 e.Å^{-3}
Charge=$367=250+2[77-30]+[53-30]

Table S1. Crystal data and structure refinement for Compound 5.

Identification code	Compound 5	
Empirical formula	$C_{26}H_{24}FNO_8S_2$	
Formula weight	561.58	
Temperature	228(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 13.3126(8) Å	α= 90°.
	b = 15.0787(8) Å	β= 114.021(3)°.
	c = 14.0844(8) Å	γ = 90°.
Volume	2582.4(3) Å^3	
Z	4	
Density (calculated)	1.444 Mg/m^3	
Absorption coefficient	0.265 mm^{-1}	
F(000)	1168	
Crystal size	0.64 x 0.24 x 0.17 mm^3	
Theta range for data collection	3.07 to 32.14°.	
Index ranges	-19<=h<=19, -22<=k<=22, -20<=l<=20	
Reflections collected	75048	
Independent reflections	17970 [R(int) = 0.0333]	
Completeness to theta = 32.14°	99.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9551 and 0.8482	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	17970 / 0 / 695	
Goodness-of-fit on F^2	1.003	
Final R indices [I>2sigma(I)]	R1 = 0.0454, wR2 = 0.1189	
R indices (all data)	R1 = 0.0655, wR2 = 0.1318	
Absolute structure parameter	-0.03(4)	
Largest diff. peak and hole	1.090 and -0.387 e.Å^{-3}	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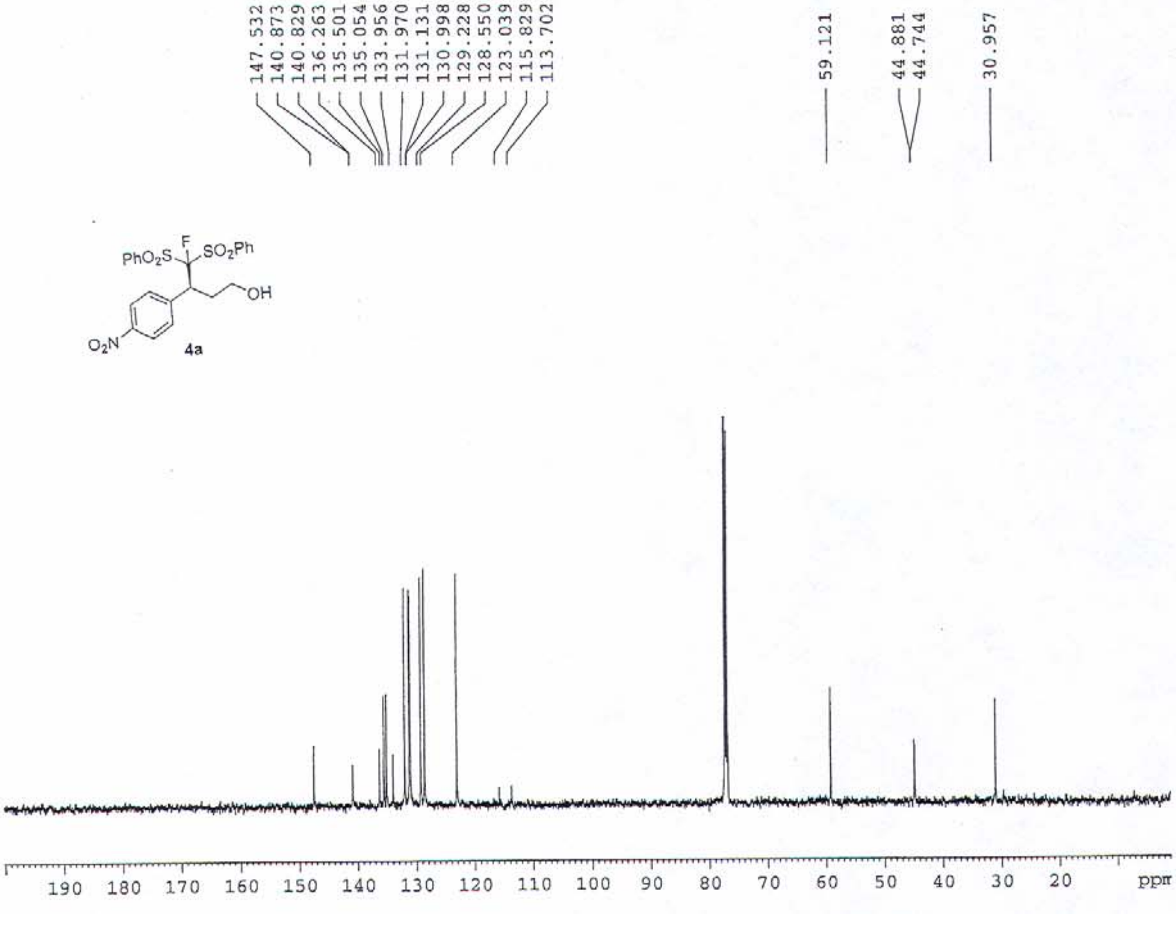
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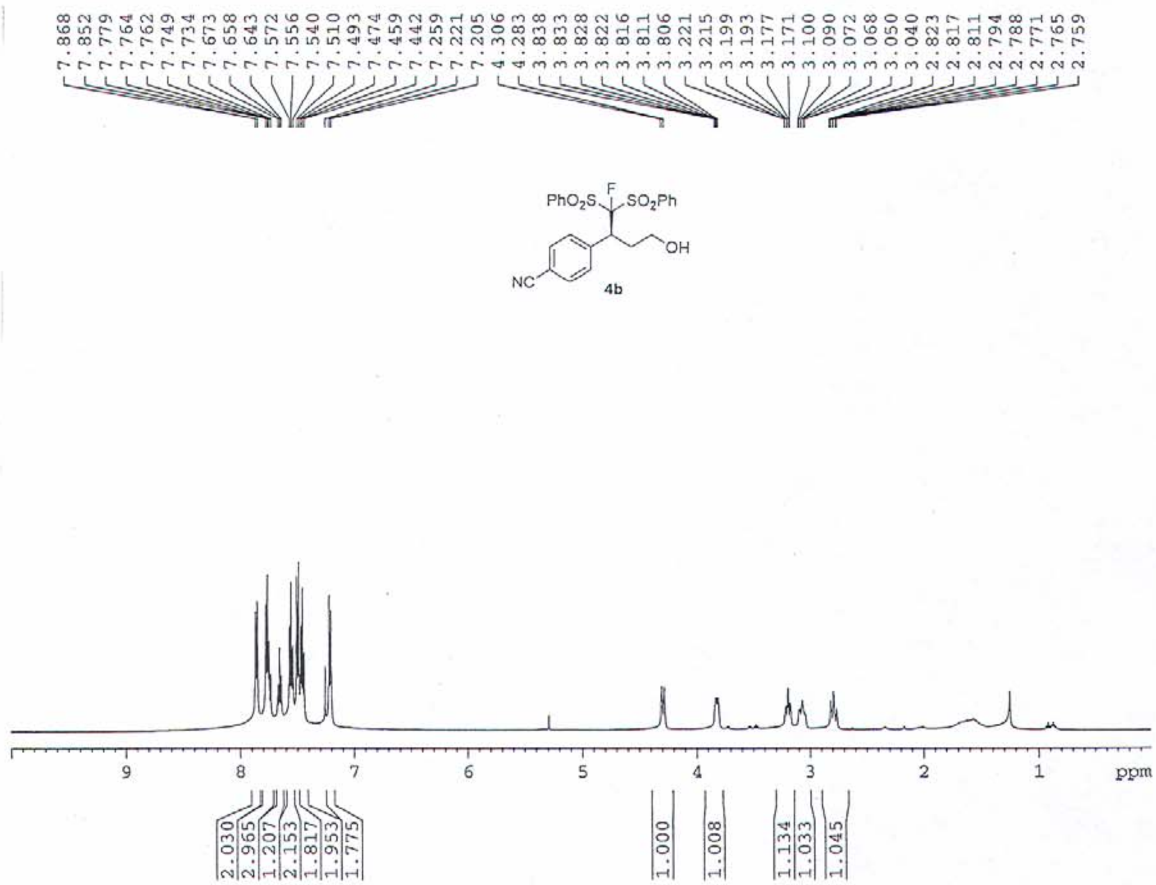
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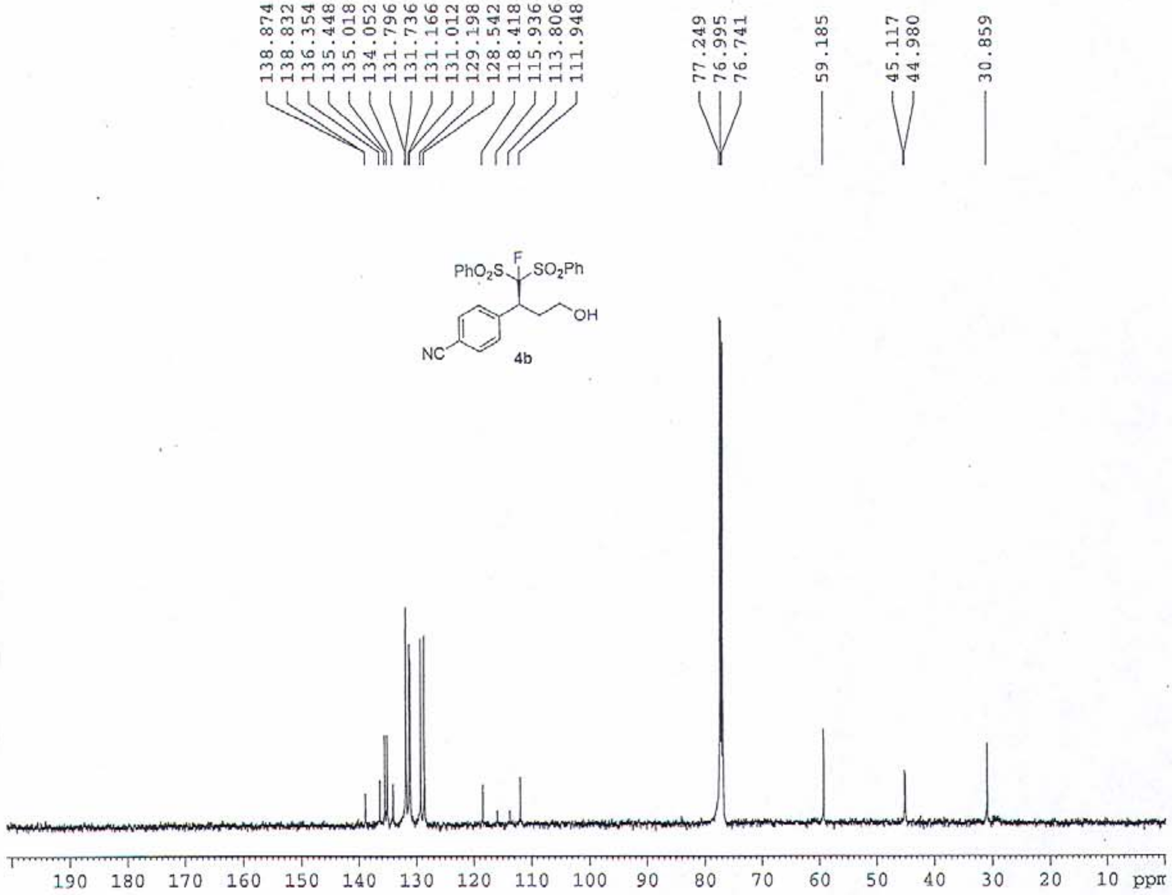
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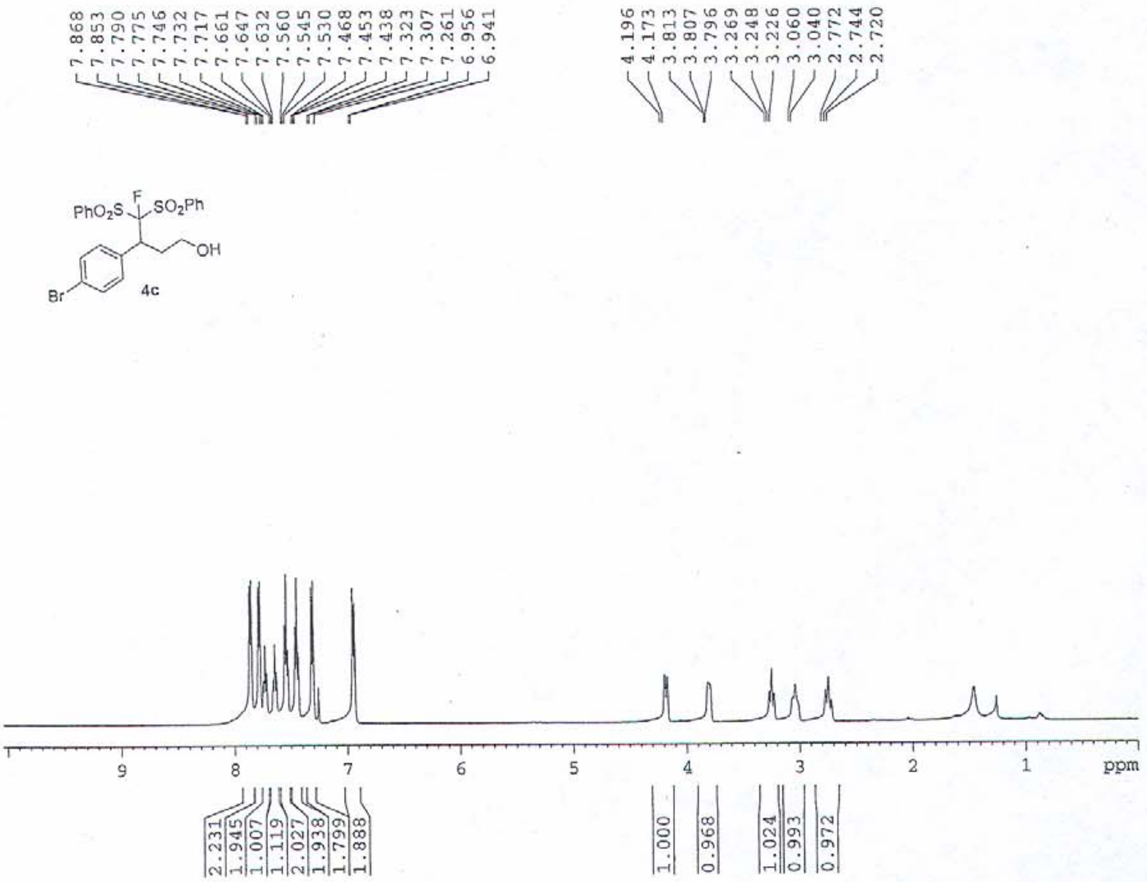
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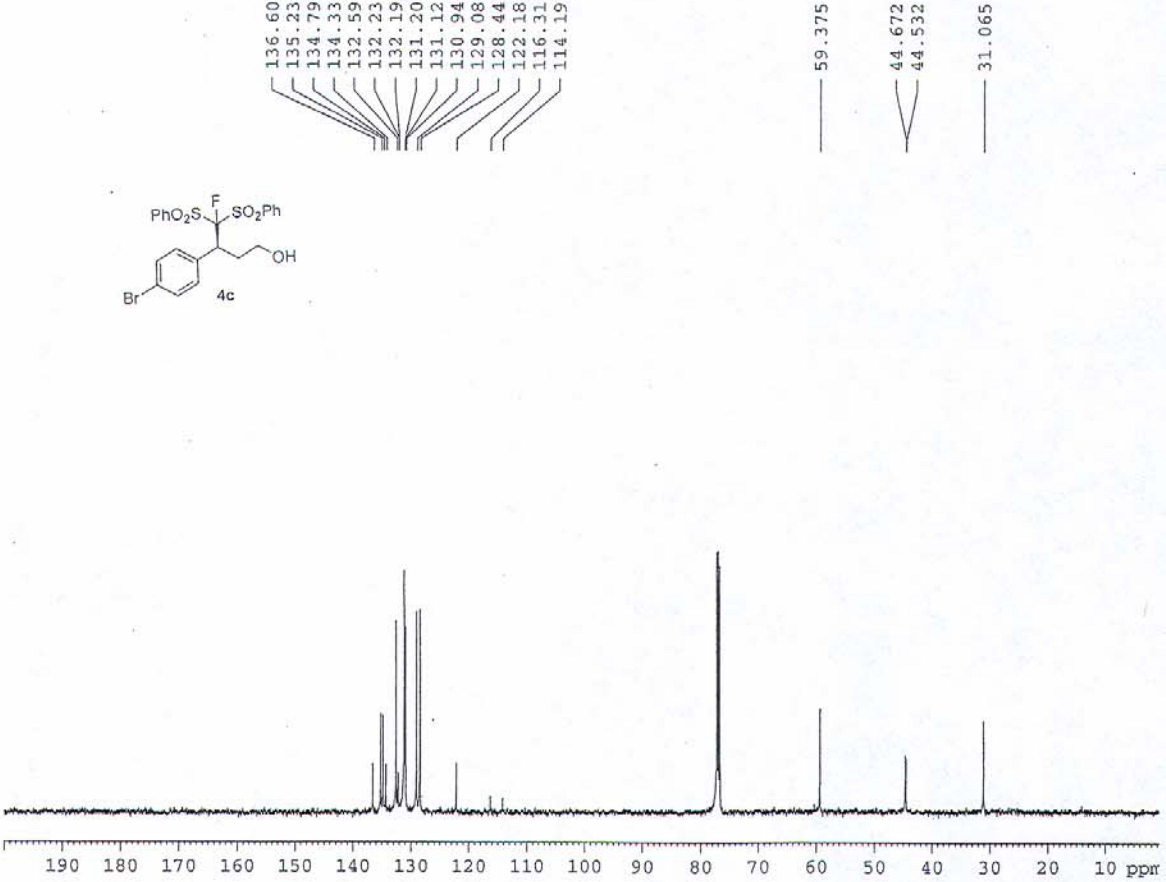
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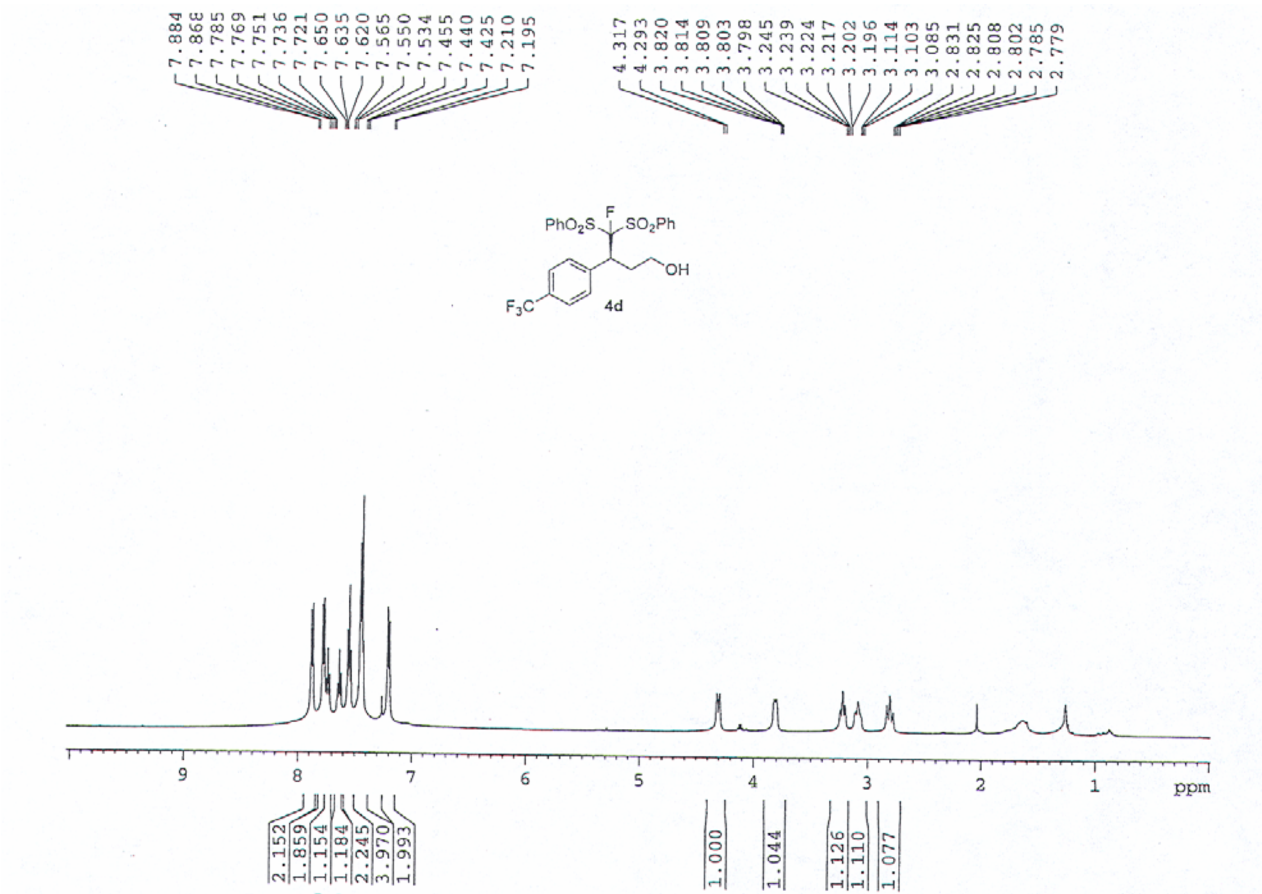
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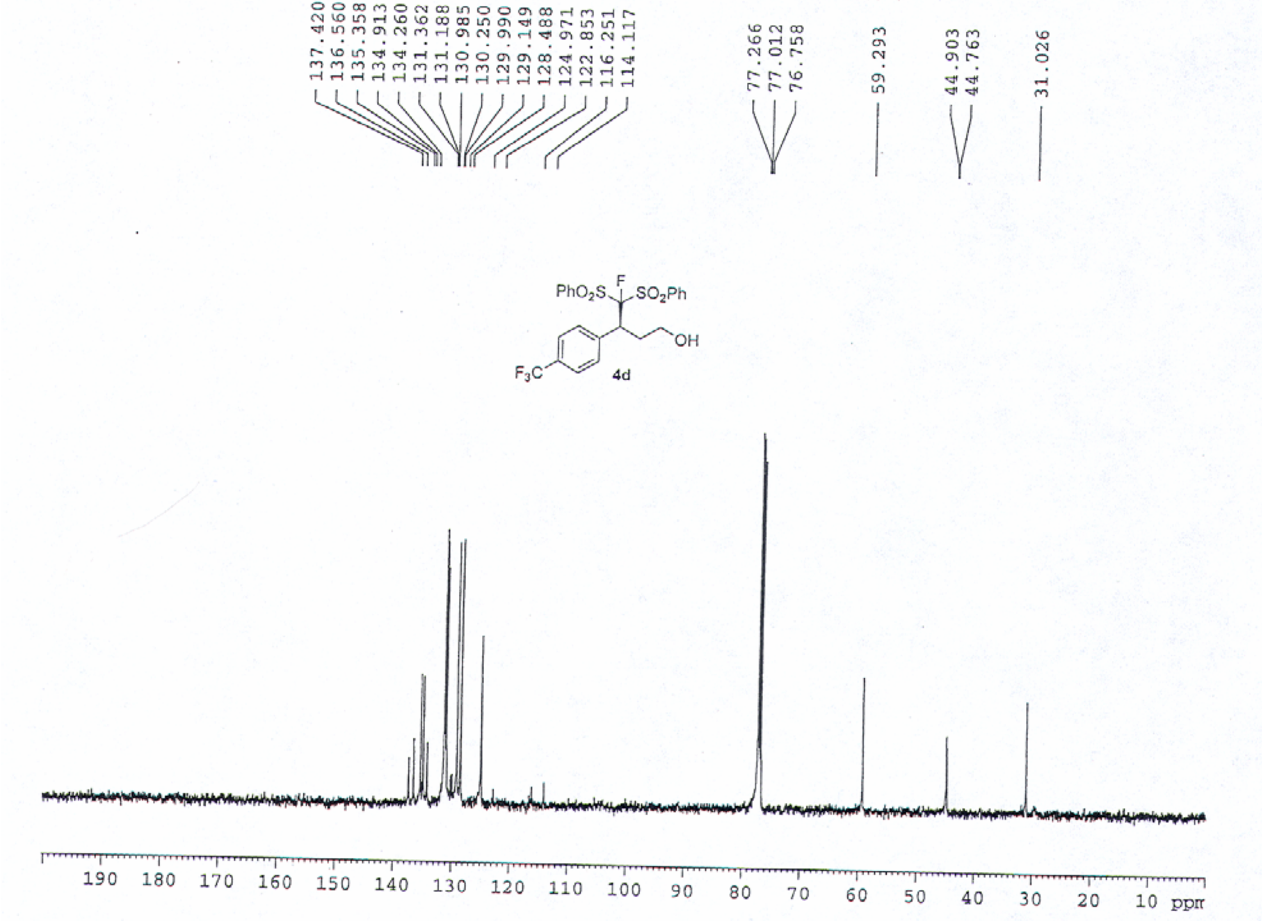
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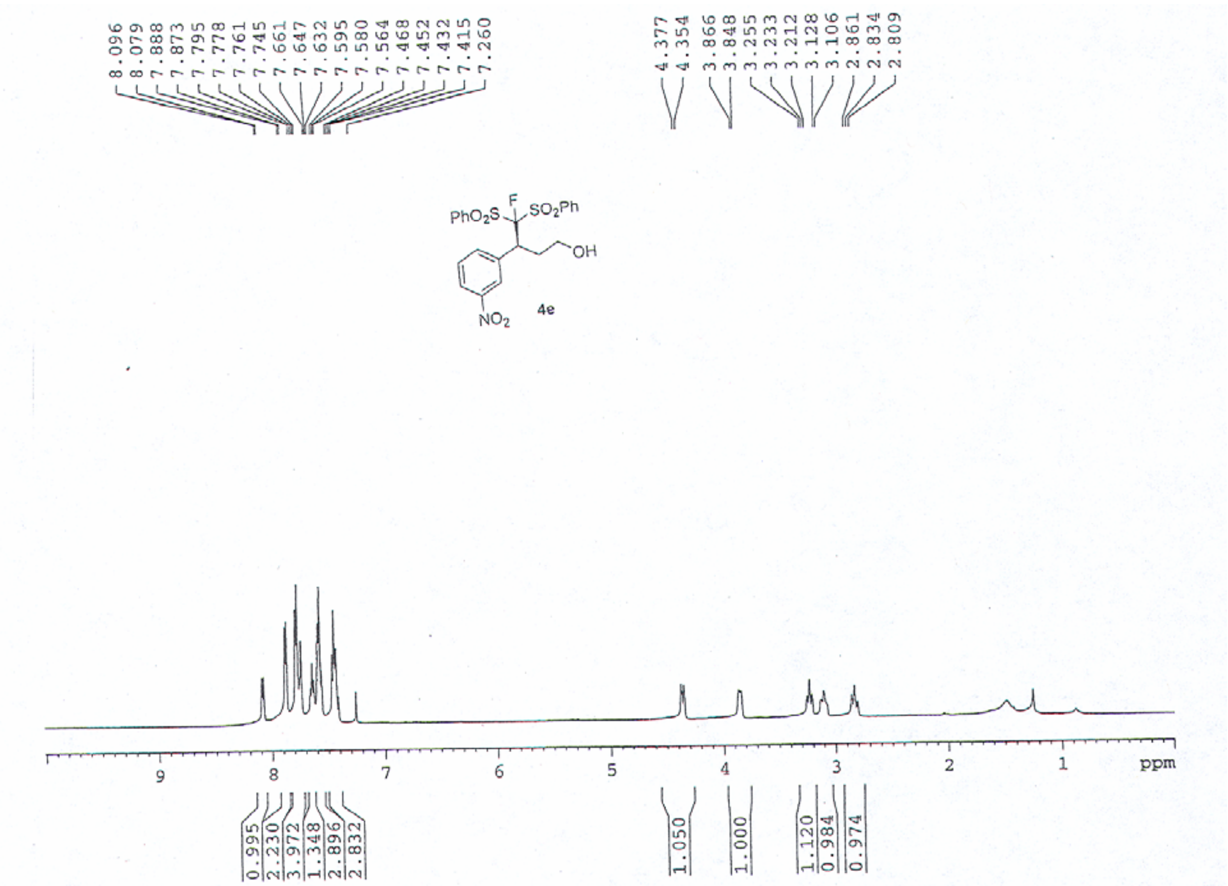
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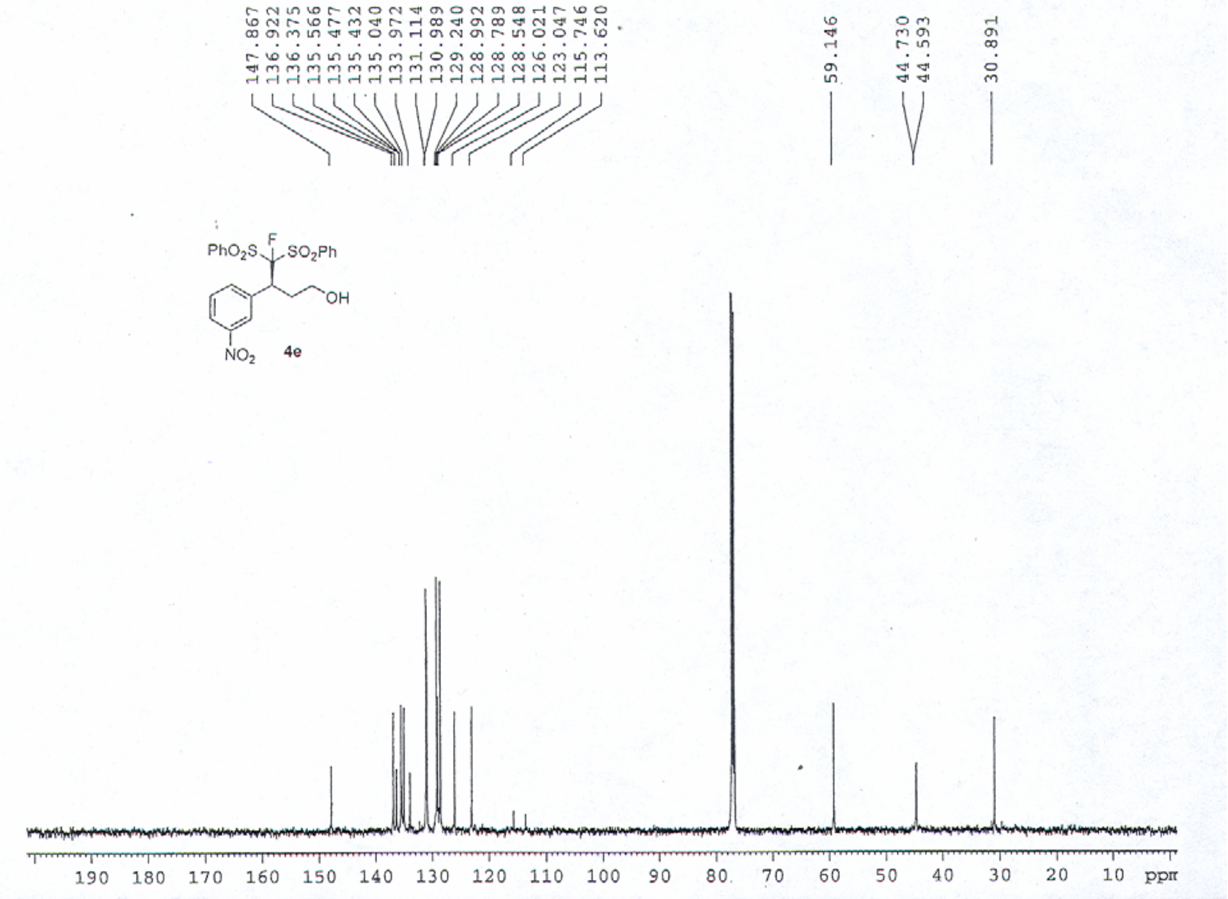
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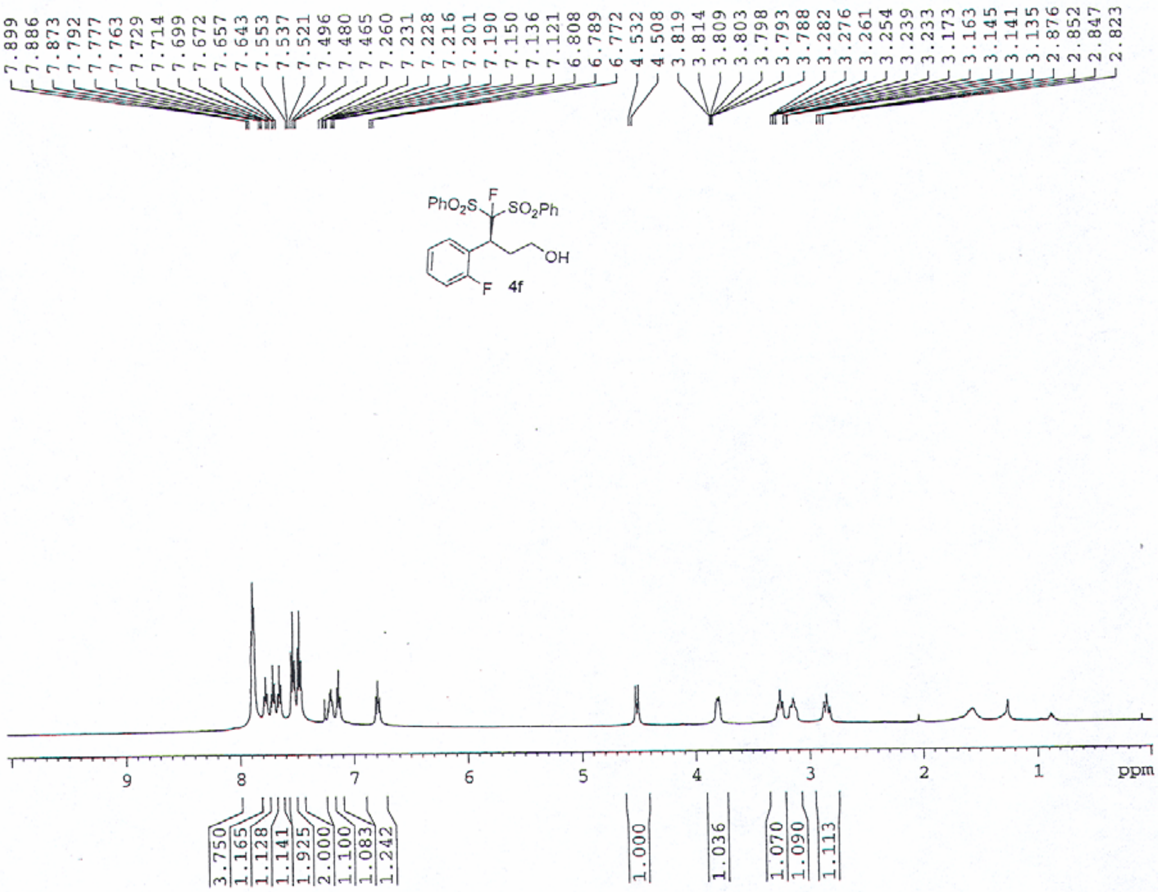
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6
5
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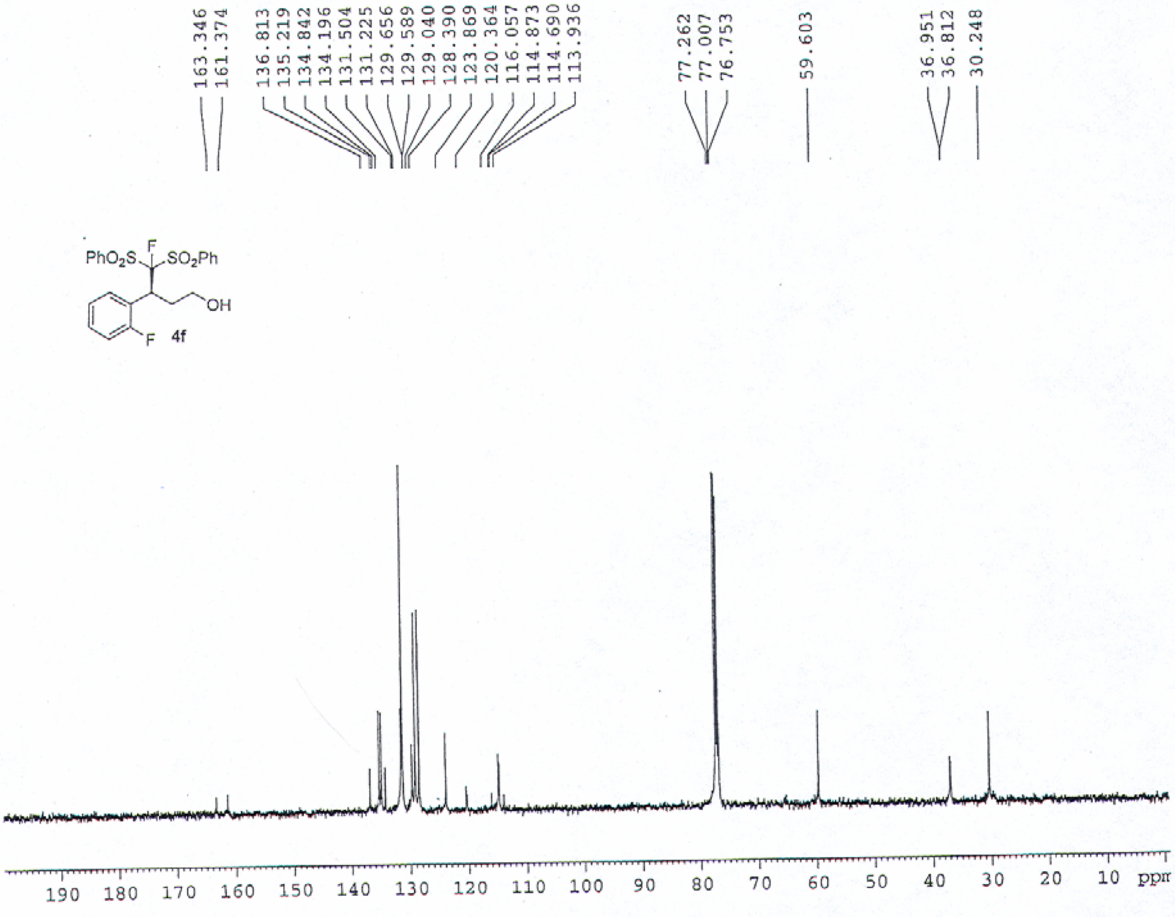
1.113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114.690
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30.248
PhO2S
F
SO2Ph
OH
F
4f
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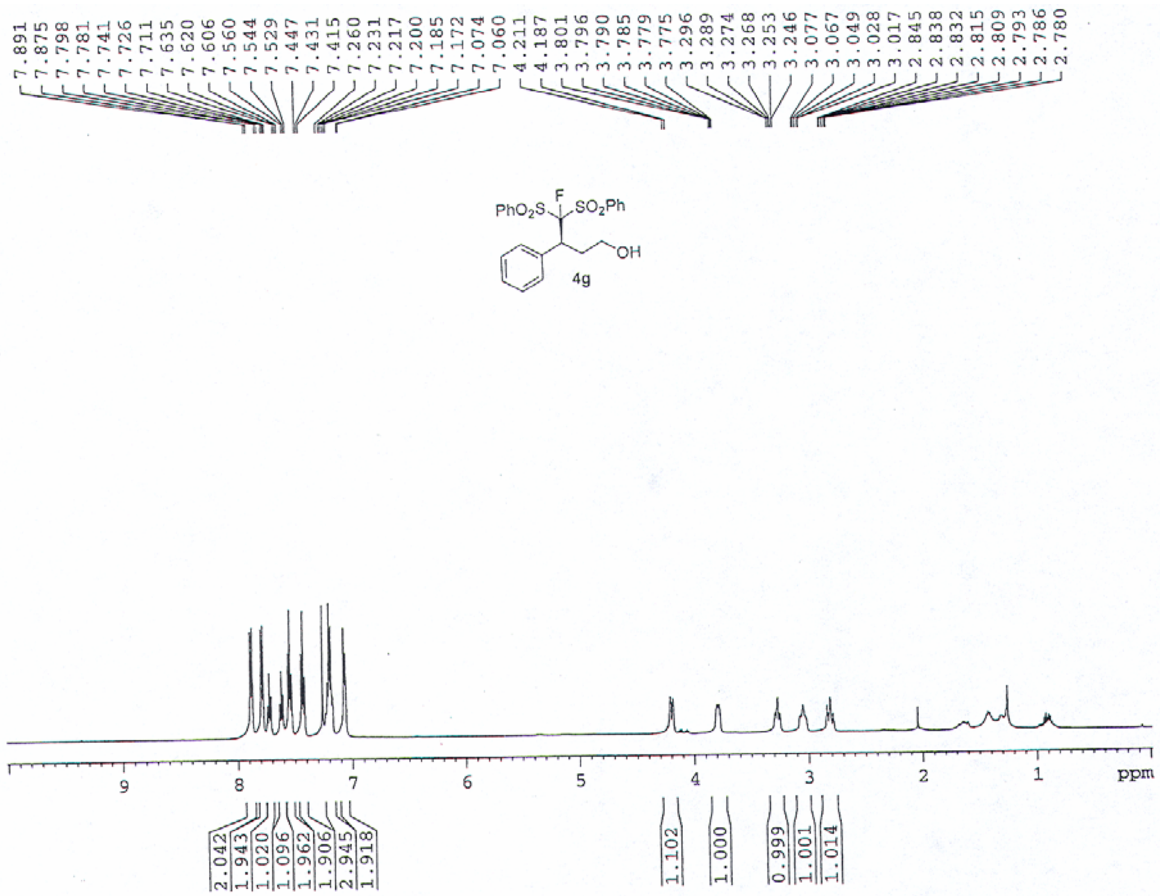
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7.529
7.447
7.431
7.415
7.260
7.231
7.217
7.200
7.185
7.172
7.074
7.060
4.211
4.187
3.801
3.796
3.790
3.785
3.779
3.775
3.296
3.289
3.274
3.268
3.253
3.246
3.077
3.067
3.049
3.028
3.017
2.845
2.838
2.832
2.815
2.809
2.793
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2.780
PhO2S
F
SO2Ph
OH
4g
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8
7
6
5
4
3
2
1
ppm
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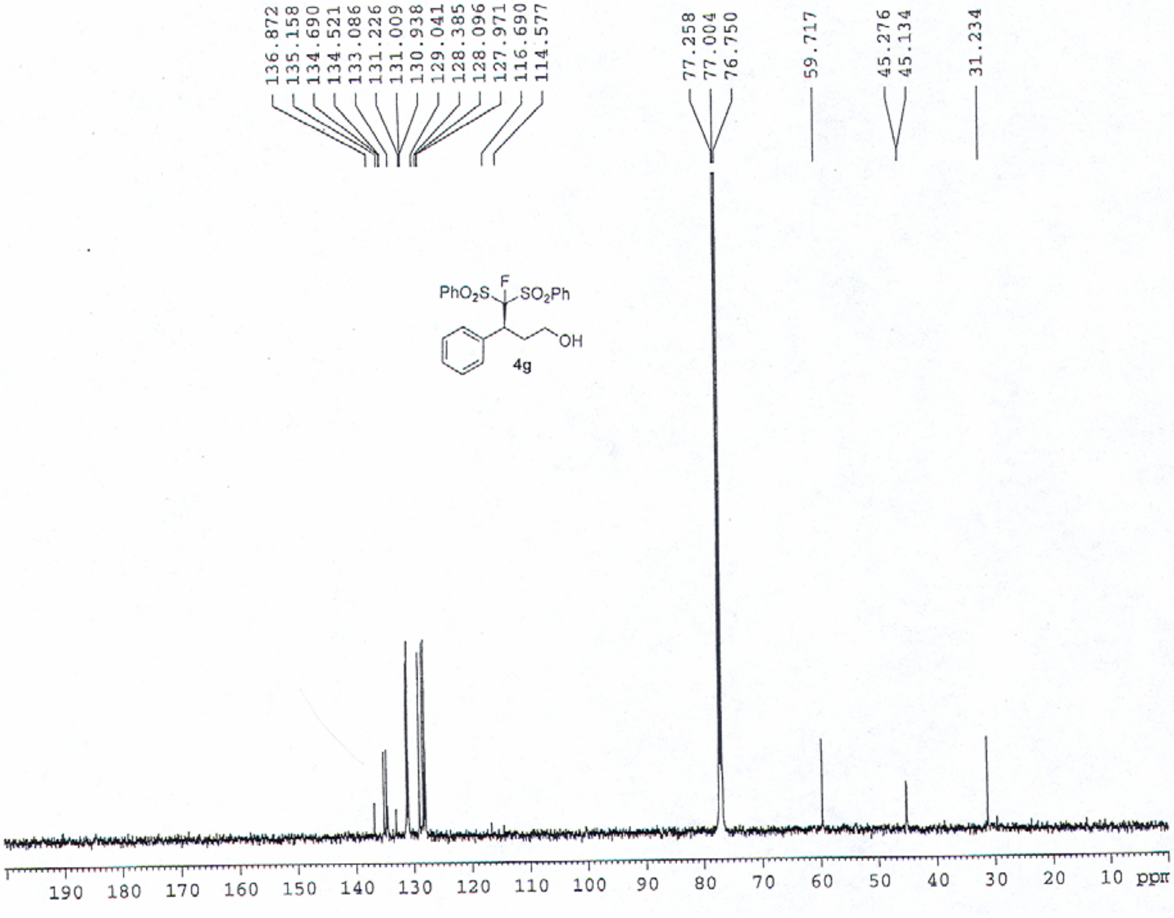
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59.717
45.276
45.134
31.234
PhO2S
F
SO2Ph
OH
4g
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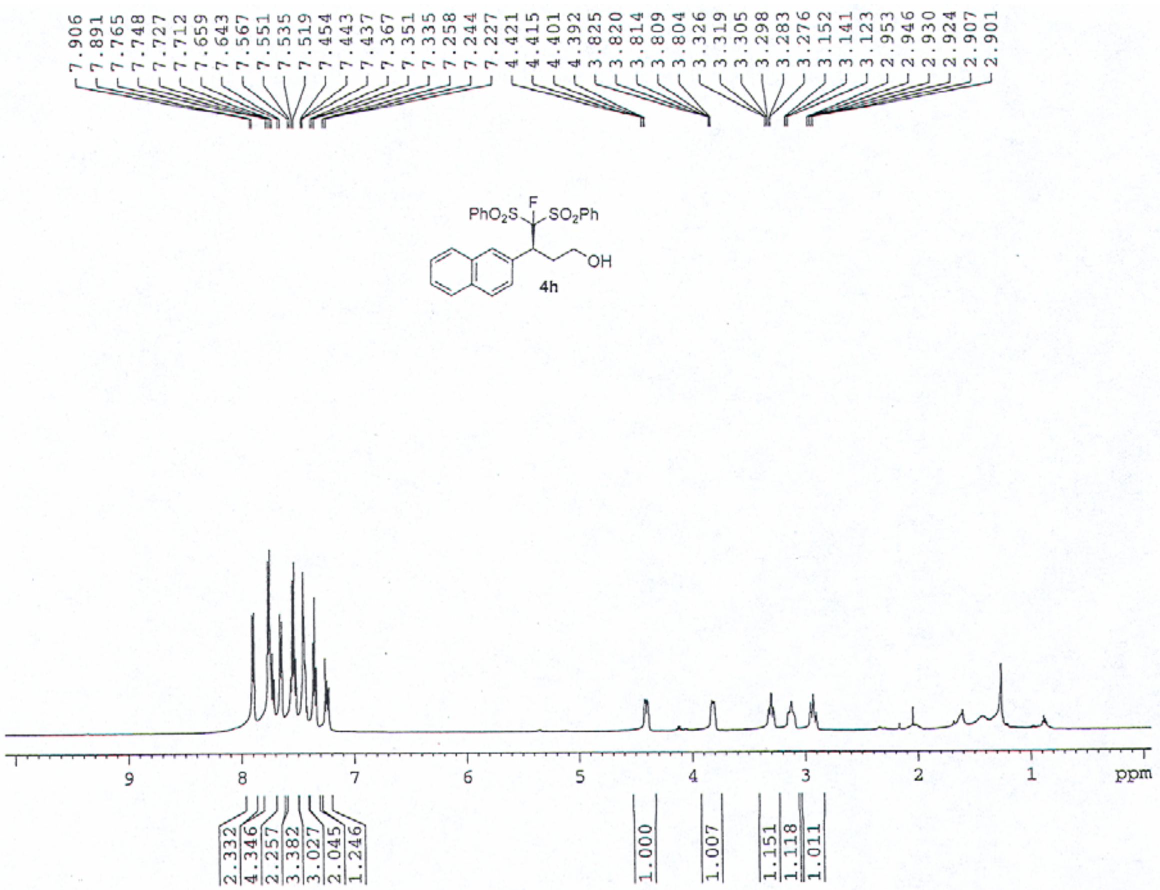
10
ppm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7.437
7.367
7.351
7.335
7.258
7.244
7.227
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4.415
4.401
4.392
3.825
3.820
3.814
3.809
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3.326
3.319
3.305
3.298
3.283
3.276
3.152
3.141
3.123
2.953
2.946
2.930
2.924
2.907
2.901
PhO2S
F
SO2Ph
OH
4h
9
8
7
6
5
4
3
2
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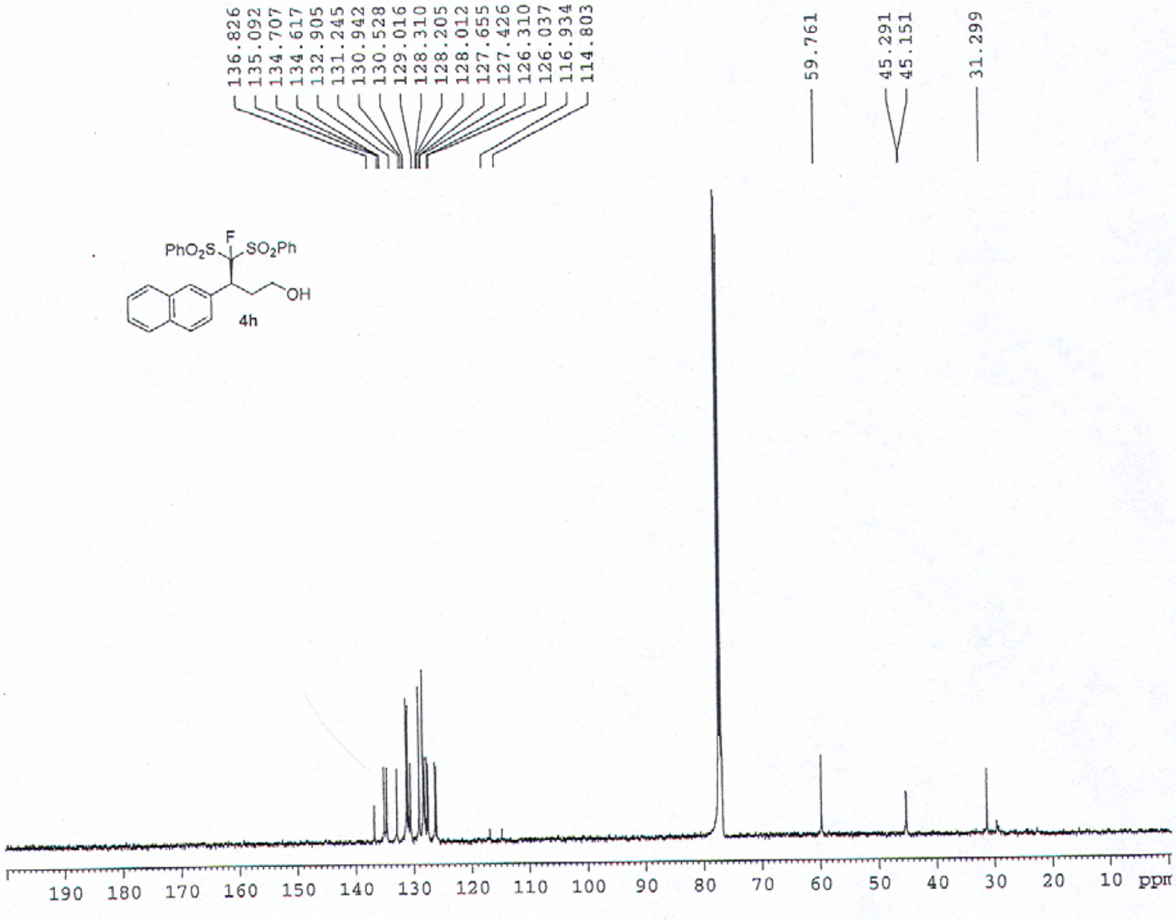
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127.426
126.310
126.037
116.934
114.803
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PhO2S
F
SO2Ph
OH
4h
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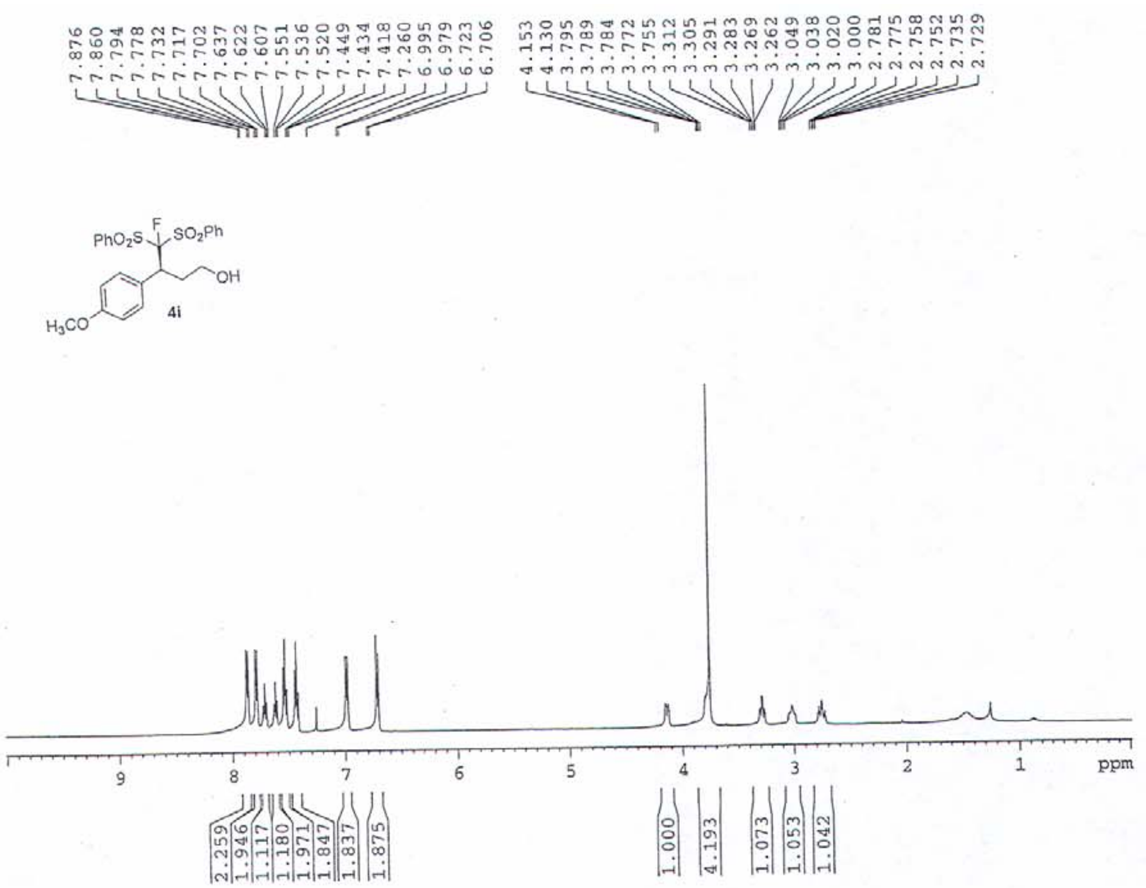
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3.049
3.038
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3.000
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2.735
2.729
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F
SO2Ph
OH
H3CO
4i
9
8
7
6
5
4
3
2
1
ppm
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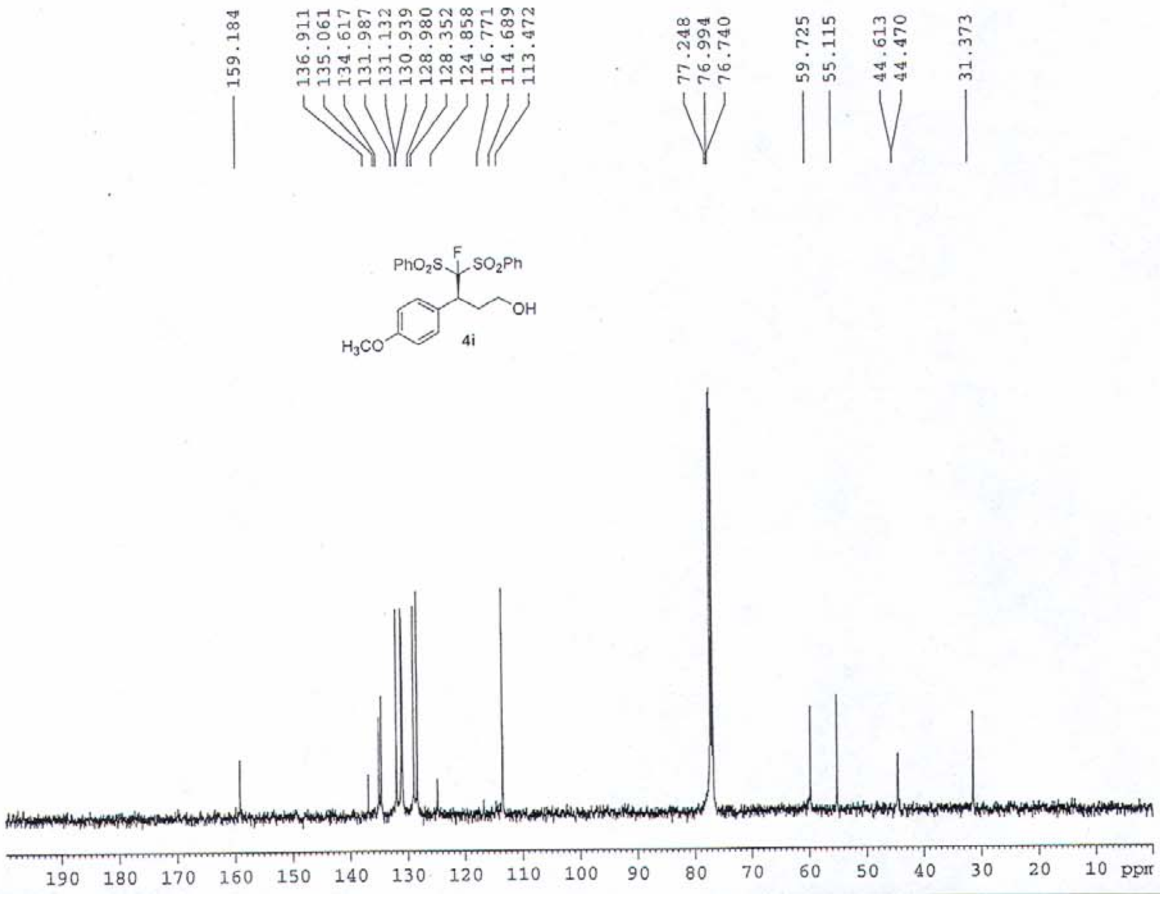
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77.248
76.994
76.740
59.725
55.115
44.613
44.470
31.373
PhO2S
F
SO2Ph
OH
H3CO
4i
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30
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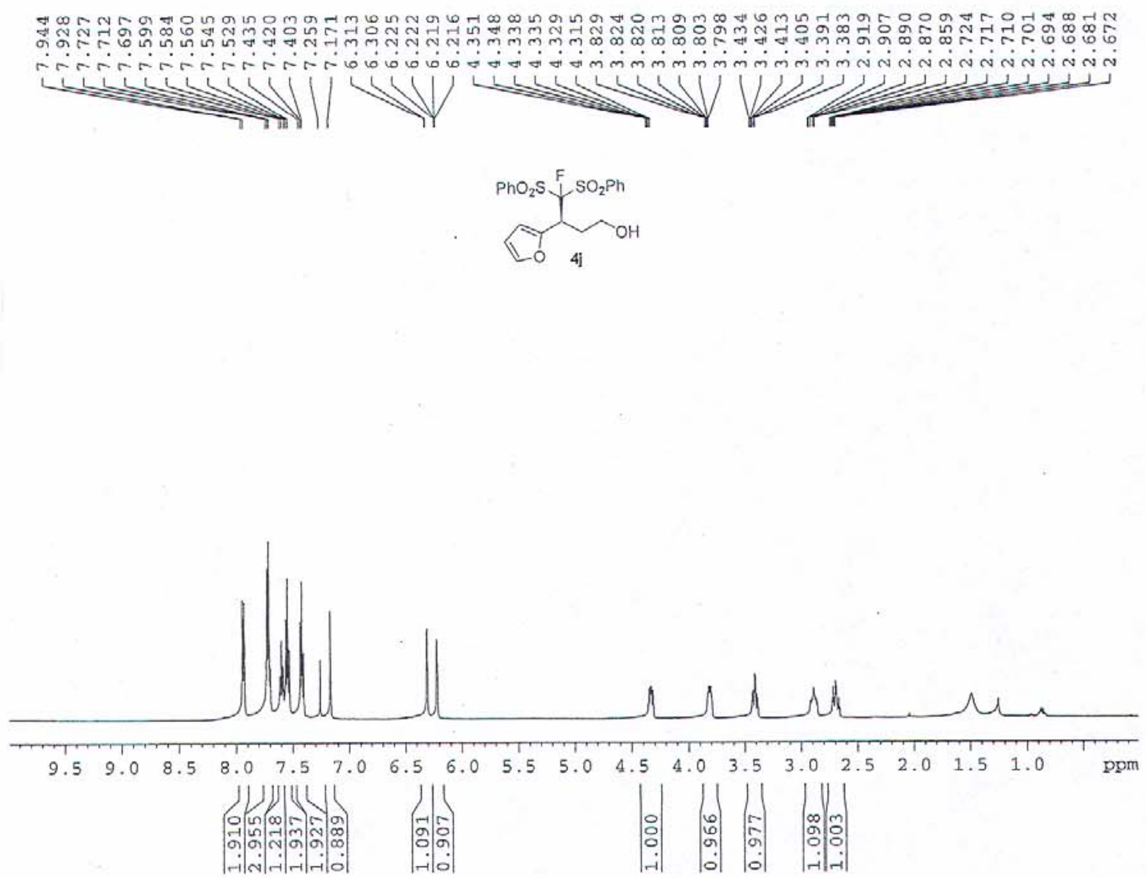
ppm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3.413
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2.907
2.890
2.870
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2.710
2.701
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2.688
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2.672
PhO2S
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SO2Ph
OH
O
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6.0
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5.0
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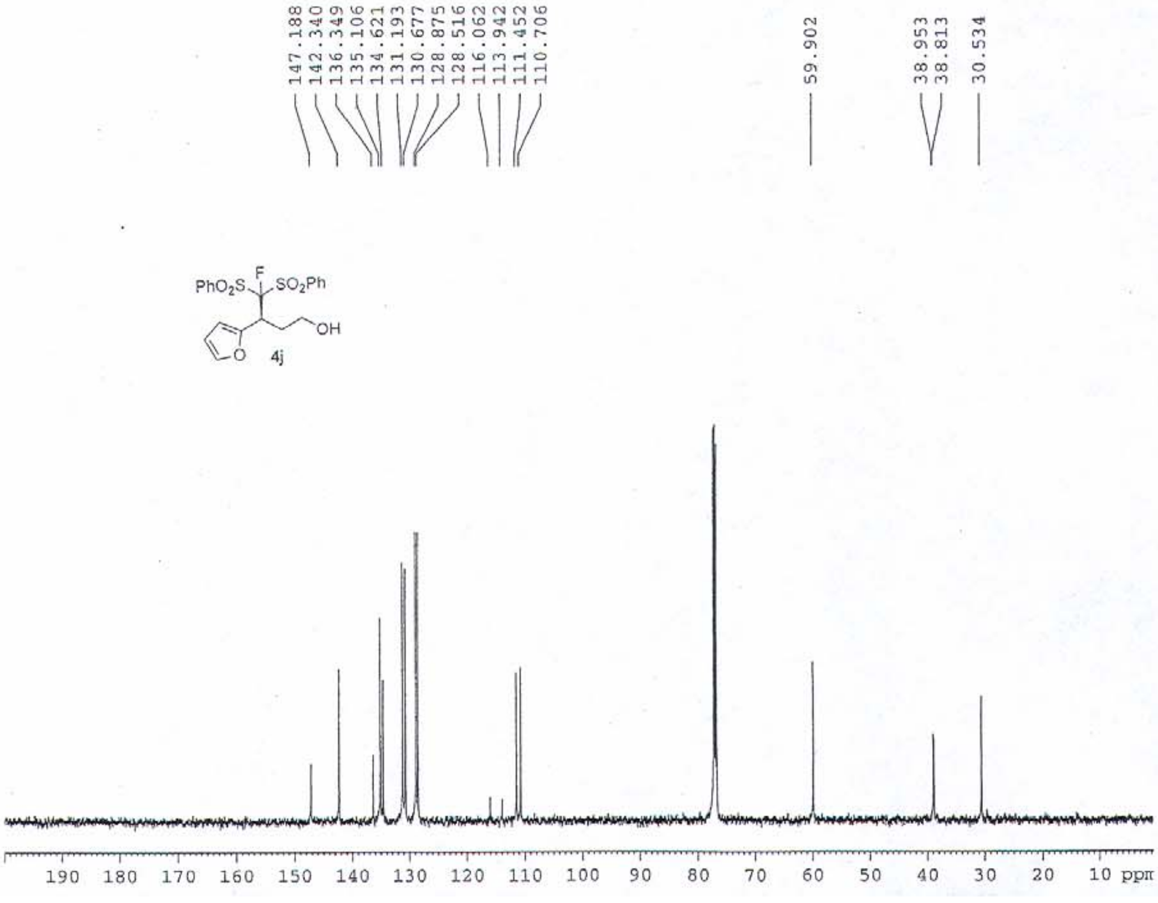
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38.813
30.534
PhO2S
F
SO2Ph
OH
O
4j
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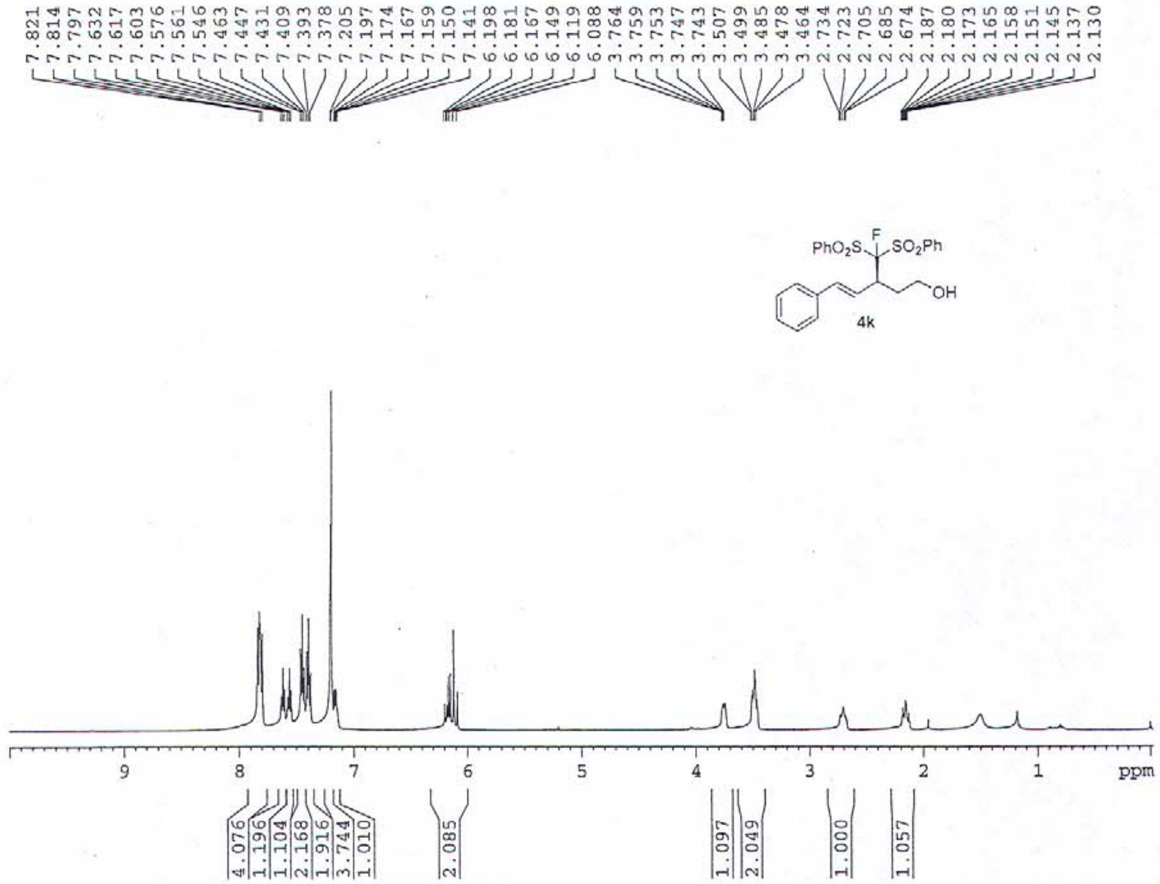
ppm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7.159
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6.167
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3.743
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3.485
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3.464
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2.723
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2.674
2.187
2.180
2.173
2.165
2.158
2.151
2.145
2.137
2.130
PhO2S
F
SO2Ph
OH
4k
9
8
7
6
5
4
3
2
1
ppm
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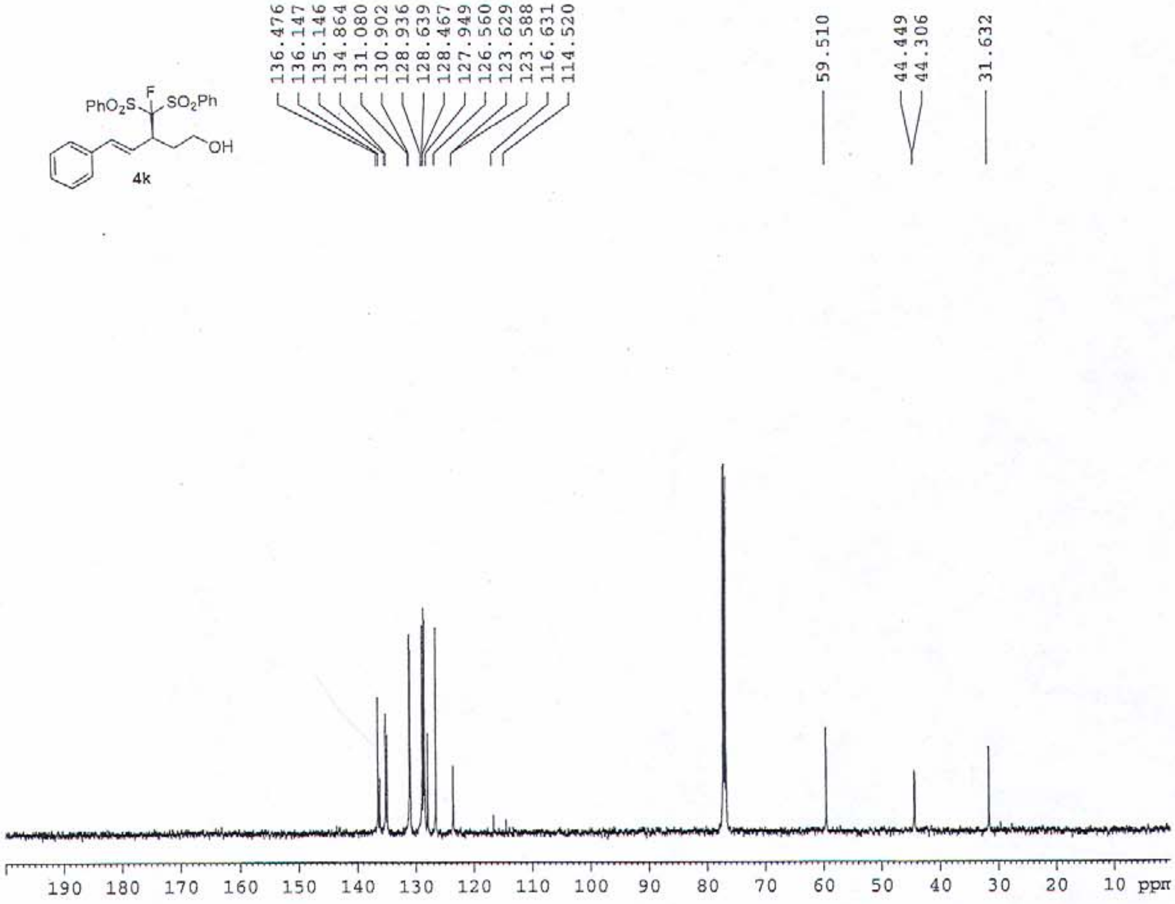
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123.588
116.631
114.520
59.510
44.449
44.306
31.632
PhO2S
F
SO2Ph
OH
4k
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160
150
140
130
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90
80
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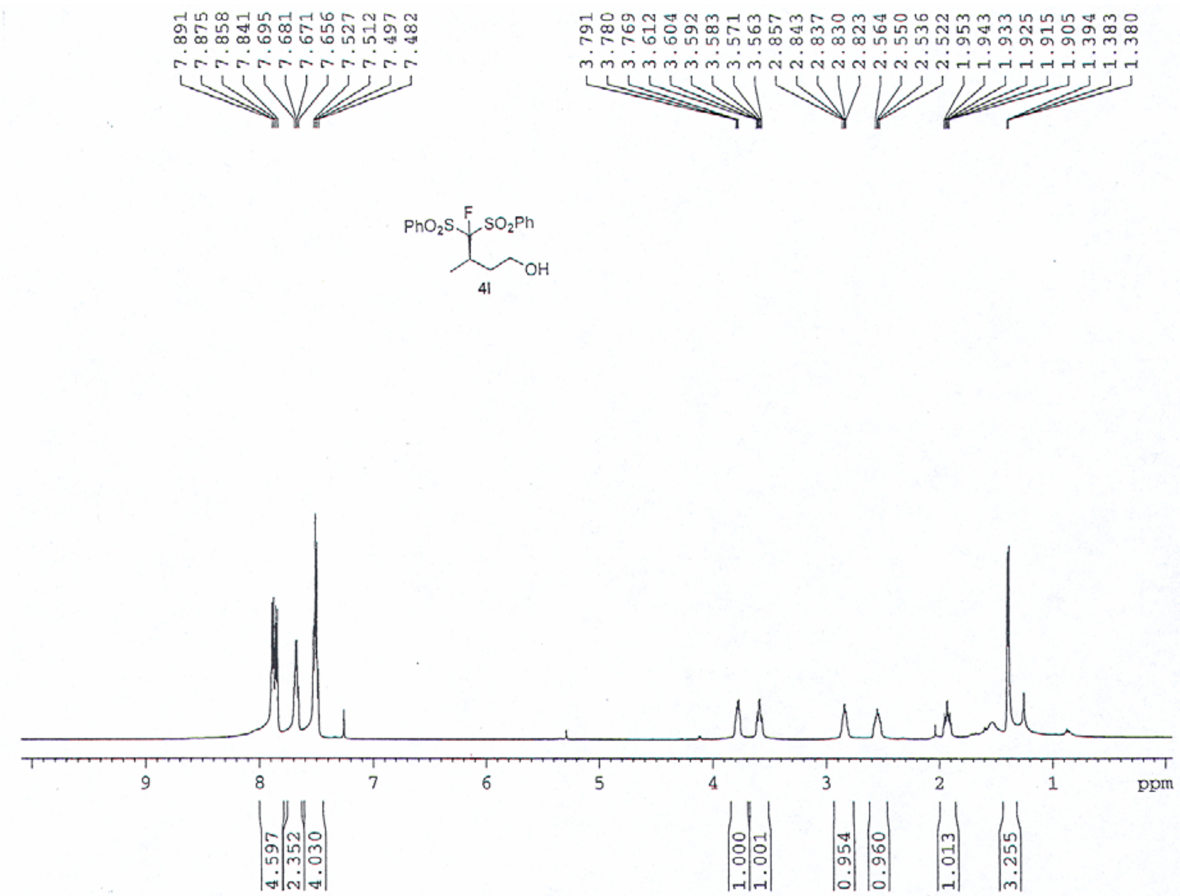
60
50
40
30
20
10
ppm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1.933
1.925
1.915
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1.394
1.383
1.380
PhO2S F SO2Ph
OH
4l
9
8
7
6
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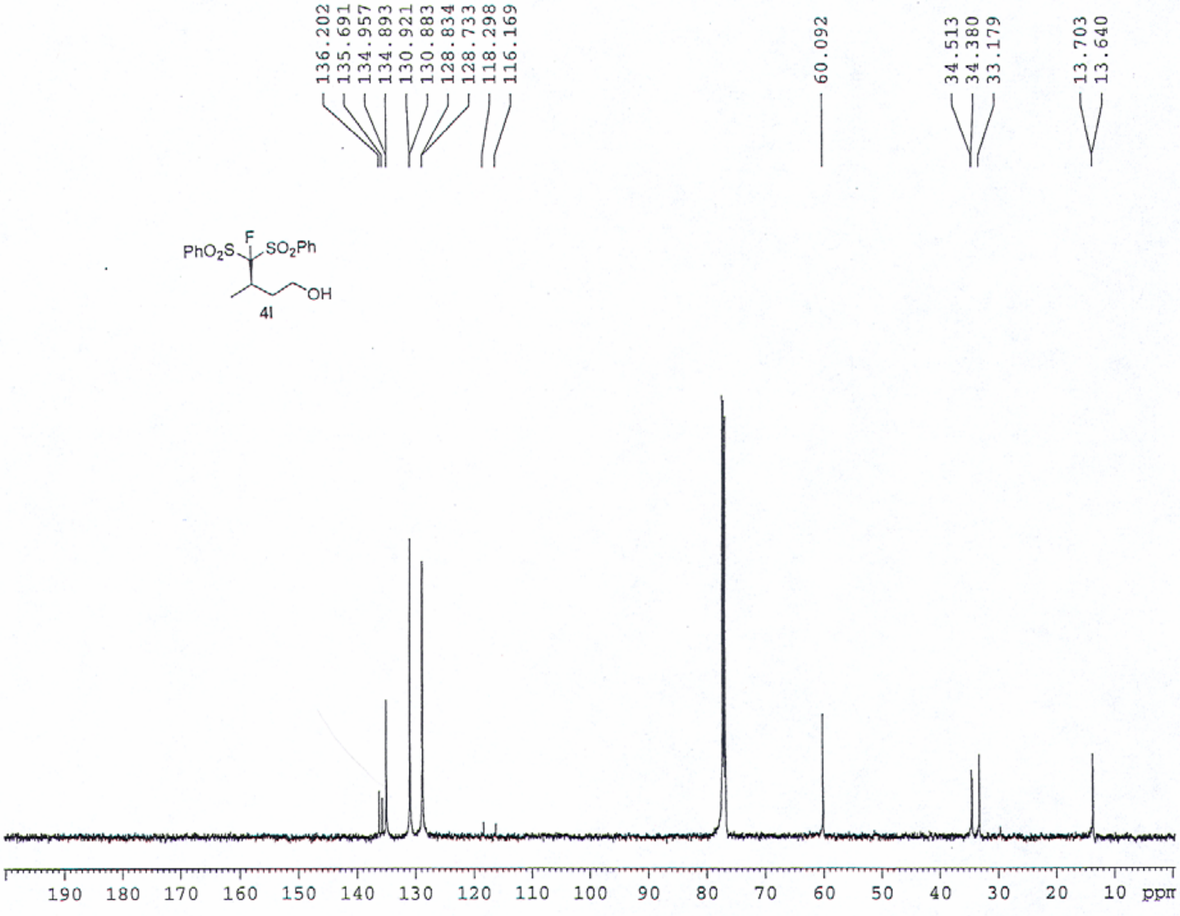
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33.179
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13.640
PhO2S F SO2Ph
OH
4l
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120
110
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80
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50
40
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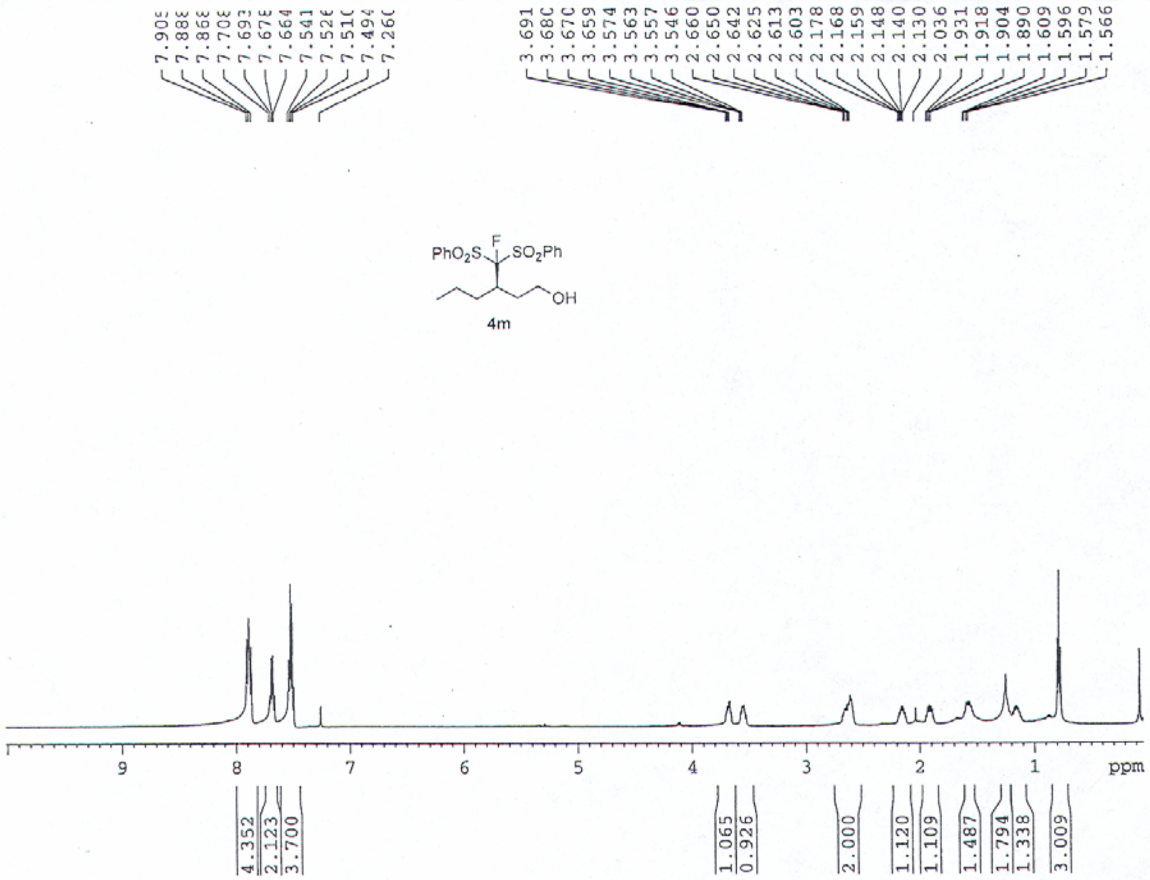
ppm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PhO2S
F
SO2Ph
OH
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8
7
6
5
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ppm
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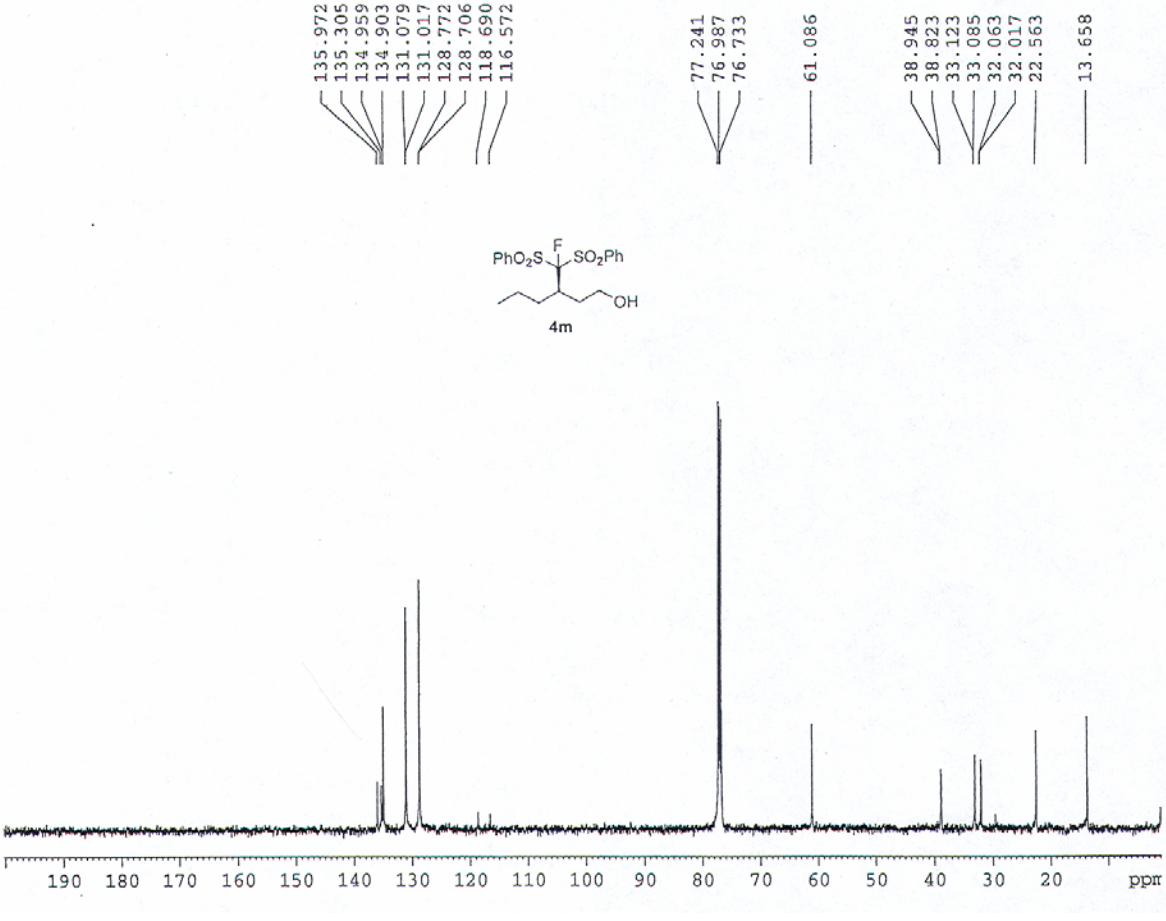
4m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76.733
61.086
38.945
38.823
33.123
33.085
32.063
32.017
22.563
13.658
PhO2S
F
SO2Ph
OH
190
180
170
160
150
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110
100
90
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20
ppm

4m

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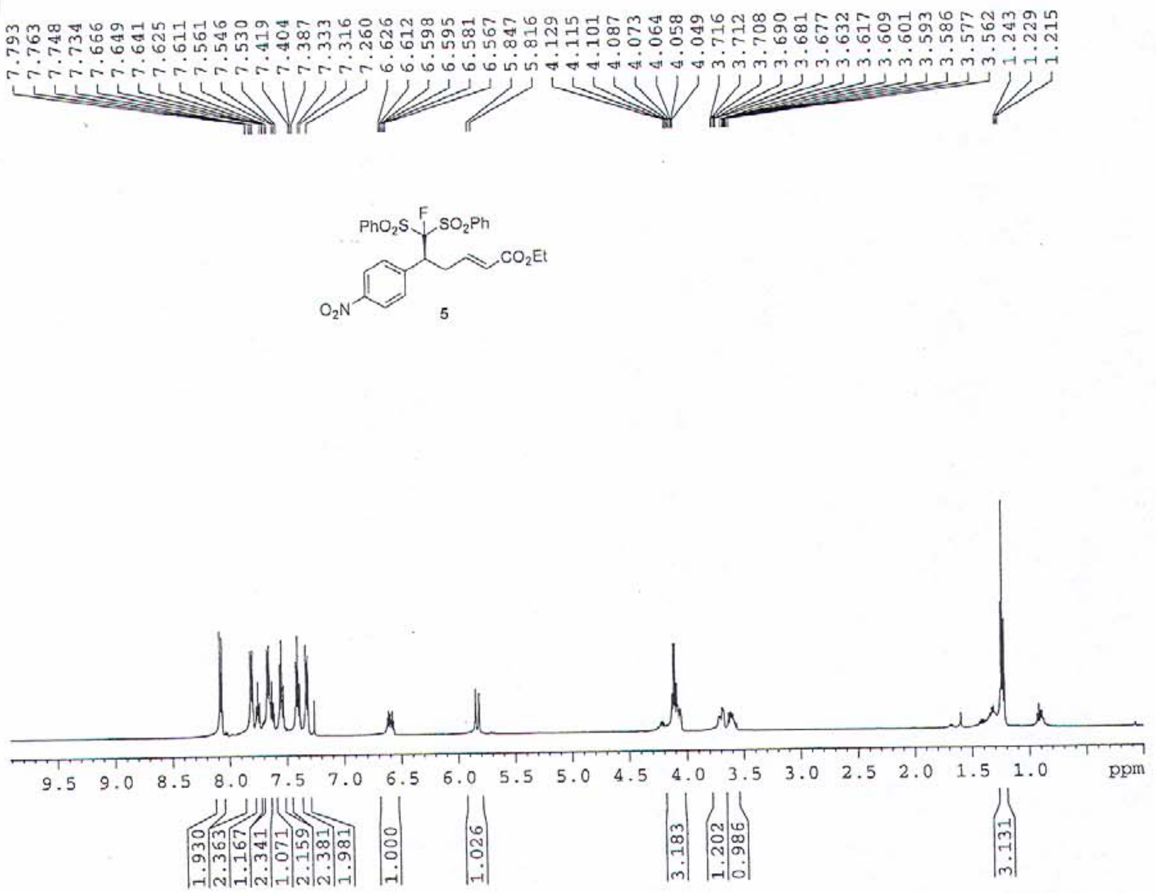

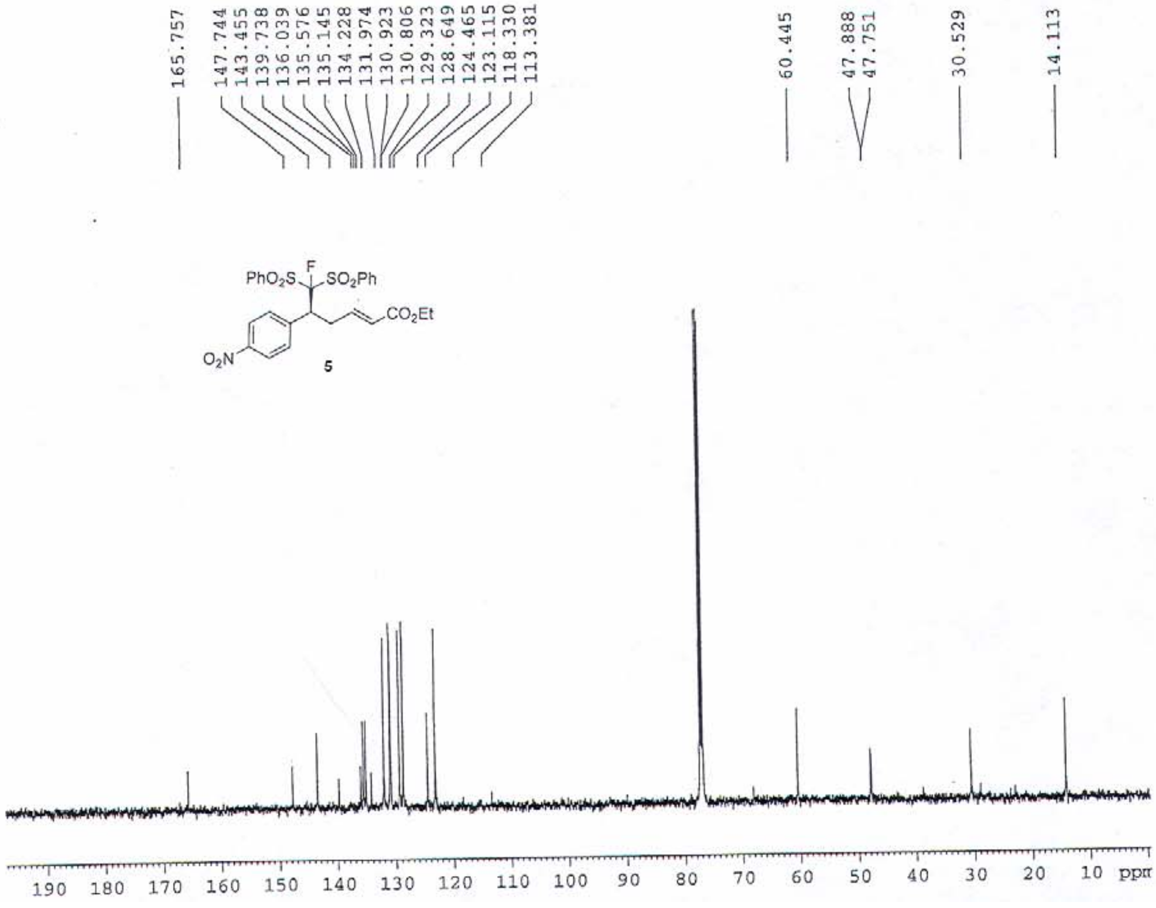

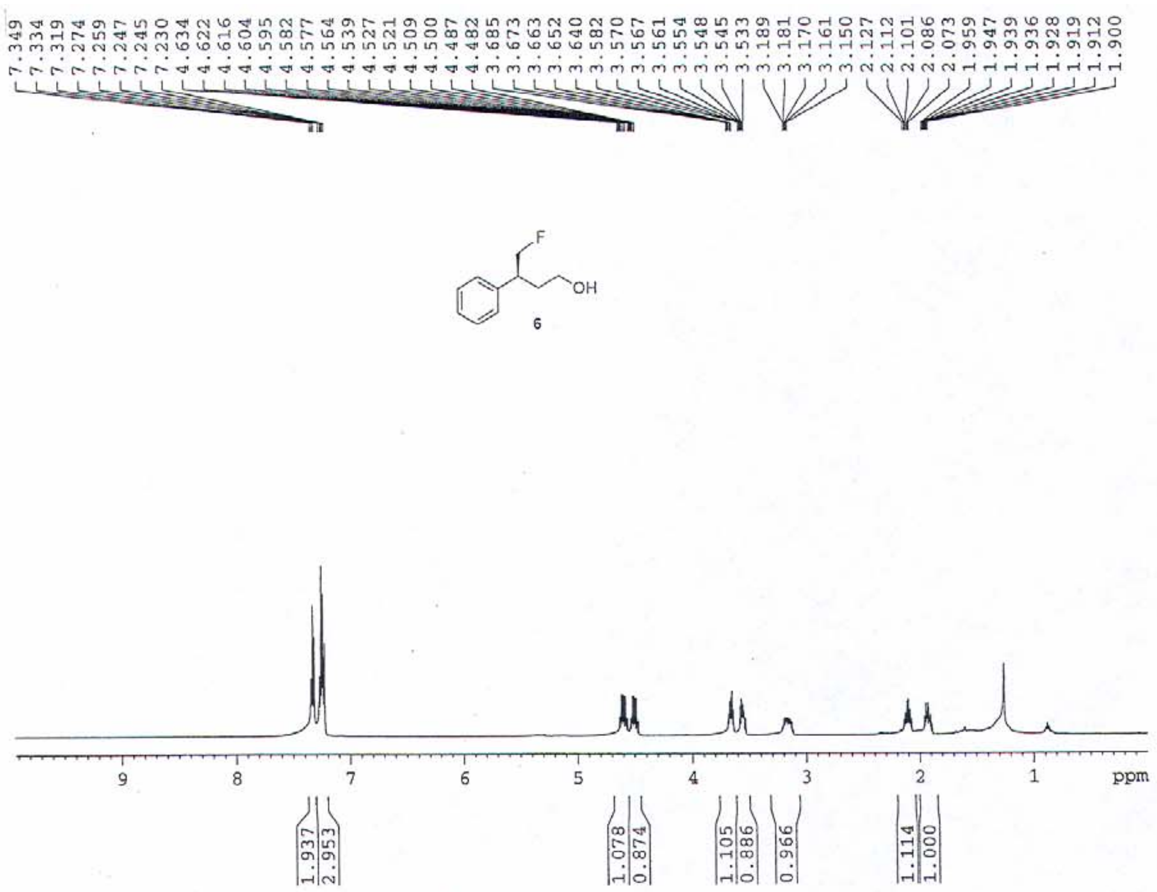
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1.936
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F
OH
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5
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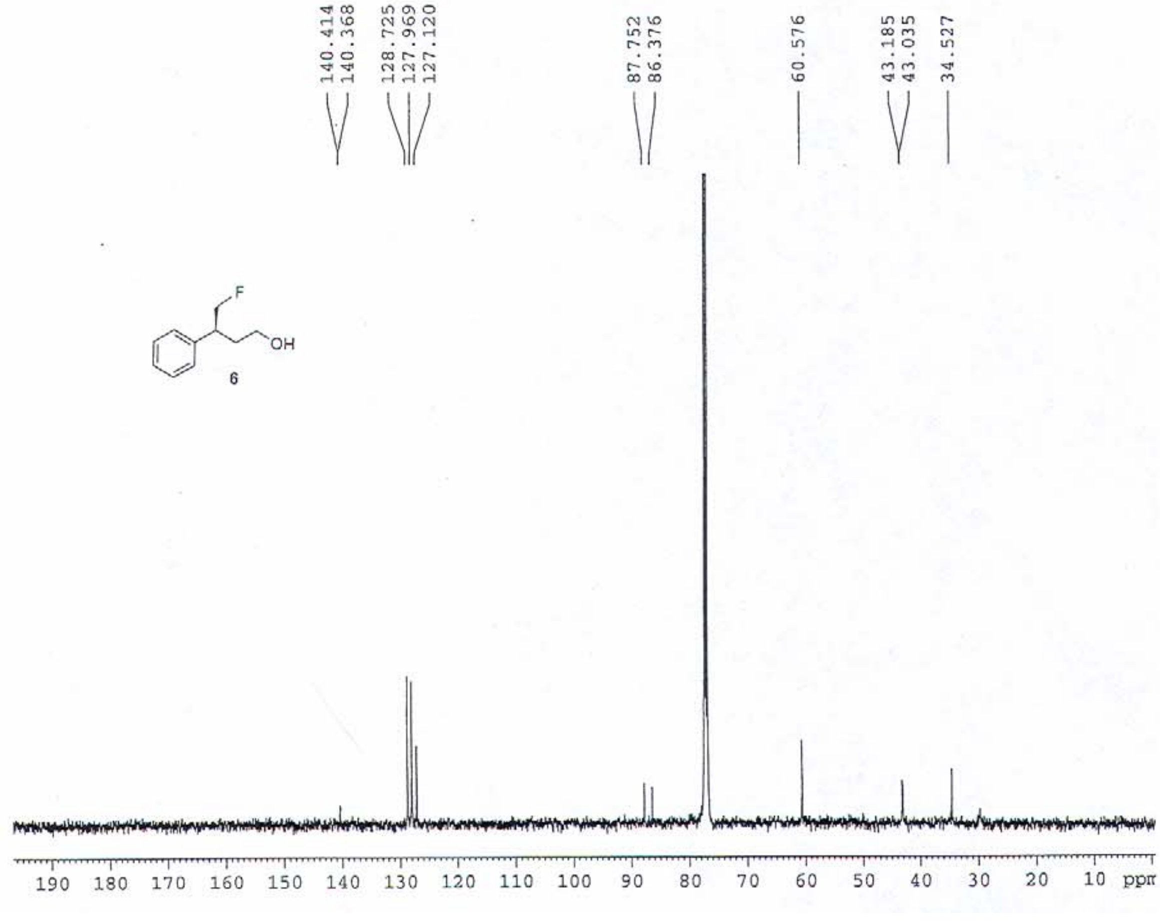
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34.527
F
OH
6
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

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Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-2-49-OD-H-1.dat
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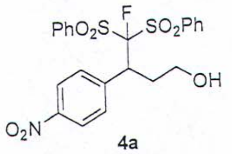
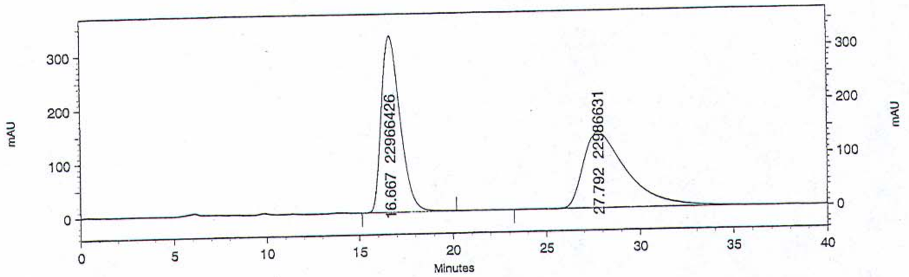


4a



SPD-20A
Ch1-254nm Results

Pk #	Retention Time	Area	Area Percent
1	16.667	22966426	49.978
2	27.792	22986631	50.022
Totals		45953057	100.000

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Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-2-61-OD-H-1.dat
Data Acquired: 12/24/2008 12:32:01 AM
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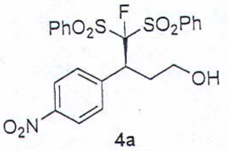
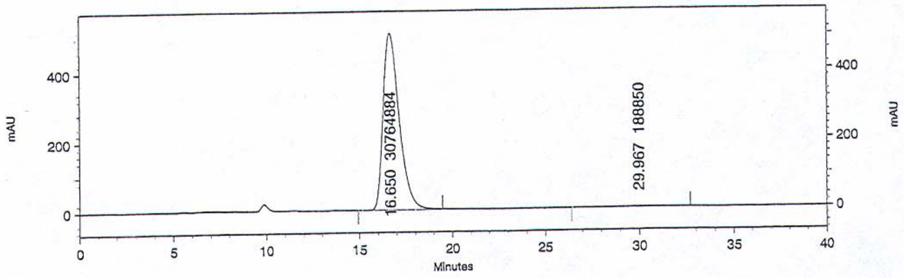
Sample ID: zsl-2-61



4a



SPD-20A
Ch1-254nm Results

Pk #	Retention Time	Area	Area Percent
1	16.650	30764884	99.390
2	29.967	188850	0.610
Totals		30953734	100.000

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Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
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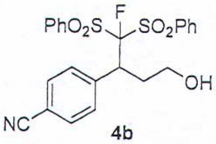
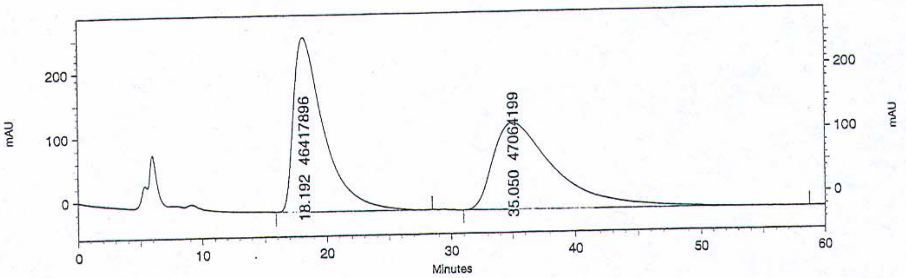


4b



SPD-20A
Ch1-210nm Results

Pk #	Retention Time	Area	Area Percent
1	18.192	46417896	49.654
2	35.050	47064199	50.346
Totals		93482095	100.000

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Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-1-69-AD-1.dat
Data Acquired: 11/24/2008 7:53:28 PM
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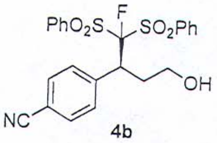
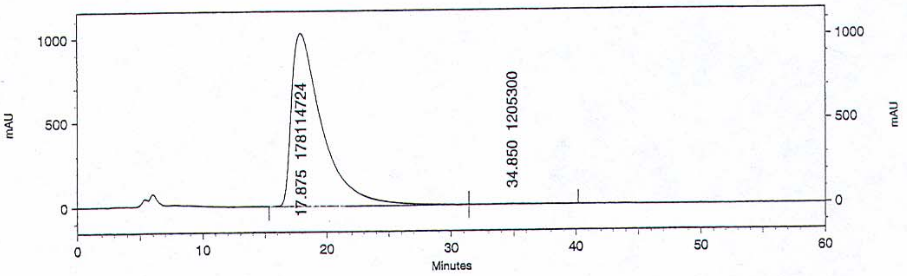
Sample ID: zsl-1-69



4b



SPD-20A
Ch1-210nm Results

Pk #	Retention Time	Area	Area Percent
1	17.875	178114724	99.328
2	34.850	1205300	0.672
Totals		179320024	100.000

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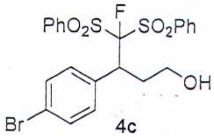
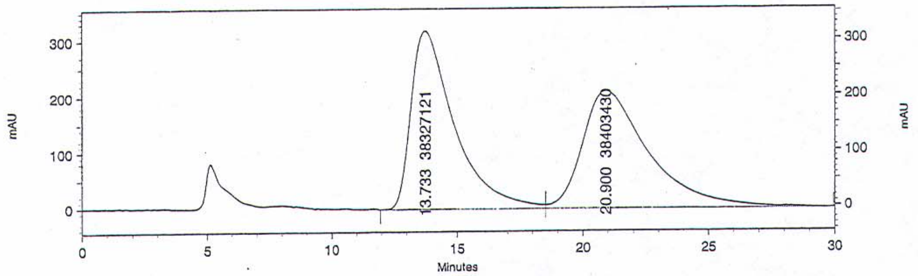
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-2-12-AD-1.dat
Data Acquired: 12/7/2008 5:01:49 PM
Date Printed: 12/17/2008 6:57:15 PM
Sample ID: zsl-2-12



4c



SPD-20A
Ch1-210nm Results

Pk #	Retention Time	Area	Area Percent
1	13.733	38327121	49.950
2	20.900	38403430	50.050

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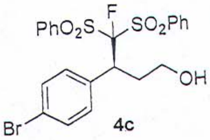
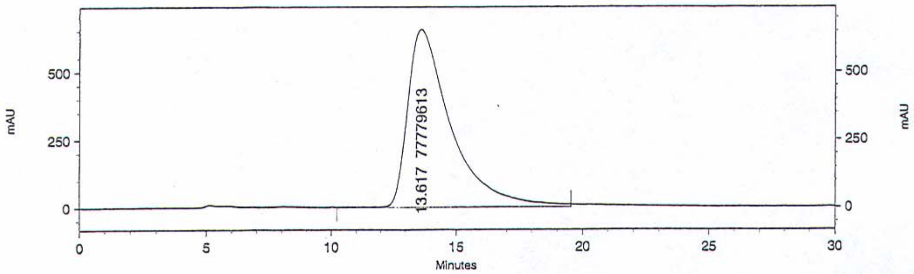
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-2-56-AD-2.dat
Data Acquired: 12/22/2008 11:00:44 AM
Date Printed: 12/22/2008 11:49:59 AM
Sample ID: zsl-2-56



4c



SPD-20A
Ch2-210nm Results

Pk #	Retention Time	Area	Area Percent
1	13.617	77779613	100.000
Totals		77779613	100.000

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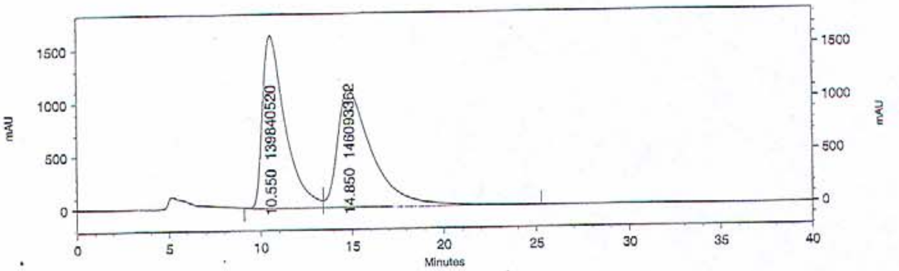
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-1-100-AD-1.dat
Data Acquired: 12/7/2008 2:53:49 PM
Date Printed: 12/22/2008 8:11:29 PM
Sample ID: zsl-1-100

4d



SPD-20A
Ch1-210nm Results

Pk #	Retention Time	Area	Area Percent
1	10.550	139840520	48.907
2	14.850	146093362	51.093
Totals		285933882	100.000

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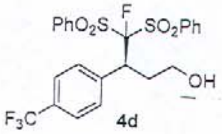
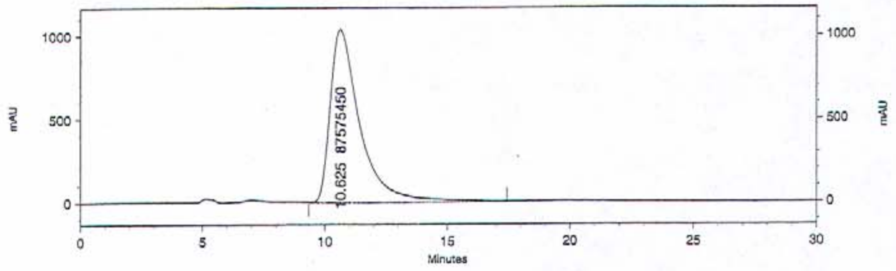
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-2-57-AD-1.dat
Data Acquired: 12/22/2008 7:41:21 PM
Date Printed: 12/22/2008 8:15:14 PM
Sample ID: zsl-2-57



4d



SPD-20A
Ch2-210nm Results

Pk #	Retention Time	Area	Area Percent
1	10.625	87575450	100.000
Totals		87575450	100.000

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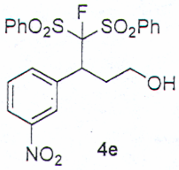
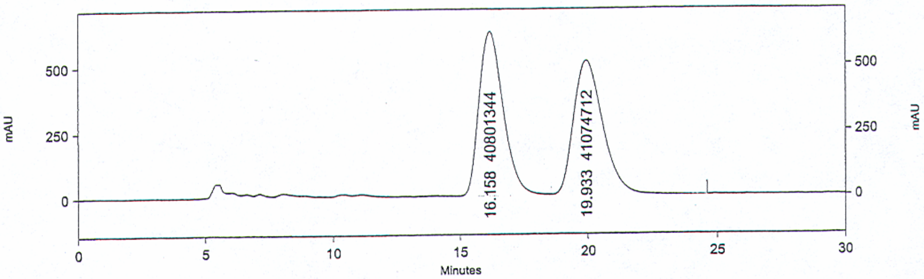
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-2-48-OD-H-3.dat
Data Acquired: 12/17/2008 12:49:51 PM
Date Printed: 12/31/2008 12:14:38 PM
Sample ID: zsl-2-48


4e



SPD-20A
Ch2-210nm Results

Pk #	Retention Time	Area	Area Percent
1	16.158	40801344	49.833
2	19.933	41074712	50.167
Totals		81876056	100.000

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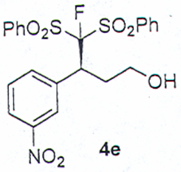
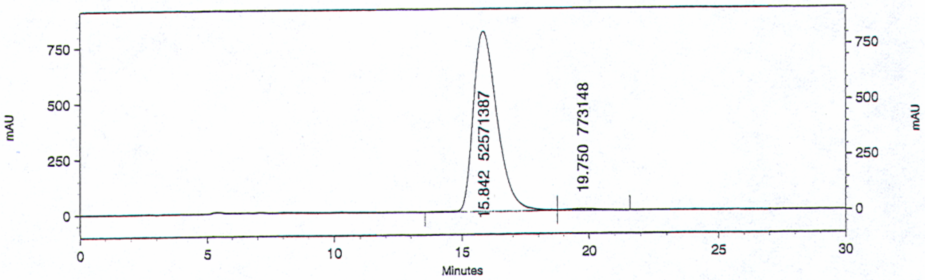
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-2-54-OD-H-4.dat
Data Acquired: 12/31/2008 11:29:08 AM
Date Printed: 12/31/2008 12:13:35 PM
Sample ID: zsl-2-54


4e



SPD-20A
Ch1-210nm Results

Pk #	Retention Time	Area	Area Percent
1	15.842	52571387	98.551
2	19.750	773148	1.449
Totals		53344535	100.000

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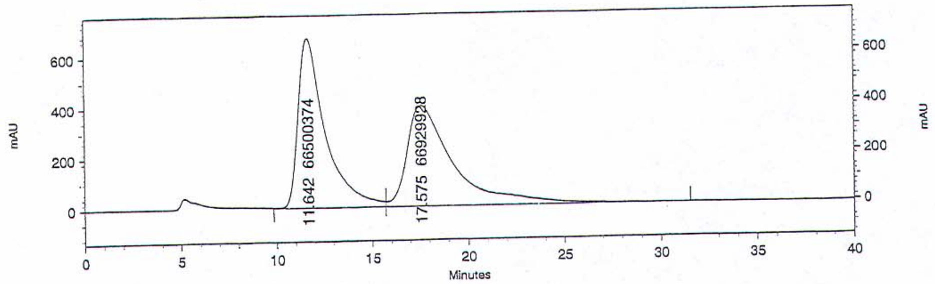
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-2-50-AD-3.dat
Data Acquired: 12/18/2008 12:08:12 PM
Date Printed: 12/29/2008 11:20:53 PM
Sample ID: zsl-2-50

4f



SPD-20A
Ch2-210nm Results

Pk #	Retention Time	Area	Area Percent
1	11.642	66500374	49.839
2	17.575	66929928	50.161
Totals		133430302	100.000

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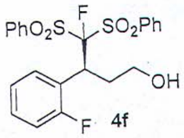
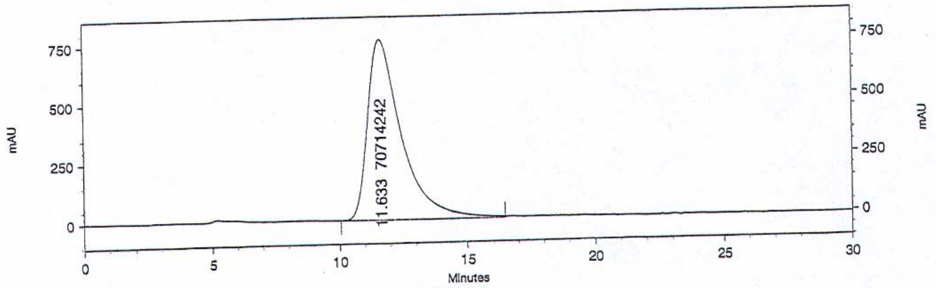
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-2-75-AD-1.dat
Data Acquired: 12/29/2008 10:36:59 PM
Date Printed: 12/29/2008 11:20:47 PM
Sample ID: zsl-2-75



4f



SPD-20A
Ch2-210nm Results

Pk #	Retention Time	Area	Area Percent
1	11.633	70714242	100.000
Totals		70714242	100.000

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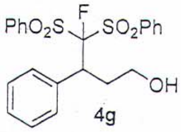
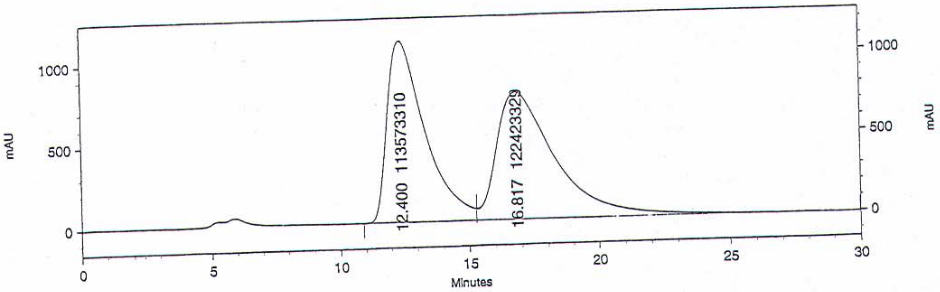
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-1-61-AD-2.dat
Data Acquired: 11/23/2008 5:33:32 PM
Date Printed: 12/15/2008 5:28:29 PM
Sample ID: zsl-1-61



4g



SPD-20A
Ch1-210nm Results

Pk #	Retention Time	Area	Area Percent
1	12.400	113573310	48.125
2	16.817	122423329	51.875
Totals		235996639	100.000

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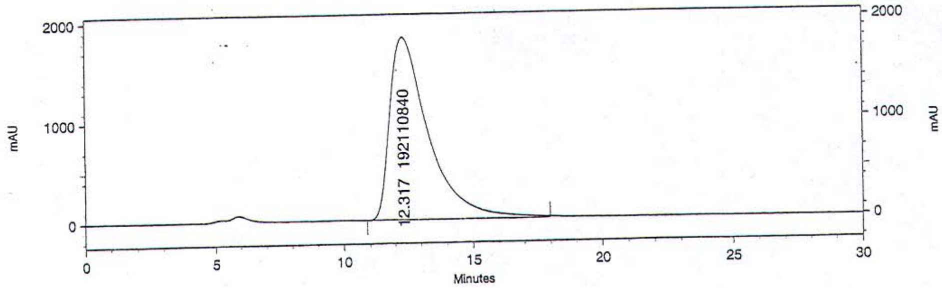
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-1-62-AD-1.dat
Data Acquired: 11/23/2008 7:07:55 PM
Date Printed: 12/15/2008 5:29:30 PM
Sample ID: zsl-1-62

4g



SPD-20A
Ch1-210nm Results

Pk #	Retention Time	Area	Area Percent
1	12.317	192110840	100.000
Totals		192110840	100.000

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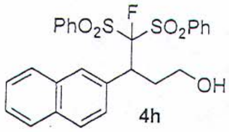
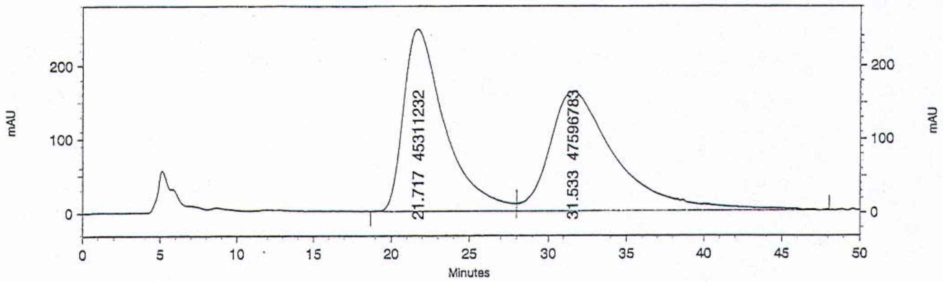
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-1-97-AD-2.dat
Data Acquired: 12/3/2008 9:02:42 PM
Date Printed: 12/23/2008 6:45:30 PM
Sample ID: zsl-1-97



4h



SPD-20A
Ch1-210nm Results

Pk #	Retention Time	Area	Area Percent
1	21.717	45311232	48.770
2	31.533	47596783	51.230
Totals		92908015	100.000

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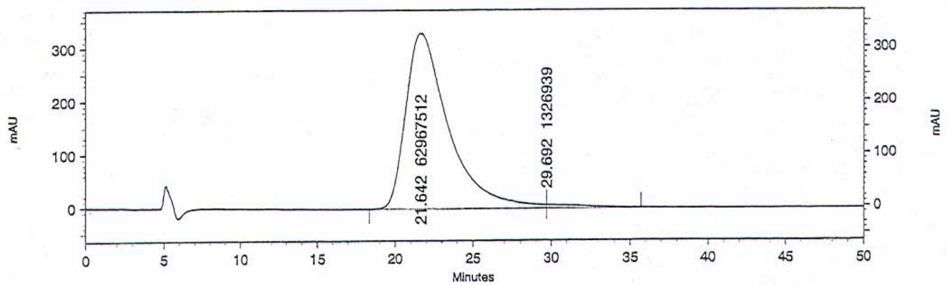
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-2-60-AD-1.dat
Data Acquired: 12/23/2008 5:49:36 PM
Date Printed: 12/23/2008 6:42:21 PM
Sample ID: zsl-2-60

4h



SPD-20A
Ch1-210nm Results

Pk #	Retention Time	Area	Area Percent
1	21.642	62967512	97.936
2	29.692	1326939	2.064
Totals		64294451	100.000

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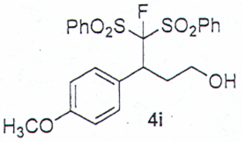
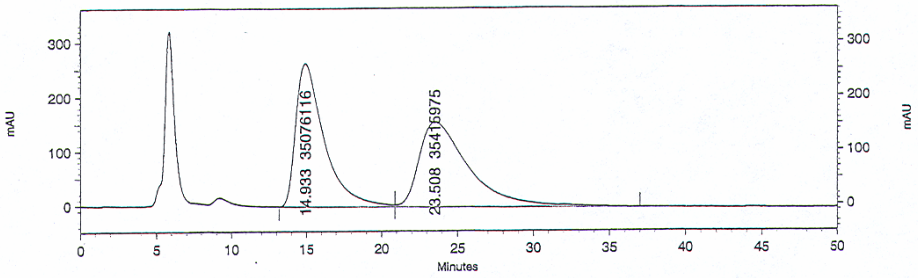
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-1-75-AD-1.dat
Data Acquired: 11/26/2008 7:55:14 PM
Date Printed: 12/15/2008 5:23:09 PM
Sample ID: zsl-1-75



4i



SPD-20A
Ch1-210nm Results

Pk #	Retention Time	Area	Area Percent
1	14.933	35076116	49.759
2	23.508	35415975	50.241
Totals		70492091	100.000

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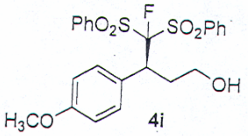
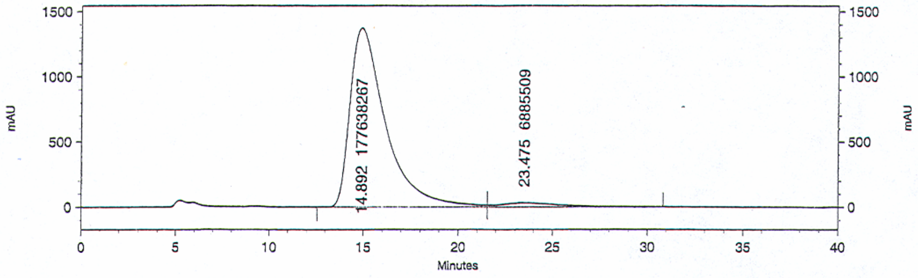
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-1-76-AD-2.dat
Data Acquired: 11/27/2008 10:11:54 AM
Date Printed: 12/15/2008 5:24:20 PM
Sample ID: zsl-1-76



4i



SPD-20A
Ch1-210nm Results

Pk #	Retention Time	Area	Area Percent
1	14.892	177638267	96.269
2	23.475	6885509	3.732
Totals		184523776	100.000

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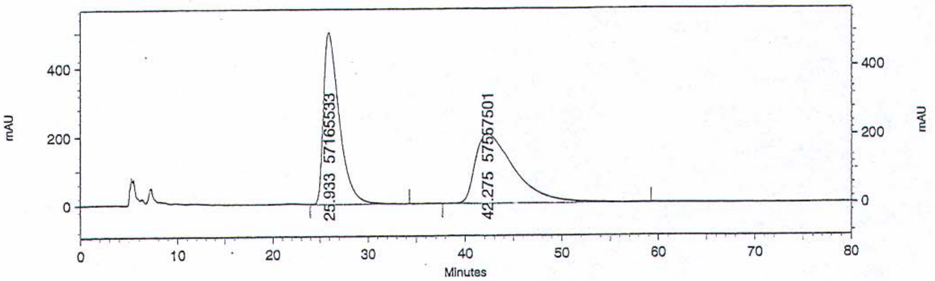
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-1-96-AS-H-5.dat
Data Acquired: 12/21/2008 10:02:21 PM
Date Printed: 12/21/2008 11:34:11 PM
Sample ID: zsl-1-96

4j



SPD-20A
Ch2-210nm Results

Pk #	Retention Time	Area	Area Percent
1	25.933	57165533	49.829
2	42.275	57557501	50.171
Totals		114723034	100.000

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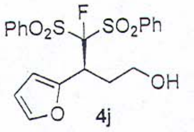
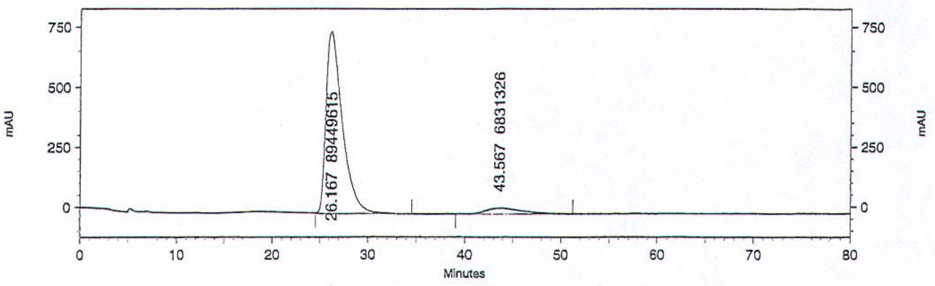
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-2-55-AS-H-1.dat
Data Acquired: 12/21/2008 8:38:04 PM
Date Printed: 12/21/2008 10:36:47 PM
Sample ID: zsl-2-55



4j



SPD-20A
Ch2-210nm Results

Pk #	Retention Time	Area	Area Percent
1	26.167	89449615	92.905
2	43.567	6831326	7.095
Totals		96280941	100.000

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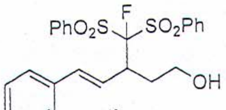
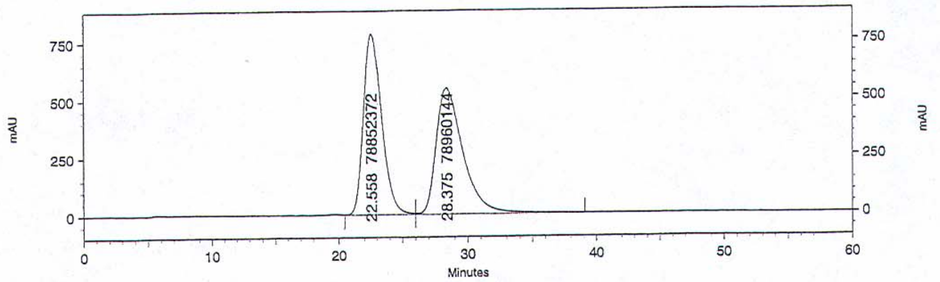
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-2-69-AS-H-1.dat
Data Acquired: 12/25/2008 10:51:38 PM
Date Printed: 12/28/2008 8:21:16 PM
Sample ID: zsl-2-69



4k



SPD-20A
Ch2-254nm Results

Pk #	Retention Time	Area	Area Percent
1	22.558	78852372	49.966
2	28.375	78960144	50.034
Totals		157812516	100.000

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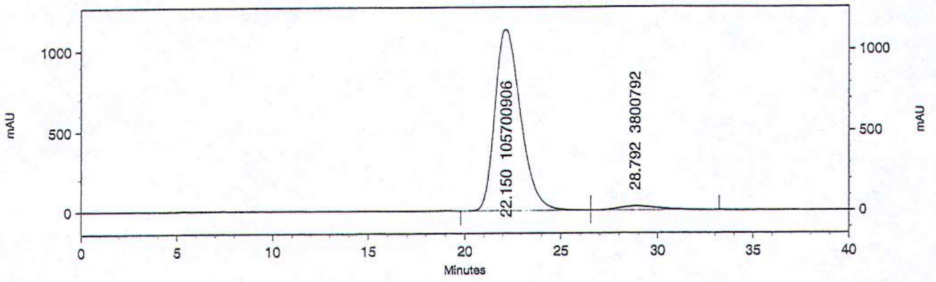
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-2-73-AS-H-1.dat
Data Acquired: 12/28/2008 7:44:33 PM
Date Printed: 12/28/2008 8:28:22 PM
Sample ID: zsl-2-73

4k



SPD-20A
Ch2-254nm Results

Pk #	Retention Time	Area	Area Percent
1	22.150	105700906	96.529
2	28.792	3800792	3.471
Totals		109501698	100.000

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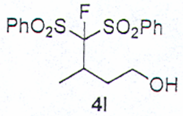
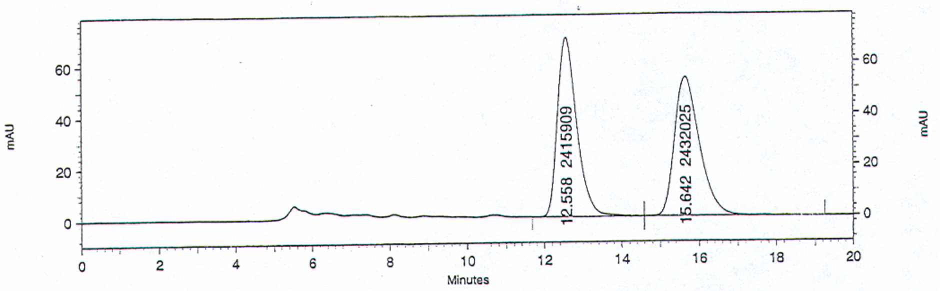
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-2-28-OD-H-3.dat
Data Acquired: 12/11/2008 8:33:46 PM
Date Printed: 12/15/2008 3:23:51 PM
Sample ID: zsl-2-28



4l



SPD-20A
Ch2-254nm Results

Pk #	Retention Time	Area	Area Percent
1	12.558	2415909	49.834
2	15.642	2432025	50.166
Totals		4847934	100.000

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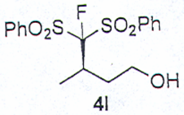
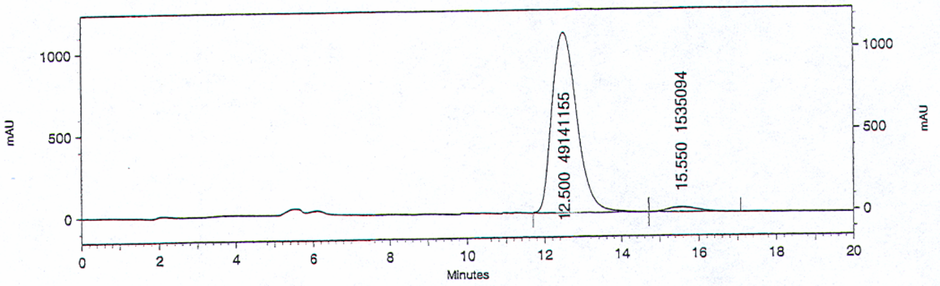
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-2-37-OD-H-1.dat
Data Acquired: 12/15/2008 10:17:08 AM
Date Printed: 12/15/2008 3:09:04 PM
Sample ID: zsl-2-37



4l



SPD-20A
Ch2-210nm Results

Pk #	Retention Time	Area	Area Percent
1	12.500	49141155	96.971
2	15.550	1535094	3.029
Totals		50676249	100.000

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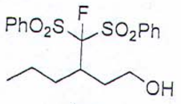
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-2-23-OD-H-1.dat
Data Acquired: 12/11/2008 11:00:53 PM
Date Printed: 12/23/2008 9:39:33 PM
Sample ID: zsl-2-23



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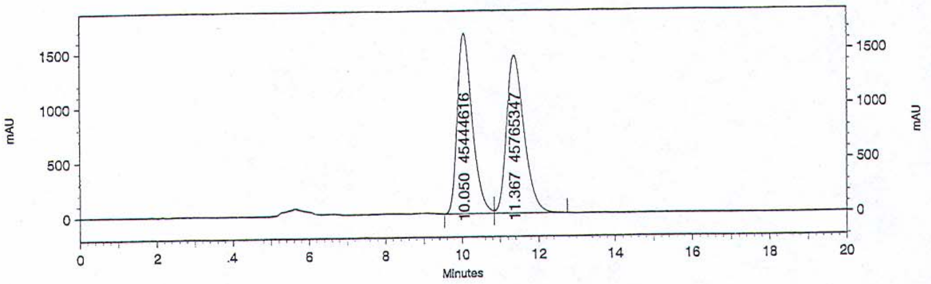

SPD-20A
Ch1-210nm Results

Pk #	Retention Time	Area	Area Percent
1	10.050	45444616	49.824
2	11.367	45765347	50.176
Totals		91209963	100.000

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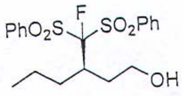
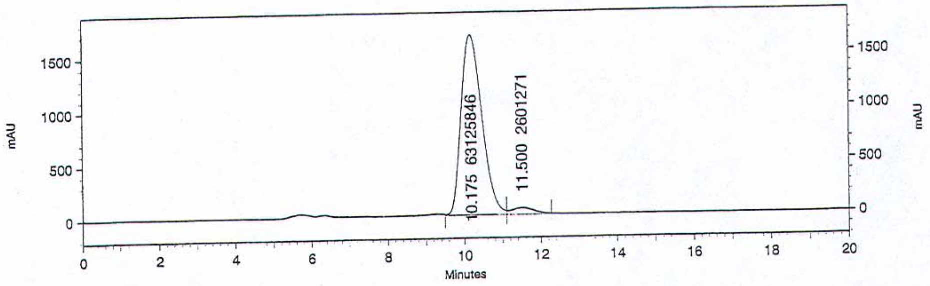
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-2-58-~~AD~~-1.dat
OD-H
Data Acquired: 12/23/2008 9:16:15 PM
Date Printed: 12/23/2008 9:40:22 PM
Sample ID: zsl-2-58



4m



SPD-20A
Ch1-210nm Results

Pk #	Retention Time	Area	Area Percent
1	10.175	63125846	96.042
2	11.500	2601271	3.958
Totals		65727117	100.000

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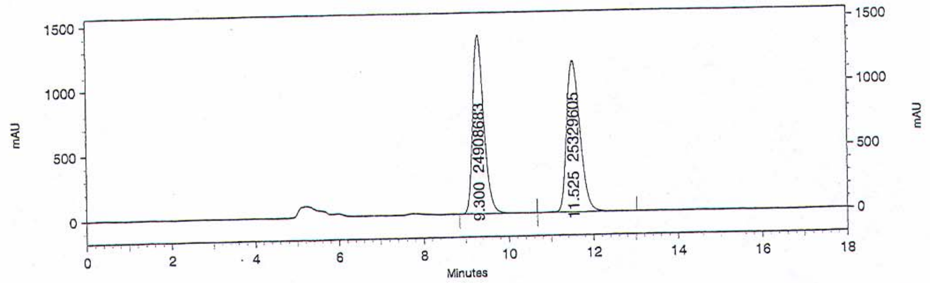
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-2-74-AS-H-2.dat
Data Acquired: 12/28/2008 10:54:18 AM
Date Printed: 12/28/2008 11:19:49 AM
Sample ID: zsl-2-74

6



SPD-20A
Ch1-210nm Results

Pk #	Retention Time	Area	Area Percent
1	9.300	24908683	49.581
2	11.525	25329605	50.419
Totals		50238288	100.000

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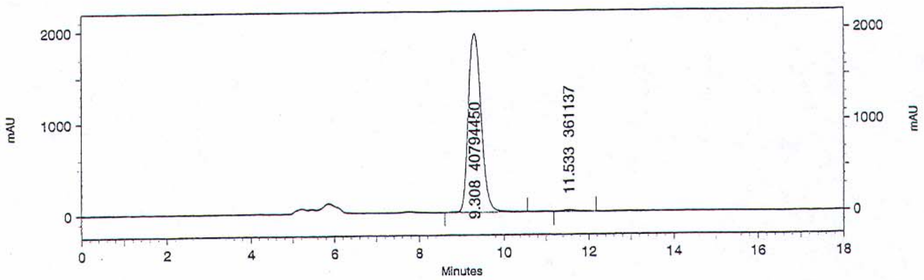
Method Name: C:\EZStart\Projects\Default\Method\zls1869.met
Data File: C:\EZStart\Projects\Default\Data\zsl-2-72-AS-H-1.dat
Data Acquired: 12/28/2008 11:17:00 AM
Date Printed: 12/28/2008 11:39:14 AM
Sample ID: zsl-2-72

6



SPD-20A
Ch1-210nm Results

Pk #	Retention Time	Area	Area Percent
1	9.308	40794450	99.123
2	11.533	361137	0.877
Totals		41155587	100.000