Supplementary Material (ESI) for Chemical Communications

Supporting Information

Asymmetric Synthesis of 2-Substituted Pyrrolidines by Addition of Grignard Reagents to γ-Chlorinated *N*-(*tert*-Butanesulfinyl) Imine

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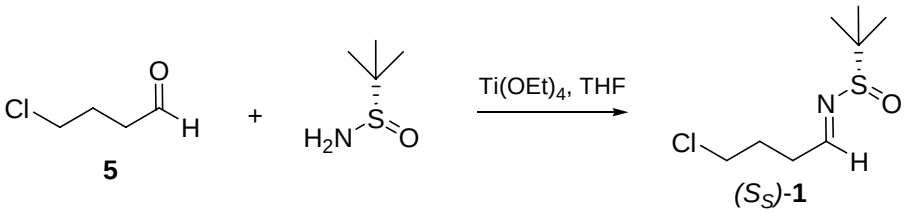
Experimental Section

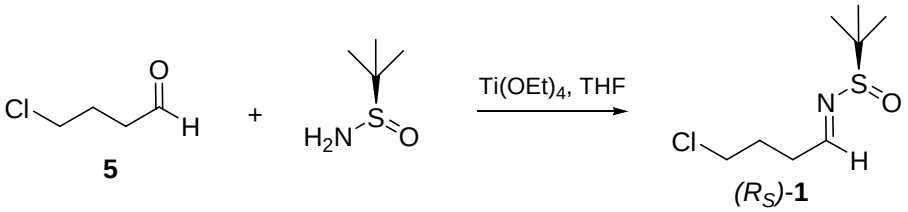
General Information: All the reactions were performed under dry nitrogen gas in glasswares that was flame-dried and equipped with a magnetic stirring bar. Tetrahydrofuran (THF) was freshly distilled from the sodium complex of benzophenone before use. Thin-layer chromatography (TLC) was performed using E. Merck silica gel 60 F254 pre-coated plates (0.25 mm). Flash chromatography was performed using Baker silica gel (40 μm particle size). All compounds were judged pure by TLC analysis (single spot/ two solvent systems) using a UV lamp or PMA for detection purposes. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker FT-NMR spectrometer at 500 and 125 MHz, respectively. High-resolution mass spectroscopy (HRMS) was carried out on a ThermoFinnigan MAT-900 in electrospray mode. Other mass spectra were obtained on a Micromass LCT in electrospray mode. Melting points were measured on a Büchi 533 melting point apparatus. All the reagents were purchased from commercial suppliers and used without further purification.

General procedure (GP1) for the synthesis of γ-Chloro *N*-sulfinyl aldimine 1: A 500 mL, three-necked, round-bottomed flask was charged with 4-chlorobutanal (**5**) (100.0 mmol), THF (100 mL), *tert*-butanesulfinamide (100.0 mmol), and $Ti(OEt)_4$ (110.0 mmol) under nitrogen atm. The reaction mixture was stirred at rt for 16 h. After completion of the reaction, charged the saturated NaCl solution (100 mL), ethyl acetate (100 mL) and

stirred for 1 h at rt. The solids were removed by filtration and filtrate was washed with water (2 × 50 mL). The organic phase was evaporated under vacuum to dryness to obtained crude product. The crude product was purified by flash column chromatography (silica gel, 10% ethyl acetate in heptanes) affords the pure γ-Chloro *N*-sulfinyl aldimine **1.**

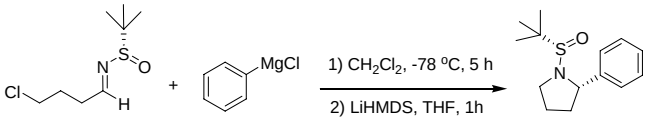


γ-chloro *N*-sulfinyl aldimine (S_S) 1 : Following the general procedure (GP1), the reaction of 4-chlorobutanal (**5**) (10.6 g, 100.0 mmol) with (S_S)-*tert*-butanesulfinamide (12.1 g, 100.0 mmol) and $Ti(OEt)_4$ (25.0 g, 110.0 mmol) gave 20.4 g (98%) of pure γ-Chloro *N*-sulfinyl aldimine (S_S) **1** as viscous oil. $[\alpha]^{25}_D = +231.1°$. (c 1.13, $CHCl_3$).1H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.20 (s, 9 H) 2.07 - 2.20 (m, 2 H) 2.65 - 2.78 (m, 2 H) 3.56 - 3.68 (m, 2 H) 8.11 (t, *J*=3.94 Hz, 1 H). ^{13}C NMR (125 MHz, CHLOROFORM-*d*) δ 22.33, 27.99, 33.13, 43.97, 56.64, 167.87. HRMS (EI) Calcd for $C_8H_{17}ClNOS$ [M+H]: 210.0719, Found 210.0700.

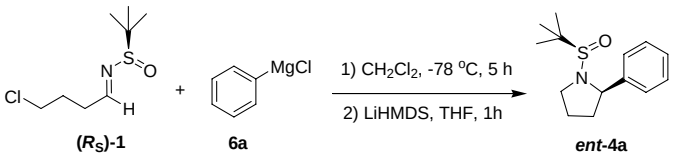


γ-chloro *N*-sulfinyl aldimine (R_S) 1 : Following the general procedure (GP1), the reaction of 4-chlorobutanal (**5**) (1.06 g, 10.0 mmol) with (R_S)-*tert*-butanesulfinamide (1.2 g, 10.0 mmol) and $Ti(OEt)_4$ (2.5 g, 11.0 mmol) gave 1.98 g (95%) of pure γ-Chloro *N*-sulfinyl aldimine (R_S) **1** as viscous oil. $[\alpha]^{25}_D = +229.2°$. (c 1.01, $CHCl_3$). ^{1}H NMR (500 MHz, CHLOROFORM-*d*) δ ppm 1.20 (s, 9 H) 2.14 (dd, *J*=6.46, 4.26 Hz, 2 H) 2.66 - 2.75 (m, 2 H) 3.58 - 3.69 (m, 2 H) 8.11 (t, *J*=3.94 Hz, 1 H). ^{13}C NMR (125 MHz, CHLOROFORM-*d*) δ 22.34, 28.00, 33.13, 43.97, 56.65, 167.

General Procedure (GP2) for the Synthesis 2-substituted pyrrolidines: To a solution of aldimine **1** (5 mmol) in CH_2Cl_2 (25 mL) at –78 °C under nitrogen was added the Grignard reagents **6** (6 mmol). After stirring for 5 h at –78 °C reaction mixture was quenched with saturated NH_4Cl (20 mL) then separated the organic layer and washed with water. The organic layer was removed in vacuum to give crude product. The crude product was dissolved in THF (20 mL) at 23 °C under nitrogen was added the LiHMDS (7.5 mmol). After stirring for 1 h at 23 °C reaction mixture was quenched with saturated NH_4Cl (20 mL) then extracted with ethyl acetate (2 x 25 mL). The combined organic layers were washed with water (20 mL) and solvent was removed in vacuum to give crude product. The crude product were purified by column chromatography (silica gel, ethyl acetate/hexanes) affords the pure 2-substituted pyrrolidines **4**.

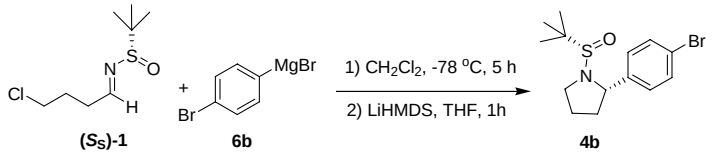


(*S*)-1-((*S*)-2-Methyl-propane-2-sulfinyl)-2-phenyl-pyrrolidine (4a) : Following the general procedure (GP2), the reaction of γ-chloro *N*-sulfinyl aldimine (S_S)-1 (1.05 g, 5.0 mmol) with phenylmagnisum chloride (3.0 mL, 6.0 mmol) and LiHMDS (7.5 mL, 7.5 mmol) afforded pyrrolidine **4a** (1.14 g, 91%) as a white solid, mp = 80-81 °C, $[\alpha]^{25}_D = -140.4°$. (c 1.07, $CHCl_3$). ^{1}H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.05 (s, 9 H) 1.66 - 1.95 (m, 3 H) 2.16 (dd, *J*=11.35, 8.51 Hz, 1 H) 3.45 - 3.61 (m, 1 H) 3.61 - 3.73 (m, 1 H) 5.07 (dd, *J*=8.20, 2.84 Hz, 1 H) 7.16 - 7.39 (m, 5 H). ^{13}C NMR (125 MHz, CHLOROFORM-*d*) δ 23.11, 24.22, 36.64, 54.92, 57.47, 126.53, 126.36, 144.67. HRMS (EI) Calcd for $C_{14}H_{22}NOS$ [M+H]: 252.1402, Found 252.1413.



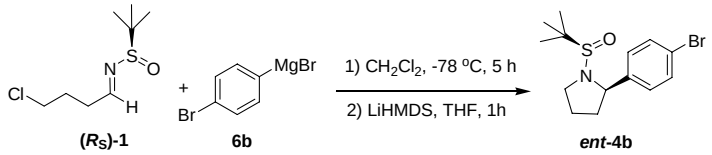
(*R*)-1-((*R*)-2-Methyl-propane-2-sulfinyl)-2-phenyl-pyrrolidine (*ent*-4a) : Following the general procedure (GP2), the reaction of γ-chloro *N*-sulfinyl aldimine (R_S)-1 (1.05 g, 5.0 mmol) with phenylmagnisum chloride (3.0 mL, 6.0 mmol) and LiHMDS (7.5 mL, 7.5 mmol) afforded pyrrolidine *ent*-**4a** (1.12 g, 90%) as a white solid, mp = 80-82 °C, $[\alpha]^{25}_D$

= + 141.2°. (c 1.10, $CHCl_3$). ^{1}H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.05 (s) 1.82 (m) 2.16 (dd, *J*=11.35, 8.51 Hz) 3.56 (m) 3.66 (m) 5.07 (dd, *J*=8.04, 2.68 Hz) 7.26 (m).). ^{13}C NMR (125 MHz, CHLOROFORM-*d*) δ 23.09, 24.21, 36.64, 54.90, 57.46, 126.52, 126.36, 144.66. HRMS (EI) Calcd for $C_{14}H_{22}NOS$ [M+H]: 252.1402, Found 252.1422.



(*S*)-1-((*S*)-2-Methyl-propane-2-sulfinyl)-2-(4-bromophenyl)-pyrrolidine (4b):

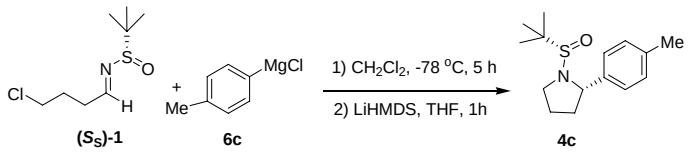
Following the general procedure (GP2), the reaction of γ-chloro *N*-sulfinyl aldimine (S_S)-1 (1.05 g, 5.0 mmol) with 4-bromophenylmagnisum bromide (6.0 mL, 6.0 mmol) and LiHMDS (7.5 mL, 7.5 mmol) afforded pyrrolidine **4b** (1.35 g, 83%) as white solid, mp = 100-112 °C, $[\alpha]^{25}_D = -164.3°$ (c 1.13, $CHCl_3$). ^{1}H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.05 (s, 9 H) 1.60 - 1.93 (m, 3 H) 2.16 (dd, *J*=11.98, 9.14 Hz, 1 H) 3.47 - 3.58 (m, 1 H) 3.61 - 3.70 (m, 1 H) 5.02 (dd, *J*=8.20, 2.84 Hz, 1 H) 7.14 (d, *J*=8.51 Hz, 2 H) 7.37 - 7.49 (m, 2 H). ^{13}C NMR (125 MHz, CHLOROFORM-*d*) δ 23.06, 24.19, 36.49, 54.86, 56.96, 57.48, 120.36, 128.28, 131.48, 143.78. HRMS (EI) Calcd for $C_{14}H_{21}BrNOS$ [M+H]: 330.0527, Found 330.0539.



(*R*)-1-((*R*)-2-Methyl-propane-2-sulfinyl)-2-(4-bromophenyl)-pyrrolidine (*ent*-4b):

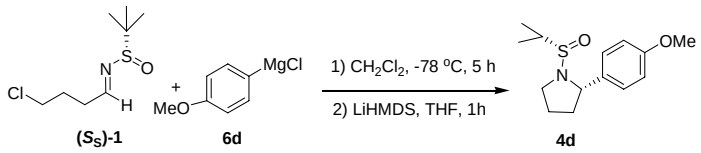
Following the general procedure (GP2), the reaction of γ-chloro *N*-sulfinyl aldimine (R_S)-1 (1.05 g, 5.0 mmol) with 4-bromophenylmagnisum bromide (6.0 mL, 6.0 mmol) and LiHMDS (7.5 mL, 7.5 mmol) afforded pyrrolidine *ent*-**4b** (1.39 g, 85%) as white solid, mp = 110-111 °C, $[\alpha]^{25}_D$ = + 163.1° (c 1.02, $CHCl_3$). ^{1}H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.05 (s, 9 H) 1.66 - 1.94 (m, 3 H) 2.08 - 2.21 (m, 1 H) 3.51 - 3.58 (m, 1 H) 3.60 - 3.68 (m, 1 H) 5.02 (dd, *J*=8.20, 2.84 Hz, 1 H) 7.13 (d, *J*=8.20 Hz, 2 H) 7.43 (d, *J*=8.20 Hz, 2 H). ^{13}C NMR (125 MHz, CHLOROFORM-*d*) δ 23.07, 24.19,

36.50, 54.89, 56.96, 57.50, 120.36, 128.29, 131.49, 143.81. HRMS (EI) Calcd for $C_{14}H_{21}BrNOS$ [M+H]: 330.0527, Found 330.0529.



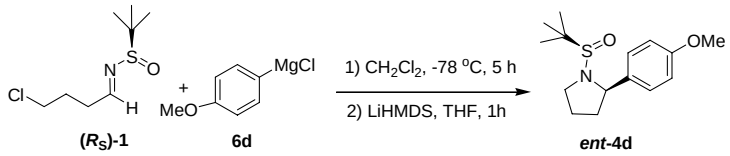
(*S*)-1-((*S*)-2-Methyl-propane-2-sulfinyl)-2-(4-methylphenyl)-pyrrolidine (4c):

Following the general procedure (GP2), the reaction of γ-chloro *N*-sulfinyl aldimine (S_S)-1 (1.05 g, 5.0 mmol) with 4-methylphenylmagnisum chloride (2.0 mL, 6.0 mmol) and LiHMDS (7.5 mL, 7.5 mmol) afforded pyrrolidine **4c** (1.13 g, 86%) as white solid, mp = 98-100 °C, $[\alpha]^{25}_D = -150.2°$. (c 1.16, $CHCl_3$). ^{1}H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.06 (s, 9 H) 1.66 - 1.77 (m, 1 H) 1.78 - 1.92 (m, 2 H) 2.08 - 2.23 (m, 1 H) 2.32 (s, 3 H) 3.48 - 3.59 (m, 1 H) 3.61 - 3.71 (m, 1 H) 5.02 (dd, *J*=8.04, 2.68 Hz, 1 H) 7.07 - 7.18 (m, 4 H). ^{13}C NMR (125 MHz, CHLOROFORM-*d*) δ 21.01, 23.14, 36.66, 54.57, 57.45, 126.46, 129.03, 136.09, 141.58. HRMS (EI) Calcd for $C_{15}H_{24}NOS$ [M+H]: 266.1579, Found 265.1589.



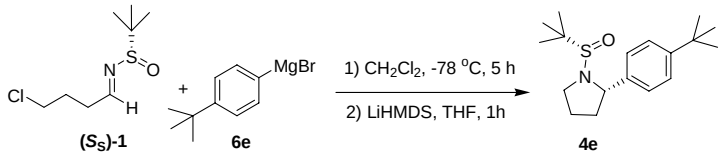
(*S*)-1-((*S*)-2-Methyl-propane-2-sulfinyl)-2-(4-methoxyphenyl)-pyrrolidine (4d):

Following the general procedure (GP2), the reaction of γ-chloro *N*-sulfinyl aldimine (S_S)-1 (1.05 g, 5.0 mmol) with 4-methoxyphenylmagnisum bromide (12.0 mL, 6.0 mmol) and LiHMDS (7.5 mL, 7.5 mmol) afforded pyrrolidine **4d** (1.20 g, 88%) as white solid, mp = 85-87 °C, $[\alpha]^{25}_D = -140.98°$. (c 1.07, MeOH). ^{1}H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.07 (s, 9 H) 1.74 (dd, *J*=7.72, 3.94 Hz, 1 H) 1.78 - 1.93 (m, 2 H) 2.05 - 2.20 (m, 1 H) 3.43 - 3.58 (m, 1 H) 3.61 - 3.70 (m, 1 H) 3.79 (s, 3 H) 4.99 (dd, *J*=7.88, 2.84 Hz, 1 H) 6.85 (d, 2 H) 7.16 (d, *J*=8.83 Hz, 21 H). ^{13}C NMR (125 MHz, CHLOROFORM-*d*) δ 23.15, 24.16, 36.62, 54.21, 55.18, 57.42, 113.72, 127.63, 136.63, 158.29. HRMS (EI) Calcd for $C_{15}H_{24}NO_2S$ [M+H]: 282.1528, Found 282.1526.



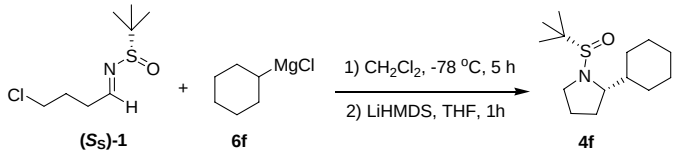
(*R*)-1-((*R*)-2-Methyl-propane-2-sulfinyl)-2-(4-methoxyphenyl)-pyrrolidine (*ent*-4d):

Following the general procedure (GP2), the reaction of γ-chloro *N*-sulfinyl aldimine (S_S)-1 (1.05 g, 5.0 mmol) with 4-methoxyphenylmagnisum bromide (12.0 mL, 6.0 mmol) and LiHMDS (7.5 mL, 7.5 mmol) afforded pyrrolidine **4d** (1.17 g, 83%) as white solid, mp = 85-86 °C, $[\alpha]^{25}_D$ = + 139.98°. (c 1.13, MeOH). ^{1}H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.06 (s) 1.69 - 1.91 (m) 2.13 (dd, *J*=11.82, 9.30 Hz) 3.47 - 3.56 (m) 3.79 (s) 5.00 (dd, *J*=7.88, 3.15 Hz) 6.85 (d, *J*=8.51 Hz) 7.16 (d, *J*=8.51 Hz). ^{13}C NMR (125 MHz, CHLOROFORM-*d*) δ 23.17, 24.16, 36.65, 55.23, 57.46, 113.73, 127.66, 136.69, 158.29. HRMS (EI) Calcd for $C_{15}H_{24}NO_2S$ [M+H]: 282.1528, Found 282.1519.



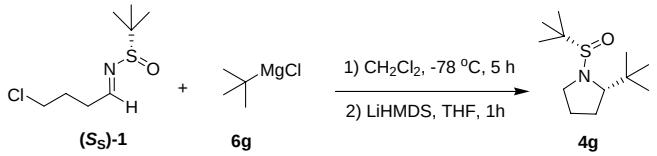
(*S*)-1-((*S*)-2-Methyl-propane-2-sulfinyl)-2-(4-*t*-butylphenyl)-pyrrolidine (4e):

Following the general procedure (GP2), the reaction of γ-chloro *N*-sulfinyl aldimine (S_S)-1 (1.05 g, 5.0 mmol) with 4-*t*-butylphenylmagnisum bromide (6.0 mL, 6.0 mmol) and LiHMDS (7.5 mL, 7.5 mmol) afforded pyrrolidine **4e** (1.30 g, 85%) as white solid, mp = 55-56 °C, $[\alpha]^{25}_D$ = – 140.03° (c 0.98, MeOH). ^{1}H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.06 (s, 9 H) 1.31 (s, 9 H) 1.71 - 1.92 (m, 3 H) 2.07 - 2.20 (m, 1 H) 3.45 - 3.54 (m, 1 H) 3.60 - 3.72 (m, 1 H) 5.02 (dd, *J*=8.04, 2.36 Hz, 1 H) 7.16 (d, *J*=8.20 Hz, 2 H) 7.31 (d, *J*=8.20 Hz, 2 H). ^{13}C NMR (125 MHz, CHLOROFORM-*d*) δ 23.18, 24.14, 31.39, 34.40, 36.49, 54.10, 57.48, 57.89, 125.17, 126.17, 141.28, 149.35. HRMS (EI) Calcd for $C_{18}H_{30}BrNOS$ [M+H]: 308.2048, Found 330.0530.



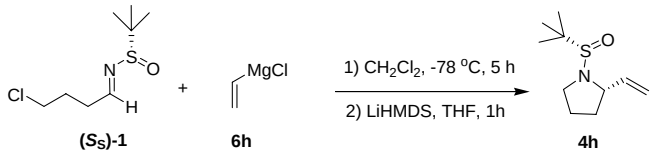
(*S*)-1-((*S*)-2-Methyl-propane-2-sulfinyl)-2-cyclohexyl pyrrolidine (4f):

Following the general procedure (GP2), the reaction of γ-chloro *N*-sulfinyl aldimine (S_S)-1 (1.05 g, 5.0 mmol) with cyclohexylmagnisum chloride (3.0 mL, 6.0 mmol) and LiHMDS (7.5 mL, 7.5 mmol) afforded pyrrolidine **4f** (1.01 g, 90%) as viscous oil, $[\alpha]^{25}_D = -40.0°$. (c 1.29, $CHCl_3$). ^{1}H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 0.86 - 1.06 (m, 2 H) 1.06 - 1.18 (m, 1 H) 1.14 - 1.31 (m, 11 H) 1.59 - 1.83 (m, 1 H) 3.11 - 3.25 (m, 1 H) 3.27 - 3.42 (m, 1 H) 3.50 - 3.64 (m, 1 H). ^{13}C NMR (125 MHz, CHLOROFORM-*d*) δ 23.49, 25.13, 26.30, 26.55, 27.13, 27.48, 30.73, 41.57, 50.06, 57.59, 62.73, HRMS (EI) Calcd for $C_{14}H_{28}NOS$ [M+H]: 258.1892, Found 258.1858.



(*S*)-1-((*S*)-2-Methyl-propane-2-sulfinyl)-2-*t*-butyl pyrrolidine (4g):

Following the general procedure (GP2), the reaction of γ-chloro *N*-sulfinyl aldimine (S_S)-1 (1.05 g, 5.0 mmol) with *t*-butylmagnisum chloride (3.0 mL, 6.0 mmol) and LiHMDS (7.5 mL, 7.5 mmol) afforded pyrrolidine **4g** (0.97 g, 84%) as viscous oil, $[\alpha]^{25}_D = -76.6°$. (c 1.04, $CHCl_3$). ^{1}H NMR (501 MHz, CHLOROFORM-*d*) □ ppm 0.96 (s, 9 H) 1.25 (s, 9 H) 1.70 - 1.93 (m, 4 H) 3.08 - 3.19 (m, 1 H) 3.28 - 3.38 (m, 1 H) 3.50 (dd, *J*=8.04, 4.26 Hz, 1 H). ^{13}C NMR (125 MHz, CHLOROFORM-*d*) δ 23.75, 25.12, 27.02, 27.40, 35.85, 48.28, 58.75, 68.30. HRMS (EI) Calcd for $C_{12}H_{25}NOS$ [M+H]: 232.1732, Found 232.1729.



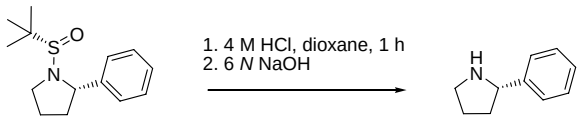
(*S*)-1-((*S*)-2-Methyl-propane-2-sulfinyl)-2-vinyl pyrrolidine (4h):

Following the general procedure (GP2), the reaction of γ-chloro *N*-sulfinyl aldimine (S_S)-1 (1.05 g, 5.0 mmol) with vinylmagnisum chloride (3.0 mL, 6.0 mmol) and LiHMDS (7.5 mL, 7.5 mmol) afforded pyrrolidine **4 h** (0.82 g, 82%) as viscous oil, $[\alpha]^{25}_D = -72.6°$. (c 1.14, $CHCl_3$). ^{1}H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.18 (s, 9 H) 1.50 - 1.65 (m, 1 H) 1.67 - 1.79 (m, 1 H) 1.82 - 1.95 (m, 1 H) 2.02 (dd, *J*=11.98, 4.10 Hz, 1 H) 2.70 - 2.87 (m, 1 H) 3.71 - 3.81 (m, 1 H) 4.03 (q, *J*=7.36 Hz, 1 H) 5.06 (d, *J*=10.72 Hz, 1 H)

5.16 (d, *J*=17.02 Hz, 1 H) 5.59 - 5.76 (m, 1 H). ^{13}C NMR (125 MHz, CHLOROFORM-*d*) δ 23.78, 25.79, 32.60, 41.26, 57.01, 67.93, 115.73, 140.18. HRMS (EI) Calcd for $C_{10}H_{19}NOS$ [M+H]: 202.1266, Found 202.1253.

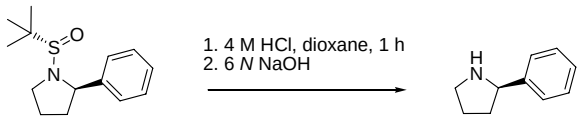
General Procedure (GP3) for the deprotection of *tert*-butanesulfinyl group from 4:

To a solution of **4** (2 mmol) in MeOH (10 mL) was added 4 M HCl solution (in dioxane, 2 mL). After the mixture was stirred at room temperature for 30 min, the mixture was concentrated to dryness and carefully dissolved in water (20 mL). The aqueous layer was washed with ethyl acetate (2 x 20 mL) and neutralized with 6N NaOH solution to pH ~13. Then, the resulting aqueous solution was extracted with ethyl acetate (3 × 30 mL). The combined organic layers were washed with brine and then dried over anhydrous Na_2SO_4. The organic layer was concentrated to dryness to obtained pure 2-substituted pyrrolidins **7**.



4a **7a**

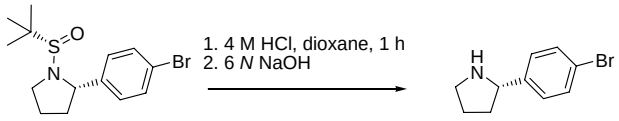
(*S*)-2-Phenyl pyrrolidine (7a): Following the general procedure (GP3), the reaction of **4a** (0.504 g, 2.0 mmol) with 4 M HCl solution (in dioxane, 2 mL) gives the (*S*)-2-phenyl pyrrolidine (7a) (291 mg, 99%) as viscous oil, $[\alpha]^{25}_{D} = -64.4°$. (c 1.02, CH_2Cl_2). ^{1}H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.61 - 1.73 (m, 1 H) 1.79 - 2.02 (m, 3 H) 2.14 - 2.24 (m, 1 H) 2.98 - 3.06 (m, 1 H) 3.17 - 3.25 (m, 1 H) 4.12 (t, *J*=7.72 Hz, 1 H) 7.18 - 7.27 (m, 2 H) 7.28 - 7.40 (m, 3 H). ^{13}C NMR (125 MHz, CHLOROFORM-*d*) δ 25.58, 34.29, 46.94, 62.57, 126.54, 126.98, 128.35, 144.66. HRMS (EI) Calcd for $C_{10}H_{14}N$ [M+H]: 148.1126, Found 148.1086.



***ent*-4a** ***ent*-7a**

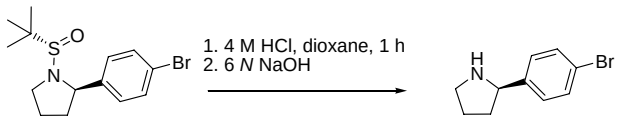
(*R*)-2-Phenyl pyrrolidine (*ent*-7a): Following the general procedure (GP3), the reaction of *ent*-**4a** (0.504 g, 2.0 mmol) with 4 M HCl solution (in dioxane, 2 mL) gives the (*R*)-2-

phenyl pyrrolidine (*ent*-7a) (290 mg, 98%) as viscous oil, $[\alpha]^{25}_{D}$ = +64.2°. (c 1.02, CH_2Cl_2). ^{1}H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.68 (m) 1.88 (m) 2.17 (m) 2.74 (br. s.) 2.99 (m) 3.18 (m) 4.09 (t, *J*=7.88 Hz) 7.28 (m). ^{13}C NMR (125 MHz, CHLOROFORM-*d*) δ 25.53, 34.20, 46.86, 62.60, 126.58, 126.87, 128.38, 144.34. HRMS (EI) Calcd for $C_{10}H_{14}N$ [M+H]: 148.1126, Found 148.1112.



4b **7b**

(*S*)-2-(4-Bromophenyl)-pyrrolidine (7b): Following the general procedure (GP3), the reaction of **4b** (658 mg, 2.0 mmol) with 4 M HCl solution (in dioxane, 2 mL) gives the (*S*)-2-(4-bromophenyl) pyrrolidine (7b) (442 mg, 98%) as viscous oil, $[\alpha]^{25}_{D}$ = − 51.9°. (c 1.27, CH_2Cl_2). ^{1}H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.50 - 1.64 (m, 1 H) 1.74 - 1.97 (m, 3 H) 2.09 - 2.21 (m, 1 H) 2.93 - 3.05 (m, 1 H) 3.11 - 3.20 (m, 1 H) 4.05 (t, *J*=7.72 Hz, 1 H) 7.22 (d, *J*=8.20 Hz, 2 H) 7.40 (d, *J*=8.51 Hz, 2 H). ^{13}C NMR (125 MHz, CHLOROFORM-*d*) δ 25.50, 34.47, 46.93, 61.82, 120.27, 128.25, 131.27, 144.17. HRMS (EI) Calcd for $C_{10}H_{13}BrN$ [M+H]: 226.0231, Found 226.0185.



***ent*-4b** ***ent*-7b**

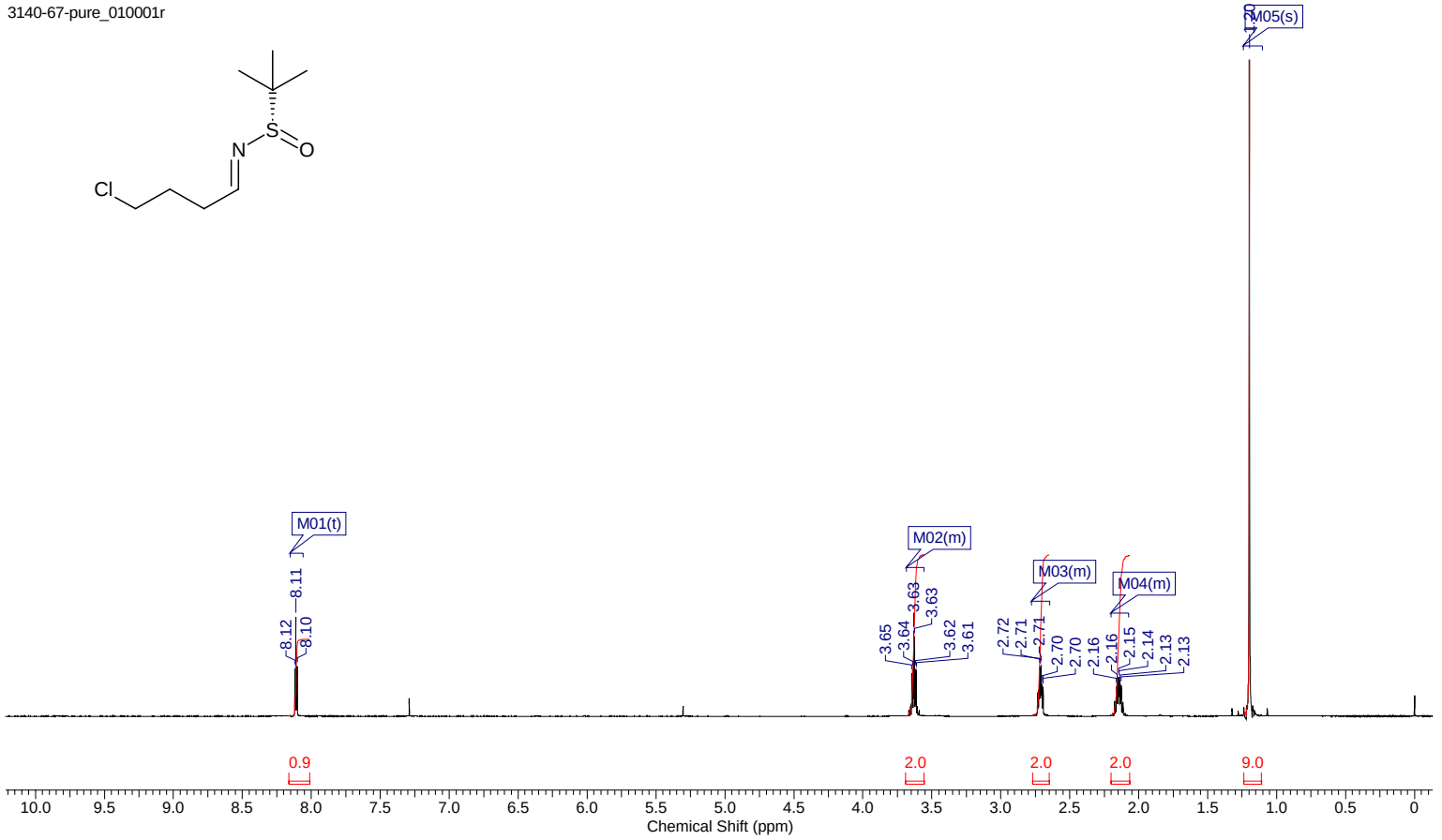
(*R*)-2-(4-Bromophenyl)-pyrrolidine (*ent*-7b): Following the general procedure (GP3), the reaction of *ent*-**4b** (658 mg, 2.0 mmol) with 4 M HCl solution (in dioxane, 2 mL) gives the (*R*)-2-(4-bromophenyl)-pyrrolidine (*ent*-7b) (440 mg, 97%) as viscous oil, $[\alpha]^{25}_{D}$ = + 52.1°. (c 1.01, CH_2Cl_2). ^{1}H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.59 (dd, *J*=12.30, 9.14 Hz) 1.85 (m) 2.16 (m) 2.99 (m) 3.15 (m) 4.05 (t, *J*=7.57 Hz) 7.22 (d, *J*=8.20 Hz) 7.41 (d, *J*=8.20 Hz).). ^{13}C NMR (125 MHz, CHLOROFORM-*d*) δ 25.50, 34.44, 46.93, 61.87, 120.36, 128.29, 131.33, 144.02. HRMS (EI) Calcd for $C_{10}H_{13}BrN$ [M+H]: 226.0231, Found 226.0217.

5/14/2009 2:35:32 PM

Acquisition Time (sec)	3.1719	***Comment***	3140-67-pure PROTON16 CDCl3 u leletra1 2			***Date***	14 Apr 2009 17:38:08
Date Stamp	14 Apr 2009 17:38:08			***File Name***	\\phuseh-S1603\LELETRA1.·.DATA\NMR data\3140-67-pure_010001r		
Frequency (MHz)	500.63	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect
Original Points Count	32768	***Owner***	drinkdo1	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	57.00	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	3093.2183	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	27.000		

1H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.20 (s, 9 H) 2.07 - 2.20 (m, 2 H) 2.65 - 2.78 (m, 2 H) 3.56 - 3.68 (m, 2 H) 8.11 (t, *J*=3.94 Hz, 1 H)
VerticalScaleFactor = 1

3140-67-pure_010001r

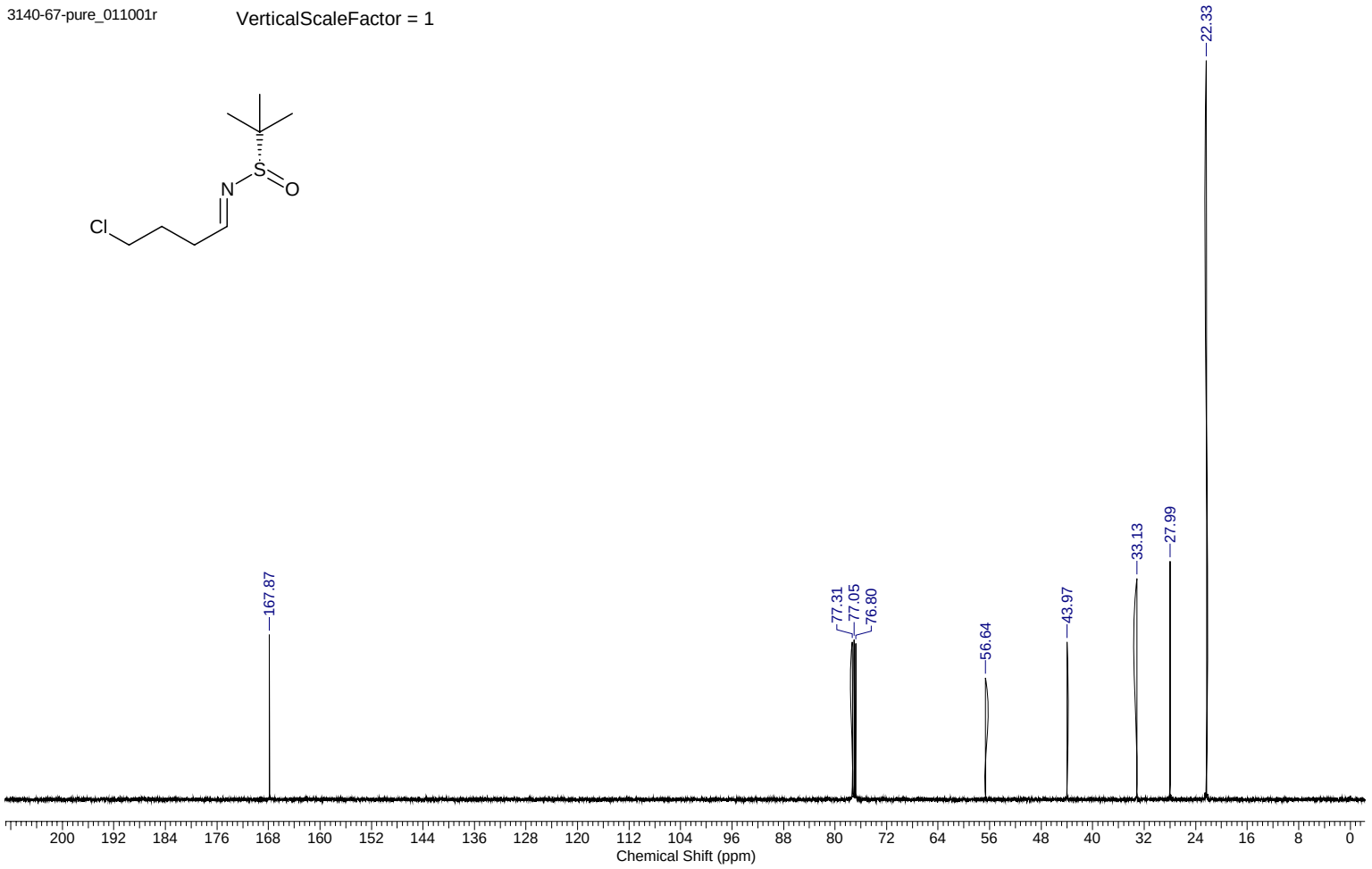


5/14/2009 2:39:59 PM

Acquisition Time (sec)	1.0912	***Comment***	3140-67-pure C13CPD256 CDCl3 u leletra1 2			***Date***	14 Apr 2009 17:46:40
Date Stamp	14 Apr 2009 17:46:40			***File Name***	\\phuseh-S1603\LELETRA1.:.DATA\NMR data\3140-67-pure_011001r		
Frequency (MHz)	125.88	***Nucleus***	13C	***Number of Transients***	256	***Origin***	spect
Original Points Count	32768	***Owner***	drinkdo1	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	13004.00	***SW(cyclical) (Hz)***	30030.03	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	12587.9785	***Sweep Width (Hz)***	30029.11	***Temperature (degree C)***	27.000		

3140-67-pure_011001r

VerticalScaleFactor = 1

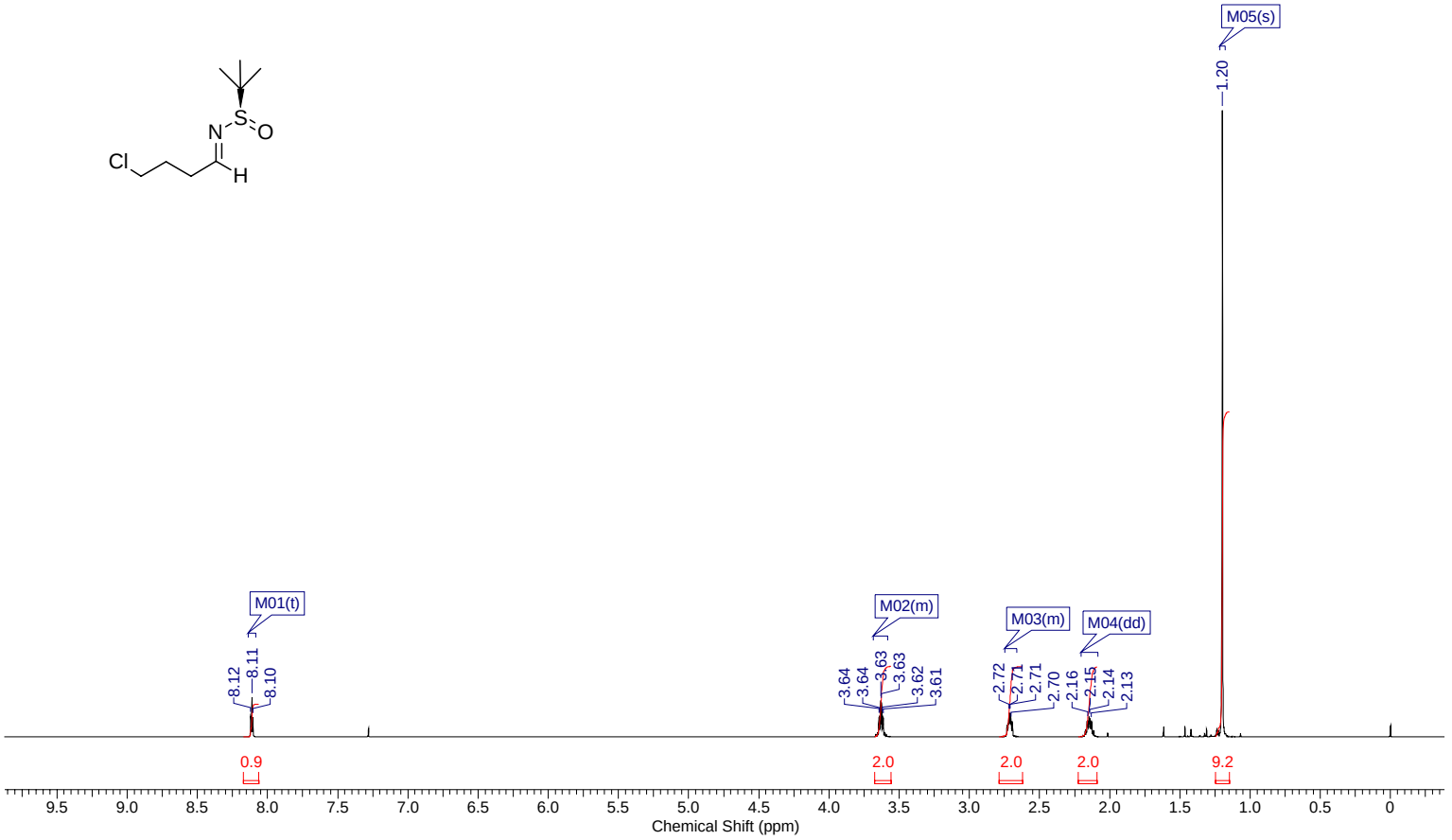


Acquisition Time (sec)	3.1719	***Comment***	3140-67-R PROTON16 CDCl3 u leletra1 22			***Date***	16 Jul 2009 21:24:16
Date Stamp	16 Jul 2009 21:24:16			***File Name***	\\phuseh-S1603\LELETRA1.:.DATA\NMR data\3140-67-R_010001r		
Frequency (MHz)	500.63	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect
Original Points Count	32768	***Owner***	nmrsu	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	101.60	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	3089.2134	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	27.000		

1H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.20 (s, 9 H) 2.14 (dd, *J*=6.46, 4.26 Hz, 2 H) 2.66 - 2.75 (m, 2 H) 3.58 - 3.69 (m, 2 H) 8.11 (t, *J*=3.94 Hz, 1 H)

3140-67-R_010001r

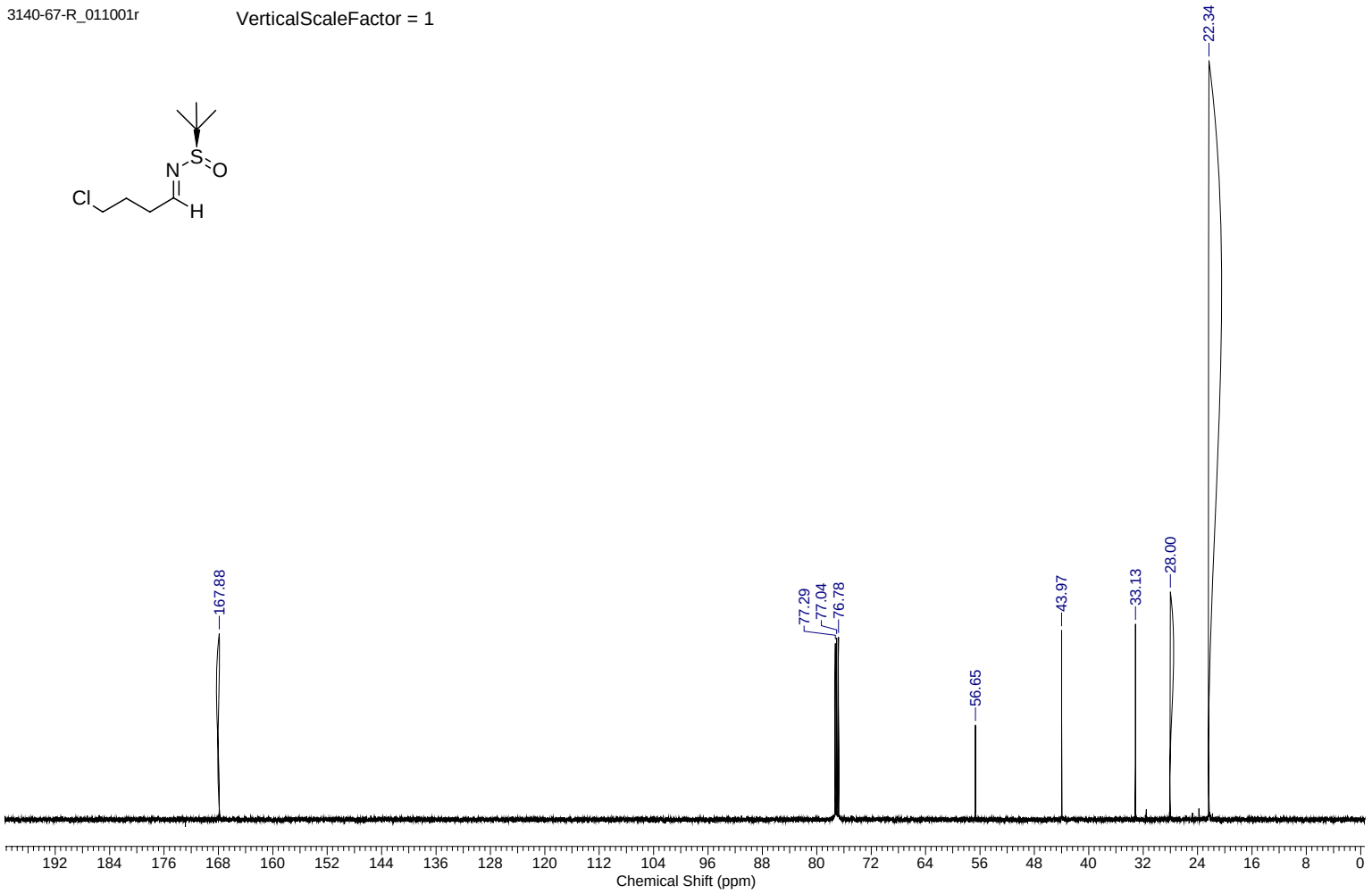
VerticalScaleFactor = 1



Acquisition Time (sec)	1.0912	***Comment***	3140-67-R C13CPD256 CDCl3 u leletra1 22			***Date***	16 Jul 2009 21:39:12
Date Stamp	16 Jul 2009 21:39:12			***File Name***	\\phuseh-S1603\LELETRA1.:.DATA\NMR data\3140-67-R_011001r		
Frequency (MHz)	125.88	***Nucleus***	13C	***Number of Transients***	256	***Origin***	spect
Original Points Count	32768	***Owner***	nmrsu	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	6502.00	***SW(cyclical) (Hz)***	30030.03	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	12587.9785	***Sweep Width (Hz)***	30029.11	***Temperature (degree C)***	27.000		

3140-67-R_011001r

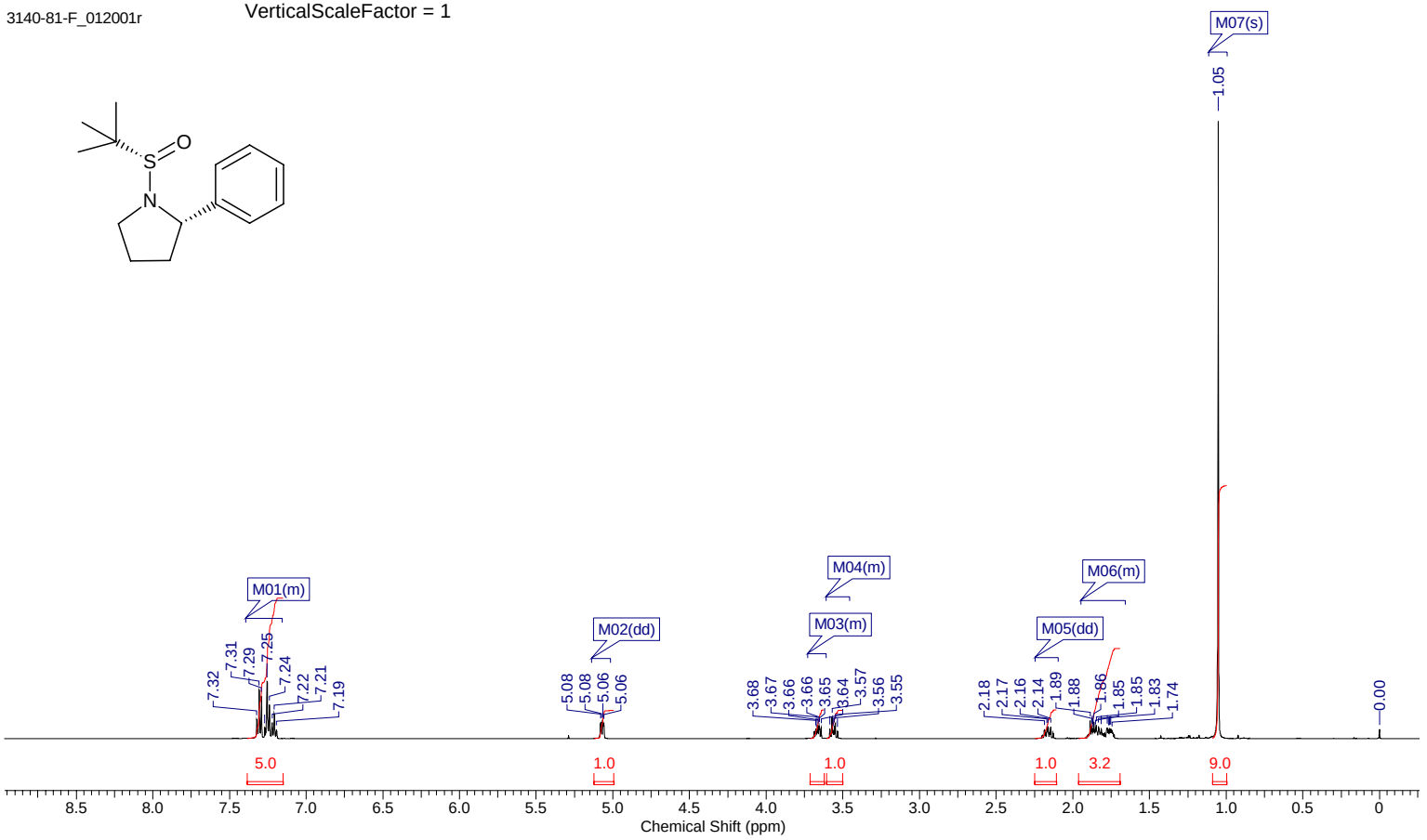
VerticalScaleFactor = 1



5/14/2009 2:48:14 PM

Acquisition Time (sec)	3.1719	***Comment***	3140-81-F PROTON1024 CDCl3 u leletra1 21			***Date***	10 May 2009 17:57:20
Date Stamp	10 May 2009 17:57:20			***File Name***	\\phuseh-S1603\LELETRA1.:DATA\NMR data\3140-81-F_012001r		
Frequency (MHz)	500.63	***Nucleus***	1H	***Number of Transients***	1024	***Origin***	spect
Original Points Count	32768	***Owner***	drinkdo1	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	71.80	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	3083.4517	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	27.000		

1H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.05 (s, 9 H) 1.66 - 1.95 (m, 3 H) 2.16 (dd, *J*=11.35, 8.51 Hz, 1 H) 3.45 - 3.61 (m, 1 H) 3.61 - 3.73 (m, 1 H) 5.07 (dd, *J*=8.20, 2.84 Hz, 1 H) 7.16 - 7.39 (m, 5 H)

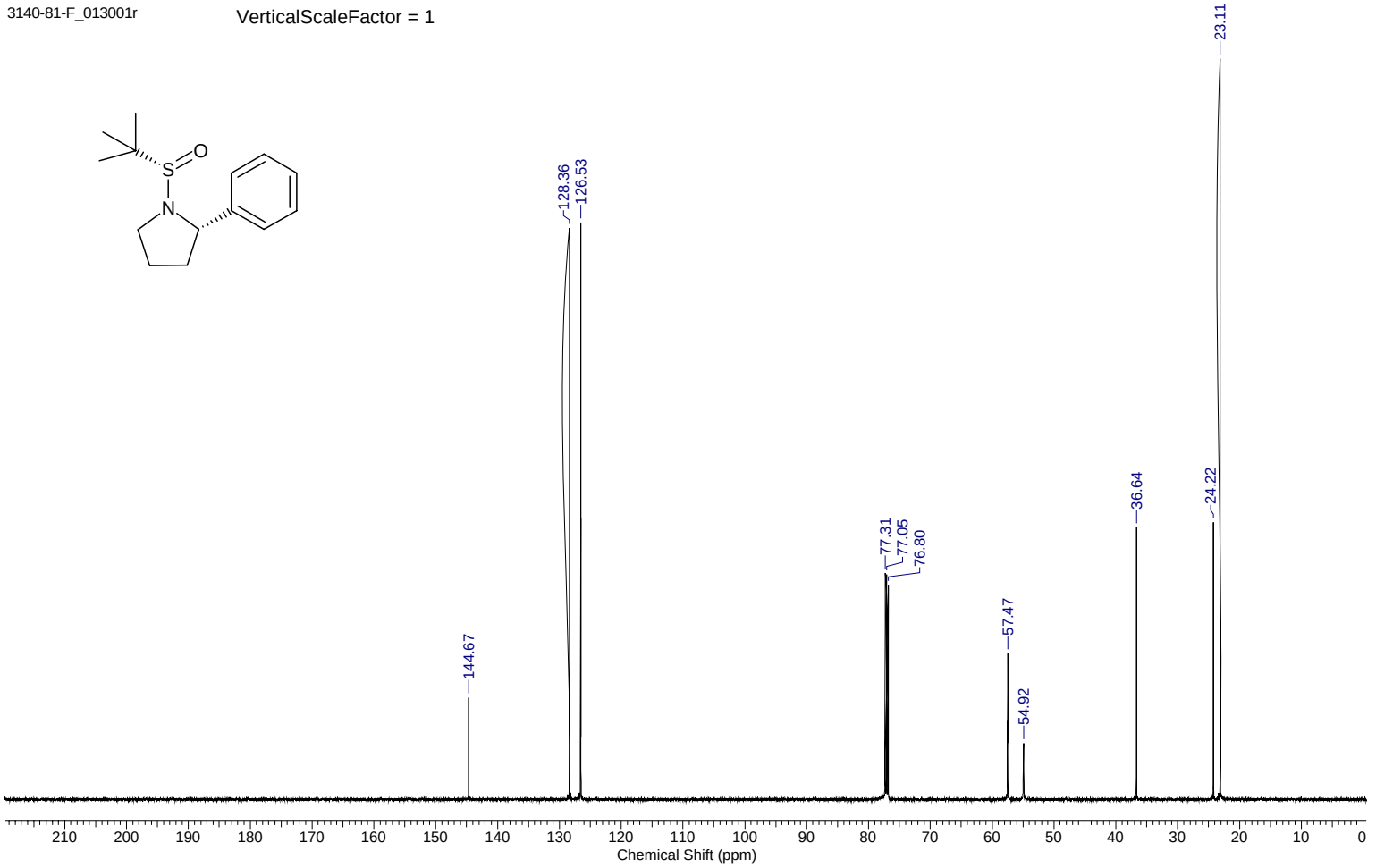


5/14/2009 2:54:02 PM

Acquisition Time (sec)	1.0912	***Comment***	3140-81-F C13CPD2048 CDCl3 u leletra1 21			***Date***	10 May 2009 18:57:04
Date Stamp	10 May 2009 18:57:04			***File Name***	\\phuseh-S1603\LELETRA1 ;·. DATA\NMR data\3140-81-F_013001r		
Frequency (MHz)	125.88	***Nucleus***	13C	***Number of Transients***	2048	***Origin***	spect
Original Points Count	32768	***Owner***	drinkdo1	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	4096.00	***SW(cyclical) (Hz)***	30030.03	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	14477.7813	***Sweep Width (Hz)***	30029.11	***Temperature (degree C)***	27.000		

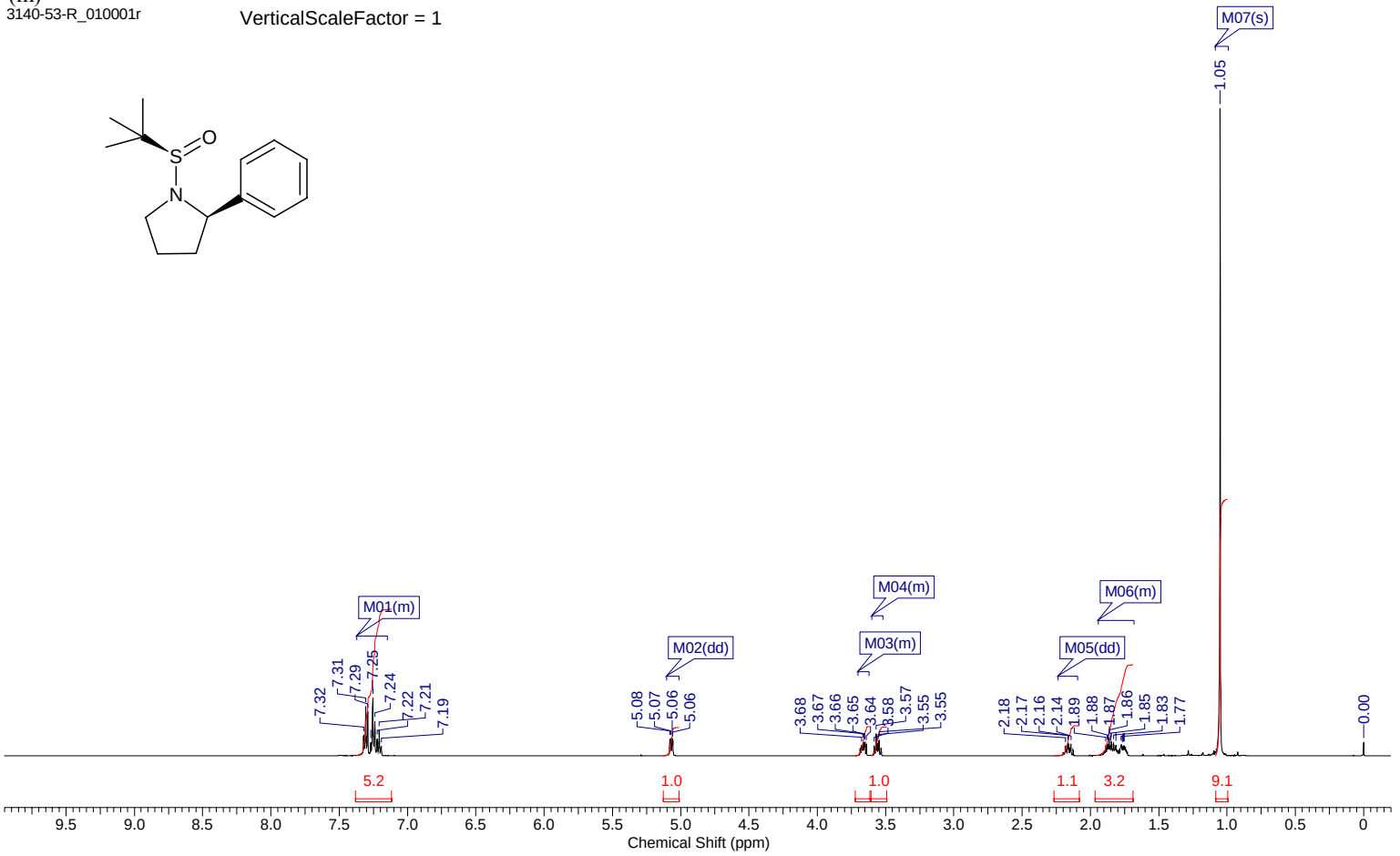
3140-81-F_013001r

VerticalScaleFactor = 1



Acquisition Time (sec)	3.1719	***Comment***	3140-53-R PROTON16 CDCl3 u leletra1 23			***Date***	16 Jul 2009 21:47:44
Date Stamp	16 Jul 2009 21:47:44			***File Name***	\\phuseh-S1603\LELETRA1.:.DATA\NMR data\3140-53-R_010001r		
Frequency (MHz)	500.63	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect
Original Points Count	32768	***Owner***	nmrsu	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	57.00	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	3083.5261	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	27.000		

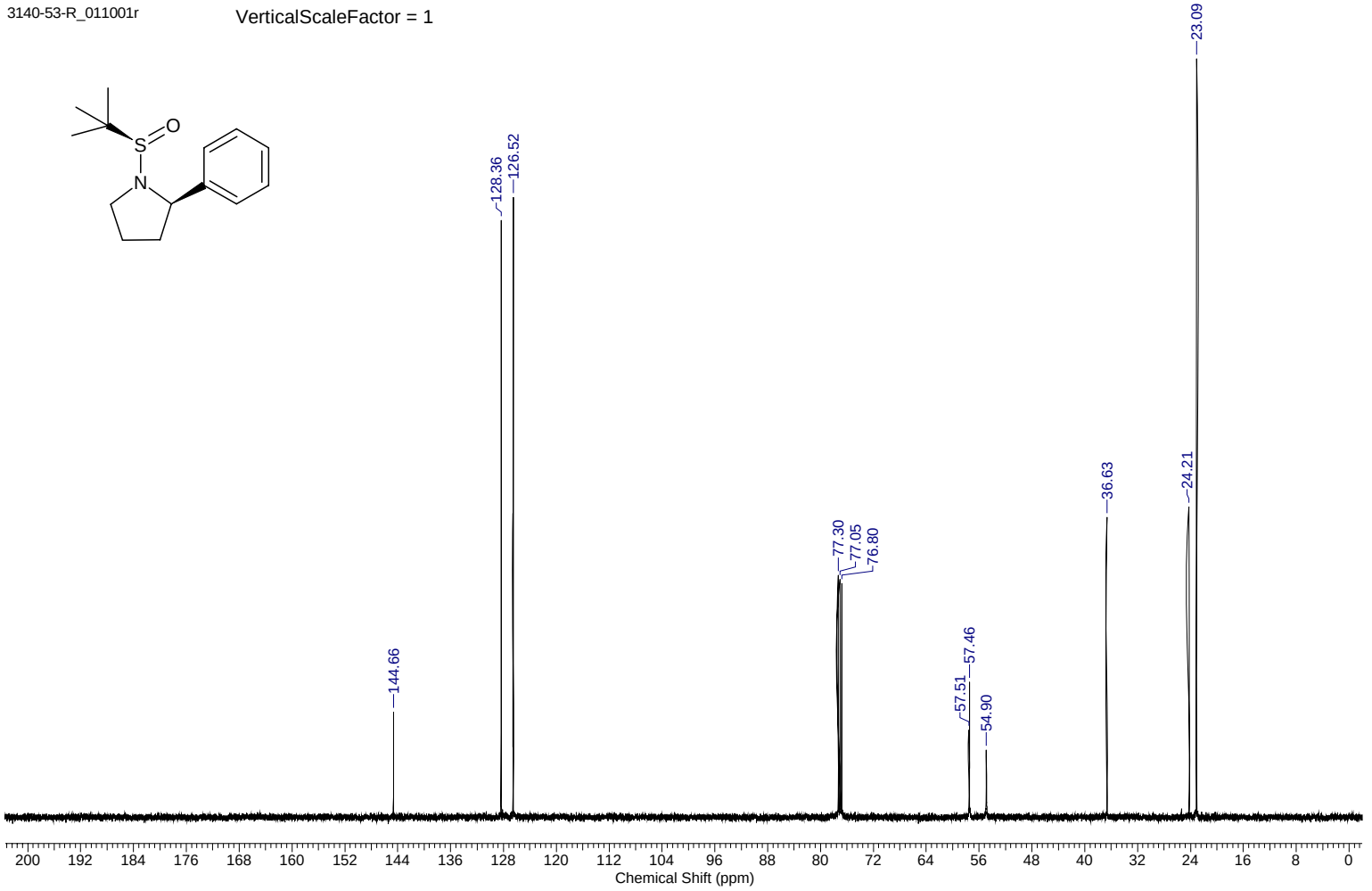
1H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.05 (s) 1.82 (m) 2.16 (dd, *J*=11.35, 8.51 Hz) 3.56 (m) 3.66 (m) 5.07 (dd, *J*=8.04, 2.68 Hz) 7.26 (m)



Acquisition Time (sec)	1.0912	***Comment***	3140-53-R C13CPD256 CDCl3 u leletra1 23			***Date***	16 Jul 2009 22:02:40
Date Stamp	16 Jul 2009 22:02:40			***File Name***	\\phuseh-S1603\LELETRA1 ·: DATA\NMR data\3140-53-R_011001r		
Frequency (MHz)	125.88	***Nucleus***	13C	***Number of Transients***	256	***Origin***	spect
Original Points Count	32768	***Owner***	nmrsu	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	13004.00	***SW(cyclical) (Hz)***	30030.03	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	12587.9785	***Sweep Width (Hz)***	30029.11	***Temperature (degree C)***	27.000		

3140-53-R_011001r

VerticalScaleFactor = 1



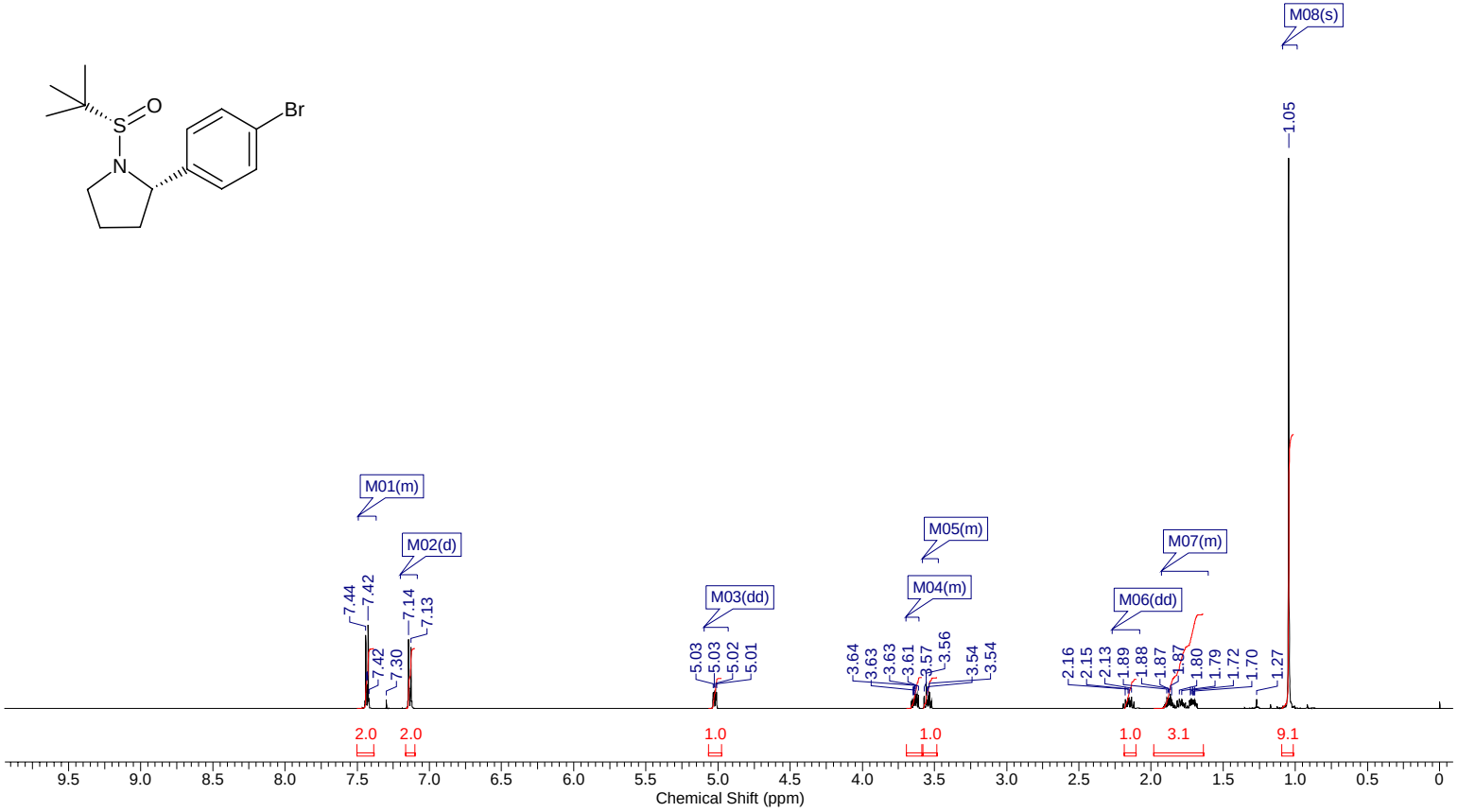
5/14/2009 3:11:08 PM

Acquisition Time (sec)	3.1719	***Comment***	PROTON CDCl3 u leletra1 11			***Date***	23 Oct 2008 06:36:48
Date Stamp	23 Oct 2008 06:36:48			***File Name***	\\phuseh-S1603\LELETRA1.-.DATA\NMR data\3105-106-F_010001r		
Frequency (MHz)	500.63	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect
Original Points Count	32768	***Owner***	nmrsu	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	50.80	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	3097.0930	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	27.000		

1H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.05 (s, 9 H) 1.60 - 1.93 (m, 3 H) 2.16 (dd, *J*=11.98, 9.14 Hz, 1 H) 3.47 - 3.58 (m, 1 H) 3.61 - 3.70 (m, 1 H) 5.02 (dd, *J*=8.20, 2.84 Hz, 1 H) 7.14 (d, *J*=8.51 Hz, 2 H) 7.37 - 7.49 (m, 2 H)

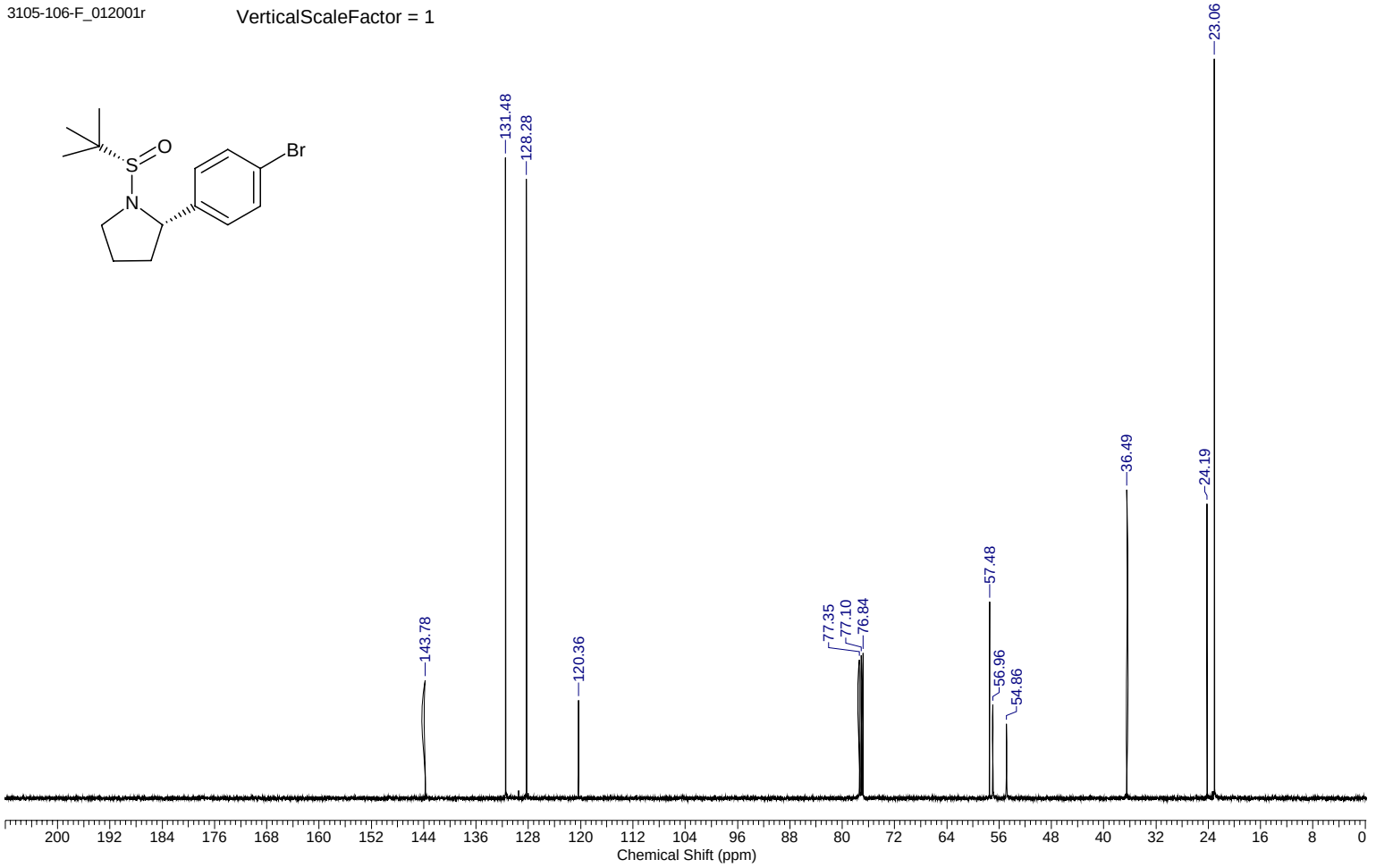
VerticalScaleFactor = 1

3105-106-F_010001r



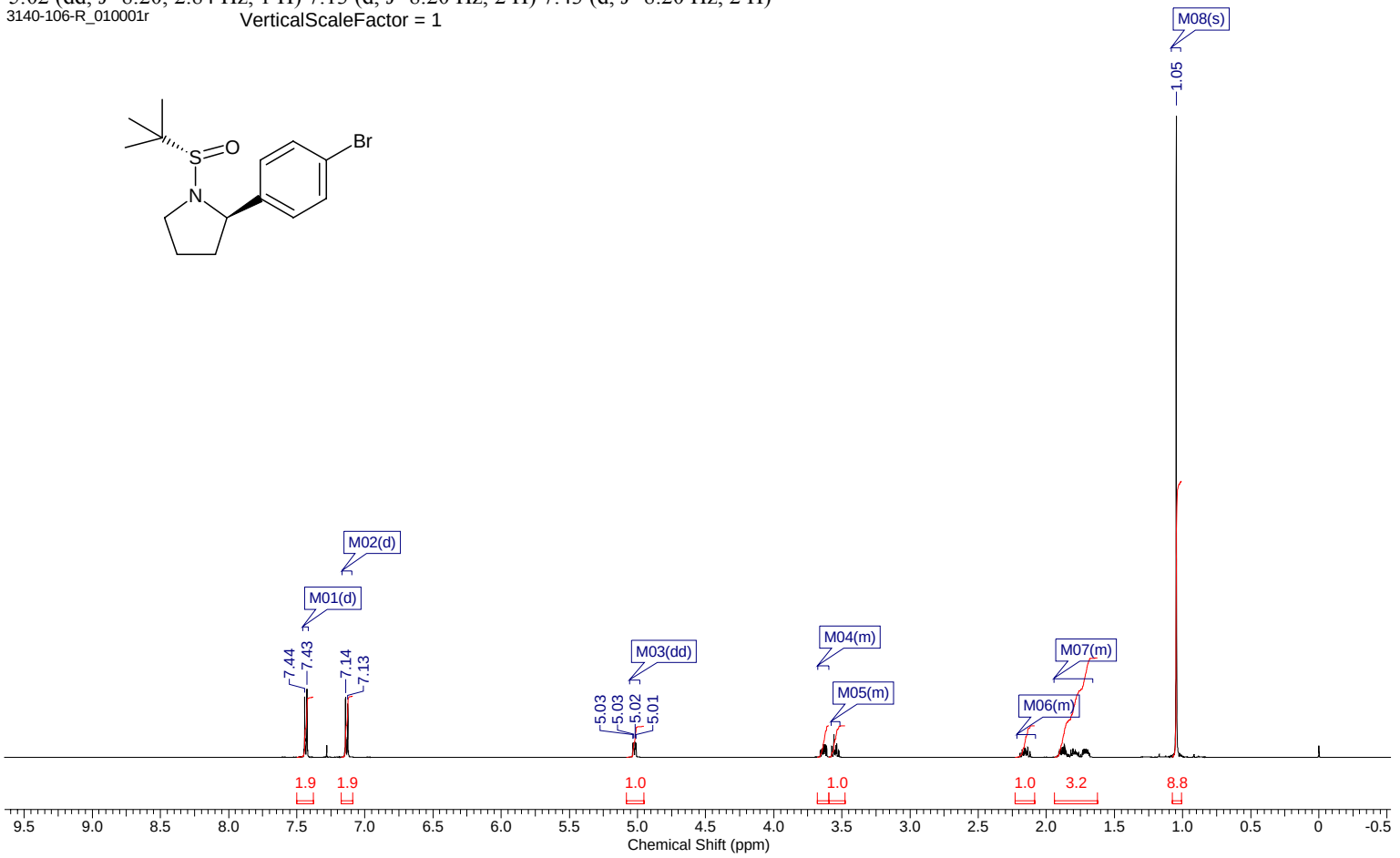
5/14/2009 3:18:24 PM

Acquisition Time (sec)	1.0912	***Comment***	C13CPD256 CDCl3 u leletra1 11			***Date***	22 Oct 2008 19:58:56
Date Stamp	22 Oct 2008 19:58:56			***File Name***	\\phuseh-S1603\LELETRA1 ;. DATA\NMR data\3105-106-F_012001r		
Frequency (MHz)	125.88	***Nucleus***	13C	***Number of Transients***	256	***Origin***	spect
Original Points Count	32768	***Owner***	nmrsu	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	11585.20	***SW(cyclical) (Hz)***	30030.03	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	12587.9785	***Sweep Width (Hz)***	30029.11	***Temperature (degree C)***	27.000		



Acquisition Time (sec)	3.1719	***Comment***	3140-106-R PROTON16 CDCl3 u leletra1 49			***Date***	24 Jul 2009 21:41:20
Date Stamp	24 Jul 2009 21:41:20			***File Name***	\\phuseh-S1603\LELETRA1.:.DATA\NMR data\3140-106-R_010001r		
Frequency (MHz)	500.63	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect
Original Points Count	32768	***Owner***	nmrsu	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	64.00	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	3088.1926	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	27.000		

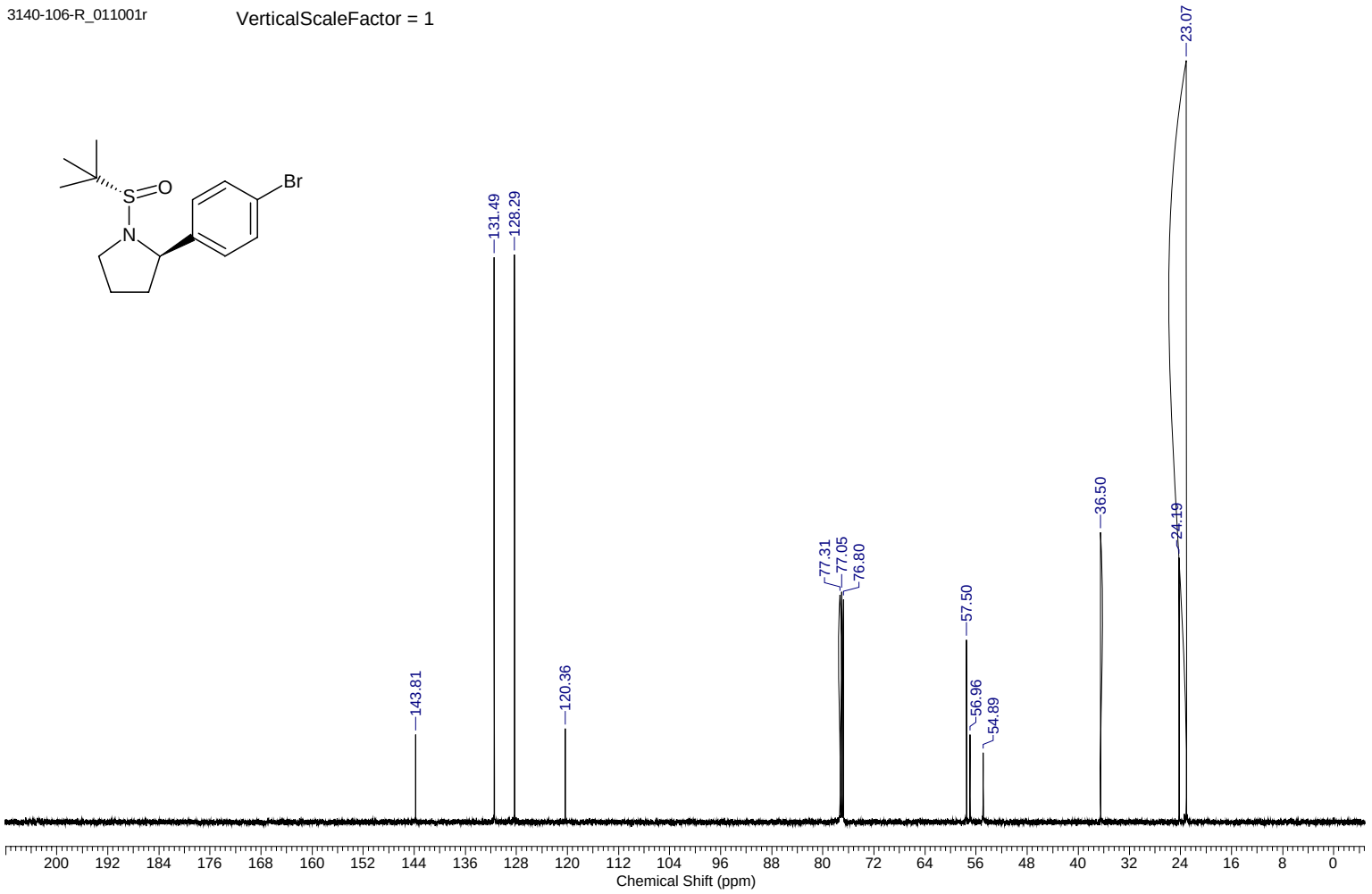
1H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.05 (s, 9 H) 1.66 - 1.94 (m, 3 H) 2.08 - 2.21 (m, 1 H) 3.51 - 3.58 (m, 1 H) 3.60 - 3.68 (m, 1 H) 5.02 (dd, *J*=8.20, 2.84 Hz, 1 H) 7.13 (d, *J*=8.20 Hz, 2 H) 7.43 (d, *J*=8.20 Hz, 2 H)



Acquisition Time (sec)	1.0912	***Comment***	3140-106-R C13CPD256 CDCl3 u leletra1 49			***Date***	24 Jul 2009 21:54:08
Date Stamp	24 Jul 2009 21:54:08			***File Name***	\\phuseh-S1603\LELETRA1.·.DATA\NMR data\3140-106-R_011001r		
Frequency (MHz)	125.88	***Nucleus***	13C	***Number of Transients***	256	***Origin***	spect
Original Points Count	32768	***Owner***	nmrsu	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	8192.00	***SW(cyclical) (Hz)***	30030.03	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	12587.9785	***Sweep Width (Hz)***	30029.11	***Temperature (degree C)***	27.000		

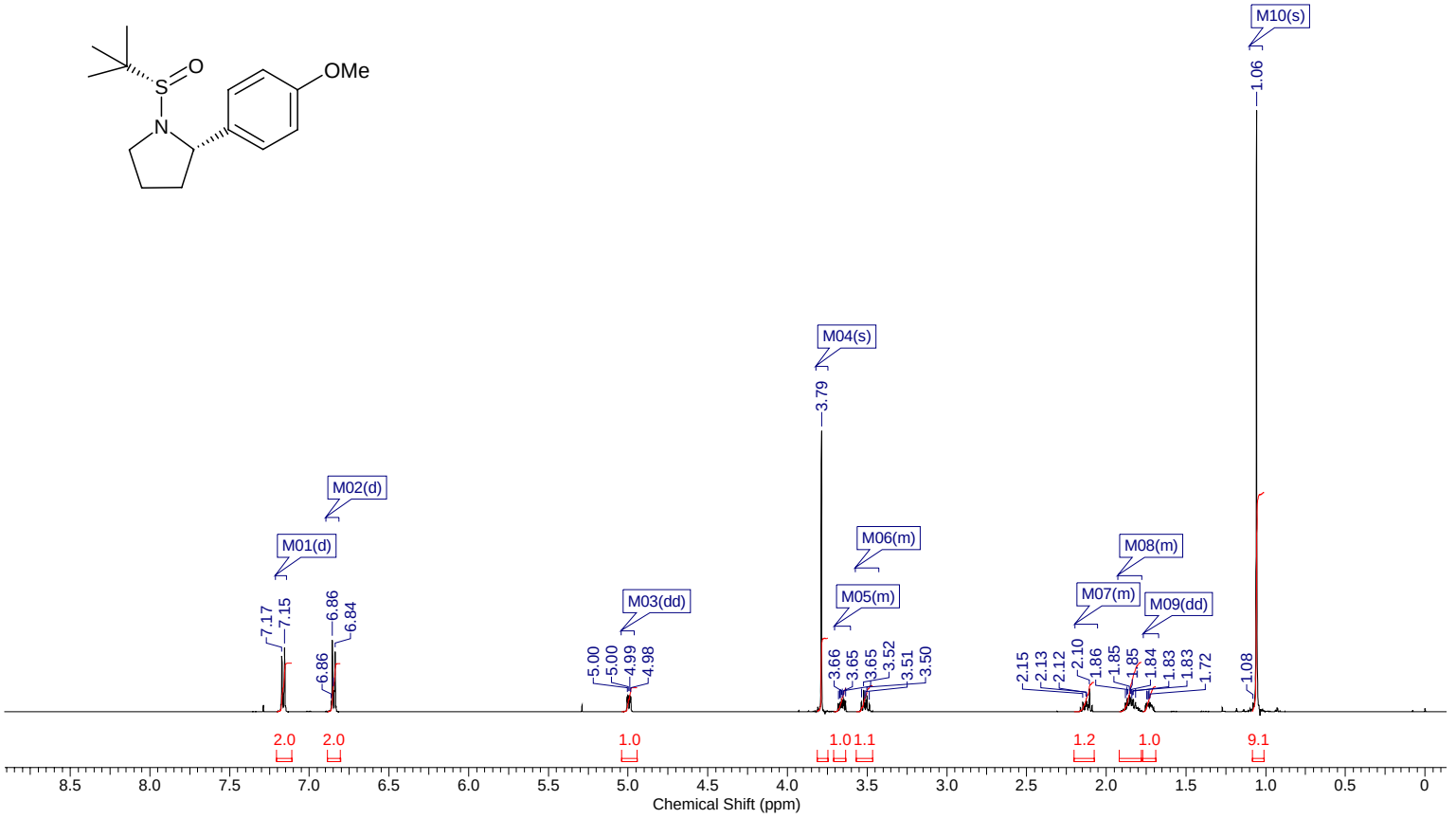
3140-106-R_011001r

VerticalScaleFactor = 1



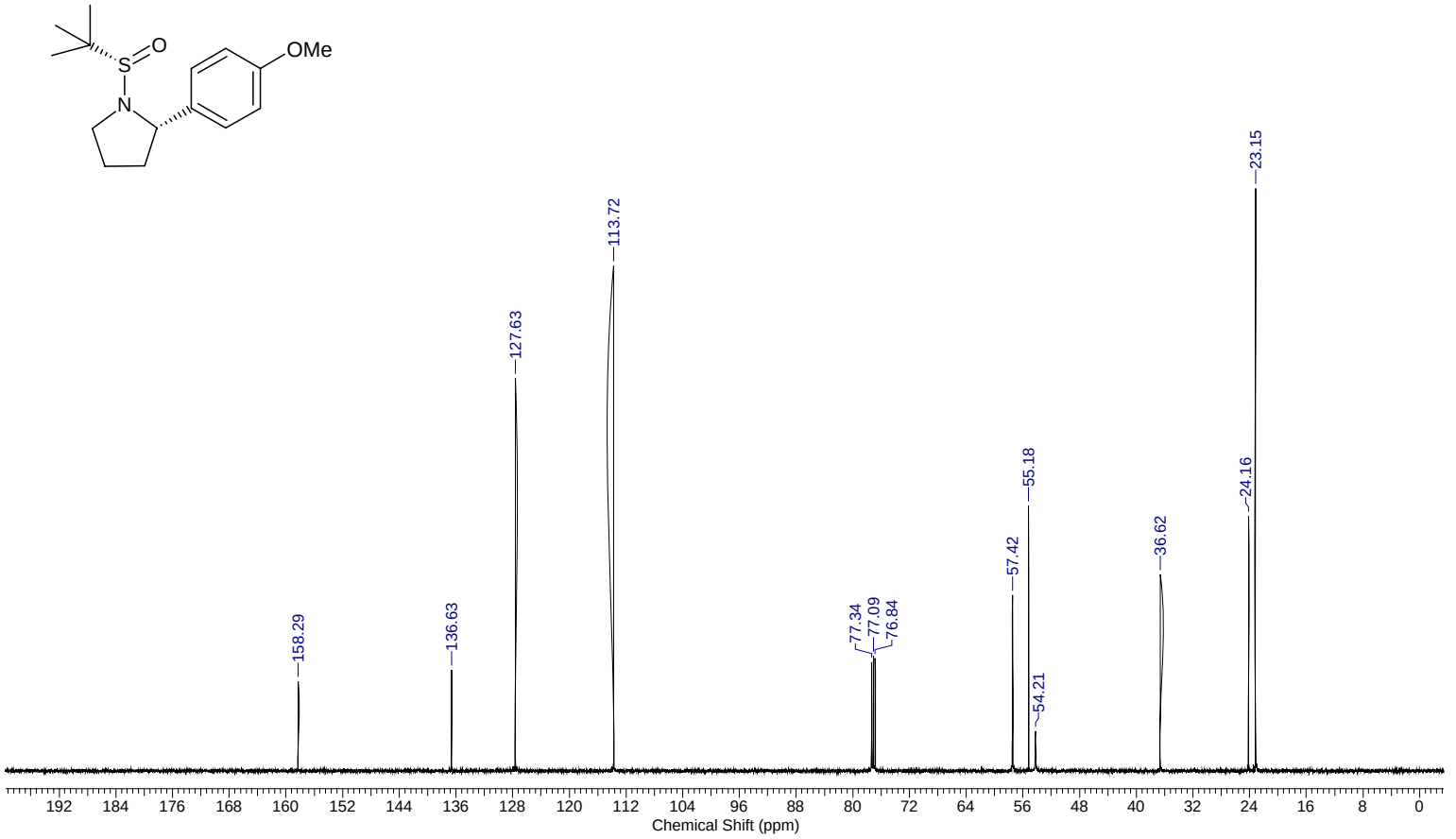
Acquisition Time (sec)	3.1719	***Comment***				***Date***	09 Jun 2009 18:35:44
Date Stamp	09 Jun 2009 18:35:44			***File Name***			
Frequency (MHz)	500.63	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect
Original Points Count	32768	***Owner***	nmrsu	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	35.90	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	3093.0684	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	27.000		

1H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.07 (s, 9 H) 1.74 (dd, *J*=7.72, 3.94 Hz, 1 H) 1.78 - 1.93 (m, 2 H) 2.05 - 2.20 (m, 1 H) 3.43 - 3.58 (m, 1 H) 3.61 - 3.70 (m, 1 H) 3.79 (s, 3 H) 4.99 (dd, *J*=7.88, 2.84 Hz, 1 H) 6.85 (d, 2 H) 7.16 (d, *J*=8.83 Hz, 21 H)
3170-5-F_010001r



Acquisition Time (sec)	1.0912	***Comment***				***Date***	09 Jun 2009 18:48:32
Date Stamp	09 Jun 2009 18:48:32			***File Name***			
Frequency (MHz)	125.88	***Nucleus***	13C	***Number of Transients***	256	***Origin***	spect
Original Points Count	32768	***Owner***	nmrsu	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	13004.00	***SW(cyclical) (Hz)***	30030.03	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	12587.9785	***Sweep Width (Hz)***	30029.11	***Temperature (degree C)***	27.000		

3170-5-F_011001r

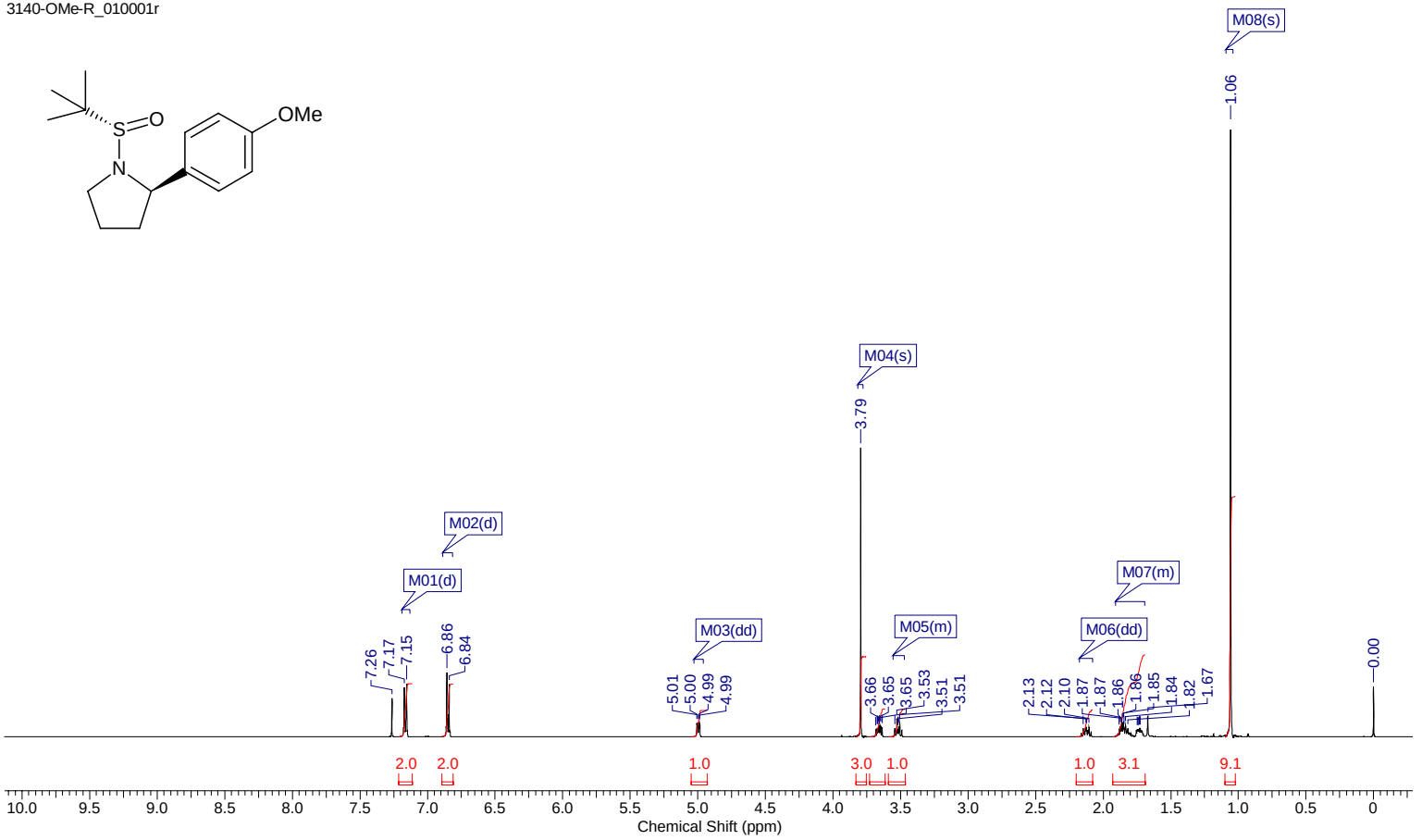


Acquisition Time (sec)	3.1719	***Comment***	3140-OMe-R PROTON16 CDCl3 u leletra1 40			***Date***	24 Jul 2009 16:17:04
Date Stamp	24 Jul 2009 16:17:04			***File Name***	\\phuseh-S1603\LELETRA1.:.DATA\NMR data\3140-OMe-R_010001r		
Frequency (MHz)	500.63	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect
Original Points Count	32768	***Owner***	nmrsu	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	256.00	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	3080.1375	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	27.000		

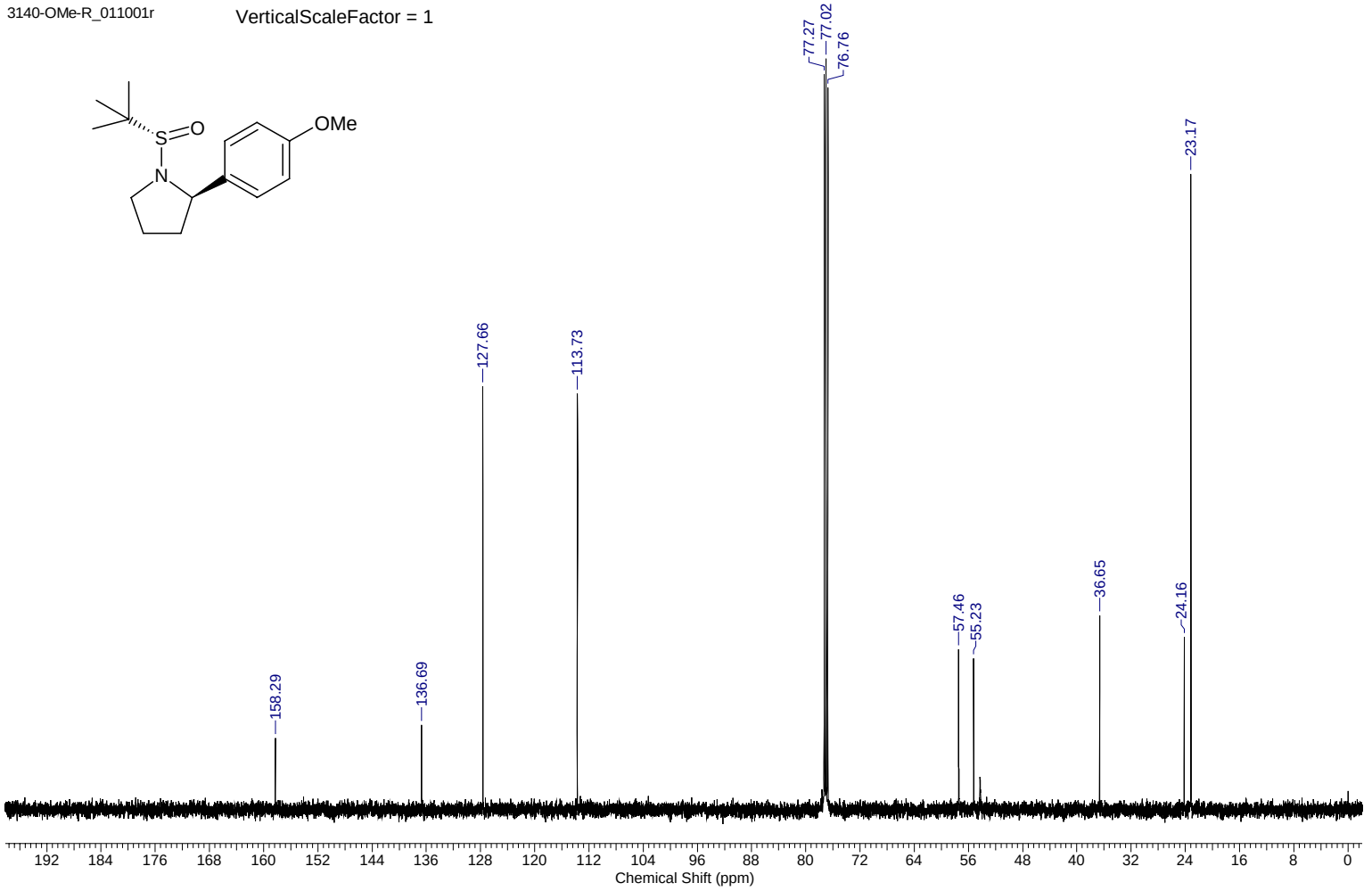
1H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.06 (s) 1.69 - 1.91 (m) 2.13 (dd, *J*=11.82, 9.30 Hz) 3.47 - 3.56 (m) 3.79 (s) 5.00 (dd, *J*=7.88, 3.15 Hz) 6.85 (d, *J*=8.51 Hz) 7.16 (d, *J*=8.51 Hz)

VerticalScaleFactor = 1

3140-OMe-R_010001r

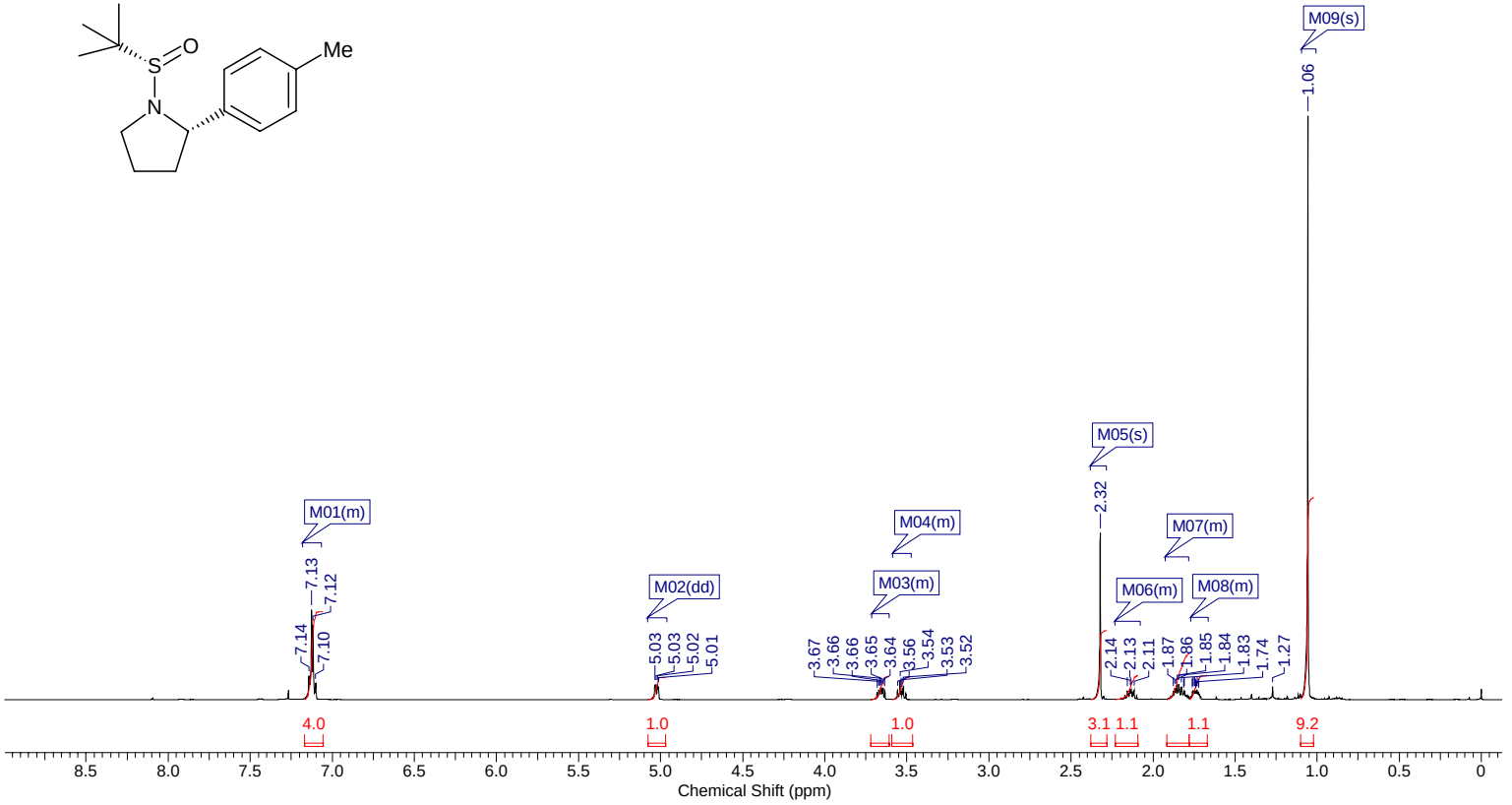


Acquisition Time (sec)	1.0912	***Comment***	3140-OMe-R C13CPD256 CDCl3 u leletra1 40			***Date***	24 Jul 2009 16:29:52
Date Stamp	24 Jul 2009 16:29:52			***File Name***	\\phuseh-S1603\LELETRA1_:_DATA\NMR data\3140-OMe-R_011001r		
Frequency (MHz)	125.88	***Nucleus***	13C	***Number of Transients***	256	***Origin***	spect
Original Points Count	32768	***Owner***	nmrsu	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	8192.00	***SW(cyclical) (Hz)***	30030.03	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	12587.9785	***Sweep Width (Hz)***	30029.11	***Temperature (degree C)***	27.000		



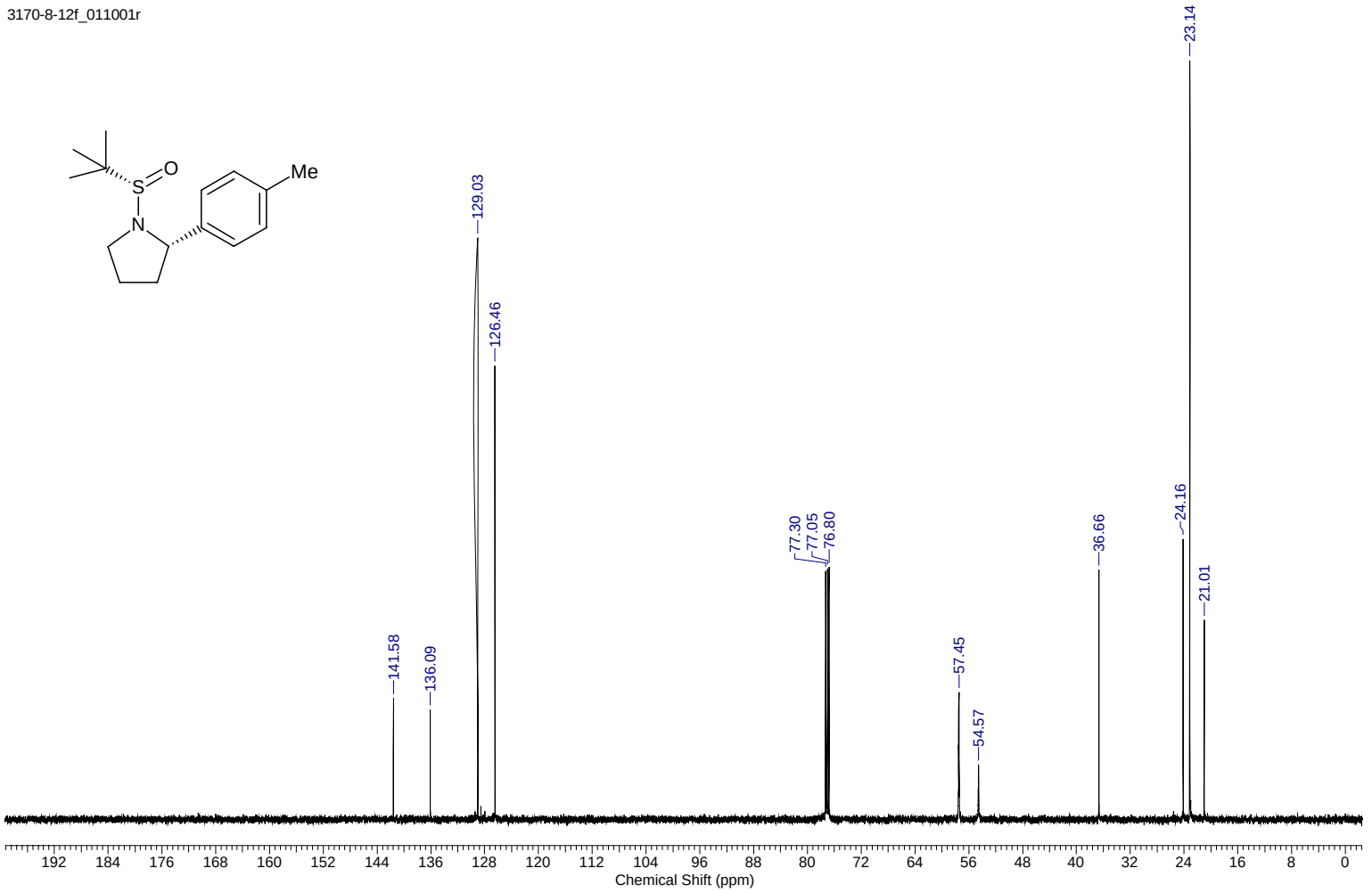
Acquisition Time (sec)	3.1719	***Comment***	3170-8-12f PROTON16 CDCl3 u dasso8 3			***Date***	15 Jun 2009 14:49:36
Date Stamp	15 Jun 2009 14:49:36			***File Name***	C:\Documents and Settings\leletra1\Desktop\sonia\3170-8-12f_010001r		
Frequency (MHz)	500.63	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect
Original Points Count	32768	***Owner***	nmrsu	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	57.00	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	3082.5256	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	27.000		

1H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.06 (s, 9 H) 1.66 - 1.77 (m, 1 H) 1.78 - 1.92 (m, 2 H) 2.08 - 2.23 (m, 1 H) 2.32 (s, 3 H) 3.48 - 3.59 (m, 1 H) 3.61 - 3.71 (m, 1 H) 5.02 (dd, *J*=8.04, 2.68 Hz, 1 H) 7.07 - 7.18 (m, 4 H)
3170-8-12f_010001r



Acquisition Time (sec)	1.0912	***Comment***				***Date***	15 Jun 2009 16:55:28
Date Stamp	15 Jun 2009 16:55:28			***File Name***			
Frequency (MHz)	125.88	***Nucleus***	13C	***Number of Transients***	256	***Origin***	spect
Original Points Count	32768	***Owner***	nmrsu	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	7298.20	***SW(cyclical) (Hz)***	30030.03	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	12587.9785	***Sweep Width (Hz)***	30029.11	***Temperature (degree C)***	27.000		

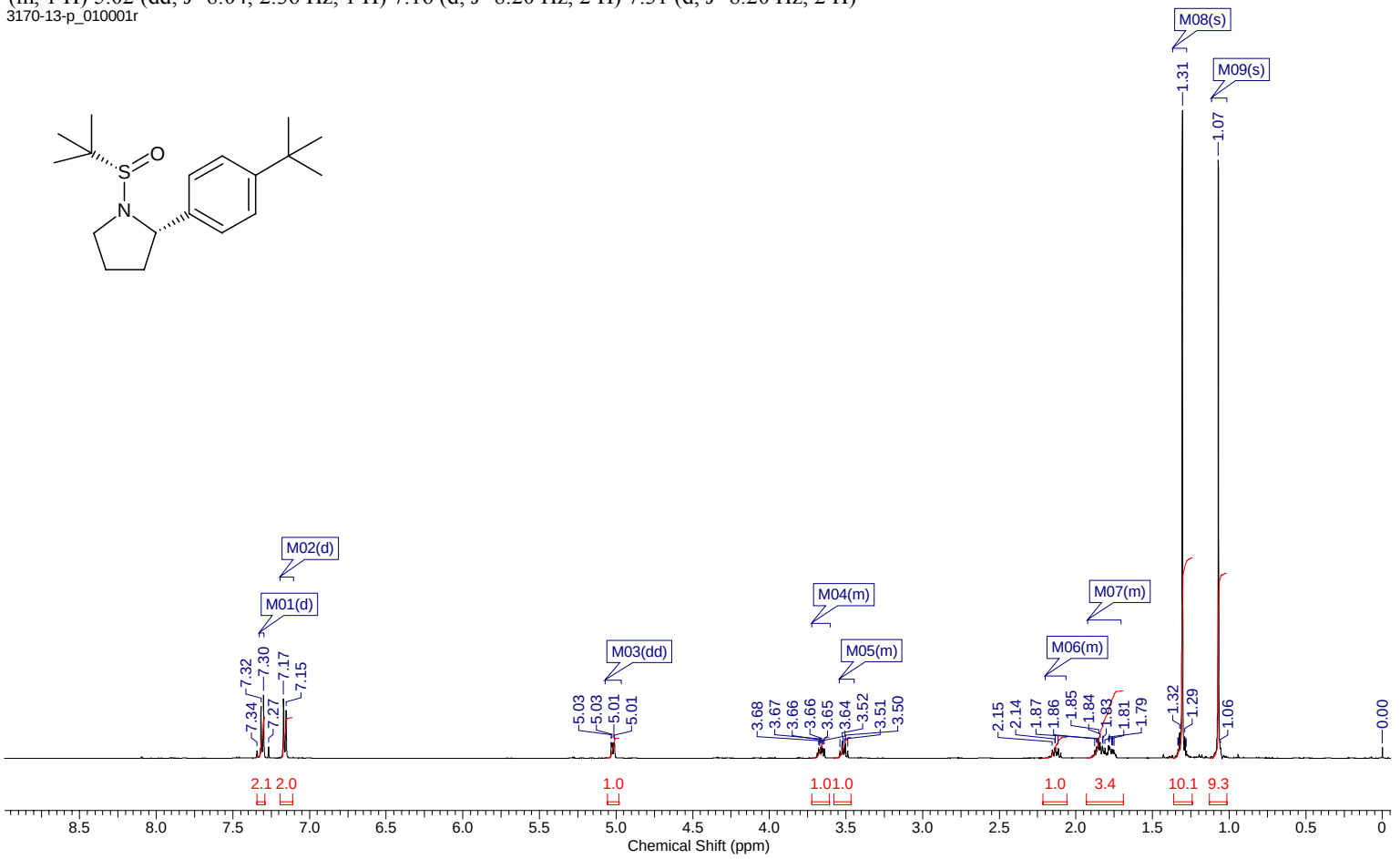
3170-8-12f_011001r



Acquisition Time (sec)	3.1719	***Comment***	3170-13-p PROTON16 CDCl3 u dasso8 22			***Date***	16 Jun 2009 20:09:36
Date Stamp	16 Jun 2009 20:09:36			***File Name***	C:\Documents and Settings\leletra1\Desktop\sonia\3170-13-p_010001r		
Frequency (MHz)	500.63	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect
Original Points Count	32768	***Owner***	nmrsu	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	57.00	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	3082.4949	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	27.000		

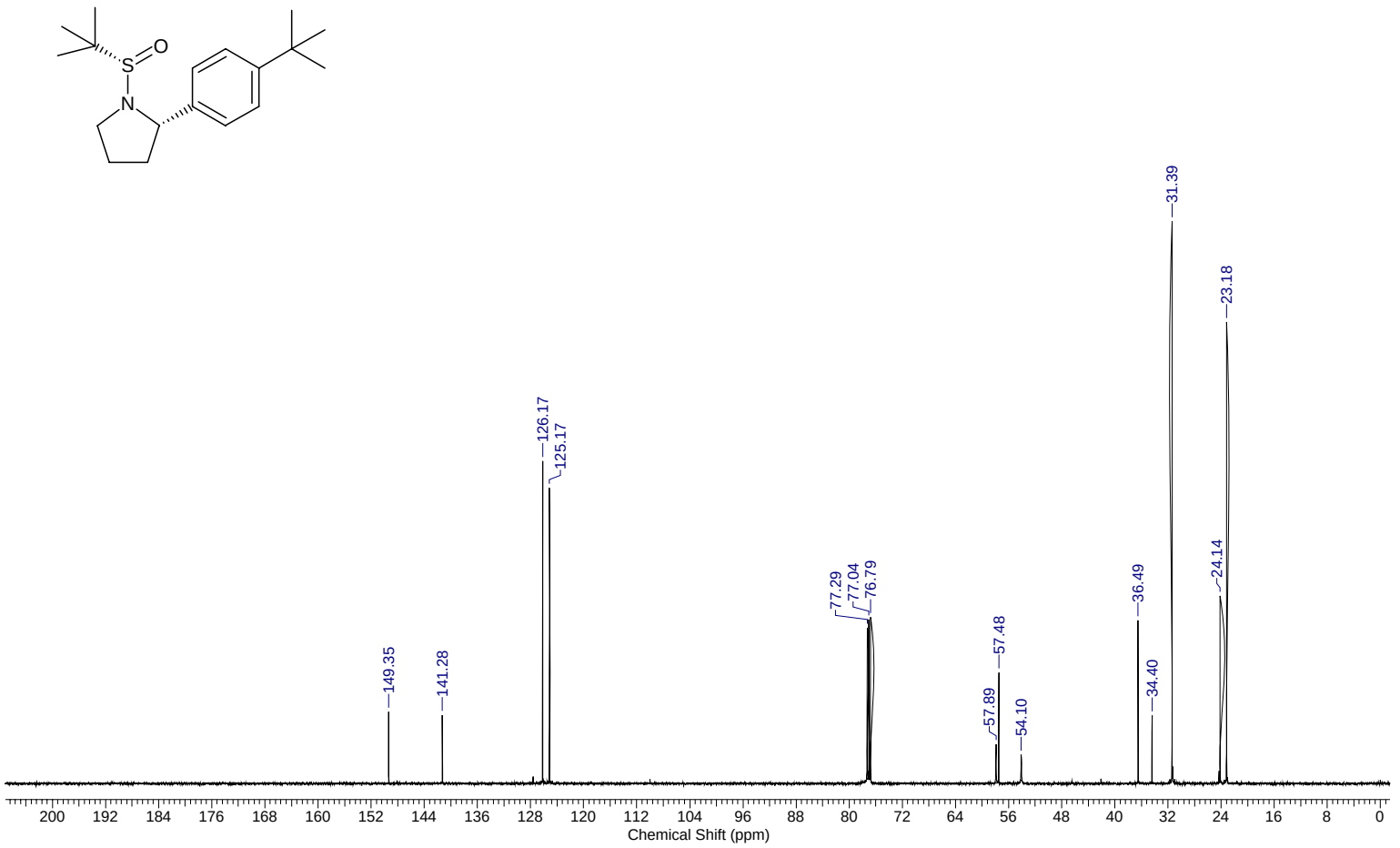
1H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.06 (s, 9 H) 1.31 (s, 9 H) 1.71 - 1.92 (m, 3 H) 2.07 - 2.20 (m, 1 H) 3.45 - 3.54 (m, 1 H) 3.60 - 3.72 (m, 1 H) 5.02 (dd, *J*=8.04, 2.36 Hz, 1 H) 7.16 (d, *J*=8.20 Hz, 2 H) 7.31 (d, *J*=8.20 Hz, 2 H)

3170-13-p_010001r



Acquisition Time (sec)	1.0912	***Comment***	3170-13-p C13CPD2048 CDCl3 u dasso8 22			***Date***	17 Jun 2009 03:18:24
Date Stamp	17 Jun 2009 03:18:24			***File Name***	C:\Documents and Settings\leletra1\Desktop\sonia\3170-13-p_012001r		
Frequency (MHz)	125.88	***Nucleus***	13C	***Number of Transients***	2048	***Origin***	spect
Original Points Count	32768	***Owner***	nmrsu	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	13004.00	***SW(cyclical) (Hz)***	30030.03	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	14477.4912	***Sweep Width (Hz)***	30029.11	***Temperature (degree C)***	27.000		

3170-13-p_012001r



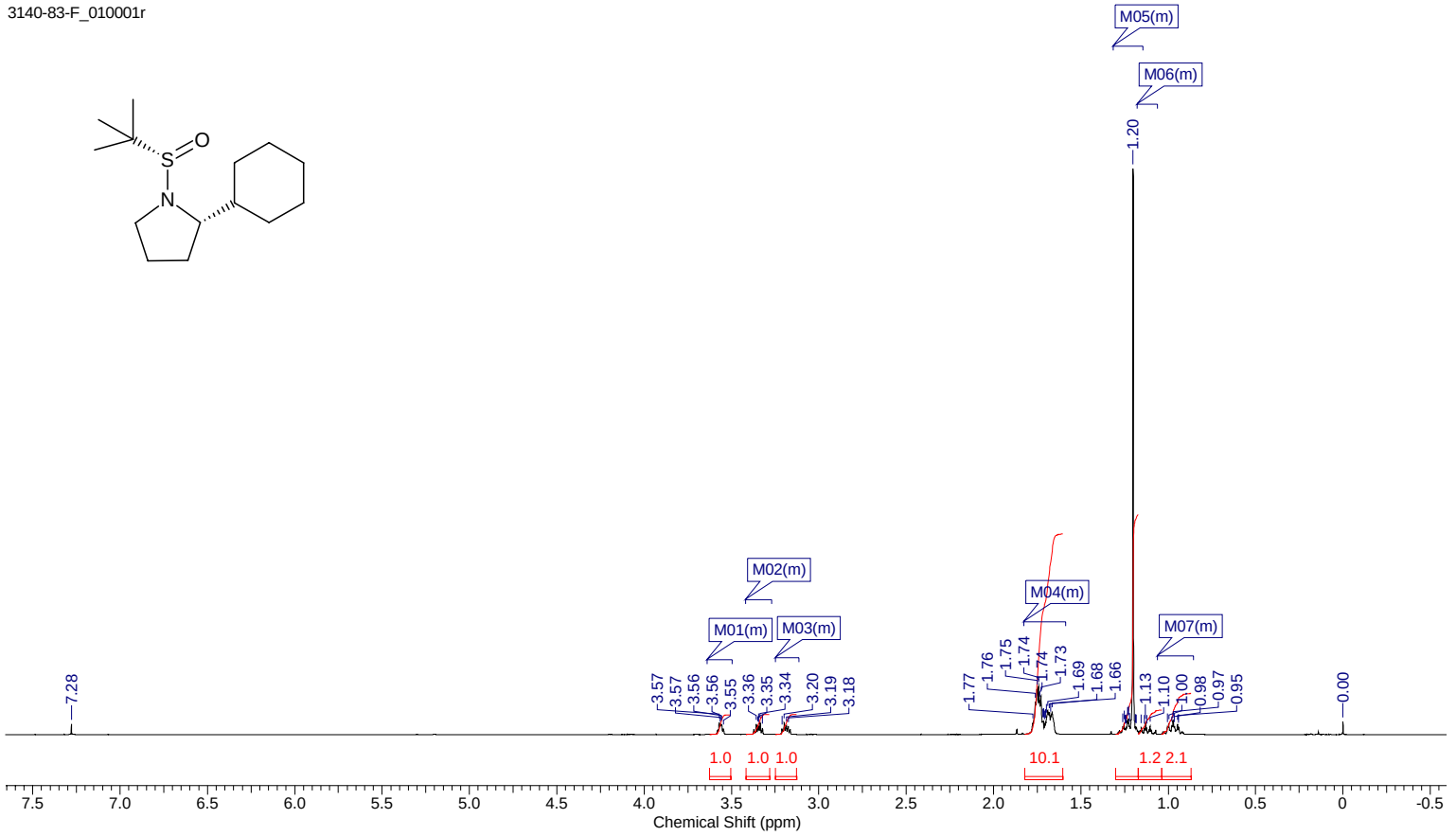
5/14/2009 3:26:28 PM

Acquisition Time (sec)	3.1719	***Comment***	3140-83-F PROTON16 CDCl3 u leletra1 43			***Date***	12 May 2009 17:10:24
Date Stamp	12 May 2009 17:10:24			***File Name***	\\phuseh-S1603\LELETRA1.:.DATA\NMR data\3140-83-F_010001r		
Frequency (MHz)	500.63	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect
Original Points Count	32768	***Owner***	drinkdo1	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	80.60	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	3087.8423	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	27.000		

1H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 0.86 - 1.06 (m, 2 H) 1.06 - 1.18 (m, 1 H) 1.14 - 1.31 (m, 11 H) 1.59 - 1.83 (m, 1 H) 3.11 - 3.25 (m, 1 H) 3.27 - 3.42 (m, 1 H) 3.50 - 3.64 (m, 1 H)

VerticalScaleFactor = 1

3140-83-F_010001r

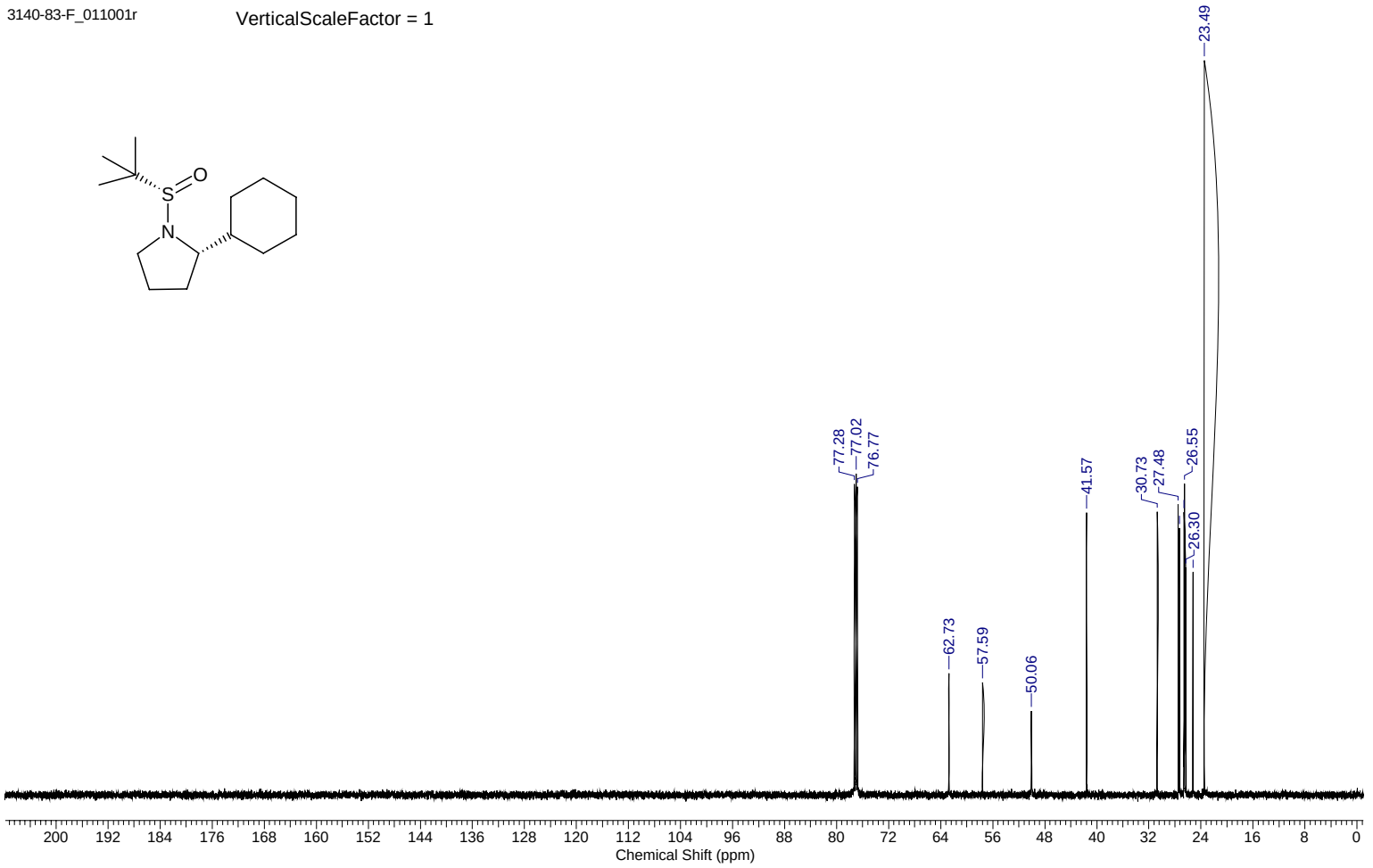


5/14/2009 3:30:05 PM

Acquisition Time (sec)	1.0912	***Comment***	3140-83-F C13CPD256 CDCl3 u leletra1 43			***Date***	12 May 2009 17:18:56
Date Stamp	12 May 2009 17:18:56			***File Name***	\\phuseh-S1603\LELETRA1_:DATA\NMR data\3140-83-F_011001r		
Frequency (MHz)	125.88	***Nucleus***	13C	***Number of Transients***	256	***Origin***	spect
Original Points Count	32768	***Owner***	drinkdo1	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	8192.00	***SW(cyclical) (Hz)***	30030.03	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	12587.9785	***Sweep Width (Hz)***	30029.11	***Temperature (degree C)***	27.000		

3140-83-F_011001r

VerticalScaleFactor = 1

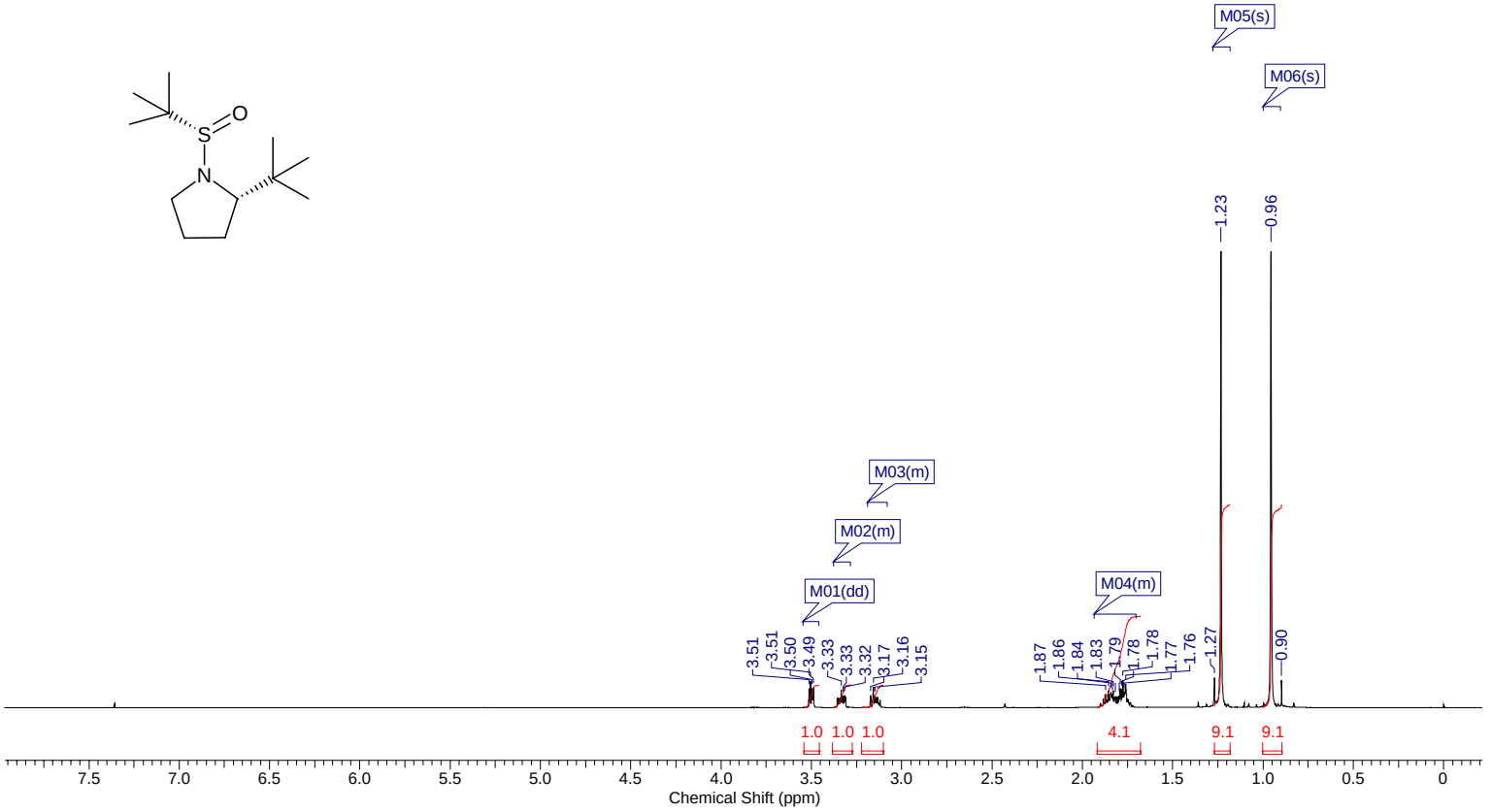


5/14/2009 3:46:41 PM

Acquisition Time (sec)	3.1719	***Comment***	3140-85-F PROTON16 CDCl3 u leletra1 8			***Date***	13 May 2009 19:16:16
Date Stamp	13 May 2009 19:16:16			***File Name***	\\phuseh-S1603\LELETRA1.:.DATA\NMR data\3140-85-F_010001r		
Frequency (MHz)	500.63	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect
Original Points Count	32768	***Owner***	drinkdo1	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	20.20	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	3127.6113	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	27.000		

1H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 0.96 (s, 9 H) 1.25 (s, 9 H) 1.70 - 1.93 (m, 4 H) 3.08 - 3.19 (m, 1 H) 3.28 - 3.38 (m, 1 H) 3.50 (dd, *J*=8.04, 4.26 Hz, 1 H)

3140-85-F_010001r VerticalScaleFactor = 1

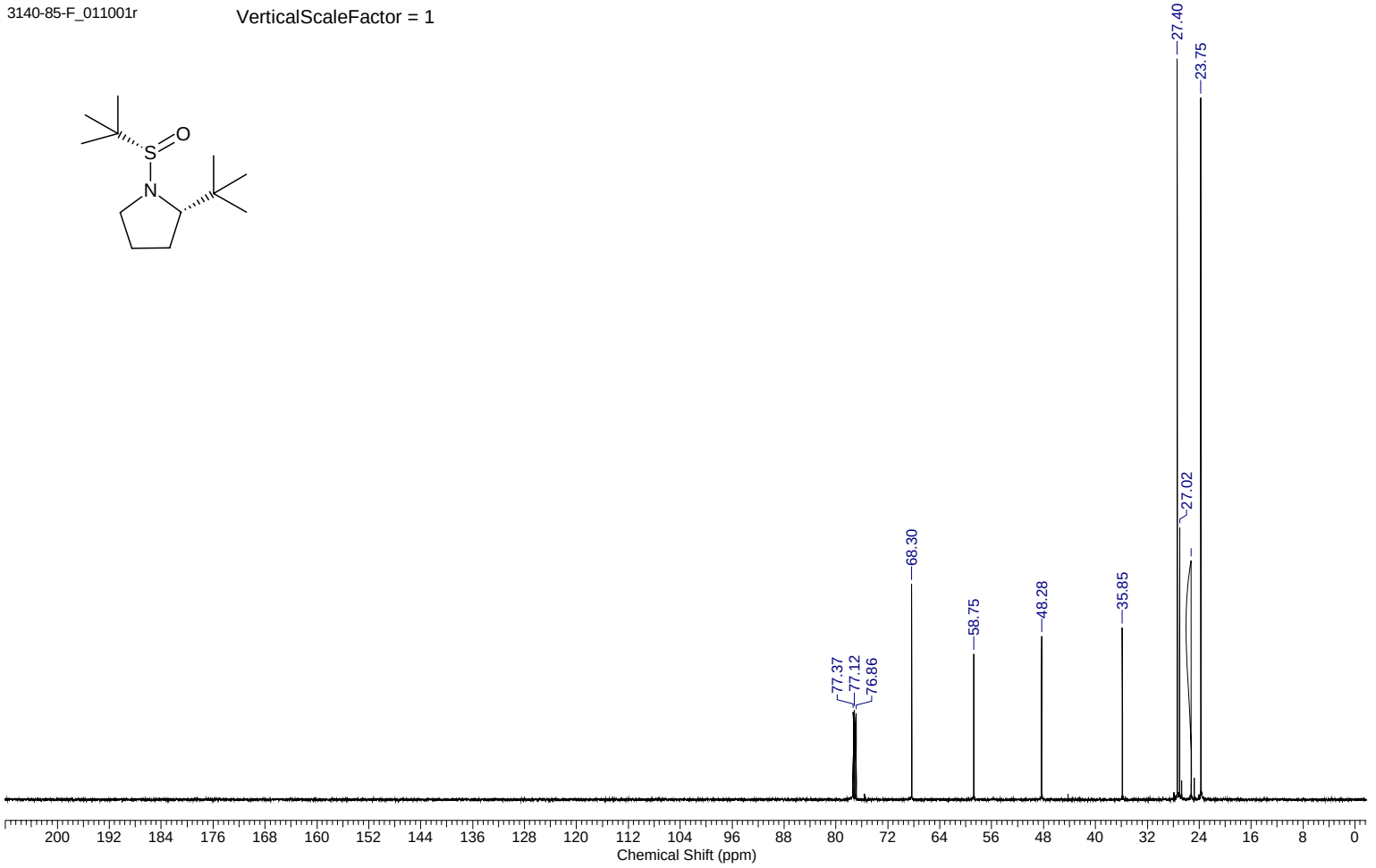


5/14/2009 3:51:36 PM

Acquisition Time (sec)	1.0912	***Comment***	3140-85-F C13CPD256 CDCl3 u leletra1 8			***Date***	13 May 2009 19:31:12
Date Stamp	13 May 2009 19:31:12			***File Name***	\\phuseh-S1603\LELETRA1 ·: DATA\NMR data\3140-85-F_011001r		
Frequency (MHz)	125.88	***Nucleus***	13C	***Number of Transients***	256	***Origin***	spect
Original Points Count	32768	***Owner***	drinkdo1	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	8192.00	***SW(cyclical) (Hz)***	30030.03	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	12587.9785	***Sweep Width (Hz)***	30029.11	***Temperature (degree C)***	27.000		

3140-85-F_011001r

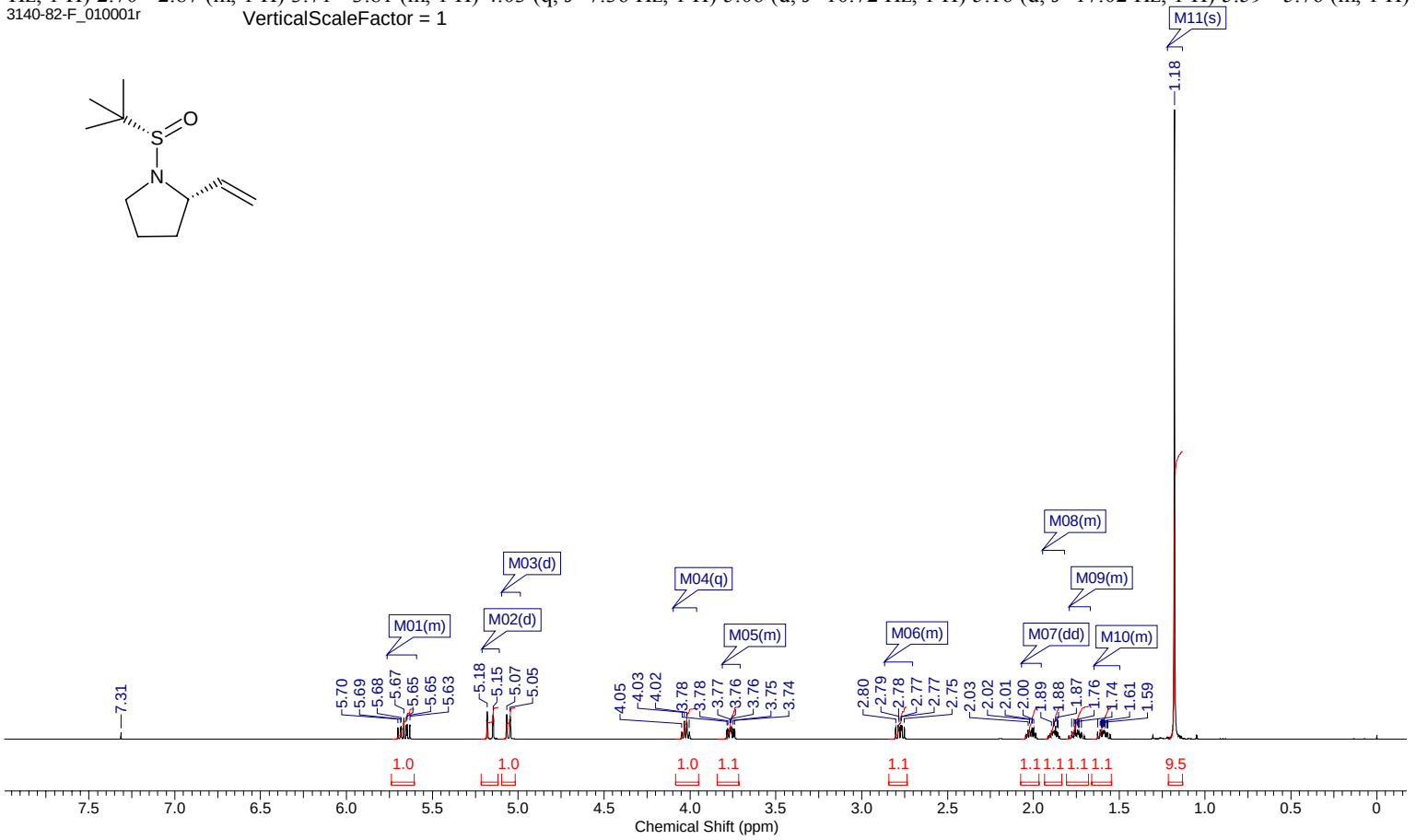
VerticalScaleFactor = 1



5/14/2009 3:57:25 PM

Acquisition Time (sec)	3.1719	***Comment***	3140-82-F PROTON16 CDCl3 u leletra1 30			***Date***	11 May 2009 19:14:08
Date Stamp	11 May 2009 19:14:08			***File Name***	\\phuseh-S1603\LELETRA1.:.DATA\NMR data\3140-82-F_010001r		
Frequency (MHz)	500.63	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect
Original Points Count	32768	***Owner***	drinkdo1	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	35.90	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	3105.2930	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	27.000		

1H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.18 (s, 9 H) 1.50 - 1.65 (m, 1 H) 1.67 - 1.79 (m, 1 H) 1.82 - 1.95 (m, 1 H) 2.02 (dd, *J*=11.98, 4.10 Hz, 1 H) 2.70 - 2.87 (m, 1 H) 3.71 - 3.81 (m, 1 H) 4.03 (q, *J*=7.36 Hz, 1 H) 5.06 (d, *J*=10.72 Hz, 1 H) 5.16 (d, *J*=17.02 Hz, 1 H) 5.59 - 5.76 (m, 1 H)

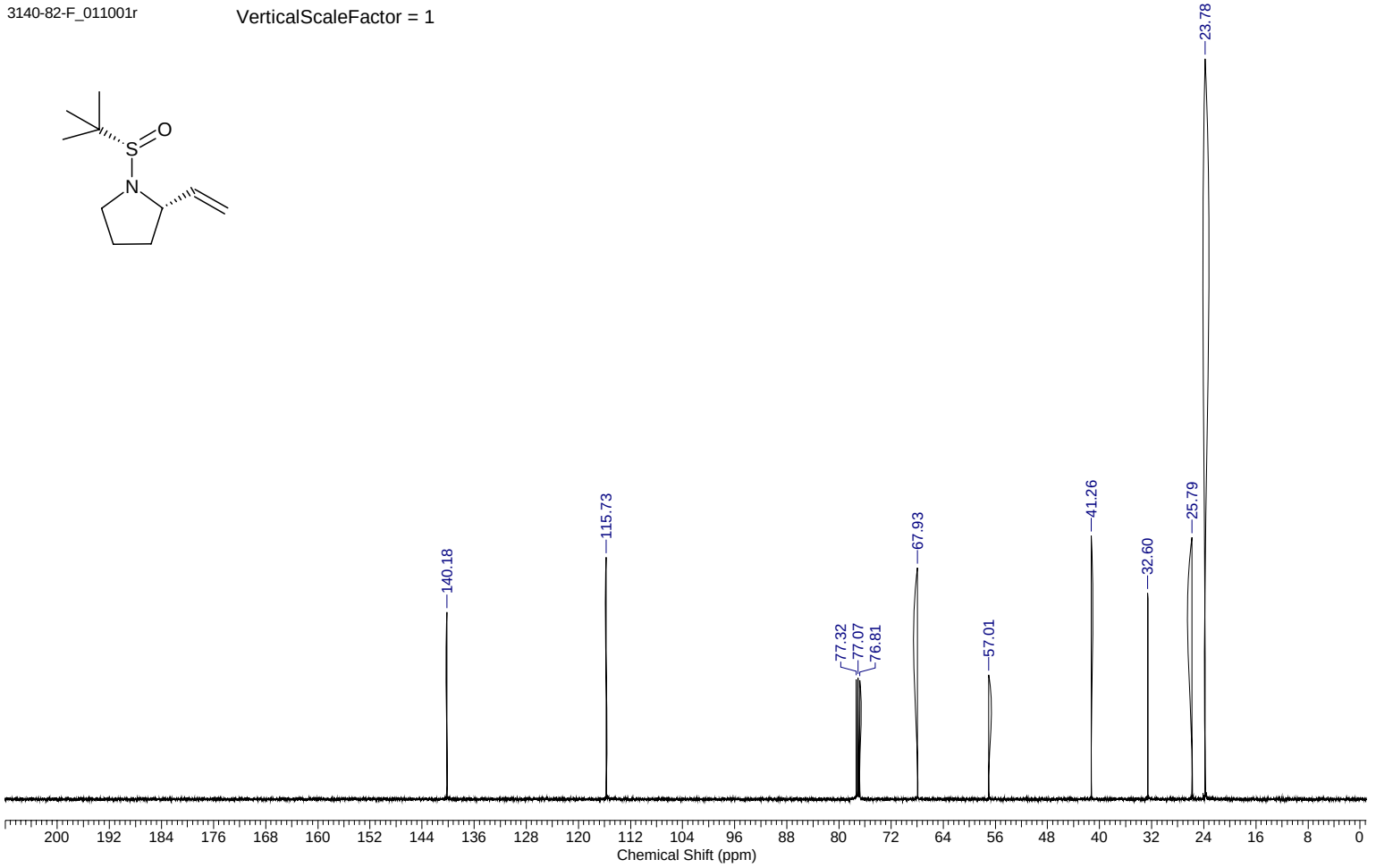


5/14/2009 4:01:45 PM

Acquisition Time (sec)	1.0912	***Comment***	3140-82-F C13CPD256 CDCl3 u leletra1 30			***Date***	11 May 2009 19:26:56
Date Stamp	11 May 2009 19:26:56			***File Name***	\\phuseh-S1603\LELETRA1_:DATA\NMR data\3140-82-F_011001r		
Frequency (MHz)	125.88	***Nucleus***	13C	***Number of Transients***	256	***Origin***	spect
Original Points Count	32768	***Owner***	drinkdo1	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	8192.00	***SW(cyclical) (Hz)***	30030.03	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	12587.9785	***Sweep Width (Hz)***	30029.11	***Temperature (degree C)***	27.000		

3140-82-F_011001r

VerticalScaleFactor = 1

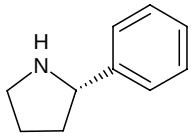


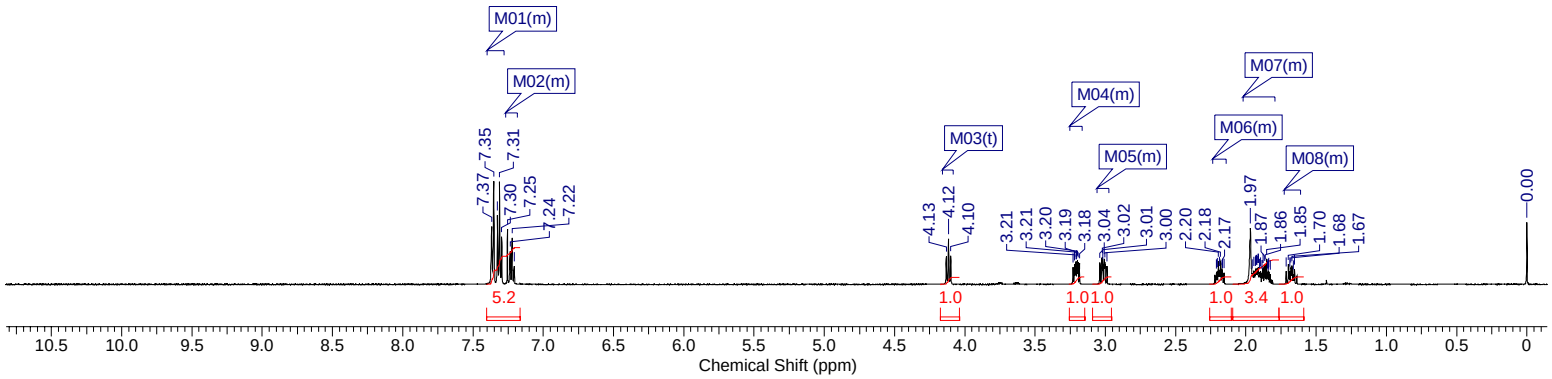
5/14/2009 1:56:05 PM

Acquisition Time (sec)	3.1719	***Comment***	3140-84-f PROTON16 CDCl3 u leletra1 10			***Date***	14 May 2009 13:07:12
Date Stamp	14 May 2009 13:07:12			***File Name***	\\phuseh-S1603\LELETRA1 :. DATA\NMR data\3140-84-F_012001r		
Frequency (MHz)	500.63	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect
Original Points Count	32768	***Owner***	drinkdo1	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	256.00	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	3075.9272	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	27.000		

1H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.61 - 1.73 (m, 1 H) 1.79 - 2.02 (m, 3 H) 2.14 - 2.24 (m, 1 H) 2.98 - 3.06 (m, 1 H) 3.17 - 3.25 (m, 1 H) 4.12 (t, *J*=7.72 Hz, 1 H) 7.18 - 7.27 (m, 2 H) 7.28 - 7.40 (m, 3 H)

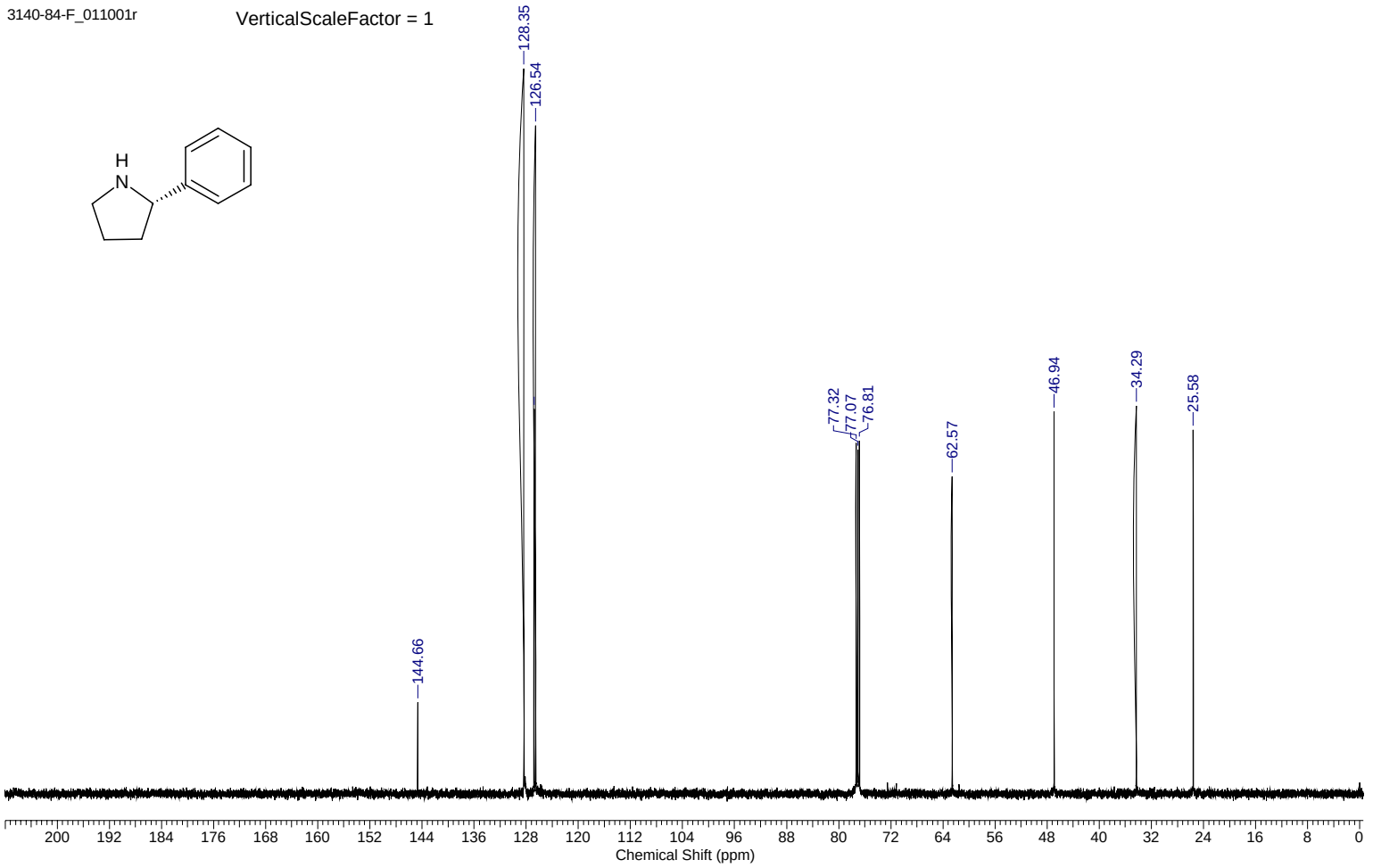
3140-84-F_012001r VerticalScaleFactor = 1





5/14/2009 2:03:50 PM

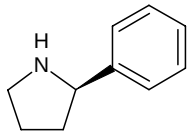
Acquisition Time (sec)	1.0912	***Comment***	3140-84-F C13CPD256 CDCl3 u leletra1 59			***Date***	13 May 2009 16:29:52
Date Stamp	13 May 2009 16:29:52			***File Name***	\\phuseh-S1603\LELETRA1 ;-.DATA\NMR data\3140-84-F_011001r		
Frequency (MHz)	125.88	***Nucleus***	13C	***Number of Transients***	256	***Origin***	spect
Original Points Count	32768	***Owner***	drinkdo1	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	14596.50	***SW(cyclical) (Hz)***	30030.03	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	12587.9785	***Sweep Width (Hz)***	30029.11	***Temperature (degree C)***	27.000		

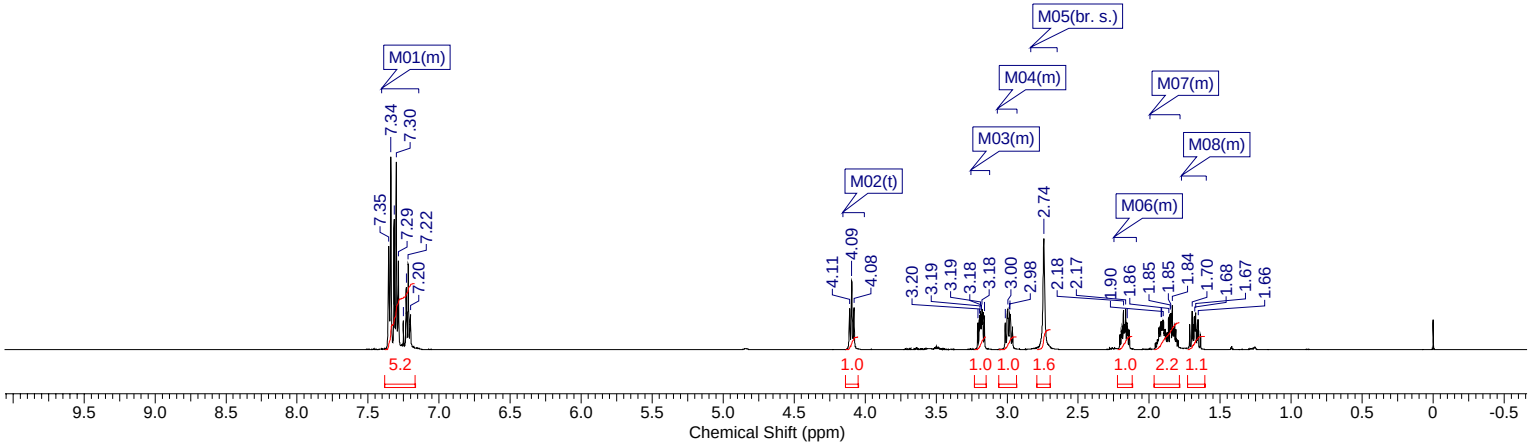


Acquisition Time (sec)	3.1719	***Comment***	3140-84-R PROTON16 CDCl3 u leletra1 45			***Date***	24 Jul 2009 18:27:12
Date Stamp	24 Jul 2009 18:27:12			***File Name***	\\phuseh-S1603\LELETRA1.:.DATA\NMR data\3140-84-R_010001r		
Frequency (MHz)	500.63	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect
Original Points Count	32768	***Owner***	nmrsu	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	50.80	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	3073.5334	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	27.000		

1H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.68 (m) 1.88 (m) 2.17 (m) 2.74 (br. s.) 2.99 (m) 3.18 (m) 4.09 (t, *J*=7.88 Hz) 7.28 (m)

3140-84-R_010001r VerticalScaleFactor = 1

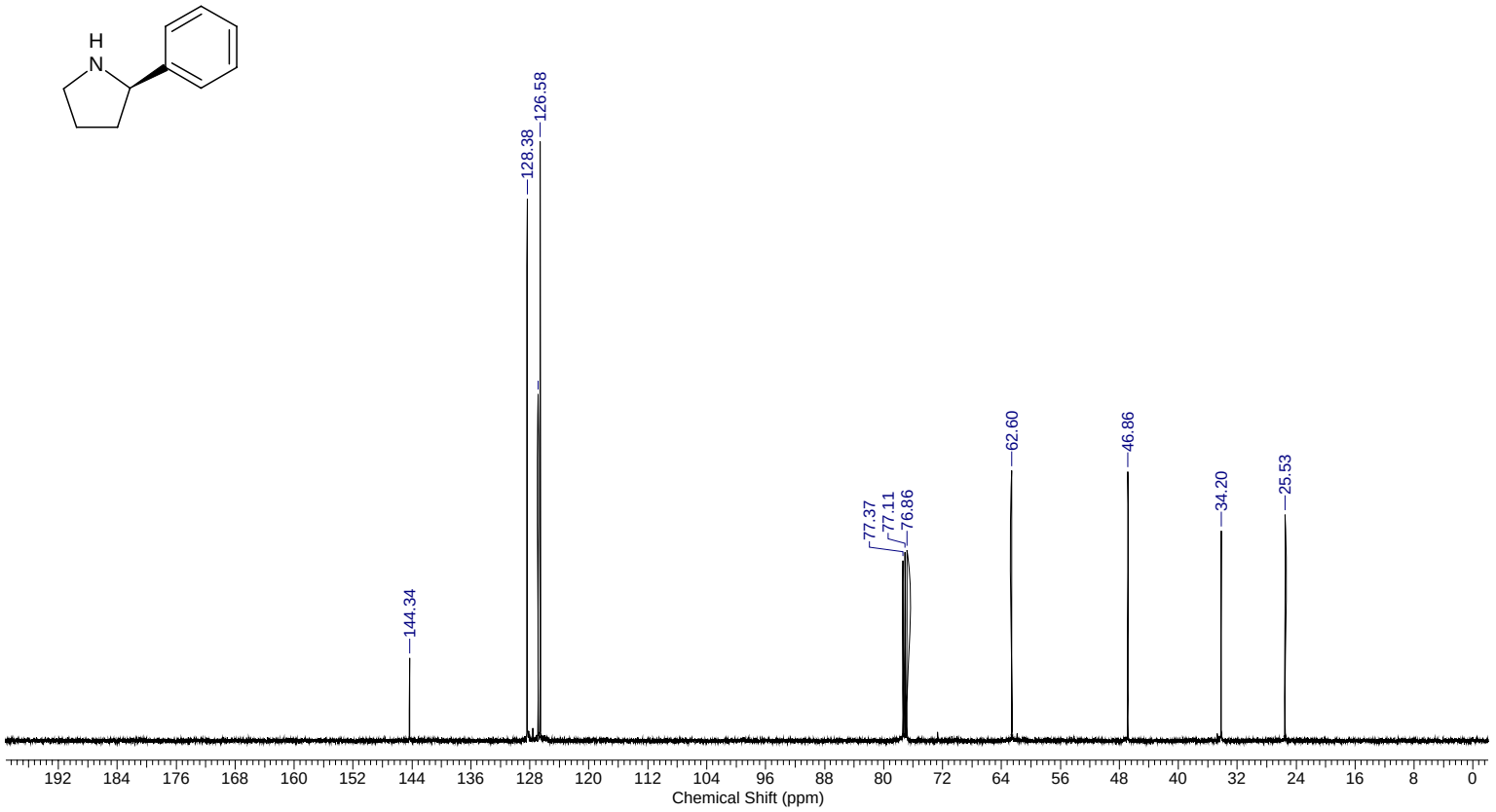




Acquisition Time (sec)	1.0912	***Comment***	3140-84-R C13CPD256 CDCl3 u leletra1 45			***Date***	24 Jul 2009 18:40:00
Date Stamp	24 Jul 2009 18:40:00			***File Name***	\\phuseh-S1603\LELETRA1.:.DATA\NMR data\3140-84-R_011001r		
Frequency (MHz)	125.88	***Nucleus***	13C	***Number of Transients***	256	***Origin***	spect
Original Points Count	32768	***Owner***	nmrsu	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	8192.00	***SW(cyclical) (Hz)***	30030.03	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	12587.9785	***Sweep Width (Hz)***	30029.11	***Temperature (degree C)***	27.000		

3140-84-R_011001r

VerticalScaleFactor = 1

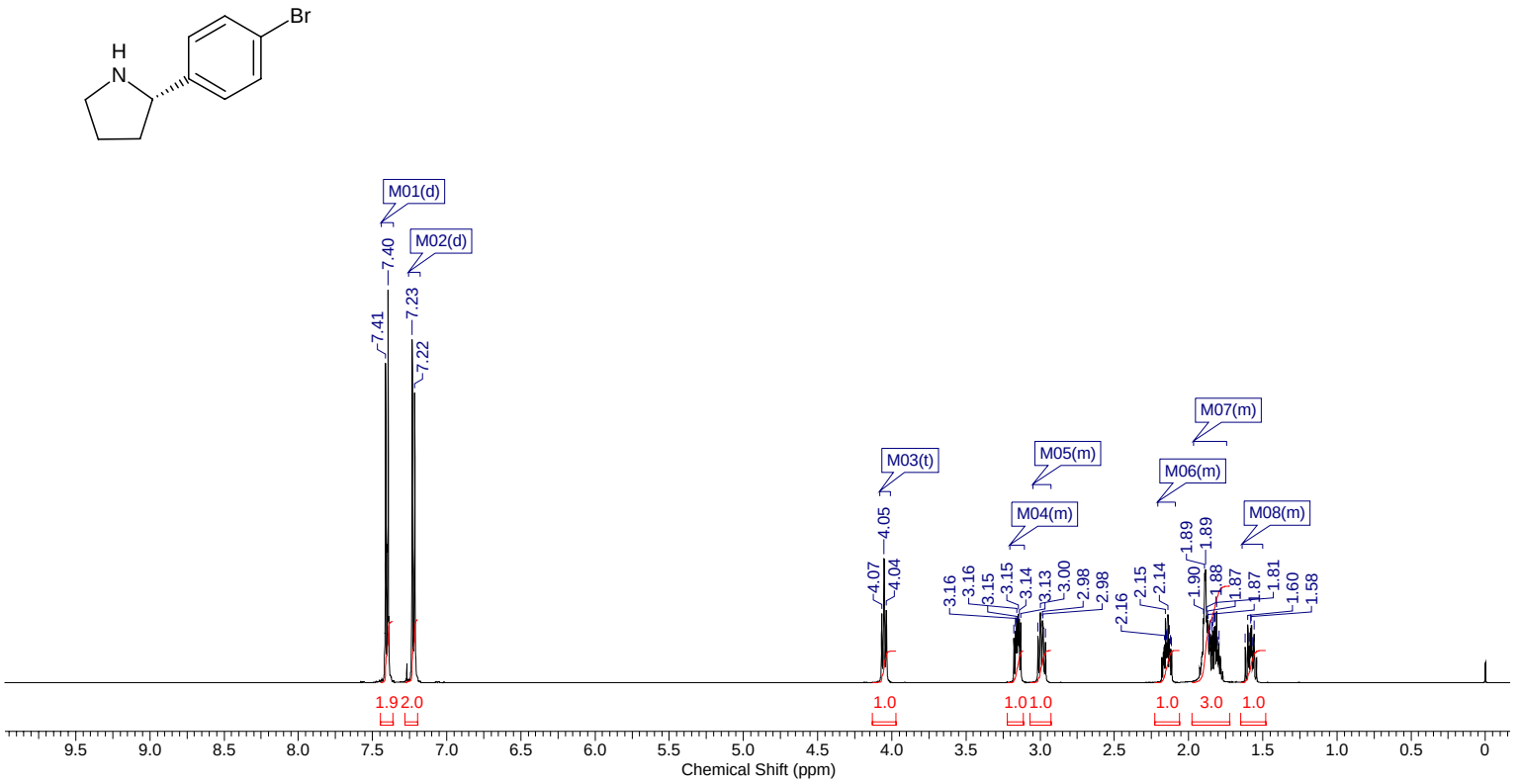


5/14/2009 2:16:12 PM

Acquisition Time (sec)	3.1719	***Comment***	3140-65-F PROTON16 CDCl3 u leletra1 44			***Date***	06 May 2009 18:48:32
Date Stamp	06 May 2009 18:48:32			***File Name***	\\phuseh-S1603\LELETRA1.:.DATA\NMR data\3140-65-F_010001r		
Frequency (MHz)	500.63	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect
Original Points Count	32768	***Owner***	drinkdo1	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	35.90	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	3082.2200	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	27.000		

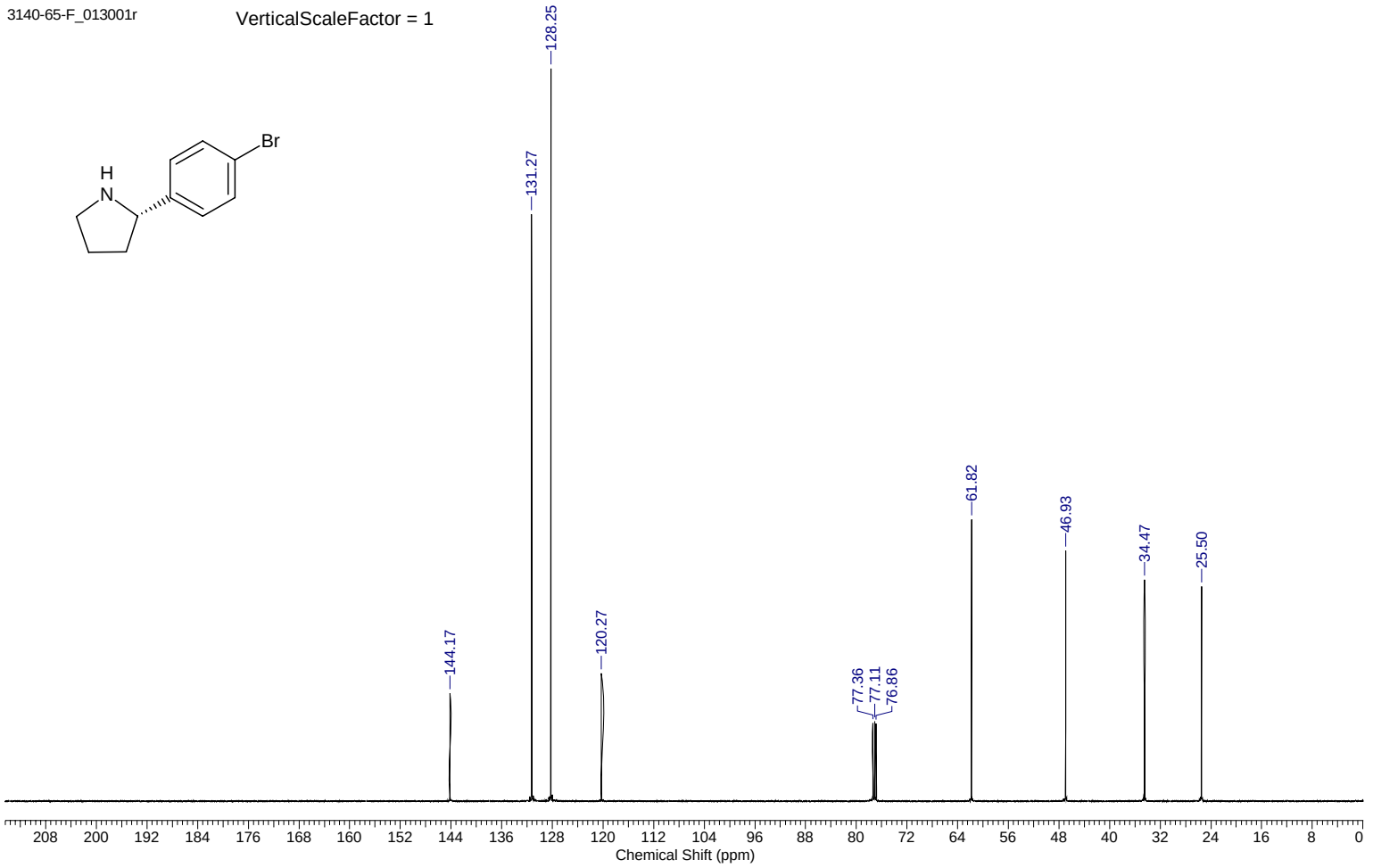
1H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.50 - 1.64 (m, 1 H) 1.74 - 1.97 (m, 3 H) 2.09 - 2.21 (m, 1 H) 2.93 - 3.05 (m, 1 H) 3.11 - 3.20 (m, 1 H) 4.05 (t, *J*=7.72 Hz, 1 H) 7.22 (d, *J*=8.20 Hz, 2 H) 7.40 (d, *J*=8.51 Hz, 2 H)

3140-65-F_010001r VerticalScaleFactor = 1



5/14/2009 2:21:19 PM

Acquisition Time (sec)	1.0912	***Comment***	3140-65-F C13CPD2048 CDCl3 u leletra1 44			***Date***	07 May 2009 04:09:36
Date Stamp	07 May 2009 04:09:36			***File Name***	\\phuseh-S1603\LELETRA1 ;·. DATA\NMR data\3140-65-F_013001r		
Frequency (MHz)	125.88	***Nucleus***	13C	***Number of Transients***	2048	***Origin***	spect
Original Points Count	32768	***Owner***	drinkdo1	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	8192.00	***SW(cyclical) (Hz)***	30030.03	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	14472.8584	***Sweep Width (Hz)***	30029.11	***Temperature (degree C)***	27.000		

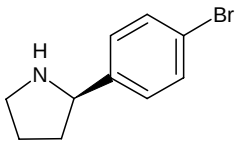


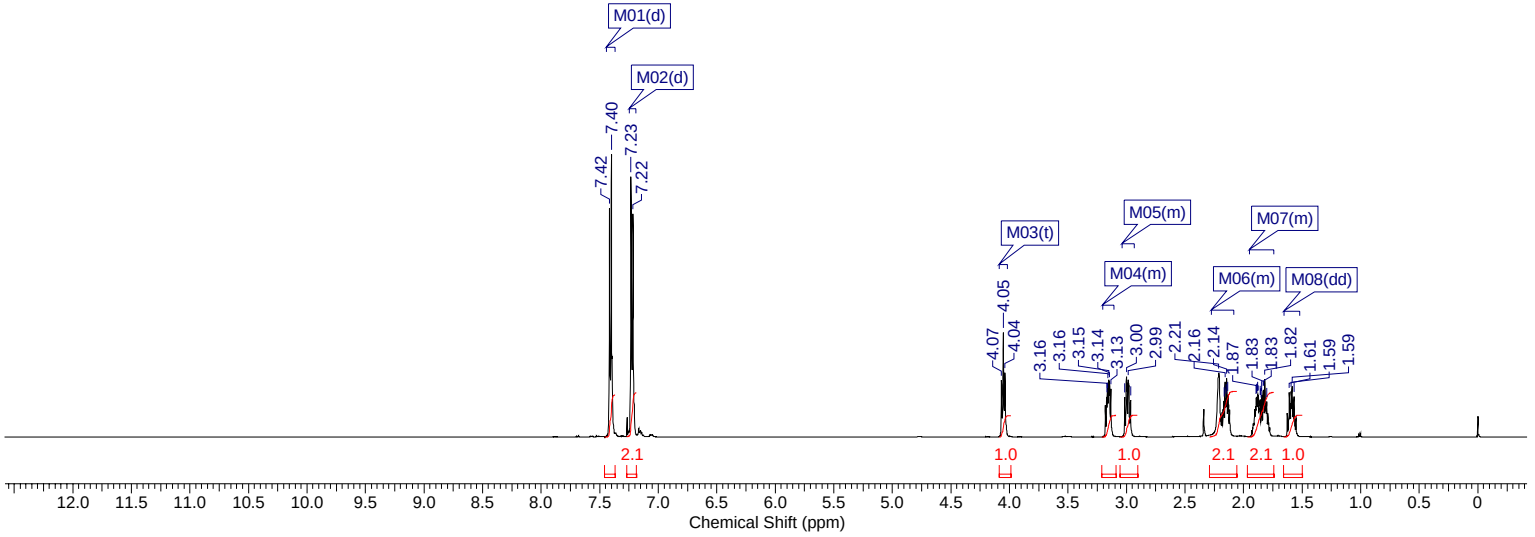
Acquisition Time (sec)	3.1719	***Comment***	3140-65-R PROTON16 CDCl3 u leletra1 44			***Date***	24 Jul 2009 18:05:52
Date Stamp	24 Jul 2009 18:05:52			***File Name***	\\phuseh-S1603\LELETRA1.:.DATA\NMR data\3140-65-R_010001r		
Frequency (MHz)	500.63	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect
Original Points Count	32768	***Owner***	nmrsu	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	45.30	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	3081.1982	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	27.000		

1H NMR (501 MHz, CHLOROFORM-*d*) δ ppm 1.59 (dd, *J*=12.30, 9.14 Hz) 1.85 (m) 2.16 (m) 2.99 (m) 3.15 (m) 4.05 (t, *J*=7.57 Hz) 7.22 (d, *J*=8.20 Hz) 7.41 (d, *J*=8.20 Hz)

VerticalScaleFactor = 1

3140-65-R_010001r





Acquisition Time (sec)	1.0912	***Comment***	3140-65-R C13CPD256 CDCl3 u leletra1 44			***Date***	24 Jul 2009 18:18:40
Date Stamp	24 Jul 2009 18:18:40			***File Name***	\\phuseh-S1603\LELETRA1.:.DATA\NMR data\3140-65-R_011001r		
Frequency (MHz)	125.88	***Nucleus***	13C	***Number of Transients***	256	***Origin***	spect
Original Points Count	32768	***Owner***	nmrsu	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	8192.00	***SW(cyclical) (Hz)***	30030.03	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	12587.9785	***Sweep Width (Hz)***	30029.11	***Temperature (degree C)***	27.000		

3140-65-R_011001r

VerticalScaleFactor = 1