Supporting Information

A simple and Efficient Method for Constructing Azepino[4,5-*b*]indole Derivatives via Acid Catalysis

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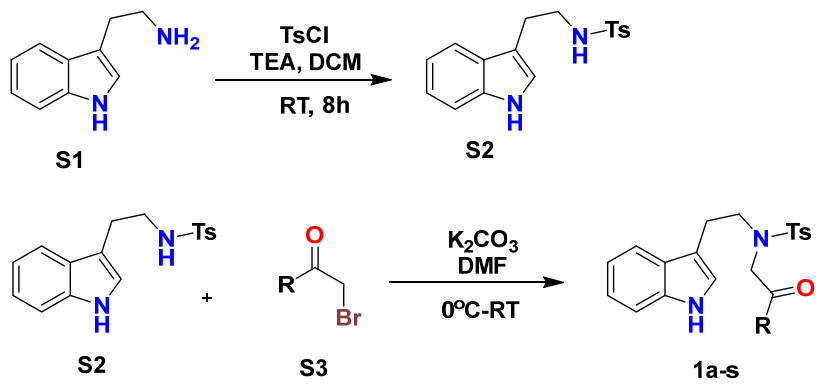
General information:

All chemicals were purchased form best grade in commercially available and are used without further purification. All solvents were purchased as HPLC grade and used without any purification or distillation. Analytical thin layer chromatography was performed on aluminium plate coated with silica gel (Merck). Gravity column chromatography was performed using 100-200 mesh silica gel and mixtures of hexane-ethyl acetate were used for elution. Visualization was accomplished using ultraviolet light (254 nm) and chemical staining with acidic potassium permanganate solution and Iodine.

Melting points was determined using "Mel-Temp" melting point apparatus and were uncorrected. Proton nuclear magnetic resonance (^{1}H NMR) and Carbon nuclear magnetic resonance (^{13}C NMR) spectra's were recorded using a Varian (400 MHz) and Oxford (400 MHz) spectrometer. All spectra were recorded at ambient temperature (298 K). Chemical shifts (δ) are quoted in ppm relative to residual solvent ($CDCl_3$: δ = 7.26 ppm for ^{1}H and δ = 77.0 for 13C). Coupling constants (*J*) are corrected and quoted to the nearest 0.1 Hz. The following abbreviations are used to indicate the multiplicity of the signals: s = singlet; d = doublet; t = triplet; q = quartet; m = multiplet; bs = broad singlet; dt = doublet of triplet; td = triplet of doublet; ddd = doublet of doublet of doublet. High resolution mass spectra (HRMS) were measured on a Micromass Q-TOF spectrometer using EI (electron impact, 70 eV) at the *J*oint Center for High valued Instruments, National Sun-Yat Sen University, Kaohsiung, Taiwan.

General experiment procedure for synthesis of starting materials: (1a-s):[1-3]



To a solution of tryptamine (S1, 5 mmol) in DCM (50 mL) was added with triethyl amine (5 equiv) followed by tosyl chloride (1.5 equiv) at 0ºC. Then the reaction mixture was stirred at RT for 8 hr and reaction monitored by TLC. After reaction completed, reaction mass portioned between water and dichloromethane. The combined organic layer was washed with ammonium chloride solution and dried on sodium sulphate and evaporated under vacuum to obtain crude. The crude was purified through column chromatography (100-200 silica gel; EA/Hexane).

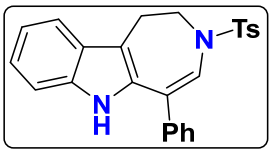
Then the obtained compound (**S2**; 2 mmol) in DMF (10 ml) was added to K_2CO_3 (1.5 equiv) at 0ºC and stirred at same temperature for 30 min. Then the respective 2-bromoacetophenones (**S3**, 1.2 equiv) were added and slowly heated to RT and stirred till the completion of starting materials (~30 min to 2 hr). After reaction completed (monitored by TLC), reaction mass quenched with ice-cold water and extracted into EtOAc. The combined organic layer was washed with ice-cold water and sat. NH_4Cl. Then it was dried and evaporated to give crude residue. The obtained crude material passed through flash column chromatography to give pure product in high yields (**1a-s**).

General Experiment Procedure for Compound 2a-s:

An oven-dried 50 mL RB flask charged with **1** (0.5 mmol) in 1,2-Dichloroethane (0.15 M) was added with TfOH (0.2 equiv), and stirred at respective temperature for given time. After reaction completed (monitored by TLC), reaction mass was partitioned between water and Dichloromethane, then the combined organic phases washed with brine solution, dried on sodium sulphate and evaporated under vacuum. The resulting crude material was purified by flash column chromatography on silica gel (100-200 mesh) with suitable ratio of hexane and ethyl acetate to afford the desired product (**3**). The identity and purity of the compounds were determined by ^{1}HNMR, ^{13}CNMR, and HRMS data.

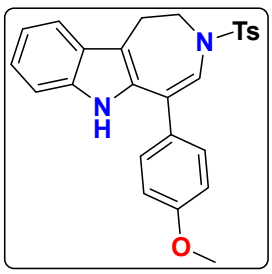
Spectral Characterization Data for Final Compounds (2a-v):

5-Phenyl-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-*b*]indole (2a): Yield: 96 %; yellow solid; m.p. 192-194 °C; ^{1}H

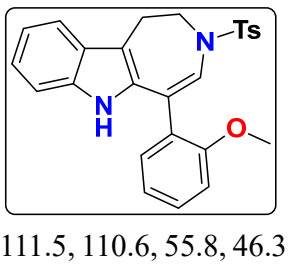


NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.4 Hz, 2H), 7.54 (s, 1H), 7.49-7.40(m, 6H), 7.29 (d, *J* = 8.4 Hz, 2H), 7.16-7.03 (m, 3H), 6.88 (s, 1H), 3.88 (t, *J* = 4.4 Hz, 2H), 3.04 (t, *J* = 4.4 Hz, 2H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.0, 138.6, 136.1, 134.6, 130.6, 129.9, 129.7, 128.9, 128.4, 128.0, 126.9, 126.6, 122.3, 119.6, 118.1, 117.9, 113.6, 110.6, 46.3, 26.4, 21.5; HRMS-ESI (m/z): calcd for $C_{25}H_{23}N_2O_2S$ $[M+H]^+$: 415.1402 found 415.1474.

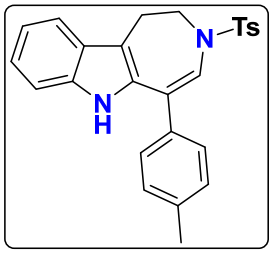
5-(4-Methoxyphenyl)-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-*b*]indole (2b): Yield: 92 %; brown solid; m.p. 184-186 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.4 Hz, 2H), 7.54 (s, 1H), 7.43 (d, *J* = 7.6 Hz, 1H), 7.34 (d, *J* = 8.4 Hz, 2H), 7.29 (d, *J* = 8.4 Hz, 2H), 7.16-6.97 (m, 5H), 6.84 (s, 1H), 3.88 (t, *J* = 3.2 Hz, 5H), 3.04 (t, *J* = 5.2 Hz, 2H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.5, 144.0, 136.3, 134.7, 131.14, 131.01, 130.86, 130.05, 128.59, 127.06, 126.41, 122.45, 119.7, 118.0, 117.9, 114.4, 113.6, 110.7, 55.5, 46.4, 29.7, 26.5, 21.6; HRMS-ESI (m/z): calcd for $C_{26}H_{25}N_2O_3S$ $[M+H]^+$: 445.1508 found 445.1590.



5-(2-Methoxyphenyl)-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-*b*]indole (2c) : Yield: 75%; yellow solid; m.p. 125-127 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.0 Hz, 2H), 7.45-7.39 (m, 3H), 7.30-7.24 (m, 3H), 7.13-7.01 (m, 5H), 6.83 (s, 1H), 3.92 (t, *J* = 5.2 Hz, 2H), 3.73 (s, 3H), 3.02(t, *J* = 5.2 Hz, 2H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.0, 143.9, 136.7, 134.8, 132.4, 131.5, 129.9, 128.7, 127.1, 122.14, 122.12, 119.5, 118.0, 114.8, 112.8, 111.5, 110.6, 55.8, 46.3, 26.4, 21.6; HRMS-ESI (m/z): calcd for $C_{26}H_{25}N_2O_3S$ $[M+H]^+$: 445.1508 found 445.1566.

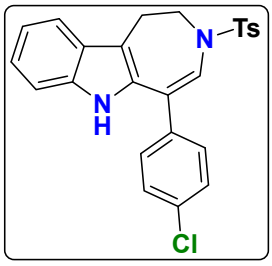


5-(*p*-Ttolyl)-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-*b*]indole (**2d**): Yield: 95%; yellow solid; m.p. 159-161 °C;



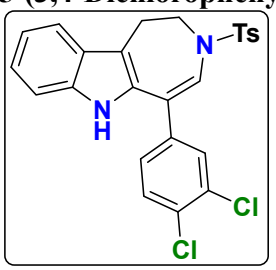
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.4 Hz, 2H), 7.60 (s, 1H), 7.46 (d, *J* = 7.6 Hz, 1H), 7.35-7.26 (m, 6H), 7.18-7.06 (m, 3H), 6.89 (s, 1H), 3.90 (t, *J* = 4.8 Hz, 2H), 3.07 (t, *J* = 4.8 Hz, 2H), 2.46 (s, 3H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.9, 137.7, 136.2, 135.5, 134.6, 130.8, 129.9, 129.6, 129.5, 128.4, 126.9, 126.3, 122.3, 119.5, 118.1, 117.9, 113.5, 110.5, 46.3, 26.4, 21.5, 21.1 HRMS-ESI (m/z): calcd for $C_{26}H_{25}N_2O_2S$ $[M+H]^+$: 429.1558 found 429.1624.

5-(4-Chlorophenyl)-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-*b*]indole (2e) : Yield: 89%; yellow solid; m.p. 161-



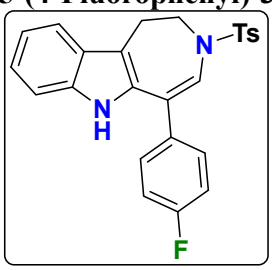
163 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.4 Hz, 2H), 7.48-7.43 (m, 4H), 7.38 (d, *J* = 4Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.17 (d, *J* = 8.0 Hz, 1H), 7.12 (t, *J* = 8.0 Hz, 1H), 7.07 (t, *J* = 8.0 Hz, 1H), 6.86 (s, 1H), 3.87 (t, *J* = 4.0 Hz, 2H), 3.04 (t, *J* = 4.0 Hz, 2H), 2.40 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.2, 137.1, 136.1, 134.8, 131.1, 130.3, 130.1, 129.3, 128.4, 127.0, 126.8, 122.6, 119.8, 118.0, 116.8, 114.1, 110.7, 46.4, 26.5, 21.6; HRMS-ESI (m/z): calcd for $C_{25}H_{22}ClN_2O_2S$ $[M+H]^+$: 449.1012 found 449.1080.

5-(3,4-Dichlorophenyl)-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-*b*]indole (2f) : Yield: 82 %; brown solid; m.p.



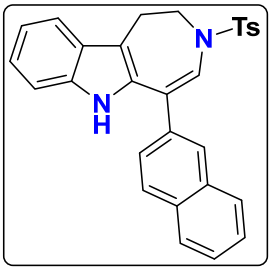
210-212 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.8 Hz, 2H), 7.55 (dd, *J* =6.4, 4.0 Hz, 2H), 7.44 (d, *J* = 7.6 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.30-7.26 (m, 1H), 7.21 (d, *J* = 7.6 Hz, 1H), 7.14 (t, *J* = 6.8 Hz, 1H), 7.08 (t, *J* = 8.0 Hz, 1H), 6.89 (s, 1H), 3.86 (t, *J* = 4.8 Hz, 2H), 3.03 (t, *J* = 4.8 Hz, 2H), 2.42 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.3, 138.7, 135.9, 134.9, 133.2, 132.4, 131.6, 131.0, 130.1, 129.8, 129.1, 128.4, 127.16, 127.11, 122.8, 120.0, 118.0, 115.5, 114.4, 110.8, 46.5, 26.4, 21.6; HRMS-ESI (m/z): calcd for $C_{25}H_{21}Cl_2N_2O_2S$ $[M+H]^+$: 483.0623 found 483.0679.

5-(4-Fluorophenyl)-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-*b*]indole (2g) : Yield: 80 %; brown solid; m.p. 158-



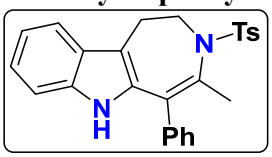
160 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.4 Hz, 2H), 7.45-7.38 (m, 4H), 7.30 (d, *J* = 8.0 Hz, 2H), 7.25(s, 1H), 7.18-7.04 (m, 5H), 6.85 (s, 1H), 3.87 (t, *J* = 4.4 Hz, 2H), 3.04 (t, *J* = 4.4 Hz, 2H), 2.40 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.5 (J_F = 245.8 Hz),144.1, 136.1, 134.7, 134.5, 134.4, 131.4 (d, J_F = 10.0 Hz), 130.5, 130.0, 128.4, 126.9, 126.6, 122.5, 119.7, 117.9, 116.9, 116.4 (d, J_F = 20.0 Hz), 113.8, 110.6, 46.3, 26.4, 21.5; HRMS-ESI (m/z): calcd for $C_{25}H_{21}FN_2O_2S$ $[M]^+$: 432.1308 found 432.1291.

5-(Naphthalen-2-yl)-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-b]indole (2h) : Yield: 75 %; yellow solid; m.p.



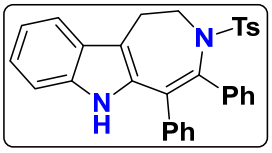
207-209 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97-7.89 (m, 4H), 7.77 (d, *J* = 8.4 Hz, 2H), 7.60-7.54 (m, 4H), 7.48 (d, *J* = 8.4 Hz, 1H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.15-7.07 (m, 3H) 7.01 (s, 1H), 3.94 (t, *J* = 8.0, 4.0 Hz, 2H), 3.11 (t, *J* = 8.0, 4.0 Hz, 2H), 2.42 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.04, 136.14, 135.98, 134.74, 133.50, 132.82, 130.73, 129.97, 128.62, 128.56, 128.40, 127.98, 127.73, 127.66, 126.98, 126.87, 126.66, 126.44, 122.42, 119.65, 118.10, 117.95, 113.81, 110.65, 46.47, 26.51, 21.52; HRMS-ESI (m/z): calcd for $C_{29}H_{25}N_2O2S$ $[M+H]^+$: 465.1558 found 465.1613.

4-Methyl-5-phenyl-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-*b*]indole (**2k**) : Yield: 57%; white solid; m.p. 201-



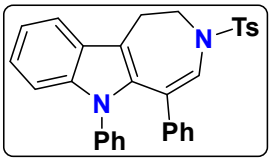
203 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 8.0 Hz, 2H), 7.49-7.42 (m, 3H), 7.37 (d, *J* = 8.0 Hz, 1H), 7.23 (d, *J* = 8.4 Hz, 2H), 7.10-7.00 (m, 3H), 6.85 (d, *J* = 8.0 Hz, 3H), 4.01 (s, 2H), 3.27 (t, *J* = 8.0 Hz, 2H), 2.05 (d, *J* = 8.0 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.0, 138.1, 136.1, 134.6, 131.4, 130.0, 129.6, 129.1, 128.6, 128.3, 126.6, 122.8, 119.4, 118.6, 113.9, 110.3, 49.7, 26.4, 21.9, 21.2; HRMS-ESI (m/z): calcd for $C_{26}H_{24}NaN_2O_2S$ $[M+Na]^+$: 451.1558 found 451.1450.

4,5-Diphenyl-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-*b*]indole (**2l**): Yield: 30%; yellow solid; m.p. 274-276 °C;



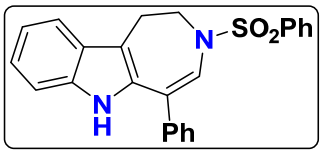
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 8.0 Hz, 1H), 7.38 (s, 1H), 7.30-7.24 (m, 4H), 7.21-7.13 (m, 4H), 7.13-7.09 (m, 1H), 7.02-6.97 (m, 3H), 6.94-6.89 (m, 4H), 4.13 (s, 2H), 3.53 (s, 2H), 2.23 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.6, 137.9, 137.7, 137.5, 135.0, 130.9, 130.6, 130.5, 128.9, 128.8, 128.69, 128.67, 127.6, 127.1, 127.0, 126.9, 123.1, 119.6, 118.9, 114.4, 110.5, 49.9, 29.7, 28.5, 21.3; HRMS-ESI (m/z): calcd for $C_{31}H_{26}NaN_2O_2S$ $[M+Na]^+$: 513.1715 found 513.1096.

5,6-Diphenyl-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-*b*]indole (**2m**): Yield: 45%; yellow solid; m.p. 126-128 °C;



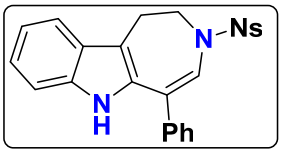
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.0 Hz, 2H), 7.54-7.52 (m, 1H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.16-7.00 (m, 7H), 6.95-6.90 (m, 5H), 6.82-6.80 (m, 2H), 4.06 (t, *J* = 4.8 Hz, 2H), 3.09 (t, *J* = 4.8 Hz, 2H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.0, 139.9, 138.7, 138.5, 135.5, 134.1, 129.8, 128.5, 128.4, 127.9, 127.6, 127.5, 127.2, 126.9, 126.4, 126.3, 122.7, 120.3, 119.6, 118.8, 117.5, 110.5, 53.2, 29.6, 24.1, 21.5; HRMS-ESI (m/z): calcd for $C_{31}H_{27}N_2O_2S$ $[M+H]^+$: 491.1715 found 491.1779.

5-Phenyl-3-(phenylsulfonyl)-1,2,3,6-tetrahydroazepino[4,5-*b*]indole (**2n**) : Yield: 79 %; yellow solid; m.p.



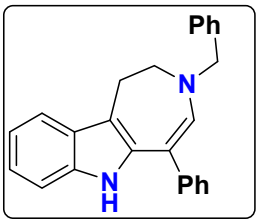
166-168 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89-7.86 (m, 2H), 7.60-7.42 (m, 10H), 7.18-7.05 (m, 3H), 6.89 (s, 1H), 3.92 (t, *J* = 4.4 Hz, 2H), 3.08 (t, *J* = 4.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.1, 138.5, 134.6, 133.0, 130.5, 129.7, 129.3, 129.0, 128.4, 128.0, 126.9, 126.4, 122.4, 119.6, 118.4, 117.9, 113.7, 110.6, 46.4, 26.5; HRMS-ESI (m/z): calcd for $C_{24}H_{21}N_2O_2S$ $[M+H]^+$: 401.1245 found 401.1313.

3-((2-Nitrophenyl)sulfonyl)-5-phenyl-1,2,3,6-tetrahydroazepino[4,5-*b*]indole (2o): Yield: 75%; red solid; m.p.

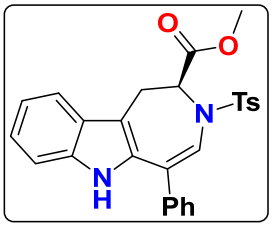


205-207 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07-8.05 (m, 1H), 7.73-7.65 (m, 3H), 7.60 (s, 1H), 7.52-7.43 (m, 6H), 7.19-7.06 (m, 3H), 6.81 (s, 1H), 4.26 (t, *J* = 4.8 Hz, 2H), 3.19(t, *J* = 5.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.1, 138.2, 134.7, 134.0, 132.1, 131.9, 130.6, 130.3, 129.6, 129.7, 129.0, 128.3, 128.2, 125.8, 124.5, 122.7, 119.76, 119.74, 118.1, 113.8, 110.7, 46.8, 26.7; HRMS-ESI (m/z): calcd for $C_{24}H_{19}N_3O_4S$ $[M+H]^+$: 446.1096 found 446.1174.

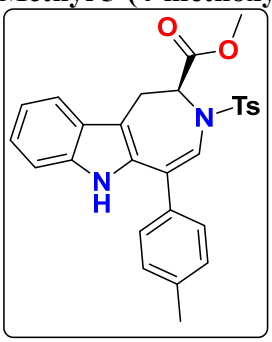
3-benzyl-5-phenyl-1,2,3,6-tetrahydroazepino[4,5-b]indole (**2p**): Yield: 30%; brown oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.61 (bs, 1H), 7.44-7.33 (m, 11H), 7.32-7.24 (m, 1H), 7.04-7.00 (m, 2H), 6.40 (s, 2H), 3.43 (t, *J* = 8.4 Hz, 2H), 3.09-3.05 (m, 2H); HRMS-ESI (m/z): calcd for $C_{25}H_{22}N_2$ $[M]^+$: 350.1783 found 350.1777. (Note: Compound started decomposing over a period of time; not enough stable to record ^{13}CNMR)



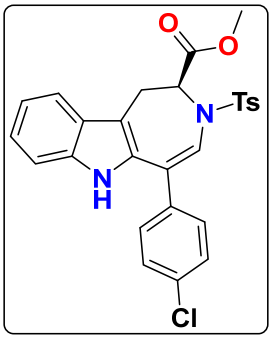
Methyl 5-phenyl-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-*b*]indole-2-carboxylate (**2q**): Yield: 82%; yellow solid; m.p. 88-90 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 8.4 Hz, 2H), 7.55 (s, 1H), 7.49-7.40 (m, 6H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.15-7.05 (m, 3H), 6.99 (d, *J* = 0.8 Hz, 1H), 5.82 (t, *J* = 4.8 Hz, 1H), 3.93 (dd, *J* = 16.0, 4.8 Hz, 1H), 3.28 (s, 3H), 2.81 (dd, *J* = 16.0, 4.8 Hz, 1H), 2.42 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.9, 144.3, 138.6, 135.5, 135.0, 131.1, 129.8, 129.6, 128.9, 128.3, 127.9, 127.2, 124.3, 122.3, 119.6, 117.6, 116.4, 110.6, 57.6, 52.0, 28.2, 21.5; HRMS-ESI (m/z): calcd for $C_{27}H_{25}N_2O_4S$ $[M+H]^+$: 473.1457 found 473.1530.

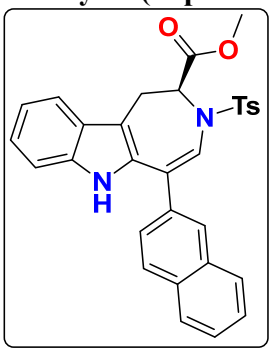


Methyl 5-(4-methoxyphenyl)-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-*b*]indole-2-carboxylate (**2r**): Yield: 89%; yellow solid; m.p. 88-90 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.4 Hz, 2H), 7.51 (s, 1H), 7.46 (d, *J* = 6.4 Hz, 1H), 7.34-7.29 (m, 4H), 7.14-7.03 (m, 3H), 6.98-6.92 (m, 3H), 5.56 (t, *J* = 3.6 Hz, 1H), 3.90 (dd, *J* = 16.0, 4.8 Hz, 1H), 3.87 (s, 3H), 3.27 (s, 3H), 2.78 (dd, *J* = 16.0, 4.8 Hz, 1H), 2.40 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.0, 159.4, 144.2, 135.6, 134.9, 131.5, 130.87, 130.82, 129.8, 128.4, 127.2, 123.9, 122.2, 119.6, 117.6, 116.1, 114.2, 110.5, 110.4, 57.6, 55.3, 52.0, 28.2, 21.5; HRMS-ESI (m/z): calcd for $C_{28}H_{26}NaN_2O_5S$ $[M+Na]^+$: 525.1562 found 525.1458.



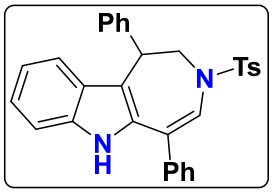
Methyl 5-(4-chlorophenyl)-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-*b*]indole-2-carboxylate (**2s**): Yield: 69%; yellow solid; m.p. 94-96 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 8.4 Hz, 2H), 7.47-7.40 (m, 4H), 7.36-7.31 (m, 4H), 7.15-7.04 (m, 3H), 6.96 (d, *J* = 0.8 Hz, 1H), 5.56 (t, *J* = 3.2 Hz, 1H), 3.91 (dd, *J* = 16.0, 4.8 Hz, 1H) 3.25 (s, 3H), 2.77 (dd, *J* = 16.0, 4.8 Hz, 1H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.8, 144.4, 137.1, 135.4, 135.1, 133.9, 130.9, 130.8, 129.9, 129.1, 128.3, 127.3, 124.5, 122.5, 119.8, 117.6, 114.9, 110.8, 110.6, 57.6, 52.1, 28.2, 21.6; HRMS-ESI (m/z): calcd for $C_{27}H_{24}ClN_2O_4S$ $[M+H]^+$: 507.1067 found 507.1154.



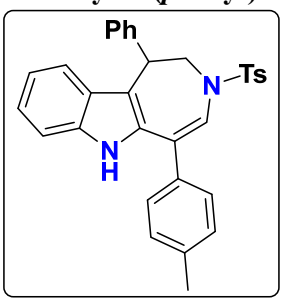
Methyl 5-(naphthalen-2-yl)-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-*b*]indole-2-carboxylate (**2t**): Yield: 86%; 

yellow solid; m.p. 116-118 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91-7.86 (m, 4H), 7.72-7.74 (m, 2H), 7.56-7.47 (m, 5H), 7.31 (d, *J* = 8.4 Hz, 2H), 7.10-7.05 (m, 4H), 5.61-5.58 (m, 1H), 3.94 (dd, *J* = 16.0, 4.8 Hz, 1H), 3.29 (s, 3H), 2.82 (dd, *J* = 16.0, 4.8 Hz, 1H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.9, 144.3, 136.0, 135.5, 135.1, 133.5, 132.8, 131.3, 129.9, 128.5, 128.4, 128.3, 128.0, 127.7, 127.5, 127.3, 126.6, 126.3, 124.6, 122.3, 119.7, 117.6, 116.3, 110.7, 110.6, 57.7, 52.1, 28.2, 21.5; HRMS-ESI (m/z): calcd for $C_{31}H_{26}NaN_2O_4S$ $[M+Na]^+$: 545.1613 found 545.1510.

1,5-diphenyl-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-*b*]indole (2u): Yield: 75%; white solid; m.p. 237-239 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (s, 1H), 7.55-7.46 (m, 7H), 7.27-7.14 (m, 8H), 7.07-7.03 (m, 1H), 6.79 (d, *J* = 8.0 Hz, 1H), 6.89-6.85 (m, 2H), 4.75 (dd, *J* = 6.4, 2.8 Hz, 1H), 4.20 (dd, *J* = 13.2, 6.4Hz, 1H), 3.85 (dd, *J* = 13.6, 2.8, Hz, 1H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.7, 141.6, 138.4, 135.9, 134.8, 131.2, 129.8, 129.7, 129.0, 128.48, 128.43, 128.3, 128.2, 128.0, 127.0, 126.7, 122.3, 119.5, 119.3, 118.2, 115.7, 110.5, 53.4, 44.4, 21.4; HRMS-ESI (m/z): calcd for $C_{31}H_{26}NaN_2O_2S$ $[M+Na]^+$: 513.1715 found 513.1611.

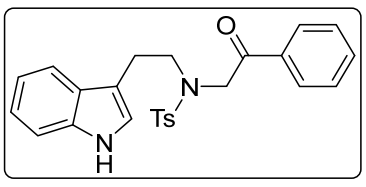


5-Phenyl-1-(*p*-tolyl)-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-*b*]indole (**2v**): Yield: 71%; white solid; m.p. 178-180 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (s, 1H), 7.54-7.46 (m, 7H), 7.20-7.13 (m, 3H), 7.07-6.97 (m, 6H), 6.89-6.85 (m, 2H), 4.71 (dd, *J* = 6.8, 2.8 Hz, 1H), 4.18 (dd, *J* = 6.8, 2.8 Hz, 1H), 3.84 (dd, *J* = 13.4, 2.8 Hz, 1H), 2.39 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.6, 138.6, 138.5, 136.1, 136.0, 134.8, 131.1, 129.79, 129.70, 129.1, 129.0, 128.4, 128.2, 128.1, 128.0, 127.0, 122.3, 119.5, 119.3, 118.2, 115.9, 110.4, 53.4, 44.0, 21.5, 21.1; HRMS-ESI (m/z): calcd for $C_{32}H_{28}NaN_2O_2S$ $[M+Na]^+$: 527.1871 found 527.1761.



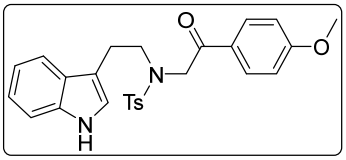
Spectral Characterization Data for Starting materials (1a-v):

N-(2-(1H-Indol-3-yl)ethyl)-4-methyl-N-(2-oxo-2-phenylethyl)benzenesulfonamide (1a): Yield: 90%; white solid; m.p. 139-141 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (s, 1H), 7.82-7.79 (m, 2H), 7.75 (d, *J* = 8.0 Hz, 2H), 7.58-7.54 (m, 1H), 7.48 (d, *J* = 7.6 Hz, 1H), 7.41 (t, *J* = 8.4 Hz, 2H), 7.28 (dd, *J* = 16.0, 8.4 Hz, 3H), 7.14 (t, *J* = 8.0 Hz, 1H), 7.05 (t, *J* = 8.0 Hz, 1H), 6.95 (s, 1H), 4.70 (s, 2H), 3.56 (t, *J* = 8.0 Hz, 2H), 3.00 (t, *J* = 8.0 Hz, 2H), 2.40 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.3, 143.4, 136.8, 136.2, 134.9, 133.7, 129.6, 128.5, 128.0, 127.2, 122.2, 122.1, 119.5, 118.7, 112.4, 56.6, 48.8, 24.8, 21.6; HRMS-ESI (m/z): calcd for



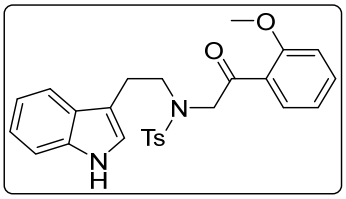
$C_{25}H_{24}NaN_2O_3S$ $[M+Na]^+$: 455.1508 found 455.1391.

N-(2-(1H-Indol-3-yl)ethyl)-N-(2-(4-methoxyphenyl)-2-oxoethyl)-4-methylbenzenesulfonamide (1b) :



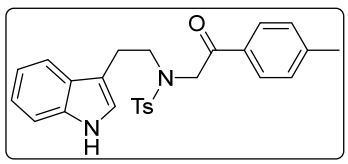
Yield: 92%; yellow solid; m.p. 175-177 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (s, 1H), 7.83 (d, *J* = 8.0 Hz, 2H), 7.75 (d, *J* = 8.0 Hz, 2H), 7.49 (d, *J* = 8.0 Hz, 2H), 7.28 (dd, *J* = 16.0, 8.0 Hz, 3H), 7.14 (t, *J* = 12.0, 8.0 Hz, 1H), 7.06 (t, *J* = 16.0, 8.0 Hz, 1H), 6.96 (s, 1H), 6.89 (d, *J* = 12.0 Hz, 2H), 4.64 (s, 2H), 3.85 (s, 3H), 3.54 (t, *J* = 16.0, 8.0 Hz, 2H), 2.99 (t, *J* = 16.0, 8.0 Hz, 2H), 2.40 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.7, 163.8, 143.3, 136.6, 136.1, 130.4, 129.5, 127.9, 127.5, 127.1, 122.09, 122.04, 119.4, 118.6, 113.9, 112.4, 111.0, 55.4, 53.3, 48.8, 24.7, 21.5; HRMS-ESI (m/z): calcd for $C_{26}H_{26}NaN_2O_4S$ $[M+Na]^+$: 485.1613 found 485.1488.

N-(2-(1H-Indol-3-yl)ethyl)-N-(2-(2-methoxyphenyl)-2-oxoethyl)-4-methylbenzenesulfonamide (1c) :



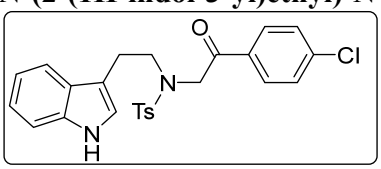
Yield: 90%; white solid; m.p. 164-166 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (s, 1H), 7.74 (d, *J* = 8.0 Hz, 2H), 7.67 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.51-7.44 (m, 2H), 7.30 (d, *J* = 8.0 Hz, 1H), 7.24 (t, *J* = 8.0, 4.0 Hz, 2H), 7.14 (t, *J* = 12.0, 4.0 Hz, 1H), 7.04 (t, *J* =12.0, 8.0 Hz, 1H), 6.99-6.95 (m, 2H), 6.91 (d, *J* = 8.0 Hz, 1H), 4.72 (s, 2H), 3.81 (s, 3H), 3.57 (t, *J* = 16.0, 8.0 Hz, 2H), 3.03 (t, *J* = 16.0, 8.0 Hz, 2H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.0, 143.0, 137.4, 136.2, 134.4, 130.7, 129.4, 127.6, 127.3, 125.6, 122.13, 122.10, 120.9, 119.4, 118.8, 112.8, 111.5, 111.1, 57.4, 55.5, 48.7, 24.8, 21.6; HRMS-ESI (m/z): calcd for $C_{26}H_{26}NaN_2O_4S$ $[M+Na]^+$: 485.1613 found 485.1488.

N-(2-(1H-Indol-3-yl)ethyl)-4-methyl-N-(2-oxo-2-(p-tolyl)ethyl)benzenesulfonamide (1d) :



Yield: 80 %; yellow solid; m.p. 144-146 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (s, 1H), 7.75 (t, *J* = 16.0, 8.0 Hz, 3H), 7.50 (d, *J* = 8.0 Hz, 1H), 7.31 (d, *J* = 12.0 Hz, 1H), 7.27 (d, *J* = 8.0 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.18-7.14 (m, 1H), 7.09-7.05 (m, 1H), 6.97 (d, *J* = 2.0 Hz, 1H), 4.69 (s, 2H), 3.57 (dd, 12.0, 8.0 Hz, 2H), 3.01 (t, *J* = 16.0, 8.0 Hz, 2H), 2.41 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.9, 144.7143.4, 136.8, 136.2, 132.5, 129.6, 129.5, 128.2, 127.6, 127.2, 122.2, 122.1, 119.5, 118.7, 112.5, 111.2, 53.4, 48.9, 24.8, 21.8, 21.6; HRMS-ESI (m/z): calcd for $C_{26}H_{27}N_2O_3S$ $[M+H]^+$: 447.1664 found 447.1736.

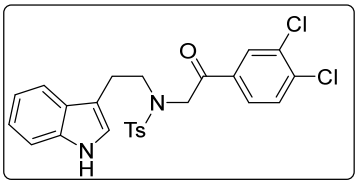
N-(2-(1H-indol-3-yl)ethyl)-N-(2-(4-chlorophenyl)-2-oxoethyl)-4-methylbenzenesulfonamide (1e): Yield: 65



%; sticky brown oil; ^{1}H NMR (400 MHz, DMSO-d6) δ 10.7 (bs, 1H), 7.99 (d, *J* = 8.4 Hz, 2H), 7.75 (d, 8.4 Hz, 2H), 7.61 (d, *J* = 8.4 Hz, 2H), 7.38 (t, 7.6 Hz, 3H), 7.30 (d, *J* = 8.0 Hz, 1H), 7.08-7.02 (m, 2H), 6.94 (t, *J* = 7.2 Hz, 1H), 4.94 (s, 2H), 3.42-3.38 (m, 2H), 2.90-2.88 (m, 2H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.7, 143.0, 138.5. 136.9,

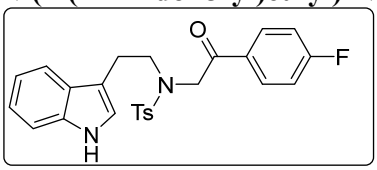
136.1, 133.4, 129.9, 129.6, 128.8, 126.9, 126.8, 122.8, 118.2, 117.9, 11.3, 110.5, 53.8, 49.2, 24.1, 20.9; HRMS-ESI (m/z): calcd for $C_{25}H_{23}ClN_2O_3S$ $[M+Na]^+$: 489.1010 found 489.0999.

N-(2-(1H-indol-3-yl)ethyl)-N-(2-(3,4-dichlorophenyl)-2-oxoethyl)-4-methylbenzenesulfonamide (1f): The



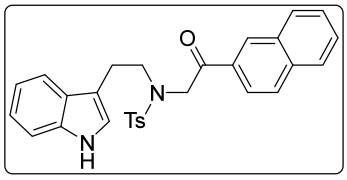
crude was taken into next without any purification and the product was confirmed by HRMS. HRMS-ESI (m/z): calcd for $C_{25}H_{23}Cl_2N_2O_3S$ $[M+H]^+$: 501.0801 found 501.0789.

N-(2-(1H-indol-3-yl)ethyl)-N-(2-(4-fluorophenyl)-2-oxoethyl)-4-methylbenzenesulfonamide (1g): The crude



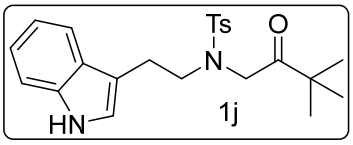
was taken into next without any purification and the product was confirmed by HRMS. HRMS-ESI (m/z): calcd for $C_{25}H_{23}FNaN_2O_3S$ $[M+Na]^+$: 473.1063 found 473.2012.

N-(2-(1H-indol-3-yl)ethyl)-4-methyl-N-(2-(naphthalen-2-yl)-2-oxoethyl)benzenesulfonamide (1h):



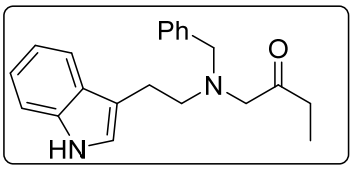
Yield: 75 %; yellow solid; m.p. 184-186 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (s, 1H), 7.92-7.85 (m, 4H), 7.77 (d, *J* = 8.0 Hz, 2H), 7.67-7.54 (m, 3H), 7.47 (d, *J* = 8.0 Hz, 1H), 7.36-7.23 (m, 3H), 7.12-7.08 (m, 1H), 7.02-6.97 (m, 2H), 4.83 (s, 2H), 3.60 (t, *J* = 16.0, 8.0 Hz, 2H), 3.02 (t, *J* = 16.0, 8.0 Hz, 2H), 2.42 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.9, 143.0, 136.9, 136.5, 136.0, 135.4, 129.5, 129.3, 129.27, 129.22, 128.5, 128.3, 127.4, 127.1, 126.7, 126.63, 126.61, 123.1, 122.4, 122.2, 121.3, 118.7, 118.6, 118.0, 111.3, 111.1, 53.2, 48.6, 43.0, 39.9, 39.7, 39.5, 25.3, 24.5, 21.2, 21.1; HRMS-ESI (m/z): calcd for $C_{29}H_{26}NaN_2O_3S$ $[M+Na]^+$: 505.1664 found 505.1545.

N-(2-(1H-indol-3-yl)ethyl)-N-(3,3-dimethyl-2-oxobutyl)-4-methylbenzenesulfonamide (1i): Yield: 85%;

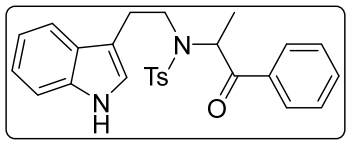


pale-yellow sticky mass; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (bs, 1H), 7.71 (d, *J* = 8.0 Hz, 2H), 7.49 (d, *J* = 8.0 Hz, 1H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.26-7.24 (m, 3H), 7.17 (t, *J* = 8.0 Hz, 1H), 7.08 (t, *J* = 8.4 Hz, 1H), 7.01 (s, 1H), 4.20 (s, 1H), 3.51 (t, *J* = 7.2 Hz, 2H), 3.01 (t, *J* = 7.2 Hz, 2H), 2.40 (s, 3H), 0.99 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.7, 143.2, 137.2, 136.3, 129.4, 127.5, 127.1, 122.2, 119.6, 118.6, 112.7, 111.2, 51.4, 48.2, 43.0, 29.7, 26.2, 25.2, 21.6; HRMS-ESI (m/z): calcd for $C_{23}H_{28}NaN_2O_3S$ $[M+Na]^+$: 435.1718 found 435.1822.

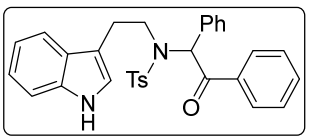
1-((2-(1H-indol-3-yl)ethyl)(benzyl)amino)butan-2-one (1j): Yield: 65%; brown sticky mass; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (bs, 1H), 7.48 (d, *J* = 8.0, 1H), 7.36-7.22 (m, 6H), 7.15 (td, *J* = 8.0, 1.2 Hz, 1H), 7.06 (td, *J* = 8.0, 0.8 Hz, 1H), 6.94 (d, *J* = 2.0 Hz, 1H), 2.97-2.85 (m, 4H), 2.37 (q, *J* = 7.2 Hz, 2H), 0.95 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 211.8, 138.7, 136.1, 128.9, 128.2, 127.3, 127.1, 121.8, 121.6, 119.1, 118.6, 113.8, 111.0, 63.3, 58.8, 55.1, 33.1, 23.2, 7.5; HRMS-ESI (m/z): calcd for $C_{21}H_{24}N_2O$ $[M+H]^+$: 321.1967 found 321.1928.



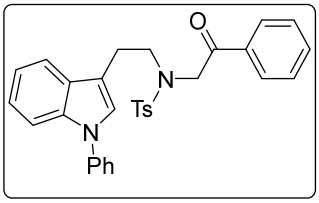
N-(2-(1H-indol-3-yl)ethyl)-4-methyl-N-(1-oxo-1-phenylpropan-2-yl)benzenesulfonamide (1k): Yield: 70%; yellow sticky mass; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (d, *J* = 8.0 Hz, 2H), 8.00 (s, 1H), 7.71 (d, *J* = 8.0 Hz, 2H), 7.62-7.55 (m, 2H), 7.47 (t, *J* = 8.0 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 1H), 7.24-7.10 (m, 4H), 6.90 (s, 1H), 5.66 (dd, *J* = 16.0, 8.0 Hz, 1H), 3.49-3.30 (m, 2H), 3.02-2.93 (m, 1H), 2.87-2.77 (m, 1H), 2.37 (s, 3H), 1.19 (d, *J* = 8.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.9, 143.5, 136.9, 136.1, 135.2, 133.5, 129.6, 128.9, 128.7, 127.4, 127.2, 122.0, 121.9, 119.4, 118.8, 112.7, 111.0, 55.6, 45.6, 27.2, 21.4, 13.7; HRMS-ESI (m/z): calcd for $C_{26}H_{26}NaN_2O_3S$ $[M+Na]^+$: 469.1664 found 469.1556.



N-(2-(1H-Indol-3-yl)ethyl)-4-methyl-N-(2-oxo-1,2-diphenylethyl)benzenesulfonamide (1l): Yield: 72%; yellow solid; m.p. 135-137 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (s, 1H), 7.74-7.71 (m, 2H), 7.67-7.61 (m, 2H), 7.51-7.46 (m, 1H), 7.41-7.32 (m, 6H), 7.28-7.09 (m, 5H), 7.02-6.98 (m, 1H), 6.76 (s, 1H), 6.72 (d, *J* = 2.4 Hz, 1H), 3.73-3.65 (m, 1H), 3.54-3.46 (m, 1H), 3.07-2.99 (m, 1H), 2.36 (s, 3H), 2.14-2.06 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.3, 143.2, 136.9, 136.0, 135.3, 134.8, 133.4, 129.9, 129.5, 129.4, 129.3, 128.9, 128.6, 128.4, 127.3, 127.2, 126.9, 121.76, 121.73, 119.0, 118.8, 113.1, 110.9, 65.1, 47.6, 27.0, 21.4; HRMS-ESI (m/z): calcd for $C_{31}H_{28}NaN_2O_3S$ $[M+Na]^+$: 531.1821 found 531.1712.

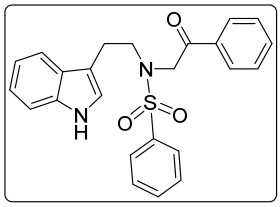


4-Methyl-N-(2-oxo-2-phenylethyl)-N-(2-(1-phenyl-1H-indol-3-yl)ethyl)benzenesulfonamide (1m) :

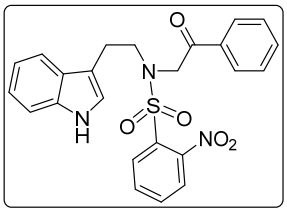


Yield: 76 %; yellow sticky mass; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85-7.82 (m, 2H), 7.76 (d, *J* = 8.0 Hz, 2H), 7.57-7.37 (m, 10H), 7.33-7.24 (m, 3H), 7.20-7.10 (m, 3H), 4.75 (s, 2H), 3.64-3.60 (m, 2H), 3.06 (t, *J* = 8.0 Hz, 2H), 2.39 (s, 3H); 194.2, 143.3, 139.5, 136.7, 135.9, 134.8, 133.6, 129.6, 129.5, 128.7, 128.5, 127.9, 127.4, 127.0, 126.1, 125.8, 123.9, 122.5, 120.1, 118.9, 113.5, 110.5, 53.5, 48.7, 24.6, 21.5; HRMS-ESI (m/z): calcd for $C_{31}H_{28}NaN_2O_3S$ $[M+Na]^+$: 531.1821 found 531.1694.

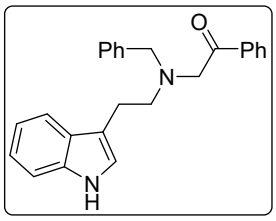
***N*-(2-(1H-Indol-3-yl)ethyl)-*N*-(2-oxo-2-phenylethyl)benzenesulfonamide (1n)**: Yield: 86 %; yellow solid; m.p. 142-144 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (s, 1H), 7.87 (d, *J* = 8.0 Hz, 2H), 7.89 (d, *J* = 2.0 Hz, 2H), 7.57-7.54 (m, 2H), 7.50-7.45 (m, 3H), 7.41 (t, *J* = 8.0 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 1H), 7.14 (t, *J* = 8.0 Hz, 1H), 7.05 (t, *J* = 8.0 Hz, 1H), 6.95 (d, *J* = 4.0 Hz, 1H), 4.71 (s, 2H), 3.60 (dd, *J* = 12.0, 8.0 Hz, 2H), 3.01 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.2, 139.8, 136.2, 134.9, 133.8, 132.7, 129.0, 128.8, 128.0, 127.5, 127.1, 127.0, 122.26, 122.21, 119.6, 118.6, 112.4, 111.2, 53.5, 48.9, 24.8; HRMS-ESI (m/z): calcd for $C_{24}H_{22}NaN_2O_3S$ $[M+Na]^+$: 441.1351 found 441.1235.



N-(2-(1H-Indol-3-yl)ethyl)-2-nitro-N-(2-oxo-2-phenylethyl)benzenesulfonamide (1o): Yield: 78 %; yellow solid; m.p. 140-142 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.0 Hz, 1H), 7.97 (s, 1H), 7.77 (d, *J* = 8.0 Hz, 2H), 7.60-7.47 (m, 5H), 7.41 (t, *J* = 8.0 Hz, 2H),7.28 (d, *J* = 8.0 Hz, 1H), 7.13 (t, *J* = 8.0 Hz, 1H), 7.04 (t, *J* = 8.0 Hz, 1H), 6.98 (s, 1H), 4.86 (s, 2H), 3.78 (t, *J* = 8.0 Hz, 2H), 3.03 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.9, 147.7, 136.2, 134.7, 133.8, 133.6, 133.2, 131.7, 130.8, 128.8, 127.9,127.0, 124.0, 122.4, 122.2, 119.6, 118.5, 112.0, 56.3, 48.9, 24.6; HRMS-ESI (m/z): calcd for $C_{24}H_{21}NaN_3O_5S$ $[M+Na]^+$: 468.1202 found 468.1088.

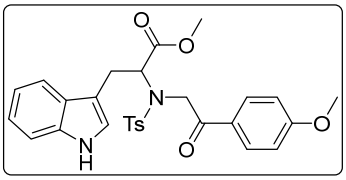


2-((2-(1H-indol-3-yl)ethyl)(benzyl)amino)-1-phenylethan-1-one (1p): Yield: 82 %; brown sticky mass; m.p. 140-142 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 (bs, 1H), 7.76 (d, *J* = 7.2 Hz, 2H), 7.54-7.48 (m, 2H), 7.49-7.24 (m, 9H), 7.12 (td, J = 8.0, 1.2 Hz, 1H), 7.05 (td, J = 7.6, 0.8 Hz, 1H), 6.96 (d, J = 1.2 Hz ,1H), 4.11 (bs, 4H), 3.25-3.23 (m, 2H), 3.13-3.09 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.1, 133.5, 129.8, 128.5, 128.0, 127.1, 122.1, 121.9, 119.2, 118.6, 119.1, 58.4, 54.4, 22.6; HRMS-ESI (m/z): calcd for $C_{25}H_{24}N_2O$ $[M+H]^+$: 369.1967 found 369.1959.



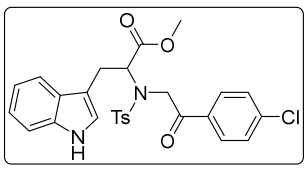
Methyl *N*$^{\alpha}$-(2-oxo-2-phenylethyl)-*N*$^{\alpha}$-tosyl-L-tryptophanate (1q) : Yield: 70 %; yellow solid; m.p. 118-120 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (s, 1H), 7.96 (d, *J* = 4.0 Hz, 2H), 7.83 (d, *J* = 12.0 Hz, 2H), 7.60 (t, *J* = 16.0, 8.0 Hz, 1H), 7.48 (t, *J* = 16.0, 8.0 Hz, 2H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.31 (d, *J* = 8.0 Hz, 1H), 7.23 (t, *J* = 16.0, 8.0 Hz, 2H), 7.15 (t, *J* = 12.0, 8.0 Hz, 1H), 7.08-7.00 (m, 1H), 6.94 (d, *J* = 4.0 Hz, 1H), 5.10 (s, 2H), 4.65 (dd, *J* = 8.0, 4.0 Hz, 1H), 3.41 (s, 3H), 3.27-3.12 (m, 2H), 2.40 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.3, 171.3, 143.6, 136.6, 136.1, 135.2, 133.6, 129.5, 129.3, 128.8, 128.2, 128.0, 127.1, 127.0, 123.3, 122.1, 119.6, 118.5, 111.2, 109.8, 59.2, 52.0, 50.5, 26.8, 21.6; HRMS-ESI (m/z): calcd for $C_{27}H_{26}N_2O_5S$ $[M+Na]^+$: 513.1562 found 513.1443.

Methyl *N*$^{\alpha}$-(2-(4-methoxyphenyl)-2-oxoethyl)-*N*$^{\alpha}$-tosyl-L-tryptophanate (1r): Yield: 77 %; yellow solid; m.p.

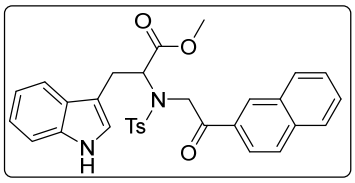


100-102 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (s, 1H), 7.95-7.92 (m, 2H), 7.84-7.81 (m, 2H), 7.39 (d, *J* = 8.0 Hz, 1H), 7.29-7.20 (m, 3H), 7.16-7.12 (m, 1H), 7.07-7.03 (m, 1H), 6.96-6.92 (m, 3H), 5.05 (s, 2H), 4.64 (dd, *J* = 12.0, 8.0, Hz, 1H), 3.87 (s, 3H), 3.40 (s, 3H), 3.26-3.11 (m, 2H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.6, 171.2, 163.8, 143.4, 136.6, 136.0, 130.2, 129.2, 128.08, 128.03, 126.9, 123.2, 121.9, 119.4, 118.3, 113.9, 111.1, 109.7, 59.2, 55.4, 51.8, 49.5, 26.7, 21.5; HRMS-ESI (m/z): calcd for $C_{28}H_{28}NaN_2O_6S$ $[M+Na]^+$: 546.1668 found 546.1560.

Methyl *N*$^{\alpha}$-(2-(4-chlorophenyl)-2-oxoethyl)-*N*$^{\alpha}$-tosyl-L-tryptophanate (1s): Yield: 54 %; yellow solid; m.p.

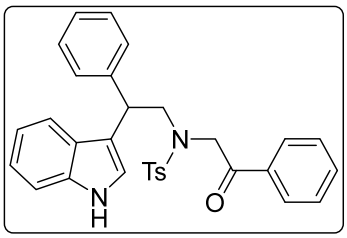


80-82 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (s, 1H), 7.90-7.86 (m, 2H), 7.81 (d, *J* = 8.0 Hz, 2H), 7.44 (d, *J* = 8.0 Hz, 2H), 7.39 (d, *J* = 4.0 Hz, 1H), 7.29 (d, *J* = 8.0 Hz, 1H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.18-7.14 (m, 1H), 7.08-7.04 (m, 1H), 6.94 (d, *J* = 4.0 Hz, 2H), 5.03 (s, 2H), 4.65 (dd, *J* = 8.0, 4.0 Hz, 1H), 3.41 (s, 3H), 3.23 (dd, *J* = 12.0, 8.0Hz, 1H), 3.15-3.10 (m, 1H), 2.40 (s, 3H); 193.2, 171.2, 143.6, 140.0, 136.3, 136.0, 133.8, 129.36, 129.31, 129.0, 128.0, 126.9, 123.1, 122.1, 119.5, 118.3, 111.1, 109.6, 59.0, 51.9, 49.8, 26.7, 21.5; HRMS-ESI (m/z): calcd for $C_{27}H_{25}ClNaN_2O_5S$ $[M+Na]^+$: 547.1173 found 547.1085.



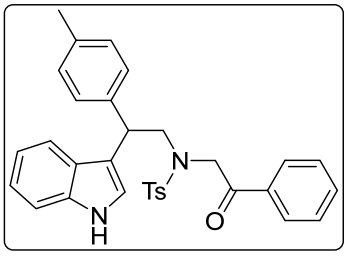
Methyl *N*$^{\alpha}$-(2-(naphthalen-2-yl)-2-oxoethyl)-*N*$^{\alpha}$-tosyltryptophanate (1t): The crude was taken to next without any purification and the product was confirmed by HRMS. HRMS-ESI (m/z): calcd for $C_{31}H_{28}N_2NaO_5S$ $[M+Na]^+$: 563.1611 found 563.1611.

***N*-(2-(1H-Indol-3-yl)-2-phenylethyl)-4-methyl-*N*-(2-oxo-2-phenylethyl)benzenesulfonamide (1u)** : Yield: 88



%; white solid; m.p. 162-164 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (s, 1H), 7.69 (d, *J* = 8.0 Hz, 2H), 7.56-7.48 (m, 3H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.34-7.09 (m, 11H), 7.05 (d, *J* = 4.0 Hz, 1H), 7.00-6.96 (m, 1H), 4.66 (d, *J* = 16.0 Hz, 1H), 4.56 (t, *J* = 16.0, 8.0 Hz, 1H), 4.24-4.13 (m, 2H), 3.76 (dd, *J* = 12.0 8.0 Hz, 1H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.3, 143.2, 142.1, 136.8, 136.3, 134.7, 133.5, 19.4, 128.59, 128.56, 128.1, 127.6, 127.5, 126.7, 126.6, 122.1, 122.0, 119.5, 119.2, 116.1, 111.0, 53.2, 52.4, 42.6, 21.5; HRMS-ESI (m/z): calcd for $C_{31}H_{28}NaN_2O_3S$ $[M+Na]^+$: 531.1821 found 531.1713.

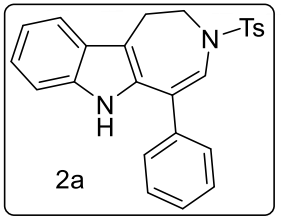
***N*-(2-(1H-Indol-3-yl)-2-(p-tolyl)ethyl)-4-methyl-*N*-(2-oxo-2-phenylethyl)benzenesulfonamide (1v)** :



Yield: 77 %; yellow solid; m.p. 139-141 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (s, 1H), 7.69 (d, *J* = 8.0 Hz, 2H), 7.57-7.48 (m, 3H), 7.41 (d, *J* = 8.0 Hz, 1H), 7.34-7.22 (m, 5H), 7.13-7.09 (m, 3H), 7.04-6.96 (m, 4H), 4.65 (d, *J* = 16.0 Hz, 1H), 4.23-4.16 (m, 2H), 7.74 (dd, *J* = 12.0 8.0 Hz, 1H), 2.39 (s, 3H), 2.24 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.3, 143.2, 139.1, 136.8, 136.3, 136.2, 134.7, 133.4, 129.4, 129.2, 128.5, 128.0, 127.6, 127.5, 126.6, 122.1, 122.0, 119.5, 119.3, 116.4, 111.0, 53.1, 52.3, 42.2, 21.5, 20.9; HRMS-ESI (m/z): calcd for $C_{32}H_{30}NaN_2O_3S$ $[M+Na]^+$: 545.1977 found 545.1872.

References:

1. Tosylation of tryptamine and tryptophan compounds: D. L. Priebbenow, L. C. Henderson, F. M. Pfeffer, S. G. Stewart, *J. Org. Chem*., **2010**, *75*, 1787–1790.
2. For *N*-Arylation of Tryptamine: Jon. C. Antilla, A. Klapars, S. L. Buchwald, *J. Am. Chem. Soc*., **2002**, *124*, 11684 – 11688.
3. Synthesis of methyl ester of Tryptophan derivatives: H. Song, J. Yang, W. Chen, Y. Qin, *Org. Lett.*, **2006**, *8*, 6011–6014.



SIVA-07-154

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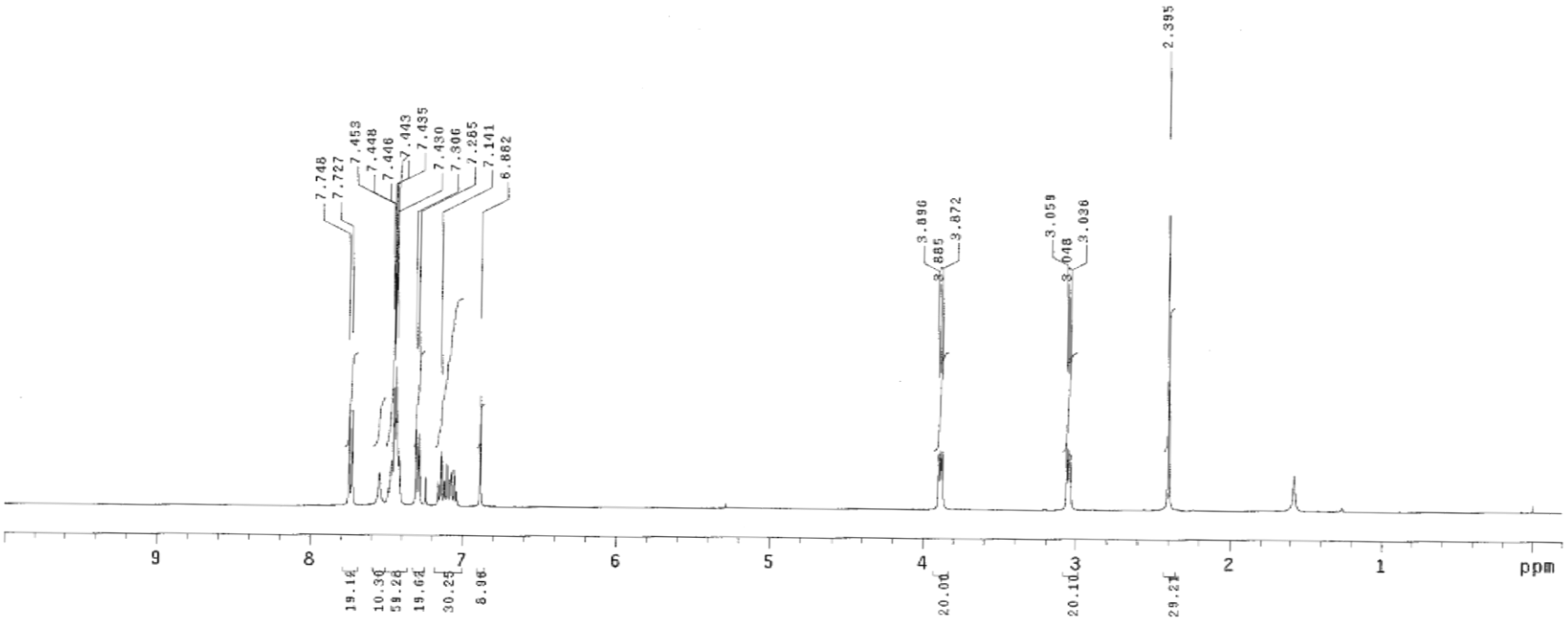
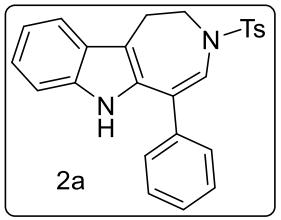
Ambient temperature
Total 64 repetitions





SIVA-07-154

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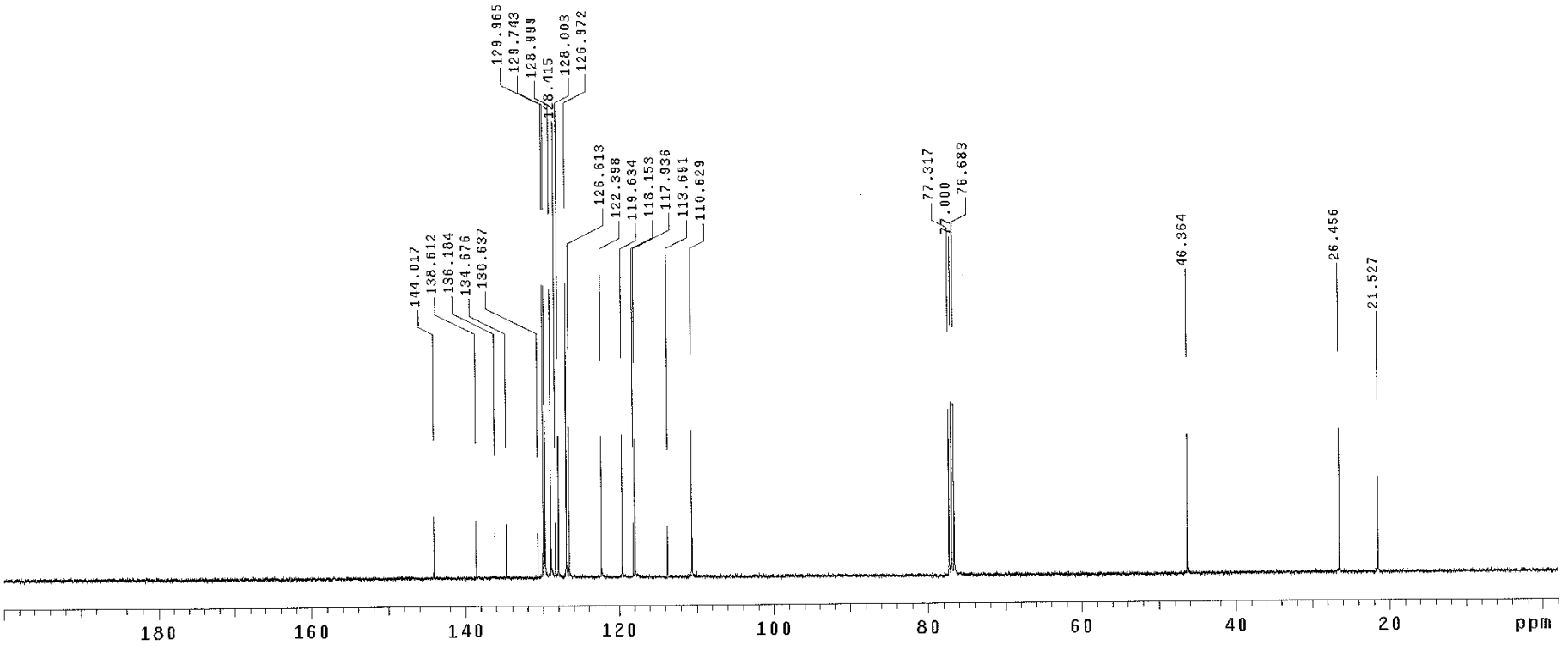
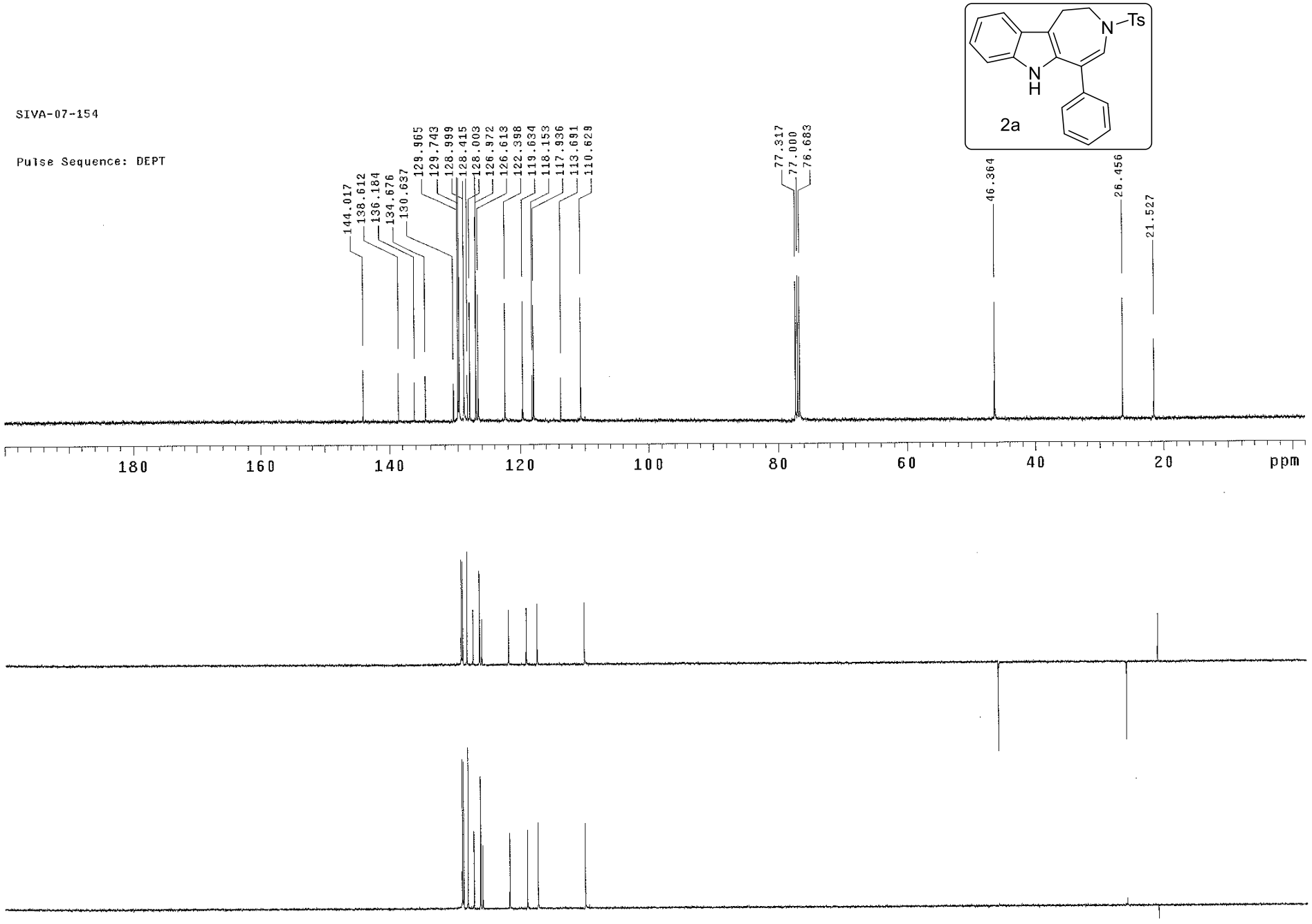
Solvent: cdcl3
Ambient temperature
Total 64000 repetitions




SIVA-07-154
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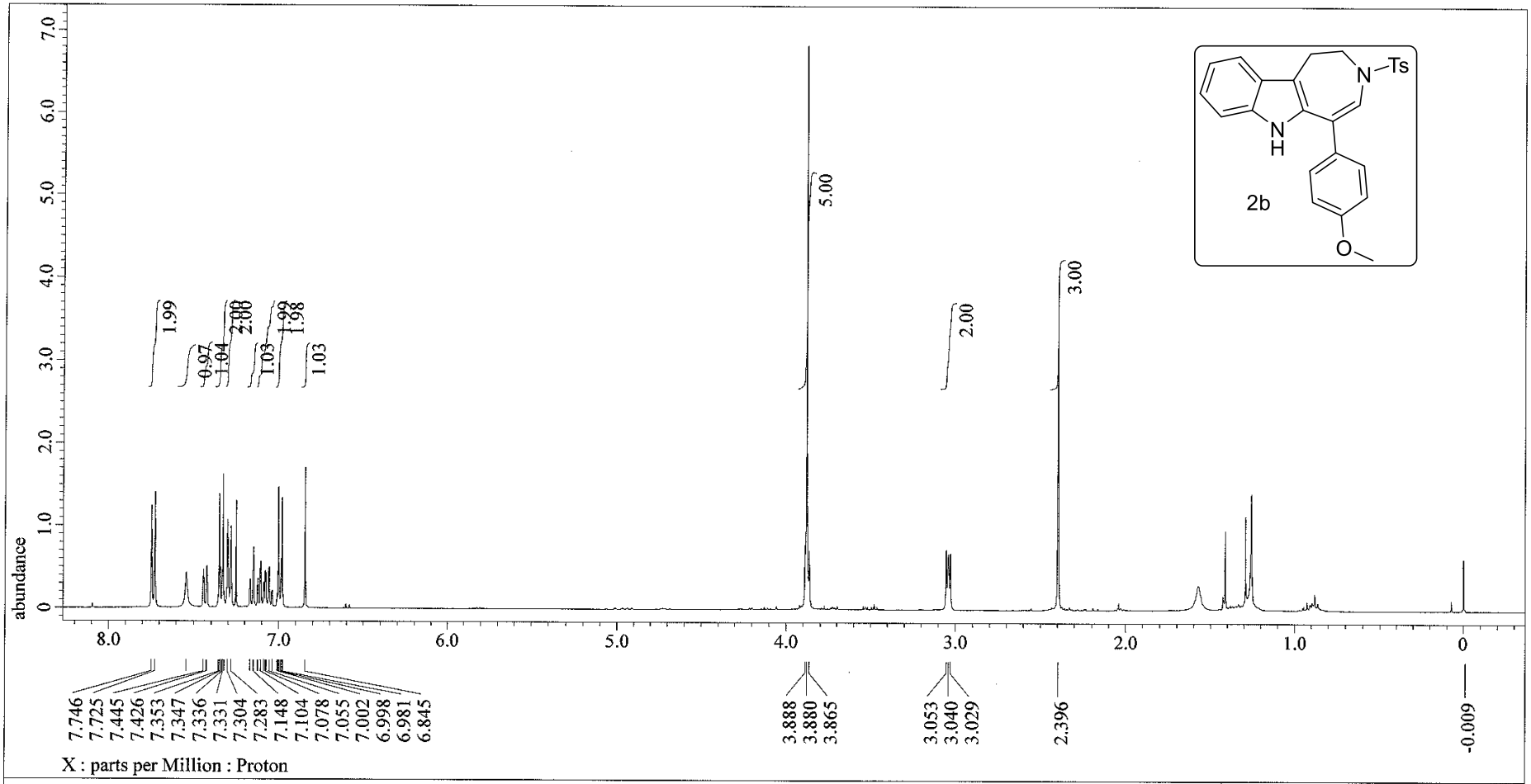
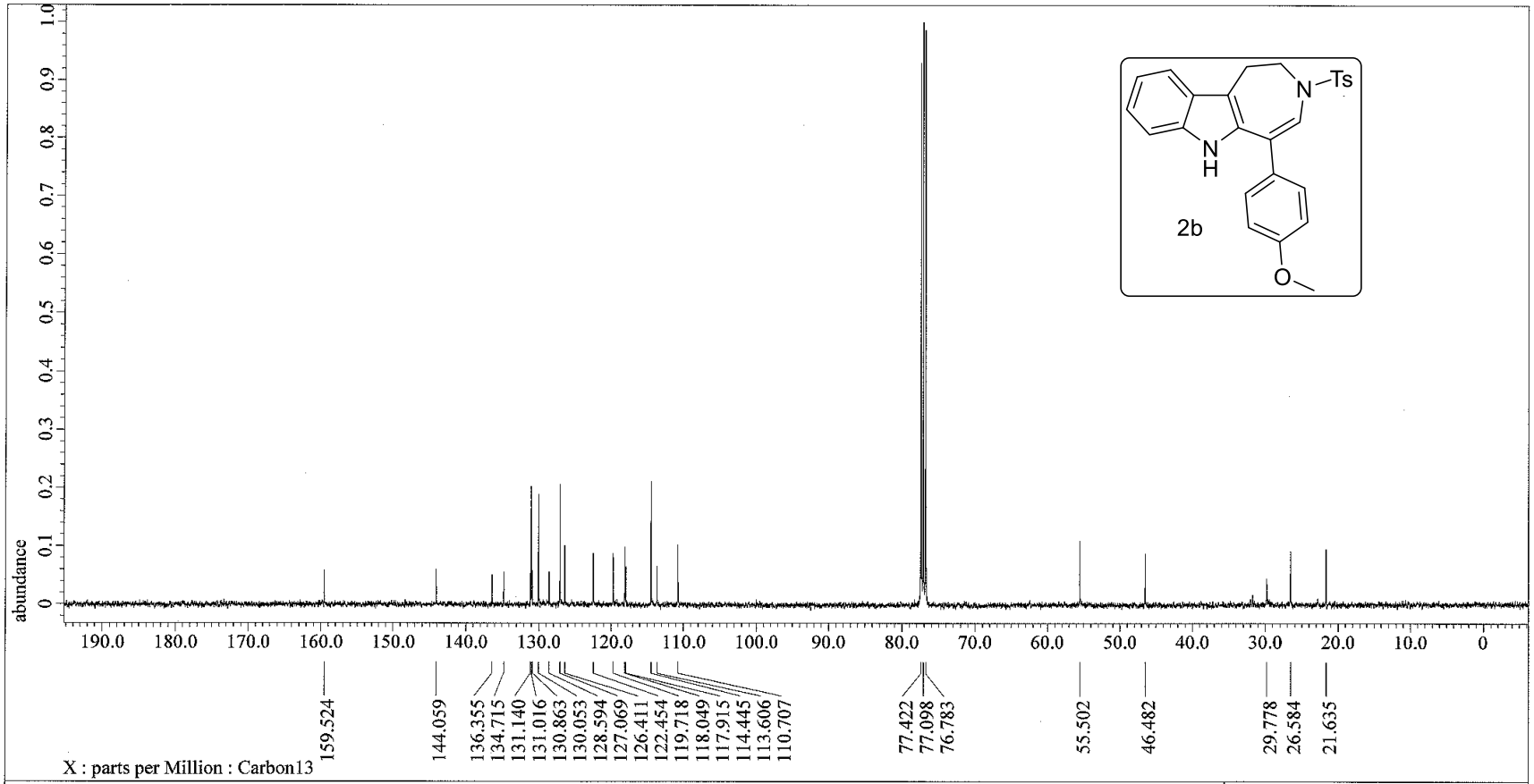
119.634
118.153
117.936
113.691
110.629
77.317
77.000
76.683
46.364
26.456
21.527
180
160
140
120
100
80
60
40
20
ppm
N
Ts
N
H
2a



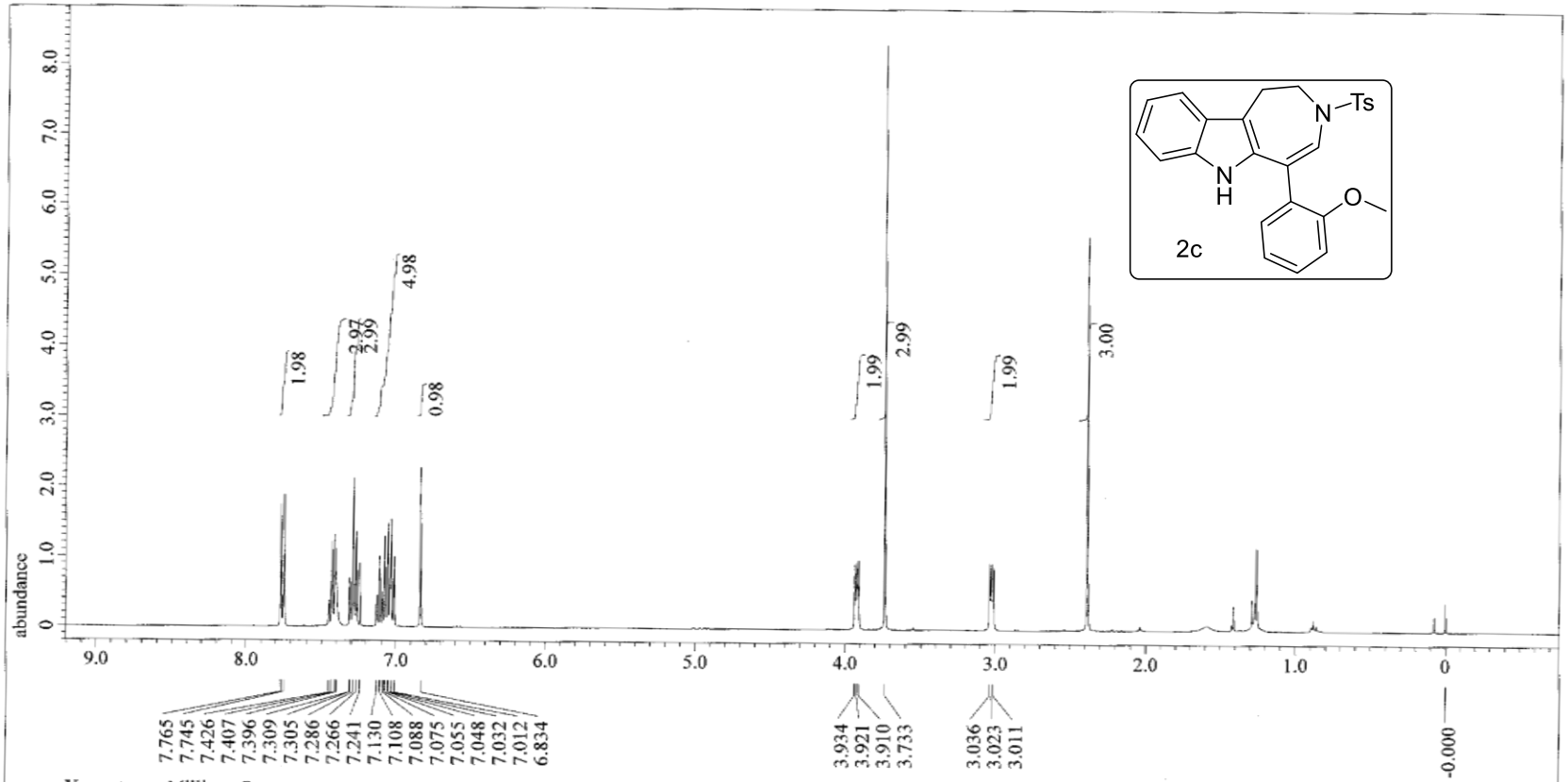
Filename	= HOU-03-10_Proton-1-3.jdf	Spectrometer	= DELTA2_NMR	Tri_Freq	= 399.78219838[MHz]
Author	= delta			Tri_Offset	= 5[ppm]
Experiment	= proton.jxp	Field_Strength	= 9.389766[T] (400[MHz])	Clipped	= FALSE
Sample_Id	= HOU-03-10	X_Acq_Duration	= 2.18365952[s]	Scans	= 23
Solvent	= CHLOROFORM-D	X_Domain	= 1H	Total_Scans	= 23
Creation_Time	= 7-JUL-2016 11:36:59	X_Freq	= 399.78219838[MHz]		
Revision_Time	= 7-JUL-2016 11:36:02	X_Offset	= 5[ppm]	Relaxation_Delay	= 5[s]
Current_Time	= 7-JUL-2016 11:36:27	X_Points	= 16384	Recvr_Gain	= 40
		X_Prescans	= 1	Temp_Get	= 26.2[dC]
Comment	= single_pulse	X_Resolution	= 0.45794685[Hz]	X_90_Width	= 12.795[us]
Data_Format	= 1D COMPLEX	X_Sweep	= 7.5030012[kHz]	X_Acq_Time	= 2.18365952[s]
Dim_Size	= 13107	X_Sweep_Clipped	= 6.00240096[kHz]	X_Angle	= 45[deg]
Dim_Title	= Proton	Irr_Domain	= Proton	X_Atn	= 2.4[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	X_Pulse	= 6.3975[us]
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Mode	= Off
Site	= JNM-ECS400	Tri_Domain	= Proton	Tri_Mode	= Off

JEOL RESONANCE



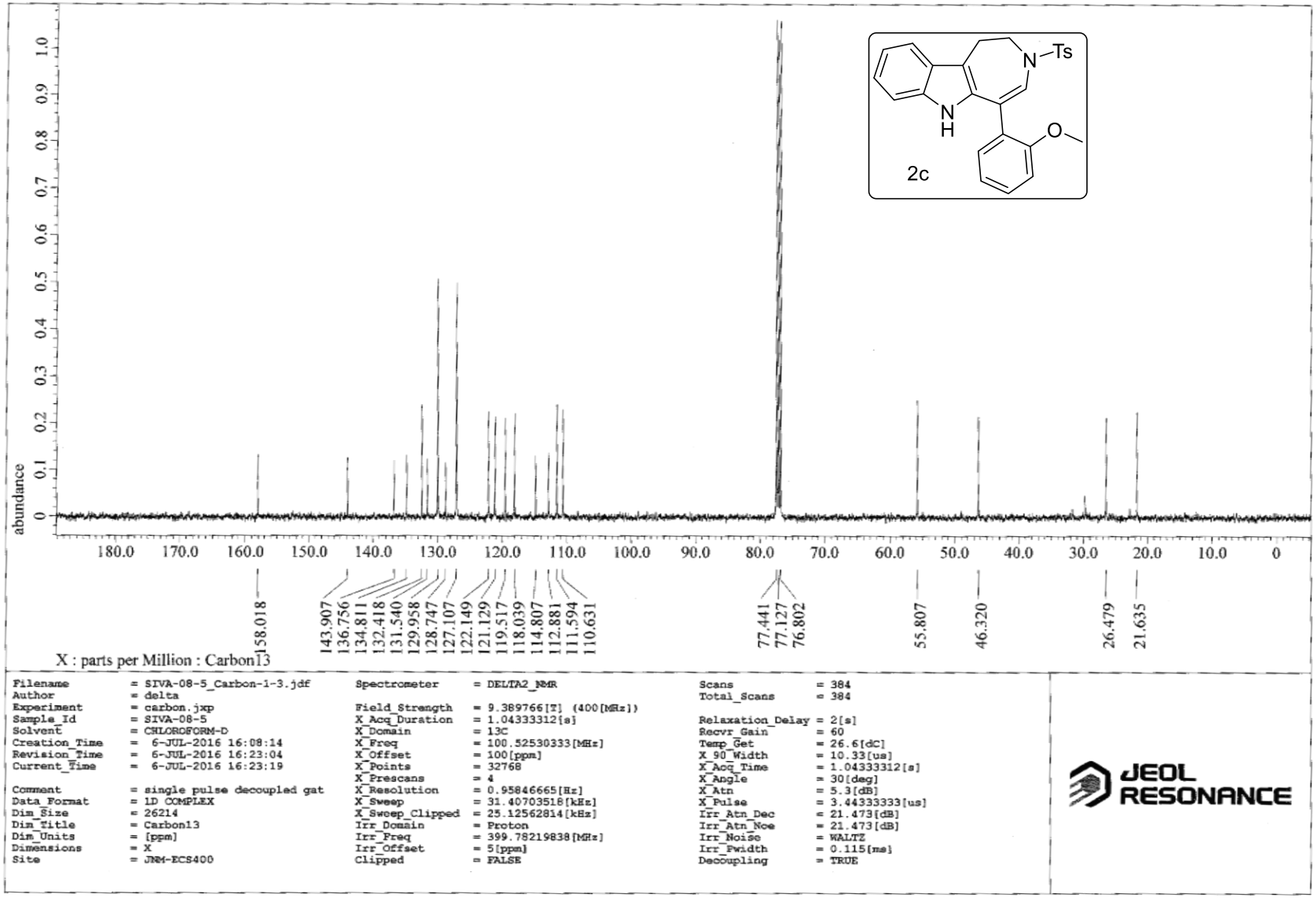
Filename	= HOU-03-10_Carbon-1-3.jdf	Spectrometer	= DELTA2_NMR	Scans	= 873
Author	= delta			Total_Scans	= 873
Experiment	= carbon.jxp	Field_Strength	= 9.389766[T] (400[MHz])		
Sample_Id	= HOU-03-10	X_Acq_Duration	= 1.04333312[s]	Relaxation_Delay	= 2[s]
Solvent	= CHLOROFORM-D	X_Domain	= 13C	Recvr_Gain	= 60
Creation_Time	= 7-JUL-2016 11:42:11	X_Freq	= 100.52530333[MHz]	Temp_Get	= 25.9[dC]
Revision_Time	= 7-JUL-2016 12:21:33	X_Offset	= 100[ppm]	X_90_Width	= 10.33[us]
Current_Time	= 7-JUL-2016 12:22:48	X_Points	= 32768	X_Acq_Time	= 1.04333312[s]
		X_Prescans	= 4	X_Angle	= 30[deg]
Comment	= single pulse decoupled gat	X_Resolution	= 0.95846665[Hz]	X_Atn	= 5.3[dB]
Data_Format	= 1D COMPLEX	X_Sweep	= 31.40703518[kHz]	X_Pulse	= 3.44333333[us]
Dim_Size	= 26214	X_Sweep_Clipped	= 25.12562814[kHz]	Irr_Atn_Dec	= 21.473[dB]
Dim_Title	= Carbon13	Irr_Domain	= Proton	Irr_Atn_Noe	= 21.473[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	Irr_Noise	= WALTZ
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Pwidth	= 0.115[ms]
Site	= JNM-ECS400	Clipped	= FALSE	Decoupling	= TRUE

JEOL RESONANCE



Filename	= SIVA-08-5_Proton-1-3.jdf	Spectrometer	= DELTA2_NMR	Tri_Freq	= 399.78219838[MHz]
Author	= delta			Tri_Offset	= 5[ppm]
Experiment	= proton.jxp	Field_Strength	= 9.389766[T] (400[MHz])	Clipped	= FALSE
Sample_Id	= SIVA-08-5	X_Acq_Duration	= 2.18365952[s]	Scans	= 16
Solvent	= CHLOROFORM-D	X_Domain	= 1H	Total_Scans	= 16
Creation_Time	= 6-JUL-2016 16:03:52	X_Freq	= 399.78219838[MHz]		
Revision_Time	= 6-JUL-2016 16:01:59	X_Offset	= 5[ppm]	Relaxation_Delay	= 5[s]
Current_Time	= 6-JUL-2016 16:02:08	X_Points	= 16384	Recvr_Gain	= 36
		X_Prescans	= 1	Temp_Get	= 26.5[dC]
Comment	= single_pulse	X_Resolution	= 0.45794685[Hz]	X_90_Width	= 12.795[us]
Data_Format	= 1D COMPLEX	X_Sweep	= 7.5030012[kHz]	X_Acq_Time	= 2.18365952[s]
Dim_Size	= 13107	X_Sweep_Clipped	= 6.00240096[kHz]	X_Angle	= 45[deg]
Dim_Title	= Proton	Irr_Domain	= Proton	X_Atn	= 2.4[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	X_Pulse	= 6.3975[us]
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Mode	= Off
Site	= JNM-ECS400	Tri_Domain	= Proton	Tri_Mode	= Off

JEOL RESONANCE

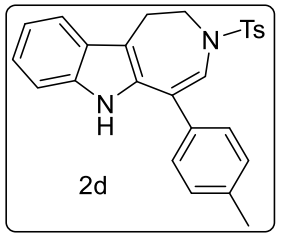


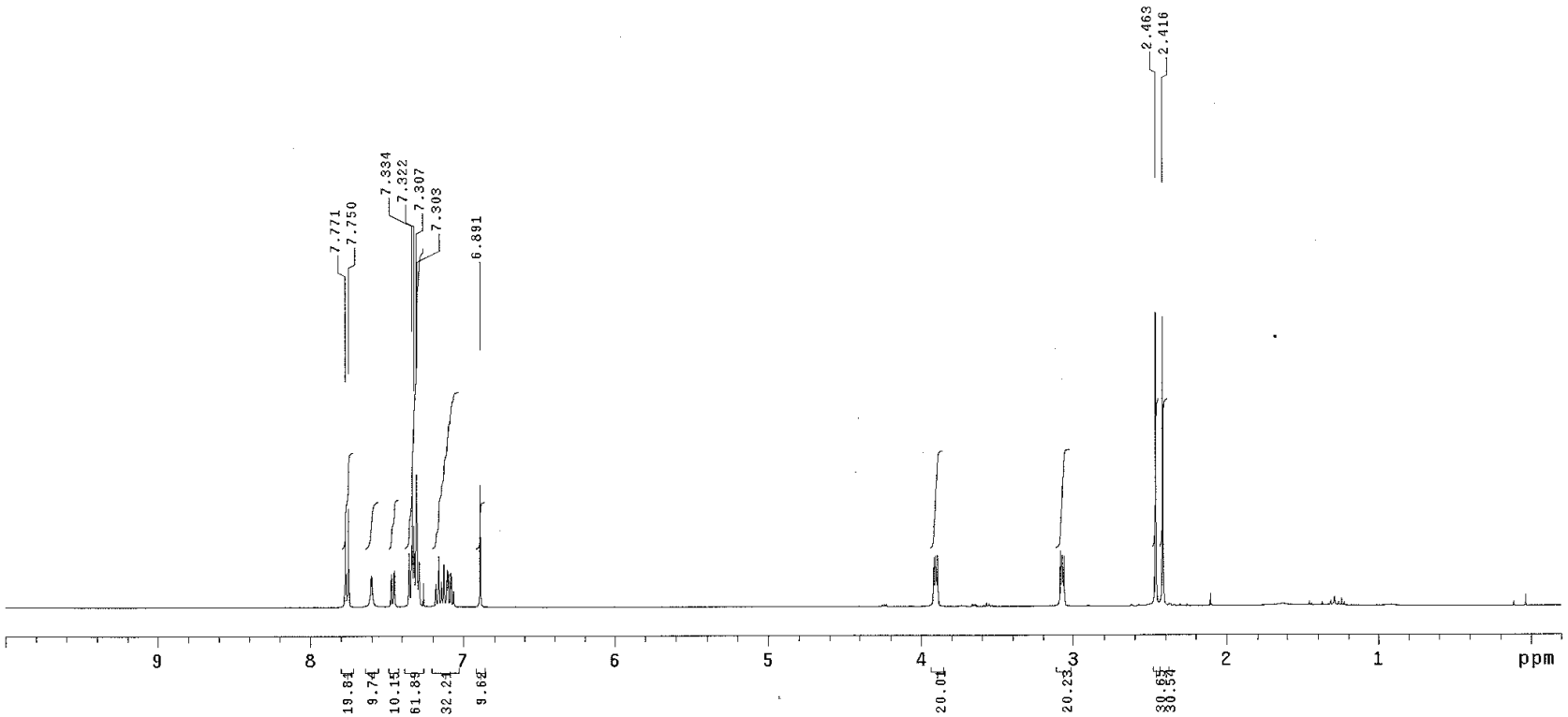
Filename	= SIVA-08-5_Carbon-1-3.jdf	Spectrometer	= DELTA2_NMR	Scans	= 384
Author	= delta			Total_Scans	= 384
Experiment	= carbon.jxp	Field_Strength	= 9.389766[T] (400[MHz])		
Sample_Id	= SIVA-08-5	X_Acq_Duration	= 1.04333312[s]	Relaxation_Delay	= 2[s]
Solvent	= CHLOROFORM-D	X_Domain	= 13C	Recvr_Gain	= 60
Creation_Time	= 6-JUL-2016 16:08:14	X_Freq	= 100.52530333[MHz]	Temp_Get	= 26.6[dC]
Revision_Time	= 6-JUL-2016 16:23:04	X_Offset	= 100[ppm]	X_90_Width	= 10.33[us]
Current_Time	= 6-JUL-2016 16:23:19	X_Points	= 32768	X_Acq_Time	= 1.04333312[s]
		X_Prescans	= 4	X_Angle	= 30[deg]
Comment	= single pulse decoupled gat	X_Resolution	= 0.95846665[Hz]	X_Atn	= 5.3[dB]
Data_Format	= 1D COMPLEX	X_Sweep	= 31.40703518[kHz]	X_Pulse	= 3.44333333[us]
Dim_Size	= 26214	X_Sweep_Clipped	= 25.12562814[kHz]	Irr_Atn_Dec	= 21.473[dB]
Dim_Title	= Carbon13	Irr_Domain	= Proton	Irr_Atn_Noe	= 21.473[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	Irr_Noise	= WALTZ
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Pwidth	= 0.115[ms]
Site	= JNM-ECS400	Clipped	= FALSE	Decoupling	= TRUE

JEOL RESONANCE

Hou-02-129

Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Jul 6 2016
Solvent: cdcl3
Ambient temperature
Total 32 repetitions

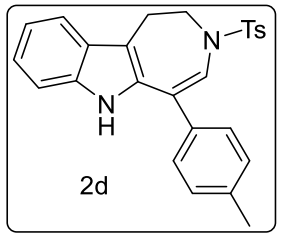


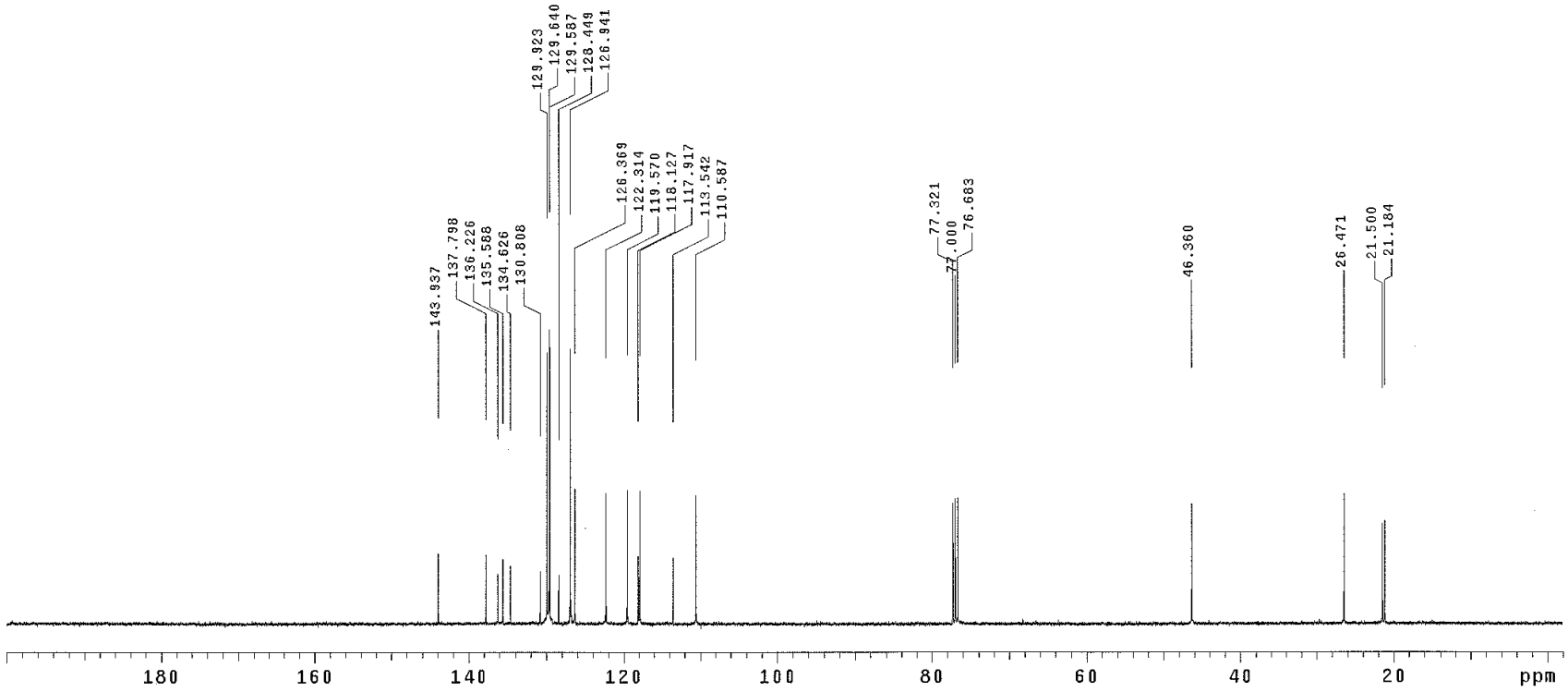


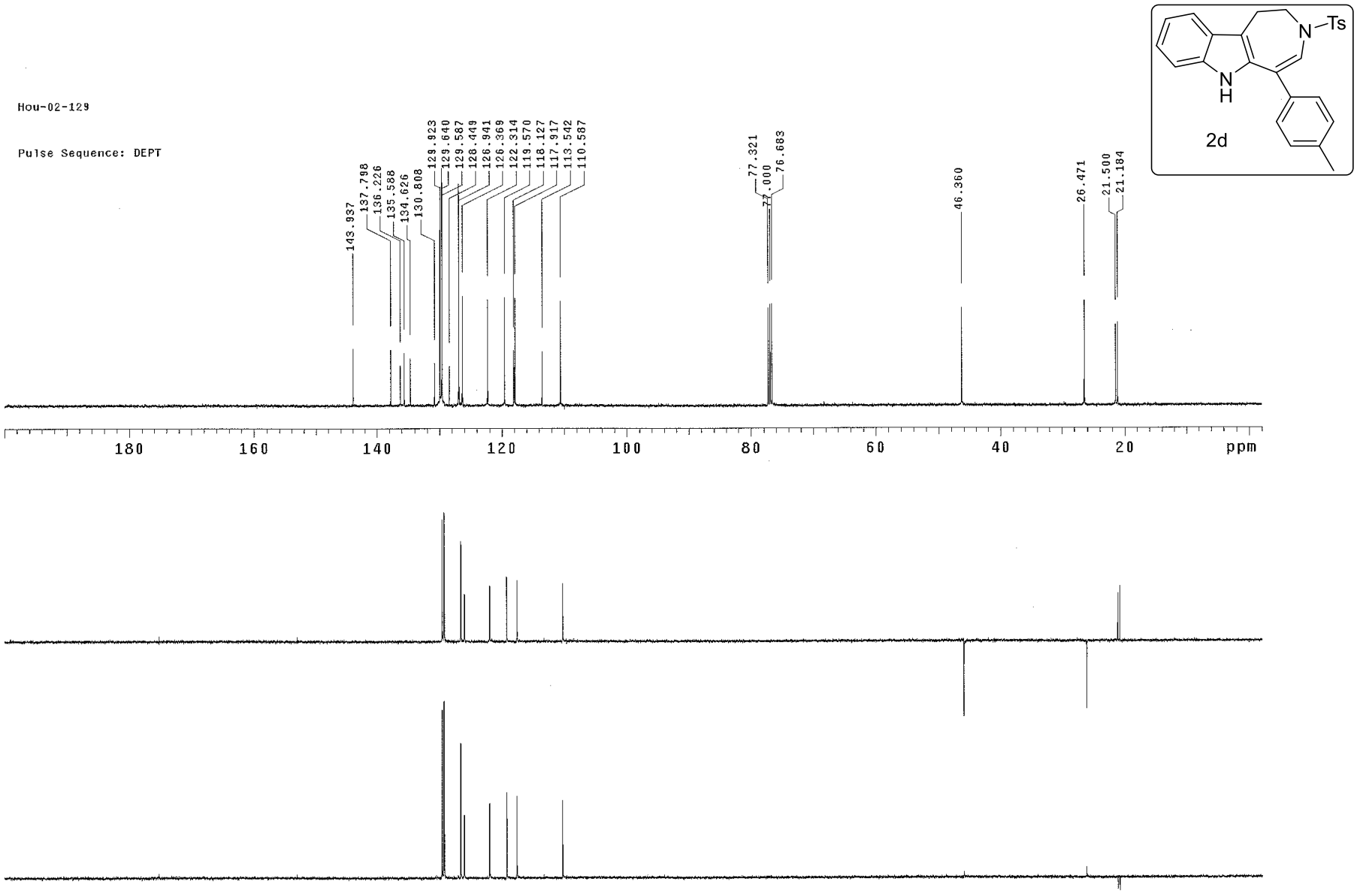
Hou-02-129

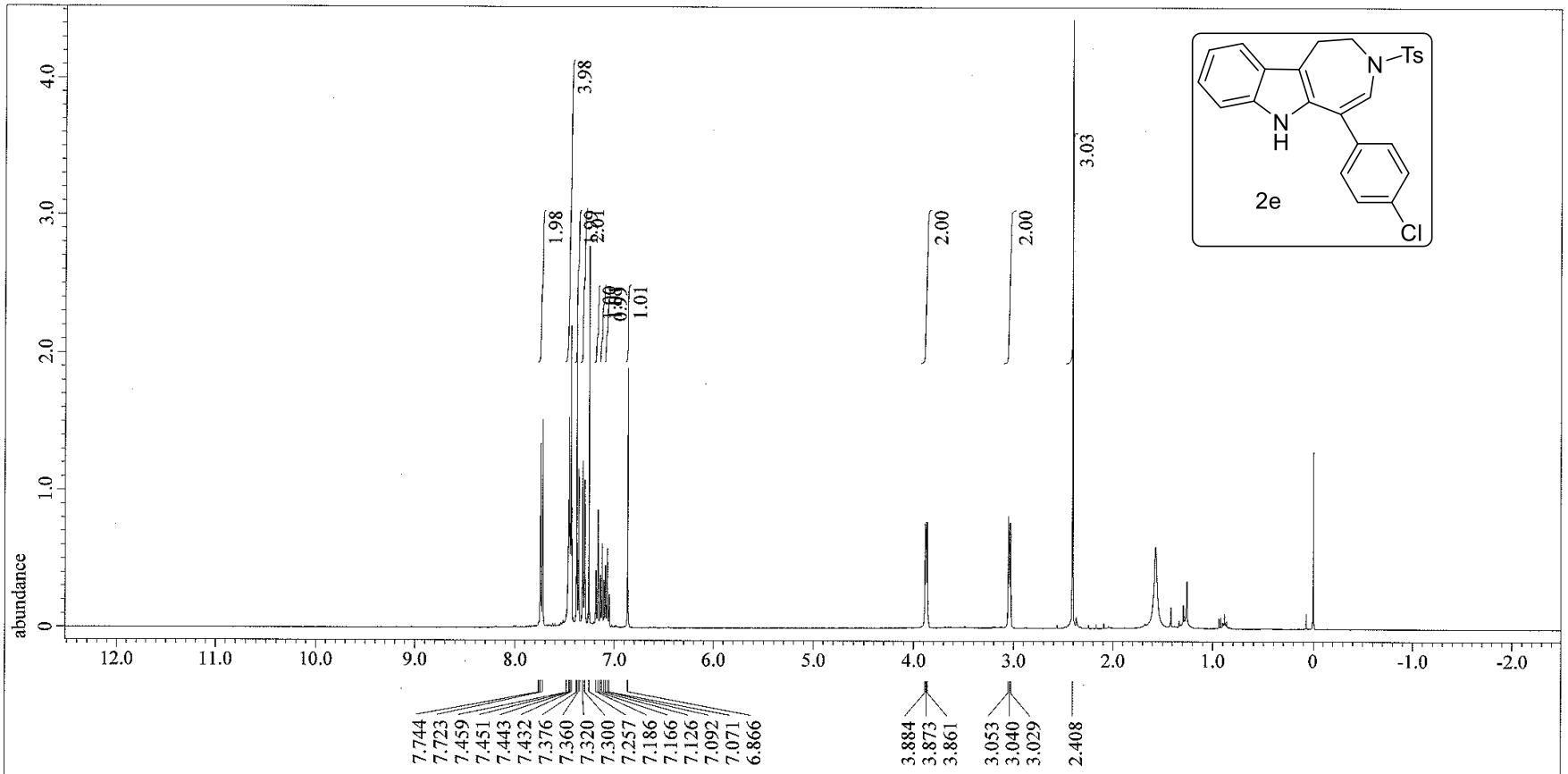
Pulse Sequence: s2pul

Mercury-400BB "MerPlus400"
Date: Jul 6 2016
Solvent: cdcl3
Ambient temperature
Total 704 repetitions





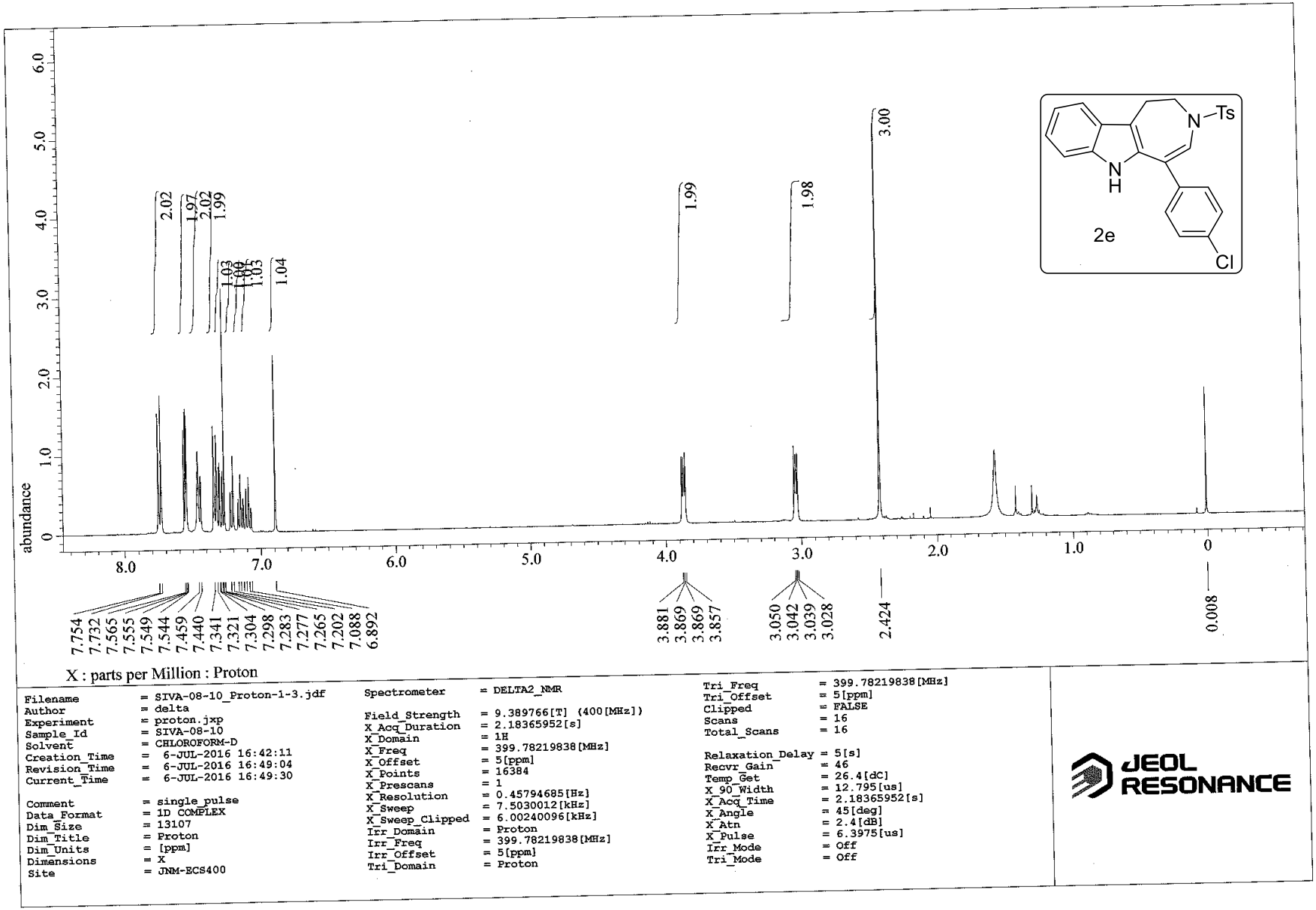

Hou-02-129
Pulse Sequence: DEPT
143.937
137.798
136.226
135.588
134.626
130.808
129.923
129.640
129.587
128.449
126.941
126.369
122.314
119.570
118.127
117.917
113.542
110.587
77.321
77.000
76.683
46.360
26.471
21.500
21.184
180
160
140
120
100
80
60
40
20
ppm
N
Ts
N
H
2d



X : parts per Million : Proton

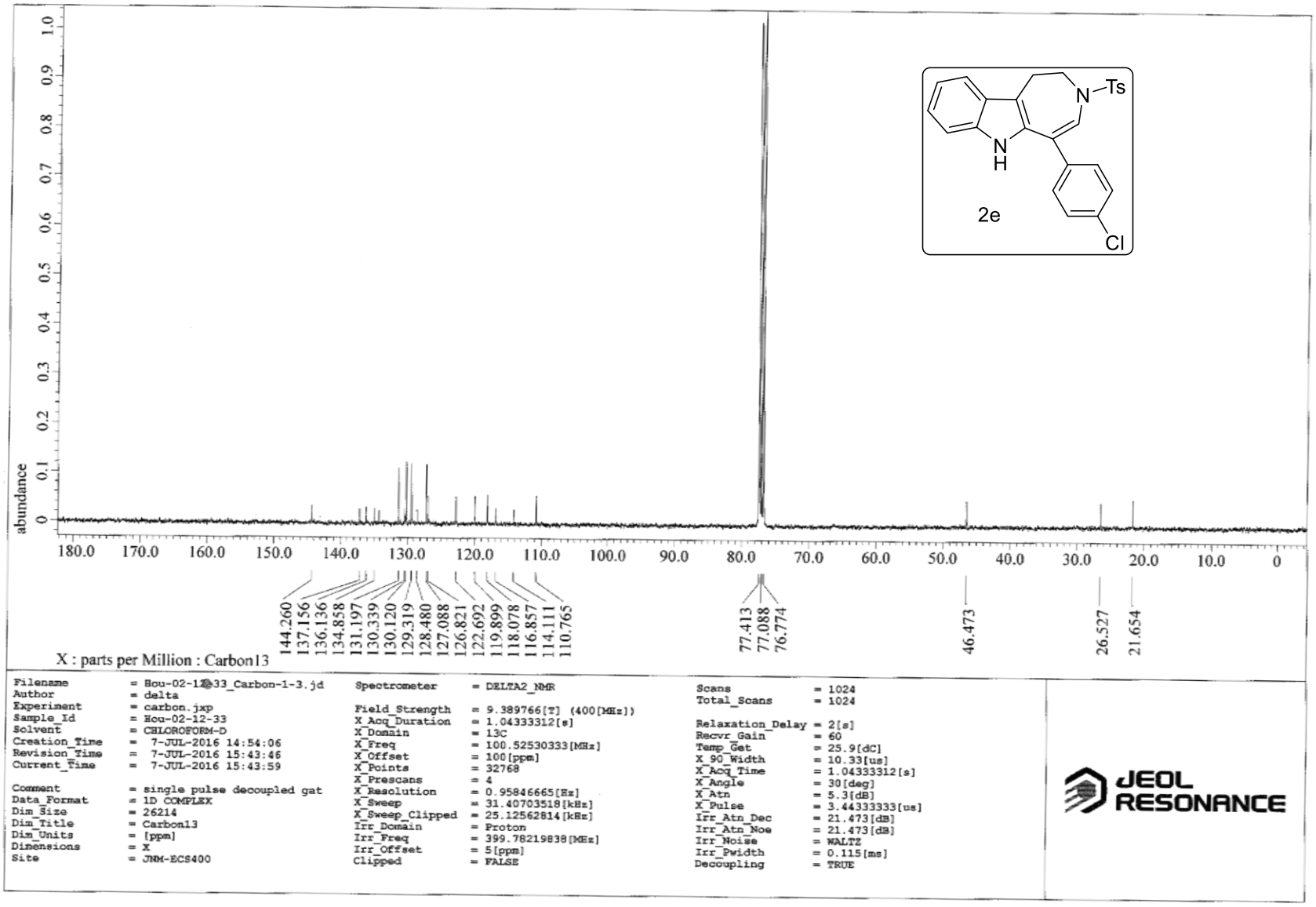
Filename	= Hou-02-133_Proton-1-8.jdf	Spectrometer	= DELTA2_NMR	Tri_Freq	= 399.78219838[MHz]
Author	= delta			Tri_Offset	= 5[ppm]
Experiment	= proton.jxp	Field_Strength	= 9.389766[T] (400[MHz])	Clipped	= FALSE
Sample_Id	= Hou-02-12-33	X_Acq_Duration	= 2.18365952[s]	Scans	= 16
Solvent	= CHLOROFORM-D	X_Domain	= 1H	Total_Scans	= 16
Creation_Time	= 7-JUL-2016 14:49:40	X_Freq	= 399.78219838[MHz]		
Revision_Time	= 25-JUL-2016 14:57:09	X_Offset	= 5[ppm]	Relaxation_Delay	= 5[s]
Current_Time	= 25-JUL-2016 14:57:33	X_Points	= 16384	Recvr_Gain	= 46
		X_Prescans	= 1	Temp_Get	= 25.6[dC]
Comment	= single_pulse	X_Resolution	= 0.45794685[Hz]	X_90_Width	= 12.795[us]
Data_Format	= 1D COMPLEX	X_Sweep	= 7.5030012[kHz]	X_Acq_Time	= 2.18365952[s]
Dim_Size	= 13107	X_Sweep_Clipped	= 6.00240096[kHz]	X_Angle	= 45[deg]
Dim_Title	= Proton	Irr_Domain	= Proton	X_Atn	= 2.4[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	X_Pulse	= 6.3975[us]
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Mode	= Off
Site	= JNM-ECS400	Tri_Domain	= Proton	Tri_Mode	= Off

JEOL RESONANCE



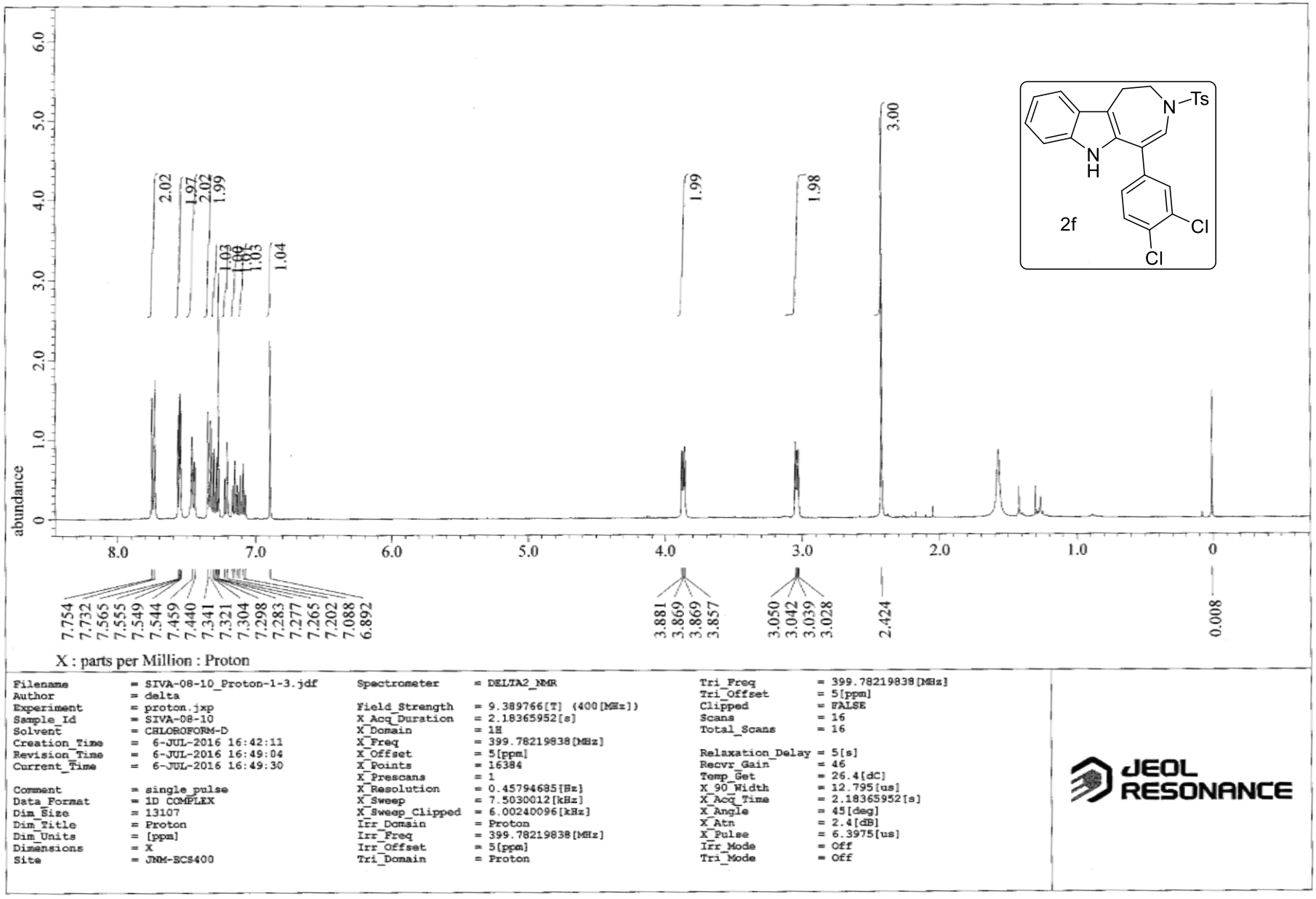
Filename	= SIVA-08-10_Proton-1-3.jdf	Spectrometer	= DELTA2_NMR	Tri_Freq	= 399.78219838[MHz]
Author	= delta			Tri_Offset	= 5[ppm]
Experiment	= proton.jxp	Field_Strength	= 9.389766[T] (400[MHz])	Clipped	= FALSE
Sample_Id	= SIVA-08-10	X_Acq_Duration	= 2.18365952[s]	Scans	= 16
Solvent	= CHLOROFORM-D	X_Domain	= 1H	Total_Scans	= 16
Creation_Time	= 6-JUL-2016 16:42:11	X_Freq	= 399.78219838[MHz]		
Revision_Time	= 6-JUL-2016 16:49:04	X_Offset	= 5[ppm]	Relaxation_Delay	= 5[s]
Current_Time	= 6-JUL-2016 16:49:30	X_Points	= 16384	Recvr_Gain	= 46
		X_Prescans	= 1	Temp_Get	= 26.4[dC]
Comment	= single_pulse	X_Resolution	= 0.45794685[Hz]	X_90_Width	= 12.795[us]
Data_Format	= 1D COMPLEX	X_Sweep	= 7.5030012[kHz]	X_Acq_Time	= 2.18365952[s]
Dim_Size	= 13107	X_Sweep_Clipped	= 6.00240096[kHz]	X_Angle	= 45[deg]
Dim_Title	= Proton	Irr_Domain	= Proton	X_Atn	= 2.4[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	X_Pulse	= 6.3975[us]
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Mode	= Off
Site	= JNM-ECS400	Tri_Domain	= Proton	Tri_Mode	= Off

JEOL RESONANCE



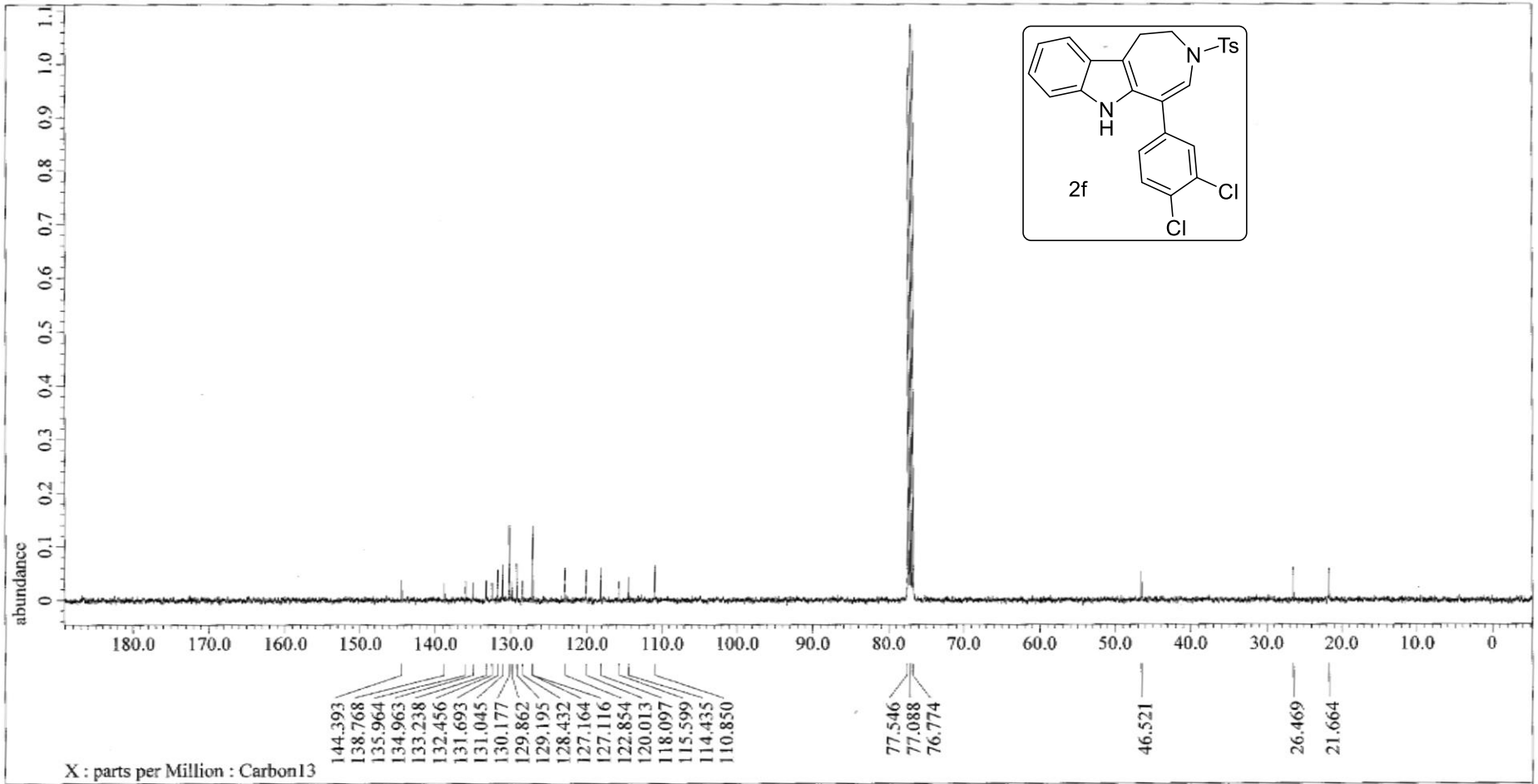
Filename	= Hou-02-12-33_Carbon-1-3.jd	Spectrometer	= DELTA2_NMR	Scans	= 1024
Author	= delta			Total_Scans	= 1024
Experiment	= carbon.jxp	Field_Strength	= 9.389766[T] (400[MHz])		
Sample_Id	= Hou-02-12-33	X_Acq_Duration	= 1.04333312[s]	Relaxation_Delay	= 2[s]
Solvent	= CHLOROFORM-D	X_Domain	= 13C	Recvr_Gain	= 60
Creation_Time	= 7-JUL-2016 14:54:06	X_Freq	= 100.52530333[MHz]	Temp_Get	= 25.9[dC]
Revision_Time	= 7-JUL-2016 15:43:46	X_Offset	= 100[ppm]	X_90_Width	= 10.33[us]
Current_Time	= 7-JUL-2016 15:43:59	X_Points	= 32768	X_Acq_Time	= 1.04333312[s]
		X_Prescans	= 4	X_Angle	= 30[deg]
Comment	= single pulse decoupled gat	X_Resolution	= 0.95846665[Hz]	X_Atn	= 5.3[dB]
Data_Format	= 1D COMPLEX	X_Sweep	= 31.40703518[kHz]	X_Pulse	= 3.44333333[us]
Dim_Size	= 26214	X_Sweep_Clipped	= 25.12562814[kHz]	Irr_Atn_Dec	= 21.473[dB]
Dim_Title	= Carbon13	Irr_Domain	= Proton	Irr_Atn_Noe	= 21.473[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	Irr_Noise	= WALTZ
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Pwidth	= 0.115[ms]
Site	= JNM-ECS400	Clipped	= FALSE	Decoupling	= TRUE

JEOL RESONANCE



Filename	= SIVA-08-10_Proton-1-3.jdf	Spectrometer	= DELTA2_NMR	Tri_Freq	= 399.78219838[MHz]
Author	= delta			Tri_Offset	= 5[ppm]
Experiment	= proton.jxp	Field_Strength	= 9.389766[T] (400[MHz])	Clipped	= FALSE
Sample_Id	= SIVA-08-10	X_Acq_Duration	= 2.18365952[s]	Scans	= 16
Solvent	= CHLOROFORM-D	X_Domain	= 1H	Total_Scans	= 16
Creation_Time	= 6-JUL-2016 16:42:11	X_Freq	= 399.78219838[MHz]		
Revision_Time	= 6-JUL-2016 16:49:04	X_Offset	= 5[ppm]	Relaxation_Delay	= 5[s]
Current_Time	= 6-JUL-2016 16:49:30	X_Points	= 16384	Recvr_Gain	= 46
		X_Prescans	= 1	Temp_Get	= 26.4[dC]
Comment	= single_pulse	X_Resolution	= 0.45794685[Hz]	X_90_Width	= 12.795[us]
Data_Format	= 1D COMPLEX	X_Sweep	= 7.5030012[kHz]	X_Acq_Time	= 2.18365952[s]
Dim_Size	= 13107	X_Sweep_Clipped	= 6.00240096[kHz]	X_Angle	= 45[deg]
Dim_Title	= Proton	Irr_Domain	= Proton	X_Atn	= 2.4[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	X_Pulse	= 6.3975[us]
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Mode	= Off
Site	= JNM-ECS400	Tri_Domain	= Proton	Tri_Mode	= Off

JEOL RESONANCE

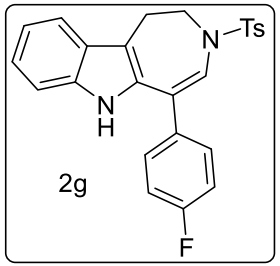


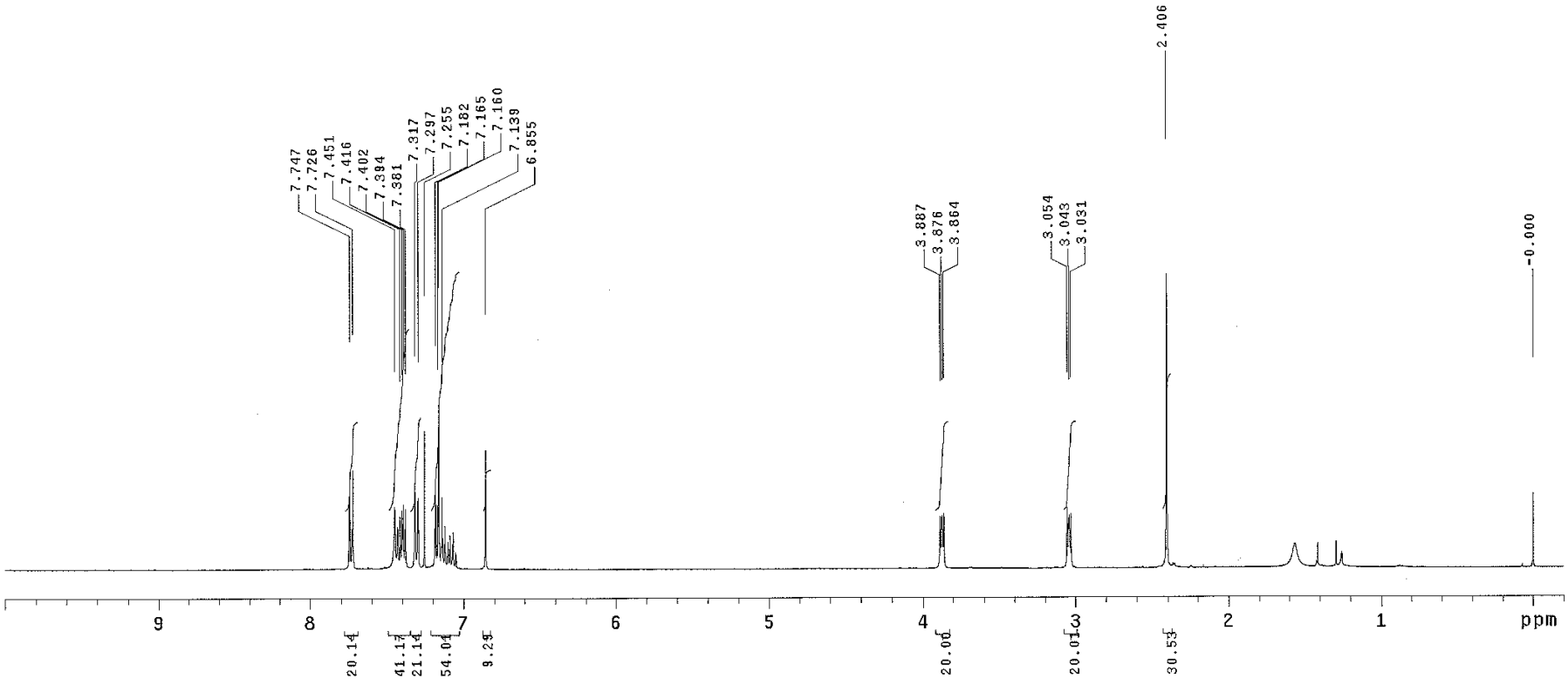
Filename	= SIVA-08-10_Carbon-1-3.jdf	Spectrometer	= DELTA2_NMR	Scans	= 688
Author	= delta			Total_Scans	= 688
Experiment	= carbon.jxp	Field_Strength	= 9.389766[T] (400[MHz])		
Sample_Id	= SIVA-08-10	X_Acq_Duration	= 1.04333312[s]	Relaxation_Delay	= 2[s]
Solvent	= CHLOROFORM-D	X_Domain	= 13C	Recvr_Gain	= 60
Creation_Time	= 6-JUL-2016 16:46:28	X_Freq	= 100.52530333[MHz]	Temp_Get	= 26.6[dC]
Revision_Time	= 6-JUL-2016 17:22:53	X_Offset	= 100[ppm]	X_90_Width	= 10.33[us]
Current_Time	= 6-JUL-2016 17:23:18	X_Points	= 32768	X_Acq_Time	= 1.04333312[s]
		X_Prescans	= 4	X_Angle	= 30[deg]
Comment	= single pulse decoupled gat	X_Resolution	= 0.95846665[Hz]	X_Atn	= 5.3[dB]
Data_Format	= 1D COMPLEX	X_Sweep	= 31.40703518[kHz]	X_Pulse	= 3.44333333[us]
Dim_Size	= 26214	X_Sweep_Clipped	= 25.12562814[kHz]	Irr_Atn_Dec	= 21.473[dB]
Dim_Title	= Carbon13	Irr_Domain	= Proton	Irr_Atn_Noe	= 21.473[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	Irr_Noise	= WALTZ
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Pwidth	= 0.115[ms]
Site	= JNM-ECS400	Clipped	= FALSE	Decoupling	= TRUE

JEOL RESONANCE

SIVA-07-191

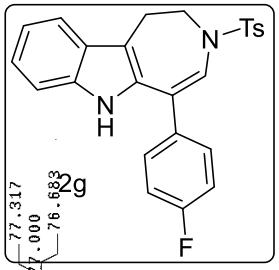
Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Jul 6 2016
Solvent: cdcl3
Ambient temperature
Total 32 repetitions

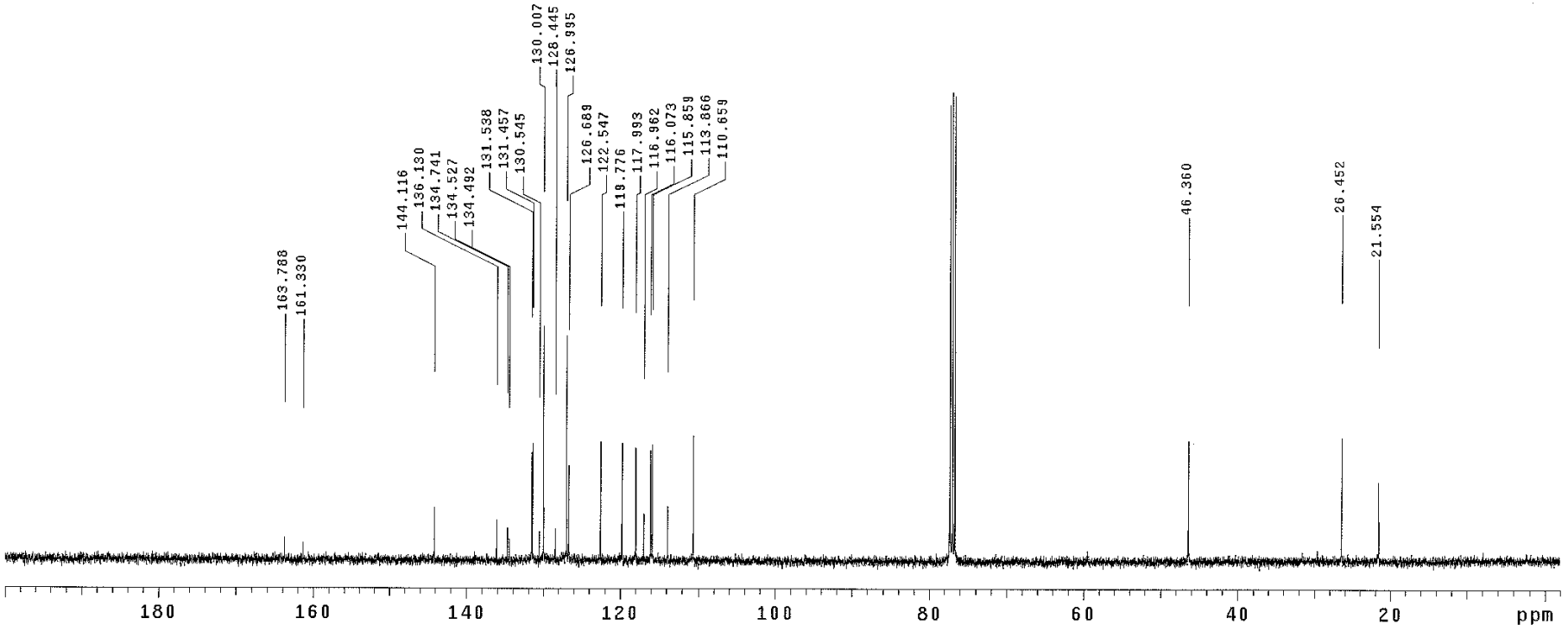


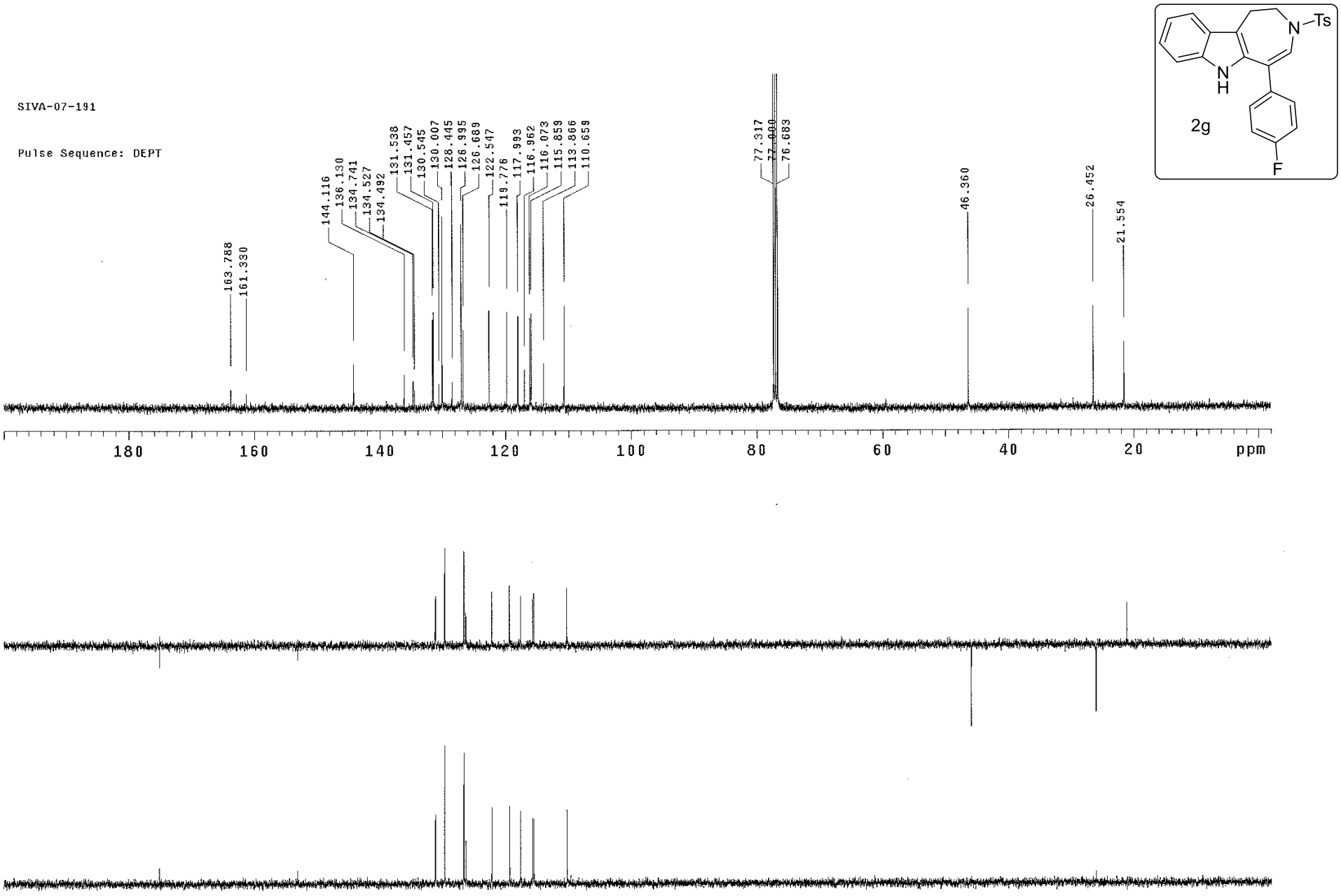


SIVA-07-191

Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Jul 6 2016
Solvent: cdcl3
Ambient temperature
Total 2096 repetitions



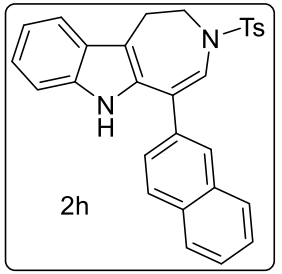


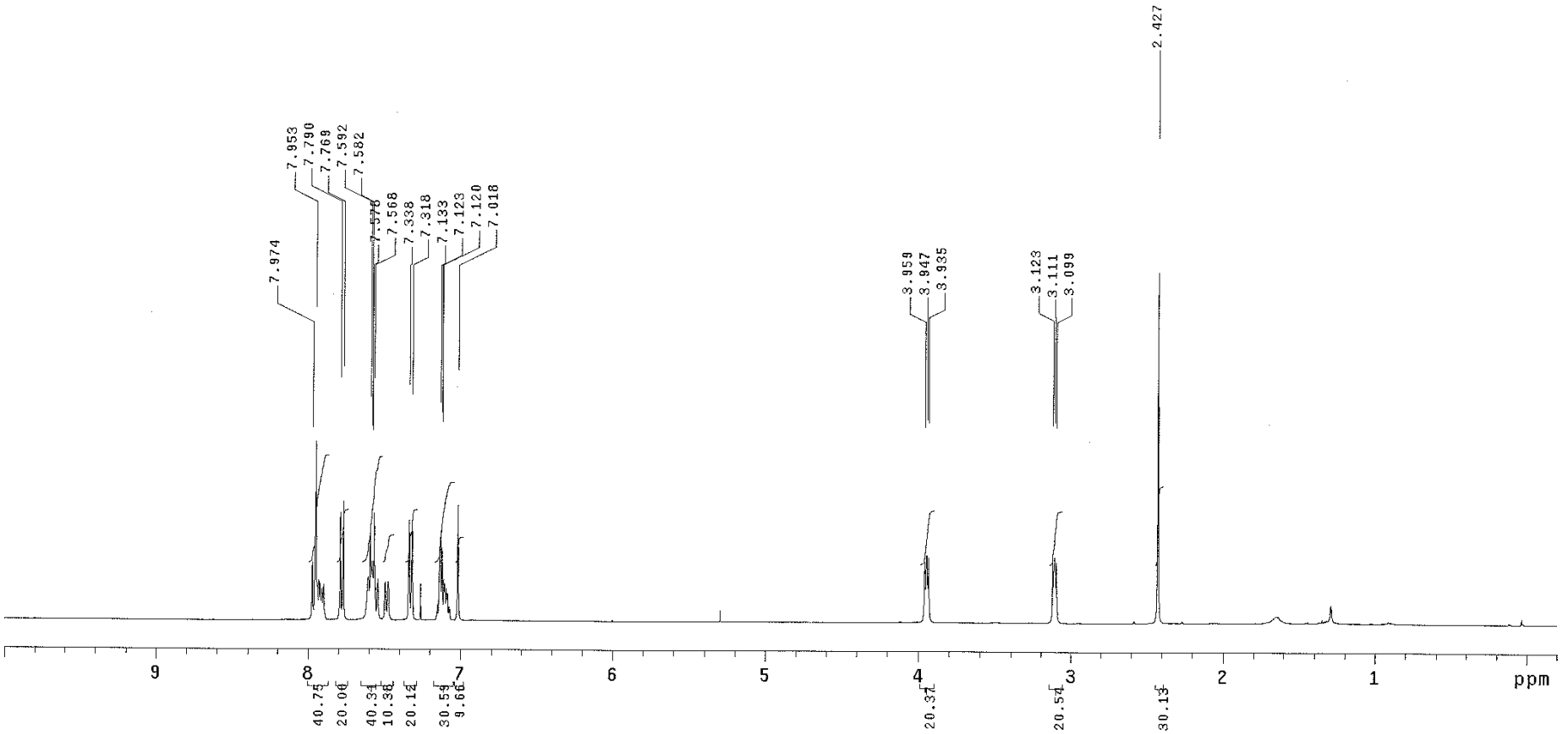

SIVA-07-191
Pulse Sequence: DEPT
163.788
161.330
144.116
136.130
134.741
134.527
134.492
131.538
131.457
130.545
130.007
128.445
126.995
126.689
122.547
119.776
117.993
116.962
116.073
115.859
113.866
110.659
77.317
77.000
76.683
46.360
26.452
21.554
180
160
140
120
100
80
60
40
20
ppm
N
Ts
N
H
2g
F

SIVA-07-184

Pulse Sequence: s2pul

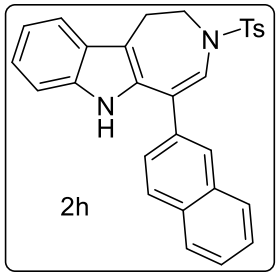
Mercury-400BB "MerPlus400"
Date: Aug 8 2016
Solvent: cdcl3
Ambient temperature
Total 32 repetitions

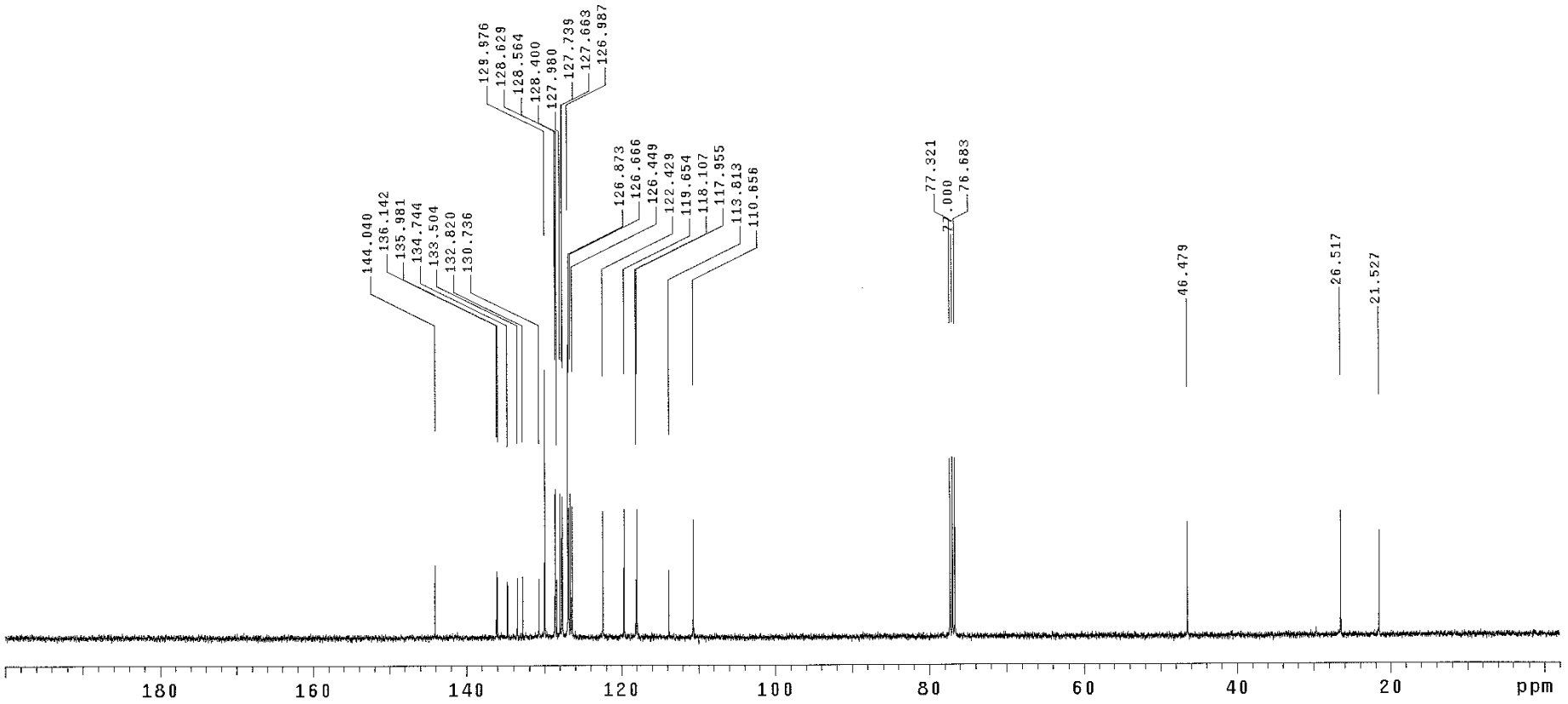


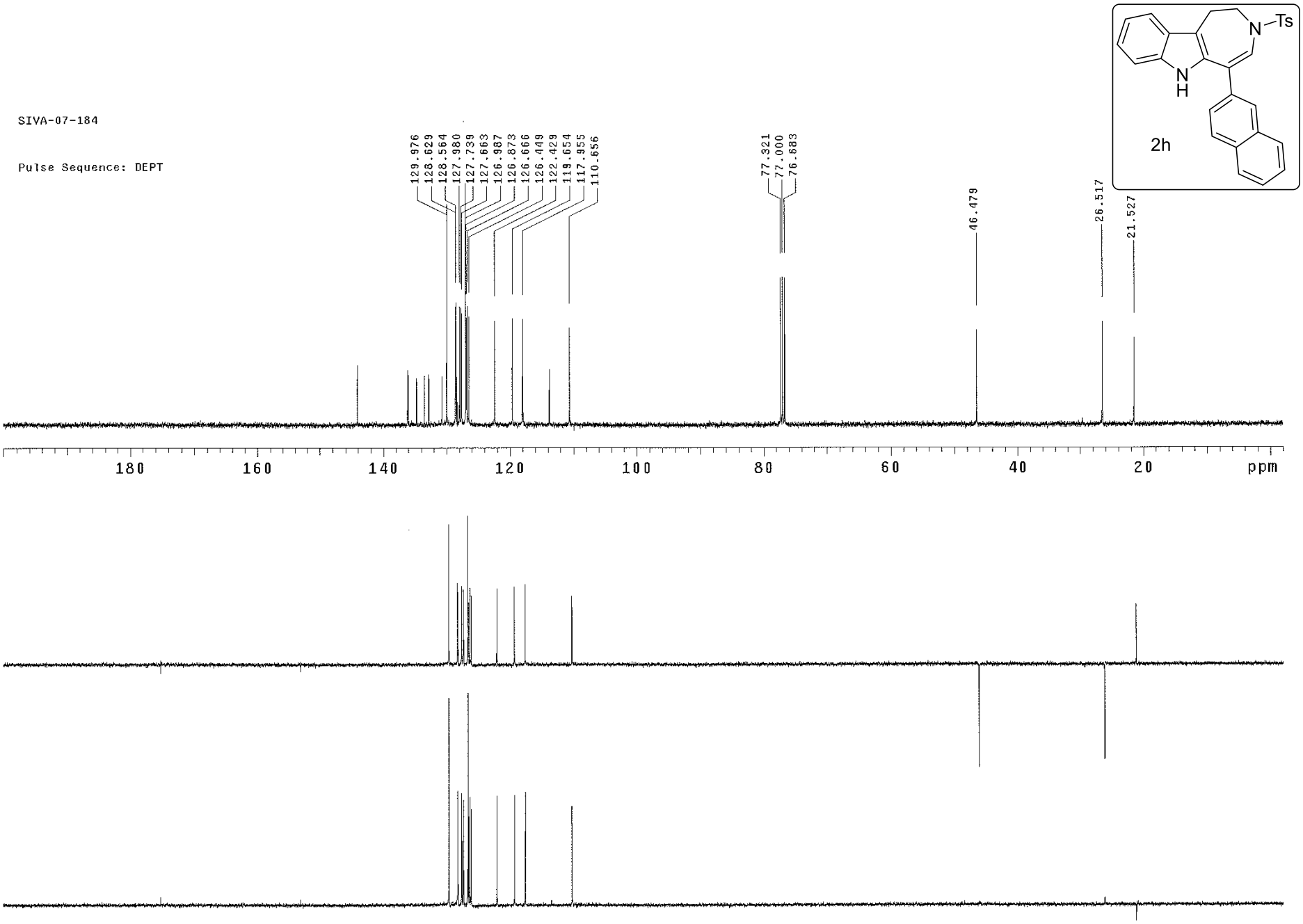


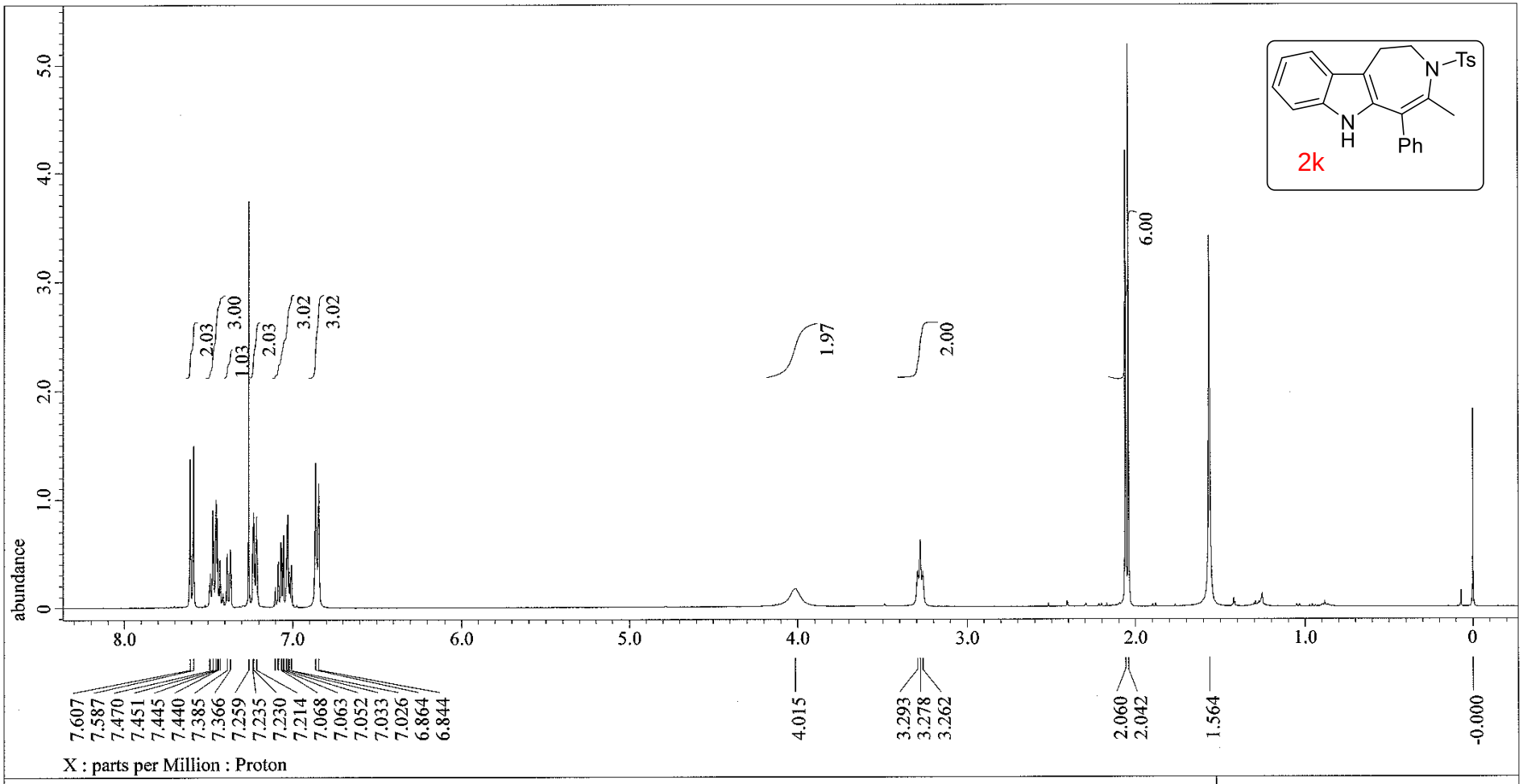
SIVA-07-184

Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Aug 8 2016
Solvent: cdcl3
Ambient temperature
Total 224 repetitions



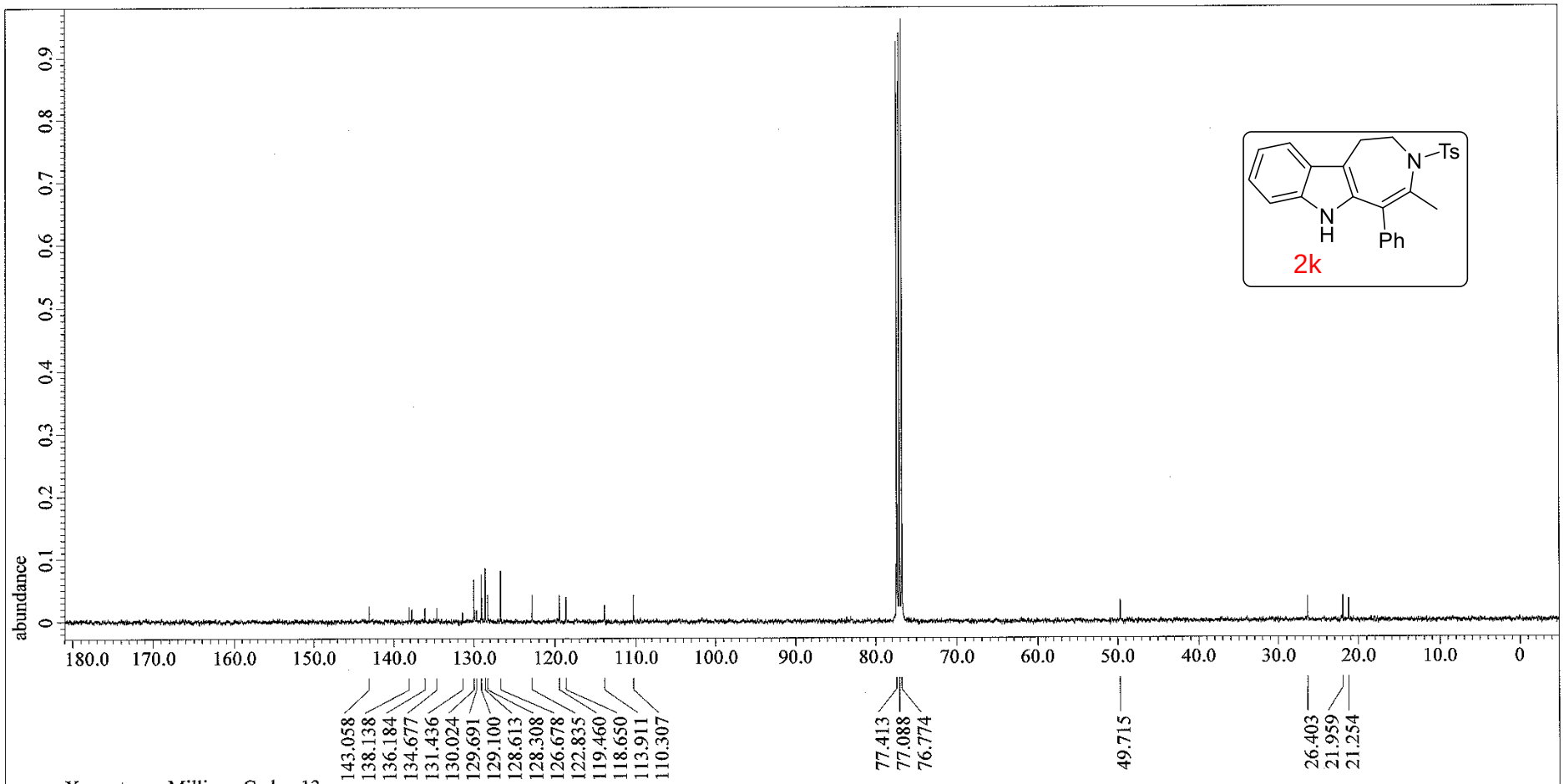



SIVA-07-184
Pulse Sequence: DEPT
129.976
128.629
128.564
127.980
127.739
127.663
126.987
126.873
126.666
126.449
122.429
119.654
117.955
110.656
77.321
77.000
76.683
46.479
26.517
21.527
180
160
140
120
100
80
60
40
20
ppm
N
Ts
N
H
2h



Filename	= Hou-03-39_Proton-1-3.jdf	Spectrometer	= DELTA2_NMR	Tri_Freq	= 399.78219838[MHz]
Author	= delta			Tri_Offset	= 5[ppm]
Experiment	= proton.jxp	Field_Strength	= 9.389766[T] (400[MHz])	Clipped	= FALSE
Sample_Id	= Hou-03-39	X_Acq_Duration	= 2.18365952[s]	Scans	= 37
Solvent	= CHLOROFORM-D	X_Domain	= 1H	Total_Scans	= 37
Creation_Time	= 11-JUL-2016 14:20:25	X_Freq	= 399.78219838[MHz]		
Revision_Time	= 11-JUL-2016 15:39:09	X_Offset	= 5[ppm]	Relaxation_Delay	= 5[s]
Current_Time	= 11-JUL-2016 15:39:17	X_Points	= 16384	Recvr_Gain	= 48
		X_Prescans	= 1	Temp_Get	= 24.4[dC]
Comment	= single_pulse	X_Resolution	= 0.45794685[Hz]	X_90_Width	= 12.795[us]
Data_Format	= 1D COMPLEX	X_Sweep	= 7.5030012[kHz]	X_Acq_Time	= 2.18365952[s]
Dim_Size	= 13107	X_Sweep_Clipped	= 6.00240096[kHz]	X_Angle	= 45[deg]
Dim_Title	= Proton	Irr_Domain	= Proton	X_Atn	= 2.4[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	X_Pulse	= 6.3975[us]
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Mode	= Off
Site	= JNM-ECS400	Tri_Domain	= Proton	Tri_Mode	= Off

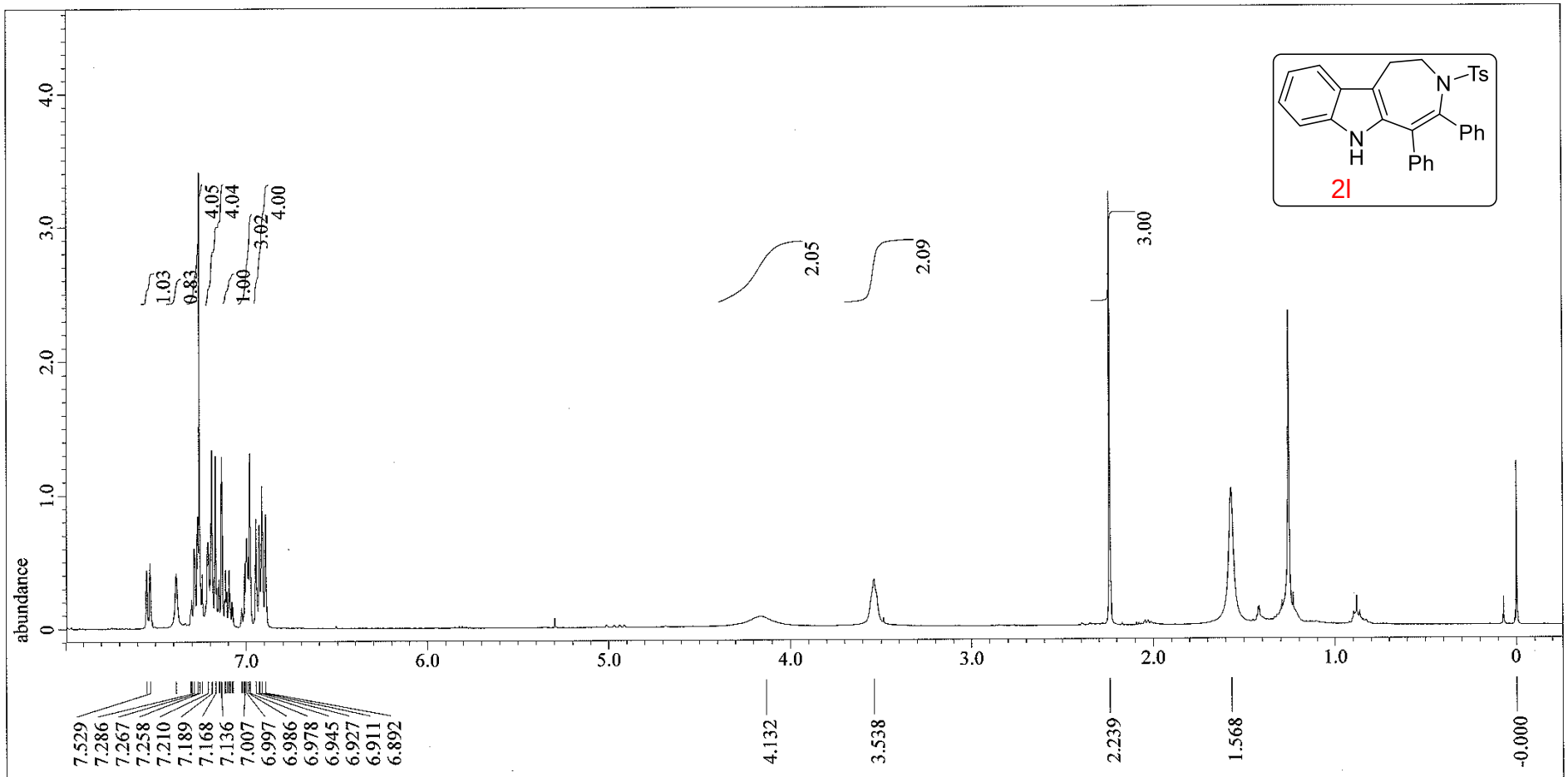
JEOL RESONANCE



X : parts per Million : Carbon13

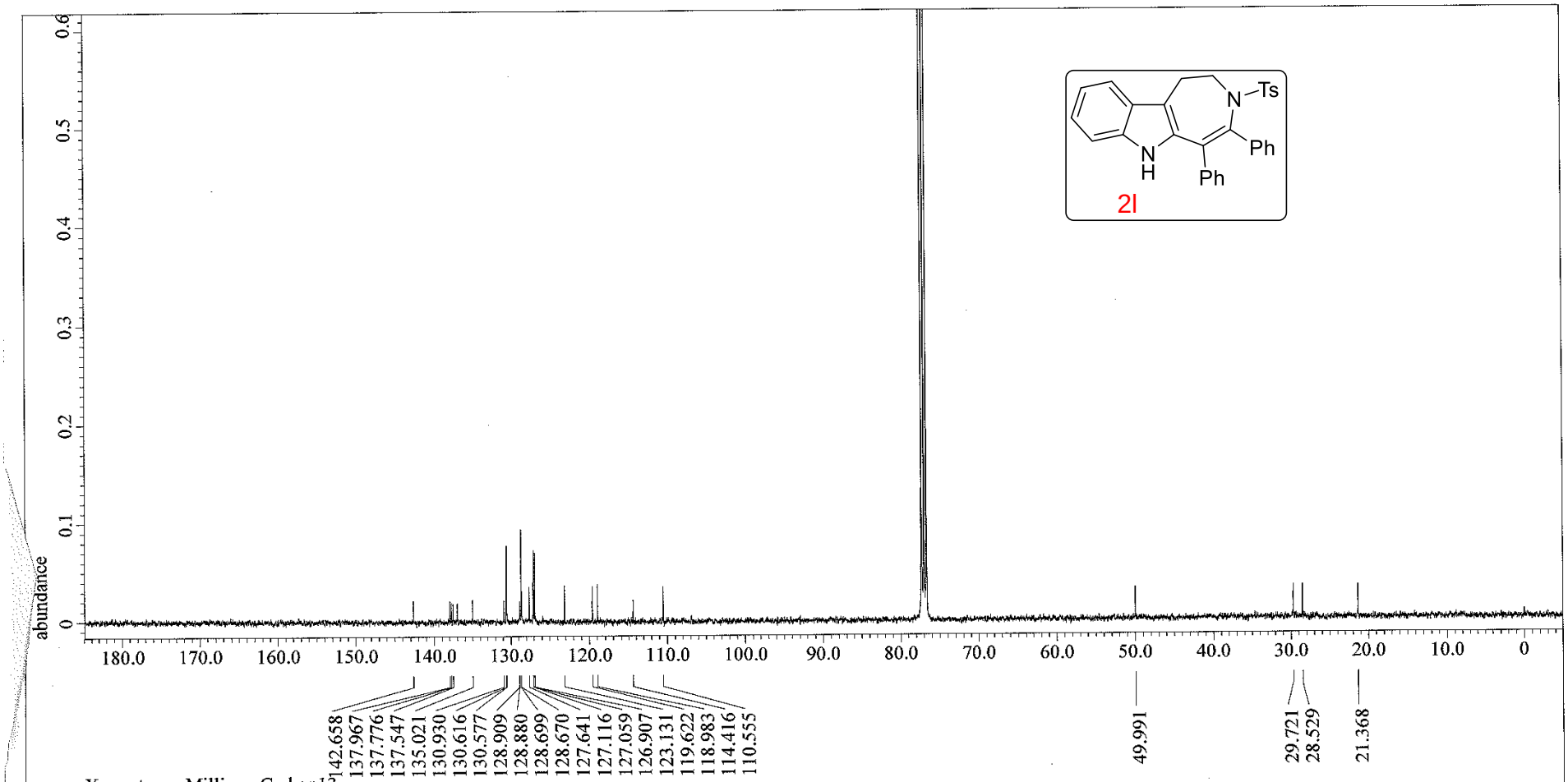
Filename	= Hou-03-39_Carbon-1-3.jdf	Spectrometer	= DELTA2_NMR	Scans	= 1430
Author	= delta			Total_Scans	= 1430
Experiment	= carbon.jxp	Field_Strength	= 9.389766[T] (400[MHz])		
Sample_Id	= Hou-03-39	X_Acq_Duration	= 1.04333312[s]	Relaxation_Delay	= 2[s]
Solvent	= CHLOROFORM-D	X_Domain	= 13C	Recvr_Gain	= 60
Creation_Time	= 11-JUL-2016 14:28:00	X_Freq	= 100.52530333[MHz]	Temp_Get	= 24.6[dC]
Revision_Time	= 11-JUL-2016 15:40:28	X_Offset	= 100[ppm]	X_90_Width	= 10.33[us]
Current_Time	= 11-JUL-2016 15:40:50	X_Points	= 32768	X_Acq_Time	= 1.04333312[s]
		X_Prescans	= 4	X_Angle	= 30[deg]
Comment	= single pulse decoupled gat	X_Resolution	= 0.95846665[Hz]	X_Atn	= 5.3[dB]
Data_Format	= 1D COMPLEX	X_Sweep	= 31.40703518[kHz]	X_Pulse	= 3.44333333[us]
Dim_Size	= 26214	X_Sweep_Clipped	= 25.12562814[kHz]	Irr_Atn_Dec	= 21.473[dB]
Dim_Title	= Carbon13	Irr_Domain	= Proton	Irr_Atn_Noe	= 21.473[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	Irr_Noise	= WALTZ
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Pwidth	= 0.115[ms]
Site	= JNM-ECS400	Clipped	= FALSE	Decoupling	= TRUE

JEOL RESONANCE



Filename	= Hou-03-65_Proton-1-3.jdf	Spectrometer	= DELTA2_NMR	Tri_Freq	= 399.78219838[MHz]
Author	= delta			Tri_Offset	= 5[ppm]
Experiment	= proton.jxp	Field_Strength	= 9.389766[T] (400[MHz])	Clipped	= FALSE
Sample_Id	= Hou-03-65	X_Acq_Duration	= 2.18365952[s]	Scans	= 64
Solvent	= NONE	X_Domain	= 1H	Total_Scans	= 64
Creation_Time	= 11-JUL-2016 15:47:30	X_Freq	= 399.78219838[MHz]		
Revision_Time	= 11-JUL-2016 15:51:53	X_Offset	= 5[ppm]	Relaxation_Delay	= 5[s]
Current_Time	= 11-JUL-2016 15:52:00	X_Points	= 16384	Recvr_Gain	= 48
		X_Prescans	= 1	Temp_Get	= 24.4[dC]
Comment	= single_pulse	X_Resolution	= 0.45794685[Hz]	X_90_Width	= 12.795[us]
Data_Format	= 1D COMPLEX	X_Sweep	= 7.5030012[kHz]	X_Acq_Time	= 2.18365952[s]
Dim_Size	= 13107	X_Sweep_Clipped	= 6.00240096[kHz]	X_Angle	= 45[deg]
Dim_Title	= Proton	Irr_Domain	= Proton	X_Atn	= 2.4[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	X_Pulse	= 6.3975[us]
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Mode	= Off
Site	= JNM-ECS400	Tri_Domain	= Proton	Tri_Mode	= Off

JEOL RESONANCE



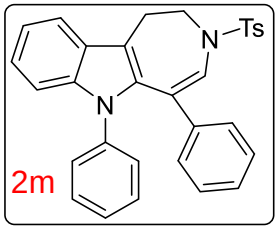
X : parts per Million : Carbon13

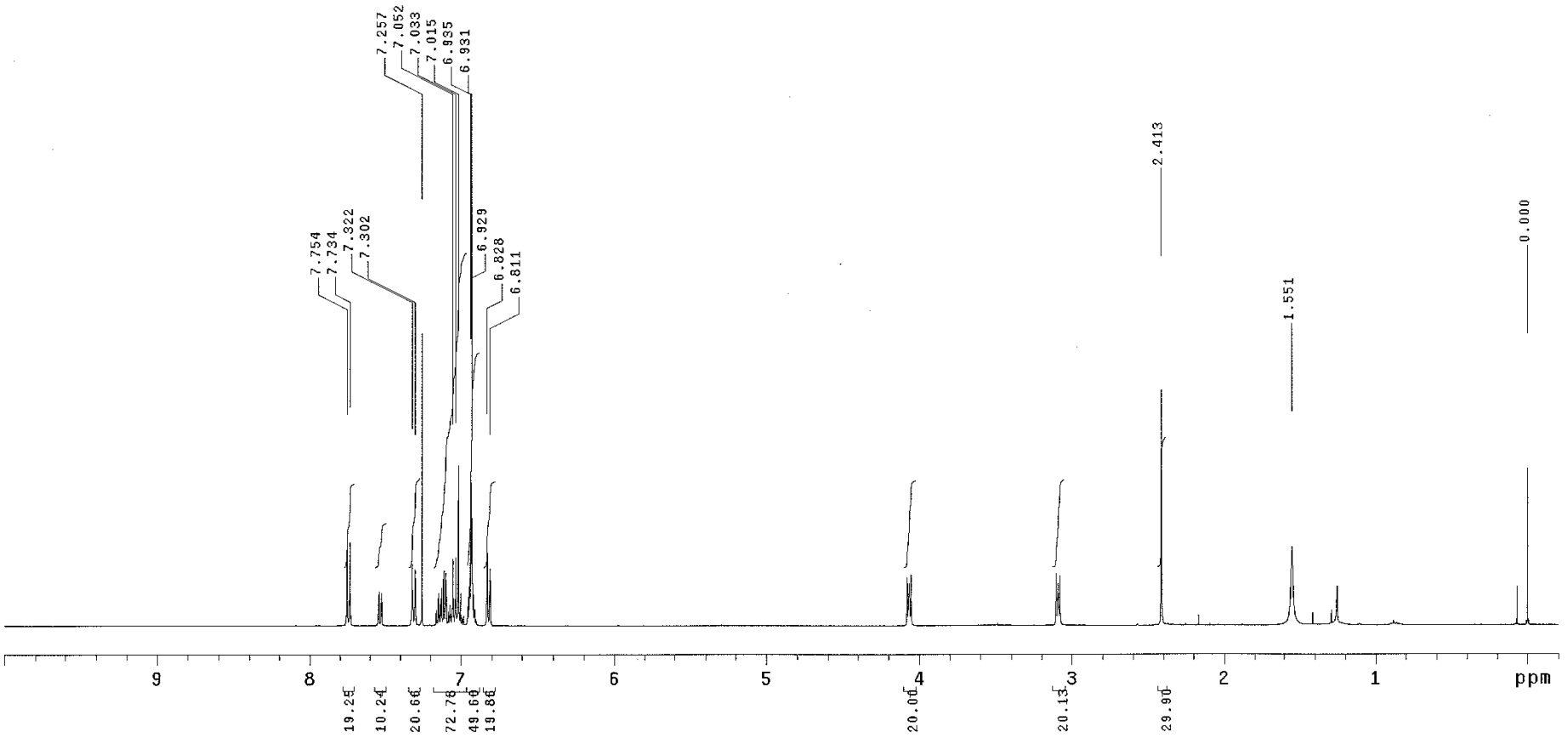
Filename	= Hou-03-65_Carbon-1-3.jdf	Spectrometer	= DELTA2_NMR	Scans	= 2292
Author	= delta			Total_Scans	= 2292
Experiment	= carbon.jxp	Field_Strength	= 9.389766[T] (400[MHz])		
Sample_Id	= Hou-03-65	X_Acq_Duration	= 1.04333312[s]	Relaxation_Delay	= 2[s]
Solvent	= NONE	X_Domain	= 13C	Recvr_Gain	= 60
Creation_Time	= 11-JUL-2016 15:58:26	X_Freq	= 100.52530333[MHz]	Temp_Get	= 24.4[dC]
Revision_Time	= 11-JUL-2016 17:49:36	X_Offset	= 100[ppm]	X_90_Width	= 10.33[us]
Current_Time	= 11-JUL-2016 17:49:57	X_Points	= 32768	X_Acq_Time	= 1.04333312[s]
		X_Prescans	= 4	X_Angle	= 30[deg]
Comment	= single pulse decoupled gat	X_Resolution	= 0.95846665[Hz]	X_Atn	= 5.3[dB]
Data_Format	= 1D COMPLEX	X_Sweep	= 31.40703518[kHz]	X_Pulse	= 3.44333333[us]
Dim_Size	= 26214	X_Sweep_Clipped	= 25.12562814[kHz]	Irr_Atn_Dec	= 21.473[dB]
Dim_Title	= Carbon13	Irr_Domain	= Proton	Irr_Atn_Noe	= 21.473[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	Irr_Noise	= WALTZ
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Pwidth	= 0.115[ms]
Site	= JNM-ECS400	Clipped	= FALSE	Decoupling	= TRUE

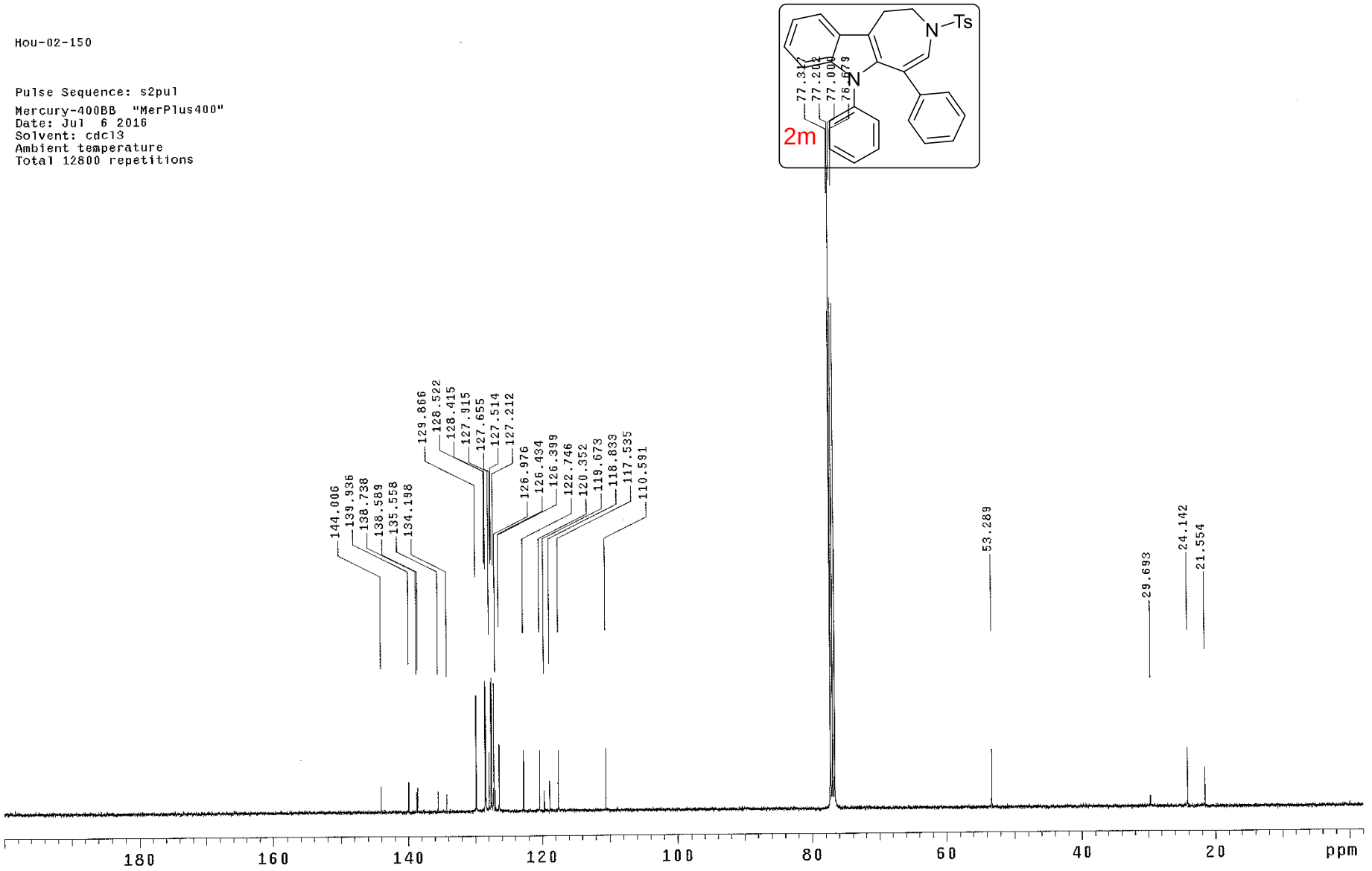
JEOL RESONANCE

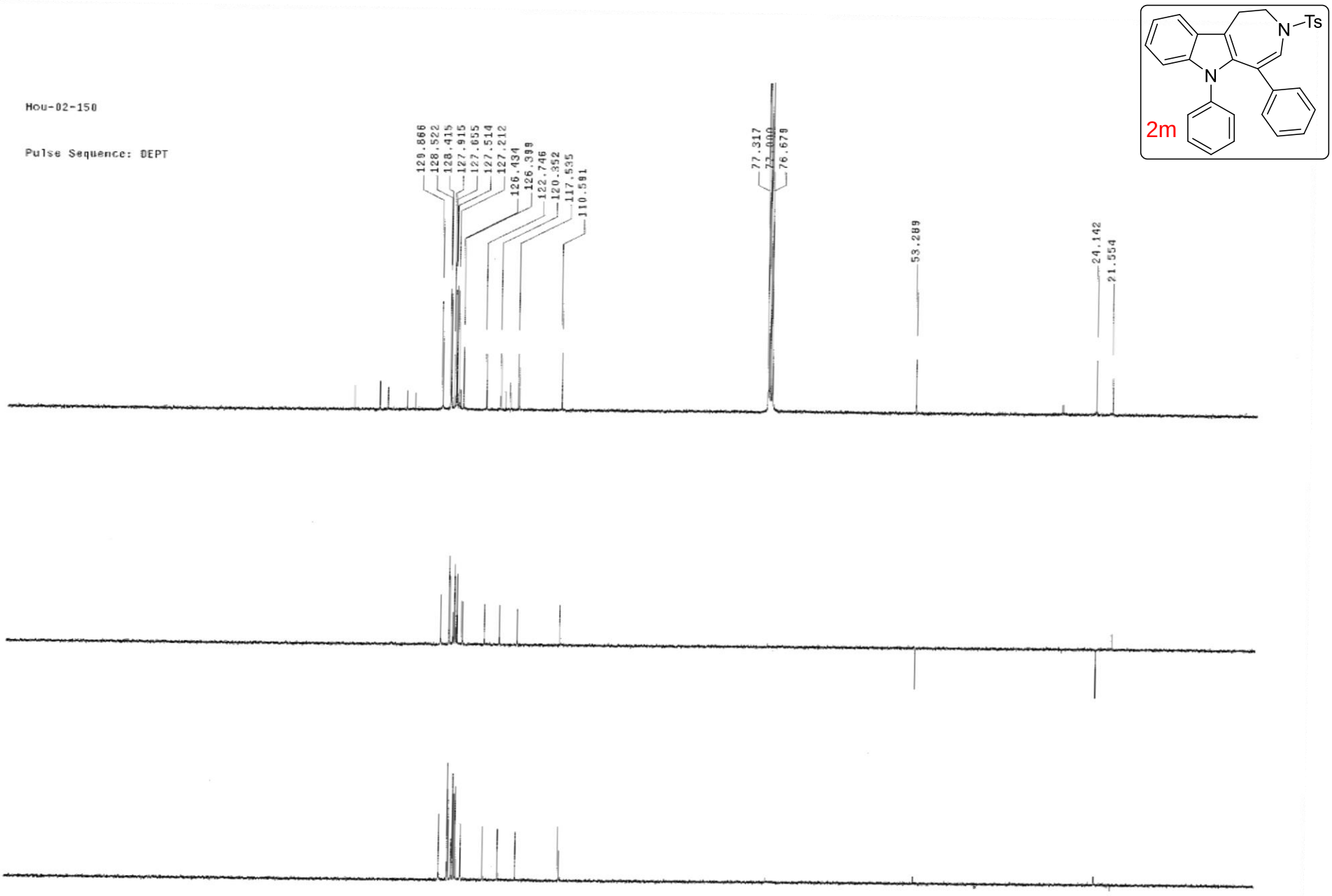
Hou-02-150

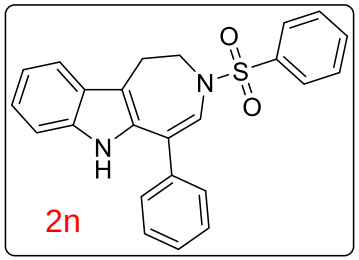
Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Jul 6 2016
Solvent: cdcl3
Ambient temperature
Total 160 repetitions






Hou-02-150
Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Jul 6 2016
Solvent: cdcl3
Ambient temperature
Total 12800 repetitions
N
Ts
N
2m
77.317
77.203
77.000
76.679
144.006
139.936
138.738
138.589
135.558
134.198
129.866
128.522
128.415
127.915
127.655
127.514
127.212
126.976
126.434
126.399
122.746
120.352
119.673
118.833
117.535
110.591
53.289
29.693
24.142
21.554
180
160
140
120
100
80
60
40
20
ppm

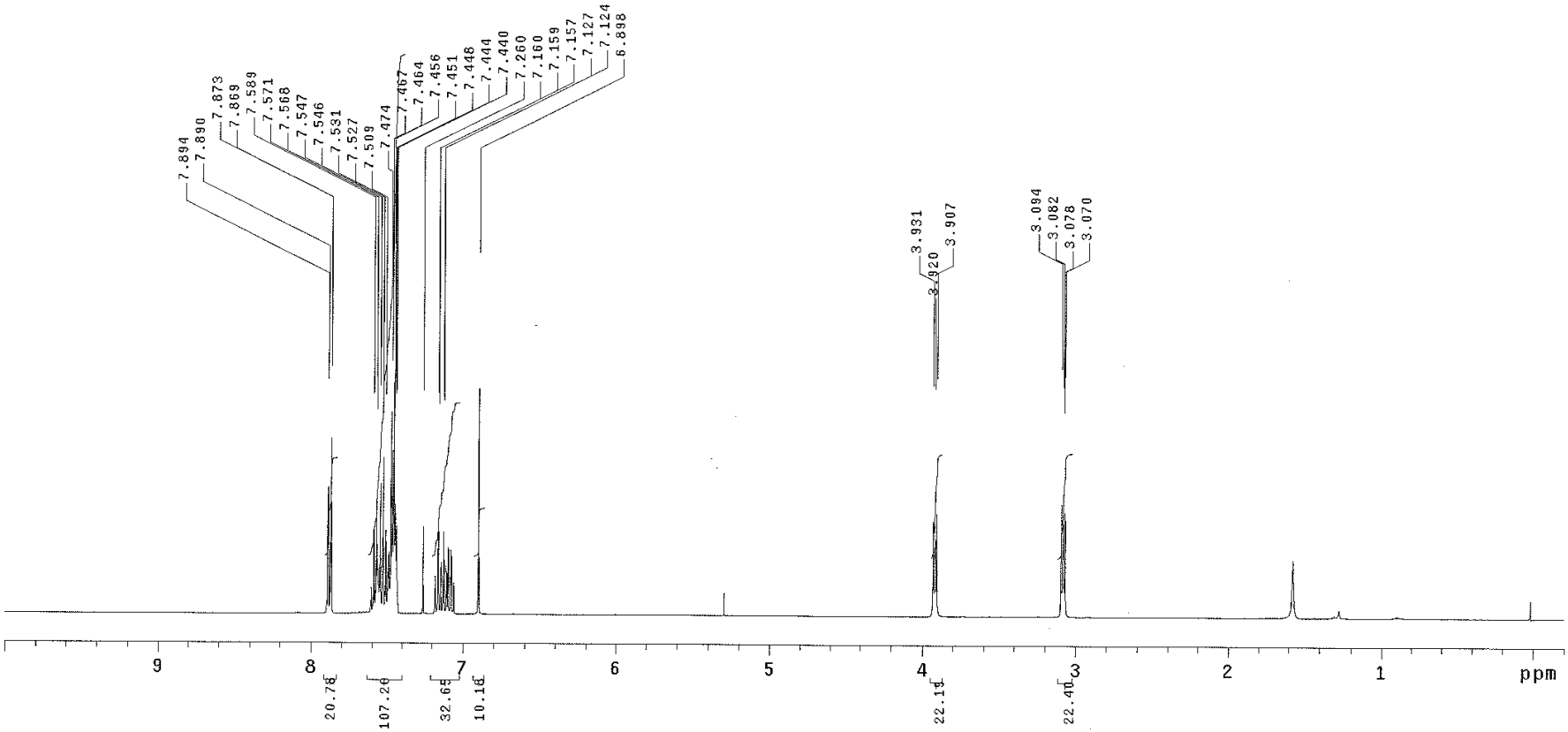

Hou-02-150
Pulse Sequence: DEPT
120.866
128.522
128.415
127.915
127.655
127.514
127.212
126.434
126.398
122.746
120.352
117.535
110.591
77.317
77.000
76.679
53.289
24.142
21.554
N
Ts
N
2m



Hou-02-154

Pulse Sequence: s2pul

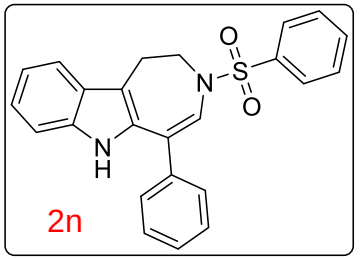
Mercury-400BB "MerPlus400"
Date: Jun 28 2016
Solvent: cdcl3
Ambient temperature
Total 32 repetitions

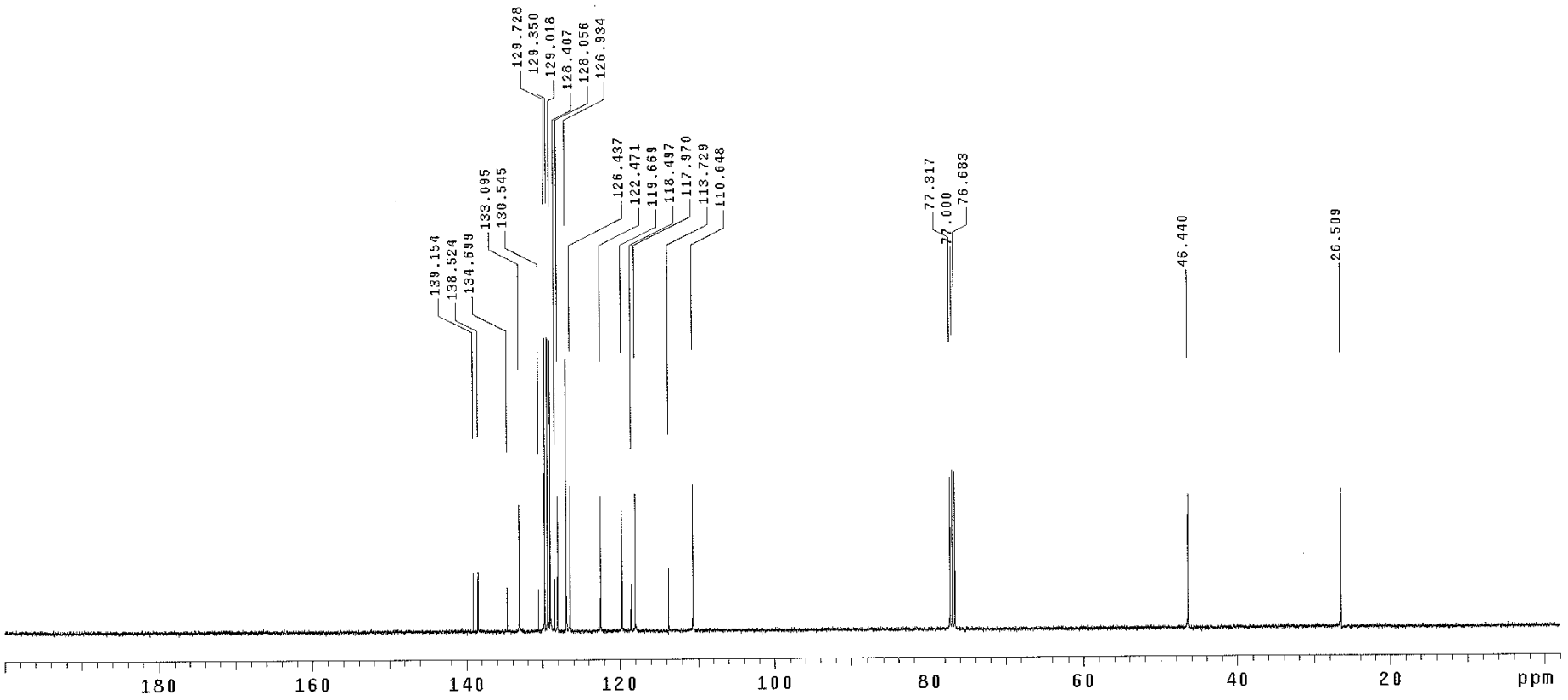


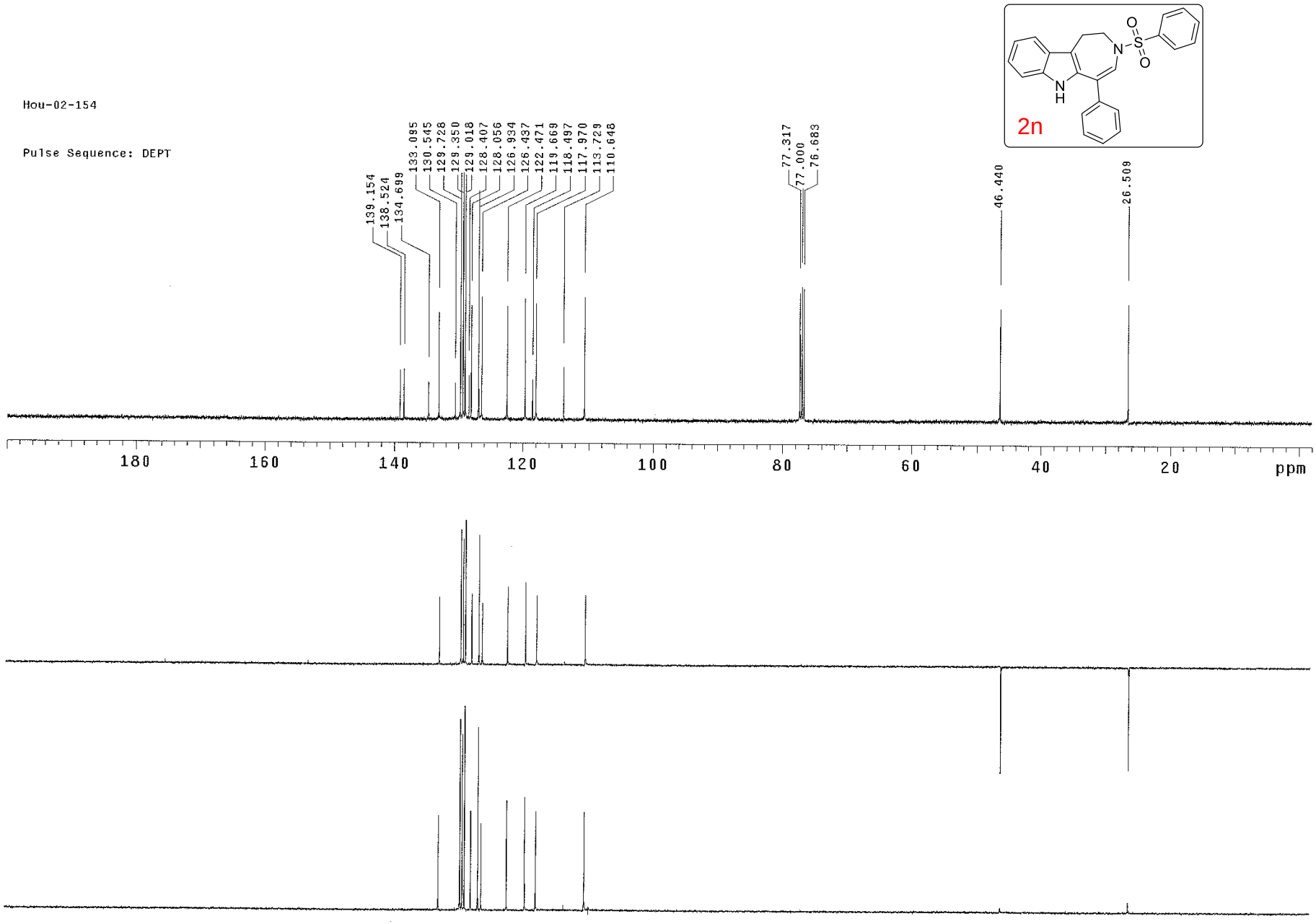
Hou-02-154

Pulse Sequence: s2pul

Mercury-400BB "MerPlus400"
Date: Jun 28 2016
Solvent: cdcl3
Ambient temperature
Total 192 repetitions

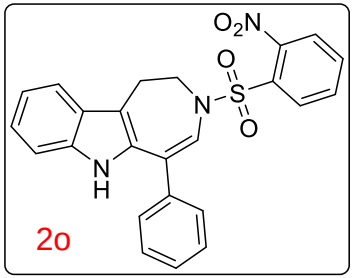




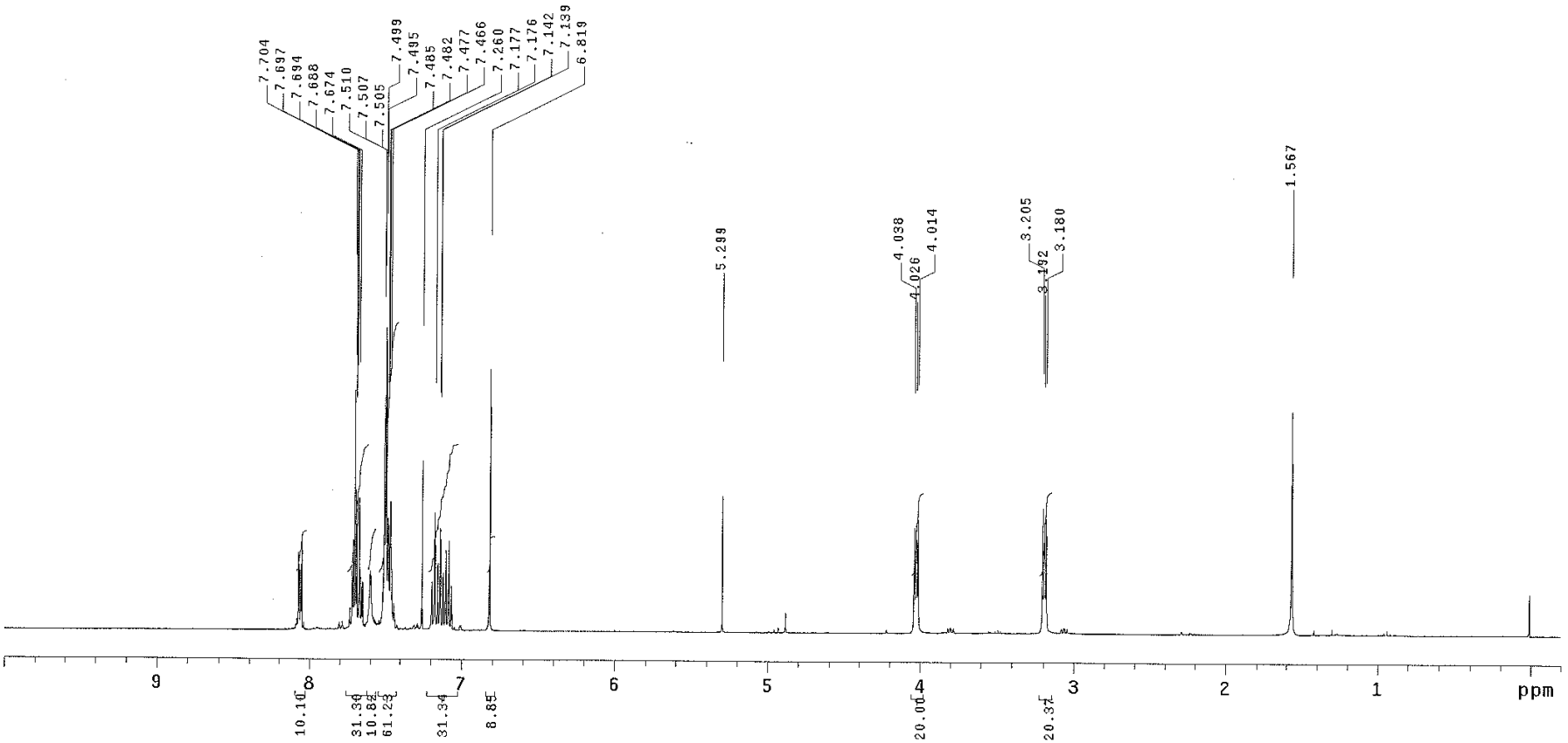

Hou-02-154
Pulse Sequence: DEPT
139.154
138.524
134.699
133.085
130.545
129.728
129.350
129.018
128.407
128.056
126.934
126.437
122.471
119.669
118.497
117.970
113.729
110.648
77.317
77.000
76.683
46.440
26.509
180
160
140
120
100
80
60
40
20
ppm
2n

Hou-02-161

Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Jun 28 2016
Solvent: cdcl3
Ambient temperature
Total 32 repetitions



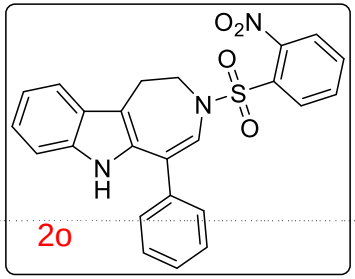
2o



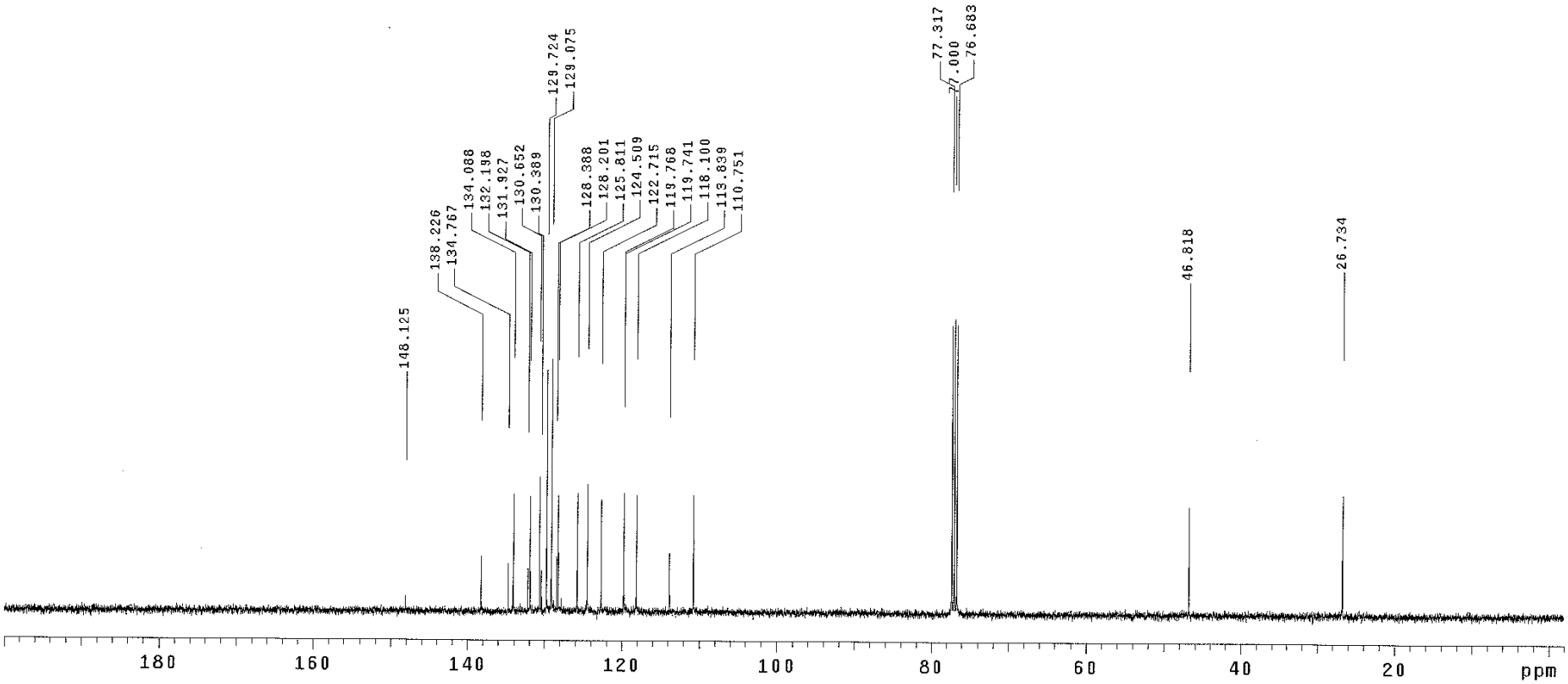
Hou-02-161

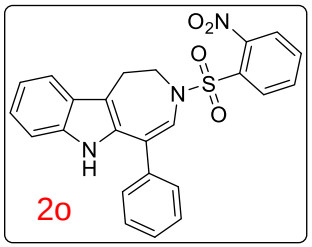
Pulse Sequence: s2pul

Mercury-400BB "MerPlus400"
Date: Jun 28 2016
Solvent: cdcl3
Ambient temperature
Total 640 repetitions



2o





Hou-02-161

Pulse Sequence: DEPT

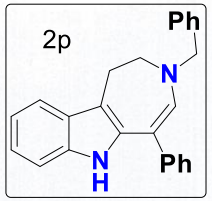
148.125
138.226
134.767
134.088
132.198
131.927
130.652
130.389
129.724
129.075
128.388
128.201
125.811
124.509
122.715
119.768
119.741
118.100
113.839
110.751
77.317
77.000
76.683
46.818
26.734

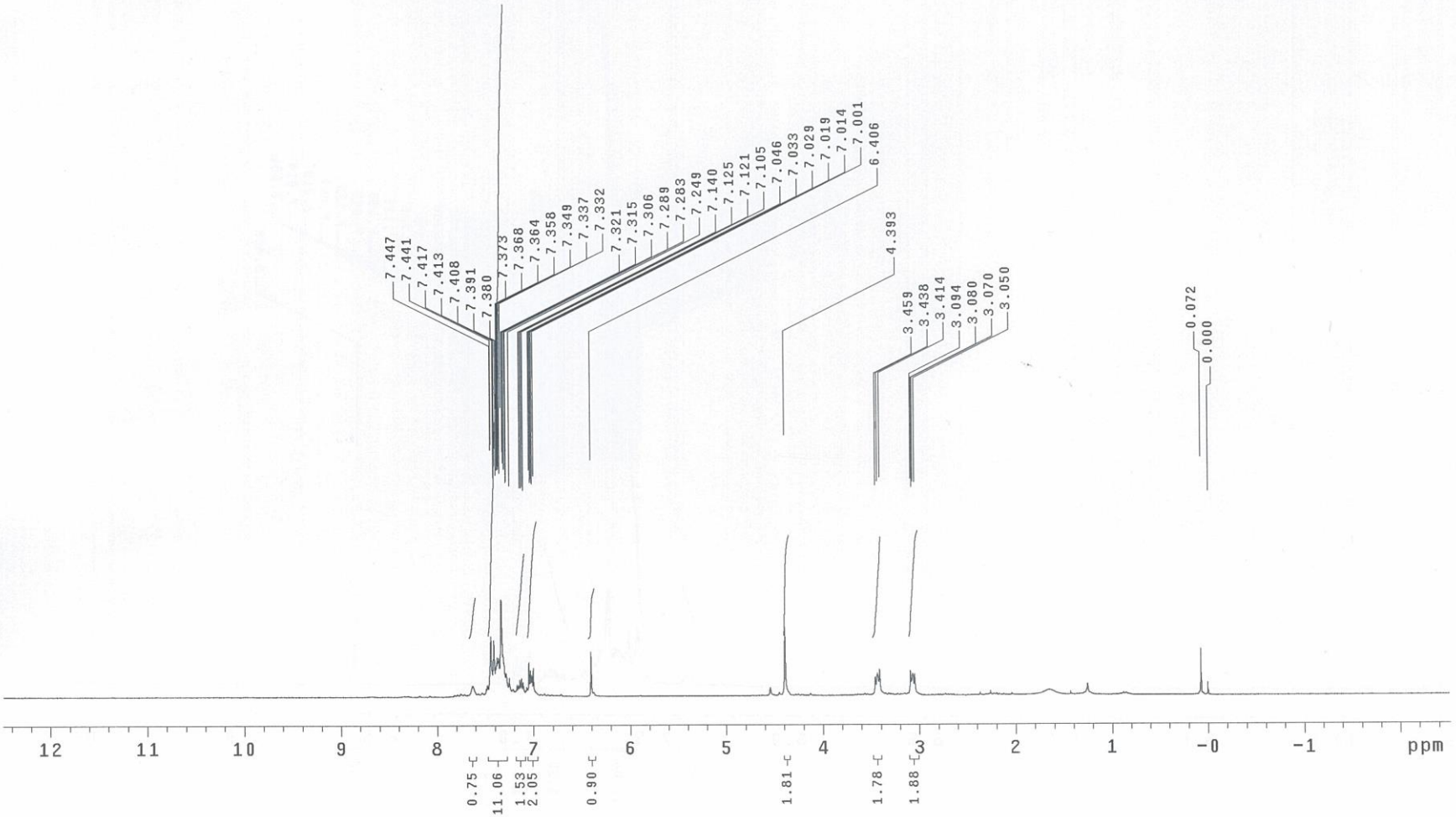
180 160 140 120 100 80 60 40 20 ppm

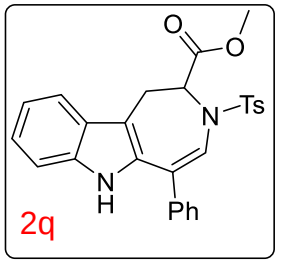
STANDARD 1H OBSERVE

Solvent: CDCl3
Ambient temperature
GEMINI-200 "oxford200"

Pulse 45.2 degrees
Acq. time 3.002 sec
Width 3000.3 Hz
72 repetitions
OBSERVE H1, 199.9678693 MHz
DATA PROCESSING
FT size 32768
Total time 2 hr, 59 min, 24 sec



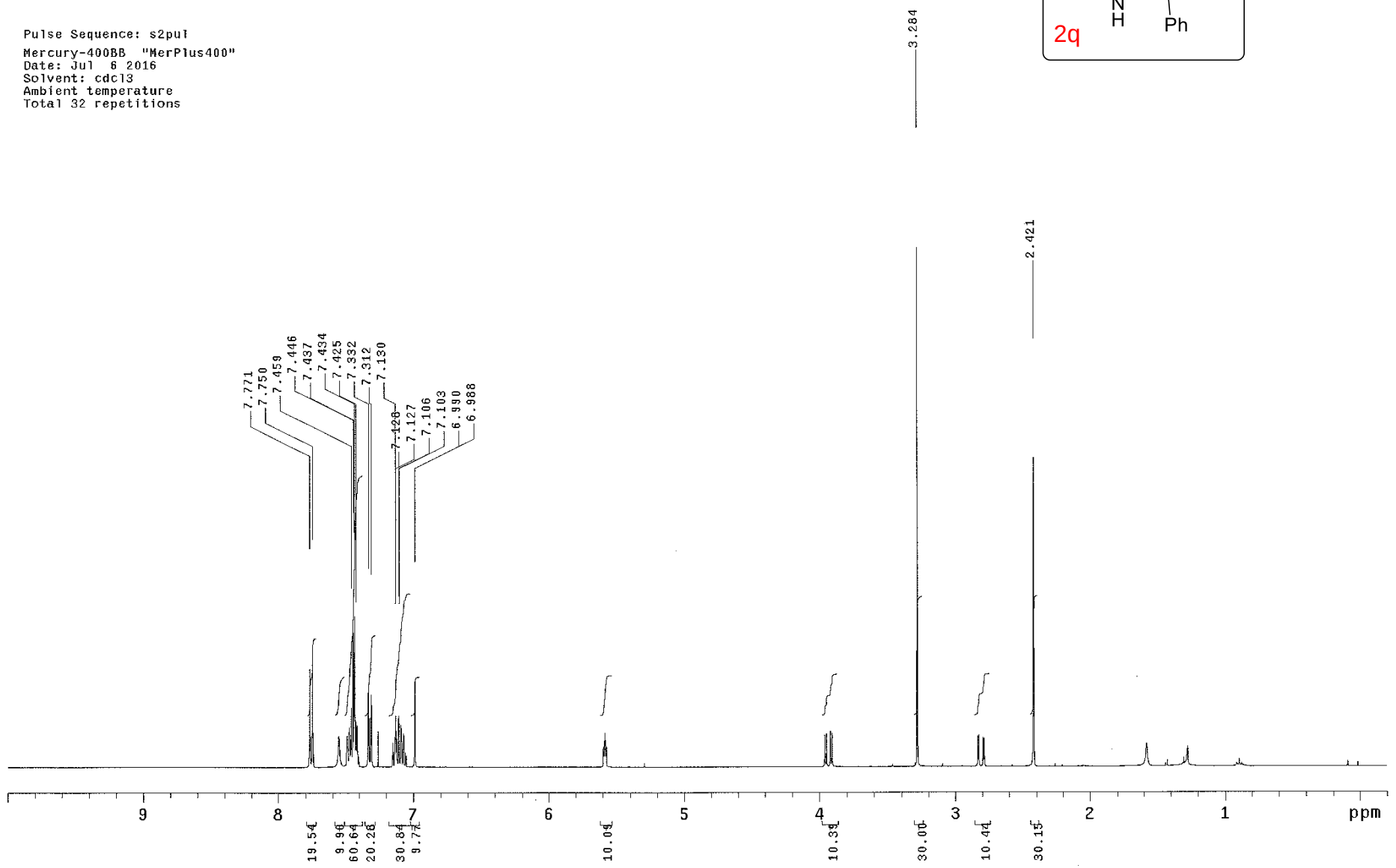


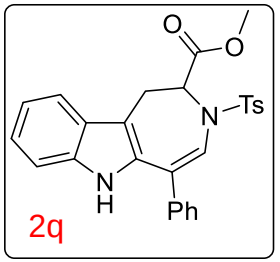


Hou-03-9

Pulse Sequence: s2pul

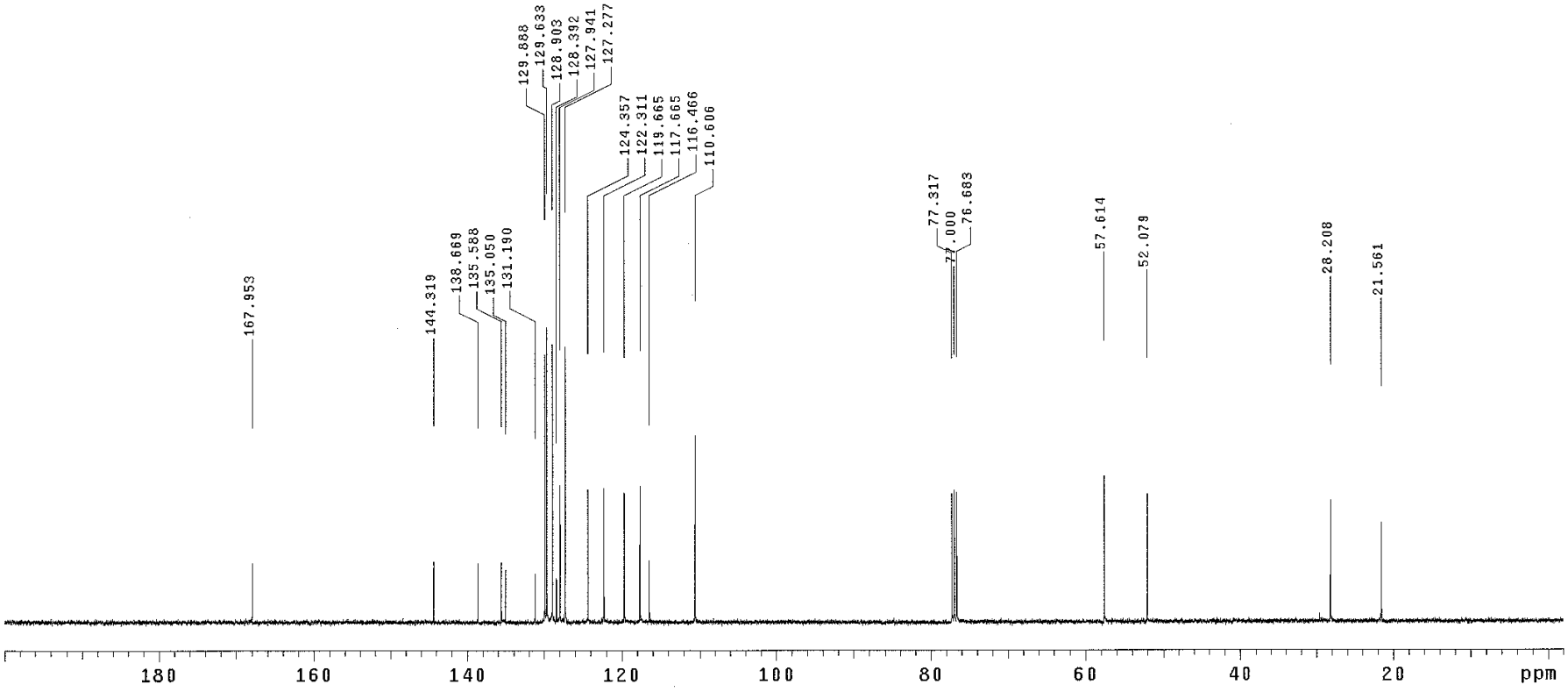
Mercury-400BB "MerPlus400"
Date: Jul 6 2016
Solvent: cdcl3
Ambient temperature
Total 32 repetitions

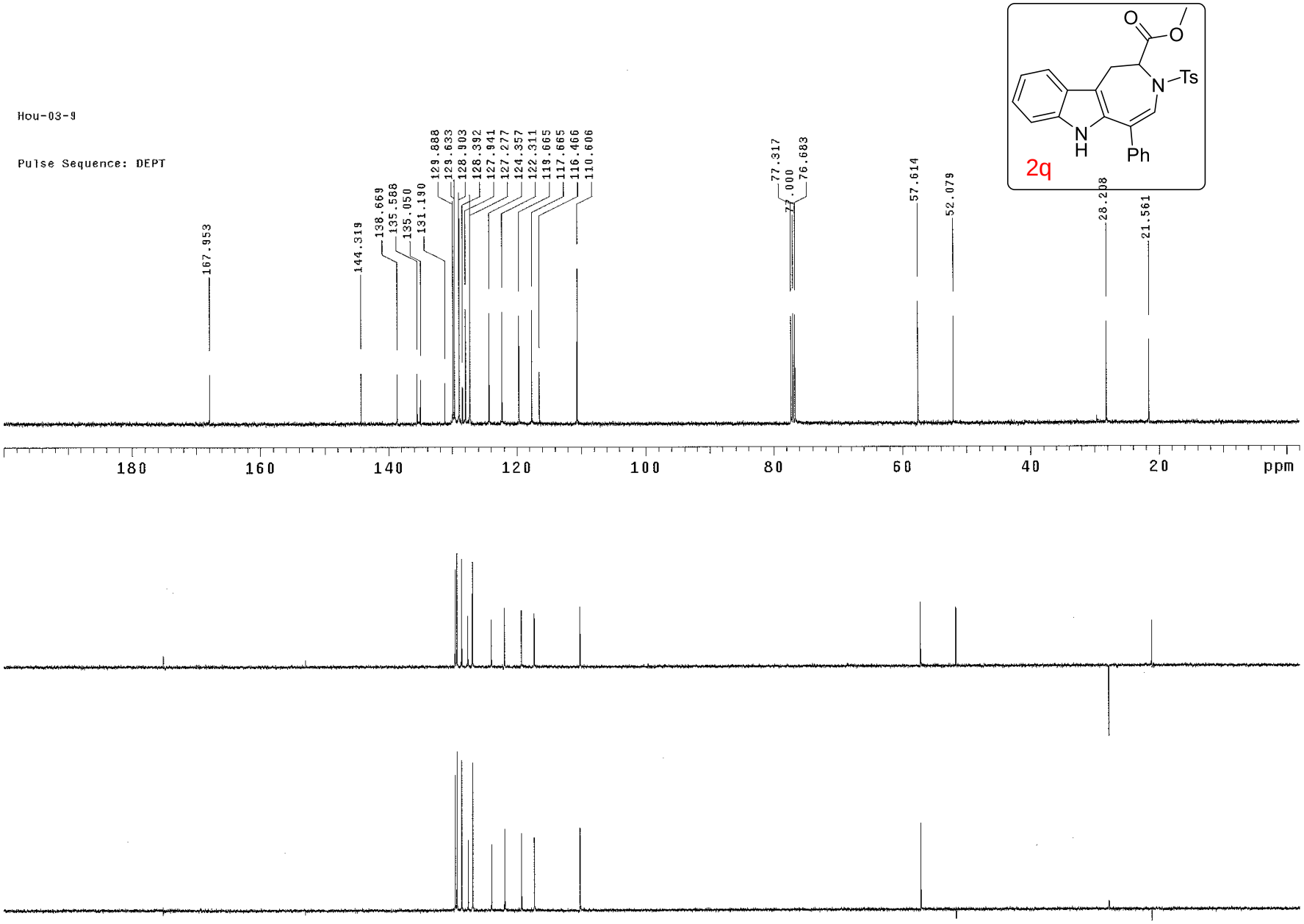




Hou-03-9

Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Jul 6 2016
Solvent: cdcl3
Ambient temperature
Total 640 repetitions

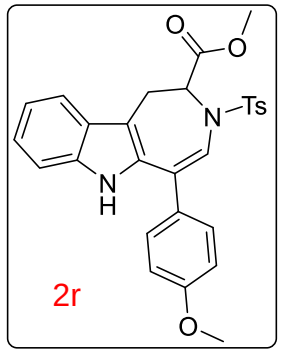



Hou-03-9
Pulse Sequence: DEPT
167.953
144.319
138.669
135.588
135.050
131.190
129.888
129.633
128.903
128.392
127.941
127.277
124.357
122.311
119.665
117.665
116.466
110.606
77.317
77.000
76.683
57.614
52.079
28.208
21.561
180
160
140
120
100
80
60
40
20
ppm
O
O
N
Ts
N
H
Ph
2q

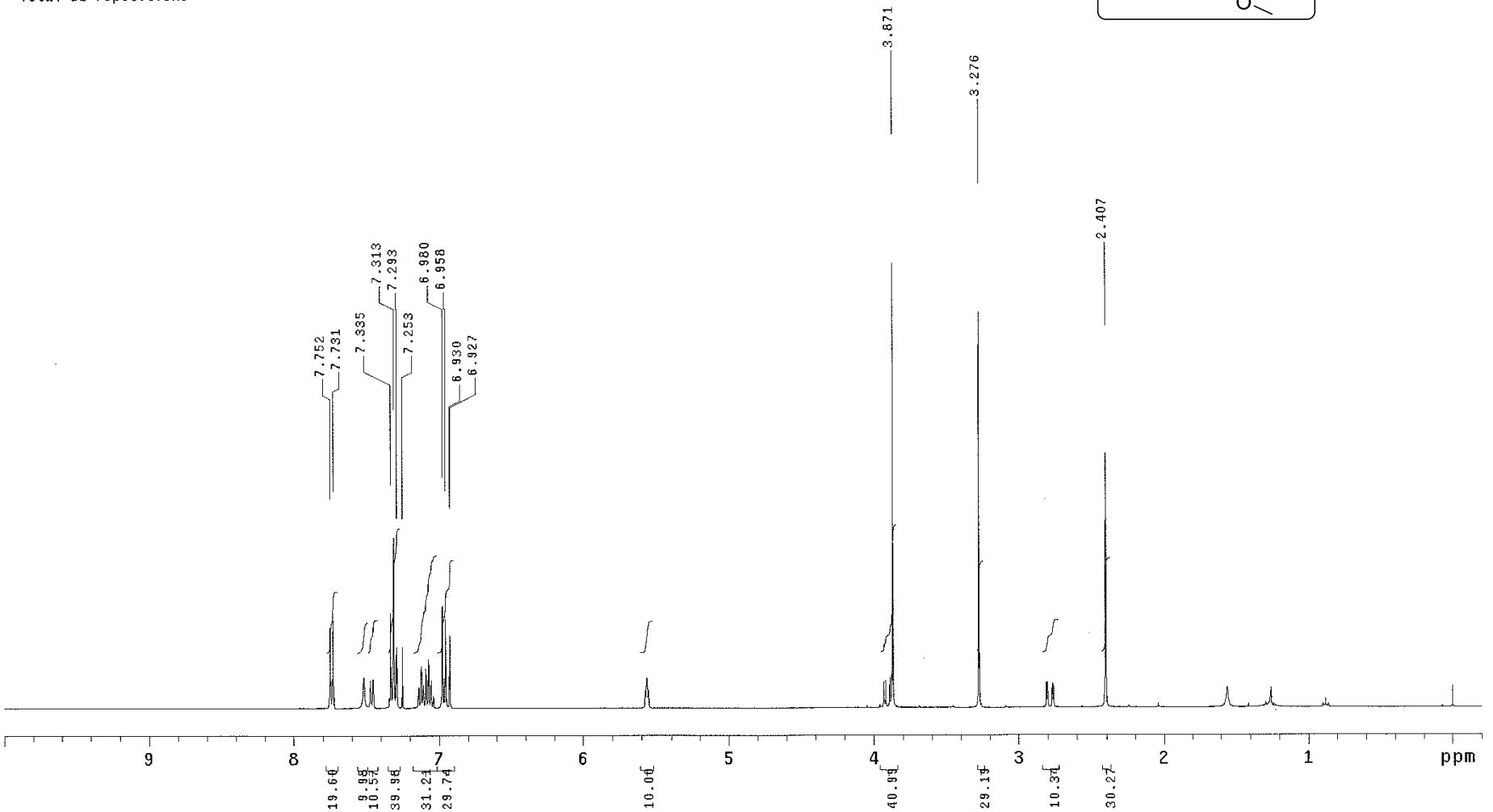
Hou-03-18

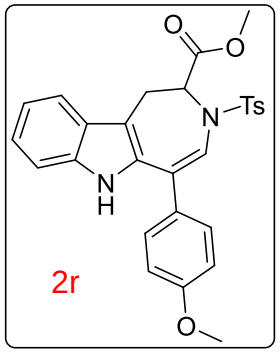
Pulse Sequence: s2pul

Mercury-400BB "MerPlus400"
Date: Jun 30 2016
Solvent: cdcl3
Ambient temperature
Total 32 repetitions



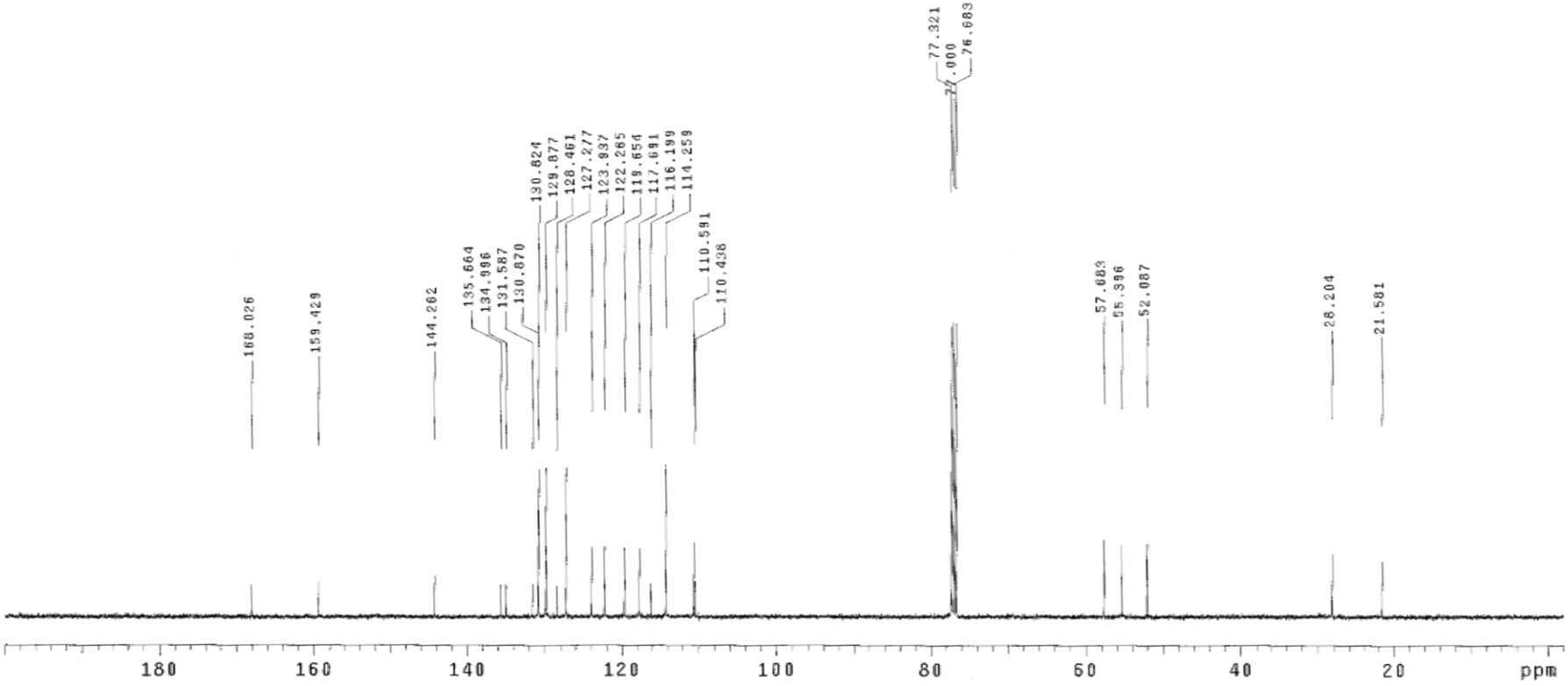
2r

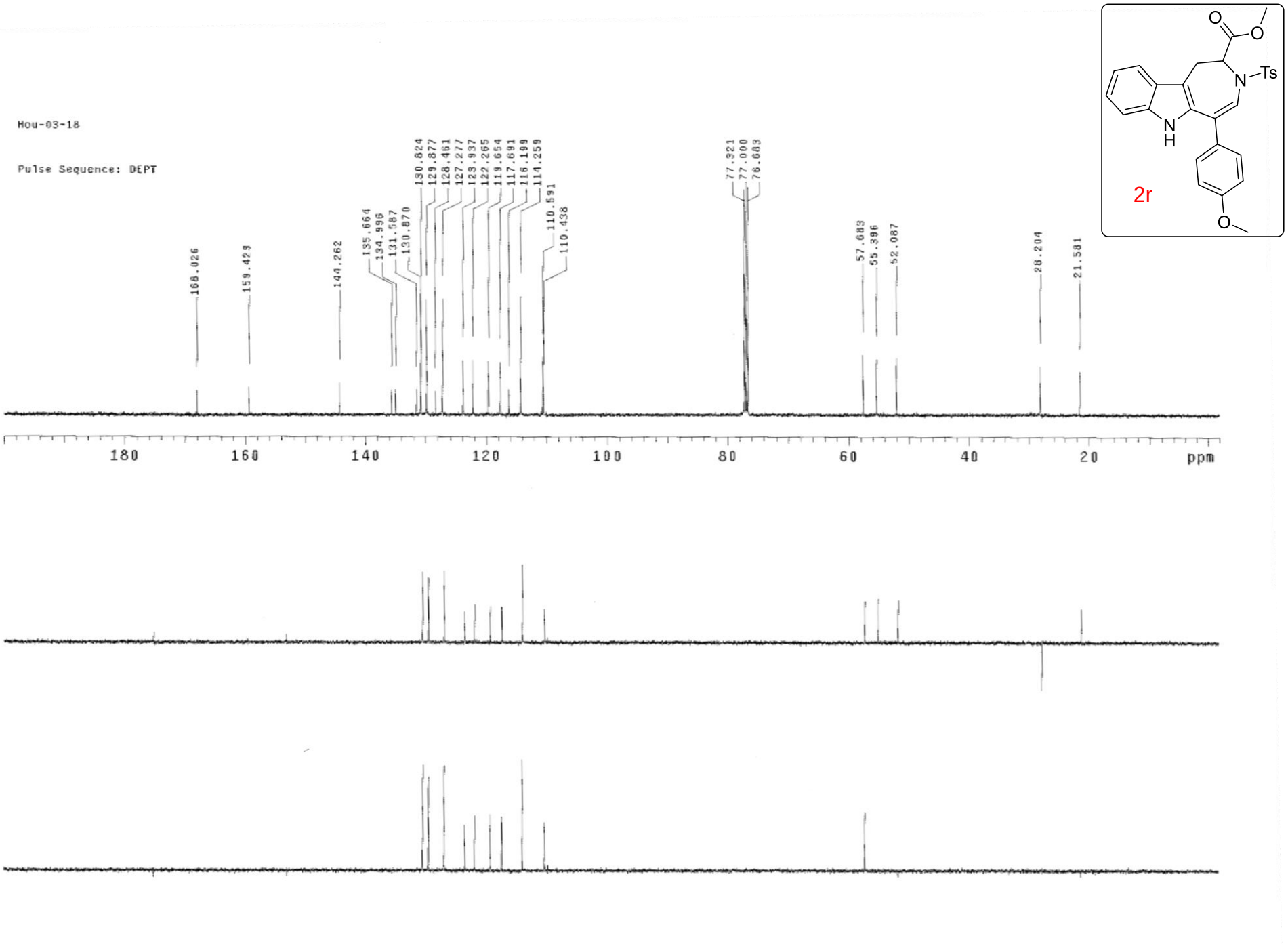




Hou-03-18

Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Jun 30 2016
Solvent: cdcl3
Ambient temperature
Total 64000 repetitions

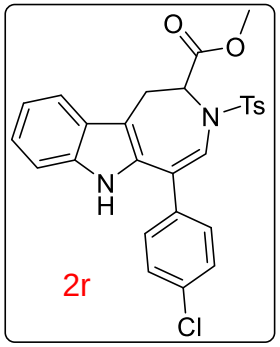


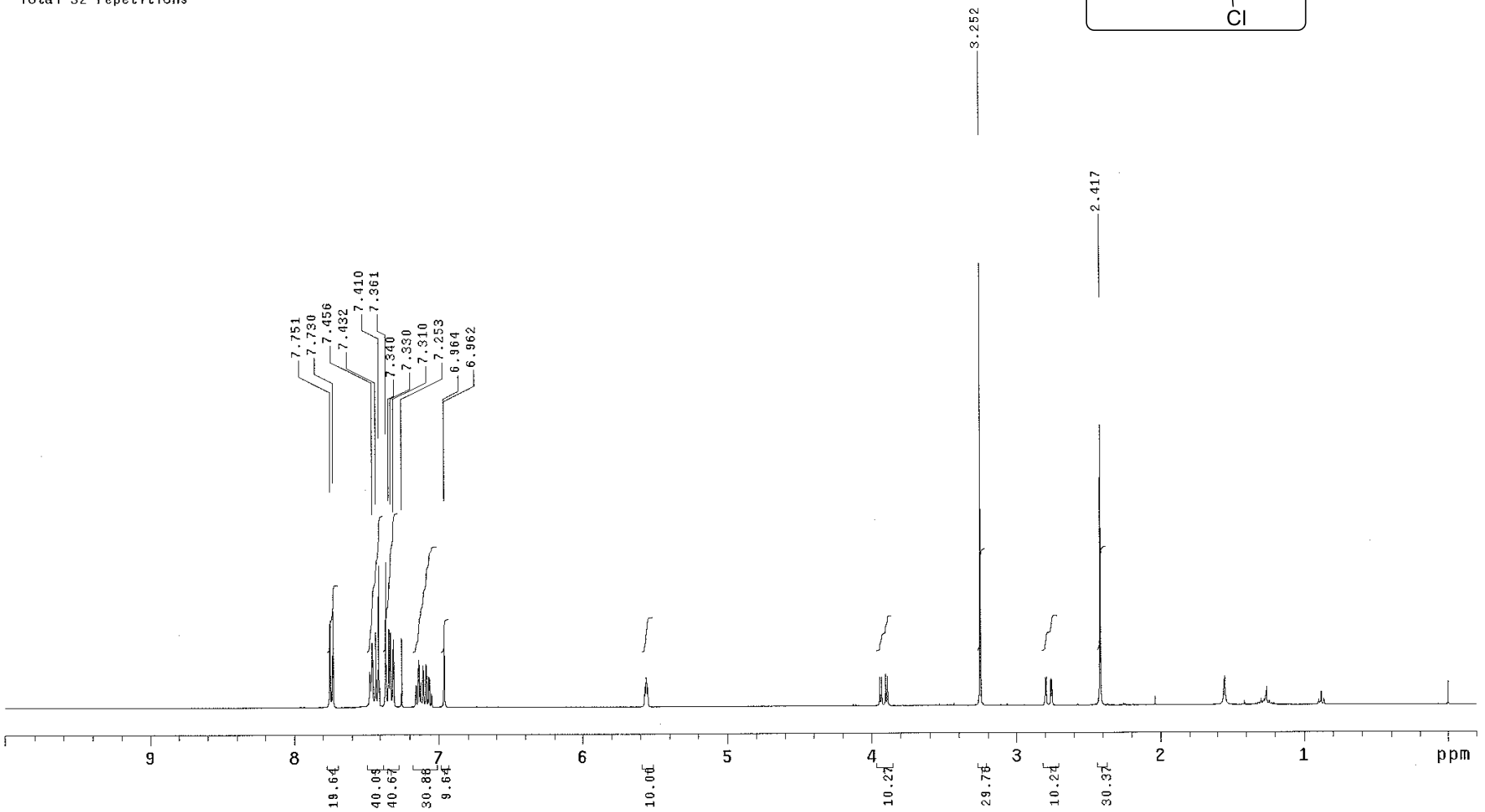

Hou-03-18
Pulse Sequence: DEPT
168.026
159.429
144.262
135.664
134.996
131.587
130.870
130.624
129.877
128.461
127.277
123.937
122.265
119.654
117.691
116.199
114.259
110.591
110.438
77.321
77.000
76.683
57.683
55.396
52.087
28.204
21.581
180
160
140
120
100
80
60
40
20
ppm
O
O
N
Ts
N
H
O
2r

Hou-03-15

Pulse Sequence: s2pul

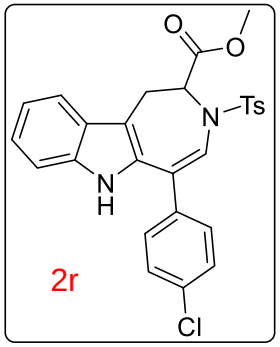
Mercury-400BB "MerPlus400"
Date: Jun 30 2016
Solvent: cdcl3
Ambient temperature
Total 32 repetitions

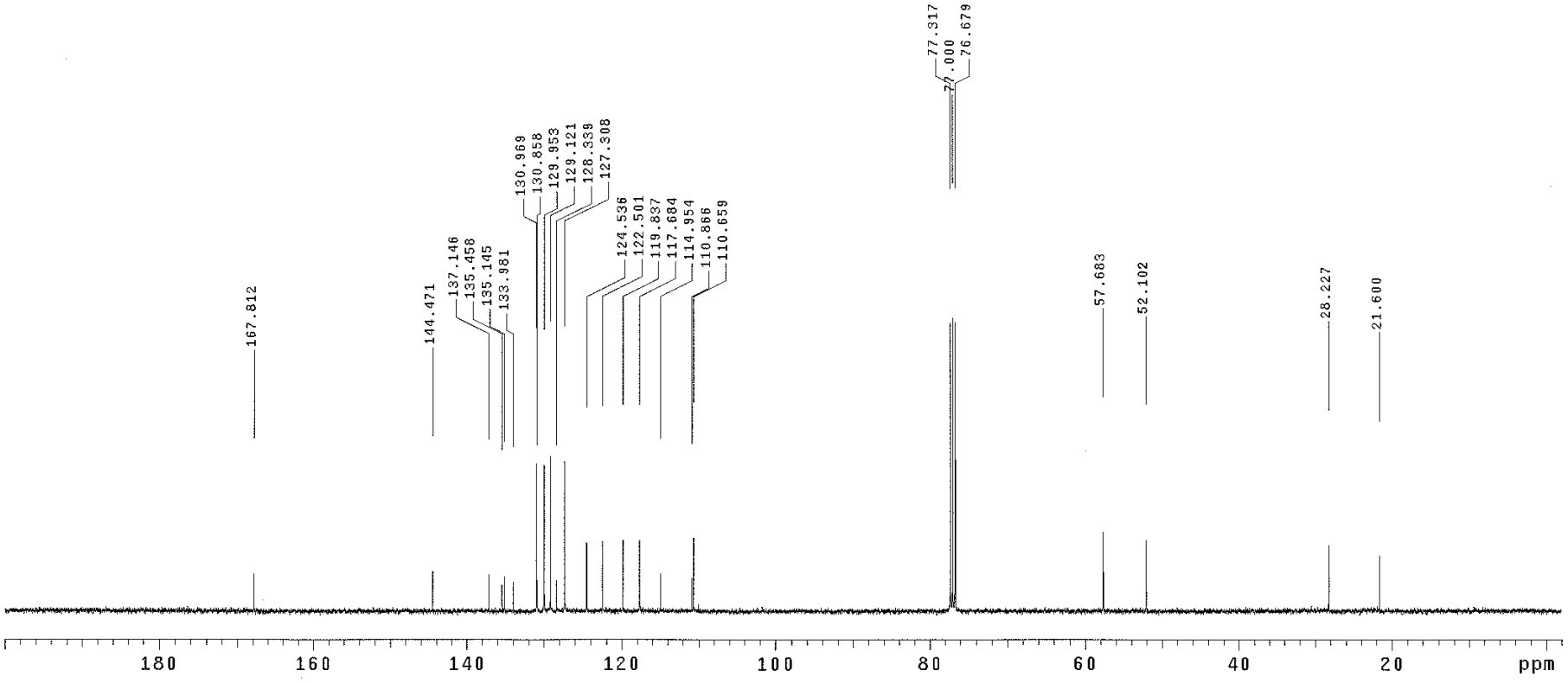


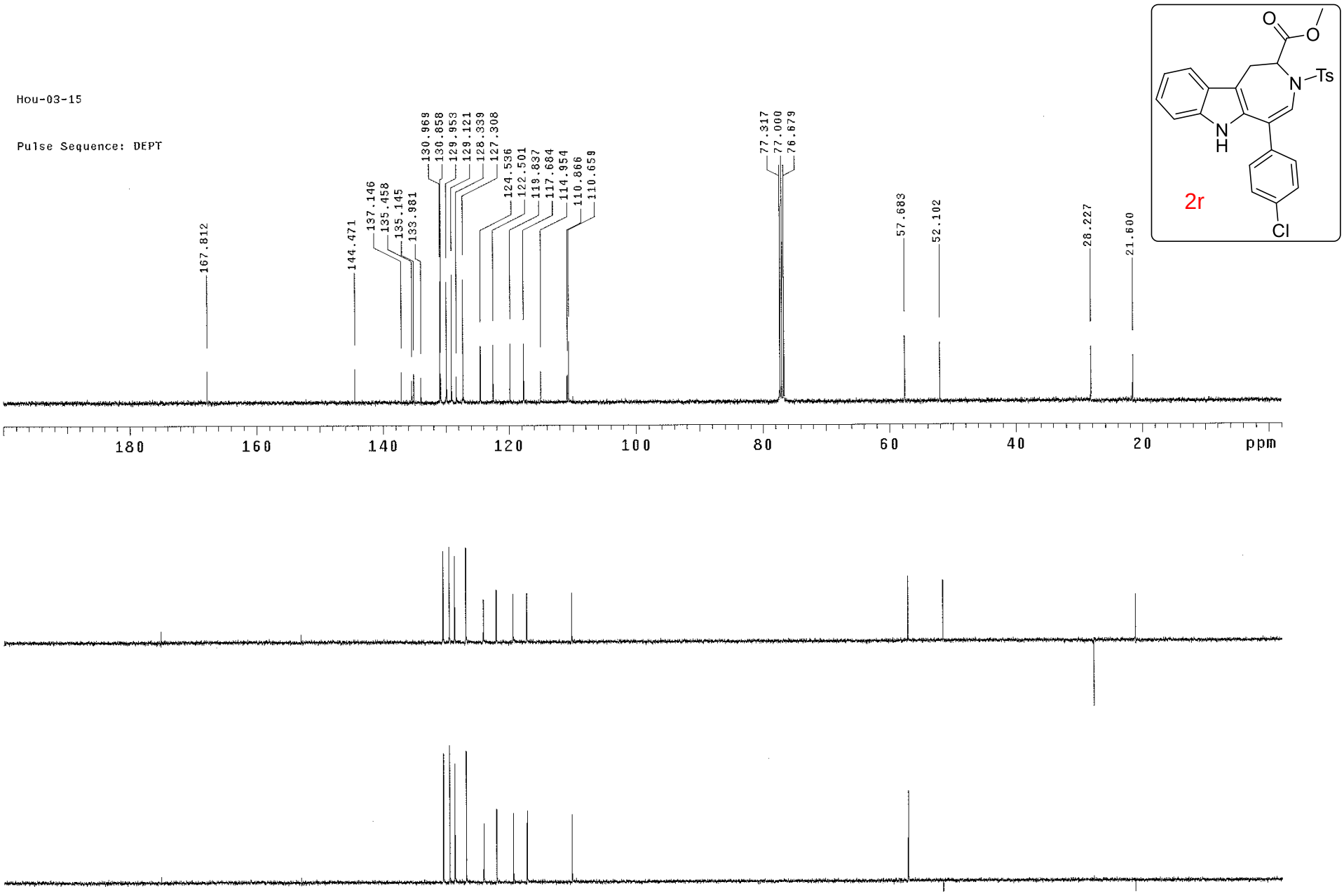


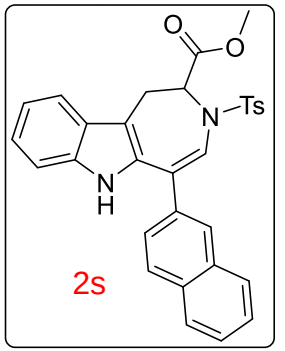
Hou-03-15

Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Jun 30 2016
Solvent: cdcl3
Ambient temperature
Total 672 repetitions



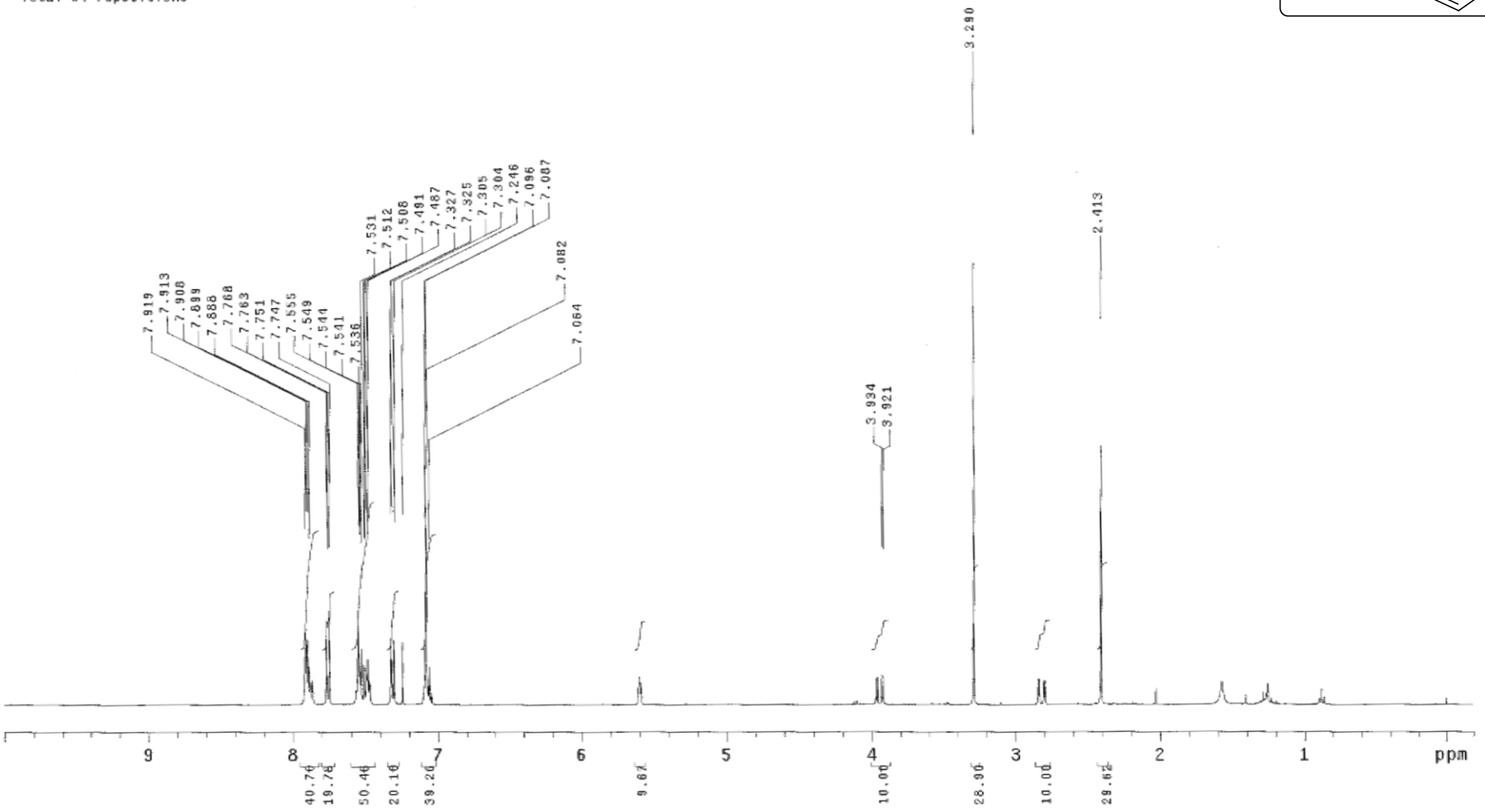



Hou-03-15
Pulse Sequence: DEPT
167.812
144.471
137.146
135.458
135.145
133.981
130.969
130.858
129.953
129.121
128.339
127.308
124.536
122.501
119.837
117.684
114.954
110.866
110.659
77.317
77.000
76.679
57.683
52.102
28.227
21.600
180
160
140
120
100
80
60
40
20
ppm
O
O
N
Ts
N
H
Cl
2r



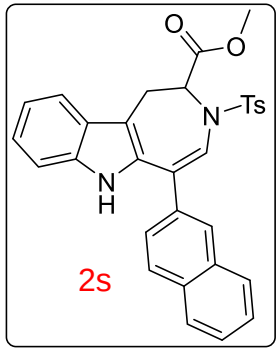
Hou-03-21

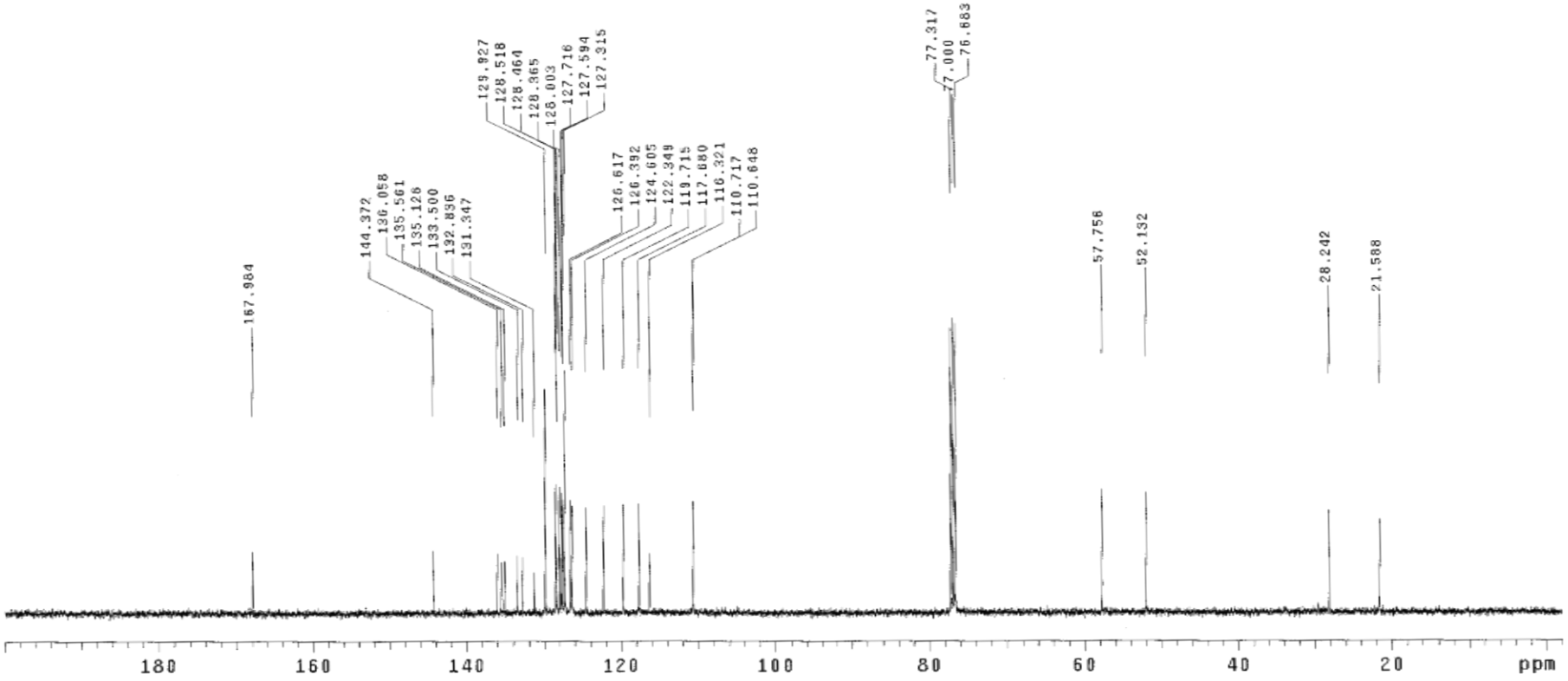
Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Jun 30 2016
Solvent: cdcl3
Ambient temperature
Total 64 repetitions

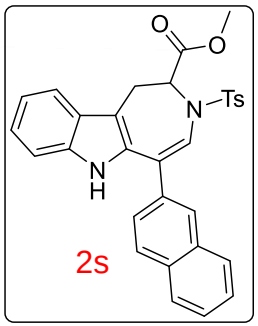


Hou-03-21

Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Jun 30 2016
Solvent: cdcl3
Ambient temperature
Total 560 repetitions





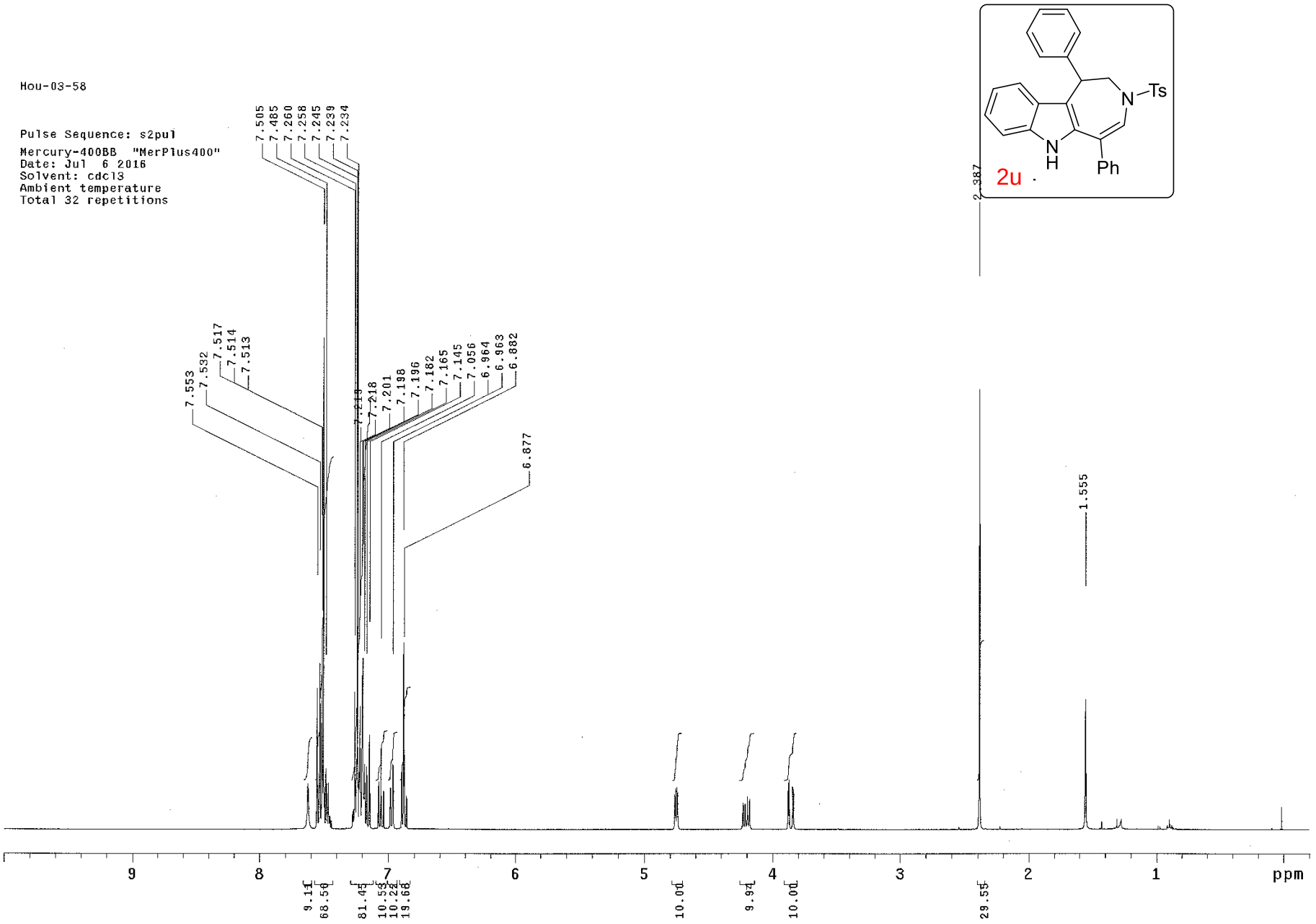


Hou-03-21

Pulse Sequence: DEPT

167.984
144.372
136.058
135.561
135.126
133.500
132.896
131.347
129.927
128.518
128.484
128.365
128.003
127.716
127.594
127.315
126.617
126.392
124.605
122.349
119.715
117.680
116.321
110.717
110.648
77.317
77.000
76.683
57.756
52.132
28.242
21.586

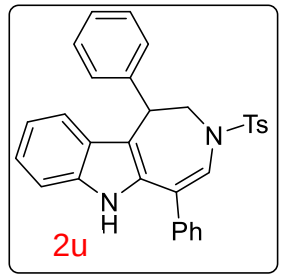
180 160 140 120 100 80 60 40 20 ppm

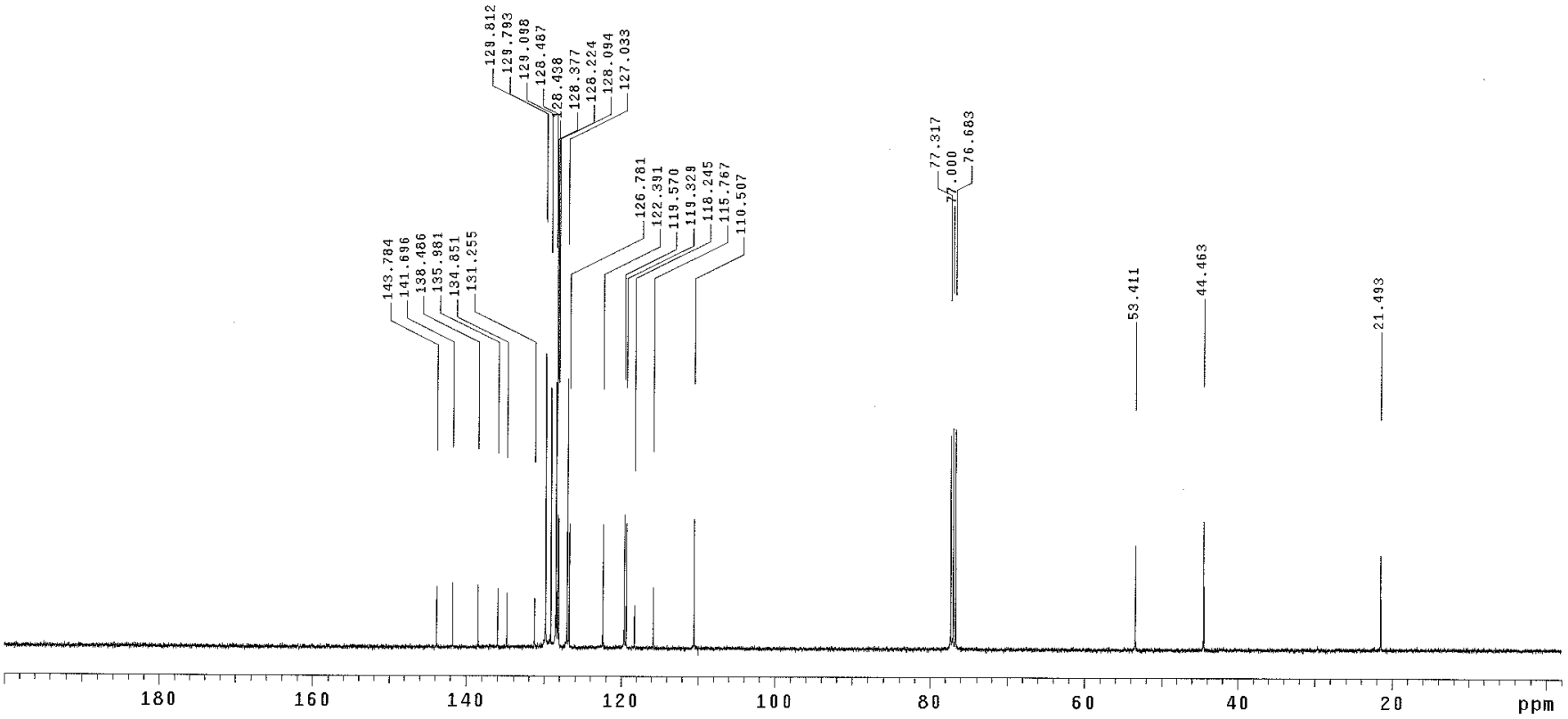

Hou-03-58
Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Jul 6 2016
Solvent: cdcl3
Ambient temperature
Total 32 repetitions
7.505
7.485
7.260
7.258
7.245
7.239
7.234
7.553
7.532
7.517
7.514
7.513
7.218
7.201
7.198
7.196
7.182
7.165
7.145
7.056
6.964
6.963
6.882
6.877
2.387
1.555
N
Ts
N
H
Ph
2u
9
8
7
6
5
4
3
2
1
ppm
9.11
68.56
81.45
10.58
10.22
19.68
10.00
9.94
10.00
29.55

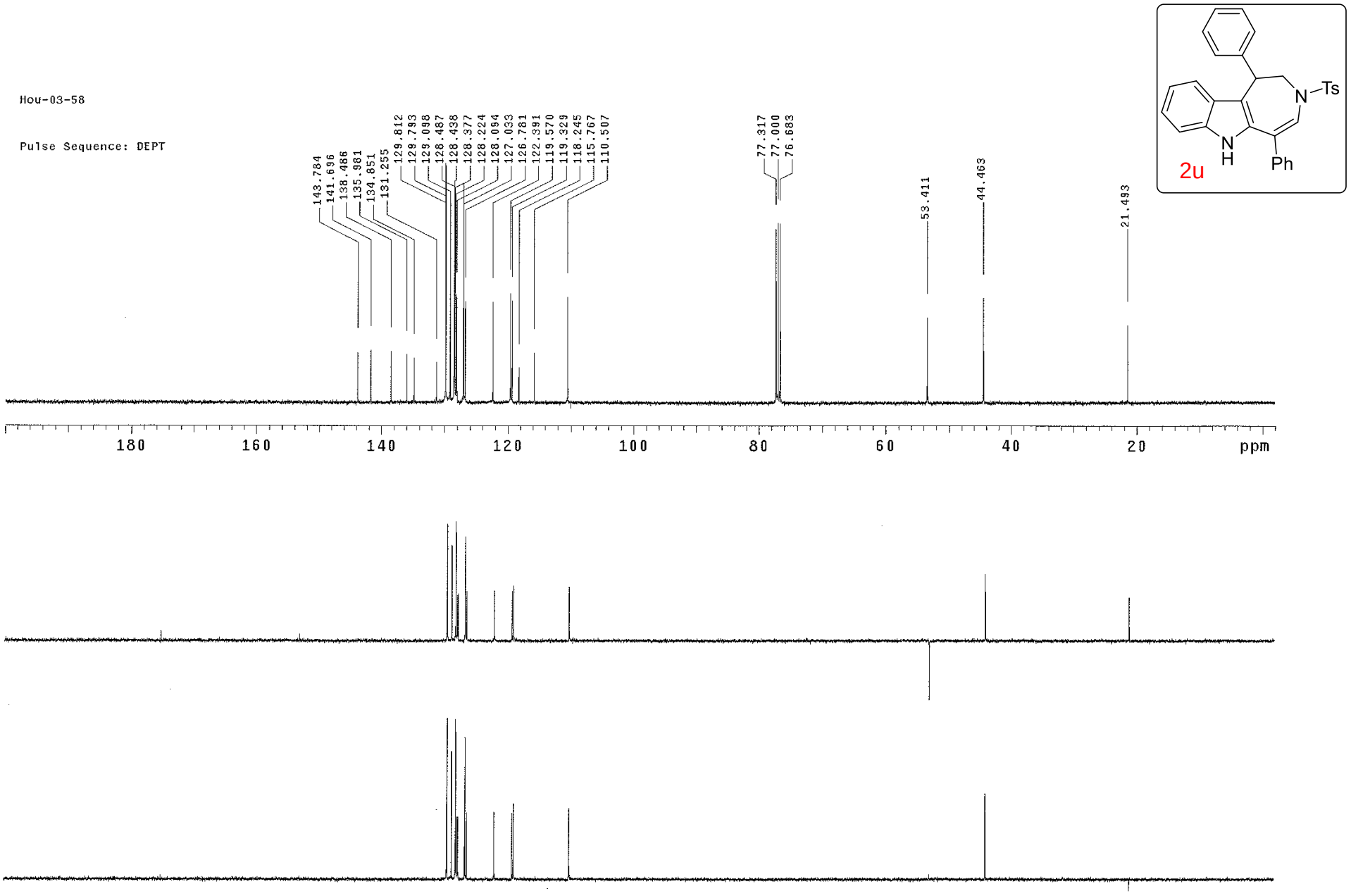
Hou-03-58

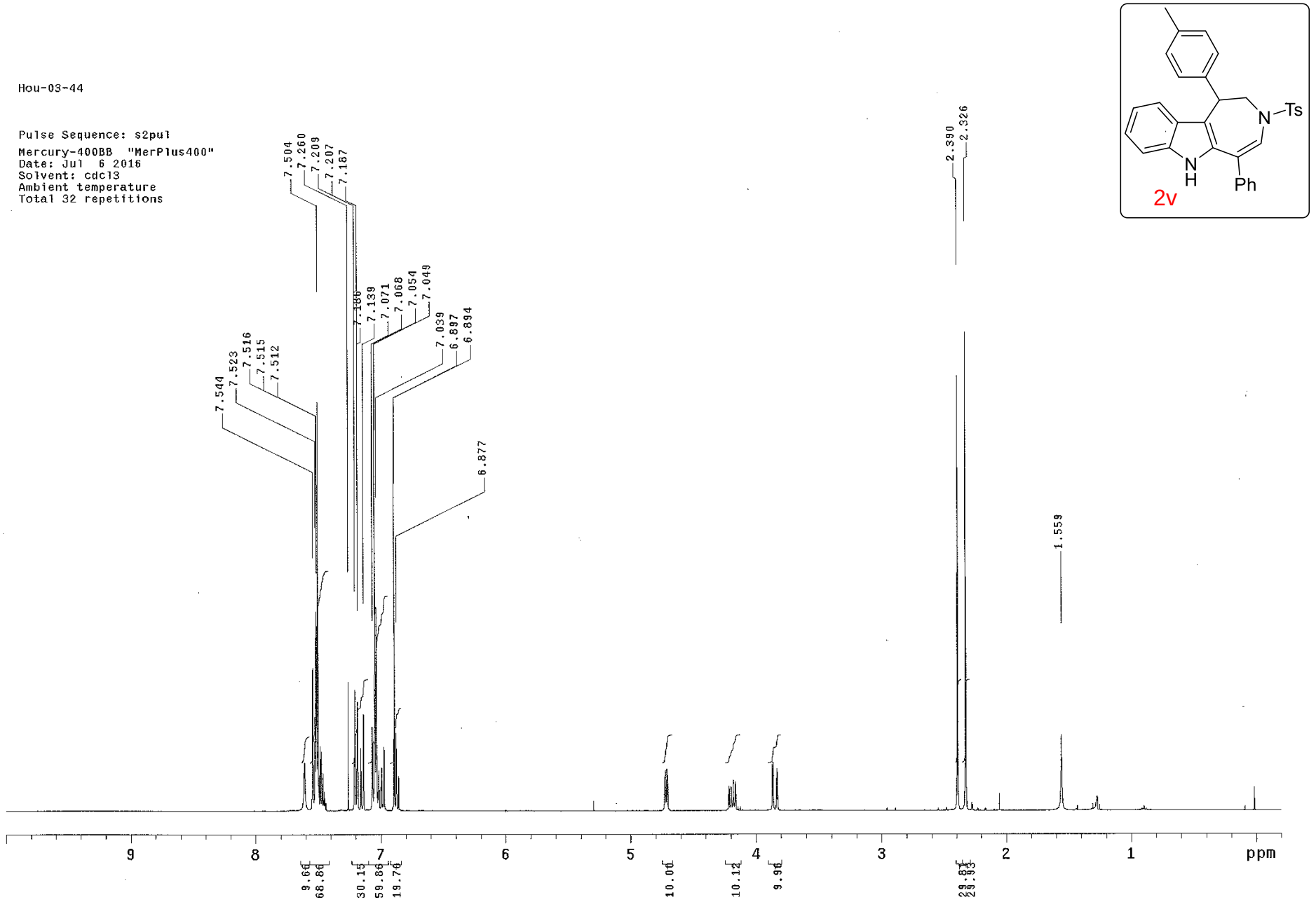
Pulse Sequence: s2pul

Mercury-400BB "MerPlus400"
Date: Jul 6 2016
Solvent: cdcl3
Ambient temperature
Total 64000 repetitions





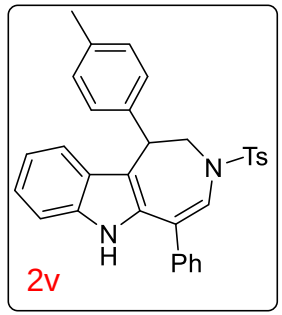

Hou-03-58
Pulse Sequence: DEPT
143.784
141.696
138.486
135.981
134.851
131.255
129.812
129.783
129.098
128.487
128.438
128.377
128.224
128.094
127.033
126.781
122.391
119.570
119.329
118.245
115.767
110.507
77.317
77.000
76.683
53.411
44.463
21.493
180
160
140
120
100
80
60
40
20
ppm
N
Ts
N
H
Ph
2u


Hou-03-44
Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Jul 6 2016
Solvent: cdcl3
Ambient temperature
Total 32 repetitions
7.544
7.523
7.516
7.515
7.512
7.504
7.260
7.209
7.207
7.187
7.139
7.071
7.068
7.054
7.049
7.039
6.887
6.894
6.877
2.390
2.326
1.559
9
8
7
6
5
4
3
2
1
ppm
9.66
68.86
30.15
59.86
19.76
10.06
10.12
9.96
29.83
29.93
N
Ts
N
H
Ph
2v

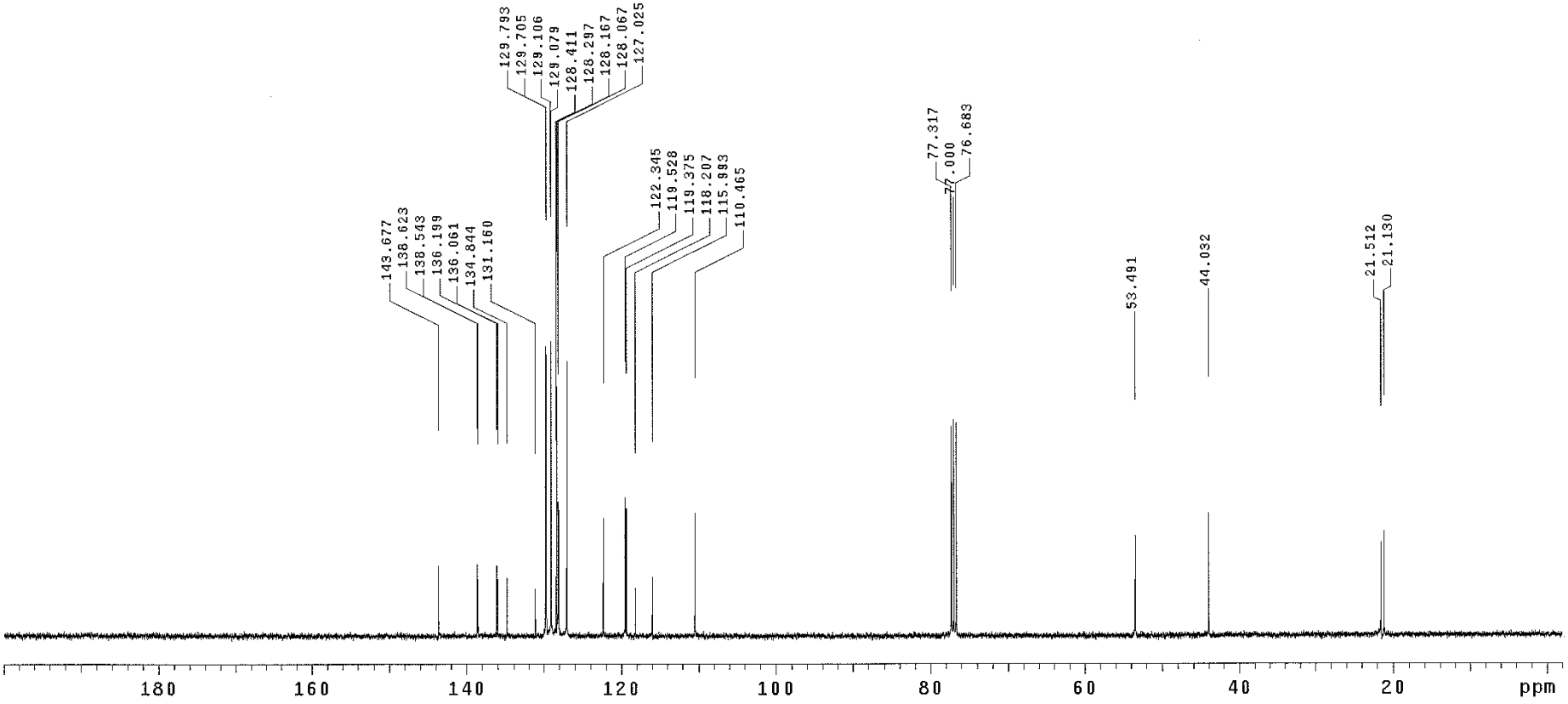
Hou-03-44

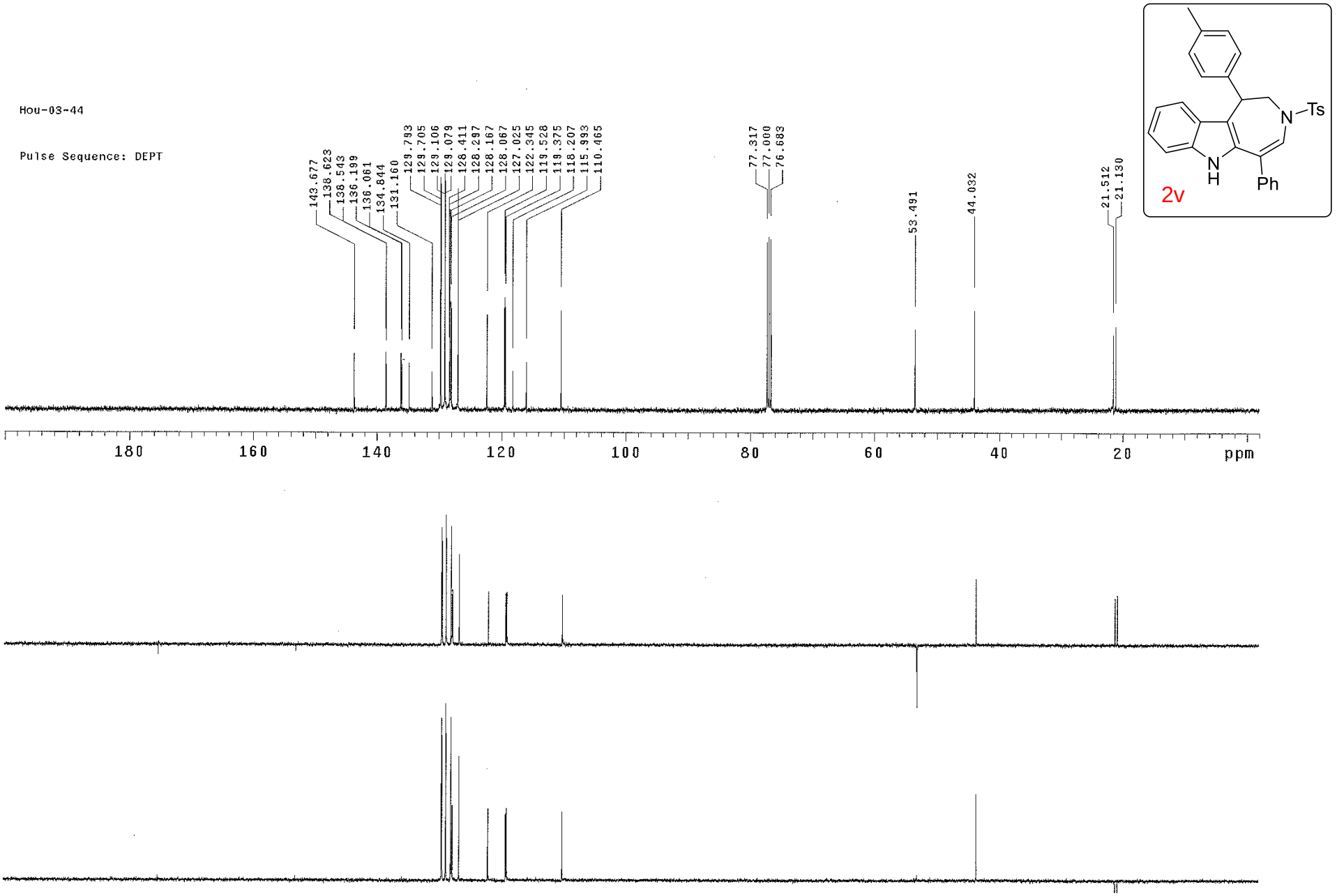
Pulse Sequence: s2pul

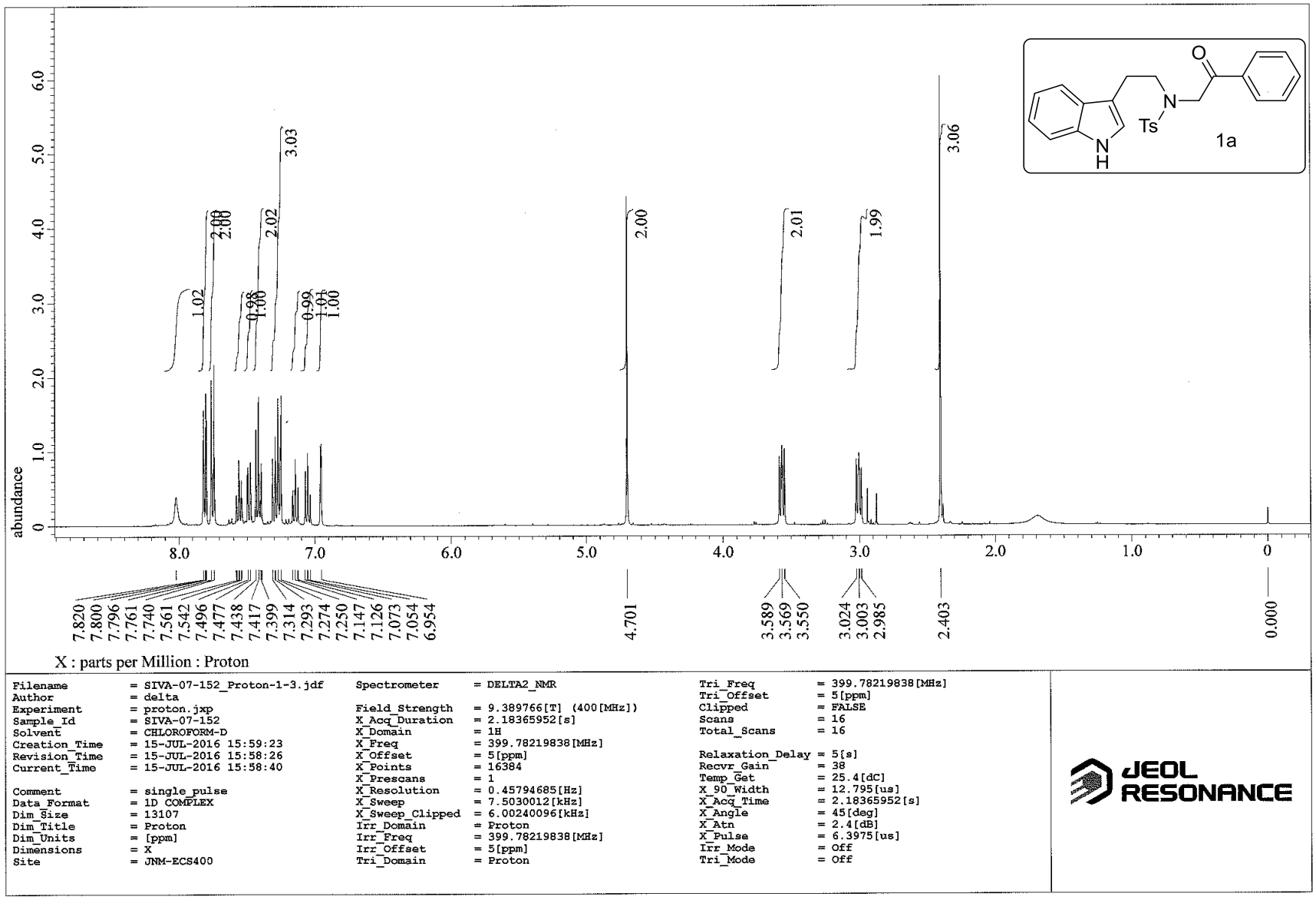
Mercury-400BB "MerPlus400"
Date: Jul 6 2016
Solvent: cdcl3
Ambient temperature
Total 976 repetitions



2v

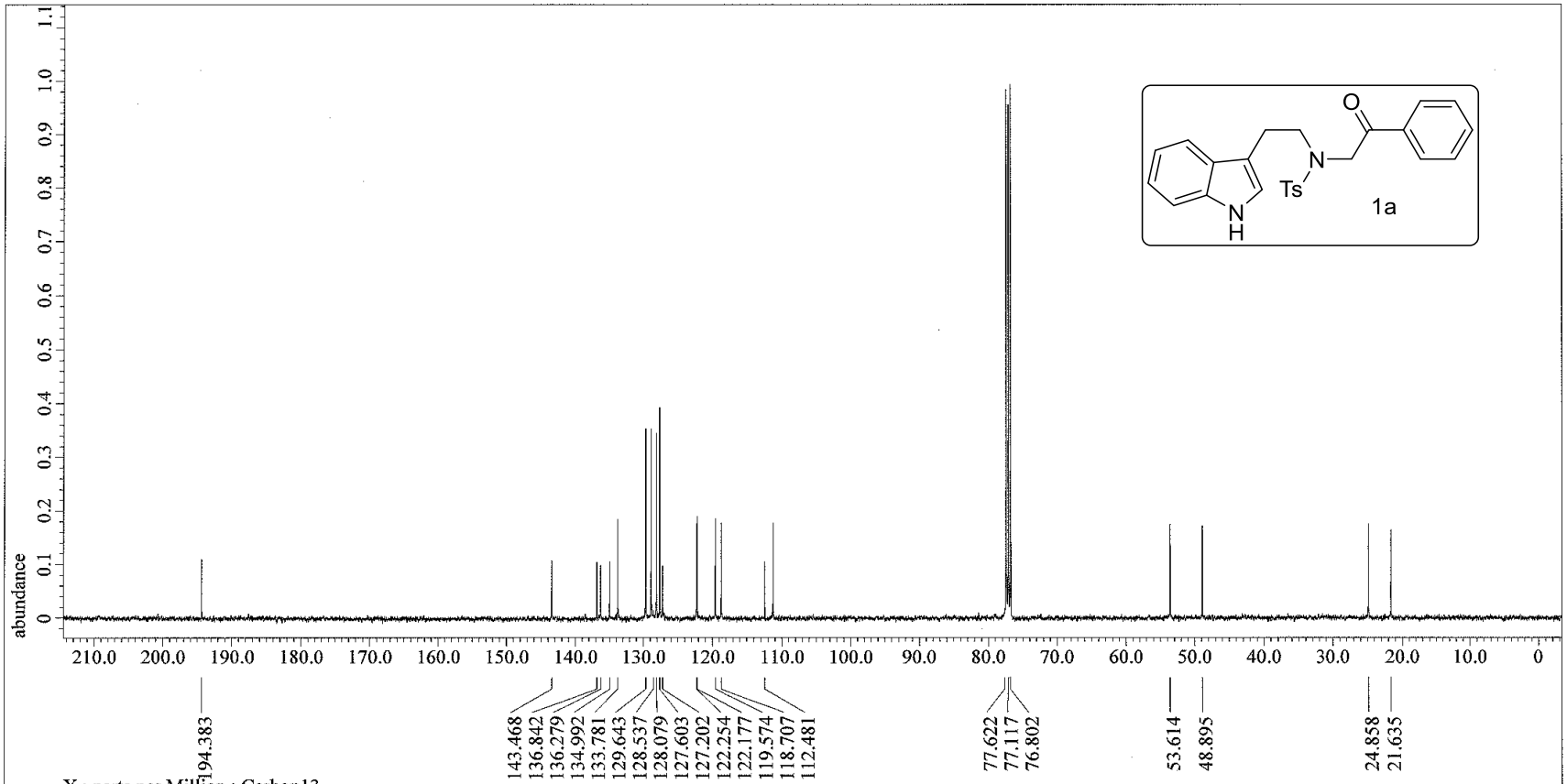



Hou-03-44
Pulse Sequence: DEPT
143.677
138.623
138.543
136.199
136.061
134.844
131.160
129.793
129.705
129.106
129.079
128.411
128.297
128.167
128.067
127.025
122.345
119.528
119.375
118.207
115.993
110.465
77.317
77.000
76.683
53.491
44.032
21.512
21.130
180
160
140
120
100
80
60
40
20
ppm
N-Ts
N
H
Ph
2v



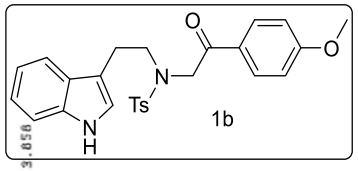
Filename	= SIVA-07-152_Proton-1-3.jdf	Spectrometer	= DELTA2_NMR	Tri_Freq	= 399.78219838[MHz]
Author	= delta			Tri_Offset	= 5[ppm]
Experiment	= proton.jxp	Field_Strength	= 9.389766[T] (400[MHz])	Clipped	= FALSE
Sample_Id	= SIVA-07-152	X_Acq_Duration	= 2.18365952[s]	Scans	= 16
Solvent	= CHLOROFORM-D	X_Domain	= 1H	Total_Scans	= 16
Creation_Time	= 15-JUL-2016 15:59:23	X_Freq	= 399.78219838[MHz]		
Revision_Time	= 15-JUL-2016 15:58:26	X_Offset	= 5[ppm]	Relaxation_Delay	= 5[s]
Current_Time	= 15-JUL-2016 15:58:40	X_Points	= 16384	Recvr_Gain	= 38
		X_Prescans	= 1	Temp_Get	= 25.4[dC]
Comment	= single_pulse	X_Resolution	= 0.45794685[Hz]	X_90_Width	= 12.795[us]
Data_Format	= 1D COMPLEX	X_Sweep	= 7.5030012[kHz]	X_Acq_Time	= 2.18365952[s]
Dim_Size	= 13107	X_Sweep_Clipped	= 6.00240096[kHz]	X_Angle	= 45[deg]
Dim_Title	= Proton	Irr_Domain	= Proton	X_Atn	= 2.4[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	X_Pulse	= 6.3975[us]
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Mode	= Off
Site	= JNM-ECS400	Tri_Domain	= Proton	Tri_Mode	= Off

JEOL RESONANCE



Filename	= SIVA-07-152_Carbon-1-3.jdf	Spectrometer	= DELTA2_NMR	Scans	= 1024
Author	= delta			Total_Scans	= 1024
Experiment	= carbon.jxp	Field_Strength	= 9.389766[T] (400[MHz])		
Sample_Id	= SIVA-07-152	X_Acq_Duration	= 1.04333312[s]	Relaxation_Delay	= 2[s]
Solvent	= CHLOROFORM-D	X_Domain	= 13C	Recvr_Gain	= 60
Creation_Time	= 15-JUL-2016 16:03:41	X_Freq	= 100.52530333[MHz]	Temp_Get	= 25.6[dC]
Revision_Time	= 15-JUL-2016 16:57:12	X_Offset	= 100[ppm]	X_90_Width	= 10.33[us]
Current_Time	= 15-JUL-2016 16:57:26	X_Points	= 32768	X_Acq_Time	= 1.04333312[s]
		X_Prescans	= 4	X_Angle	= 30[deg]
Comment	= single pulse decoupled gat	X_Resolution	= 0.95846665[Hz]	X_Atn	= 5.3[dB]
Data_Format	= 1D COMPLEX	X_Sweep	= 31.40703518[kHz]	X_Pulse	= 3.44333333[us]
Dim_Size	= 26214	X_Sweep_Clipped	= 25.12562814[kHz]	Irr_Atn_Dec	= 21.473[dB]
Dim_Title	= Carbon13	Irr_Domain	= Proton	Irr_Atn_Noe	= 21.473[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	Irr_Noise	= WALTZ
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Pwidth	= 0.115[ms]
Site	= JNM-ECS400	Clipped	= FALSE	Decoupling	= TRUE

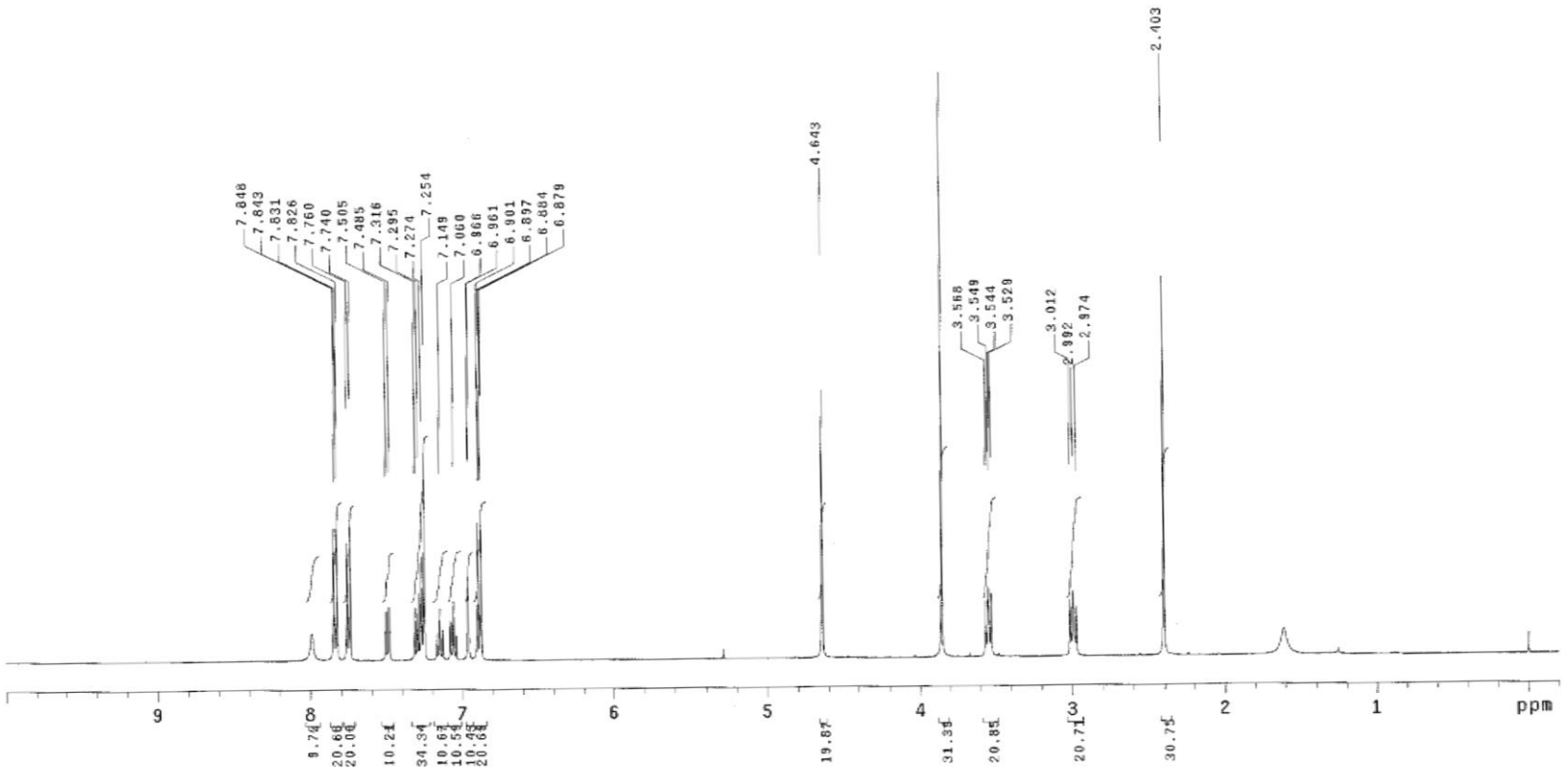
JEOL RESONANCE



SIVA-07-173

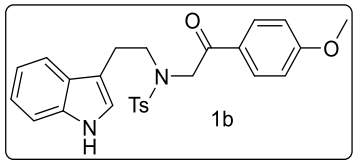
Pulse Sequence: s2pul

Mercury-400BB "MerPlus400"
Date: Aug 8 2016
Solvent: cdcl3
Ambient temperature
File: M0173-13
Total 32 repetitions

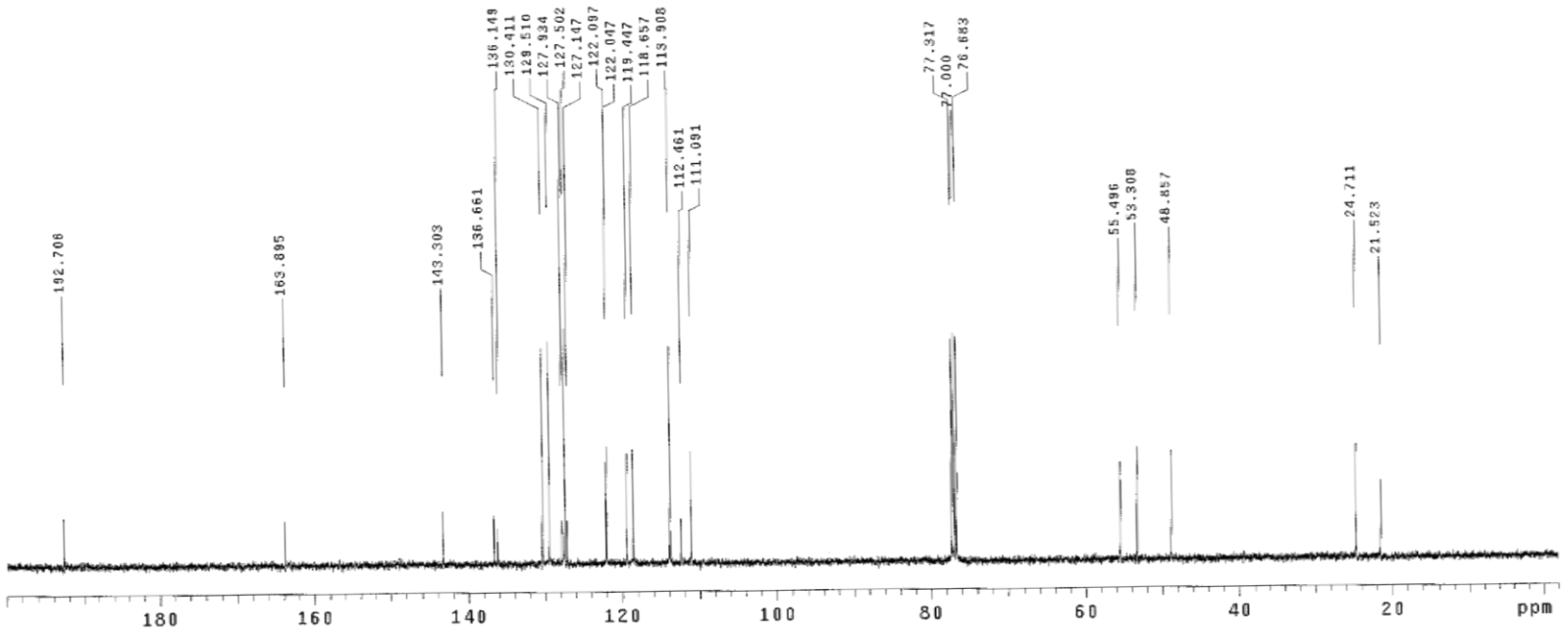


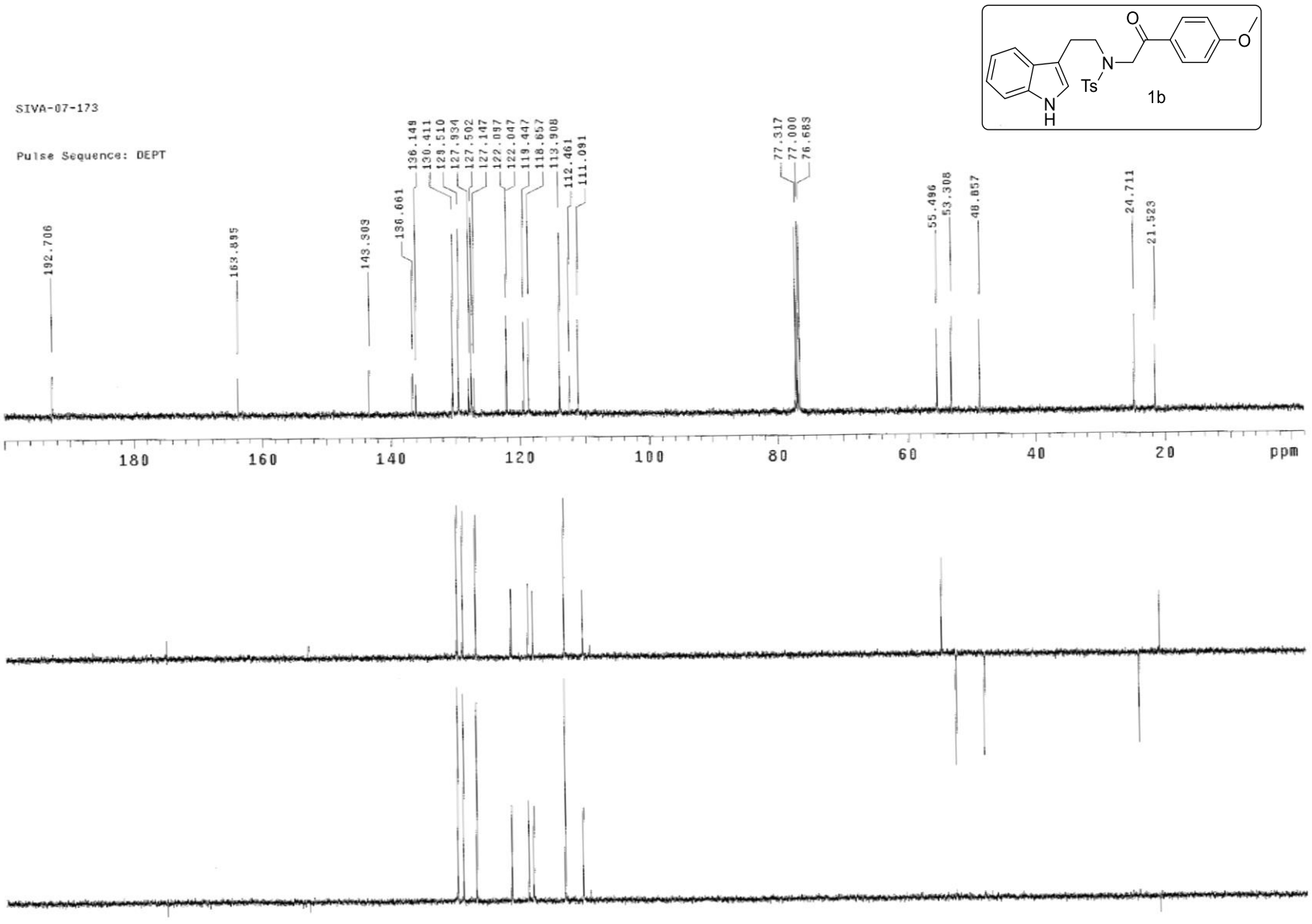
SIVA-07-173

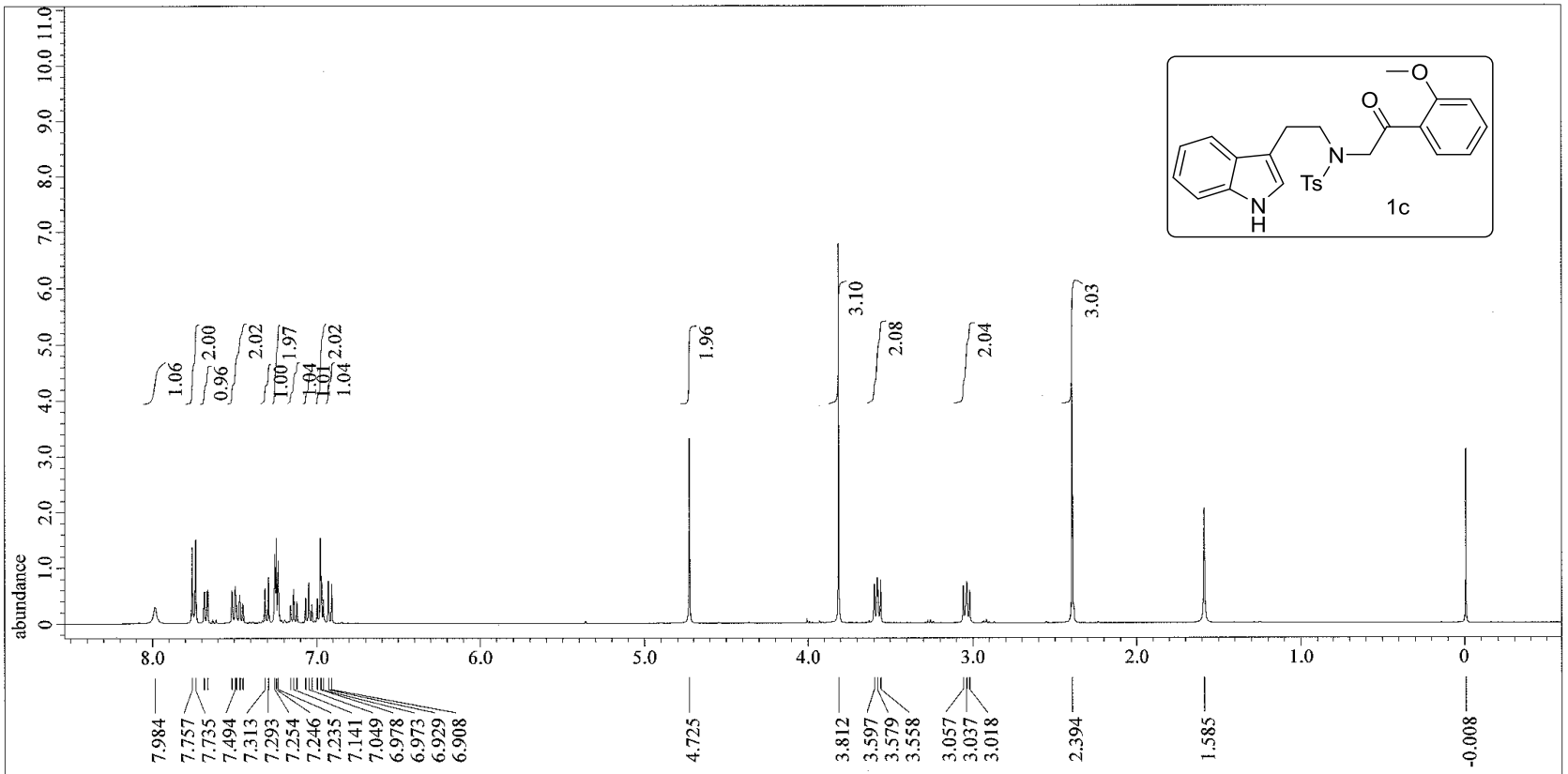
Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Aug 8 2016
Solvent: cdcl3
Ambient temperature
Total 1088 repetitions



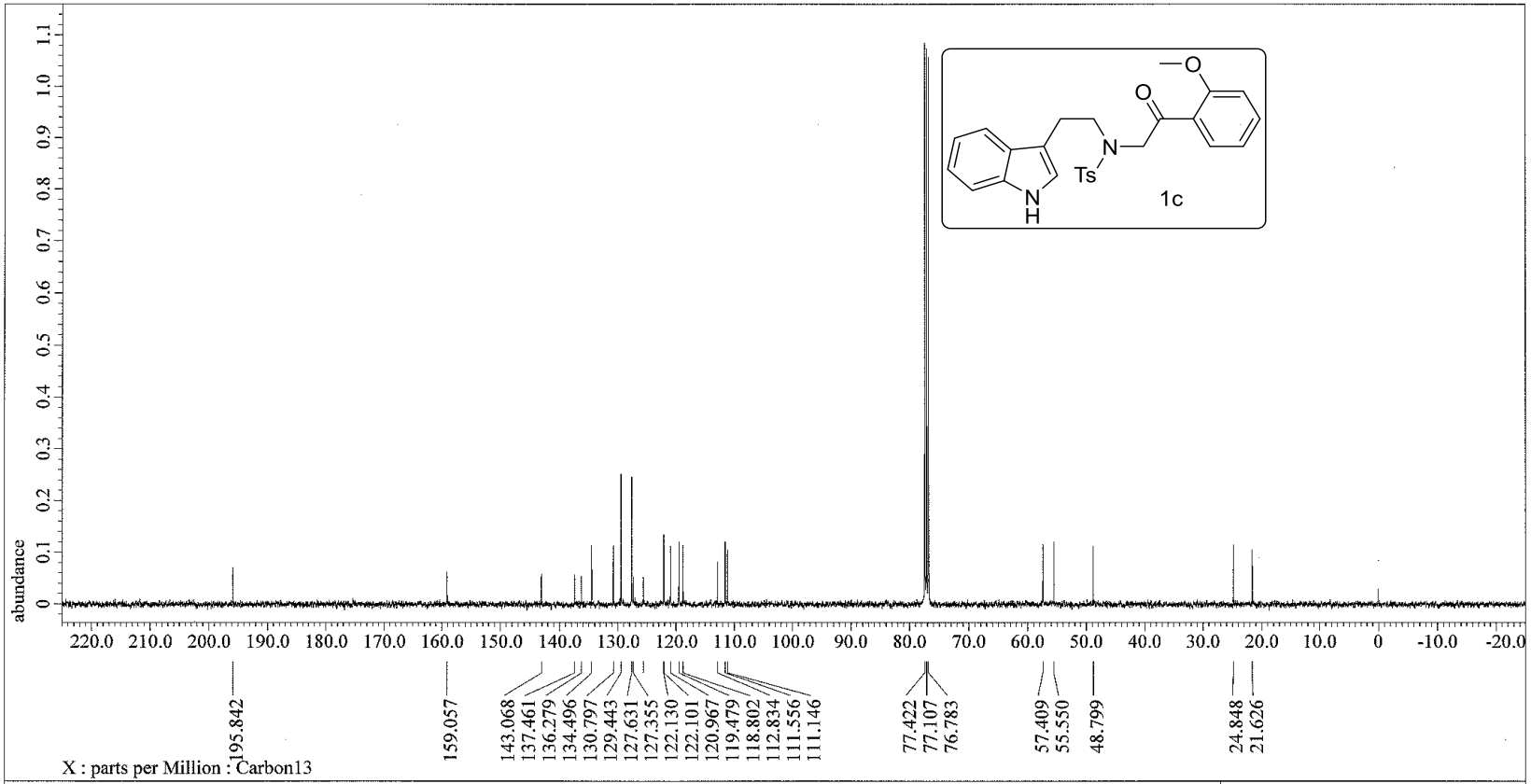
1b




SIVA-07-173
Pulse Sequence: DEPT
192.706
163.895
143.303
136.661
136.149
130.411
129.510
127.934
127.502
127.147
122.097
122.047
119.447
118.657
113.908
112.461
111.091
77.317
77.000
76.683
55.496
53.308
48.857
24.711
21.523
180
160
140
120
100
80
60
40
20
ppm
O
O
N
Ts
N
H
1b

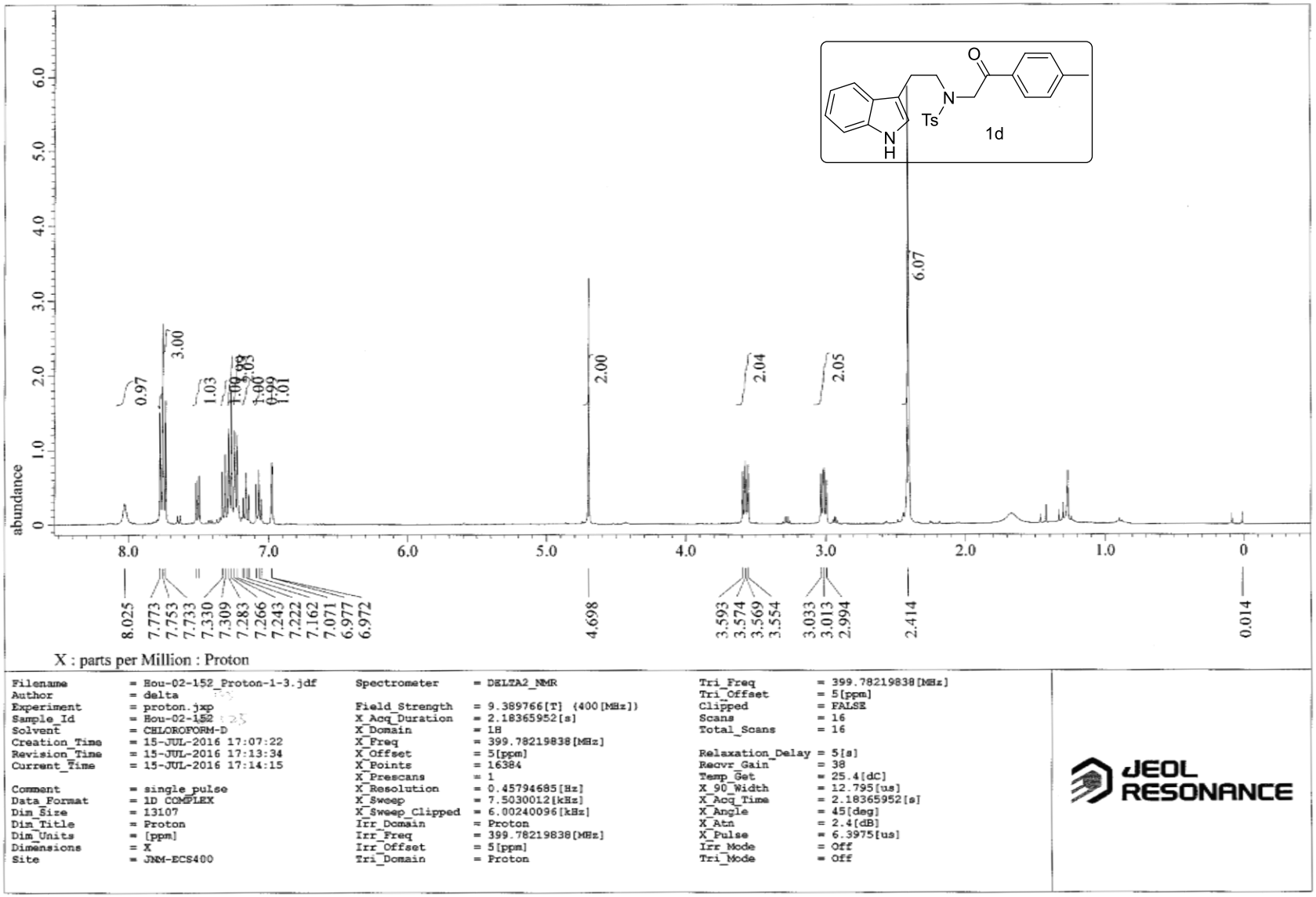


Filename	= SIVA-08-04_Proton-1-3.jdf	Spectrometer	= DELTA2_NMR	Tri_Freq	= 399.78219838[MHz]
Author	= delta			Tri_Offset	= 5[ppm]
Experiment	= proton.jxp	Field_Strength	= 9.389766[T] (400[MHz])	Clipped	= FALSE
Sample_Id	= SIVA-08-04	X_Acq_Duration	= 2.18365952[s]	Scans	= 16
Solvent	= CHLOROFORM-D	X_Domain	= 1H	Total_Scans	= 16
Creation_Time	= 20-JUL-2016 10:43:55	X_Freq	= 399.78219838[MHz]		
Revision_Time	= 20-JUL-2016 10:44:24	X_Offset	= 5[ppm]	Relaxation_Delay	= 5[s]
Current_Time	= 20-JUL-2016 10:45:12	X_Points	= 16384	Recvr_Gain	= 40
		X_Prescans	= 1	Temp_Get	= 26.5[dC]
Comment	= single_pulse	X_Resolution	= 0.45794685[Hz]	X_90_Width	= 12.795[us]
Data_Format	= 1D COMPLEX	X_Sweep	= 7.5030012[kHz]	X_Acq_Time	= 2.18365952[s]
Dim_Size	= 13107	X_Sweep_Clipped	= 6.00240096[kHz]	X_Angle	= 45[deg]
Dim_Title	= Proton	Irr_Domain	= Proton	X_Atn	= 2.4[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	X_Pulse	= 6.3975[us]
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Mode	= Off
Site	= JNM-ECS400	Tri_Domain	= Proton	Tri_Mode	= Off

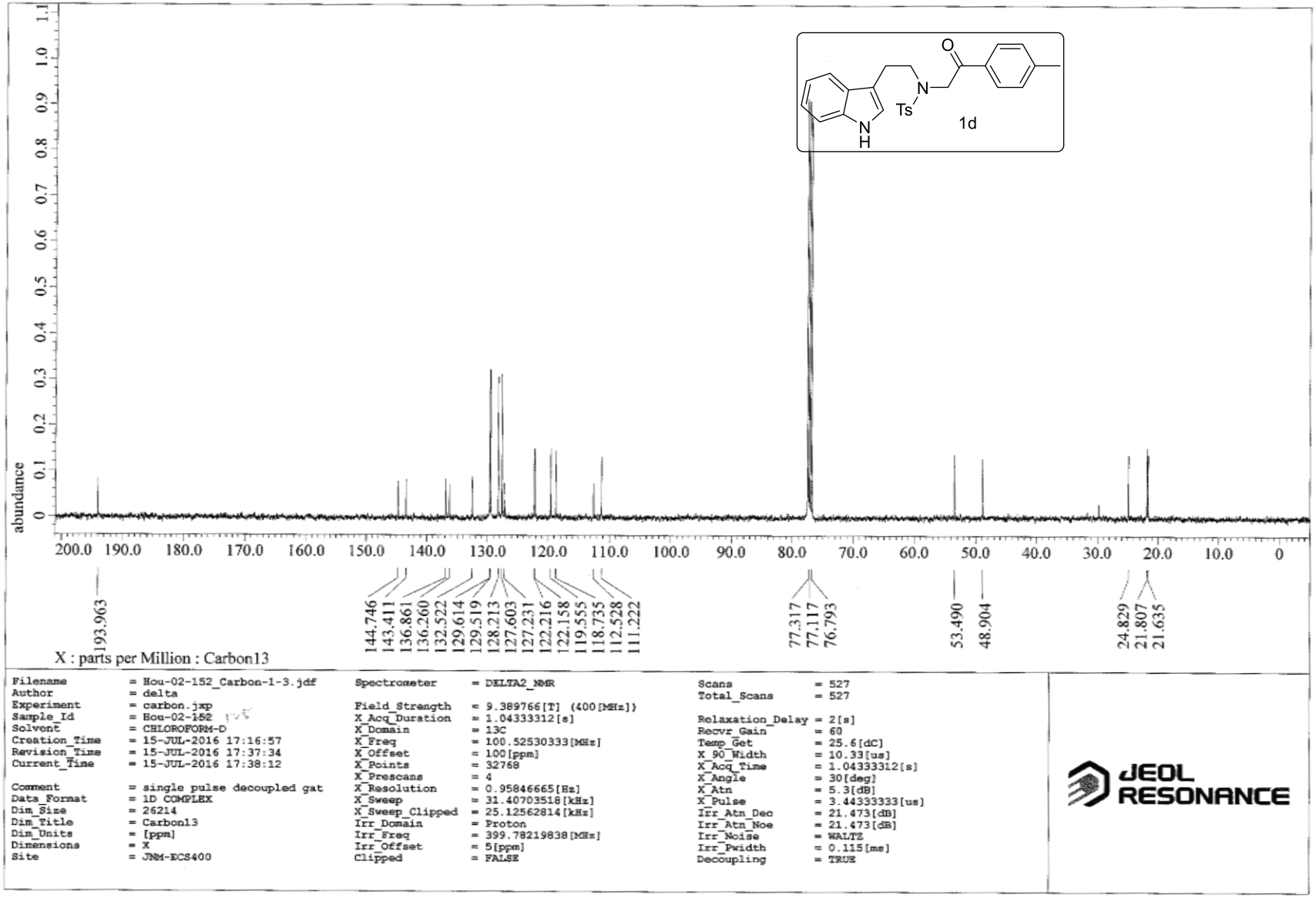


Filename	= SIVA-08-04_Carbon-1-3.jdf	Spectrometer	= DELTA2_NMR	Scans	= 614
Author	= delta			Total_Scans	= 614
Experiment	= carbon.jxp	Field_Strength	= 9.389766[T] (400[MHz])		
Sample_Id	= SIVA-08-04	X_Acq_Duration	= 1.04333312[s]	Relaxation_Delay	= 2[s]
Solvent	= CHLOROFORM-D	X_Domain	= 13C	Recvr_Gain	= 60
Creation_Time	= 20-JUL-2016 10:48:15	X_Freq	= 100.52530333[MHz]	Temp_Get	= 26.4[dC]
Revision_Time	= 20-JUL-2016 11:14:14	X_Offset	= 100[ppm]	X_90_Width	= 10.33[us]
Current_Time	= 20-JUL-2016 11:14:25	X_Points	= 32768	X_Acq_Time	= 1.04333312[s]
		X_Prescans	= 4	X_Angle	= 30[deg]
Comment	= single pulse decoupled gat	X_Resolution	= 0.95846665[Hz]	X_Atn	= 5.3[dB]
Data_Format	= 1D COMPLEX	X_Sweep	= 31.40703518[kHz]	X_Pulse	= 3.44333333[us]
Dim_Size	= 26214	X_Sweep_Clipped	= 25.12562814[kHz]	Irr_Atn_Dec	= 21.473[dB]
Dim_Title	= Carbon13	Irr_Domain	= Proton	Irr_Atn_Noe	= 21.473[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	Irr_Noise	= WALTZ
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Pwidth	= 0.115[ms]
Site	= JNM-ECS400	Clipped	= FALSE	Decoupling	= TRUE

JEOL RESONANCE



Filename	= Hou-02-152_Proton-1-3.jdf	Spectrometer	= DELTA2_NMR	Tri_Freq	= 399.78219838[MHz]
Author	= delta			Tri_Offset	= 5[ppm]
Experiment	= proton.jxp	Field_Strength	= 9.389766[T] (400[MHz])	Clipped	= FALSE
Sample_Id	= Hou-02-152	X_Acq_Duration	= 2.18365952[s]	Scans	= 16
Solvent	= CHLOROFORM-D	X_Domain	= 1H	Total_Scans	= 16
Creation_Time	= 15-JUL-2016 17:07:22	X_Freq	= 399.78219838[MHz]		
Revision_Time	= 15-JUL-2016 17:13:34	X_Offset	= 5[ppm]	Relaxation_Delay	= 5[s]
Current_Time	= 15-JUL-2016 17:14:15	X_Points	= 16384	Recvr_Gain	= 38
		X_Prescans	= 1	Temp_Get	= 25.4[dC]
Comment	= single_pulse	X_Resolution	= 0.45794685[Hz]	X_90_Width	= 12.795[us]
Data_Format	= 1D COMPLEX	X_Sweep	= 7.5030012[kHz]	X_Acq_Time	= 2.18365952[s]
Dim_Size	= 13107	X_Sweep_Clipped	= 6.00240096[kHz]	X_Angle	= 45[deg]
Dim_Title	= Proton	Irr_Domain	= Proton	X_Atn	= 2.4[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	X_Pulse	= 6.3975[us]
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Mode	= Off
Site	= JNM-ECS400	Tri_Domain	= Proton	Tri_Mode	= Off



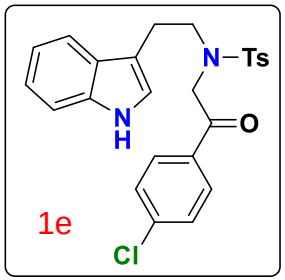
Filename	= Hou-02-152_Carbon-1-3.jdf	Spectrometer	= DELTA2_NMR	Scans	= 527
Author	= delta			Total_Scans	= 527
Experiment	= carbon.jxp	Field_Strength	= 9.389766[T] (400[MHz])		
Sample_Id	= Hou-02-152	X_Acq_Duration	= 1.04333312[s]	Relaxation_Delay	= 2[s]
Solvent	= CHLOROFORM-D	X_Domain	= 13C	Recvr_Gain	= 60
Creation_Time	= 15-JUL-2016 17:16:57	X_Freq	= 100.52530333[MHz]	Temp_Get	= 25.6[dC]
Revision_Time	= 15-JUL-2016 17:37:34	X_Offset	= 100[ppm]	X_90_Width	= 10.33[us]
Current_Time	= 15-JUL-2016 17:38:12	X_Points	= 32768	X_Acq_Time	= 1.04333312[s]
		X_Prescans	= 4	X_Angle	= 30[deg]
Comment	= single pulse decoupled gat	X_Resolution	= 0.95846665[Hz]	X_Atn	= 5.3[dB]
Data_Format	= 1D COMPLEX	X_Sweep	= 31.40703518[kHz]	X_Pulse	= 3.44333333[us]
Dim_Size	= 26214	X_Sweep_Clipped	= 25.12562814[kHz]	Irr_Atn_Dec	= 21.473[dB]
Dim_Title	= Carbon13	Irr_Domain	= Proton	Irr_Atn_Noe	= 21.473[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	Irr_Noise	= WALTZ
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Pwidth	= 0.115[ms]
Site	= JNM-ECS400	Clipped	= FALSE	Decoupling	= TRUE

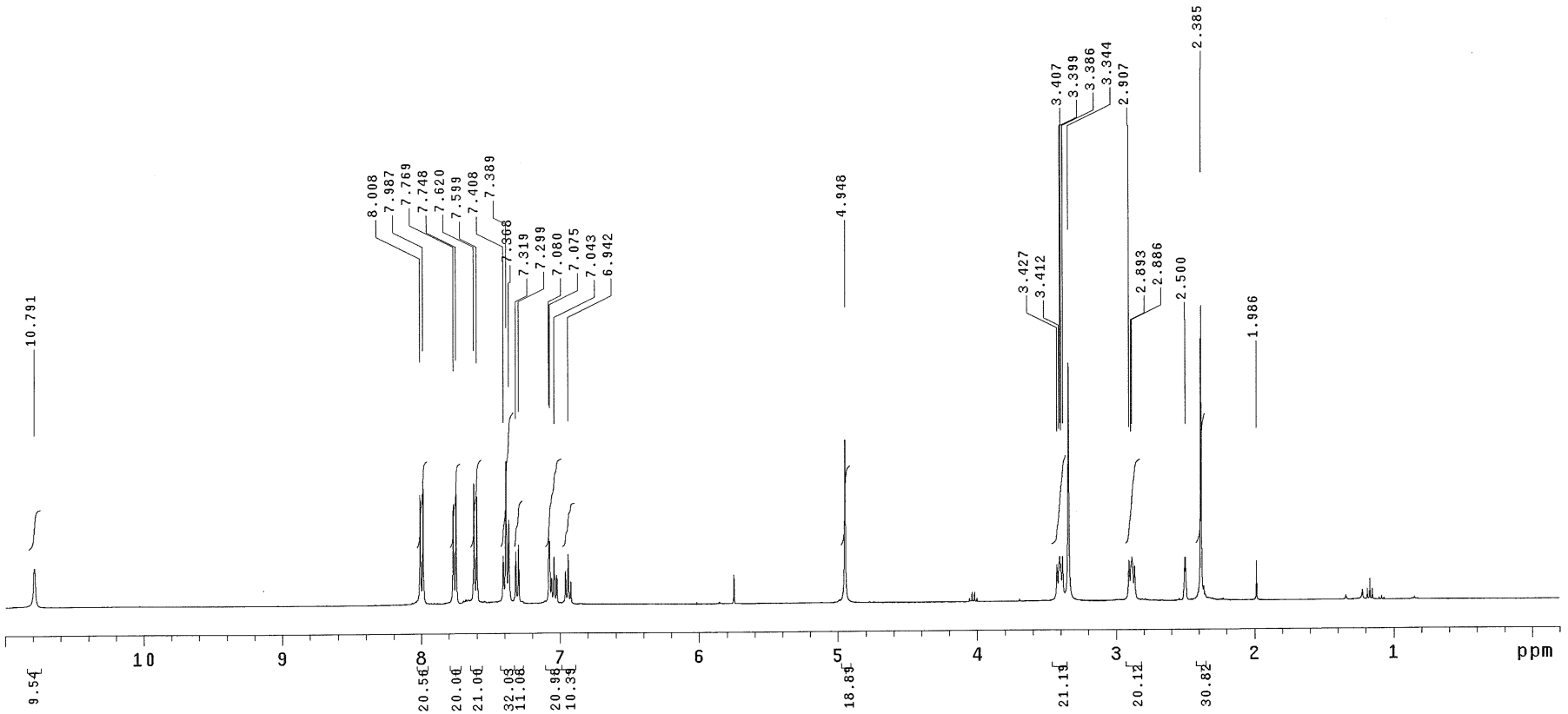
JEOL RESONANCE

Hou-Cl-1

Pulse Sequence: s2pul

Mercury-400BB "MerPlus400"
Date: Aug 30 2016
Solvent: dmso
Ambient temperature
Total 32 repetitions

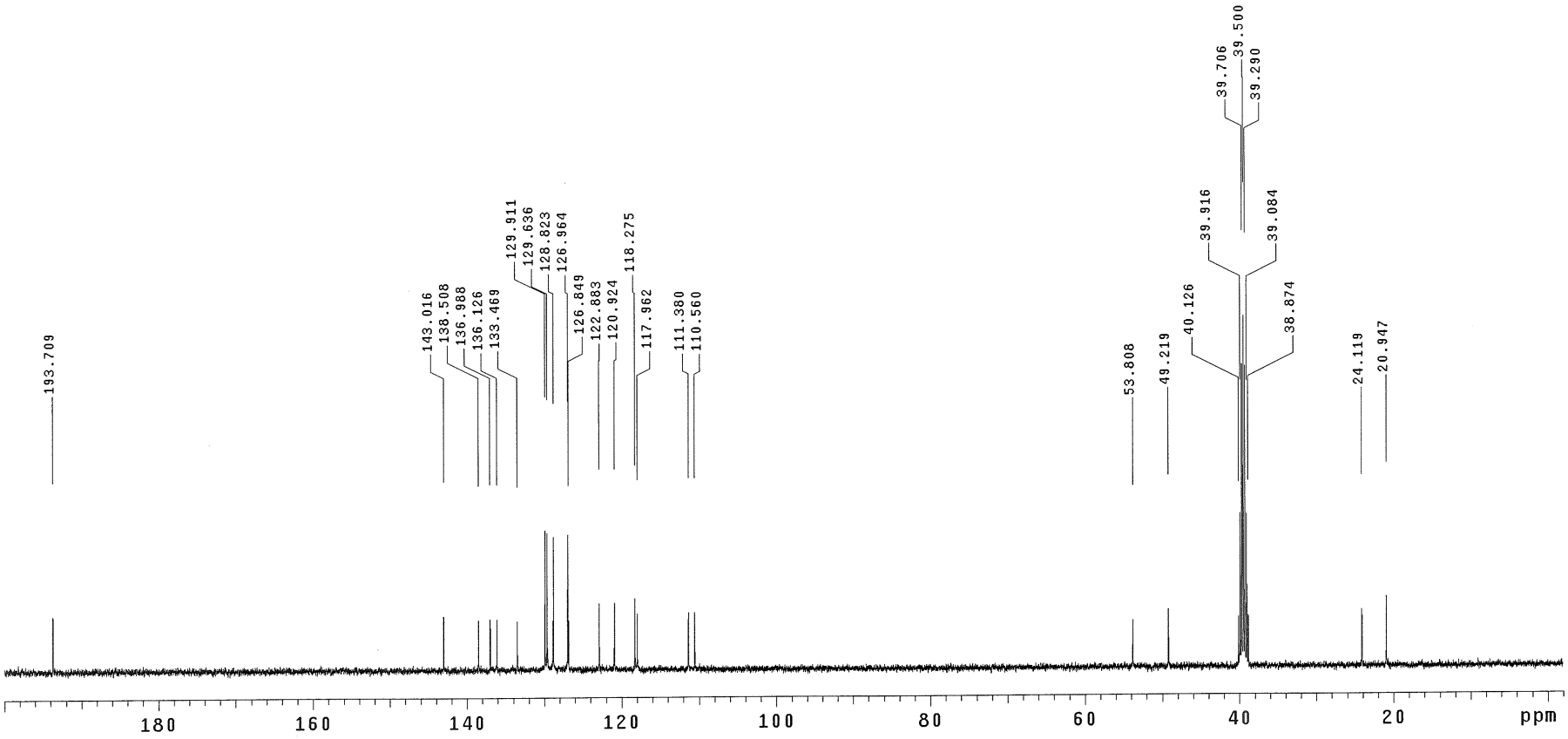




Hou-Cl-1

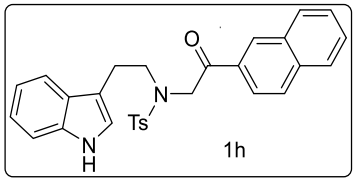
Pulse Sequence: s2pul

Mercury-400BB "MerPlus400"
Date: Aug 30 2016
Solvent: dmso
Ambient temperature
Total 672 repetitions

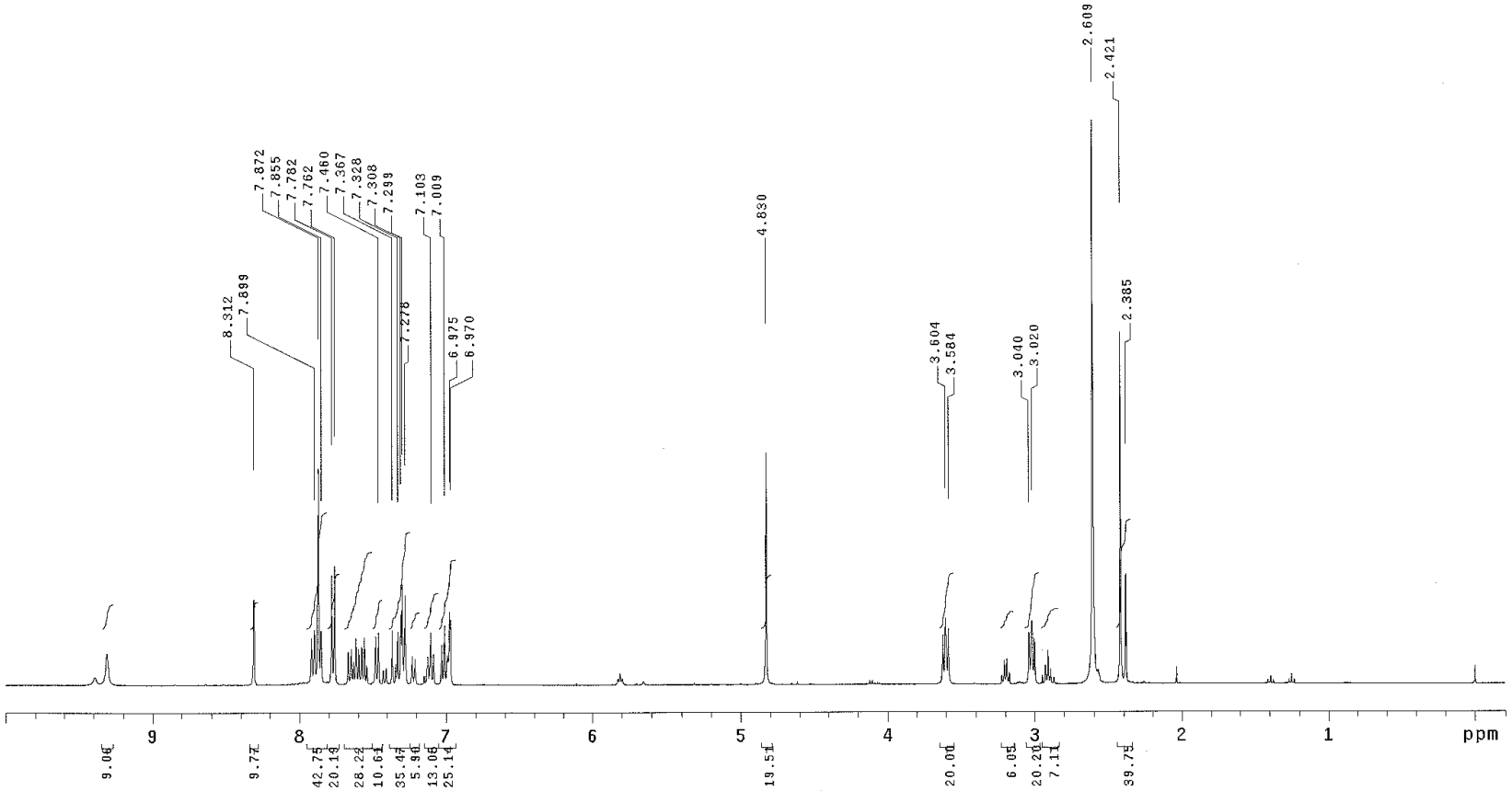


SIVA-07-176

Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Aug 8 2016
Solvent: cdcl3
Ambient temperature
Total 32 repetitions



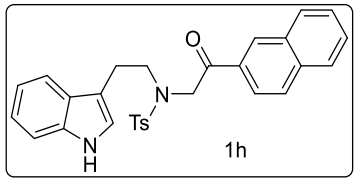
1h

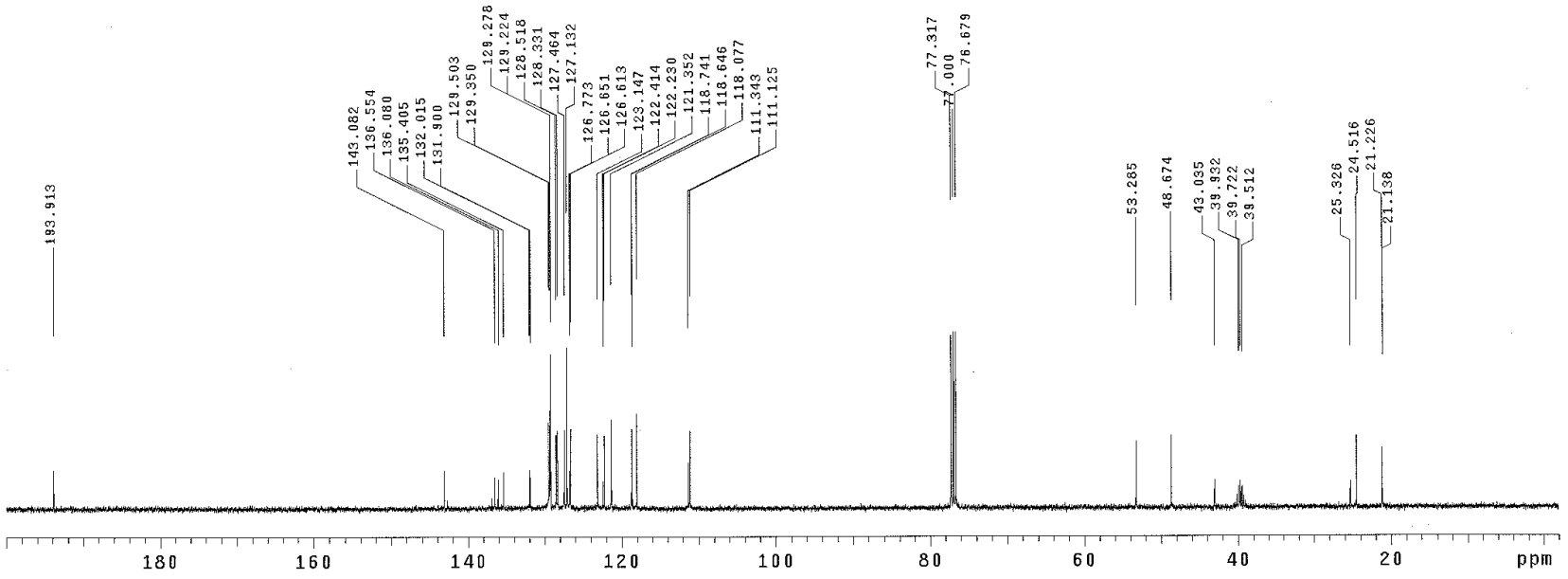


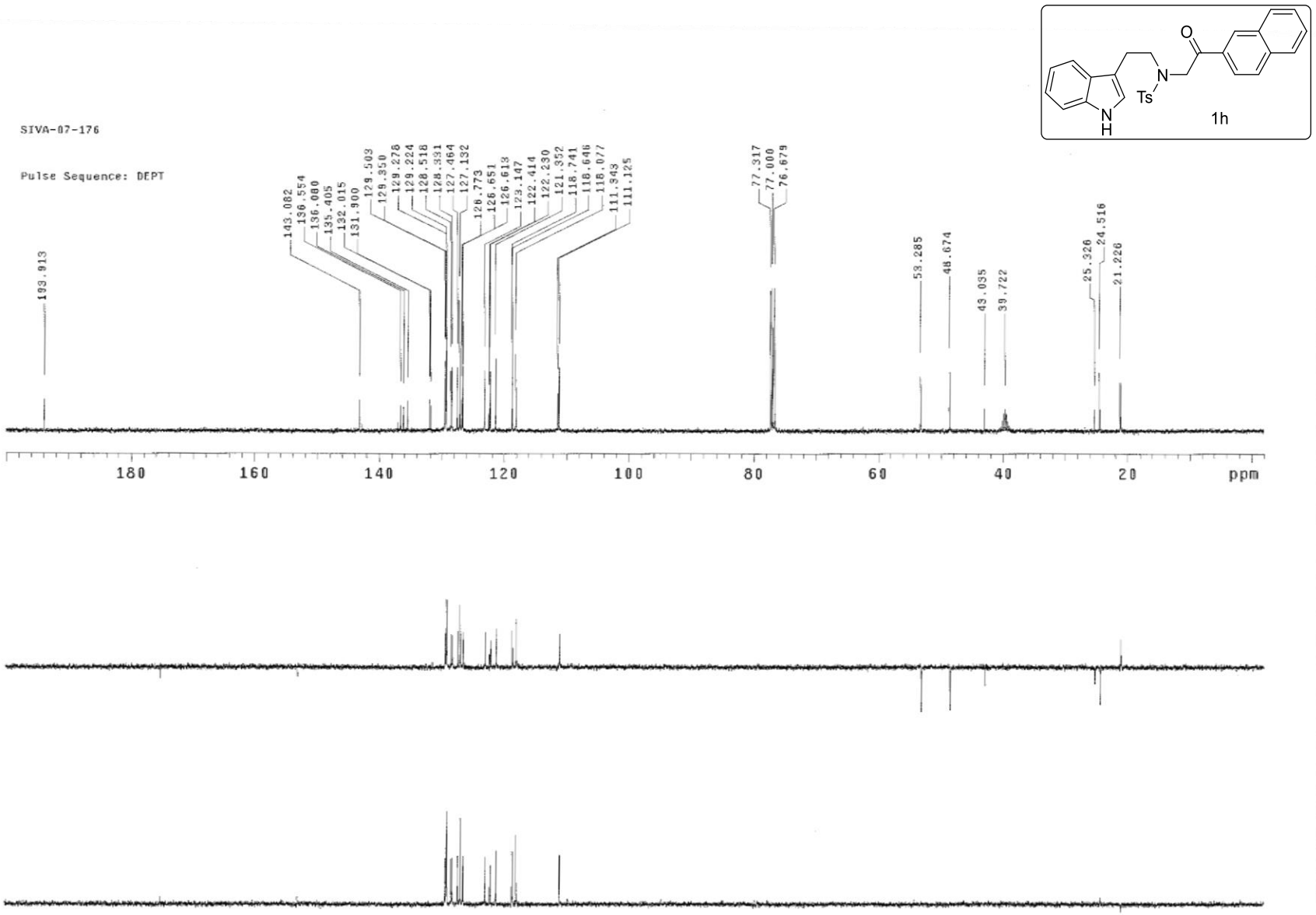
SIVA-07-176

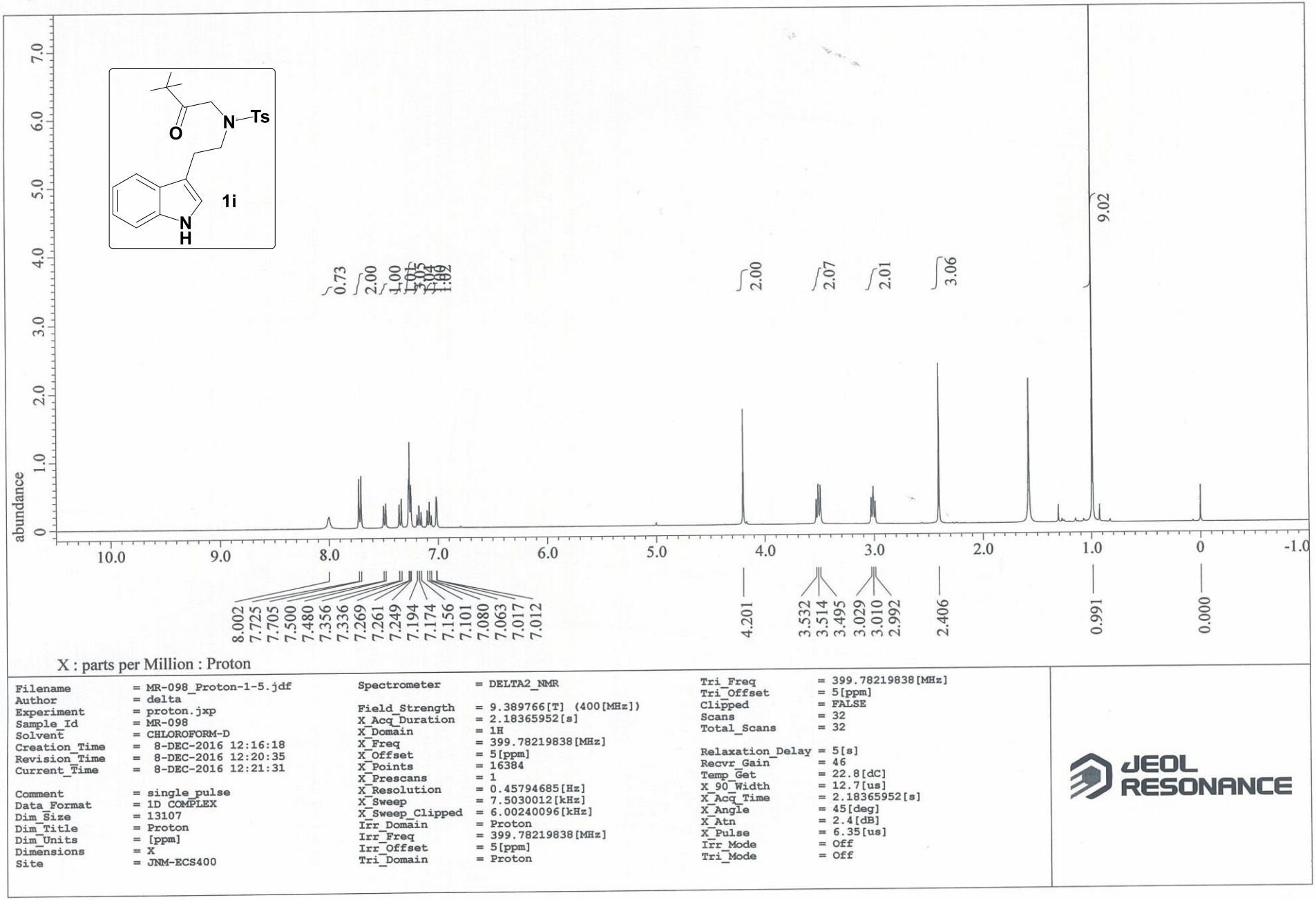
Pulse Sequence: s2pul

Mercury-400BB "MerPlus400"
Date: Aug 8 2016
Solvent: cdcl3
Ambient temperature
Total 1584 repetitions



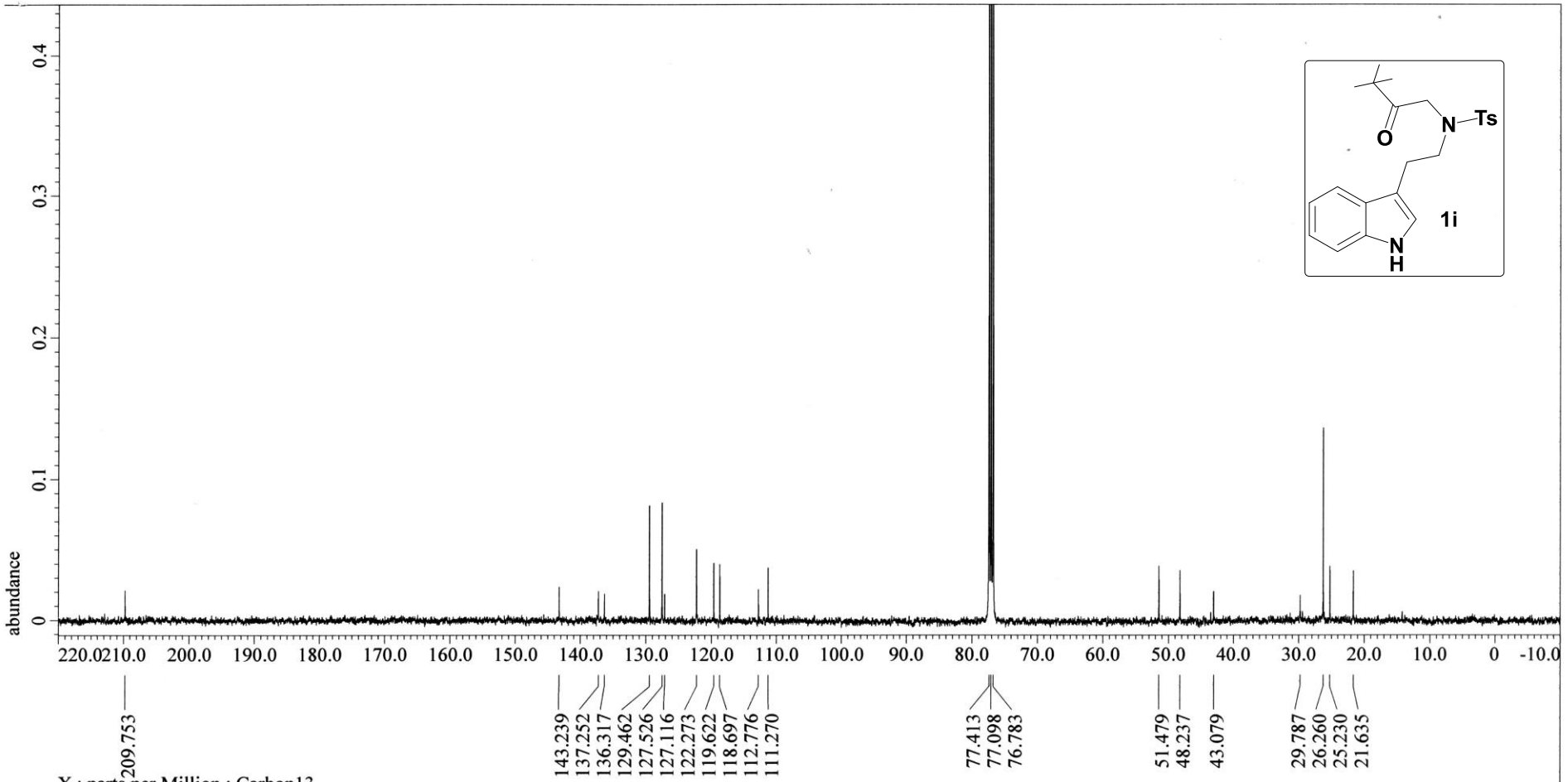



SIVA-07-176
Pulse Sequence: DEPT
193.913
143.082
136.554
136.080
135.405
132.015
131.900
129.503
129.350
129.278
129.224
128.518
128.331
127.464
127.132
126.773
126.651
126.613
123.147
122.414
122.230
121.352
118.741
118.646
118.077
111.343
111.125
77.317
77.000
76.679
53.285
48.674
43.035
39.722
25.326
24.516
21.226
180
160
140
120
100
80
60
40
20
ppm
O
N
Ts
N
H
1h



Filename	= MR-098_Proton-1-5.jdf	Spectrometer	= DELTA2_NMR	Tri_Freq	= 399.78219838[MHz]
Author	= delta			Tri_Offset	= 5[ppm]
Experiment	= proton.jxp	Field_Strength	= 9.389766[T] (400[MHz])	Clipped	= FALSE
Sample_Id	= MR-098	X_Acq_Duration	= 2.18365952[s]	Scans	= 32
Solvent	= CHLOROFORM-D	X_Domain	= 1H	Total_Scans	= 32
Creation_Time	= 8-DEC-2016 12:16:18	X_Freq	= 399.78219838[MHz]		
Revision_Time	= 8-DEC-2016 12:20:35	X_Offset	= 5[ppm]	Relaxation_Delay	= 5[s]
Current_Time	= 8-DEC-2016 12:21:31	X_Points	= 16384	Recvr_Gain	= 46
		X_Prescans	= 1	Temp_Get	= 22.8[dC]
Comment	= single_pulse	X_Resolution	= 0.45794685[Hz]	X_90_Width	= 12.7[us]
Data_Format	= 1D COMPLEX	X_Sweep	= 7.5030012[kHz]	X_Acq_Time	= 2.18365952[s]
Dim_Size	= 13107	X_Sweep_Clipped	= 6.00240096[kHz]	X_Angle	= 45[deg]
Dim_Title	= Proton	Irr_Domain	= Proton	X_Atn	= 2.4[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	X_Pulse	= 6.35[us]
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Mode	= Off
Site	= JNM-ECS400	Tri_Domain	= Proton	Tri_Mode	= Off

JEOL RESONANCE

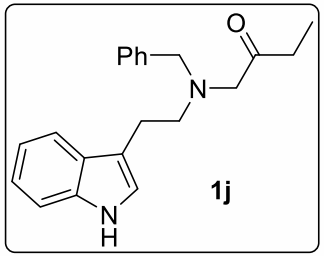


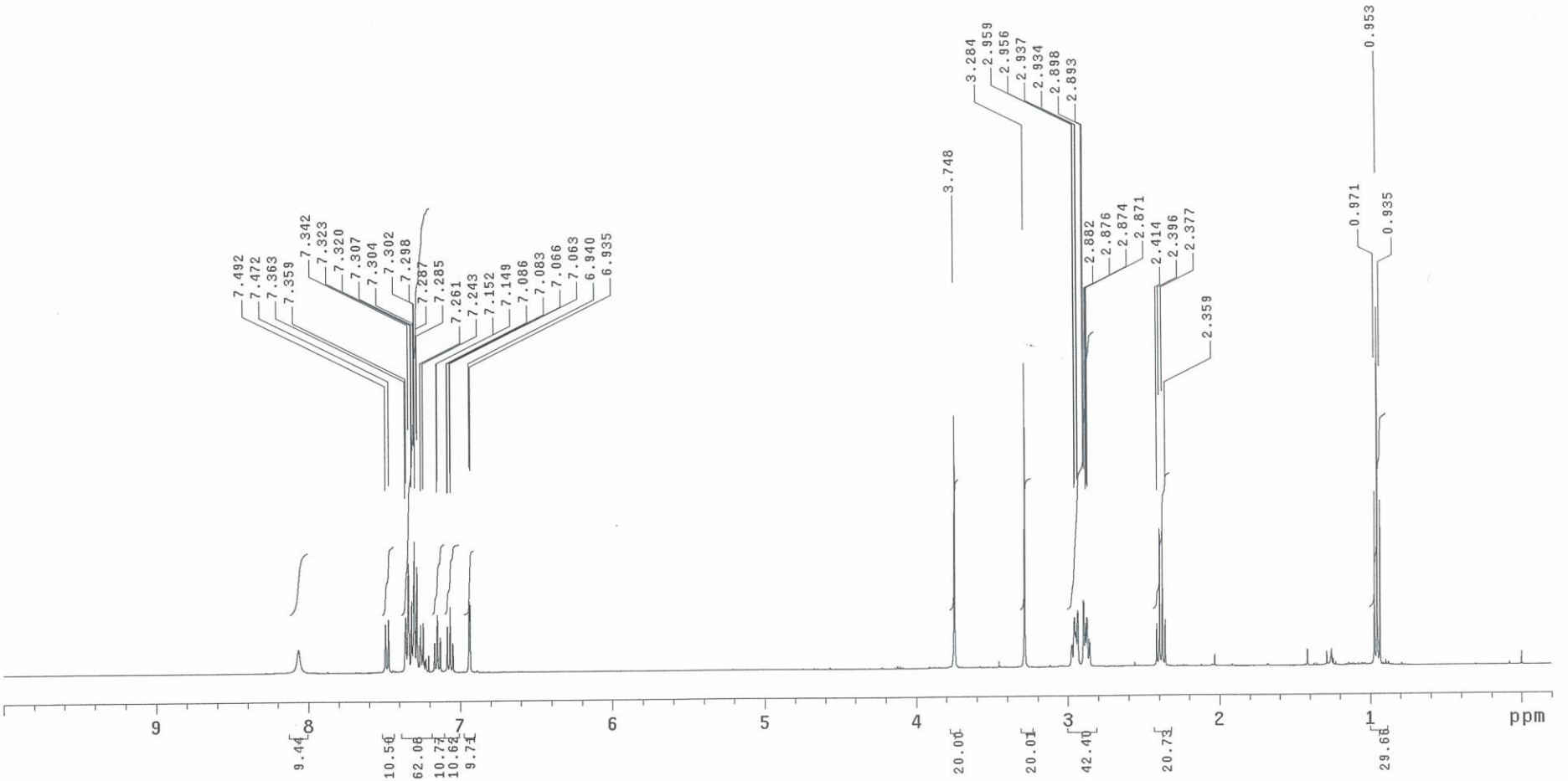
Filename	= MR-098_Carbon-1-2.jdf	Spectrometer	= DELTA2_NMR	Scans	= 3000
Author	= delta			Total_Scans	= 3000
Experiment	= carbon.jxp	Field_Strength	= 9.389766[T] (400[MHz])		
Sample_Id	= MR-098	X_Acq_Duration	= 1.04333312[s]	Relaxation_Delay	= 2[s]
Solvent	= CHLOROFORM-D	X_Domain	= 13C	Recvr_Gain	= 60
Creation_Time	= 10-DEC-2016 14:37:22	X_Freq	= 100.52530333[MHz]	Temp_Get	= 22.9[dC]
Revision_Time	= 10-DEC-2016 17:01:46	X_Offset	= 100[ppm]	X_90_Width	= 10.37[us]
Current_Time	= 10-DEC-2016 17:04:20	X_Points	= 32768	X_Acq_Time	= 1.04333312[s]
		X_Prescans	= 4	X_Angle	= 30[deg]
Comment	= single pulse decoupled gat	X_Resolution	= 0.95846665[Hz]	X_Atn	= 5.3[dB]
Data_Format	= 1D COMPLEX	X_Sweep	= 31.40703518[kHz]	X_Pulse	= 3.45666667[us]
Dim_Size	= 26214	X_Sweep_Clipped	= 25.12562814[kHz]	Irr_Atn_Dec	= 21.538[dB]
Dim_Title	= Carbon13	Irr_Domain	= Proton	Irr_Atn_Noe	= 21.538[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	Irr_Noise	= WALTZ
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Pwidth	= 0.115[ms]
Site	= JNM-ECS400	Clipped	= TRUE	Decoupling	= TRUE

JEOL RESONANCE

MR-085

Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Nov 25 2016
Solvent: cdcl3
Ambient temperature
Total 32 repetitions

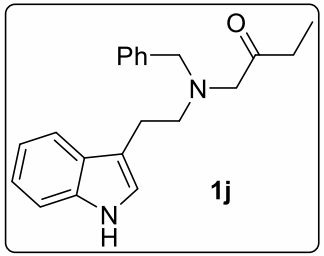


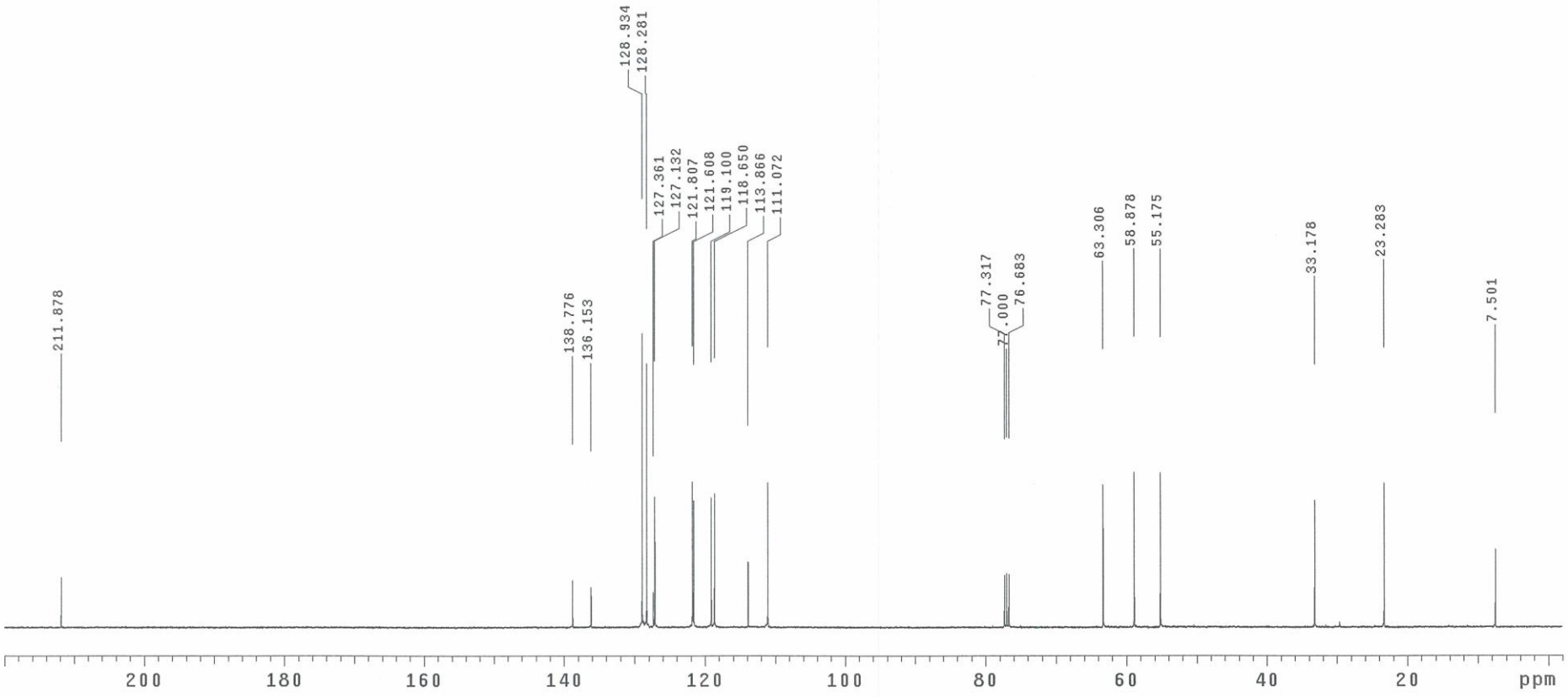


MR-085

Pulse Sequence: s2pul

Mercury-400BB "MerPlus400"
Date: Nov 25 2016
Solvent: cdcl3
Ambient temperature
Total 896 repetitions

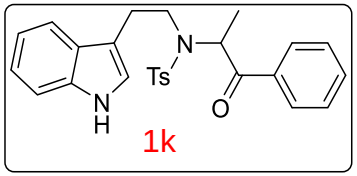


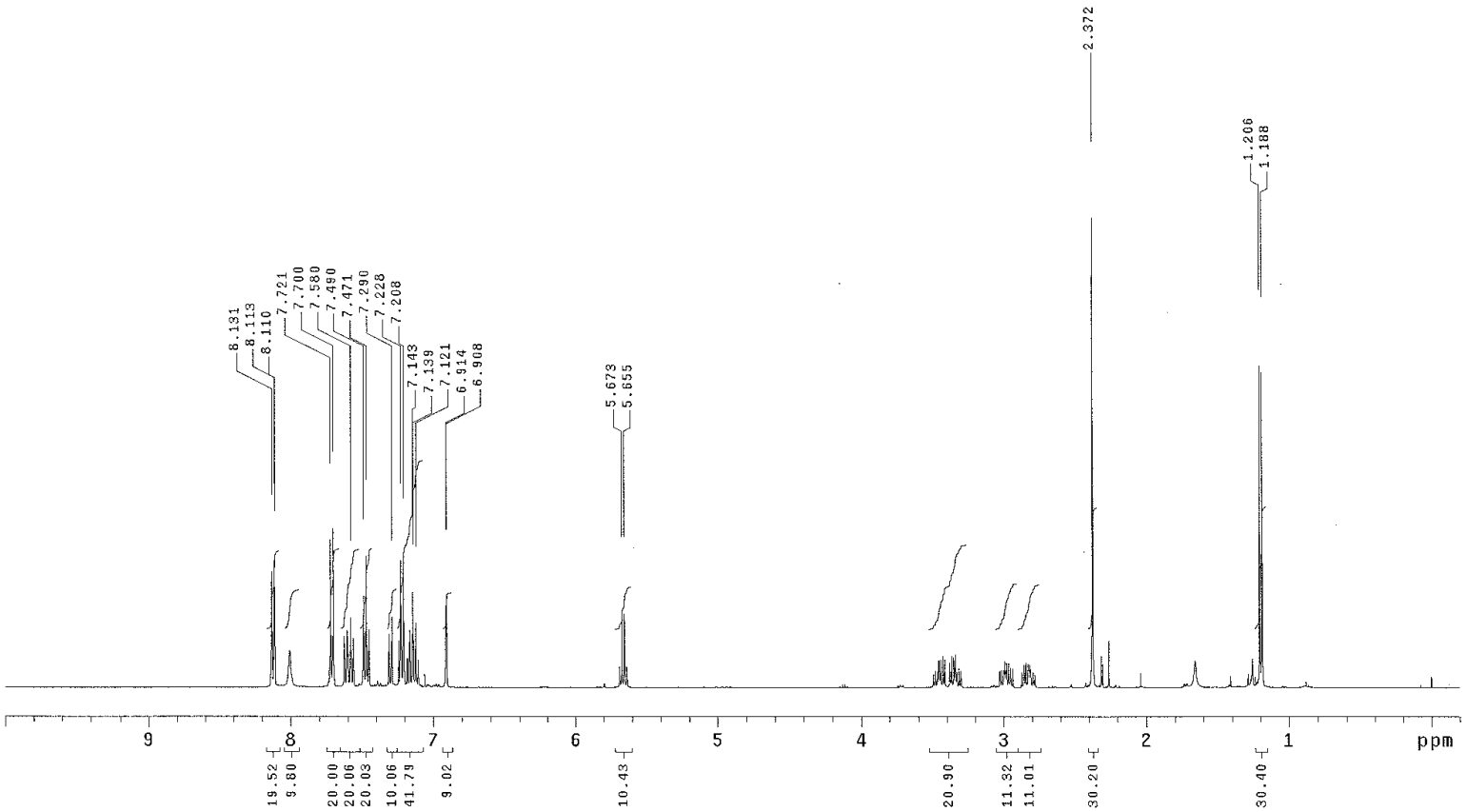


Hou-03-36

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Aug 8 2016
Solvent: CDCl3
Ambient temperature
Total 32 repetitions

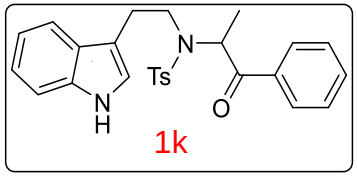


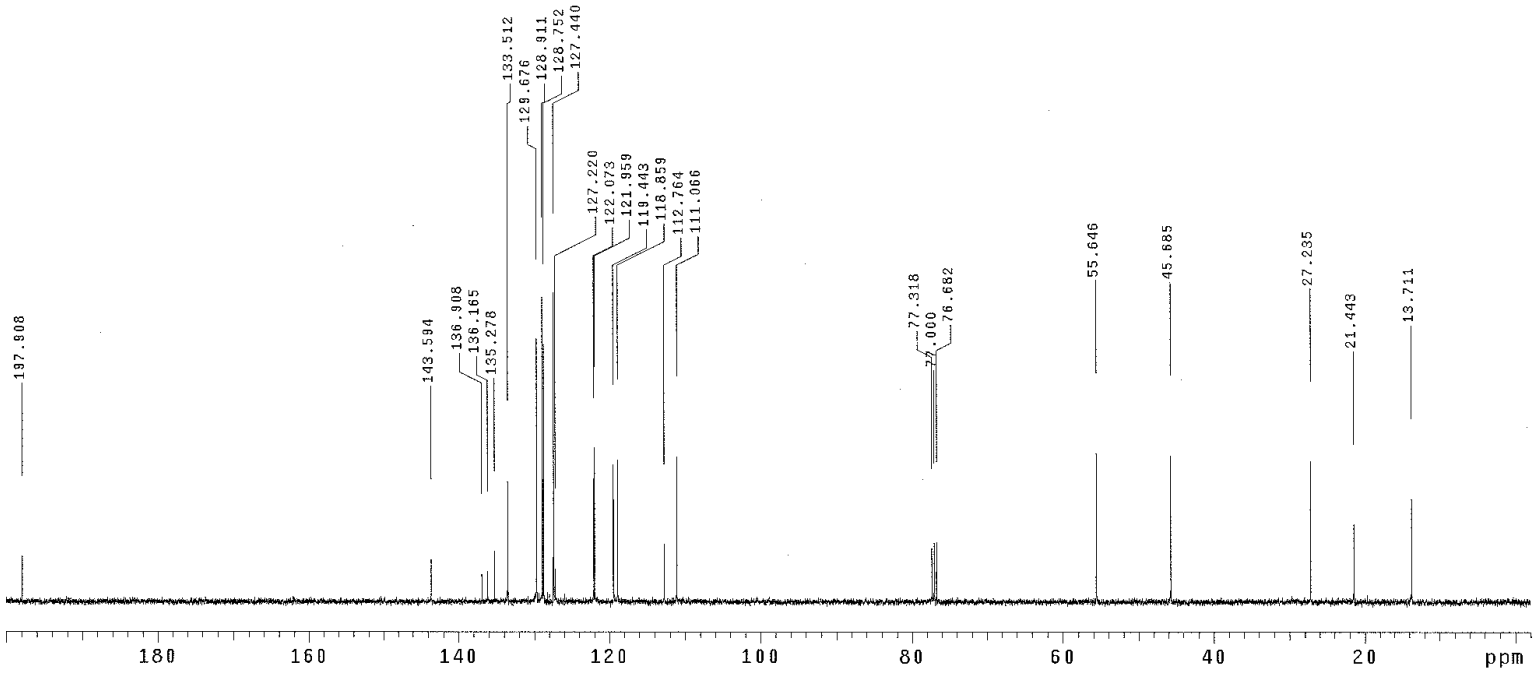


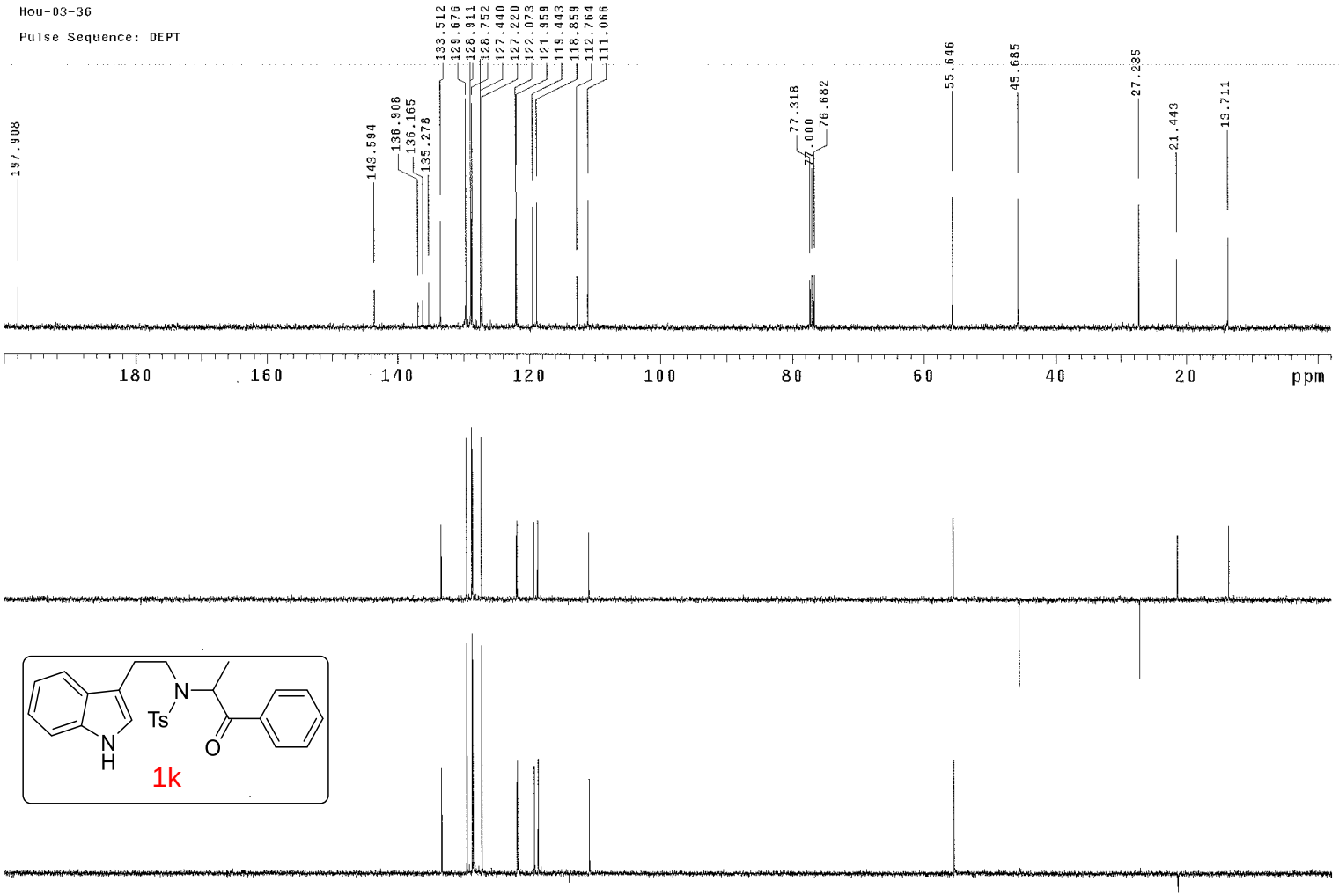
Hou-03-36

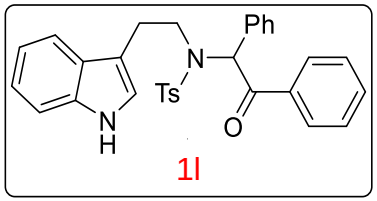
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Aug 8 2016
Solvent: CDCl3
Ambient temperature
Total 1024 repetitions





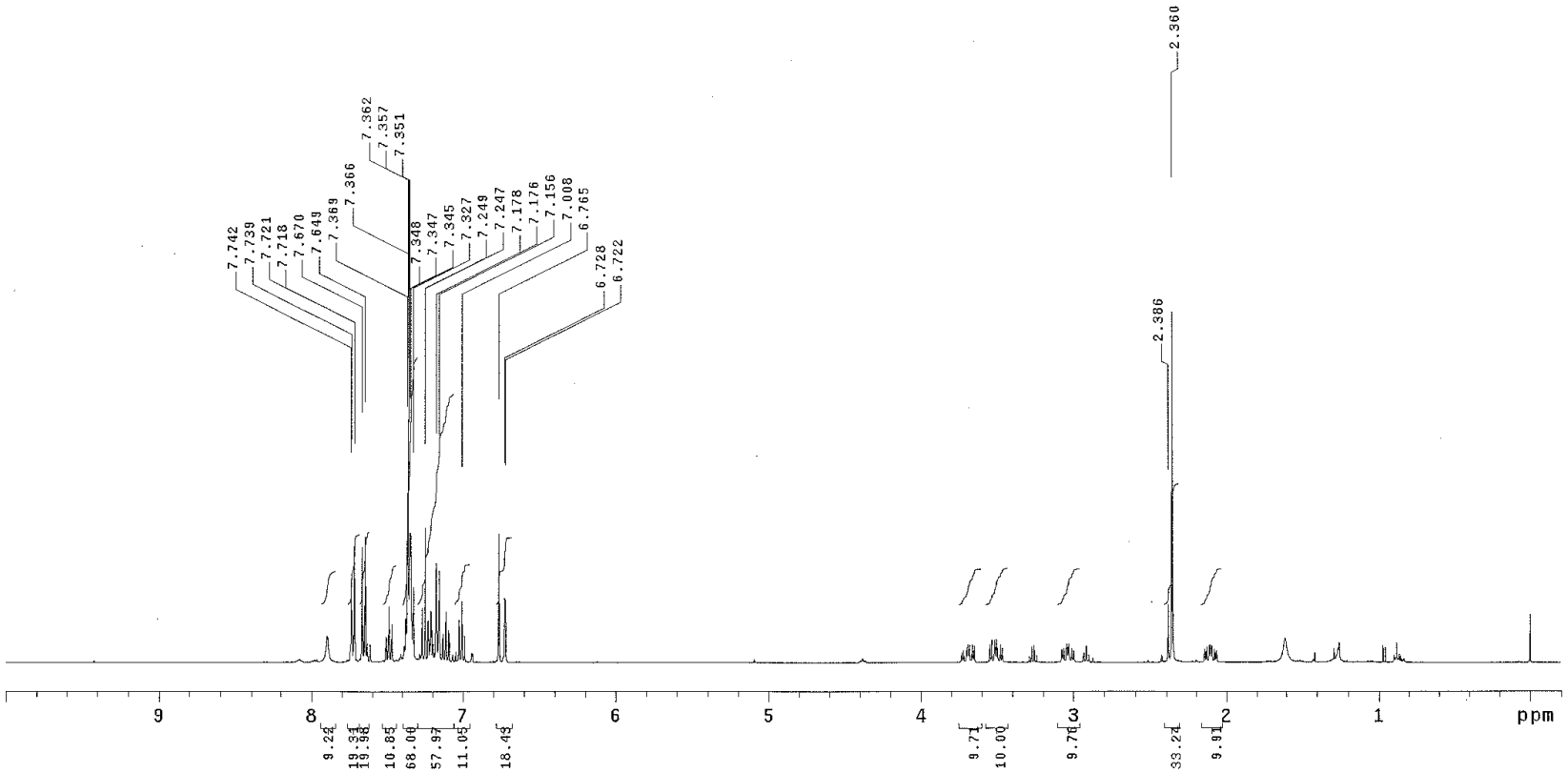

Hou-03-36
Pulse Sequence: DEPT
197.908
143.594
136.908
136.165
135.278
133.512
129.676
128.911
128.752
127.440
127.220
122.073
121.959
119.443
118.859
112.764
111.066
77.318
77.000
76.682
55.646
45.685
27.235
21.443
13.711
180
160
140
120
100
80
60
40
20
ppm
N
Ts
O
N
H
1k



1l

Hou-03-61

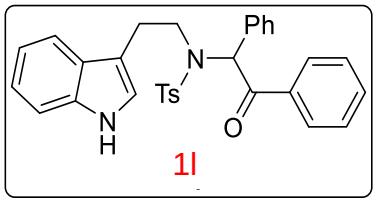
Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Aug 1 2016
Solvent: cdcl3
Ambient temperature
Total 32 repetitions



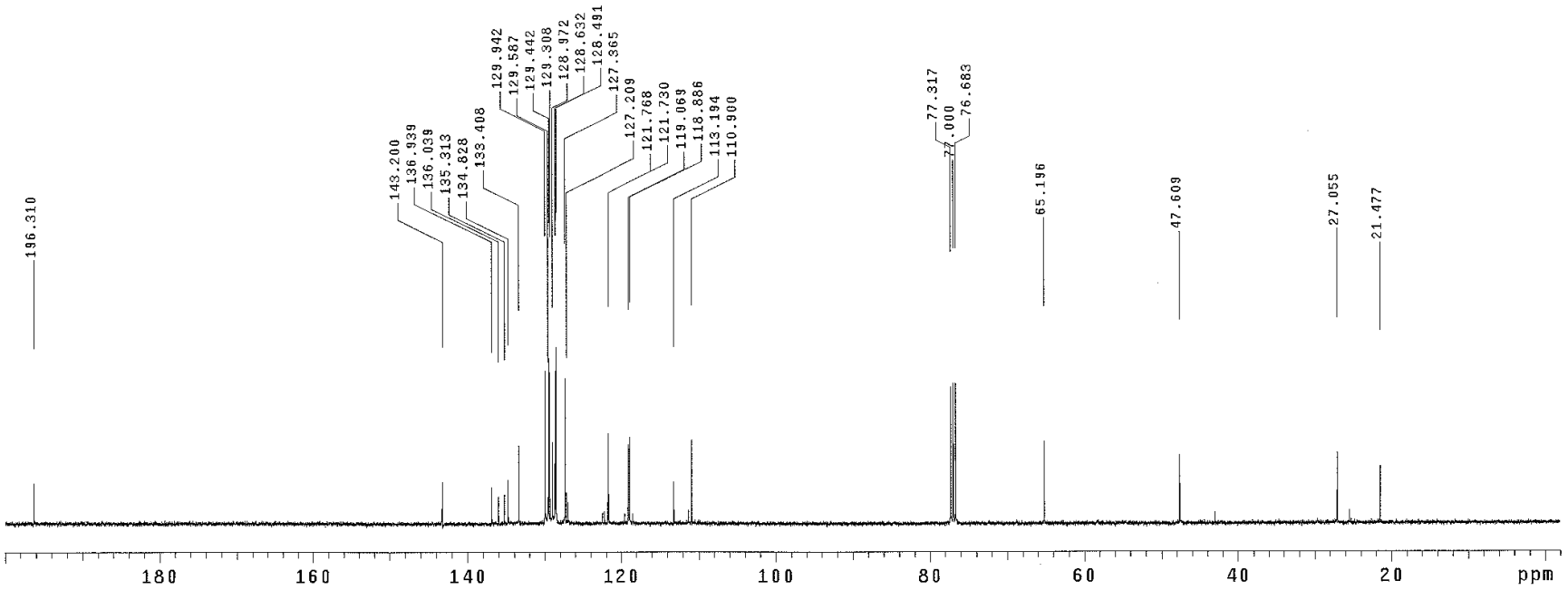
Hou-03-61

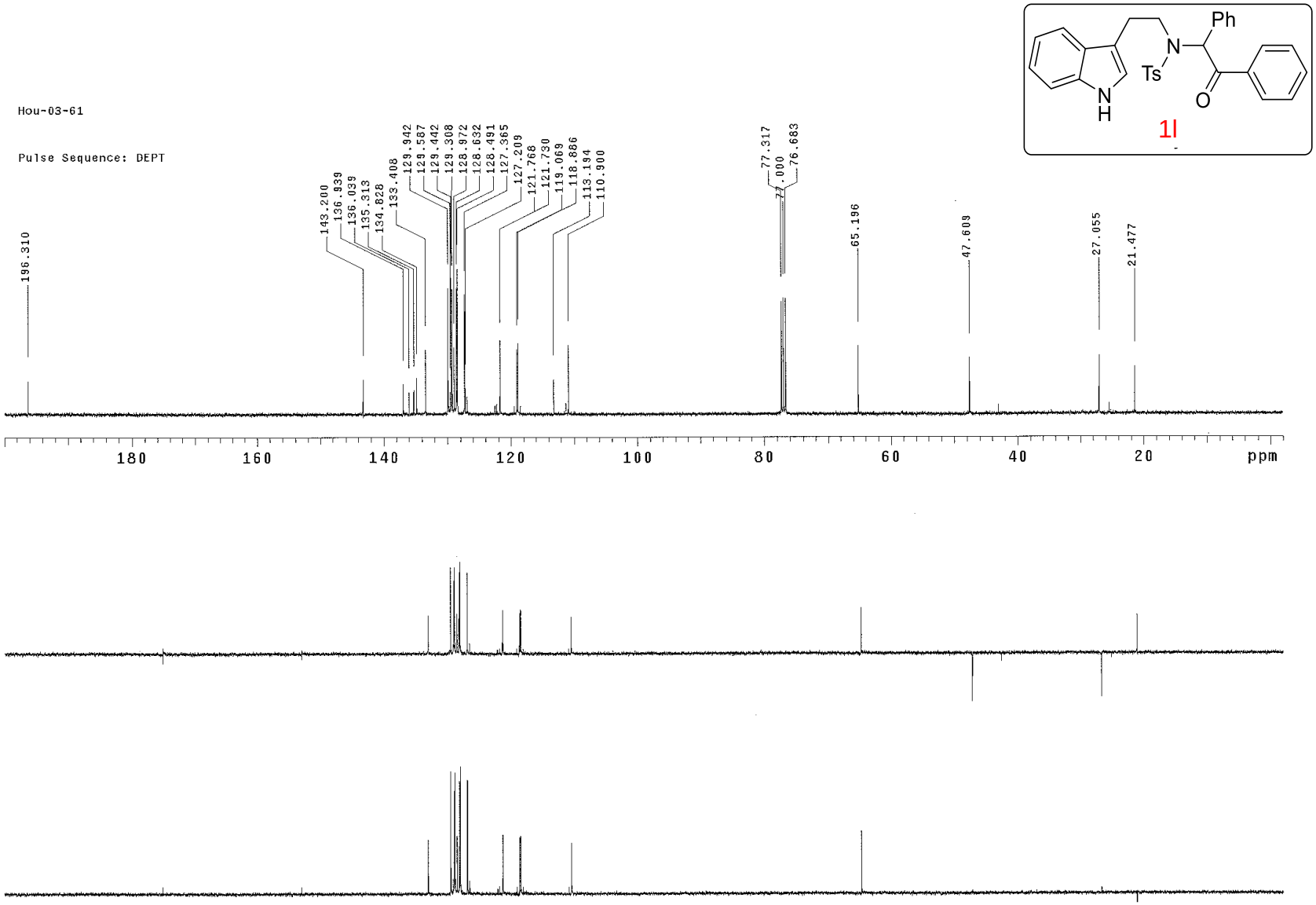
Pulse Sequence: s2pul

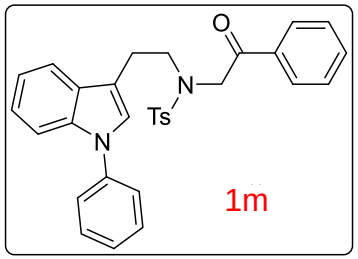
Mercury-400BB "MerPlus400"
Date: Aug 1 2016
Solvent: cdcl3
Ambient temperature
Total 1552 repetitions



1l



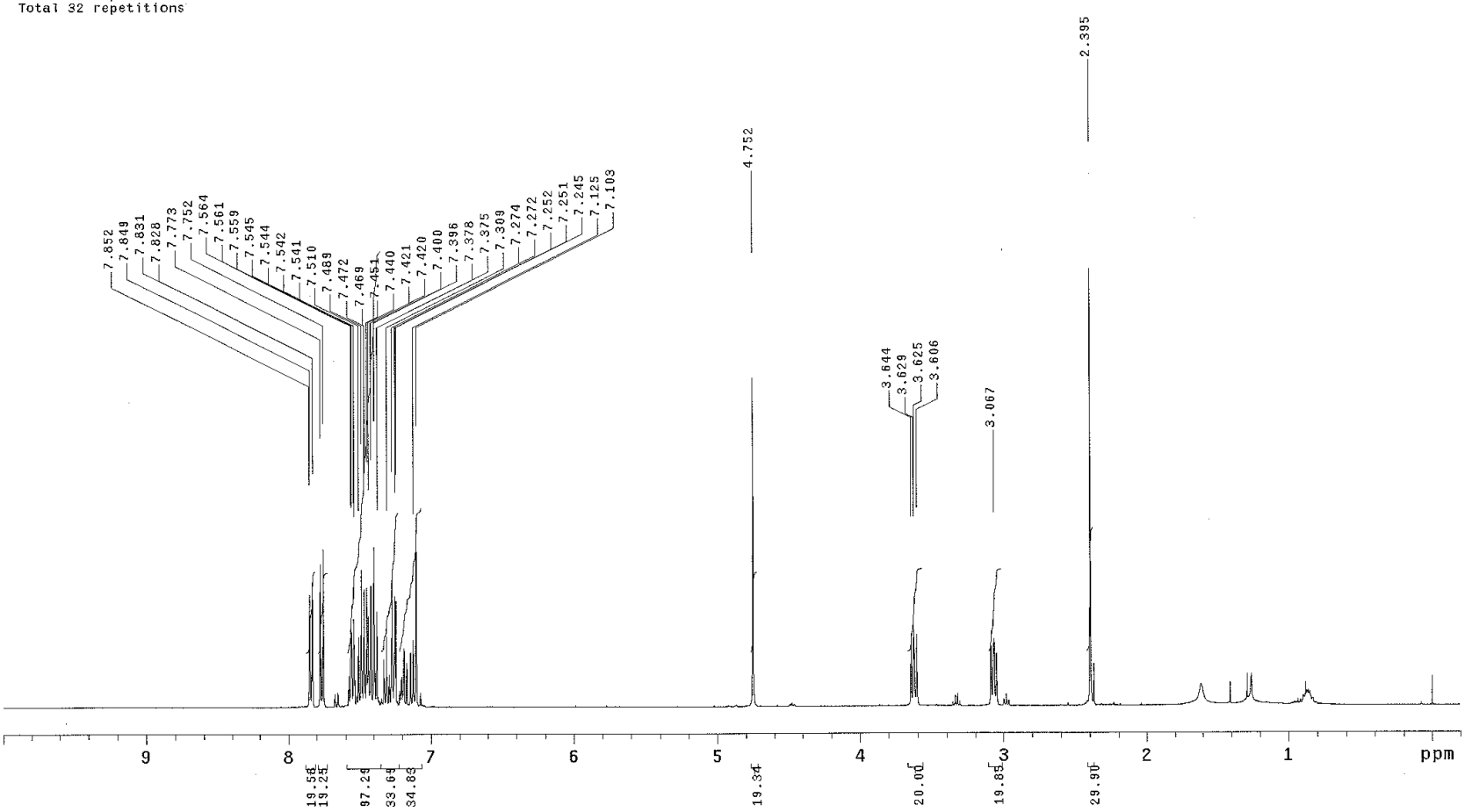

Hou-03-61
Pulse Sequence: DEPT
196.310
143.200
136.939
136.039
135.313
134.828
133.408
129.942
129.587
129.442
129.308
128.972
128.632
128.491
127.365
127.209
121.768
121.730
119.069
118.886
113.194
110.900
77.317
77.000
76.683
65.196
47.609
27.055
21.477
180
160
140
120
100
80
60
40
20
ppm
Ph
N
Ts
O
N
H
1l



Hou-02-152

Pulse Sequence: s2pul

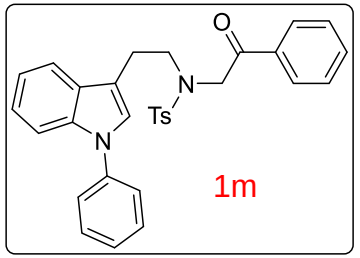
Mercury-400BB "MerPlus400"
Date: Aug 8 2016
Solvent: cdcl3
Ambient temperature
Total 32 repetitions



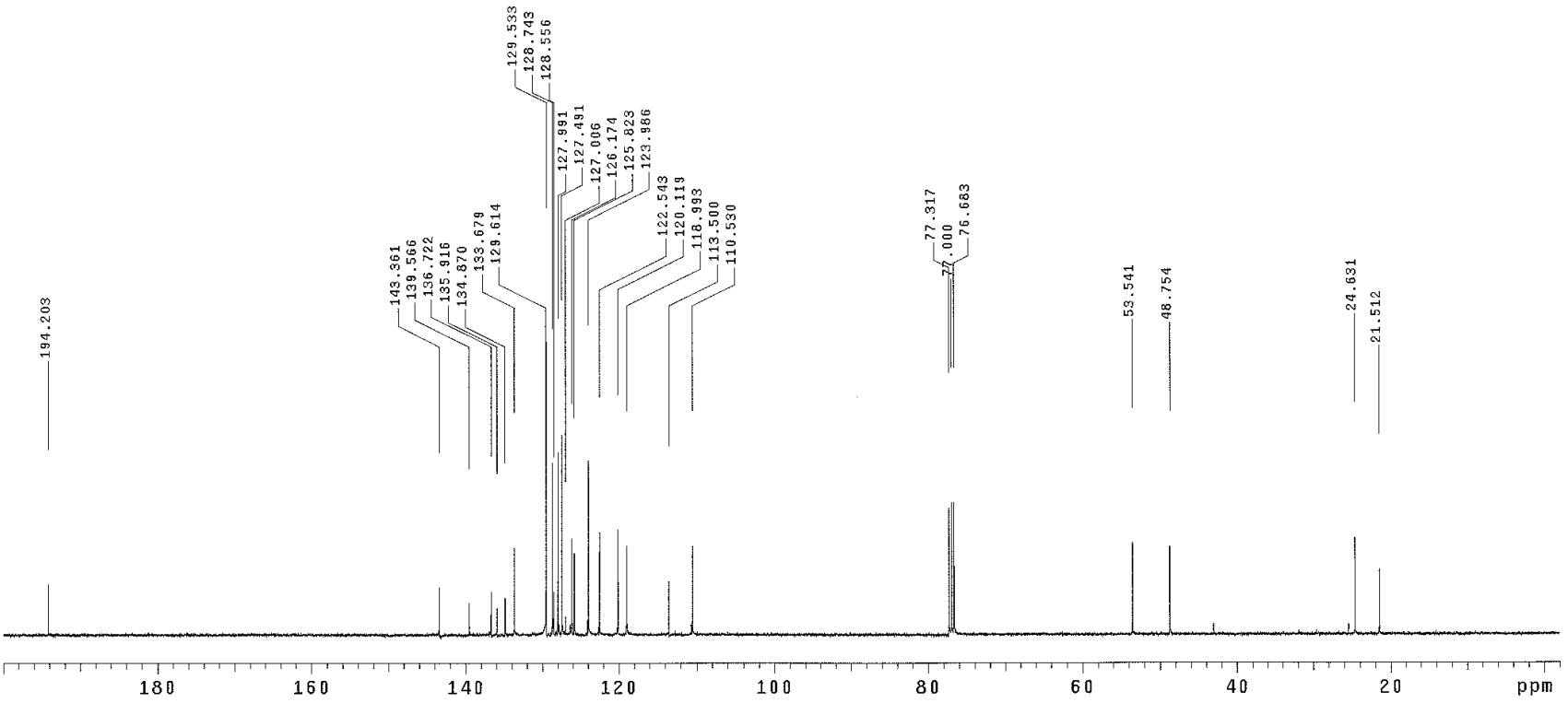
Hou-02-152

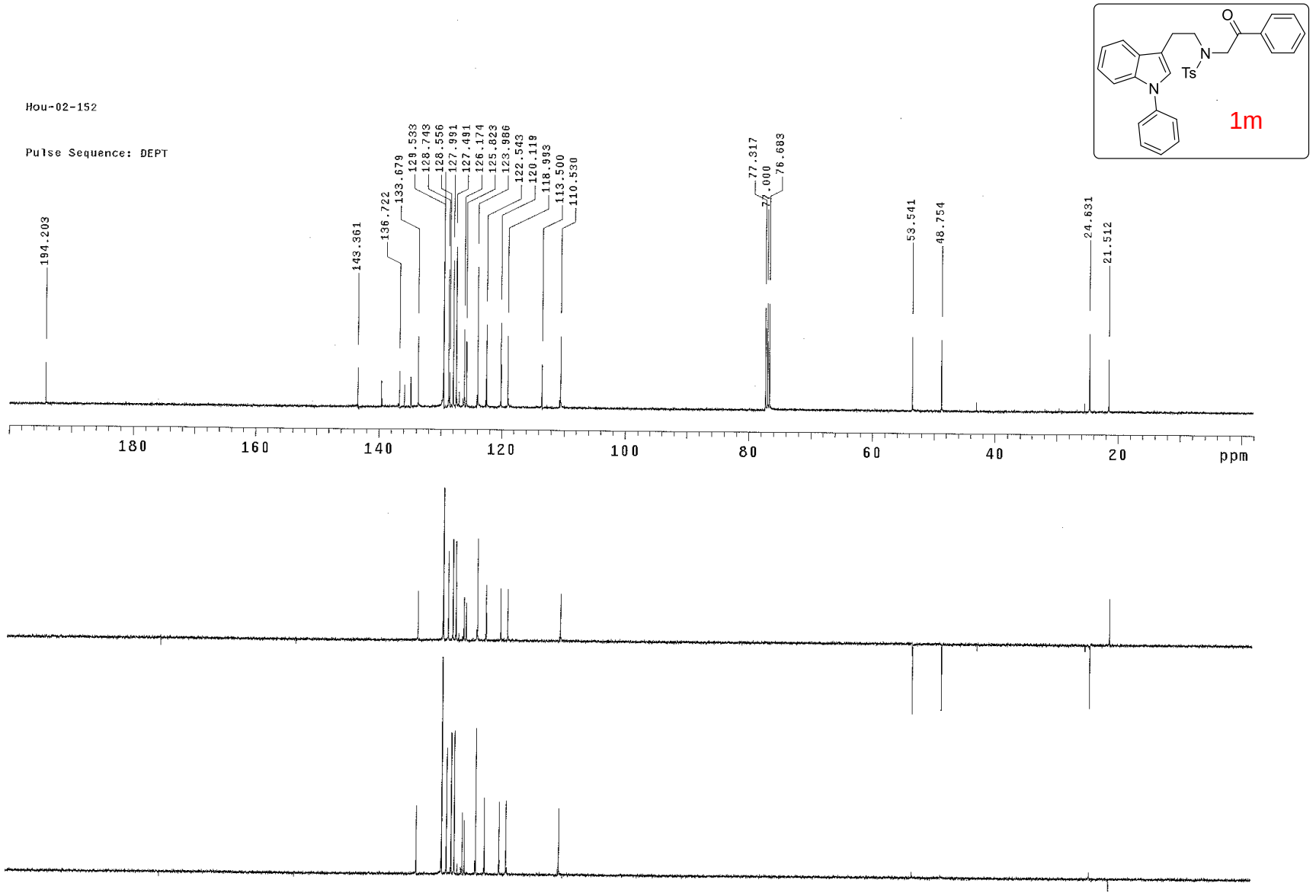
Pulse Sequence: s2pul

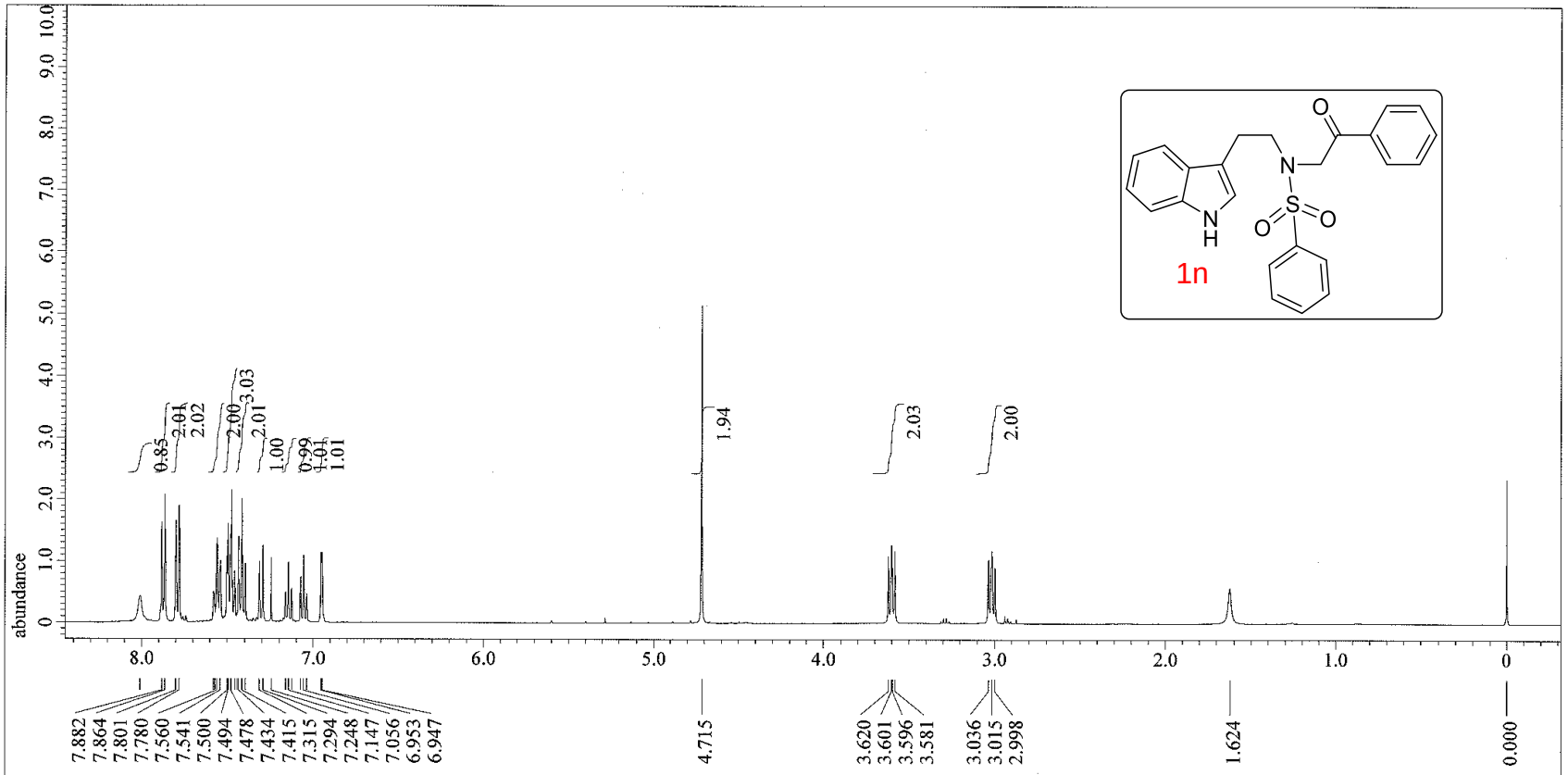
Mercury-400BB "MerPlus400"
Date: Aug 8 2016
Solvent: cdcl3
Ambient temperature
Total 2688 repetitions



1m

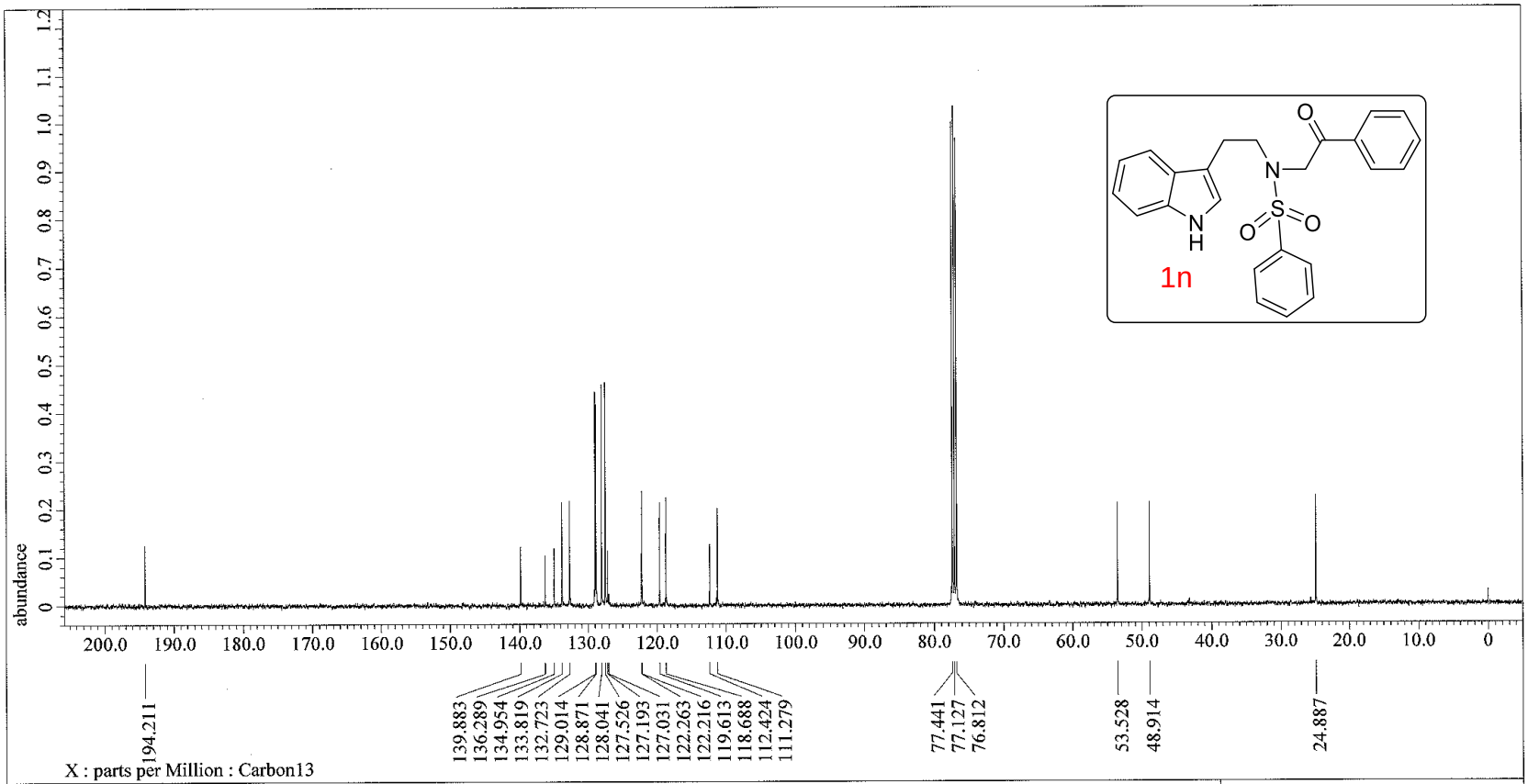



Hou-02-152
Pulse Sequence: DEPT
194.203
143.361
136.722
133.679
129.533
128.743
128.556
127.991
127.491
126.174
125.823
123.986
122.543
120.119
118.993
113.500
110.530
77.317
77.000
76.683
53.541
48.754
24.631
21.512
180
160
140
120
100
80
60
40
20
ppm
O
N
Ts
N
1m



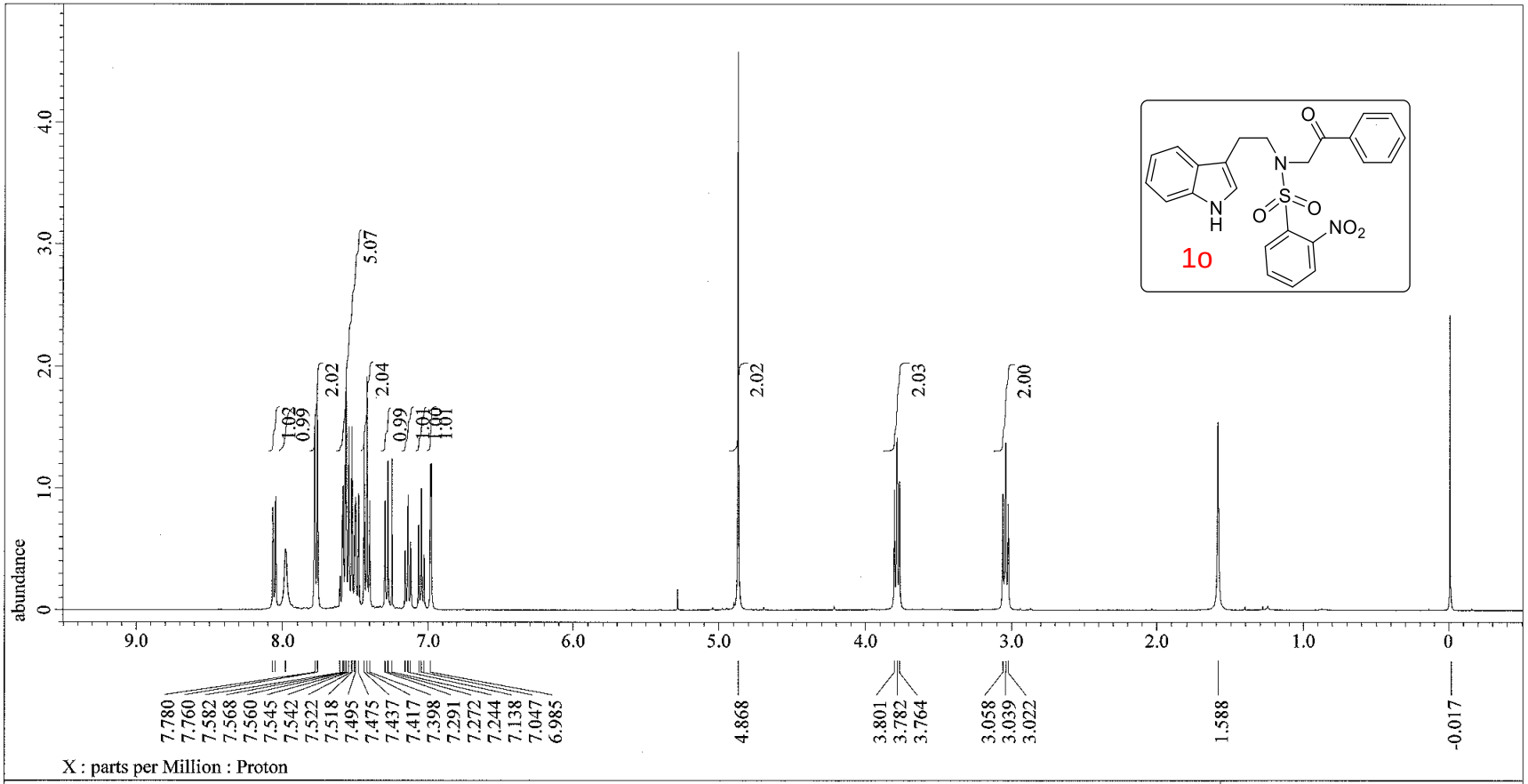
Filename	= Hou-02-151_Proton-1-5.jdf	Spectrometer	= DELTA2_NMR	Tri_Freq	= 399.78219838[MHz]
Author	= delta			Tri_Offset	= 5[ppm]
Experiment	= proton.jxp	Field_Strength	= 9.389766[T] (400[MHz])	Clipped	= FALSE
Sample_Id	= Hou-02-151	X_Acq_Duration	= 2.18365952[s]	Scans	= 16
Solvent	= CHLOROFORM-D	X_Domain	= 1H	Total_Scans	= 16
Creation_Time	= 20-JUL-2016 11:25:22	X_Freq	= 399.78219838[MHz]		
Revision_Time	= 20-JUL-2016 11:23:20	X_Offset	= 5[ppm]	Relaxation_Delay	= 5[s]
Current_Time	= 20-JUL-2016 11:24:04	X_Points	= 16384	Recvr_Gain	= 38
		X_Prescans	= 1	Temp_Get	= 26.1[dC]
Comment	= single_pulse	X_Resolution	= 0.45794685[Hz]	X_90_Width	= 12.795[us]
Data_Format	= 1D COMPLEX	X_Sweep	= 7.5030012[kHz]	X_Acq_Time	= 2.18365952[s]
Dim_Size	= 13107	X_Sweep_Clipped	= 6.00240096[kHz]	X_Angle	= 45[deg]
Dim_Title	= Proton	Irr_Domain	= Proton	X_Atn	= 2.4[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	X_Pulse	= 6.3975[us]
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Mode	= Off
Site	= JNM-ECS400	Tri_Domain	= Proton	Tri_Mode	= Off

JEOL RESONANCE

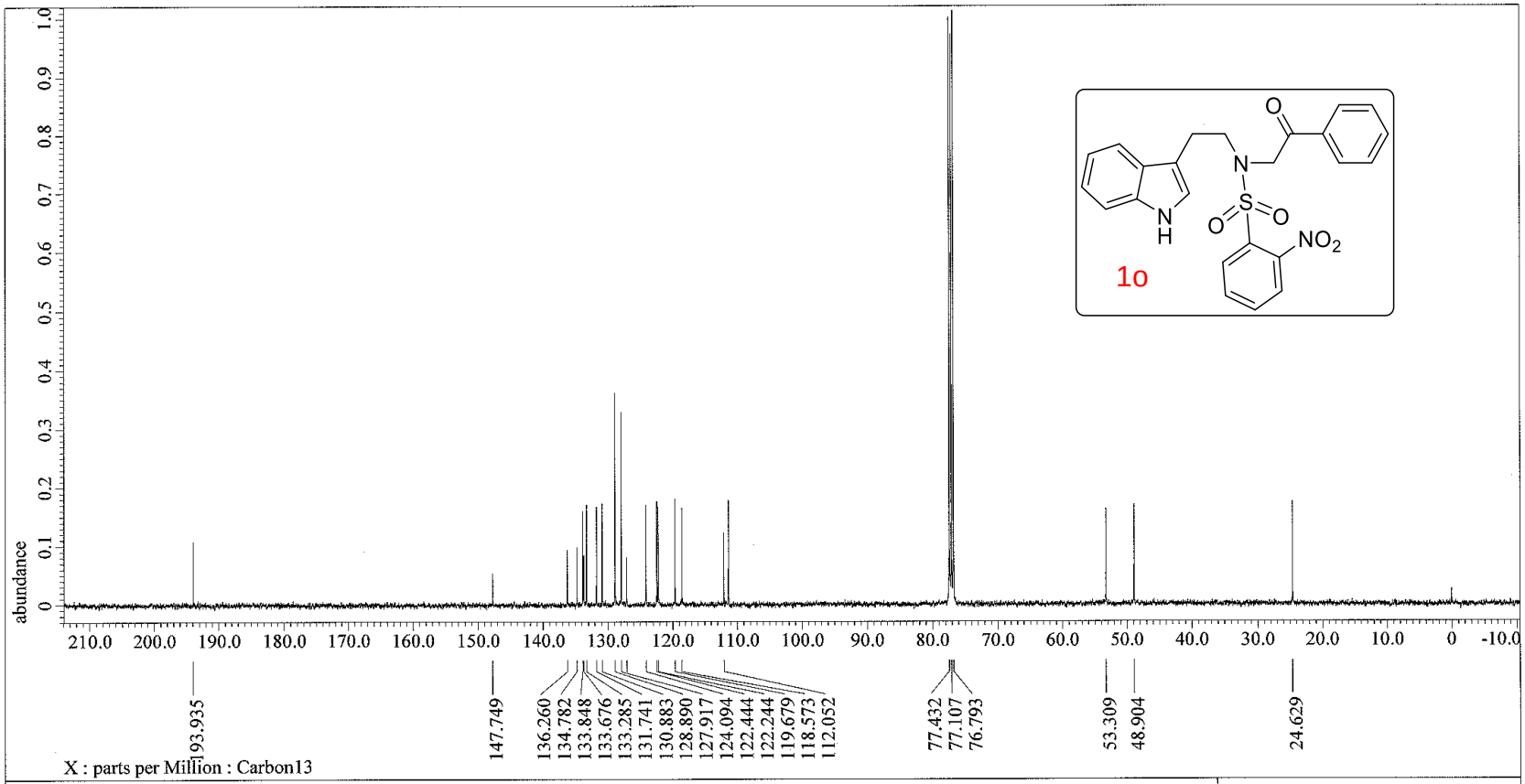


Filename	= Hou-02-151_Carbon-1-3.jdf	Spectrometer	= DELTA2_NMR	Scans	= 1024
Author	= delta			Total_Scans	= 1024
Experiment	= carbon.jxp	Field_Strength	= 9.389766[T] (400[MHz])		
Sample_Id	= Hou-02-151	X_Acq_Duration	= 1.04333312[s]	Relaxation_Delay	= 2[s]
Solvent	= CHLOROFORM-D	X_Domain	= 13C	Recvr_Gain	= 60
Creation_Time	= 20-JUL-2016 11:29:47	X_Freq	= 100.52530333[MHz]	Temp_Get	= 26[dC]
Revision_Time	= 20-JUL-2016 12:53:18	X_Offset	= 100[ppm]	X_90_Width	= 10.33[us]
Current_Time	= 20-JUL-2016 12:53:34	X_Points	= 32768	X_Acq_Time	= 1.04333312[s]
		X_Prescans	= 4	X_Angle	= 30[deg]
Comment	= single pulse decoupled gat	X_Resolution	= 0.95846665[Hz]	X_Atn	= 5.3[dB]
Data_Format	= 1D COMPLEX	X_Sweep	= 31.40703518[kHz]	X_Pulse	= 3.44333333[us]
Dim_Size	= 26214	X_Sweep_Clipped	= 25.12562814[kHz]	Irr_Atn_Dec	= 21.473[dB]
Dim_Title	= Carbon13	Irr_Domain	= Proton	Irr_Atn_Noe	= 21.473[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	Irr_Noise	= WALTZ
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Pwidth	= 0.115[ms]
Site	= JNM-ECS400	Clipped	= FALSE	Decoupling	= TRUE

JEOL RESONANCE



Filename	= Hou-02-160_Proton-1-3.jdf	Spectrometer	= DELTA2_NMR	Tri_Freq	= 399.78219838[MHz]
Author	= delta			Tri_Offset	= 5[ppm]
Experiment	= proton.jxp	Field_Strength	= 9.389766[T] (400[MHz])	Clipped	= FALSE
Sample_Id	= Hou-02-160	X_Acq_Duration	= 2.18365952[s]	Scans	= 16
Solvent	= CHLOROFORM-D	X_Domain	= 1H	Total_Scans	= 16
Creation_Time	= 20-JUL-2016 13:03:29	X_Freq	= 399.78219838[MHz]		
Revision_Time	= 20-JUL-2016 14:46:02	X_Offset	= 5[ppm]	Relaxation_Delay	= 5[s]
Current_Time	= 20-JUL-2016 14:46:37	X_Points	= 16384	Recvr_Gain	= 40
		X_Prescans	= 1	Temp_Get	= 25.5[dC]
Comment	= single_pulse	X_Resolution	= 0.45794685[Hz]	X_90_Width	= 12.795[us]
Data_Format	= 1D COMPLEX	X_Sweep	= 7.5030012[kHz]	X_Acq_Time	= 2.18365952[s]
Dim_Size	= 13107	X_Sweep_Clipped	= 6.00240096[kHz]	X_Angle	= 45[deg]
Dim_Title	= Proton	Irr_Domain	= Proton	X_Atn	= 2.4[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	X_Pulse	= 6.3975[us]
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Mode	= Off
Site	= JNM-ECS400	Tri_Domain	= Proton	Tri_Mode	= Off

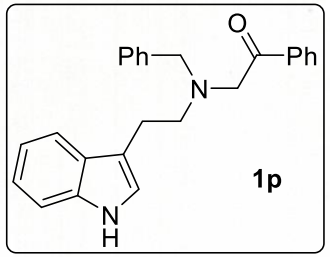


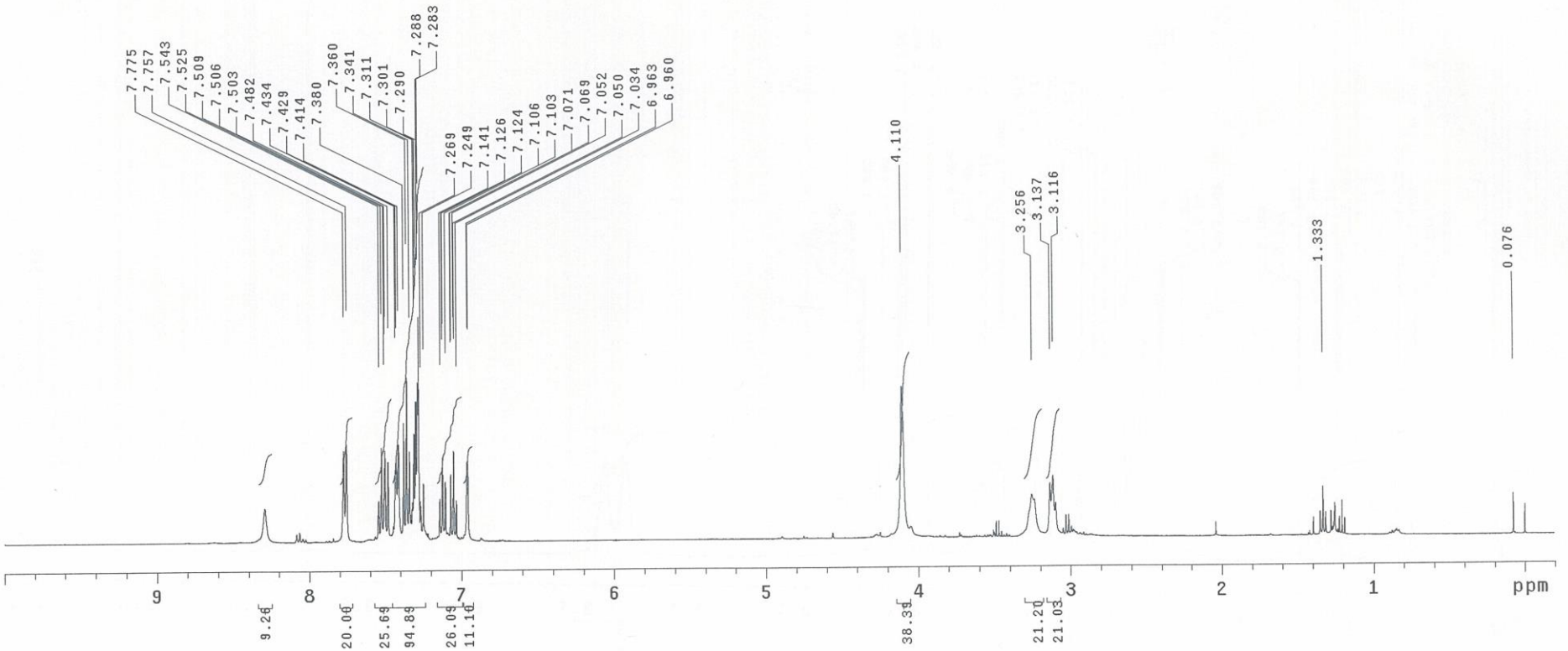
Filename	= Hou-02-160_Carbon-1-3.jdf	Spectrometer	= DELTA2_NMR	Scans	= 1024
Author	= delta			Total_Scans	= 1024
Experiment	= carbon.jxp	Field_Strength	= 9.389766[T] (400[MHz])		
Sample_Id	= Hou-02-160	X_Acq_Duration	= 1.04333312[s]	Relaxation_Delay	= 2[s]
Solvent	= CHLOROFORM-D	X_Domain	= 13C	Recvr_Gain	= 60
Creation_Time	= 20-JUL-2016 13:07:50	X_Freq	= 100.52530333[MHz]	Temp_Get	= 25.6[dC]
Revision_Time	= 20-JUL-2016 14:40:55	X_Offset	= 100[ppm]	X_90_Width	= 10.33[us]
Current_Time	= 20-JUL-2016 14:41:11	X_Points	= 32768	X_Acq_Time	= 1.04333312[s]
		X_Prescans	= 4	X_Angle	= 30[deg]
Comment	= single pulse decoupled gat	X_Resolution	= 0.95846665[Hz]	X_Atn	= 5.3[dB]
Data_Format	= 1D COMPLEX	X_Sweep	= 31.40703518[kHz]	X_Pulse	= 3.44333333[us]
Dim_Size	= 26214	X_Sweep_Clipped	= 25.12562814[kHz]	Irr_Atn_Dec	= 21.473[dB]
Dim_Title	= Carbon13	Irr_Domain	= Proton	Irr_Atn_Noe	= 21.473[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	Irr_Noise	= WALTZ
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Pwidth	= 0.115[ms]
Site	= JNM-ECS400	Clipped	= FALSE	Decoupling	= TRUE

MR-081

Pulse Sequence: s2pul

Mercury-400BB "MerPlus400"
Date: Nov 28 2016
Solvent: cdcl3
Ambient temperature
Total 32 repetitions

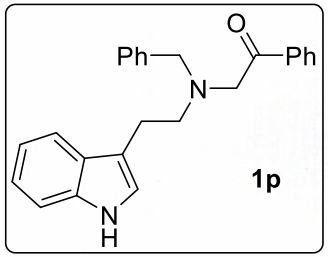


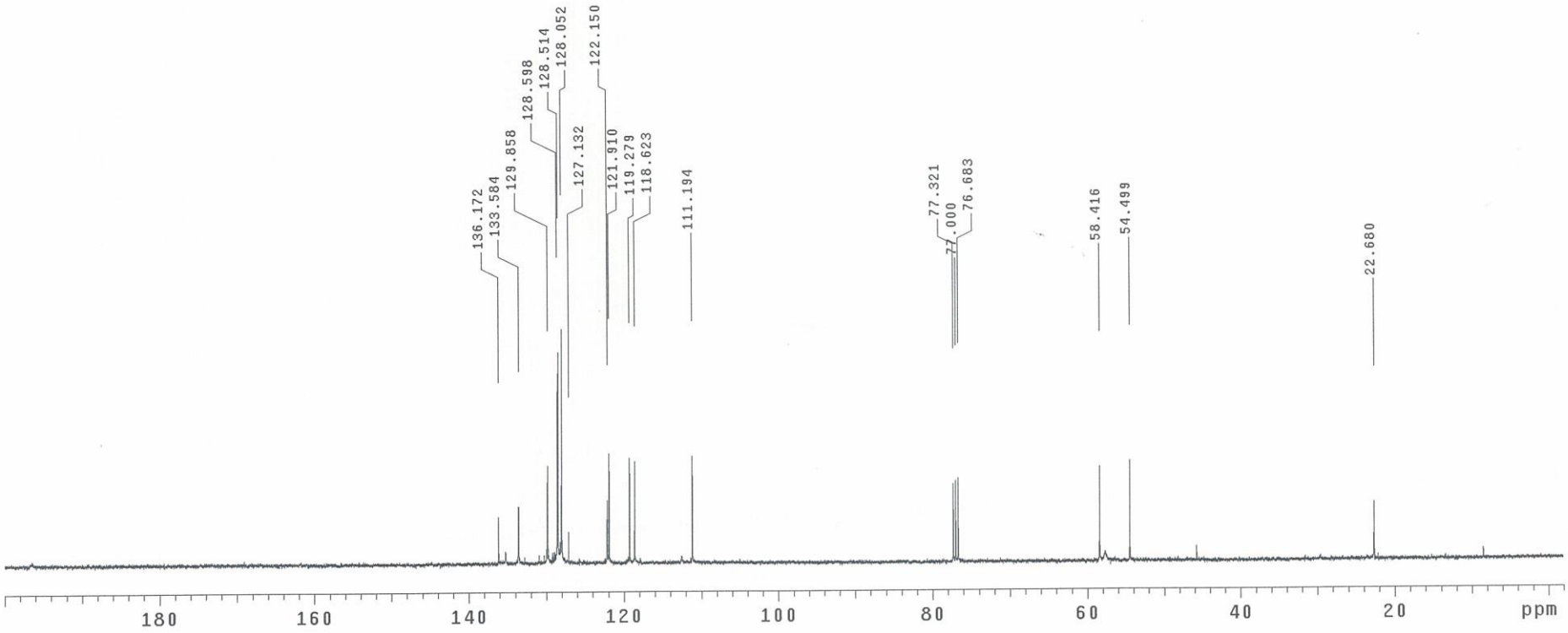


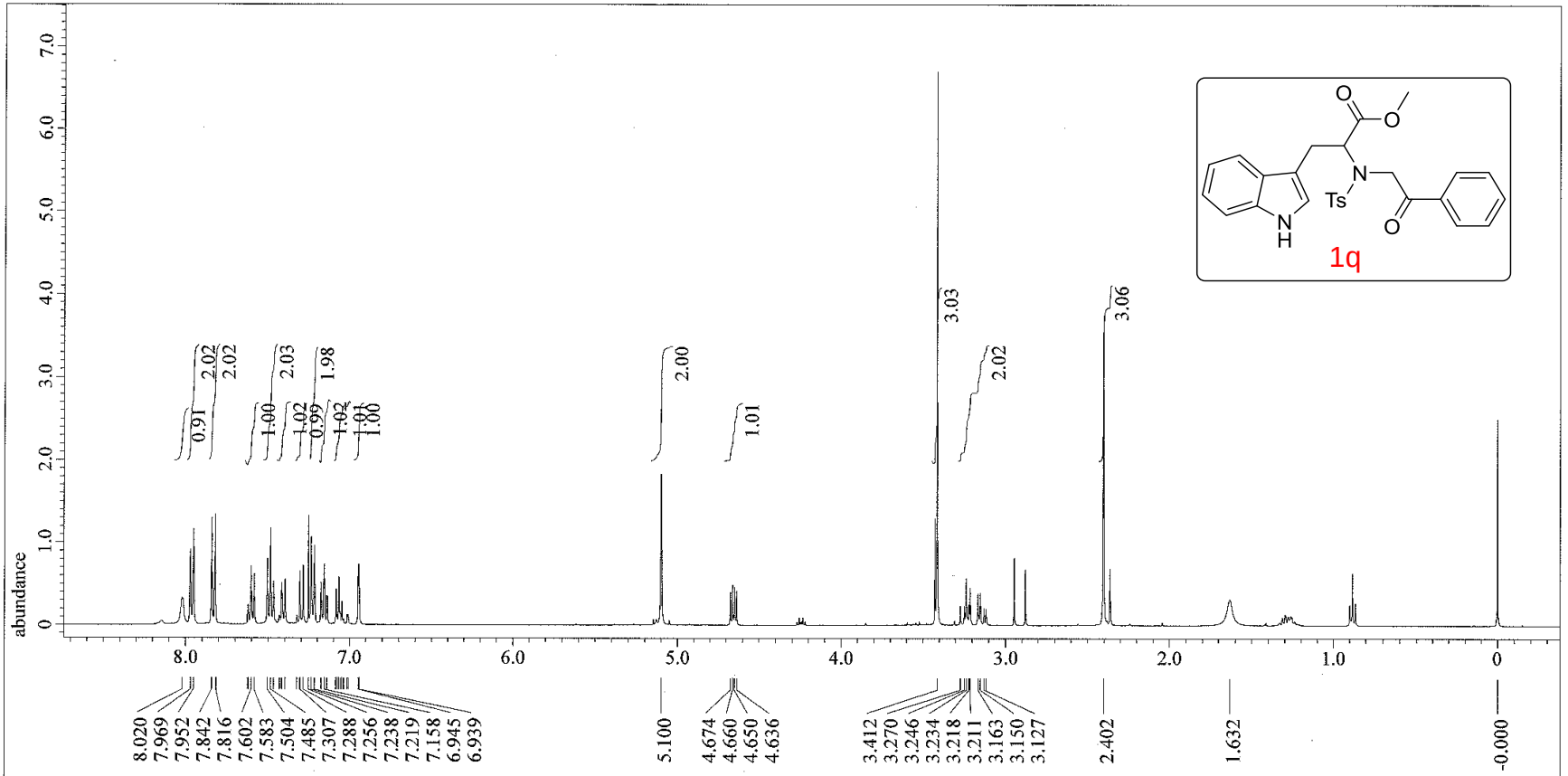
MR-081

Pulse Sequence: s2pul

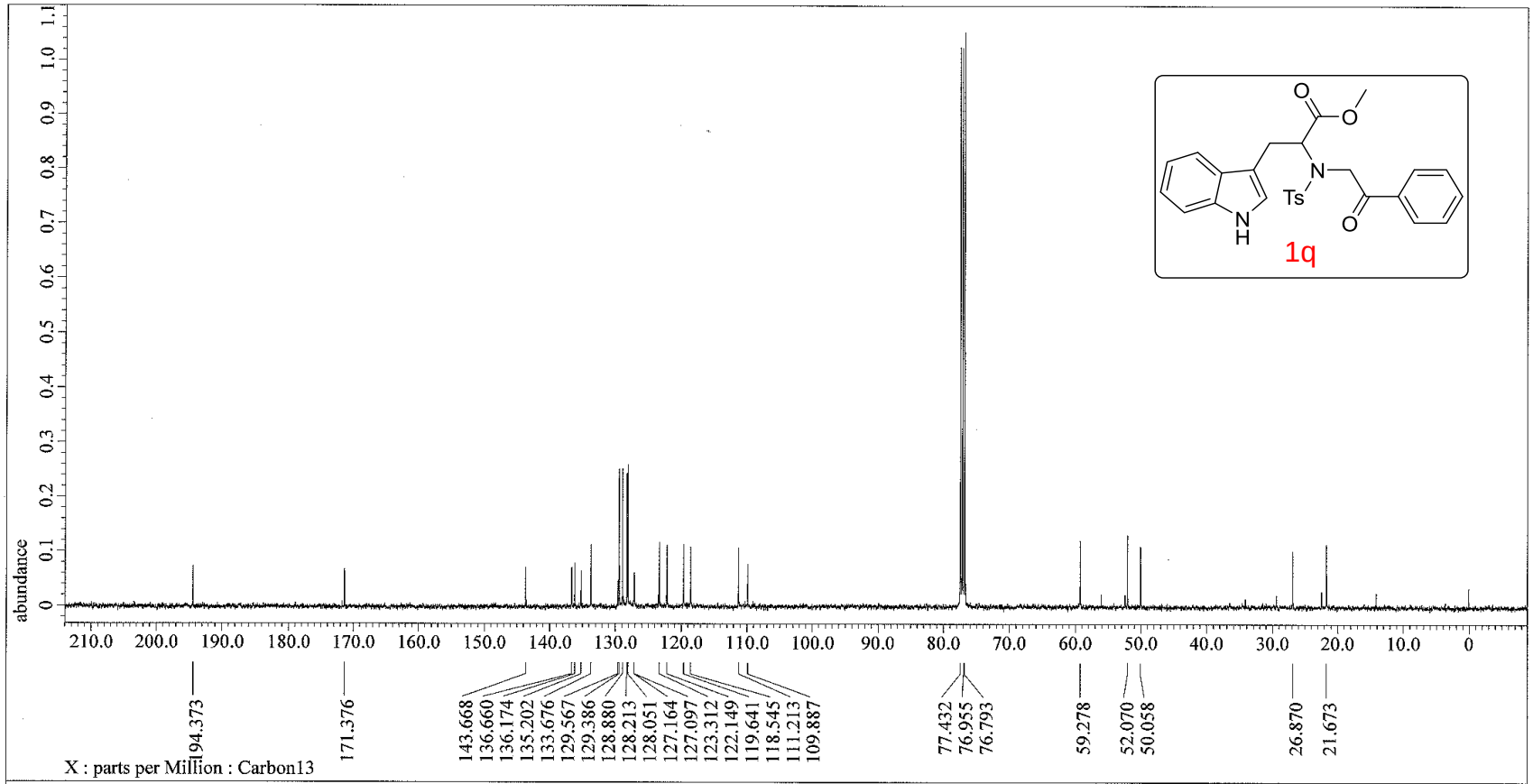
Mercury-400BB "MerPlus400"
Date: Nov 28 2016
Solvent: cdcl3
Ambient temperature
Total 1136 repetitions







Filename	= Hou-03-04_Proton-1-3.jdf	Spectrometer	= DELTA2_NMR	Tri_Freq	= 399.78219838[MHz]
Author	= delta			Tri_Offset	= 5[ppm]
Experiment	= proton.jxp	Field_Strength	= 9.389766[T] (400[MHz])	Clipped	= FALSE
Sample_Id	= Hou-03-04	X_Acq_Duration	= 2.18365952[s]	Scans	= 16
Solvent	= CHLOROFORM-D	X_Domain	= 1H	Total_Scans	= 16
Creation_Time	= 20-JUL-2016 16:13:52	X_Freq	= 399.78219838[MHz]		
Revision_Time	= 20-JUL-2016 16:16:43	X_Offset	= 5[ppm]	Relaxation_Delay	= 5[s]
Current_Time	= 20-JUL-2016 16:17:04	X_Points	= 16384	Recvr_Gain	= 38
		X_Prescans	= 1	Temp_Get	= 25.2[dC]
Comment	= single_pulse	X_Resolution	= 0.45794685[Hz]	X_90_Width	= 12.795[us]
Data_Format	= 1D COMPLEX	X_Sweep	= 7.5030012[kHz]	X_Acq_Time	= 2.18365952[s]
Dim_Size	= 13107	X_Sweep_Clipped	= 6.00240096[kHz]	X_Angle	= 45[deg]
Dim_Title	= Proton	Irr_Domain	= Proton	X_Atn	= 2.4[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	X_Pulse	= 6.3975[us]
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Mode	= Off
Site	= JNM-ECS400	Tri_Domain	= Proton	Tri_Mode	= Off

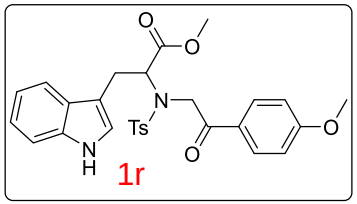


Filename	= Hou-03-04_Carbon-1-3.jdf	Spectrometer	= DELTA2_NMR	Scans	= 1024
Author	= delta			Total_Scans	= 1024
Experiment	= carbon.jxp	Field_Strength	= 9.389766[T] (400[MHz])		
Sample_Id	= Hou-03-04	X_Acq_Duration	= 1.04333312[s]	Relaxation_Delay	= 2[s]
Solvent	= CHLOROFORM-D	X_Domain	= 13C	Recvr_Gain	= 60
Creation_Time	= 20-JUL-2016 16:19:02	X_Freq	= 100.52530333[MHz]	Temp_Get	= 25.4[dC]
Revision_Time	= 20-JUL-2016 18:58:58	X_Offset	= 100[ppm]	X_90_Width	= 10.33[us]
Current_Time	= 20-JUL-2016 18:59:10	X_Points	= 32768	X_Acq_Time	= 1.04333312[s]
		X_Prescans	= 4	X_Angle	= 30[deg]
Comment	= single pulse decoupled gat	X_Resolution	= 0.95846665[Hz]	X_Atn	= 5.3[dB]
Data_Format	= 1D COMPLEX	X_Sweep	= 31.40703518[kHz]	X_Pulse	= 3.44333333[us]
Dim_Size	= 26214	X_Sweep_Clipped	= 25.12562814[kHz]	Irr_Atn_Dec	= 21.473[dB]
Dim_Title	= Carbon13	Irr_Domain	= Proton	Irr_Atn_Noe	= 21.473[dB]
Dim_Units	= [ppm]	Irr_Freq	= 399.78219838[MHz]	Irr_Noise	= WALTZ
Dimensions	= X	Irr_Offset	= 5[ppm]	Irr_Pwidth	= 0.115[ms]
Site	= JNM-ECS400	Clipped	= FALSE	Decoupling	= TRUE

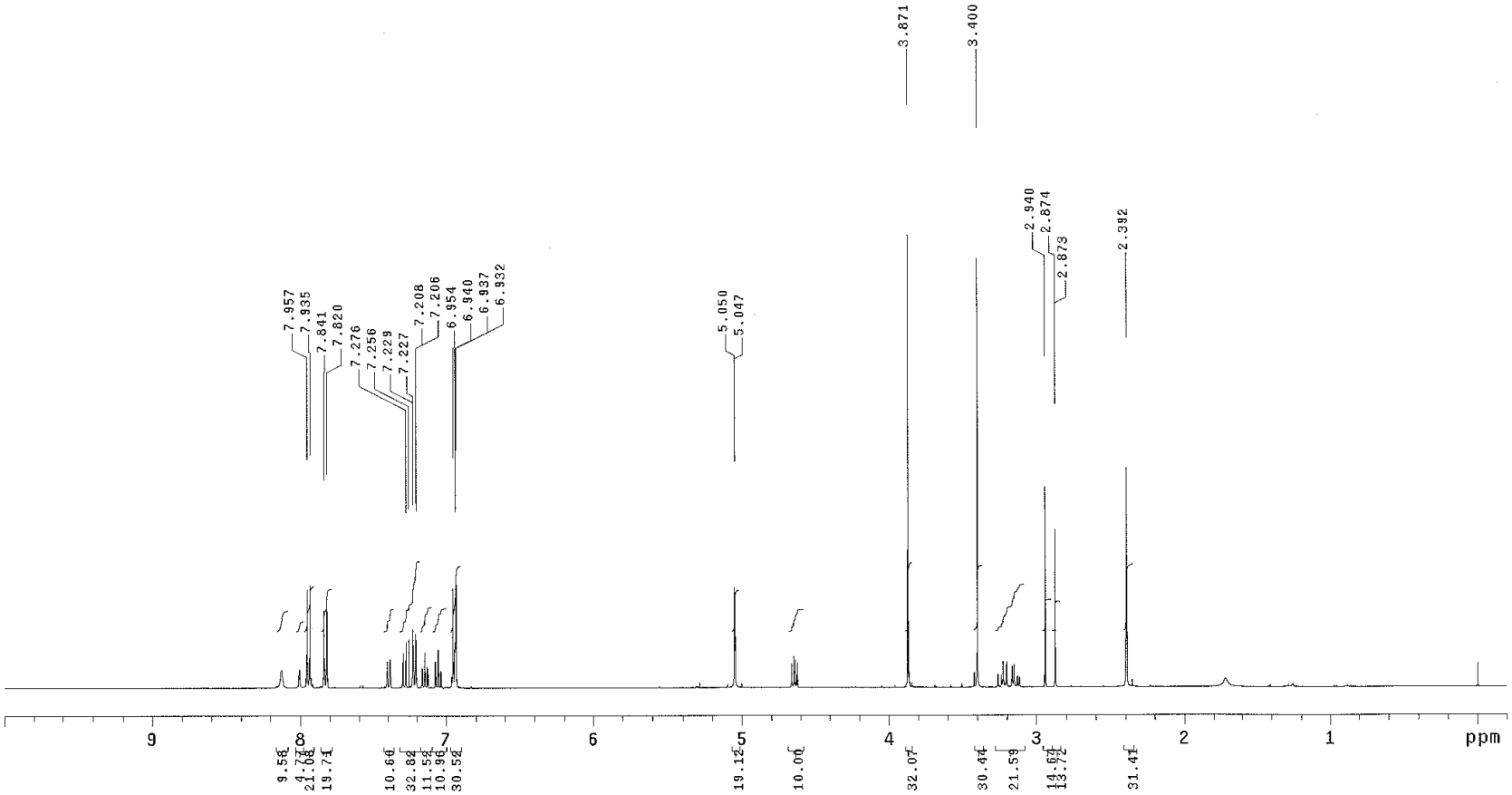
JEOL RESONANCE

Hou-03-17

Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Aug 1 2016
Solvent: cdcl3
Ambient temperature
Total 32 repetitions



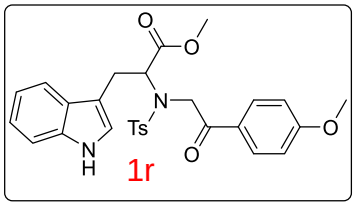
1r

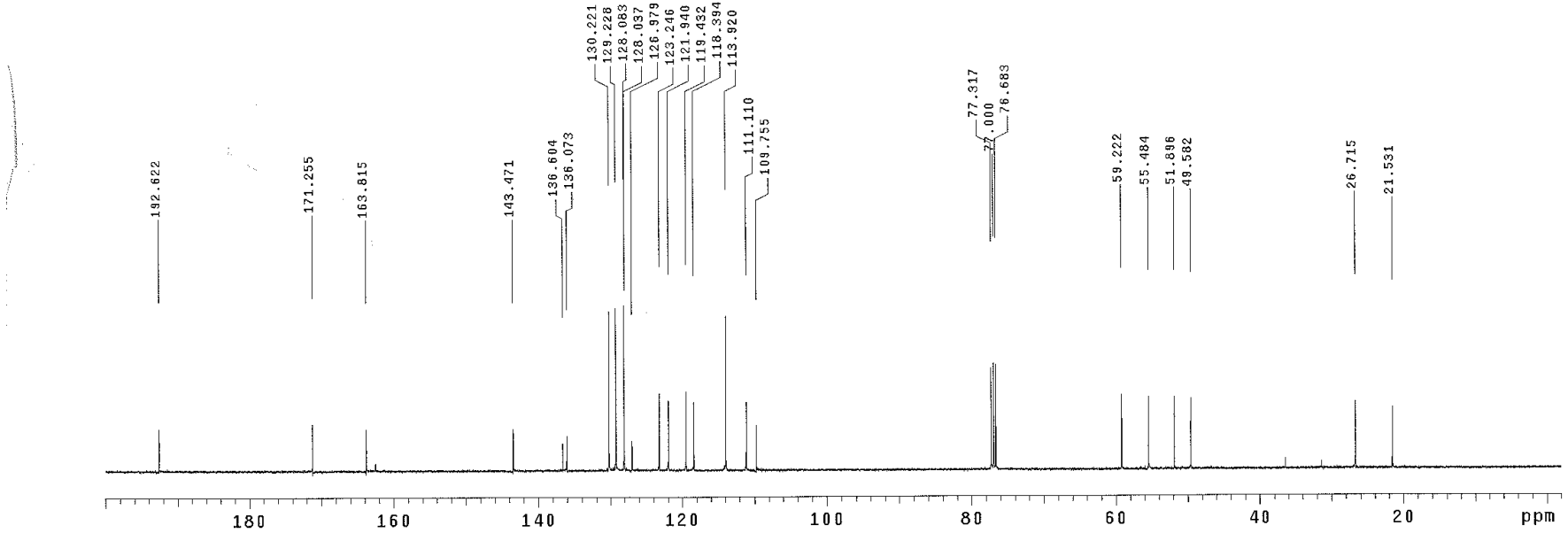


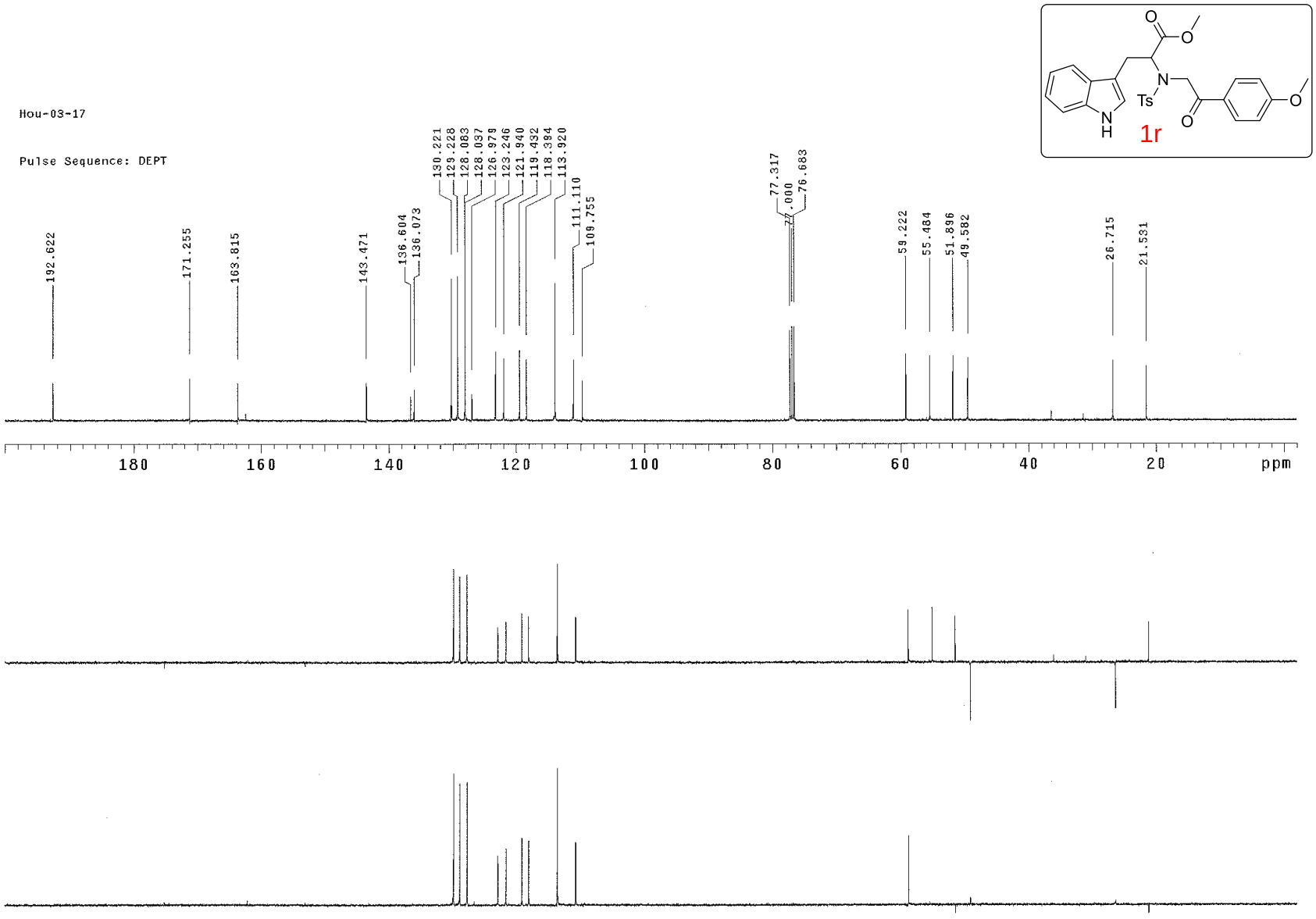
Hou-03-17

Pulse Sequence: s2pul

Mercury-400BB "MerPlus400"
Date: Aug 1 2016
Solvent: cdcl3
Ambient temperature
Total 64000 repetitions



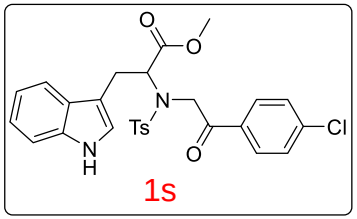



Hou-03-17
Pulse Sequence: DEPT
192.622
171.255
163.815
143.471
136.604
136.073
130.221
129.228
128.083
128.037
126.979
123.246
121.940
119.432
118.384
113.920
111.110
109.755
77.317
77.000
76.683
59.222
55.484
51.896
49.582
26.715
21.531
180
160
140
120
100
80
60
40
20
ppm
Ts
1r

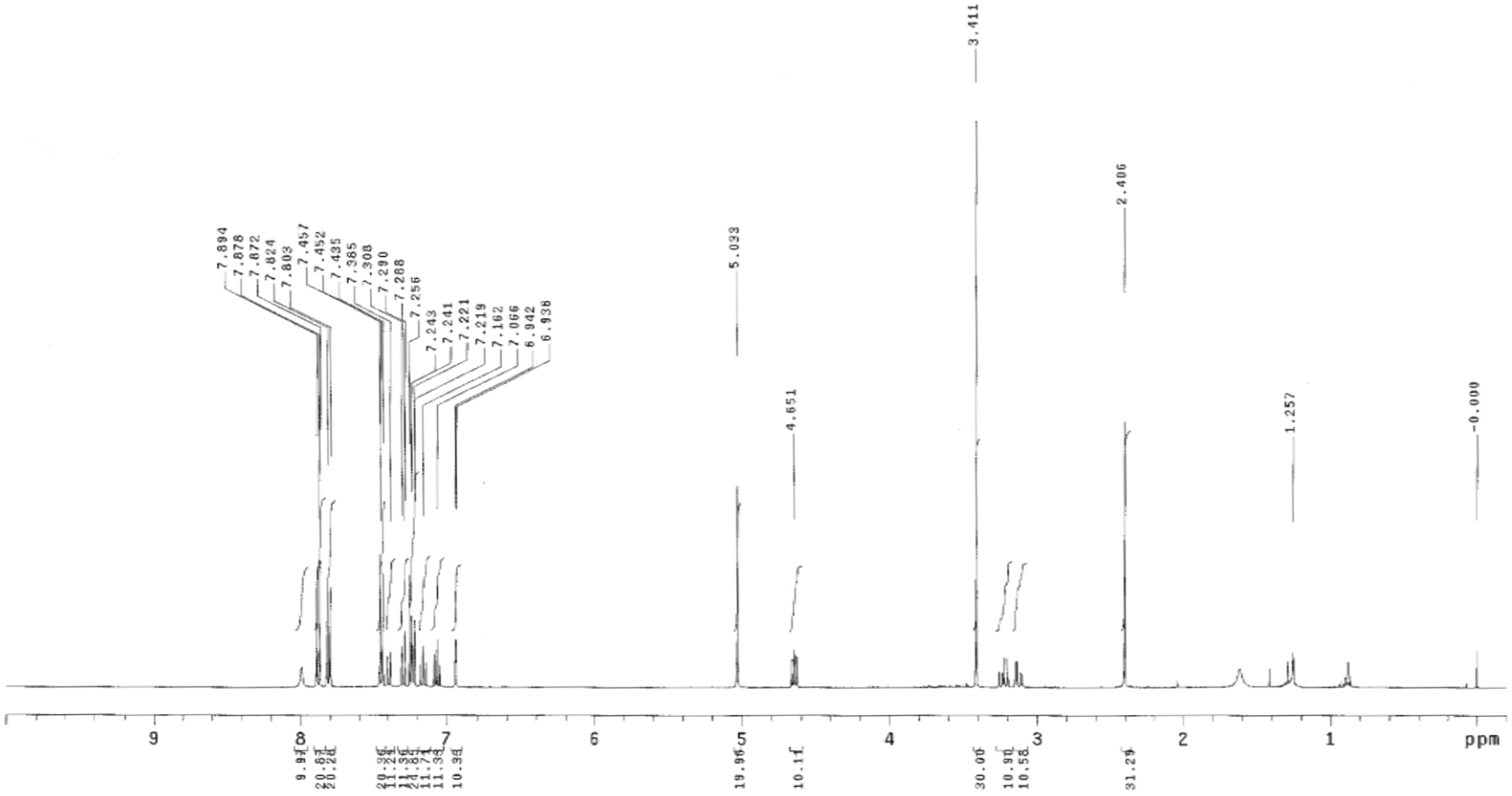
Hou-03-13

Pulse Sequence: s2pul

Mercury-400BB "MerPlus400"
Date: Aug 8 2016
Solvent: CDCl3
Ambient temperature
File: M0173-10
Total 32 repetitions



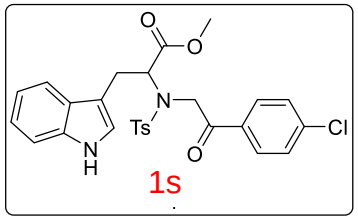
1s



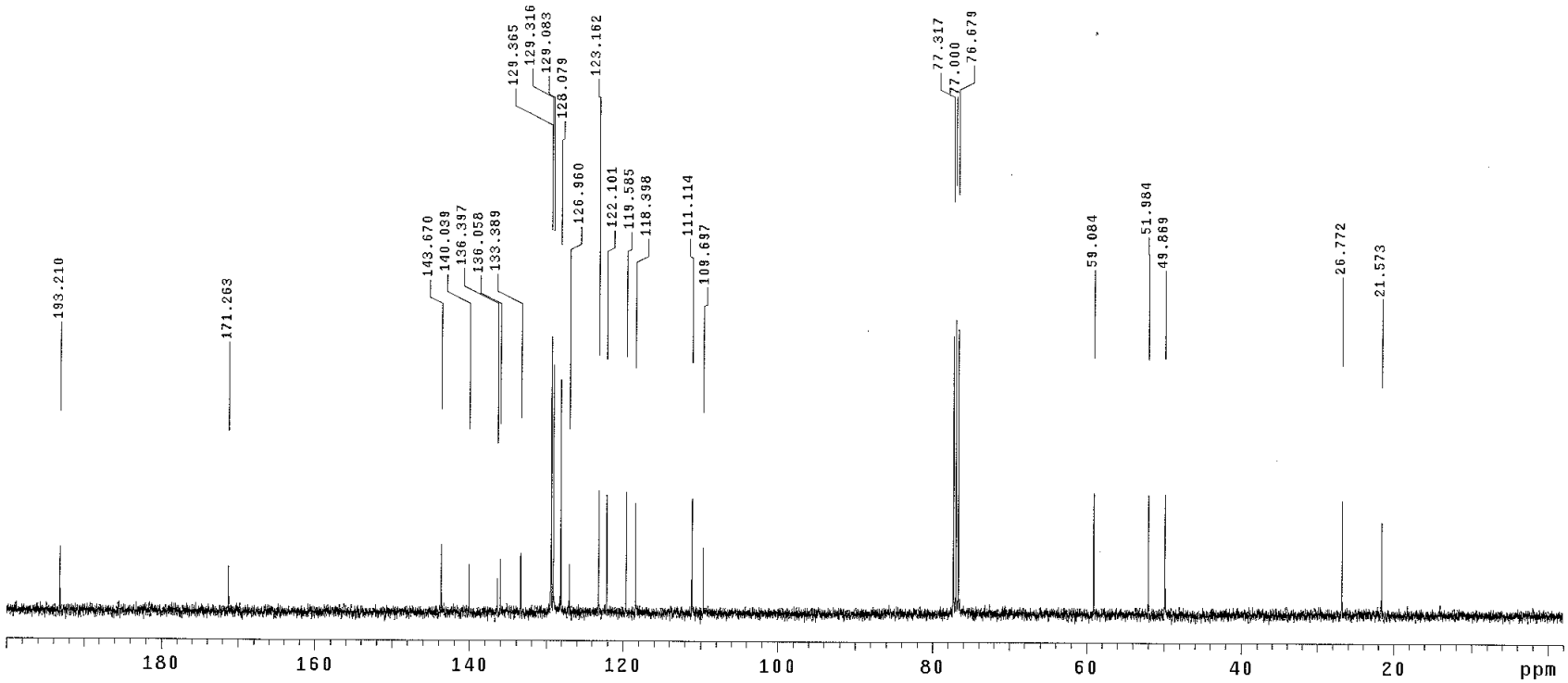
Hou-03-13

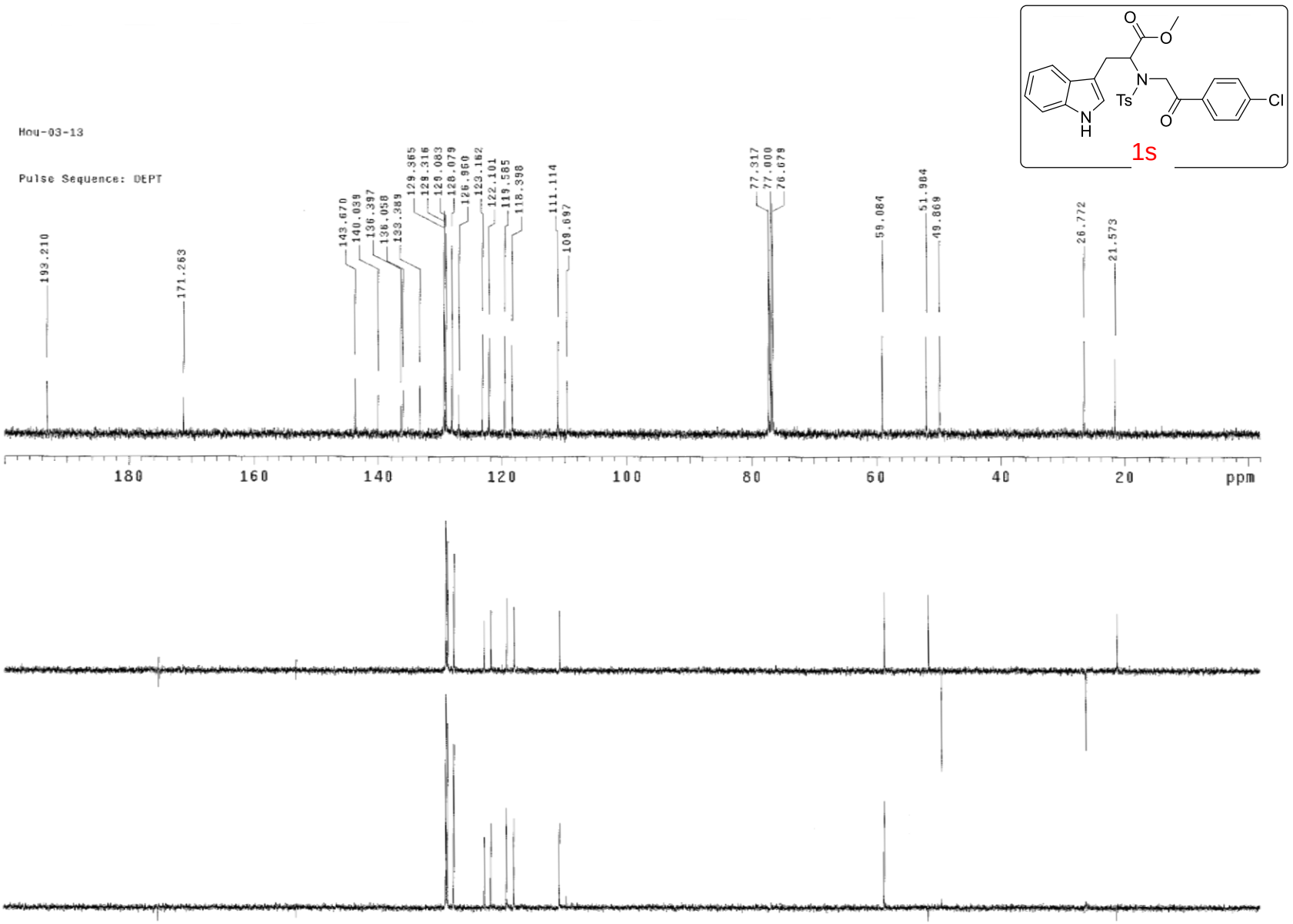
Pulse Sequence: s2pul

Mercury-400BB "MerPlus400"
Date: Aug 8 2016
Solvent: cdcl3
Ambient temperature
Total 768 repetitions



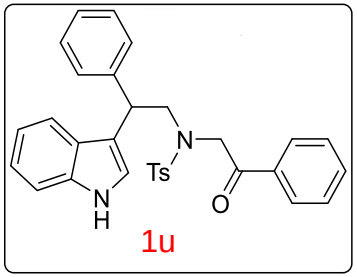
1s



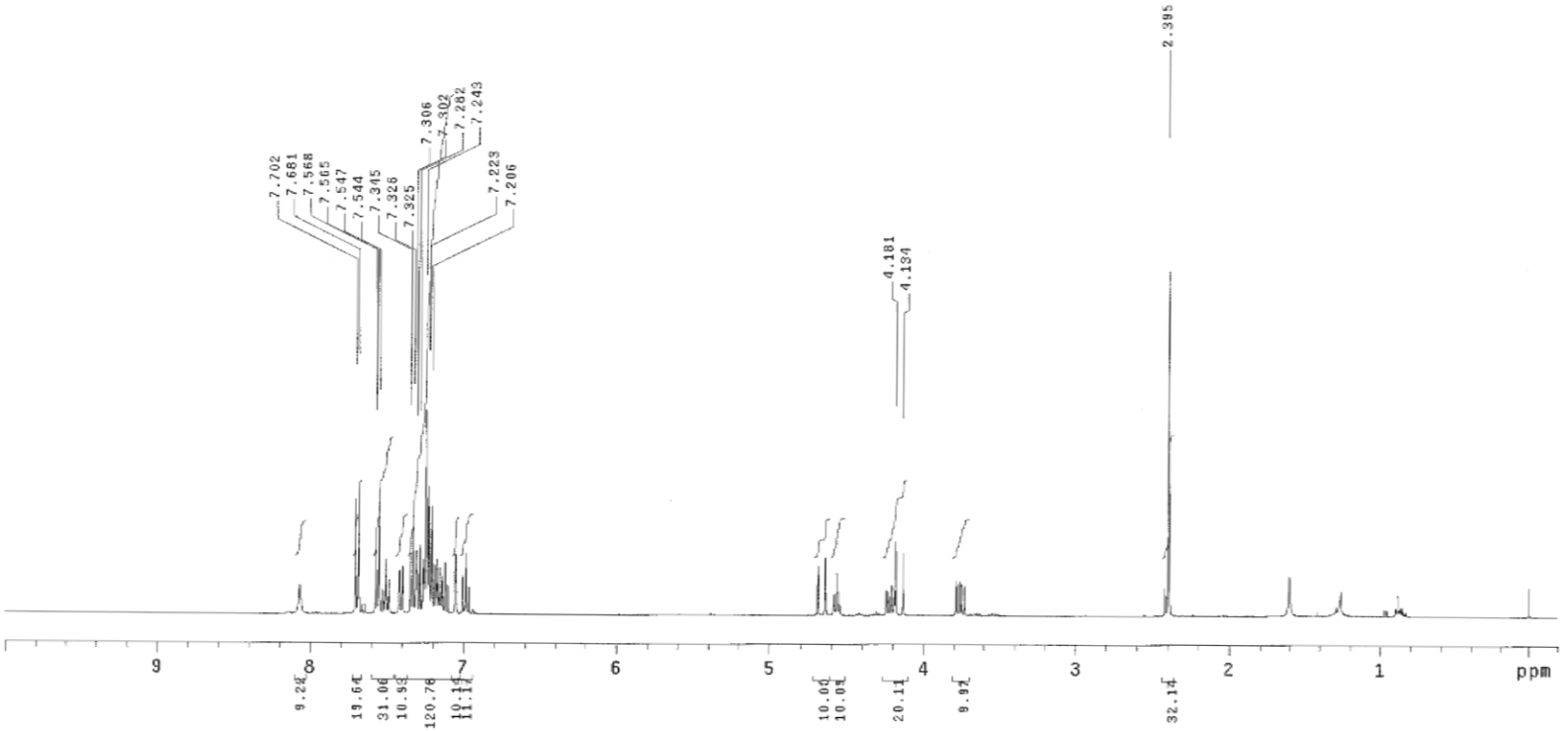

Hou-03-13
Pulse Sequence: DEPT
193.210
171.263
143.670
140.039
136.397
136.058
133.389
129.365
129.316
129.083
128.079
126.960
123.162
122.101
119.585
118.398
111.114
109.697
77.317
77.000
76.679
59.084
51.904
49.869
26.772
21.573
180
160
140
120
100
80
60
40
20
ppm
1s

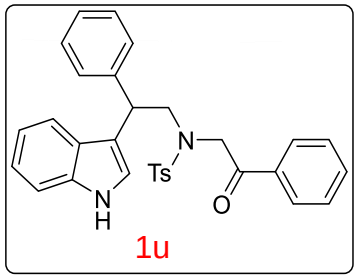
Hou-03-57

Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Aug 1 2016
Solvent: cdcl3
Ambient temperature
Total 32 repetitions



1u

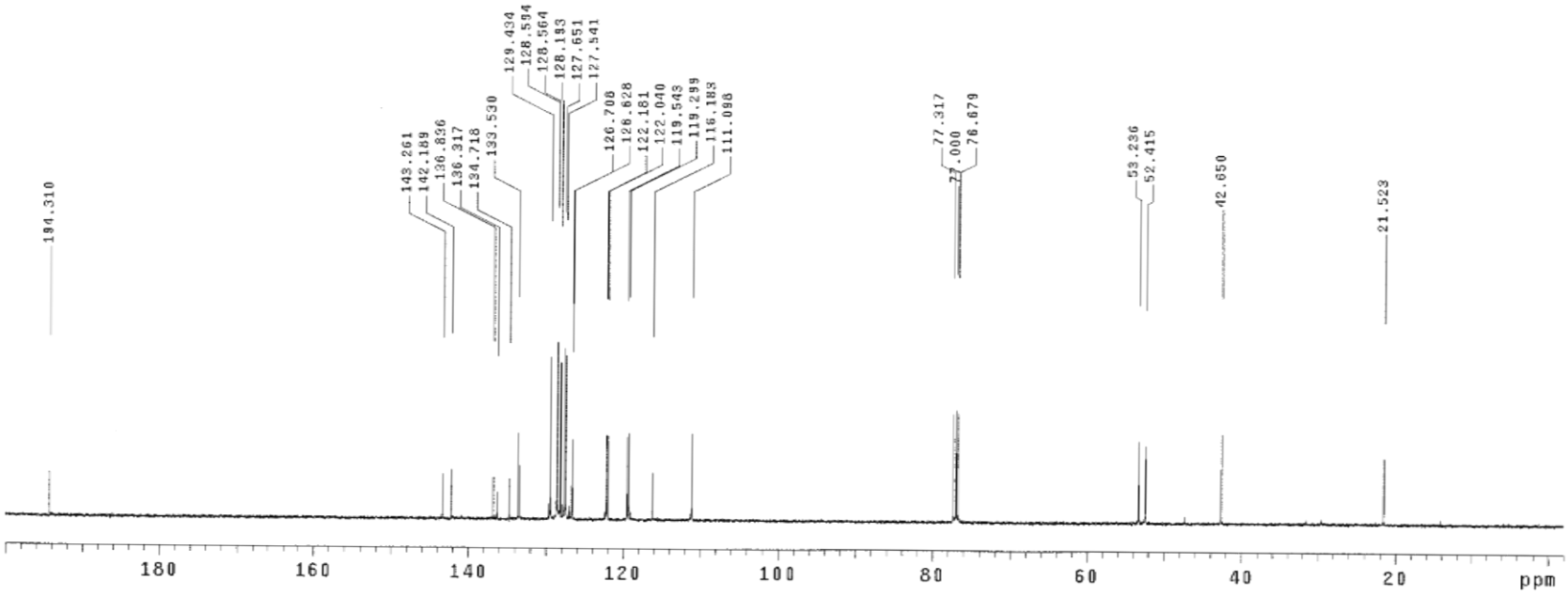


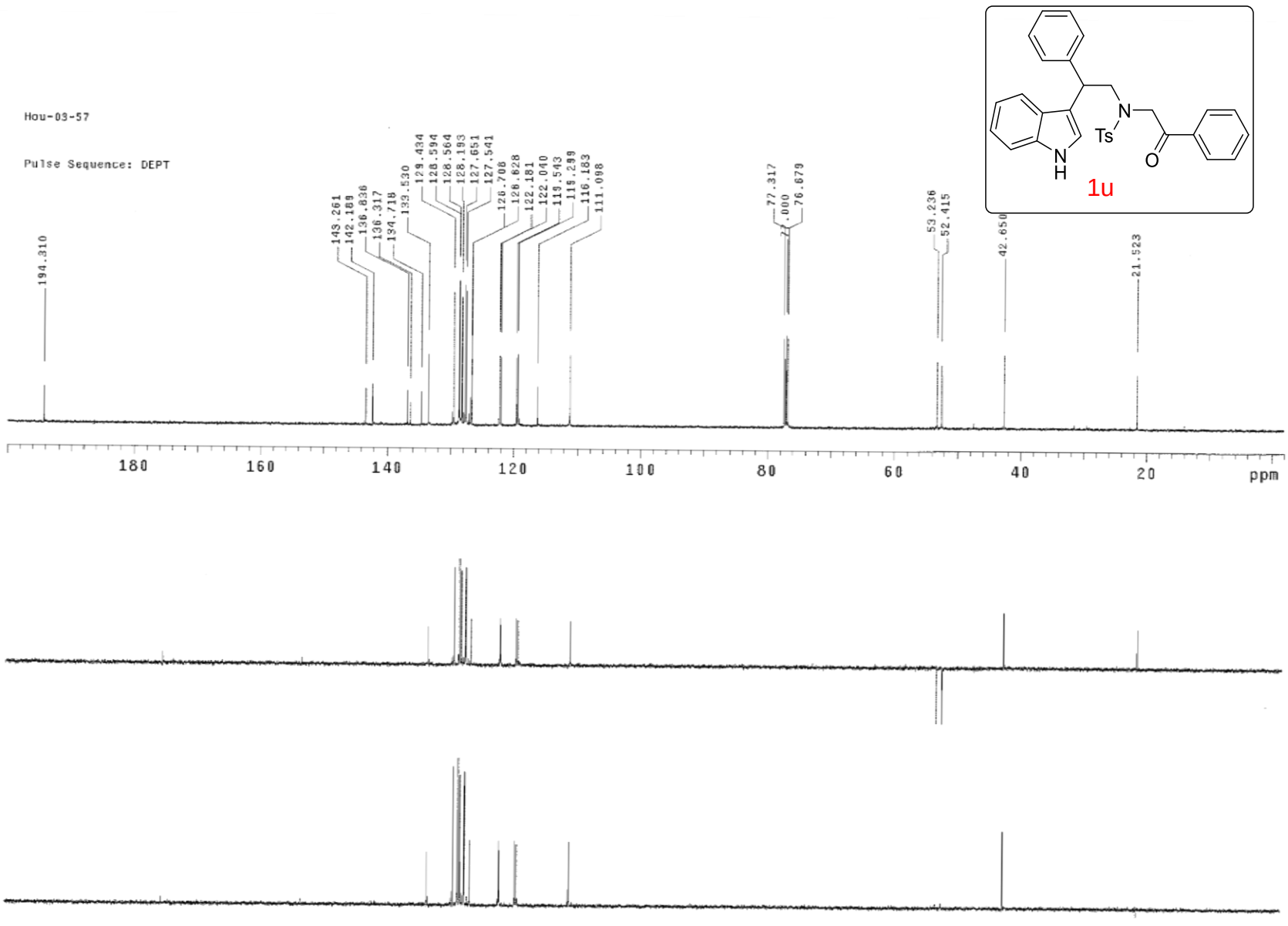


1u

Hou-08-57

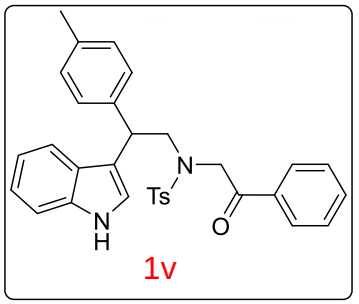
Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Aug 1 2016
Solvent: cdcl3
Ambient temperature
Total 1600 repetitions



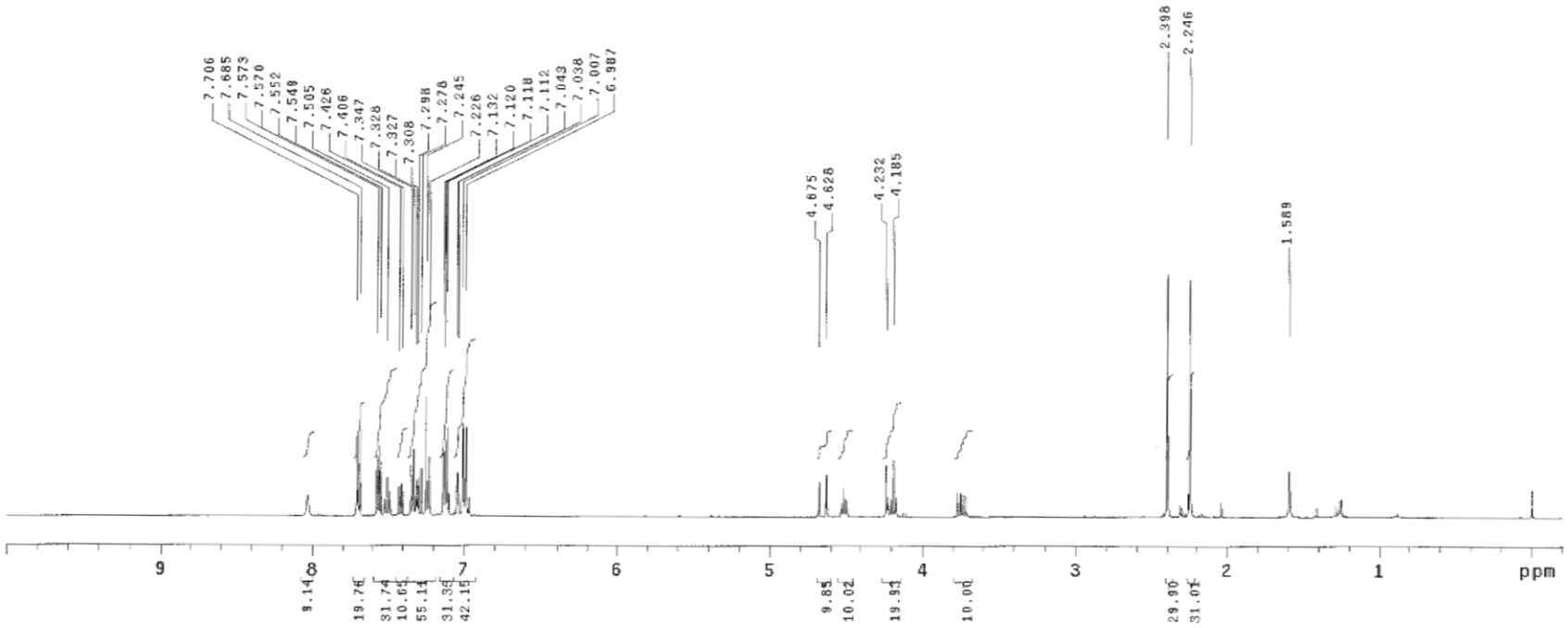

Hou-03-57
Pulse Sequence: DEPT
194.310
143.261
142.189
136.836
136.317
134.718
133.530
129.434
128.594
128.564
128.193
127.651
127.541
126.708
126.628
122.181
122.040
119.543
119.299
116.183
111.098
77.317
77.000
76.679
53.236
52.415
42.650
21.523
180
160
140
120
100
80
60
40
20
ppm
N
Ts
O
N
H
1u

Hou-03-43

Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Aug 1 2016
Solvent: cdcl3
Ambient temperature
Total 32 repetitions



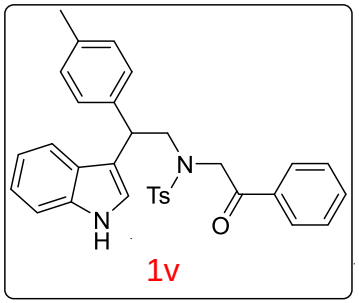
1v



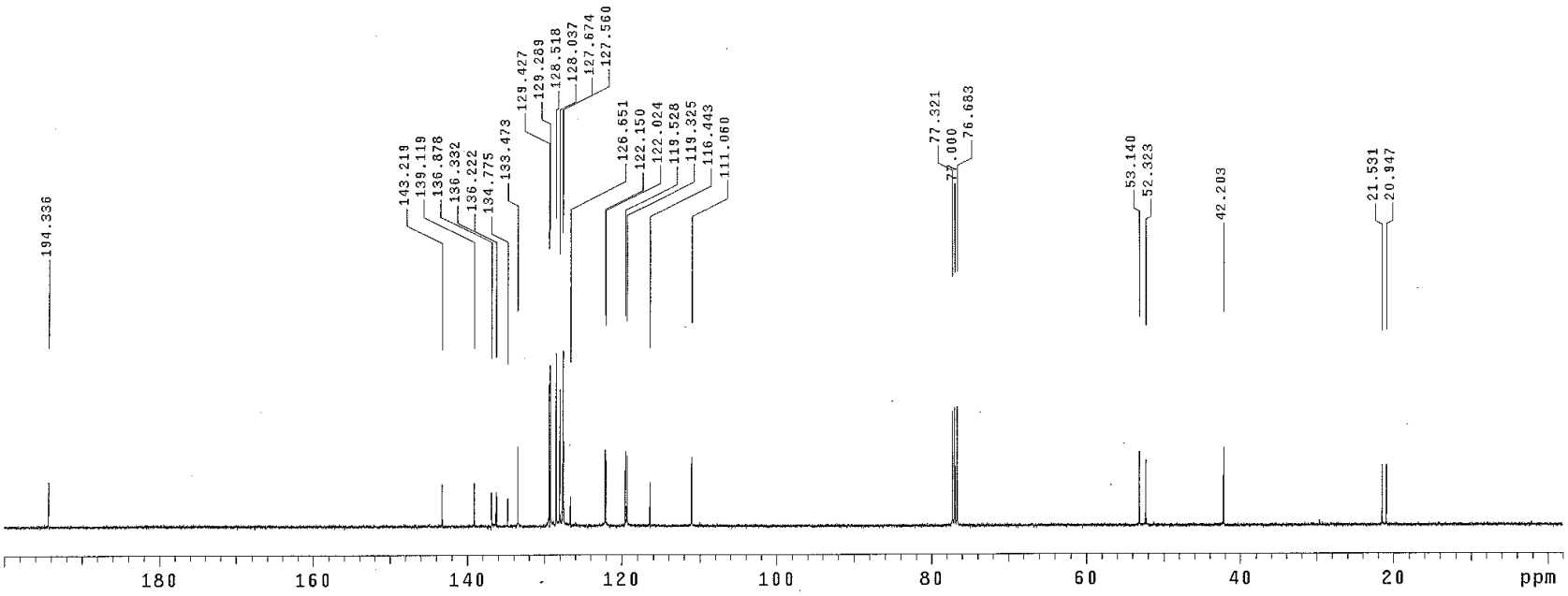
Hou-03-43

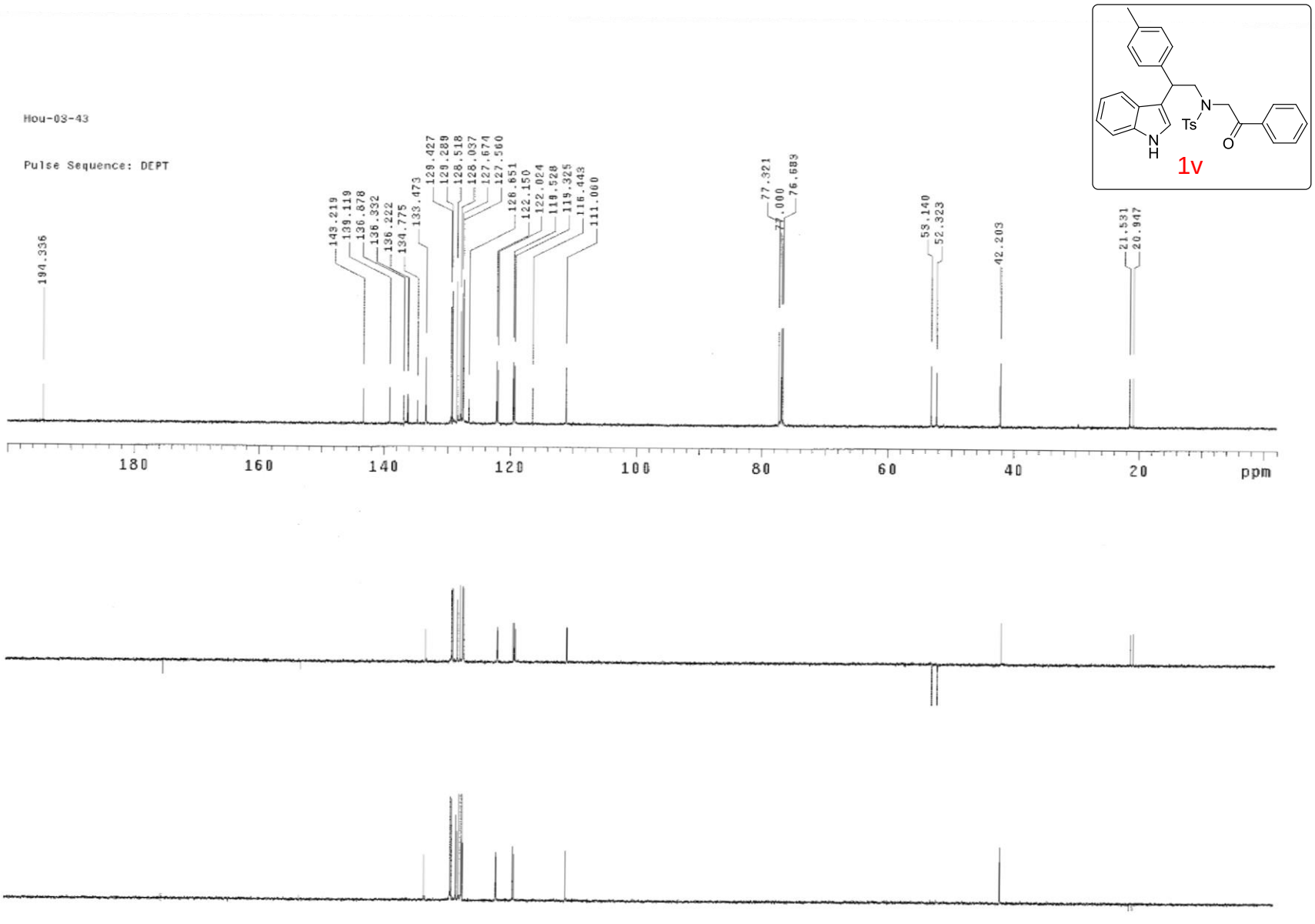
Pulse Sequence: s2pul

Mercury-400BB "MerPlus400"
Date: Aug 1 2016
Solvent: cdcl3
Ambient temperature
Total 2016 repetitions



1v




Hou-03-43
Pulse Sequence: DEPT
194.336
143.219
139.119
136.878
136.332
136.222
134.775
133.473
129.427
129.289
128.518
128.037
127.674
127.560
126.851
122.150
122.024
119.528
119.325
116.443
111.000
77.321
77.000
76.683
53.140
52.323
42.203
21.531
20.947
180
160
140
120
100
80
60
40
20
ppm
Ts
N
H
O
1v

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) nb7154

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: nb7154

```
Bond precision:  C-C = 0.0022 A          Wavelength=0.71073

Cell:          a=8.8691(4)       b=9.8046(5)       c=12.4019(6)
               alpha=99.836(2)   beta=102.889(2)   gamma=94.239(2)
Temperature:   150 K
```

	Calculated	Reported
Volume	1028.75(9)	1028.75(9)
Space group	P -1	P -1
Hall group	-P 1	?
Moiety formula	C25 H22 N2 O2 S	C25 H22 N2 O2 S
Sum formula	C25 H22 N2 O2 S	C25 H22 N2 O2 S
Mr	414.51	414.51
Dx,g cm-3	1.338	1.338
Z	2	2
Mu (mm-1)	0.182	0.182
F000	436.0	436.0
F000’	436.41	
h,k,lmax	11,12,15	11,12,15
Nref	4222	4204
Tmin,Tmax	0.920,0.937	0.921,0.937
Tmin’	0.920	

```
Correction method= # Reported T Limits: Tmin=0.921 Tmax=0.937
AbsCorr = MULTI-SCAN

Data completeness= 0.996          Theta(max)= 26.420

R(reflections)= 0.0380( 3632)     wR2(reflections)= 0.1117( 4204)

S = 1.000                 Npar= 275
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level A

```
DENSX01_ALERT_1_A  The ratio of the calculated to measured crystal density
            lies outside the range 0.80 <> 1.20
            Calculated density =      1.338
            Measured density   =      0.000
PLAT902_ALERT_1_A No (Interpretable) Reflections found in FCF ....     Please Check
```

Alert level G

```
PLAT005_ALERT_5_G No Embedded Refinement Details found  in the CIF     Please Do !
PLAT066_ALERT_1_G Predicted and Reported Tmin&Tmax Range Identical          ? Check
PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note)      0.002 Degree
PLAT380_ALERT_4_G Incorrectly? Oriented X(sp2)-Methyl Moiety .....        C7 Check
PLAT899_ALERT_4_G SHELXL97   is Deprecated and Succeeded by SHELXL       2014 Note
```

```
   2 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
   0 ALERT level C = Check. Ensure it is not caused by an omission or oversight
   5 ALERT level G = General information/check it is not something unexpected

   4 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   0 ALERT type 2 Indicator that the structure model may be wrong or deficient
   0 ALERT type 3 Indicator that the structure quality may be low
   2 ALERT type 4 Improvement, methodology, query or suggestion
   1 ALERT type 5 Informative message, check
```

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 11/08/2016; check.def file version of 04/08/2016

Datablock nb7154 - ellipsoid plot