

Novel one-pot method for the stereoselective synthesis of tetrahydropyrimidinones in low melting mixture

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Supporting Information

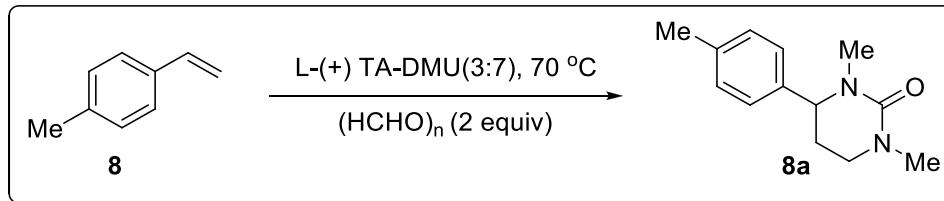
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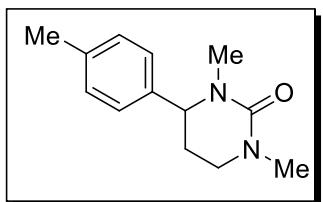
General Considerations: All the solvents were distilled prior to use. Dry solvents were prepared according to the standard procedures. All other reagents were used as received from either Aldrich or Lancaster chemical companies. Reactions requiring inert atmosphere were carried out under argon atmosphere. Infrared (IR) spectra were recorded on a JASCO 4100 FT-IR spectrometer. ¹H NMR spectra were measured on Bruker AVANCE 400 MHz and 500 MHz spectrometers. Chemical shifts were reported in ppm relative to solvent signals. ¹³C NMR spectra were recorded on Bruker 100 MHz and 125 MHz spectrometers with complete proton decoupling. Chemical shifts were reported in ppm from the residual solvent as an internal standard. The high-resolution mass spectra (HRMS) were performed on Micromass QTOF micro mass spectrometer equipped with a Harvard apparatus syringe pump. The structures were solved by direct methods (SHELXS-97) and refined by full-matrix least squares techniques against *F*2 (SHELXL-97). Hydrogen atoms were inserted from geometry consideration using the HFIX option of the program. For thin layer chromatography (TLC) analysis throughout this work, E-Merck precoated TLC plates (silica gel 60 F254 grade, 0.25 mm) were used. Acme (India) silica gel (100-200 mesh) was used for column chromatography.

Synthesis of tetrahydropyrimidinones (THPMs):

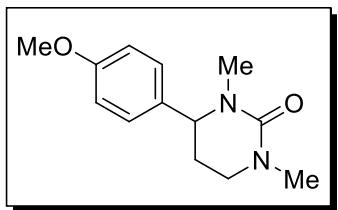
Vinyl arenes were prepared from carbonyl compounds by known procedure.¹



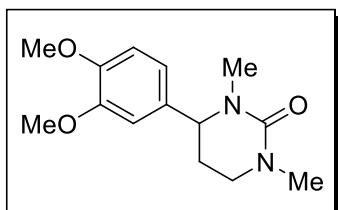
General experimental procedure A: 1.5 g of L - (+)-tartaric acid-DMU (30:70) was heated to 70 °C to obtain a clear melt. To this melt, 1 mmol of vinyl arene and 2 mmol of paraformaldehyde were added at 70 °C. The reaction was monitored by thin layer chromatography. After completion of reaction, the reaction mixture was quenched by adding water while still hot. The reaction mixture was cooled to room temperature and aqueous layer was extracted with EtOAc (3 x 5 mL) and washed with water (2 x 5 mL). The organic layer was dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The crude compound was purified by column chromatography over silica gel using 30% EtOAc/hexane to afford pure THPMs.



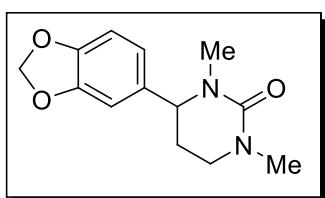
1,3-dimethyl-4-(p-tolyl)tetrahydropyrimidin-2(1H)-one 8a: Following the procedure A, the reaction of 4-methyl styrene (8) (118 mg, 1 mmol) and formaldehyde (60 mg, 2 mmol) at 70 °C for 22h afforded compound 8a in 53% yield (116 mg) as a colourless liquid; **IR** (neat): 2935, 1614, 1515, 1459, 1245, 1032 cm⁻¹; **¹H NMR** (400 MHz, CDCl₃): δ 7.16 (d, J = 8.0 Hz, 2H), 7.04 (d, J = 8.0 Hz, 2H), 4.42 (t, J = 4.4 Hz, 1H), 3.14 (dt, J = 3.6, 11.6 Hz, 1H), 3.00-3.06 (m, 1H), 2.97 (s, 3H), 2.86 (s, 3H), 2.29-2.38 (m, 4H), 1.86 (ddd, J = 4.0, 8.0, 13.2 Hz, 1H) ppm; **¹³C NMR** (100 MHz, CDCl₃): δ 157.0, 138.6, 137.2, 129.5, 126.2, 61.2, 44.1, 36.0, 34.9, 30.0, 21.1 ppm; **HRMS** (ESI) calculated for C₁₃H₁₈N₂O [M+Na]⁺ 241.1317, found 241.1328.



Synthesis of 4-(4-methoxyphenyl)-1,3-dimethyltetrahydropyrimidin-2(1H)-one 9a: Following the procedure A, the reaction of 4-methoxy styrene (**9**) (134 mg, 1 mmol) and formaldehyde (60 mg, 2 mmol) at 70 °C for 22h afforded compound **9a** in 59% yield (138 mg) as a colorless liquid; **IR** (neat): 2935, 1614, 1515, 1459, 1245, 1032 cm⁻¹; **¹H NMR** (400 MHz, CDCl₃): δ 7.06 (dd, J = 2.0, 6.8 Hz, 2H), 6.86 (dd, J = 2.0, 6.8 Hz, 2H), 4.39 (t, J = 4.4 Hz, 1H), 3.78 (s, 3H), 3.13 (dt, J = 4.0, 12.0 Hz, 1H), 3.00-3.06 (m, 1H), 2.96 (s, 3H), 2.84 (s, 3H), 2.26-2.35 (m, 1H), 1.83 (ddd, J = 4.0, 8.0, 13.2 Hz, 1H) ppm; **¹³C NMR** (100 MHz, CDCl₃): δ 159.0, 157.0, 133.5, 127.3, 114.1, 60.8, 55.3, 44.0, 35.9, 34.8, 30.0 ppm; **HRMS** (ESI) calculated for C₁₃H₁₈N₂O₂ [M + H]⁺ 235.1441, found 235.1448.



4-(3,4-dimethoxyphenyl)-1,3-dimethyltetrahydropyrimidin-2(1H)-one 10a: Following the procedure A, the reaction of vinyl arene (**10**) (150 mg, 0.91 mmol) and formaldehyde (55 mg, 1.8 mmol) at 70 °C for 14h afforded compound **10a** in 76% yield (184 mg) as a colorless liquid; **IR** (neat): 2925, 1611, 1517, 1459, 1306, 1240 cm⁻¹; **¹H NMR** (400 MHz, CDCl₃): δ 6.83 (d, J = 8.0 Hz, 1H), 6.70 (dd, J = 2.0, 8.0 Hz, 1H), 6.65 (d, J = 2.0Hz, 1H) 4.39 (t, J = 4.8 Hz, 1H), 3.86 (s, 3H), 3.86 (s, 3H), 3.15 (dt, J = 3.6, 10.4 Hz, 1H), 3.04-3.10 (m, 1H), 2.98 (s, 3H), 2.86 (s, 3H), 2.27-2.35(m, 1H), 1.88 (ddd, J = 4.0, 8.0, 12.4 Hz, 1H) ppm; **¹³C NMR** (100 MHz, CDCl₃): δ 157.1, 149.4, 148.5, 134.2, 118.5, 111.3, 109.3, 61.2, 56.1, 56.0, 44.3, 36.0, 34.9, 30.2 ppm; **HRMS** (ESI) calculated for C₁₄H₂₀N₂O₃ [M+H]⁺ 265.1547, found 265.1543.



4-(benzo[d][1,3]dioxol-5-yl)-1,3-dimethyltetrahydropyrimidin-2(1H)-one 11a: Following the procedure A, the reaction of vinyl arene (**11**) (148 mg, 1 mmol) and formaldehyde (60 mg, 2 mmol) at 70 °C for 17h afforded compound **11a** in 68% yield (168 mg) as a colorless liquid; **IR** (neat): 3447, 3037, 2923, 1632, 1302 cm⁻¹; **¹H NMR** (400 MHz, CDCl₃): δ 6.75 (d, J = 8.0 Hz, 1H), 6.62 (d, J = 1.6Hz, 1H), 6.59 (dd, J = 1.6, 7.6 Hz, 1H), 5.93 (s, 2H), 4.35 (t, J = 4.4Hz, 1H), 3.14 (ddd, J = 4.0, 11.2, 23.2 Hz, 1H), 3.04 (ddd, J = 4.0, 8.8, 11.2 Hz, 1H), 2.95 (s, 3H), 2.84 (s, 3H), 2.29 (dddd, J = 5.2, 10.8, 13.2, 14.8 Hz, 1H), 1.82 (ddd, J = 4.0, 8.0,

12.0 Hz, 1H) ppm; **¹³C NMR** (100 MHz, CDCl₃): δ 156.8, 148.2, 147.0, 135.6, 119.4, 108.4, 106.6, 101.2, 61.1, 44.0, 35.9, 34.8, 30.1 ppm; **HRMS** (ESI) calculated for C₁₃H₁₆N₂O₃ [M+H]⁺ 249.1234, found 249.1226.

1,3,4-trimethyl-4-phenyltetrahydropyrimidin-2(1H)-one 12a: Following the procedure A, the reaction of vinyl arene (**12**) (80 mg, 0.677 mmol) and formaldehyde (41 mg, 1.35 mmol) at 70 °C for 21h afforded compound **12a** in 69% yield (102 mg) as a colorless liquid; **IR** (neat): 2923, 1617, 1511, 1349, 1261, 1032 cm⁻¹; **¹H NMR** (400 MHz, CDCl₃): δ 7.33-7.37 (m, 2H), 7.21-7.28 (m, 3H), 3.03 (td, J = 4.8, 12.0Hz, 1H), 2.96 (s, 3H), 2.88-2.94 (m, 1H), 2.83 (s, 3H), 2.00-2.14 (m, 2H), 1.69 (s, 3H) ppm; **¹³C NMR** (100 MHz, CDCl₃): δ 157.3, 145.1, 128.7, 127.1, 125.8, 61.0, 43.8, 38.4, 36.2, 30.5, 26.8 ppm; **HRMS** (ESI) calculated for C₁₃H₁₈N₂O [M+Na]⁺ 241.1311, found 241.1325.

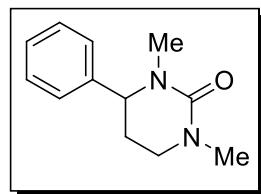
4-(4-methoxyphenyl)-1,3,4-trimethyltetrahydropyrimidin-2(1H)-one 13a: Following the procedure A, the reaction of vinyl arene (**13**) (100 mg, 0.67 mmol) and formaldehyde (40 mg, 1.3 mmol) at 70 °C for 23h afforded compound **13a** in 76% yield (127 mg) as a colorless liquid; **IR** (neat): 3454, 2932, 1621, 1511, 1455, 1353, 1257 cm⁻¹; **¹H NMR** (400 MHz, CDCl₃): δ 7.09 (d, J = 8.0 Hz, 2H), 6.84 (d, J = 8.0 Hz, 2H), 3.76 (s, 3H), 2.98-3.03 (m, 1H), 2.92 (s, 3H), 2.86-2.90 (m, 1H), 2.77 (s, 3H), 1.93-2.08 (m, 2H), 1.62 (s, 3H); **¹³C NMR** (100 MHz, CDCl₃): δ 158.5, 157.2, 137.0, 126.8, 113.9, 60.4, 55.2, 43.8, 38.4, 36.1, 30.2, 26.9 ppm; **HRMS** (ESI) calculated for C₁₄H₂₀N₂O₂ [M+H]⁺ 249.1598, found 249.1606.

4-(4-(allyloxy)phenyl)-1,3-dimethyltetrahydropyrimidin-2(1H)-one 14a: Following the procedure A, the reaction of styrene (**14**) (80 mg, 0.5 mmol) with formaldehyde (30 mg, 1 mmol) at 70 °C for 22h afforded compound **14a** in 73% yield (95 mg) as a colorless liquid; **IR** (neat): 3441, 2927, 1617, 1513, 1411, 1249 cm⁻¹; **¹H NMR** (400 MHz, CDCl₃): δ 7.03 (d, J = 8.4 Hz, 2H), 6.86 (d, J = 8.4 Hz, 2H), 5.97-6.06 (m, 1H), 5.38 (dd, J = 1.2, 17.2 Hz, 1H), 5.24 (d, J = 10.4 Hz, 1H), 4.49 (d, J = 5.2Hz, 2H), 4.38 (t, J = 4.4Hz, 1H), 3.10 (dt, J = 3.6, 11.6Hz, 1H), 2.99-3.04 (m, 1H), 2.94 (s, 3H), 2.82 (s, 3H), 2.25-2.33

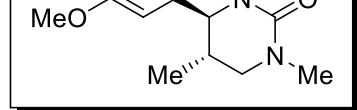
(m, 1H), 1.79-1.85 (m, 1H) ppm; **¹³C NMR** (100 MHz, CDCl₃): δ 157.9, 156.9, 133.6, 133.2, 127.2, 117.7, 114.8, 68.8, 60.7, 43.9, 35.8, 34.7, 29.9 ppm; **HRMS** (ESI) calculated for C₁₅H₂₀N₂O₂ [M+Na]⁺ 283.1417, found 283.1423.

Data for THPM derivative from styrene 1,3-dimethyl-4-phenyltetrahydropyrimidin-2(1H)-one (reference 26): Following the

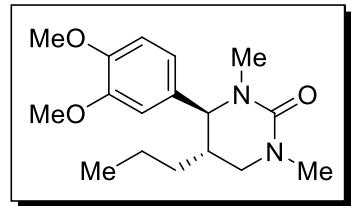
procedure A, the reaction of styrene (104 mg, 1 mmol) and formaldehyde (60 mg, 2 mmol) at 70 °C for 25h afforded compound in 14% yield (29 mg) as a colorless liquid; **IR** (neat): 2927, 2857, 2377, 1617, 1508, 1446, 1402, 1331cm⁻¹; **¹H NMR** (400 MHz, CDCl₃): δ 7.36 (t, J = 6.0 Hz, 2H), 7.26-7.29 (m, 1H), 7.17 (d, J = 6.0 Hz, 2H), 4.47 (t, J = 3.6 Hz, 1H), 3.14 (dt, J = 3.2, 9.2 Hz, 1H), 3.03-3.07 (m, 1H), 2.98 (s, 3H), 2.88 (s, 3H), 2.33-2.40 (m, 1H), 1.89 (ddd, J = 3.2, 6.0, 12.8 Hz, 1H) ppm; **¹³C NMR** (100 MHz, CDCl₃): δ 157.0, 141.7, 128.8, 127.5, 126.3, 61.4, 44.0, 36.0, 34.9, 29.9 ppm; **HRMS** (ESI) calculated for C₁₂H₁₆N₂O [M + H]⁺ 205.1324, found 205.1324



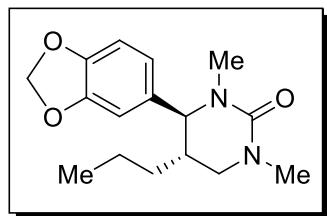
(4S,5S)-4-(3,4-dimethoxyphenyl)-1,3,5-trimethyltetrahydropyrimidin-2(1H)-one 15a: Following the procedure A, the reaction of vinyl arene (**15**) (160 mg, 0.9 mmol) and formaldehyde (56 mg, 1.8 mmol) at 70 °C for 20h afforded compound **15a** in 69% yield (102 mg, dr = 19:1) as a colorless liquid recovered starting material 71mg; **IR** (neat): 2925, 1611, 1517, 1459, 1306, 1277 cm⁻¹; **¹H NMR** (400 MHz, CDCl₃): δ 6.81 (d, J = 8.0 Hz, 1H), 6.69 (dd, J = 2.0, 8.0 Hz, 1H), 6.64 (d, J = 2.0 Hz, 1H), 3.91 (d, J = 6.0Hz, 1H), 3.86 (s, 3H), 3.85 (s, 3H), 3.18 (dd, J = 4.0, 11.6 Hz, 1H), 2.97 (s, 3H), 2.88 (dd, J = 6.4, 12.0 Hz, 1H), 2.77 (s, 3H), 2.07-2.00 (m, 1H), 1.02 (d, J = 6.8 Hz, 3H) ppm; **¹³C NMR** (100 MHz, CDCl₃): δ 157.0, 149.4, 148.5, 134.1, 119.1, 111.1, 109.3, 68.4, 56.0, 56.0, 51.4, 36.1, 34.8, 34.7, 17.1 ppm; **HRMS** (ESI) calculated for C₁₅H₂₂N₂O₃ [M+H]⁺ 279.1700, found 279.1703.



(4S,5S)-4-(3,4-dimethoxyphenyl)-1,3-dimethyl-5-propyltetrahydropyrimidin-2(1H)-one 16a: Following the procedure A, the reaction of vinyl arene (**16**) (150 mg, 0.72 mmol) and formaldehyde (43 mg, 1.44 mmol) at 70 °C for 21h afforded compound **16a** in 73% yield (164 mg, dr = 19:1) as a colorless liquid; **IR** (neat): 2927, 2857, 1617, 1508, 1446, 1402, 1331, 1305 cm⁻¹; **¹H NMR** (400 MHz, CDCl₃): δ



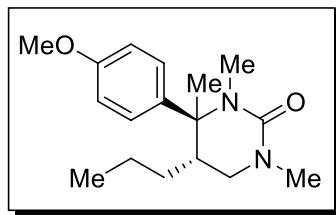
6.83 (d, J = 8.4 Hz, 1H), 6.69 (dd, J = 8.4 Hz, 1H), 6.63 (s, 1H), 4.04 (d, J = 4.0 Hz, 1H), 3.87 (s, 3H), 3.86 (s, 3H), 3.22 (dd, J = 4.0, 12.0 Hz, 1H), 2.97 (s, 3H), 2.83-2.86 (m, 4H), 1.29-1.83 (m, 5H), 0.90 (t, J = 6.8 Hz, 3H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 156.8, 149.4, 148.4, 134.5, 118.8, 111.2, 109.3, 66.5, 56.1, 56.0, 48.6, 39.4, 36.2, 35.1, 33.4, 20.5, 14.1 ppm; HRMS (ESI) calculated for $\text{C}_{17}\text{H}_{26}\text{N}_2\text{O}_3$ $[\text{M}+\text{H}]^+$ 307.2016, found 307.2042.



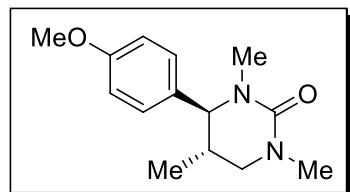
(4S,5S)-4-(benzo[d][1,3]dioxol-5-yl)-1,3-dimethyl-5-propyltetrahydropyrimidin-2(1H)-one 17a:

Following the procedure A, the reaction of vinyl arene (**17**) (100 mg, 0.52 mmol) and formaldehyde (32 mg, 1.05 mmol) at 70 °C for 18h afforded compound **17a** in 68% yield (103 mg, dr = 19:1) as a colorless liquid; IR (neat): 3002, 2355, 1608, 1266, 1041 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 6.77 (d, J = 8.0 Hz, 1H), 6.63 (d, J = 1.6 Hz, 1H), 6.61 (dd, J = 1.6, 8.0 Hz, 1H), 5.96 (s, 2H), 4.02 (d, J = 4.0 Hz, 1H), 3.23 (dd, J = 4.0, 12.0 Hz, 1H), 2.97 (s, 3H), 2.83-2.86 (m, 4H), 1.76-1.82 (m, 1H), 1.29-1.48 (m, 4H), 0.92 (t, J = 7.2 Hz, 3H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ ppm 156.7, 148.2, 147.0, 136.1, 119.7, 108.4, 106.8, 101.3, 66.5, 48.4, 39.4, 36.2, 35.1, 33.4, 20.5, 14.1 ppm; HRMS (ESI) calculated for $\text{C}_{16}\text{H}_{22}\text{N}_2\text{O}_3$ $[\text{M}+\text{H}]^+$ 291.1703, found 291.1704.

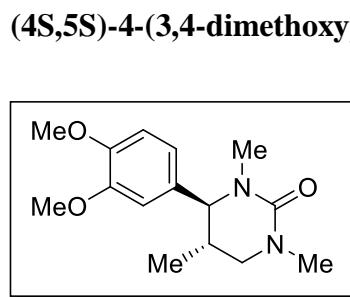
(4S,5S)-4-(4-methoxyphenyl)-1,3,4-trimethyl-5-propyltetrahydropyrimidin-2(1H)-one 18a: Following the procedure A, the



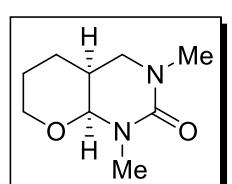
reaction of vinyl styrene (**18**) (80 mg, 0.42 mmol) and formaldehyde (26 mg, 0.84 mmol) at 70 °C for 24h afforded compound **18a** in 63% yield (84 mg, dr = 19:1) as a colorless liquid; IR (neat): 2923, 1617, 1511, 1402, 1345, 1257, 1159 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.18 (d, J = 8.0 Hz, 2H), 6.85 (d, J = 8.0 Hz, 2H), 3.80 (s, 3H), 3.00-3.14 (m, 2H), 2.98 (s, 3H), 2.55 (s, 3H), 2.01-2.07 (m, 1H), 1.43 (s, 3H), 1.24-1.28 (m, 2H), 0.98-1.06 (m, 2H), 0.72 (t, J = 7.2 Hz, 3H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 158.5, 157.4, 136.7, 127.7, 113.7, 63.9, 55.3, 49.1, 44.0, 36.1, 31.2, 30.0, 20.8, 18.2, 14.0 ppm; HRMS (ESI) calculated for $\text{C}_{17}\text{H}_{26}\text{N}_2\text{O}_2$ $[\text{M}+\text{Na}]^+$ 313.1886, found 313.1892.



(4S,5S)-4-(3-methoxyphenyl)-1,3,5-trimethyltetrahydropyrimidin-2(1H)-one 19a: Following the procedure A, the reaction of vinyl arene (**19**) (80 mg, 0.54 mmol) and formaldehyde (32 mg, 1.08 mmol) at 70 °C for 20h afforded compound **19a** in 74% yield (98 mg, dr = 19:1) as a colorless liquid; **IR** (neat): 2925, 1617, 1507, 1454, 1405, 1242, 1174 cm⁻¹; **¹H NMR** (400 MHz, CDCl₃): δ 7.06 (d, J = 4.4 Hz, 2H), 6.85 (d, J = 8.4 Hz, 2H), 3.93 (d, J = 5.2 Hz, 1H), 3.78 (s, 3H), 3.16 (dd, J = 4.0, 11.6 Hz, 1H), 2.96 (s, 3H), 2.84 (dd, J = 6.0, 11.6 Hz, 1H), 2.76 (s, 3H), 1.98-2.03 (m, 1H), 1.03 (d, J = 6.8 Hz, 3H) ppm; **¹³C NMR** (100 MHz, CDCl₃): δ 159.0, 156.9, 133.7, 127.7, 114.1, 67.9, 55.3, 51.1, 36.1, 34.7, 17.1 ppm; **HRMS** (ESI) calculated for C₁₄H₂₀N₂O₂ [M+Na]⁺ 271.1417, found 271.1430.



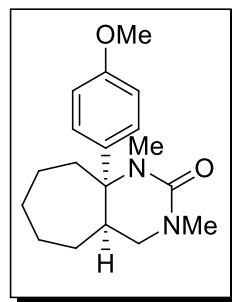
(4S,5S)-4-(3,4-dimethoxyphenyl)-1,3,5-trimethyltetrahydropyrimidin-2(1H)-one 15a from starting material 20: Following the procedure A, the reaction of vinyl arene (**20**) (80 mg, 0.44 mmol) and formaldehyde (27 mg, 0.88 mmol) at 70 °C for 16h afforded compound **15a** in 81% yield (101 mg, dr = 19:1) as a colorless liquid; **IR** (neat): 2925, 1611, 1517, 1459, 1306, 1277 cm⁻¹; **¹H NMR** (400 MHz, CDCl₃): δ 6.81 (d, J = 8.0 Hz, 1H), 6.68 (d, J = 8.0 Hz, 1H), 6.64 (s, 1H), 3.90 (d, J = 5.2 Hz, 1H), 3.85 (s, 3H), 3.84 (s, 3H), 3.17 (dd, J = 4.0, 11.6 Hz, 1H), 2.96 (s, 3H), 2.87 (dd, J = 6.4, 12.0 Hz, 1H), 2.77 (s, 3H), 2.07-2.01 (m, 1H), 1.02 (d, J = 6.8 Hz, 3H) ppm; **¹³C NMR** (100 MHz, CDCl₃): δ 156.9, 149.4, 148.5, 134.1, 119.1, 111.0, 109.2, 68.4, 56.0, 56.0, 51.4, 36.1, 34.7, 17.0 ppm; **HRMS** (ESI) calculated for C₁₅H₂₂N₂O₃ [M+H]⁺ 279.1700, found 279.1703.



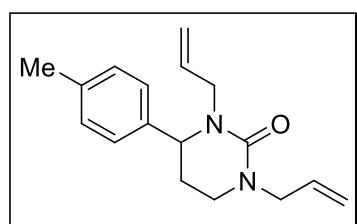
1,3-dimethylhexahydro-1H-pyranopyrimidin-2(3H)-one 21a: Following the procedure A, the reaction of 3, 4-dihydropyran (**21**) (84 mg, 1 mmol) and formaldehyde (60 mg, 2 mmol) at 70 °C for 16h afforded compound **21a** in 65% yield (119 mg) as a colorless liquid; **IR** (neat): 3429, 1637, 1286, 1234, 1103 cm⁻¹; **¹H NMR** (400 MHz, CDCl₃): δ 4.40 (d, J = 1.6 Hz, 1H), 3.91-3.95 (m, 1H), 3.57 (t, J = 11.6 Hz, 1H), 3.47 (dt, J = 2.4, 11.6 Hz, 1H), 2.96 (s, 3H), 2.86-2.91 (m, 4H), 2.17-2.22 (m, 1H), 1.63-1.83 (m, 3H), 1.32-1.38 (m, 1H) ppm; **¹³C NMR** (100

MHz, CDCl₃): δ ppm 155.6, 87.2, 66.3, 47.2, 35.7, 34.1, 31.3, 24.9, 21.3 ppm; **HRMS** (ESI) calculated for C₉H₁₆N₂O₂ [M+H]⁺ 184.1285, found 185.1297.

9a-(4-methoxyphenyl)-1,3-dimethyldecahydro-2H-cyclohepta[d]pyrimidin-2-one 22a: Following the procedure A, the reaction of vinyl arene (**22**) (80 mg, 0.39 mmol) and formaldehyde (23 mg, 0.79 mmol) at 70 °C for 14h afforded compound **22a** in 80% yield (97 mg) as a colorless liquid; **IR** (neat): 2926, 1620, 1509, 1098, 1035 cm⁻¹; **¹H NMR** (400 MHz, CDCl₃): δ 7.07 (d, J = 8.8 Hz, 2H), 6.82 (d, J = 8.4 Hz, 2H), 3.77 (s, 3H), 3.03 (dd, J = 2.8, 12.0 Hz, 1H), 2.91 (s, 3H), 2.87 (s, 3H), 2.55 (dd, J = 1.6, 11.6 Hz, 1H), 2.18 (dd, J = 8.8, 15.2 Hz, 1H), 1.80-1.98 (m, 4H), 1.62-1.78 (m, 3H), 1.49-1.54 (m, 3H) ppm; **¹³C NMR** (100 MHz, CDCl₃): δ 158.3, 157.5, 139.9, 126.7, 113.8, 66.6, 55.3, 52.1, 46.2, 37.8, 36.7, 30.1, 29.0, 27.3, 26.7, 21.6 ppm; **HRMS** (ESI) calculated for C₁₈H₂₆N₂O₂ [M+Na]⁺ 325.1886, found 325.1894.

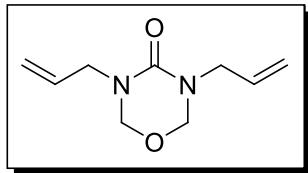


10a-(4-methoxyphenyl)-1,3-dimethyldecahydrocycloocta[d]pyrimidin-2(1H)-one 23a: Following the procedure A, the reaction of vinyl arene (**23**) (70 mg, 0.32 mmol) and formaldehyde (19 mg, 0.64 mmol) at 70 °C for 17h afforded compound **23a** in 74% yield (76 mg) as a colorless liquid; **IR** (neat): 2926, 1620, 1509, 1098, 1035 cm⁻¹; **¹H NMR** (400 MHz, CDCl₃): δ 7.18 (d, J = 8.0 Hz, 2H), 7.01 (d, J = 8.0 Hz, 2H), 3.06 (s, 3H), 2.83 (s, 3H), 2.80 (d, J = 2.4 Hz, 1H), 2.47 (d, J = 11.2 Hz, 1H), 2.31 (s, 3H), 2.03-2.16 (m, 4H), 1.49-1.88 (m, 7H), 1.23-1.32 (m, 2H) ppm; **¹³C NMR** (100 MHz, CDCl₃): δ ppm 157.8, 144.9, 136.4, 129.4, 125.1, 66.3, 54.2, 40.6, 36.7, 35.9, 30.3, 29.1, 27.9, 27.6, 23.6, 21.9, 20.9 ppm; **HRMS** (ESI) calculated for C₁₉H₂₈N₂O₂ [M+H]⁺ 317.2224, found 317.2213.

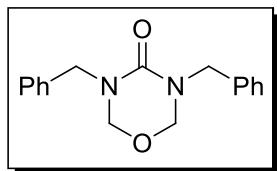


1,3-diallyl-4-p-tolyltetrahydropyrimidin-2(1H)-one 24: Diallylurea prepared by known procedure.² 1.5 g of L - (+)-tartaric acid–diallylurea (TA:DAU) (30:70) was heated to 70 °C to obtain a clear melt. To this melt, 4-methyl styrene (**8**) (118 mg, 1 mmol) and paraformaldehyde (60 mg, 2 mmol) were added at 70 °C. After completion of reaction, the reaction mixture was quenched by adding water while

still hot. The reaction mixture was cooled to room temperature and aqueous layer was extracted with EtOAc (3 x 5 mL) and washed with water (2 x 5 mL). The organic layer was dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The crude compound was purified by column chromatography over silica gel to furnish the corresponding bis-allyl THPM derivative **24** along with 3,5-diallyl-1,3,5-oxadiazinan-4-one (**25**). Colorless liquid; yield 19% (52 mg); **IR** (neat): 3441, 2927, 1617, 1513, 1411, 1249 cm⁻¹; **¹H NMR** (400 MHz, CDCl₃): δ 7.26 (d, J = 7.6 Hz, 2H), 7.15 (d, J = 7.6 Hz, 2H), 5.79-5.93 (m, 2H), 5.09-5.26 (m, 4H), 4.65 (d, J = 10.0 Hz, 1H), 3.76-3.93 (m, 5H), 3.16 (d, J = 15.2 Hz, 1H), 2.35 (s, 1H), 1.90-1.97 (m, 1H), 1.76-1.82 (m, 1H) ppm; **¹³C NMR** (100 MHz, CDCl₃): δ 159.0, 141.3, 136.9, 135.5, 133.6, 129.1, 125.7, 116.9, 115.5, 70.1, 49.6, 43.9, 43.4, 37.9, 21.2 ppm; **HRMS** (ESI) calculated for C₁₇H₂₂N₂O [M+H]⁺ 271.1806, found 271.1805.



3,5-diallyl-1,3,5-oxadiazinan-4-one 25: Colorless liquid; yield 33% (59 mg); **IR** (neat): 2927, 1617, 1513, 1411, 1249 cm⁻¹; **¹H NMR** (400 MHz, CDCl₃): δ 5.74-5.83 (m, 2H), 5.15-5.21 (m, 4H), 4.75 (s, 4H), 3.95 (d, J = 5.6 Hz, 4H) ppm; **¹³C NMR** (100 MHz, CDCl₃): δ ppm 153.7, 133.5, 117.2, 78.3, 47.0 ppm; **HRMS** (ESI) calculated for C₁₇H₁₈N₂O₂ [M+Na]⁺ 205.0947, found 205.0948.

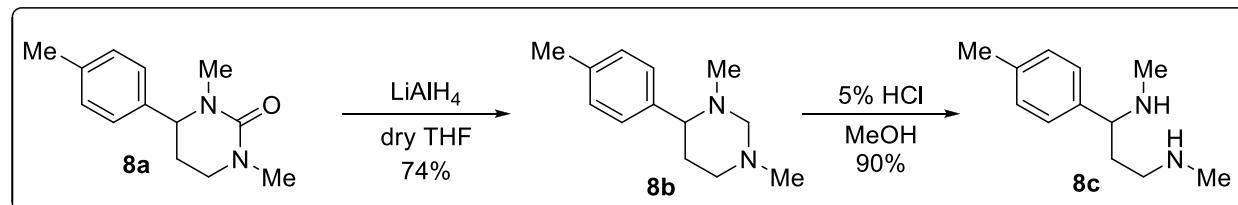


3,5-dibenzyl-1,3,5-oxadiazinan-4-one 26: Dibenzylurea prepared by known procedure.³ 1.5 g of L -(+)-tartaric acid-choline chloride (1:2) was heated to 70 °C to obtain a clear melt. To this melt, 4-methyl styrene (**8**) (59 mg, 0.5 mmol) and dibenzylurea (120 mg, 0.5 mmol) and paraformaldehyde (30 mg, 1 mmol) were added. After completion of reaction, the reaction mixture was quenched by adding water while still hot, cooled to room temperature and extracted with EtOAc (3x 5 mL). The organic layer was washed with water (2 x 5 mL), dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The crude compound was purified by column chromatography over silica gel to give 3,5-dibenzyl-1,3,5-oxadiazinan-4-one (**27**) as a colorless liquid (102 mg) in 72% yield. **IR** (neat): 2926, 1620, 1509, 1098, 1035 cm⁻¹; **¹H NMR** (400 MHz, CDCl₃): δ 7.28-7.39 (m, 10H), 4.76 (s, 4H), 4.62 (s, 4H) ppm; **¹³C NMR** (100 MHz, CDCl₃): δ ppm 154.2, 137.5, 128.8, 127.8, 127.5, 78.5, 48.1 ppm; **HRMS** (ESI) calculated for C₁₇H₁₈N₂O₂ [M+Na]⁺ 305.1260, found 305.1262.

Gram scale synthesis of THPMs derivative **8a:** 9.0 g of L -(+)-tartaric acid-DMU (30:70) was heated to 70 °C to obtain a clear melt. To this melt, 1 g of 4-methyl styrene (**8**) and 508 mg of paraformaldehyde were added at 70 °C. The reaction was monitored by thin layer chromatography. After completion of reaction, the reaction mixture was quenched by adding water while still hot. The reaction mixture was cooled to room temperature and aqueous layer was extracted with EtOAc (3 x 40 mL) and washed with water (2 x 40 mL). The organic layer was dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The crude compound was purified by column chromatography over silica gel using 30% EtOAc/hexane to afford pure **8a** (1.04g) THPM derivative in 56% yield.

Recovery and recycle of melt mixture: To recover the melt, aqueous layer was concentrated under reduced pressure. The recovered melt mixture was reused in the next cycle without any further purification. The recovered melt was azotropically dried with toluene. To perform the reaction in recovered melt 1 mmol DMU was added and allowed to stir 70 °C to obtain a clear melt. To this melt, 1 mmol of 4-methyl styrene (**8**) and 60 mg of paraformaldehyde were added at 70 °C. The reaction progress was monitored by thin layer chromatography. After completion of reaction, the reaction mixture was quenched by adding water while still hot. The reaction mixture was cooled to room temperature and aqueous layer was extracted with EtOAc (3 x 40 mL) and washed with water (2 x 40 mL). The organic layer was dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The crude compound was purified by column chromatography over silica gel using 30% EtOAc/hexane to afford pure THPM derivative **8a** (107 mg) in 49% yield.

Synthesis of 1,3-diamine:



A mixture of lithium aluminum hydride (186 mg, 5.04 mmol) and **8a** (220 mg, 1.09 mmol) in anhydrous THF (5.0 mL) was stirred for 4 h. The reaction was quenched with water (3.0 mL), and 10% aqueous sodium hydroxide (3.0 mL) was added. After being

stirred for 15 minutes, the mixture was diluted with ethyl acetate, filtered through celite pad. The filtrate was extracted with ethyl acetate three times. The combined organic layer was dried over anhydrous sodium sulfate and concentrated in vacuo to give the crude product, which was purified by silica gel chromatography.

1,3-dimethyl-4-(p-tolyl)hexahydropyrimidine 8b: 156 mg, colourless liquid, yield 74%; **IR** (neat): 2941, 2783, 2374, 2349, 1513, 1447, 1387, 1283 cm⁻¹; **¹H NMR** (400 MHz, CDCl₃): δ 7.16 (d, J = 7.6 Hz, 2H), 7.04 (d, J = 8.0 Hz, 2H), 3.67 (d, J = 8.8 Hz, 1H), 2.86 (d, J = 11.6 Hz, 2H), 2.67 (dd, J = 2.4, 11.2 Hz, 1H), 2.46 (d, J = 9.2 Hz, 1H), 2.25 (s, 6H), 2.06 (dt, J = 2.4, 12.0 Hz, 1H), 1.80-1.88 (m, 4H), 1.52 (dd, J = 2.0, 13.2 Hz, 1H) ppm; **¹³C NMR** (100 MHz, MeOH-d⁴): δ 140.6, 136.7, 129.1, 127.4, 80.1, 68.6, 54.7, 42.9, 40.6, 34.3, 21.1 ppm; **HRMS** (ESI) calculated for C₁₃H₂₀N₂ [M+H]⁺ 205.1699, found 205.1700.

N^{1,N³}-dimethyl-1-(p-tolyl)propane-1,3-diamine 8c: To the product (100 mg, 0.49 mmol) obtained above was added 5% HCl in methanol (3.0 mL). The mixture was stirred at 50 °C until TLC revealed complete conversion. and concentrated under reduced pressure to give 1,3-diamine 87 mg, white solid (moisture sensitive), yield 90%; **IR** (neat): 3403, 2978, 2743, 1465, 1267 cm⁻¹; **¹H NMR** (400 MHz, MeOH-d⁴): δ 7.34 (d, J = 8.0 Hz, 2H), 7.24 (d, J = 7.6 Hz, 2H), 4.23 (dd, J = 4.0, 10.8 Hz, 1H), 3.19 (d, J = 1.2 Hz, 1H), 2.90 (dd, J = 4.4, 11.2 Hz, 1H), 2.51-2.59 (m, 4H), 2.44 (s, 3H), 2.35 (dd, J = 4.8, 11.6 Hz, 1H), 2.28 (s, 3H) ppm; **¹³C NMR** (100 MHz, MeOH-d⁴): δ 141.9, 131.5, 130.9, 129.6, 61.9, 46.7, 33.7, 31.6, 30.2, 21.3 ppm; **HRMS** (ESI) calculated for C₁₂H₂₀N₂ [M+H]⁺ 193.1699, found 193.1700.

References:

1. (a) L. J. Rono, H. G. Yayla, D. Y. Wang, M. F. Armstrong, and R. R. Knowles, *J. Am. Chem. Soc.*, 2013, **135**, 17735; (b) T.-Q. Wang, Y.-Y. Hu, and S.-L. Zhang, *Org. Biomol. Chem.*, 2010, **8**, 2312; (c) A. M. Del Hoyo, A. G. Herraiz and M. G. Suero, *Angew. Chem., Int. Ed.*, 2017, **56**, 1610.
2. C. Iacobucci, C. Piotrowski, A. Rehkamp, C. H. Ihling and A. Sinz, *J. Am. Soc. Mass Spectrom.*, 2019, **30**, 139-148.
3. M. A. Pasha and V. P. Jayashankara, *Synth. Commun.*, 2006, **36**, 1787-1793.

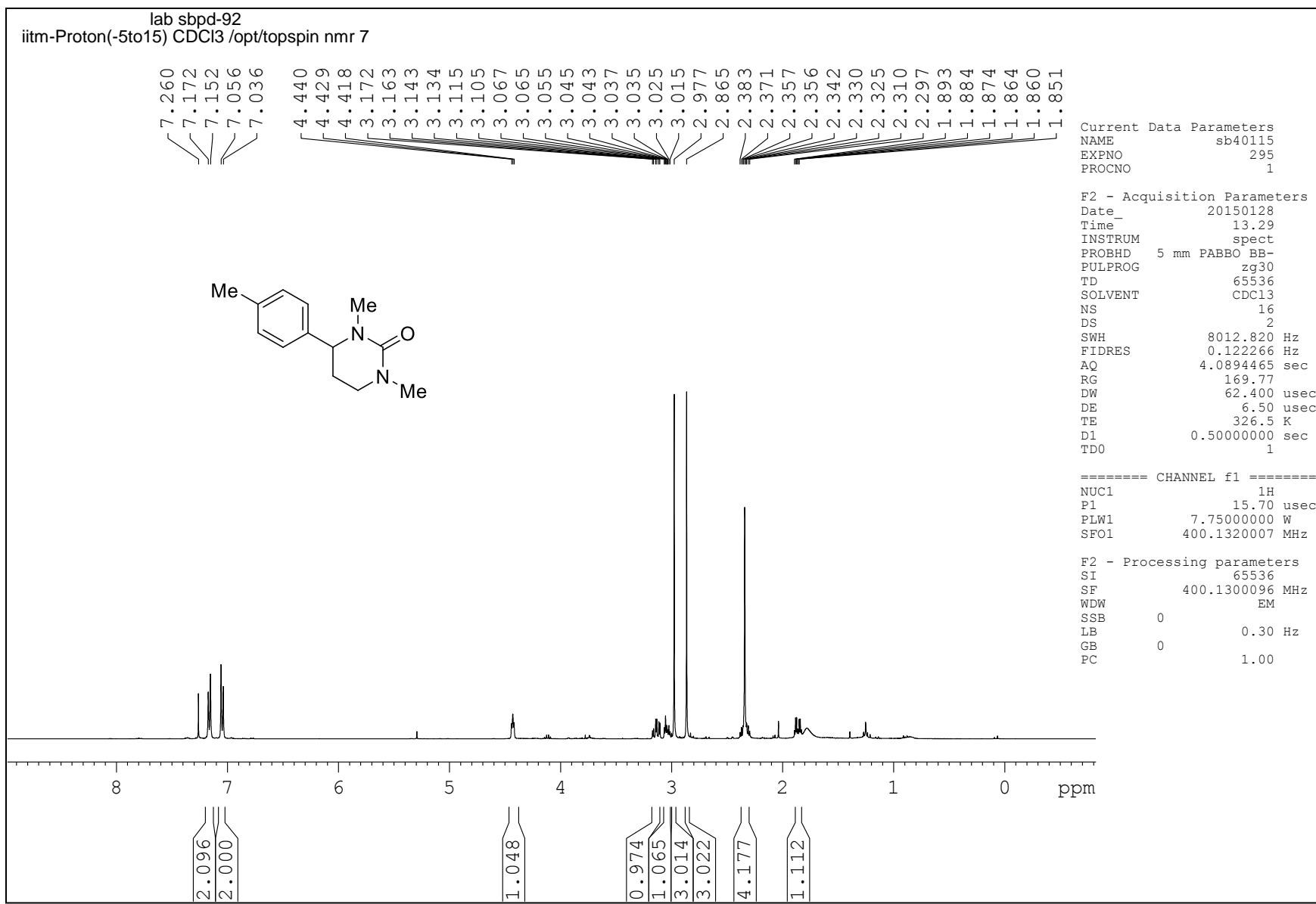


Figure 1 ^1H NMR spectrum of compound 8a

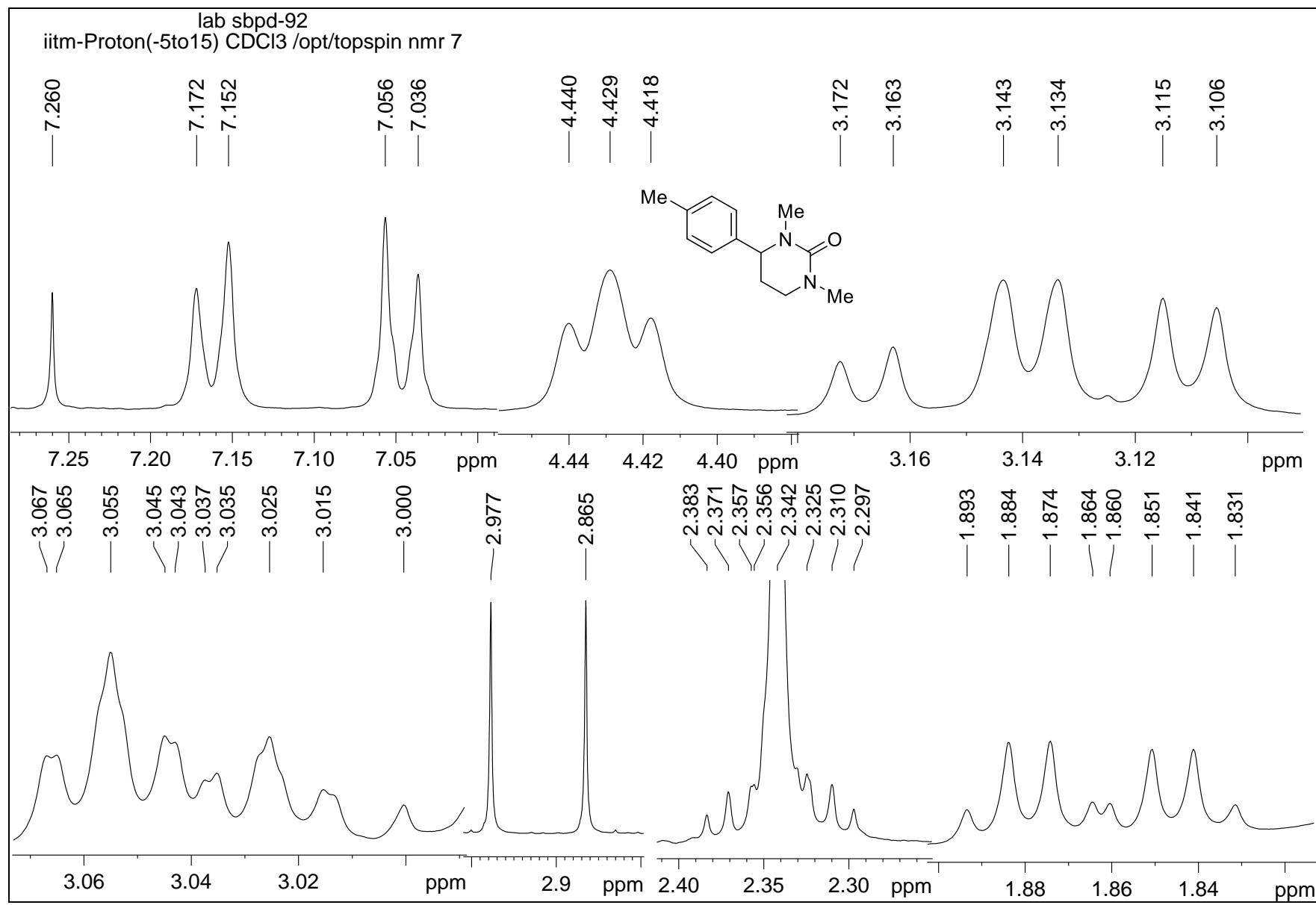
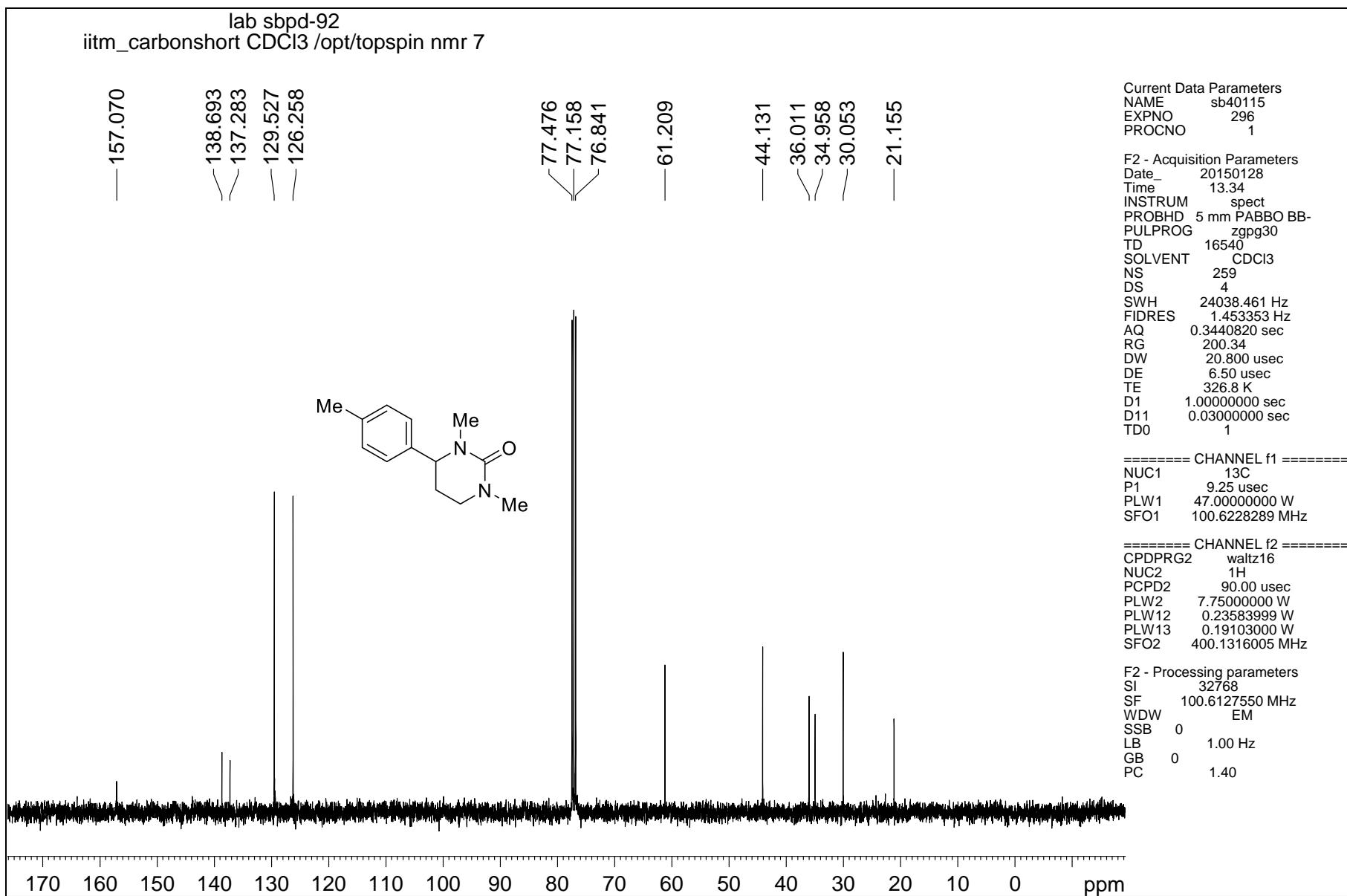


Figure 2 Expanded ¹H NMR spectrum of compound 8a



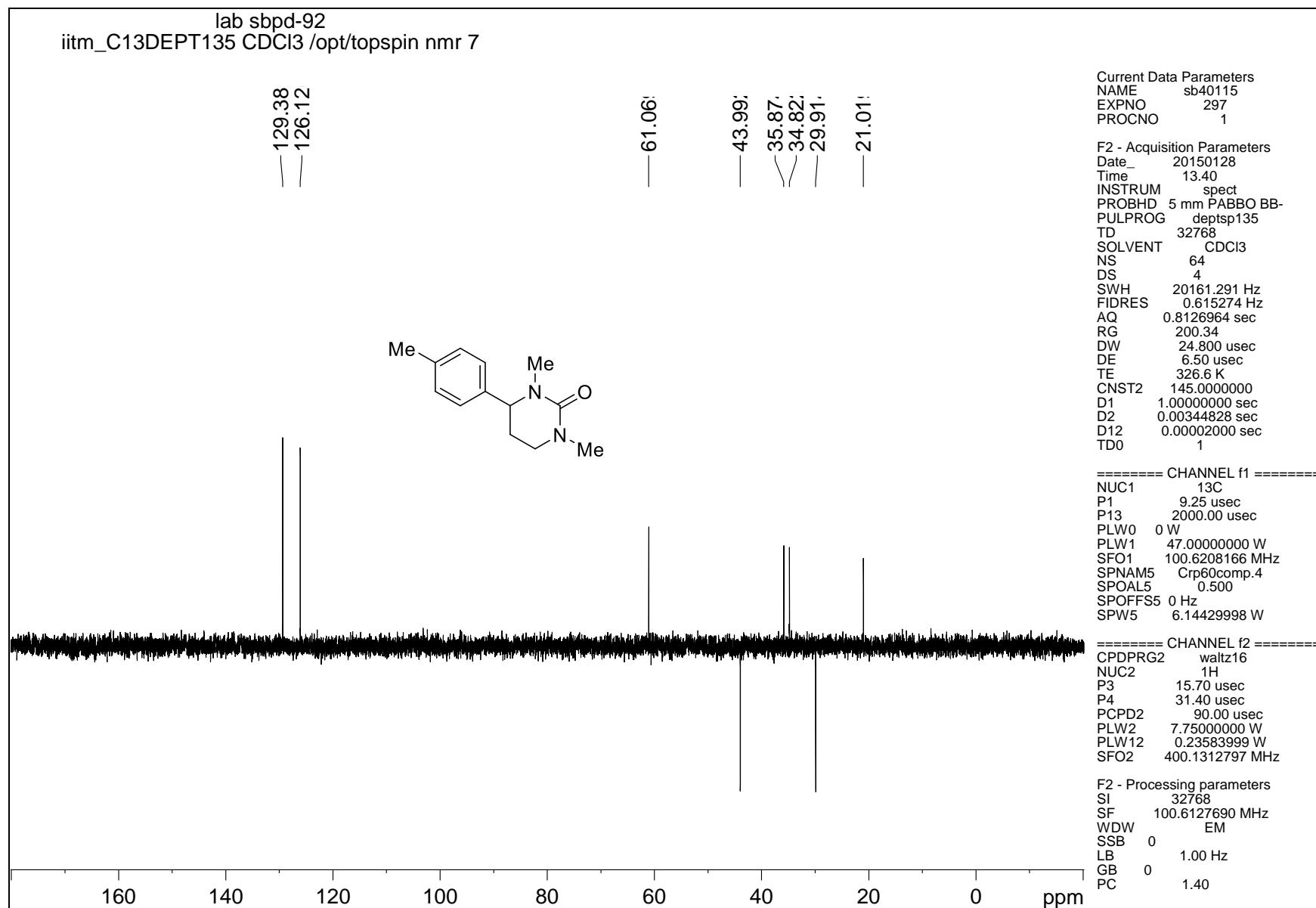


Figure 4 DEPT-135 NMR spectrum of compound 8a

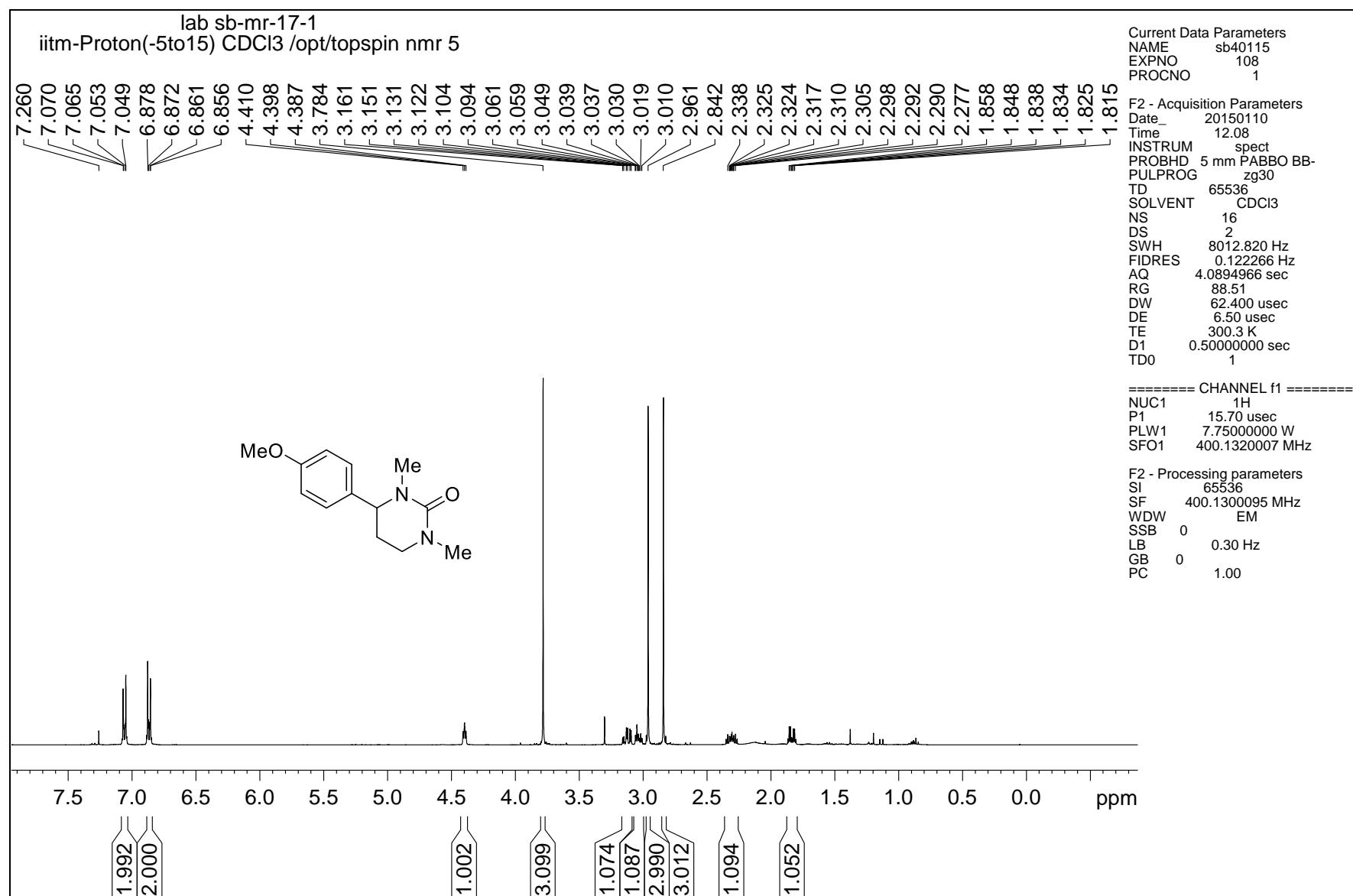


Figure 5 ¹H NMR spectrum of compound 9a

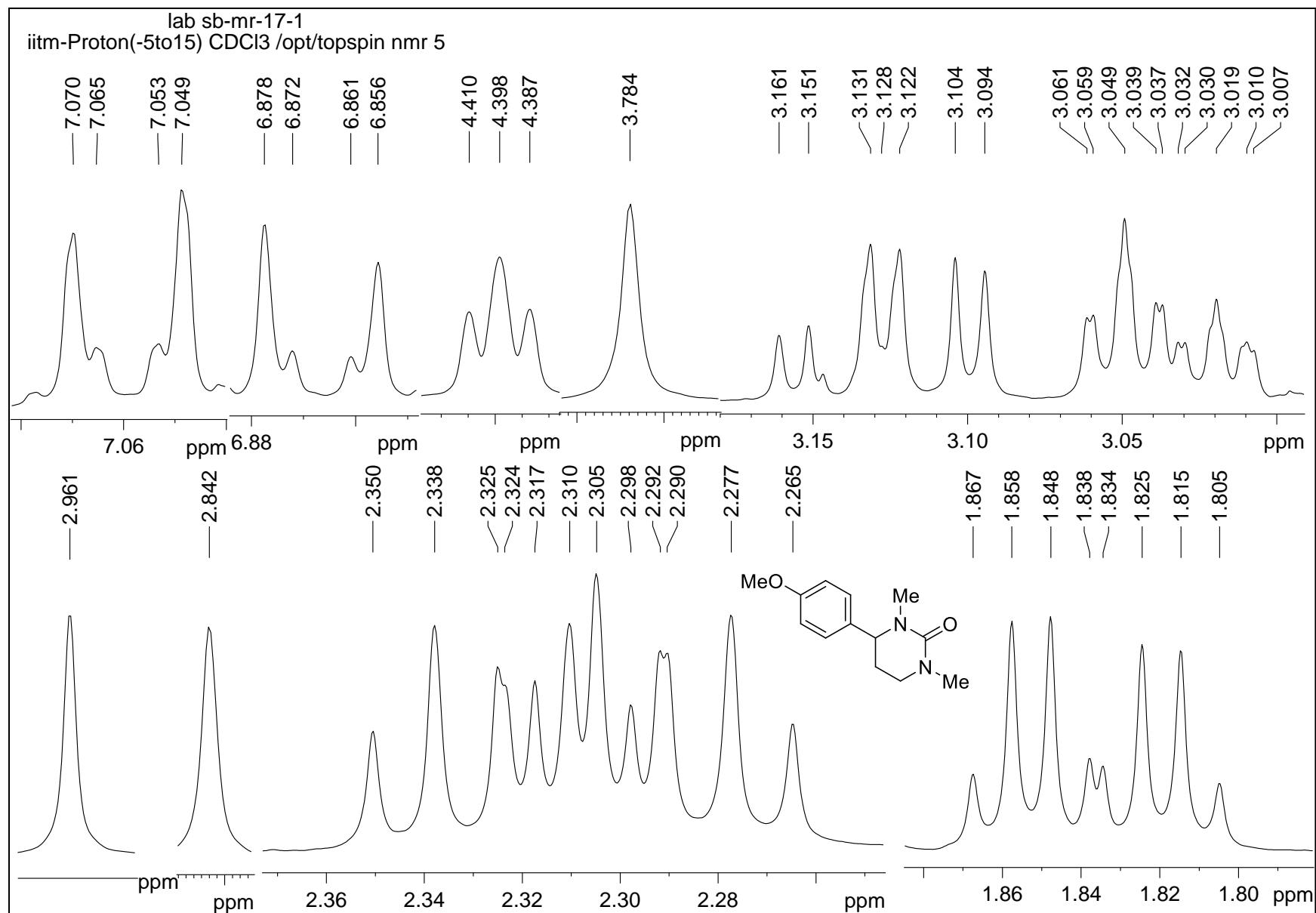


Figure 6 Expanded ^1H NMR spectrum of compound 9a

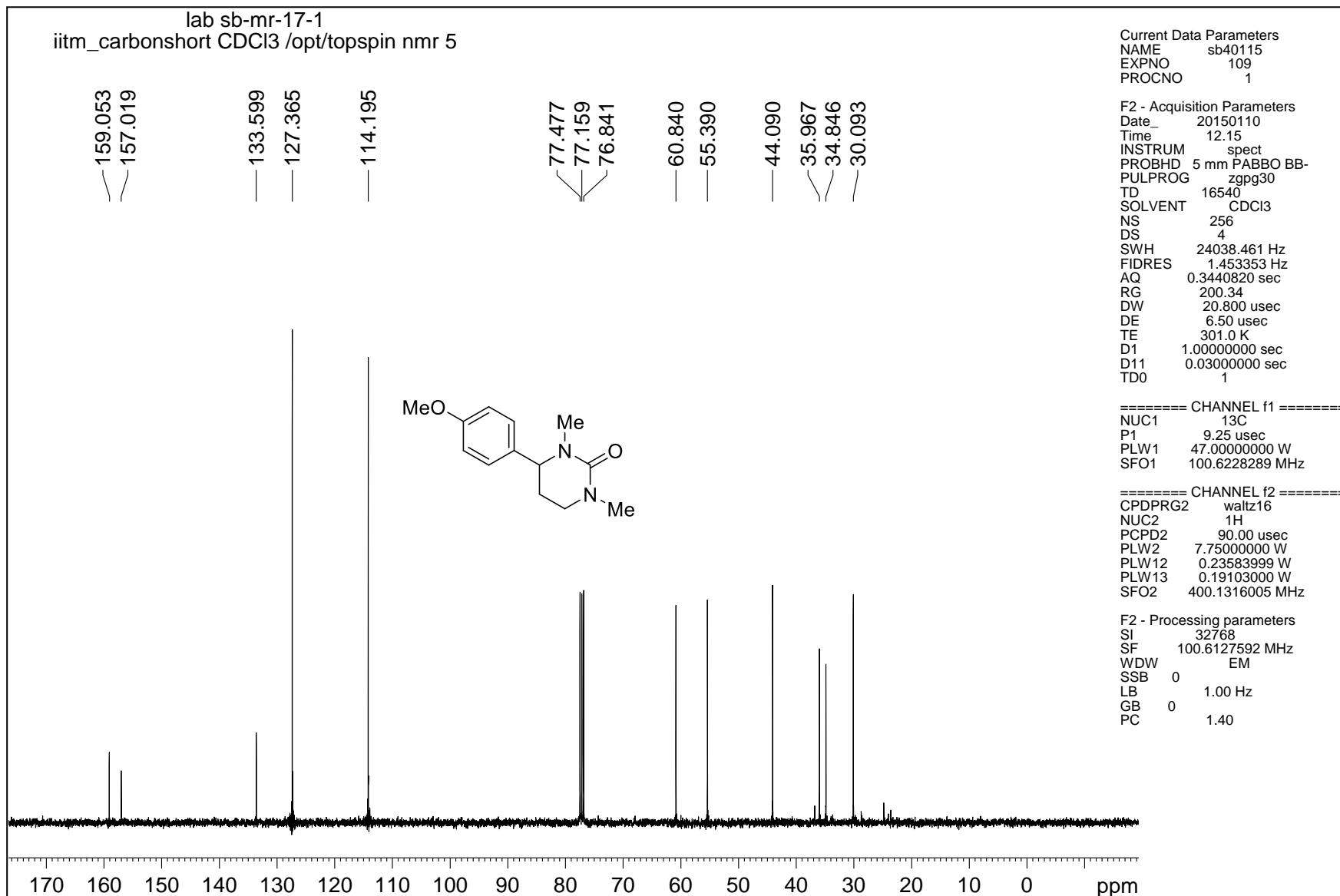
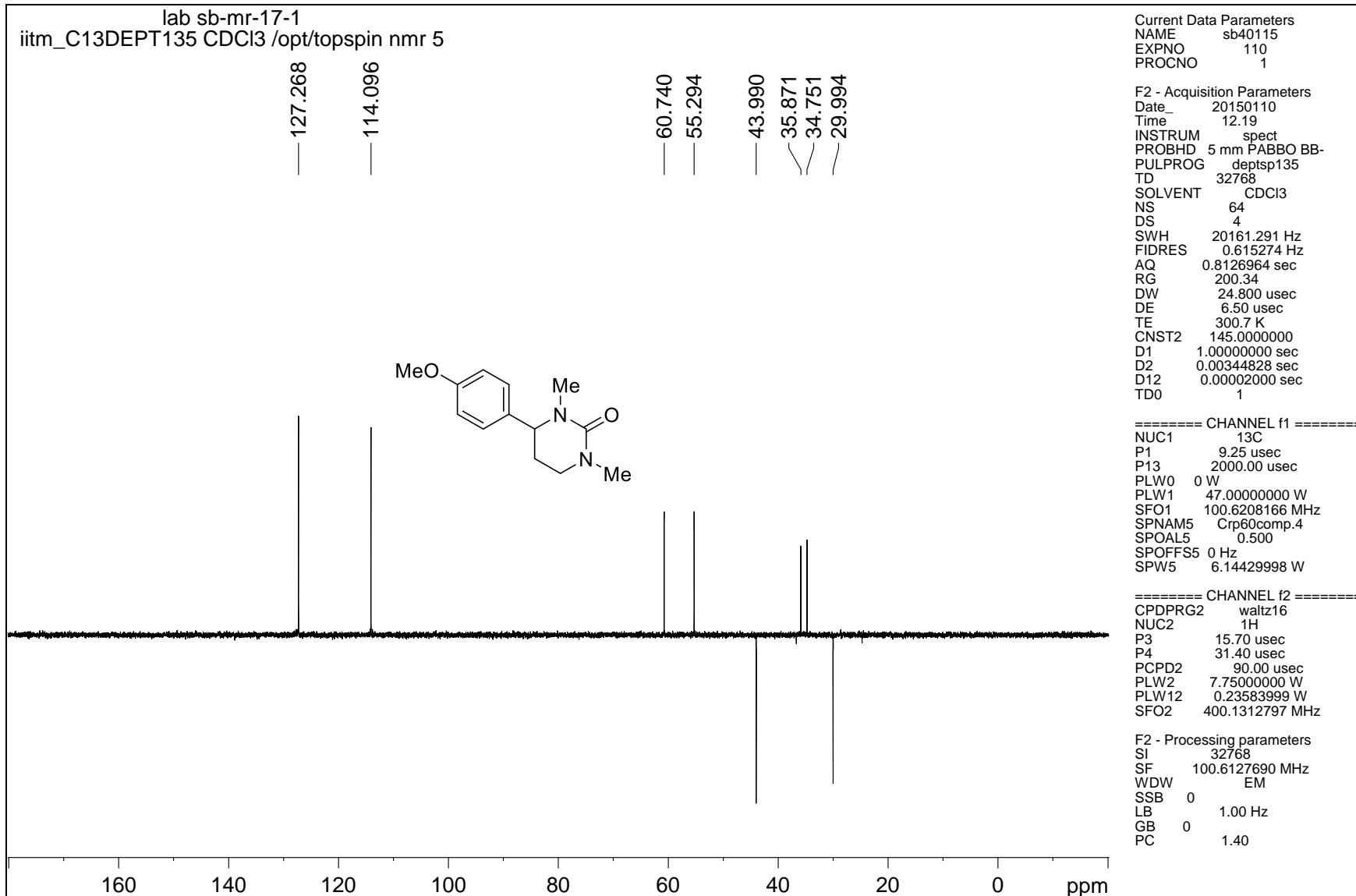
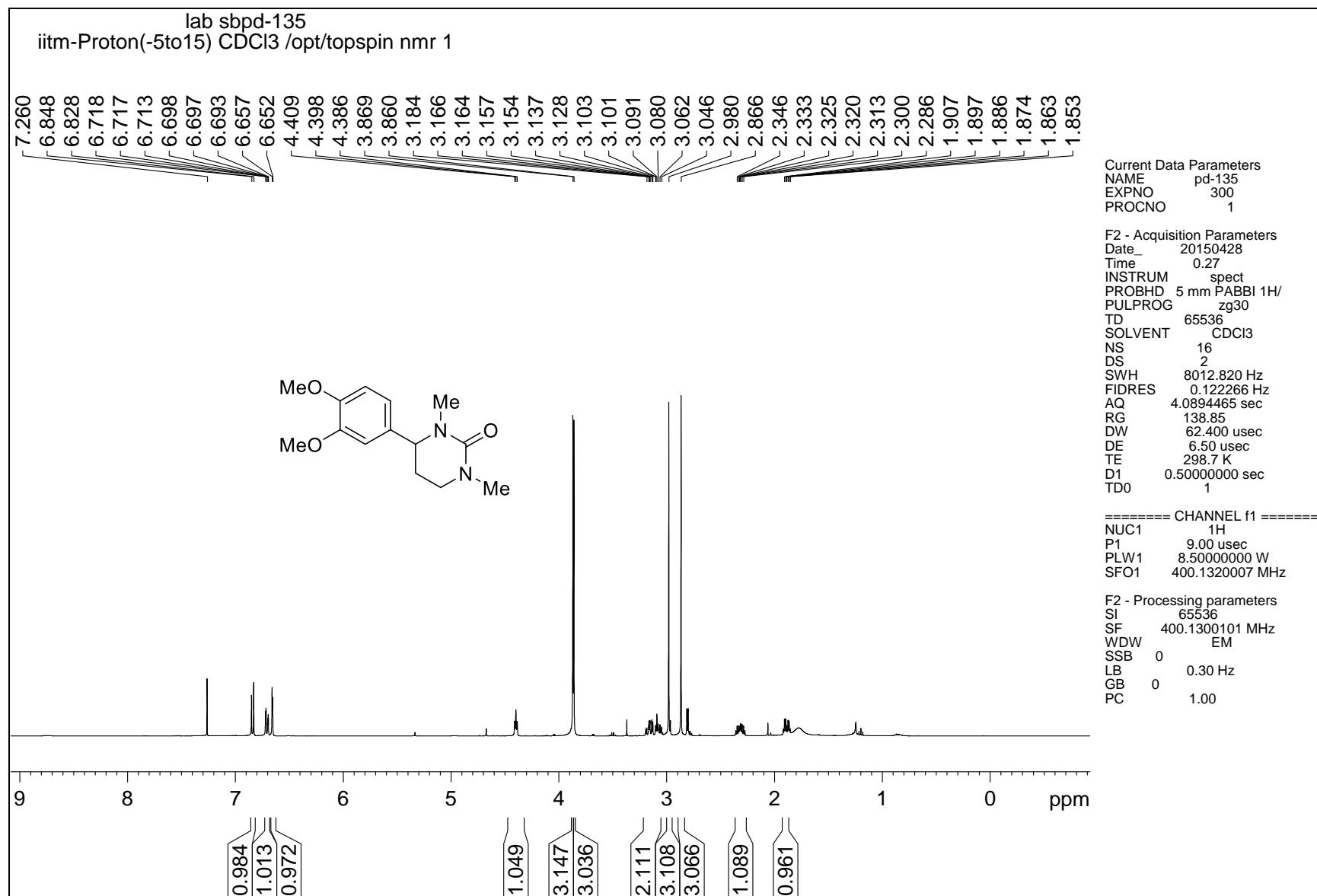
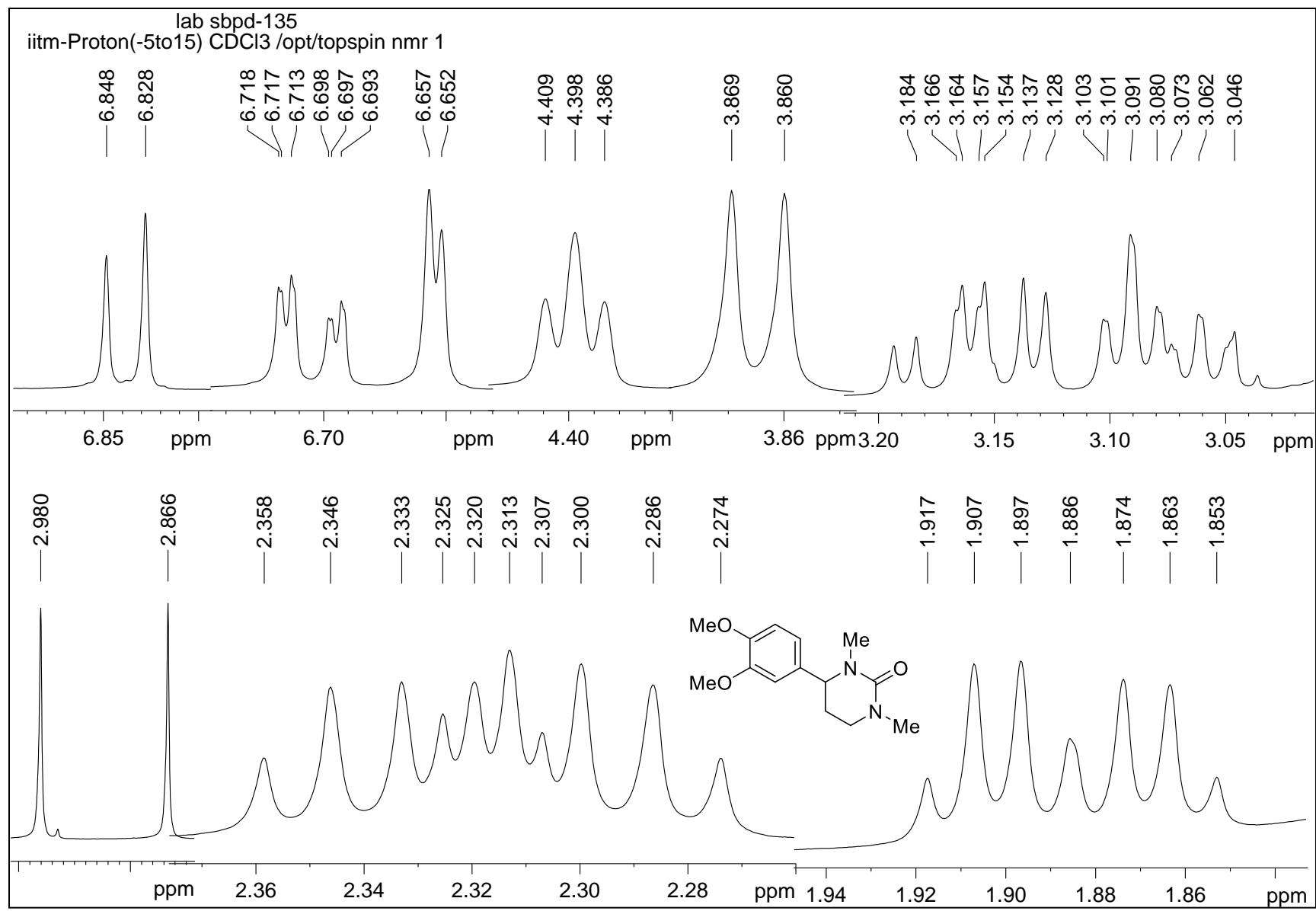


Figure 7 ¹³C NMR spectrum of compound 9a







lab sbpd-135
iitm_carbonshort CDCl₃ /opt/topspin nmr 1

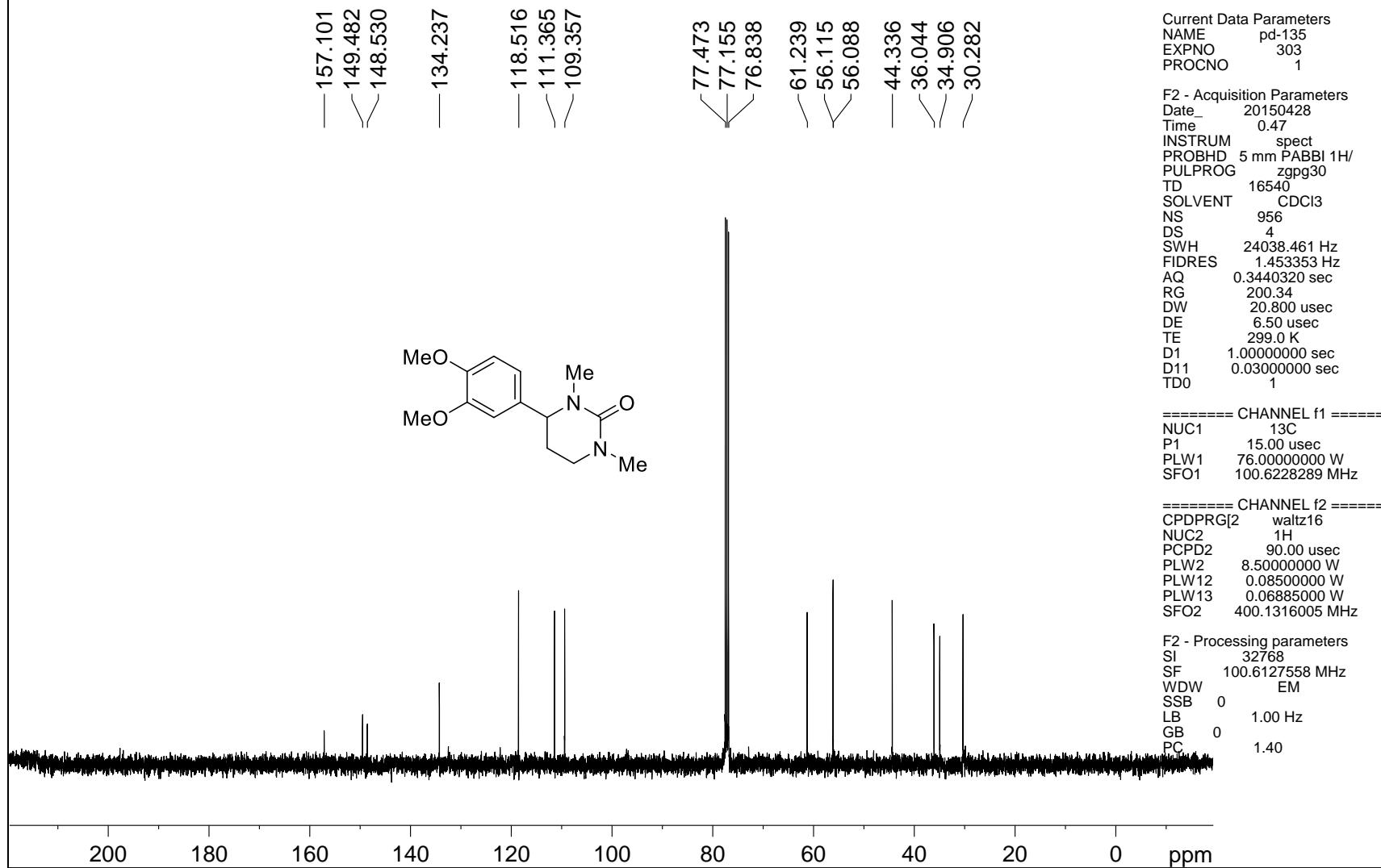
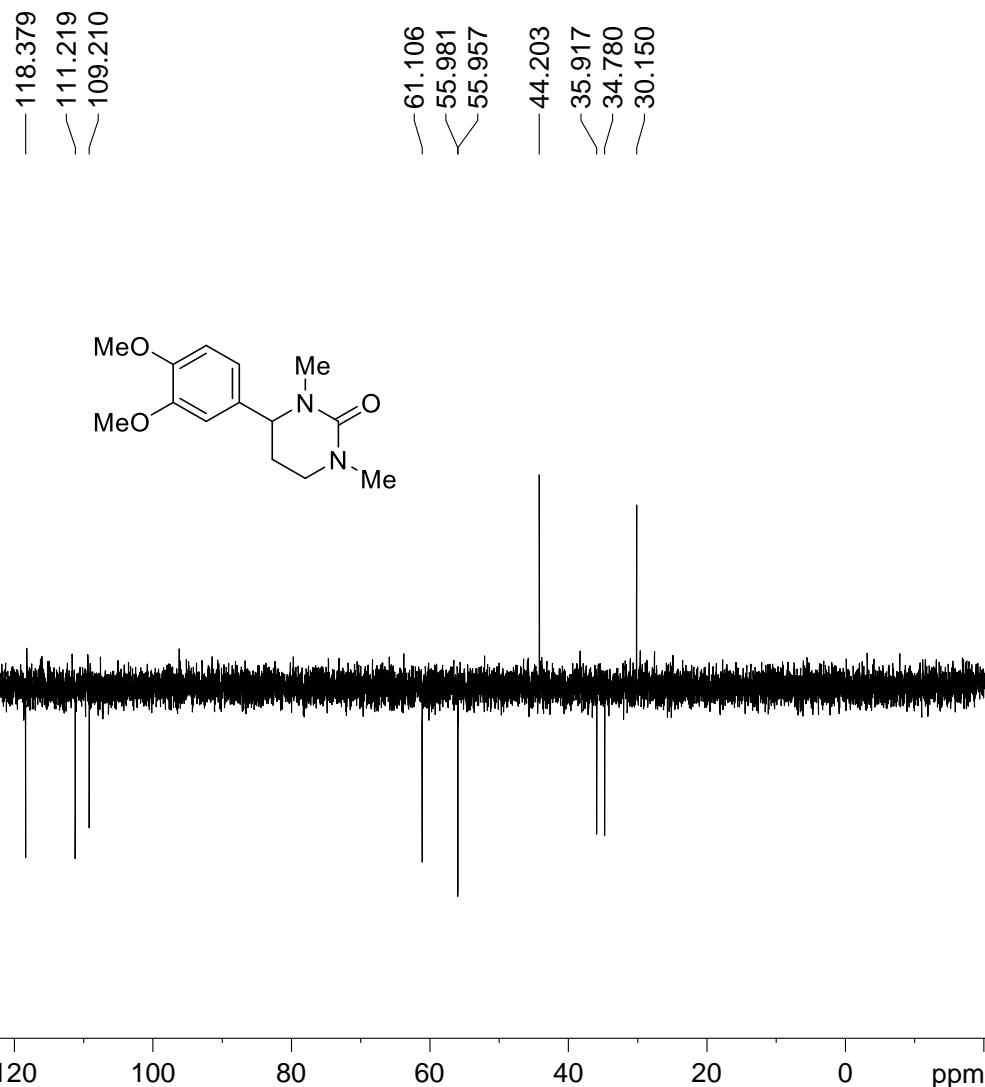


Figure 11 ¹³C NMR spectrum of compound 10a

lab sbpd-135
litm_C13DEPT135 CDCl₃ /opt/topspin nmr 1



Current Data Parameters
NAME sb40415
EXPNO 302
PROCNO 1

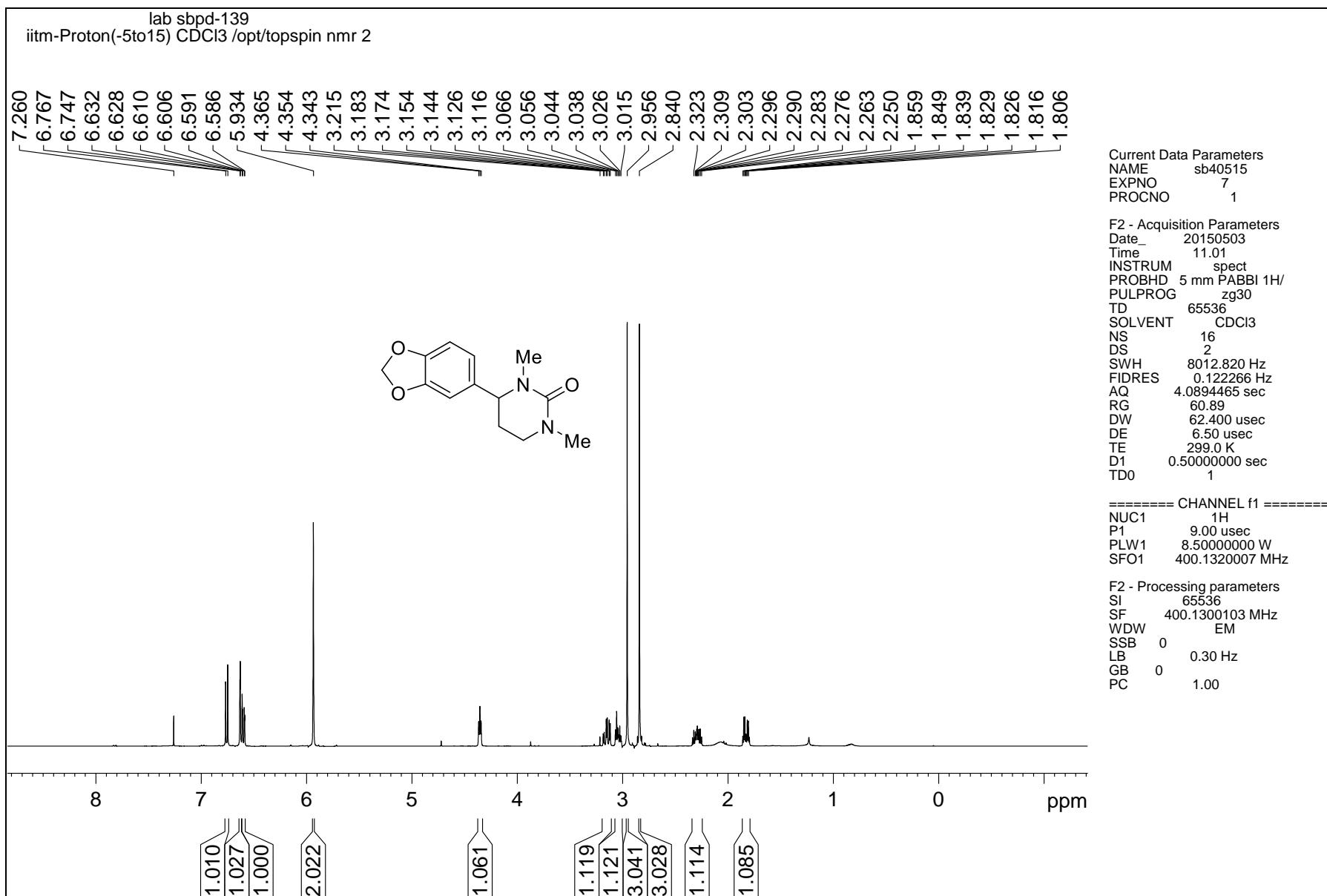
F2 - Acquisition Parameters
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PROBHD 5 mm PABBI 1H/
PULPROG deptsp135
TD 32768
SOLVENT CDCl₃
NS 64
DS 4
SWH 20161.291 Hz
FIDRES 0.615274 Hz
AQ 0.8126464 sec
RG 200.34
DW 24.800 usec
DE 6.50 usec
TE 298.9 K
CNST2 145.0000000
D1 1.0000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

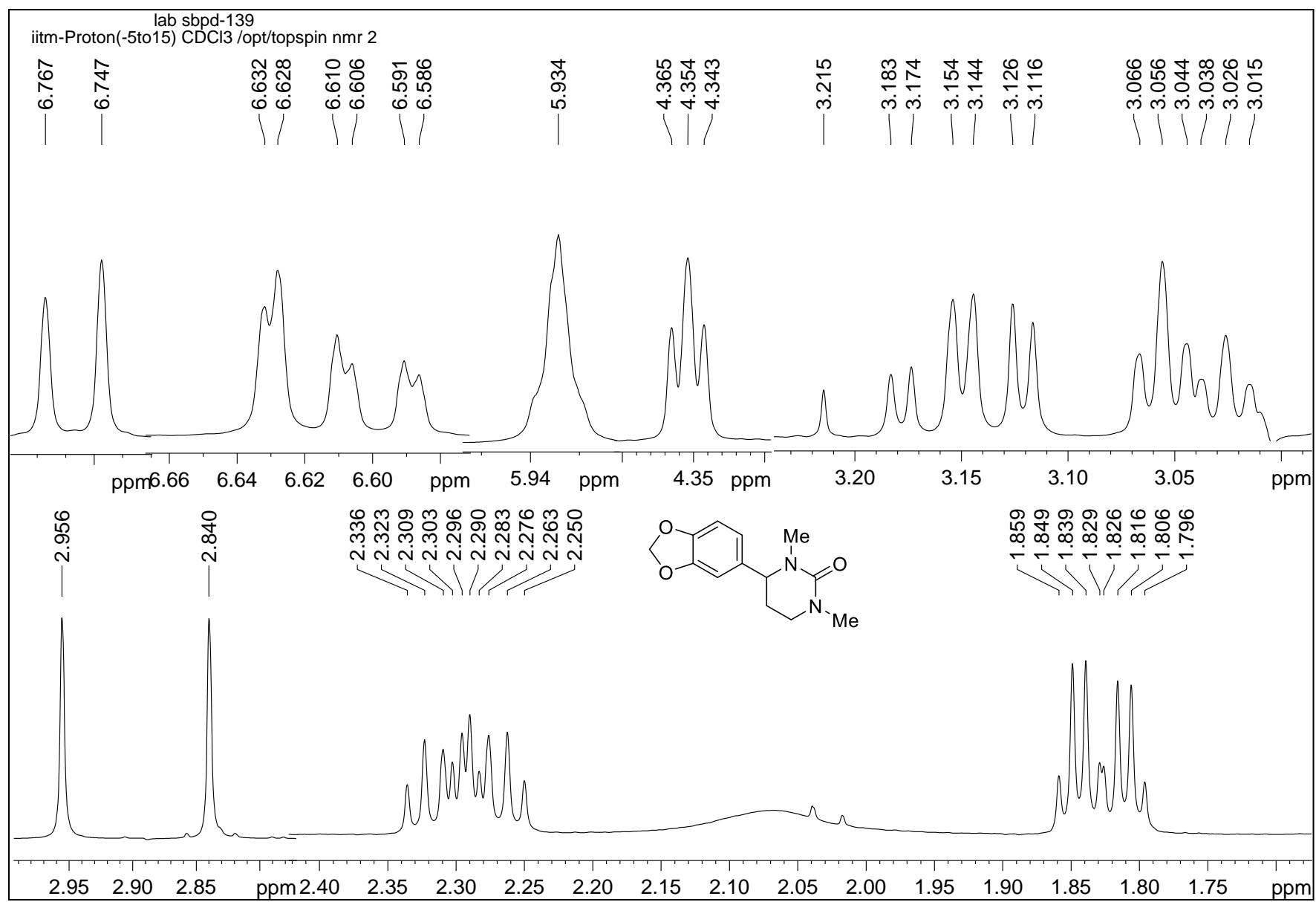
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P13 2000.00 usec
PLW0 0 W
PLW1 76.00000000 W
SFO1 100.6208166 MHz
SPNAM[5] Crp60comp.4
SPOALS5 0.500
SPOFFS5 0 Hz
SPW5 26.12700081 W

===== CHANNEL f2 =====
CPDPRG[2 waltz16
NUC2 1H
P3 9.00 usec
P4 18.00 usec
PCPD2 90.00 usec
PLW2 8.50000000 W
PLW12 0.08500000 W
SFO2 400.1312797 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure 12 DEPT-135 NMR spectrum of compound 10a





lab sbpd-139
itm_carbonshort CDCl₃ /opt/topspin nmr 2

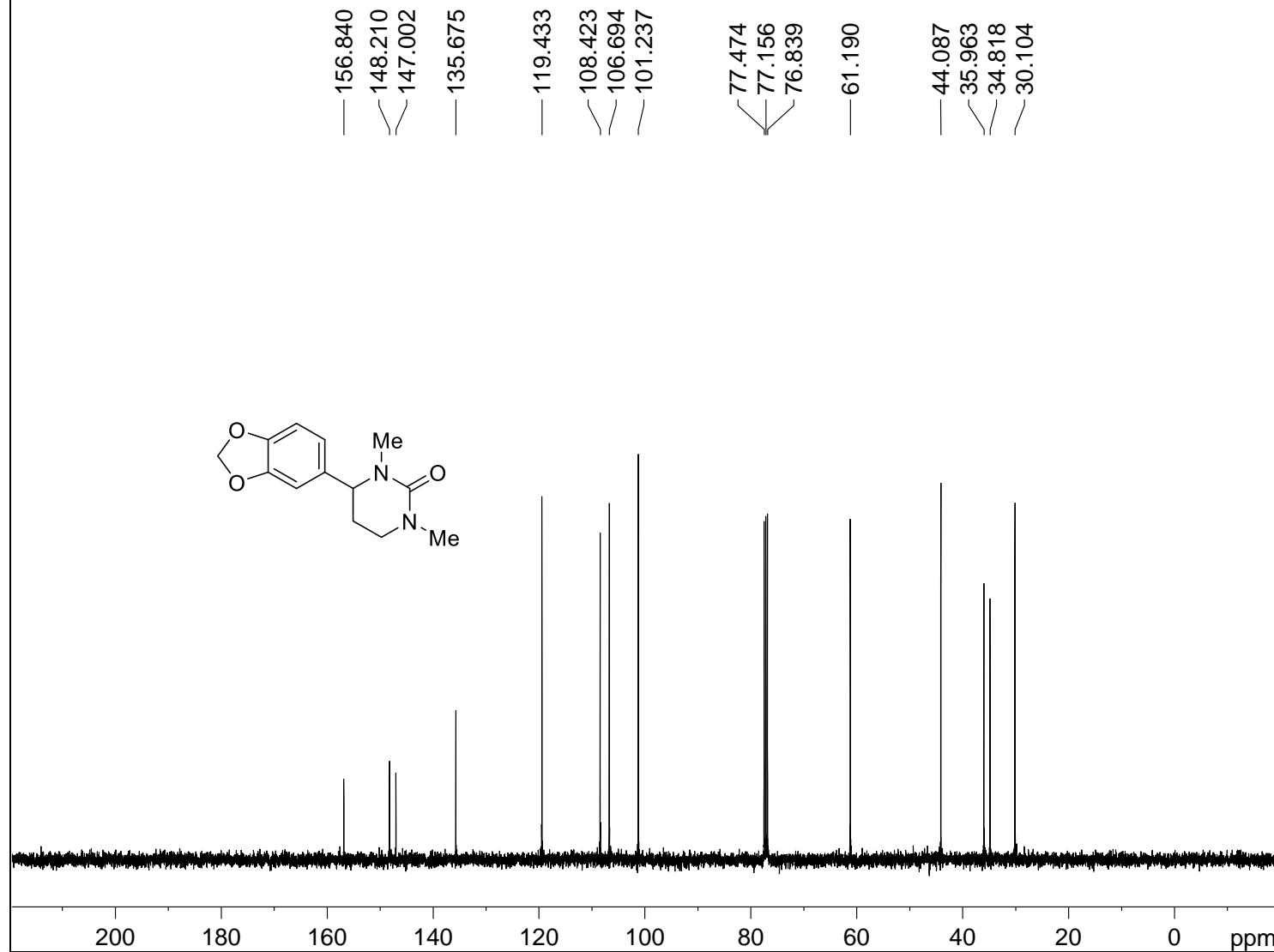


Figure 15 ¹³C NMR spectrum of compound 11a

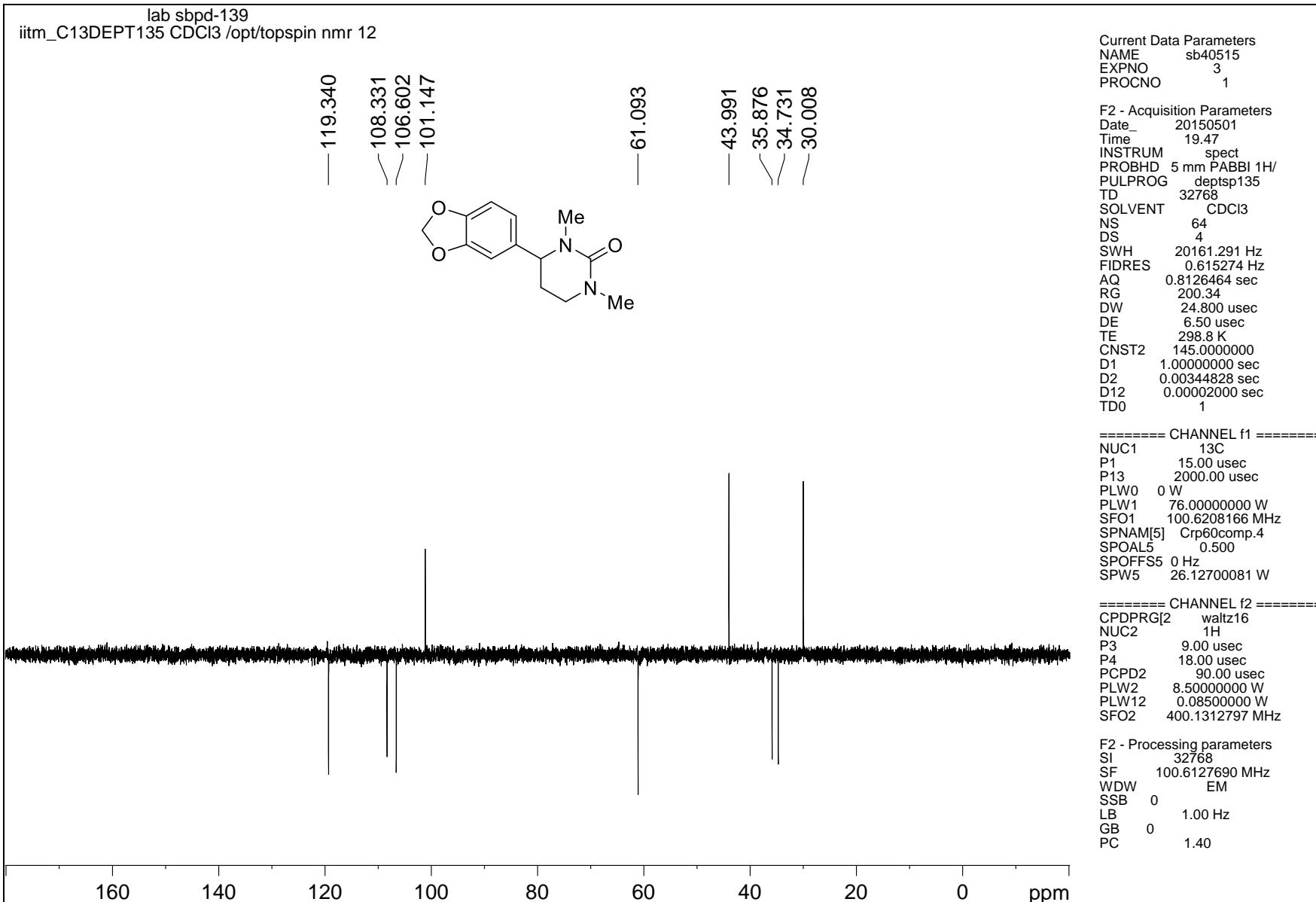
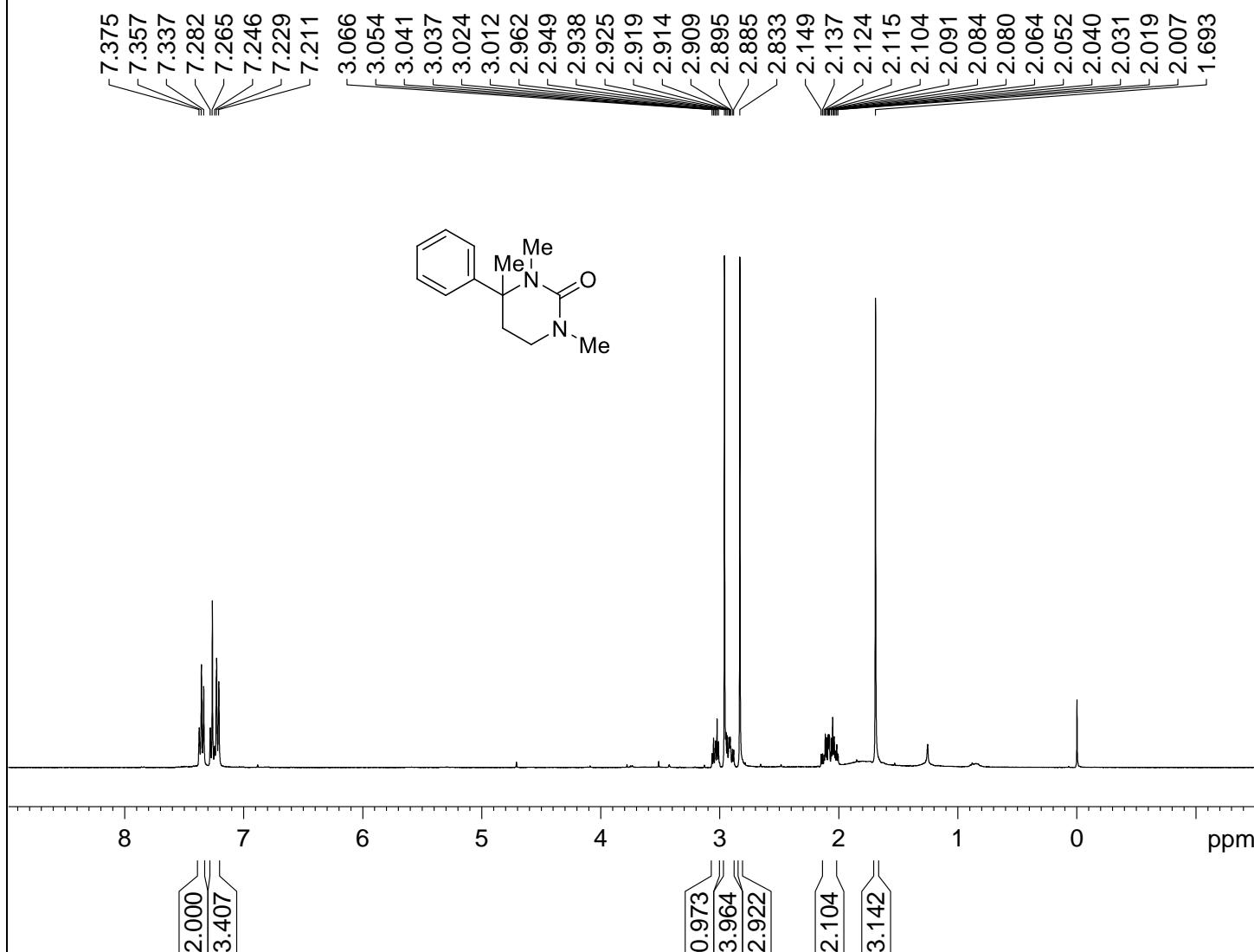


Figure 16 DEPT-135 NMR spectrum of compound 11a

lab sbpd-701
iitm-Proton(-5to15) CDCl₃ /opt/topspin nmr 2



Current Data Parameters
NAME PD-701
EXPNO 315
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190221
Time 21.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 200.34
DW 62.400 usec
DE 6.50 usec
TE 296.5 K
D1 0.5000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 400.1320007 MHz
NUC1 1H
P1 15.70 usec
PLW1 7.7500000 W

F2 - Processing parameters
SI 65536
SF 400.1300077 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure 17 ¹H NMR spectrum of compound 12a

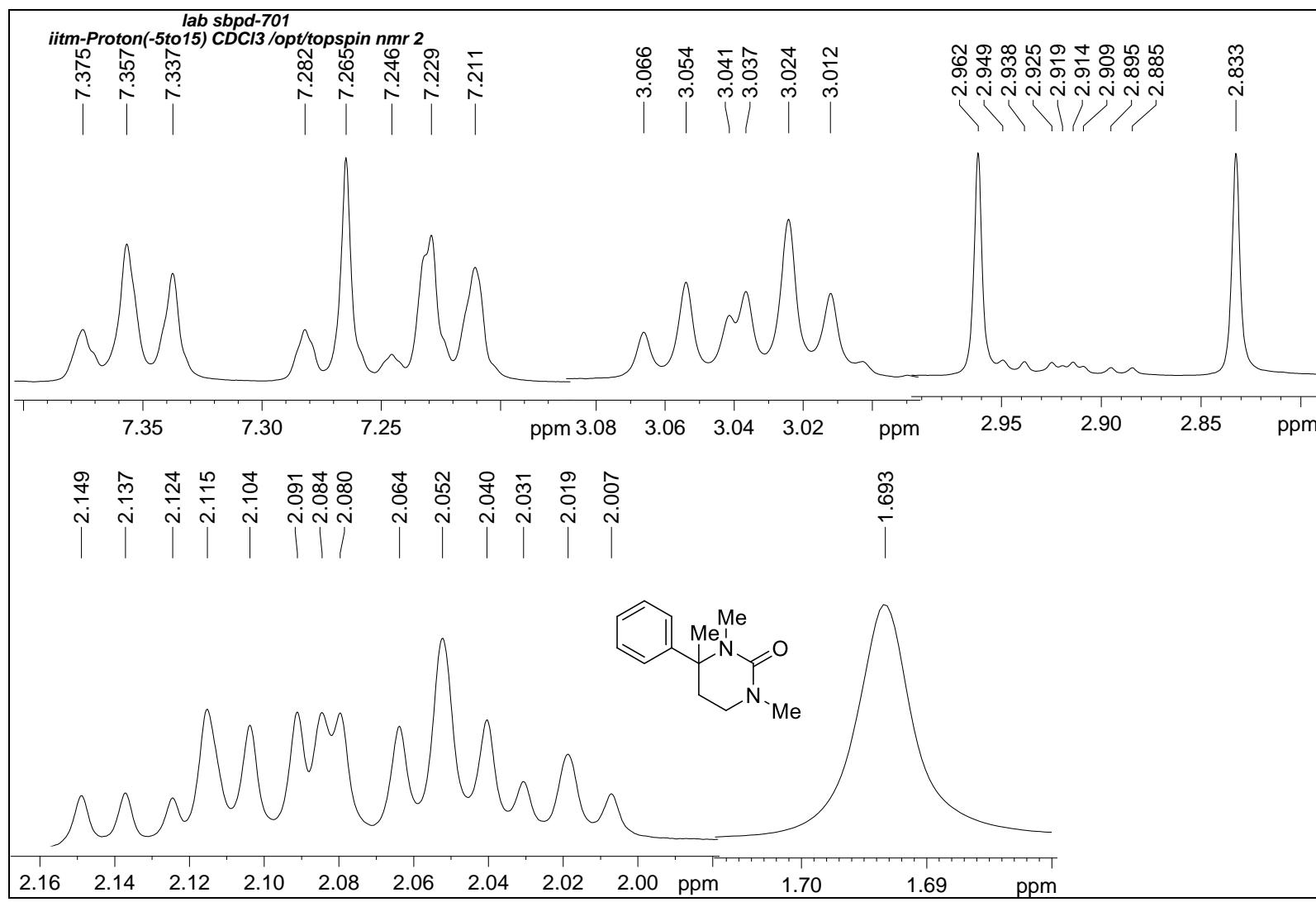
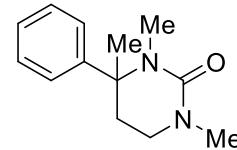


Figure 18 Expanded ^1H NMR spectrum of compound 12a

lab sbpd-701
iitm_carbonshort CDCl₃ /opt/topspin nmr 3

— 157.356
— 145.138
— 128.763
— 127.123
— 125.841

— 61.016
— 43.882
— 38.475
— 36.246
— 30.514
— 26.807



Current Data Parameters
NAME pd-701-c
EXPNO 330
PROCNO 1

F2 - Acquisition Parameters
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Time 12.42
INSTRUM spect
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PULPROG zgpg30
TD 16540
SOLVENT CDCl₃
NS 1000
DS 4
SWH 24038.461 Hz
FIDRES 1.453353 Hz
AQ 0.3440320 sec
RG 200.34
DW 20.800 usec
DE 6.50 usec
TE 296.6 K
D1 1.0000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228289 MHz
NUC1 ¹³C
P1 9.25 usec
PLW1 47.0000000 W

===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 ¹H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 7.7500000 W
PLW12 0.23583999 W
PLW13 0.11863000 W

F2 - Processing parameters
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SF 100.6127562 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Figure 19 ¹³C NMR spectrum of compound 12a

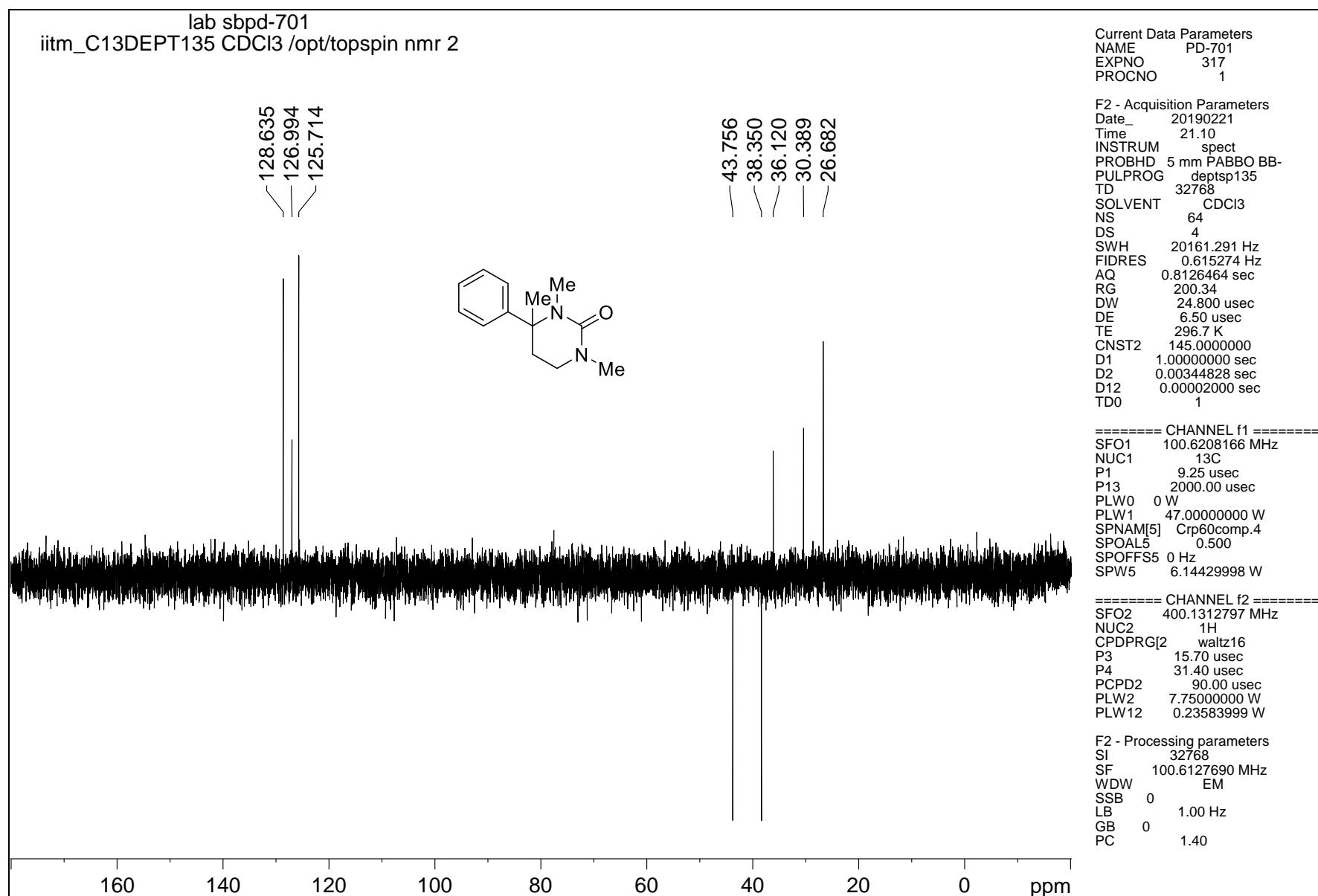
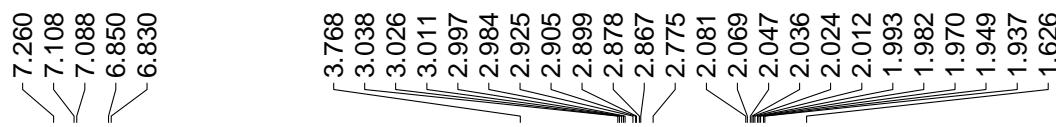


Figure 20 DEPT-135 NMR spectrum of compound 12a

lab sbpd-706
iitm-Proton(-5to15) CDCl₃ /opt/topspin nmr 13



Current Data Parameters
NAME pd-706
EXPNO 351
PROCNO 1

F2 - Acquisition Parameters
Date 20190225
Time 13.52
INSTRUM spect
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PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 67.99
DW 62.400 usec
DE 6.50 usec
TE 296.7 K
D1 0.5000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 400.1320007 MHz
NUC1 1H
P1 15.70 usec
PLW1 7.7500000 W

F2 - Processing parameters
SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure 21 ¹H NMR spectrum of compound 13a

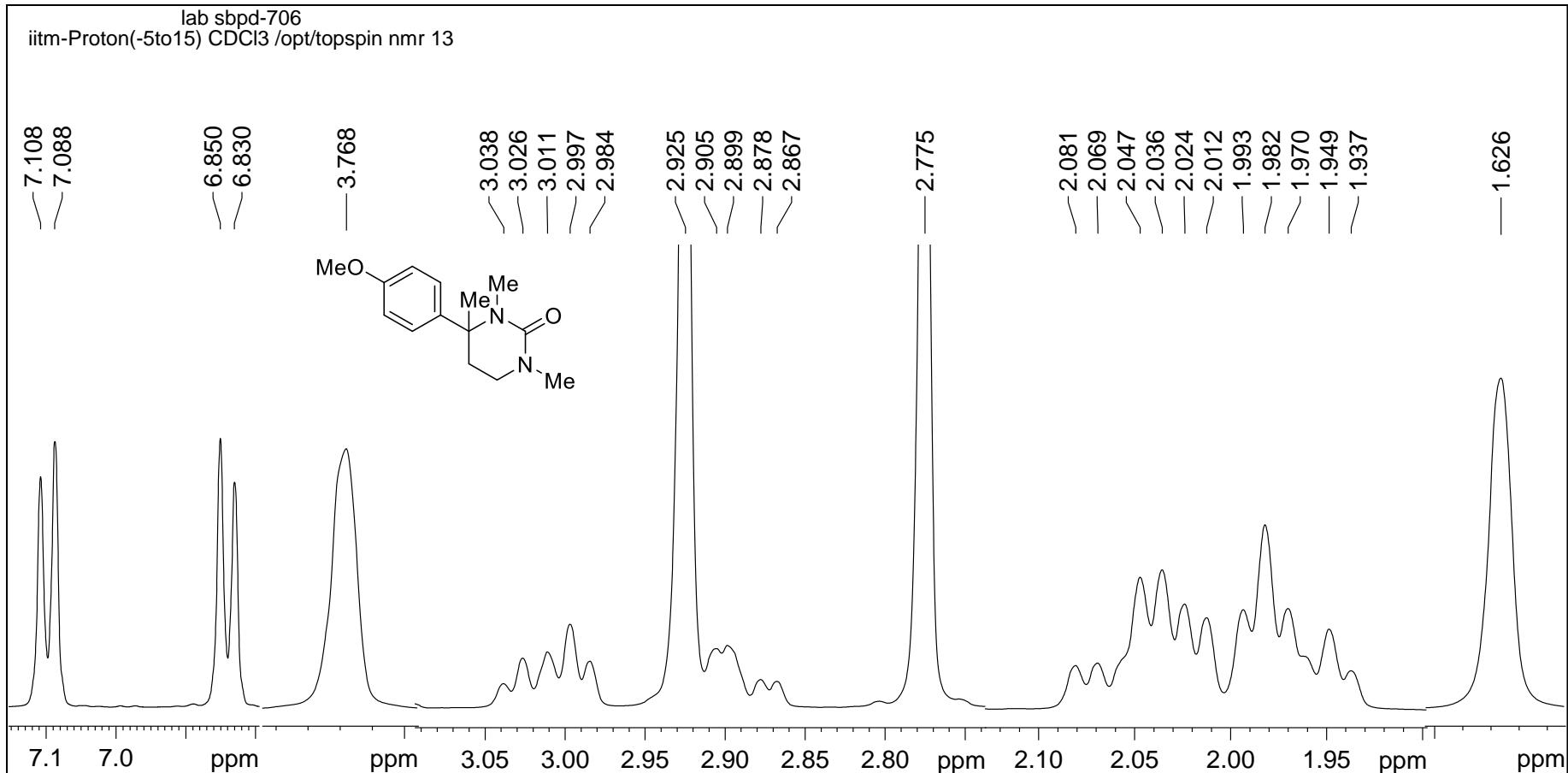


Figure 22 Expanded ¹H NMR spectrum of compound 13a

lab sbpd-706
iitm_carbonshort CDCl₃ /opt/topspin nmr 13

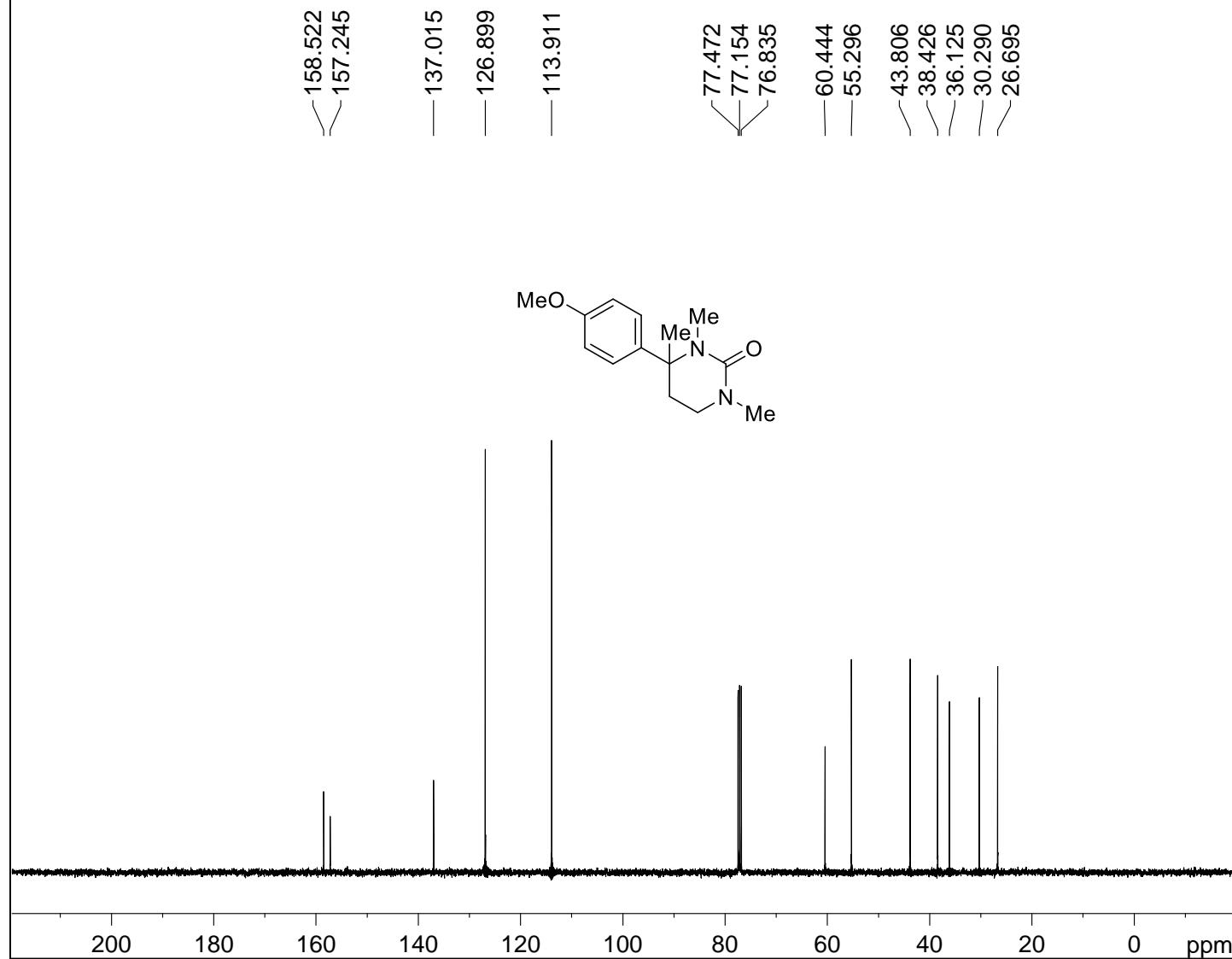


Figure 23 ¹³C NMR spectrum of compound 13a

lab sbpd-706
iitm_C13DEPT135 CDCl₃ /opt/topspin nmr 13

Current Data Parameters
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EXPNO 353
PROCNO 1

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PULPROG deptsp135
TD 32768
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DS 4
SWH 20161.291 Hz
FIDRES 0.615274 Hz
AQ 0.8126464 sec
RG 200.34
DW 24.800 usec
DE 6.50 usec
TE 297.0 K
CNST2 145.0000000
D1 1.0000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6208166 MHz
NUC1 13C
P1 9.25 usec
P13 2000.00 usec
PLW0 0 W
PLW1 47.0000000 W
SPNAM[5] Crp60comp.4
SPOALS 0.500
SPOFFS5 0 Hz
SPW5 6.14429998 W

===== CHANNEL f2 =====
SFO2 400.1312797 MHz
NUC2 1H
CPDPRG[2] waltz16
P3 15.70 usec
P4 31.40 usec
PCPD2 90.00 usec
PLW2 7.75000000 W
PLW12 0.23583999 W

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

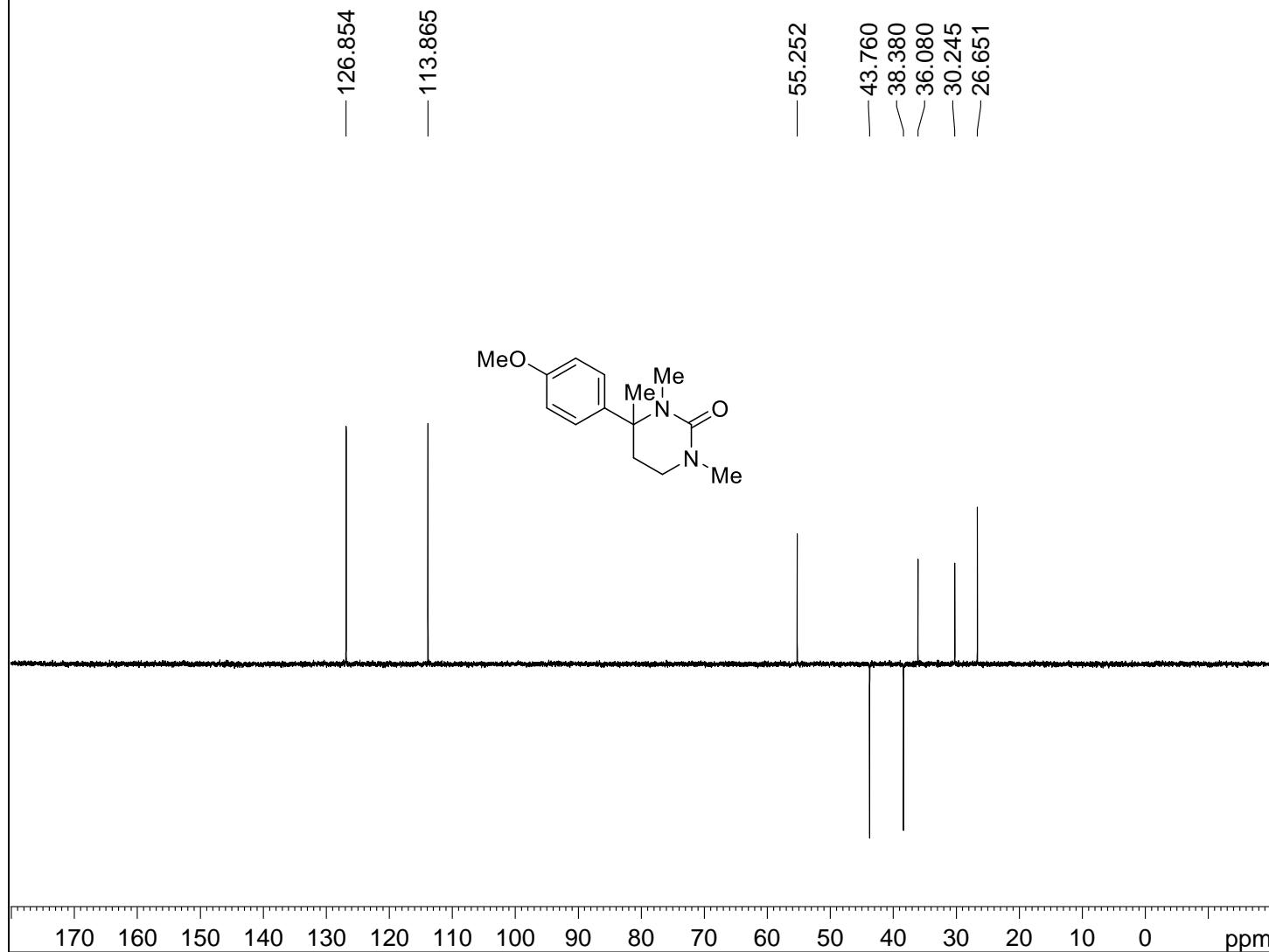
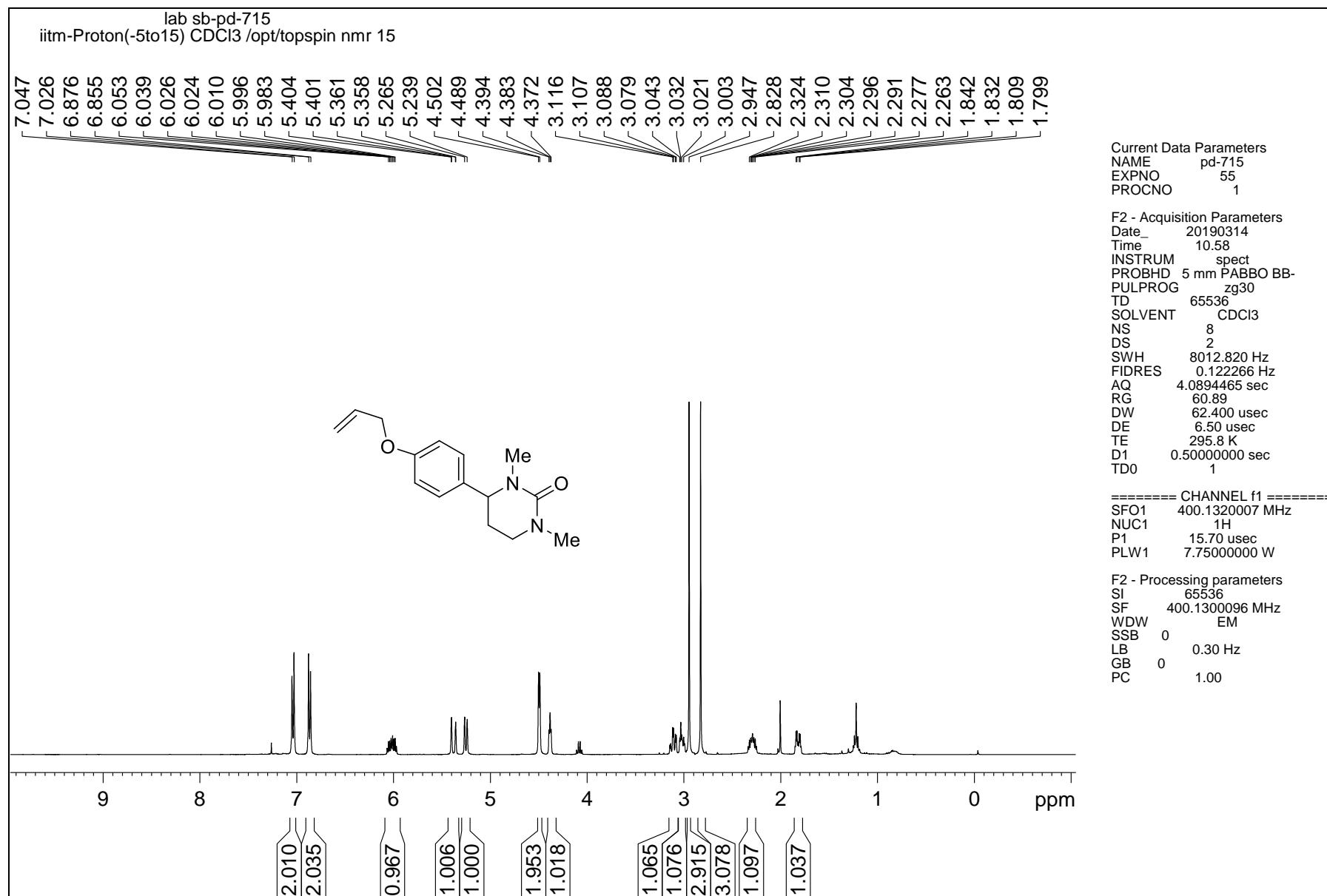


Figure 24 DEPT-135 NMR spectrum of compound 13a



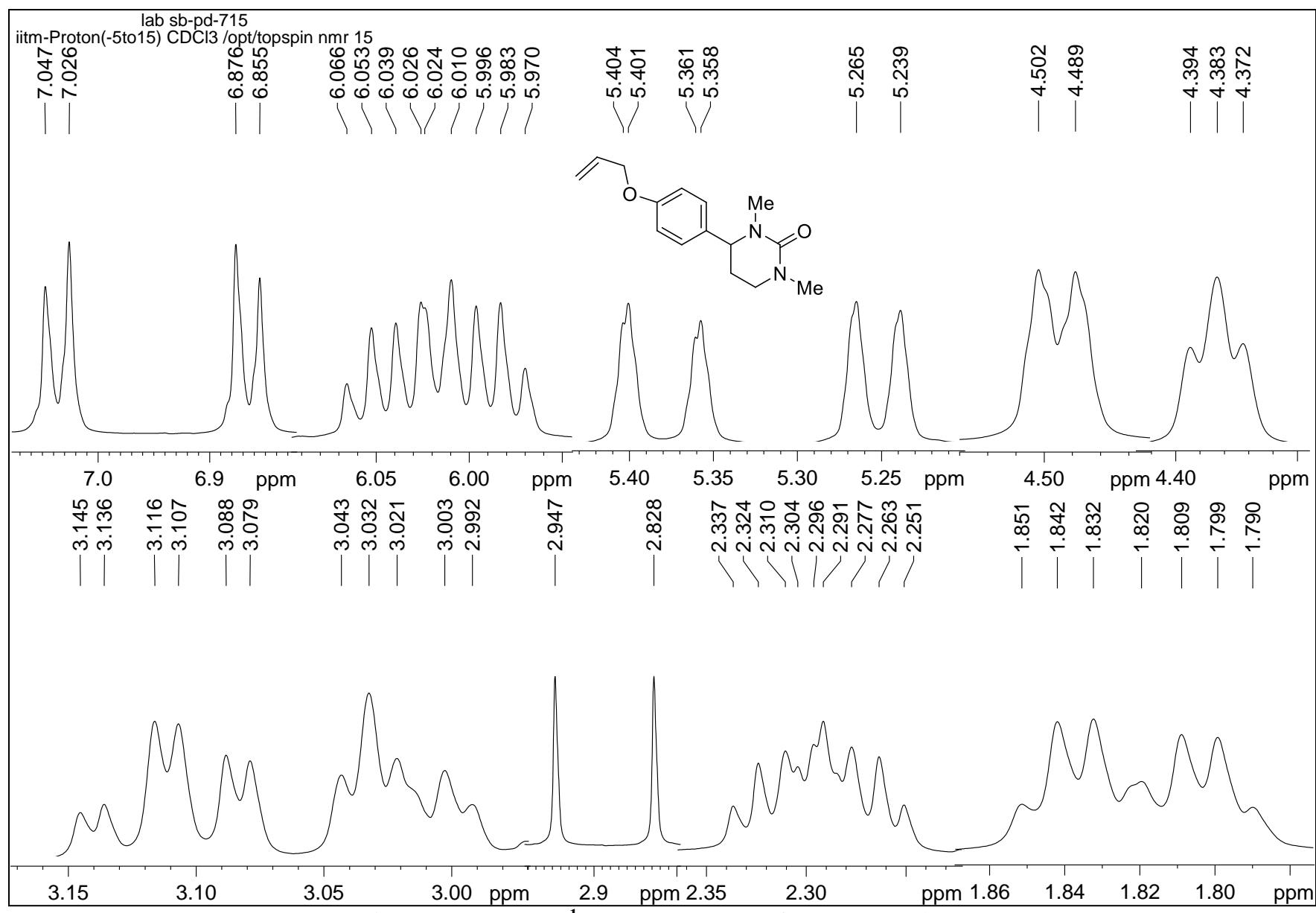


Figure 26 Expanded ^1H NMR spectrum of compound 14a

lab sb-pd-715
itm_carbonshort CDCl₃ /opt/topspin nmr 15

157.982
156.900
133.693
133.219
127.284
117.749
114.893

77.475
77.157
76.838
68.881
60.739

43.993
35.895
34.791
29.985

Current Data Parameters
NAME pd-715
EXPNO 56
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190314
Time 11.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 16540
SOLVENT CDCl₃
NS 128
DS 4
SWH 24038.461 Hz
FIDRES 1.453353 Hz
AQ 0.3440320 sec
RG 200.34
DW 20.800 usec
DE 6.50 usec
TE 296.2 K
D1 1.0000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228289 MHz
NUC1 13C
P1 9.25 usec
PLW1 47.0000000 W

===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 7.7500000 W
PLW12 0.23583999 W
PLW13 0.11863000 W

F2 - Processing parameters
SI 32768
SF 100.6127652 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

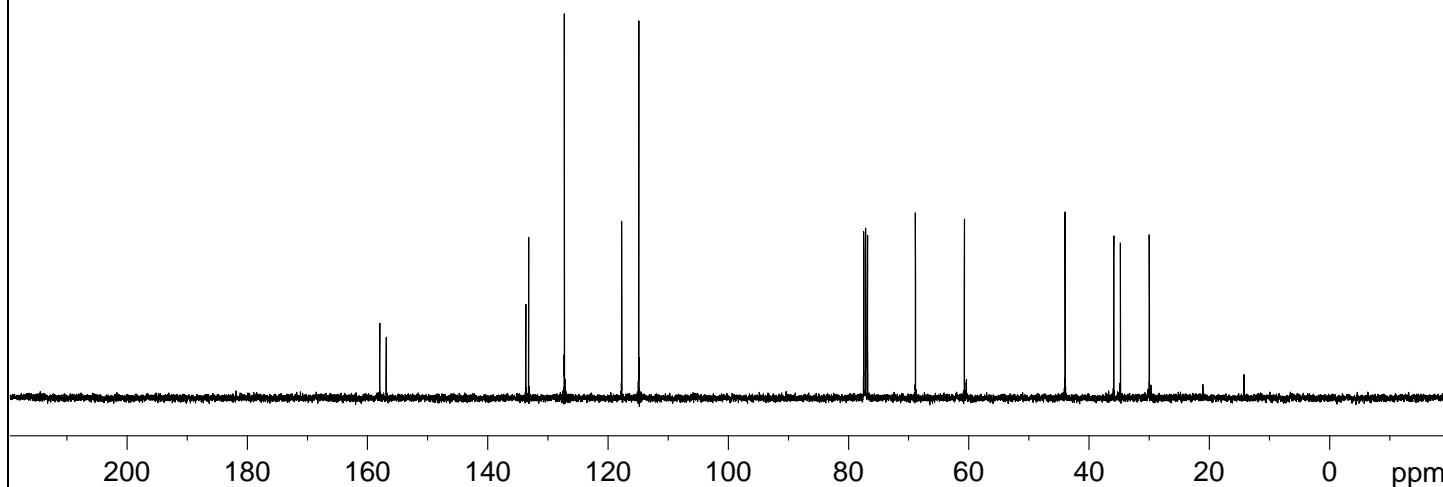
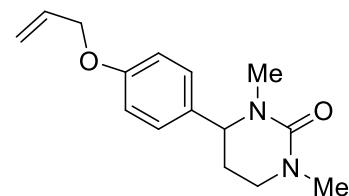


Figure 27 ¹³C NMR spectrum of compound 14a

lab sbPD-715
itm_C13DEPT135 CDCl₃ /opt/topspin nmr 13

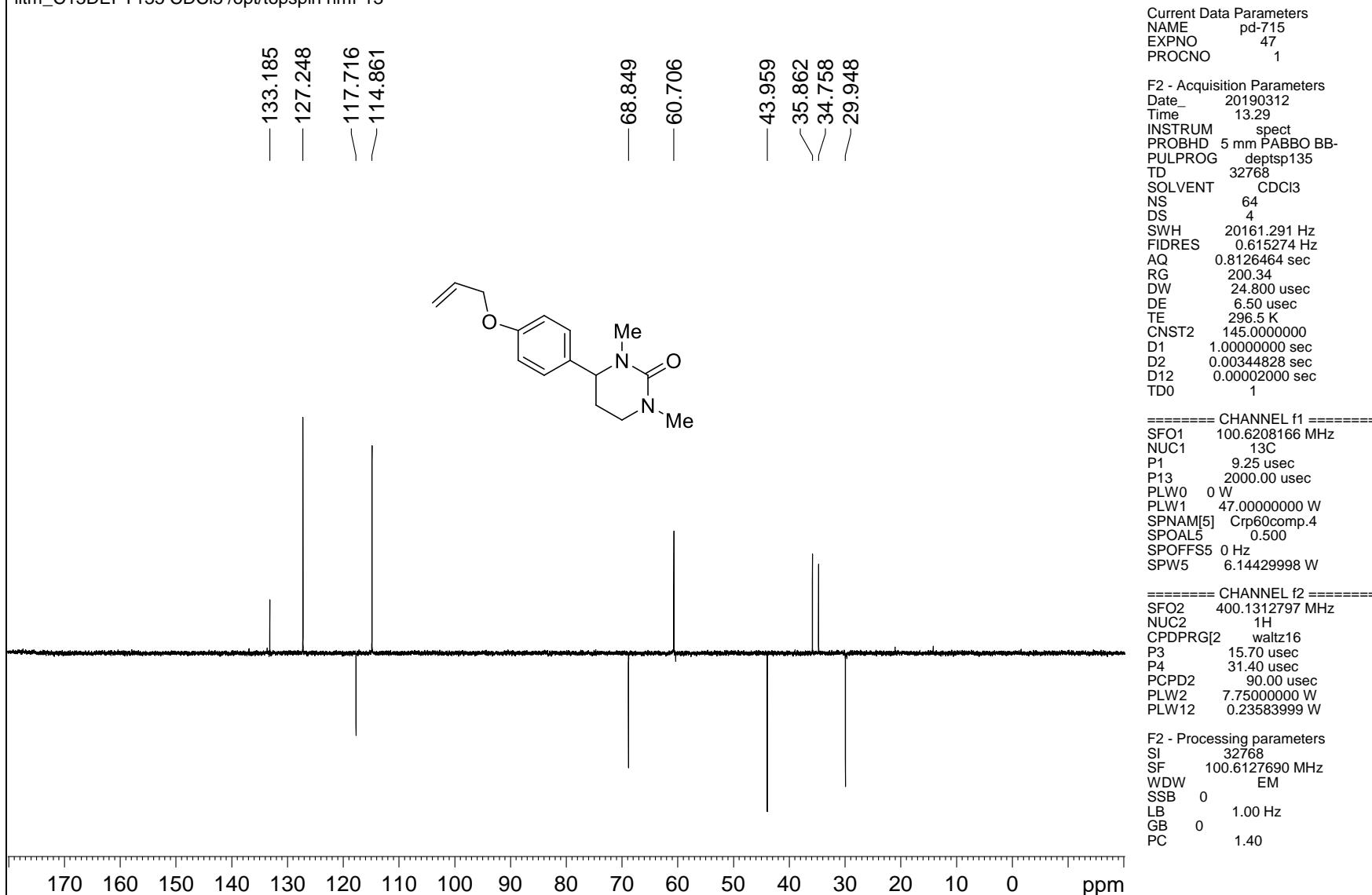


Figure 28 DEPT-135 NMR spectrum of compound 14a

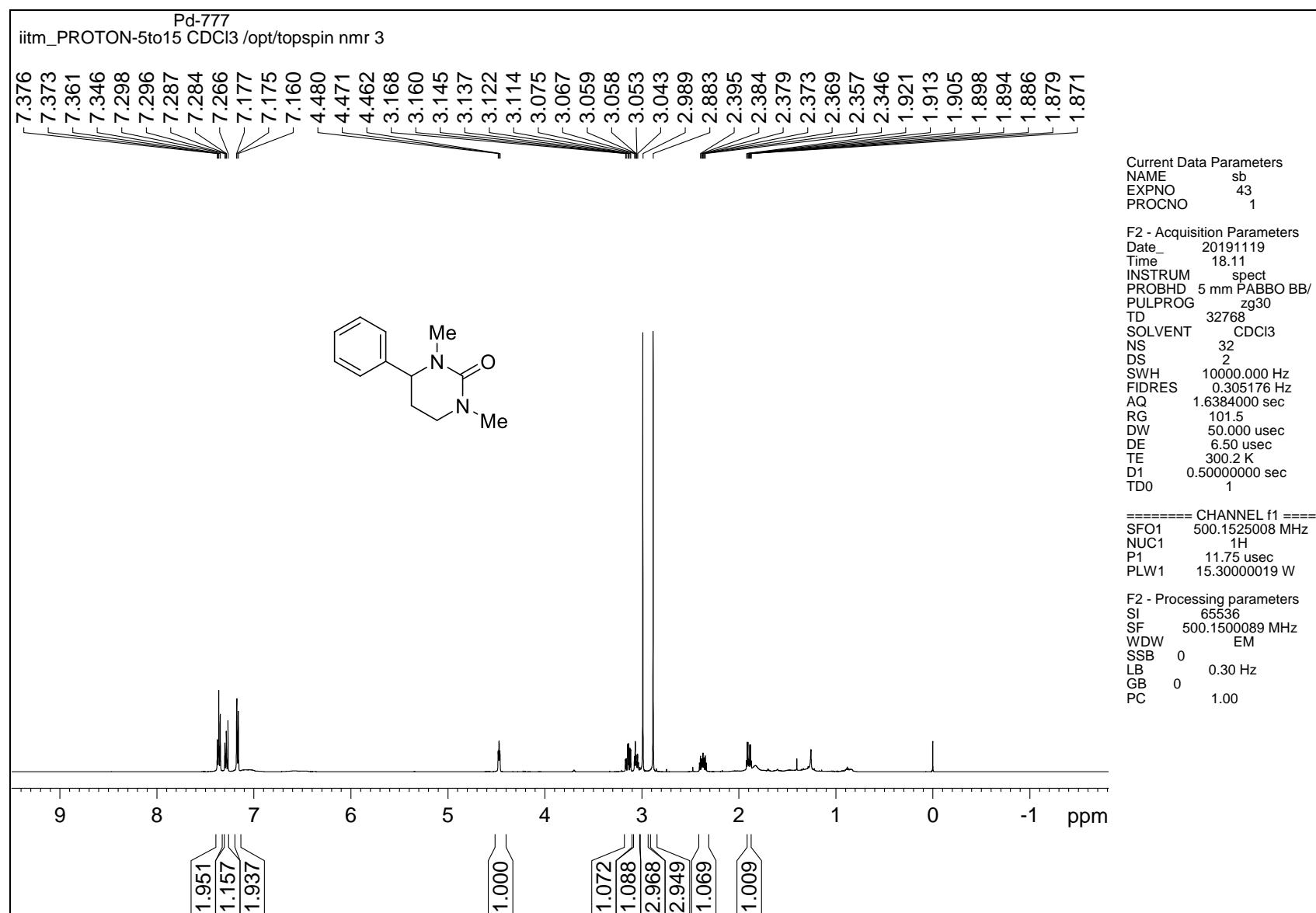


Figure 29 ^1H NMR spectrum of THPM from styrene (reference 26)

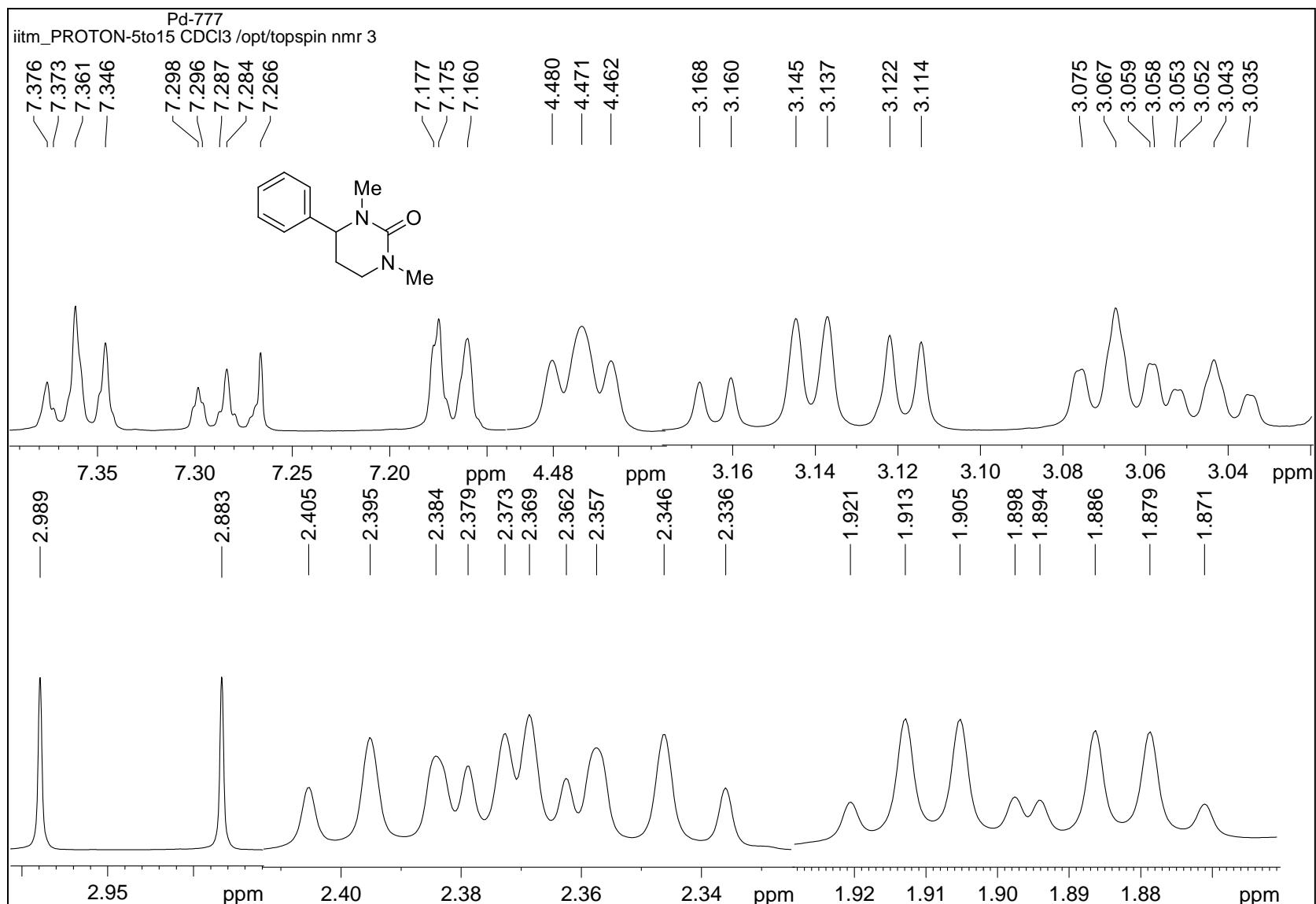


Figure 30 Expanded ^1H NMR spectrum of THPM from styrene (reference 26)

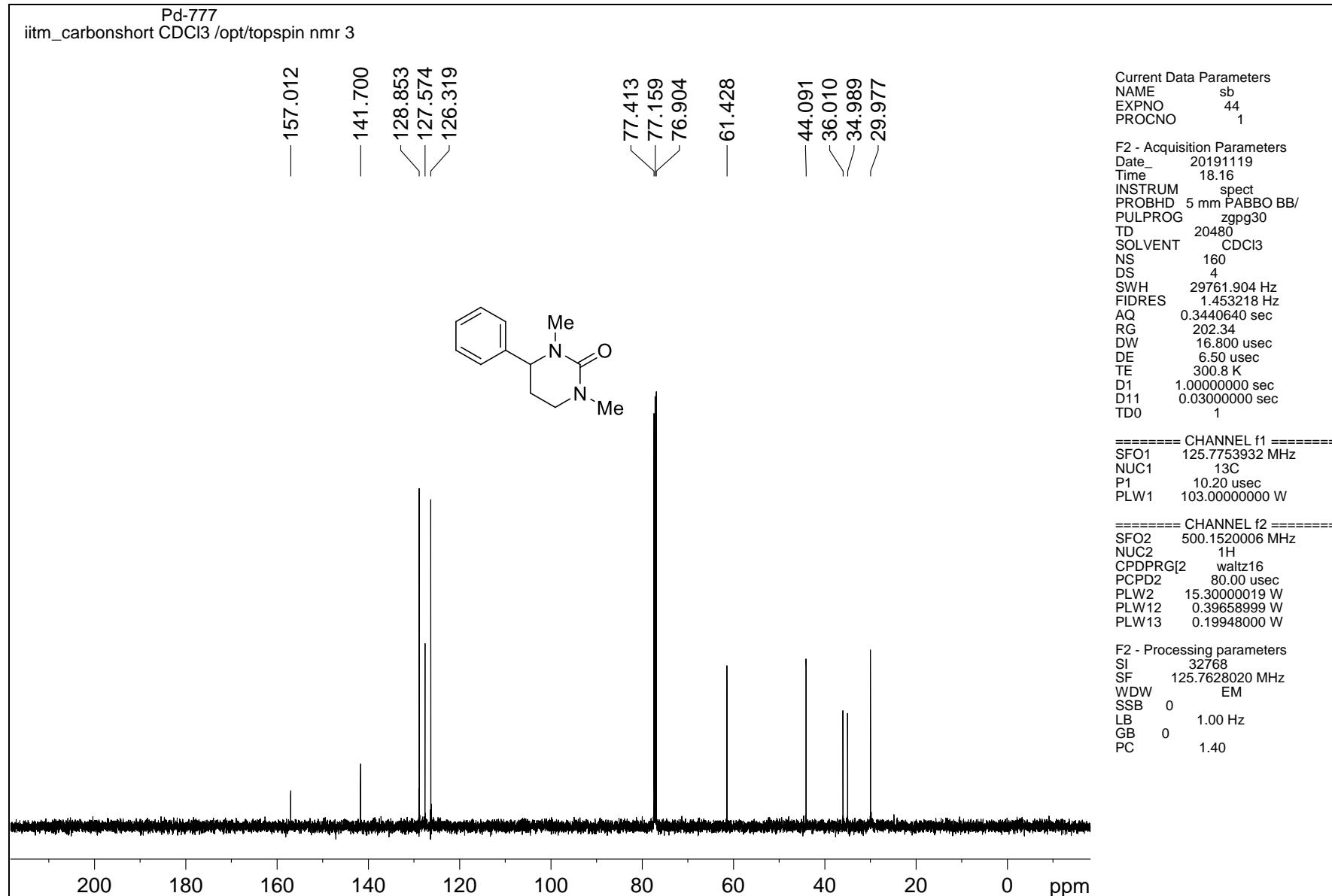


Figure 31 ¹³C NMR spectrum of THPM from styrene (reference 26)

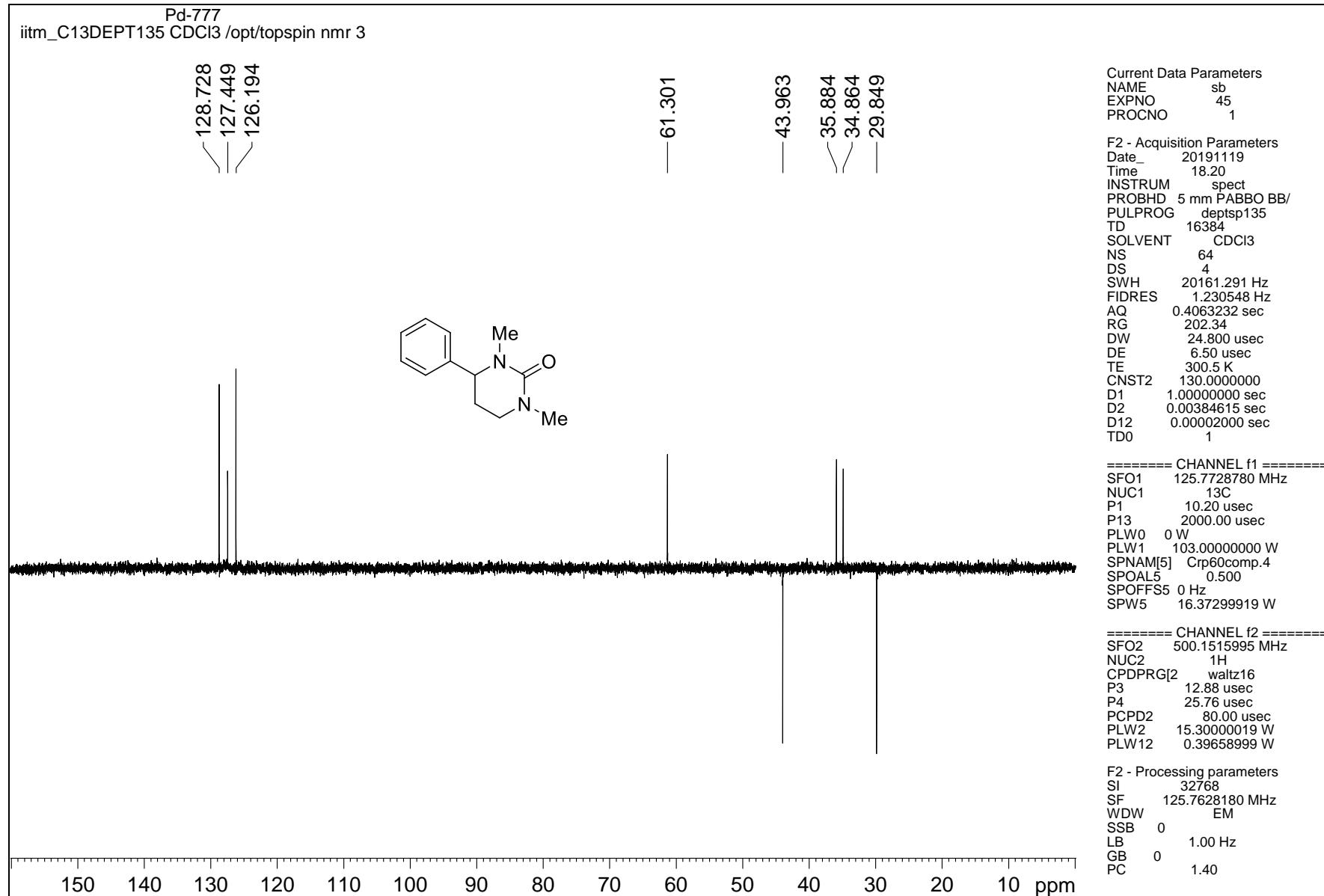
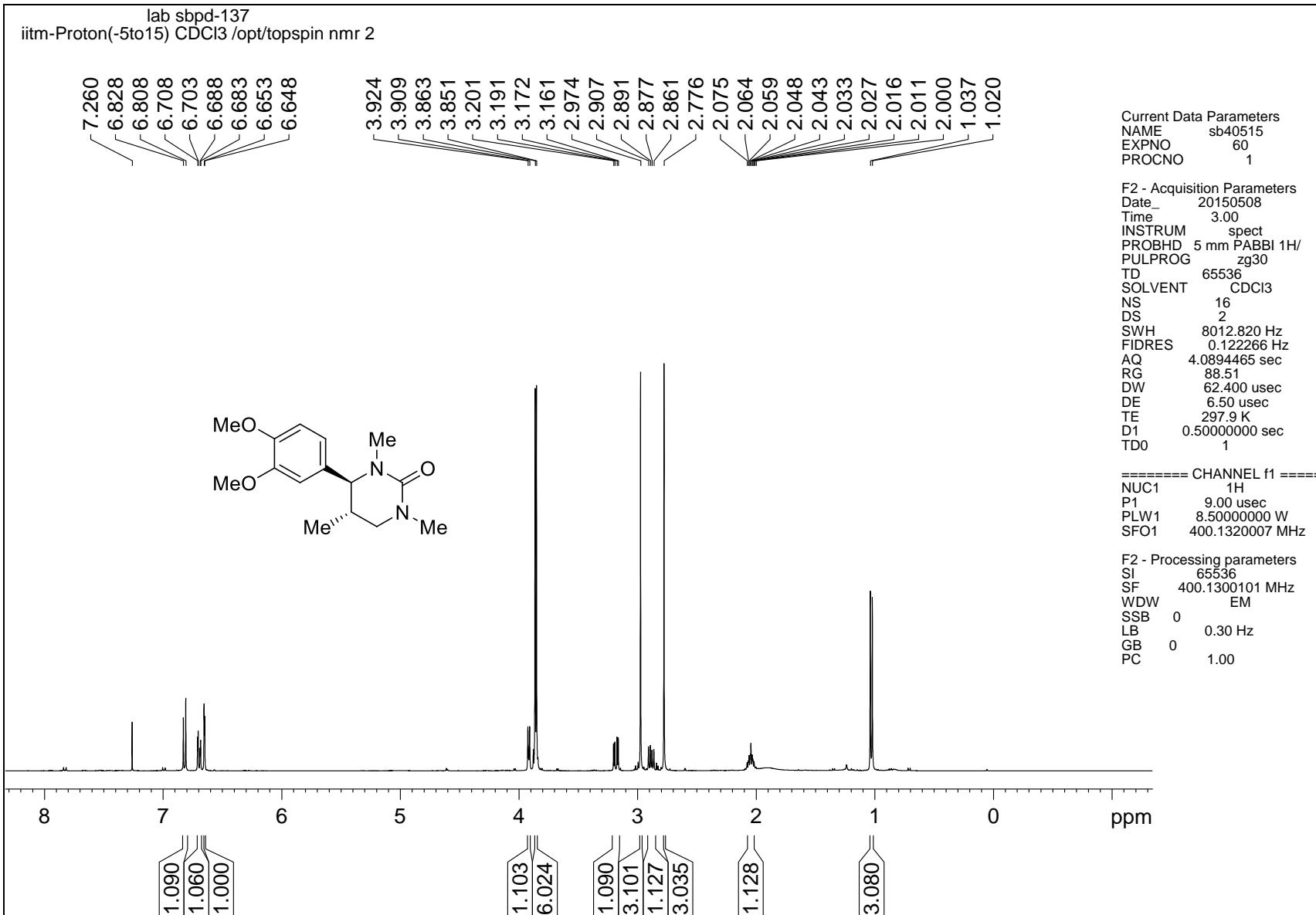


Figure 32 DEPT-135 NMR spectrum of THPM from styrene (reference 26)



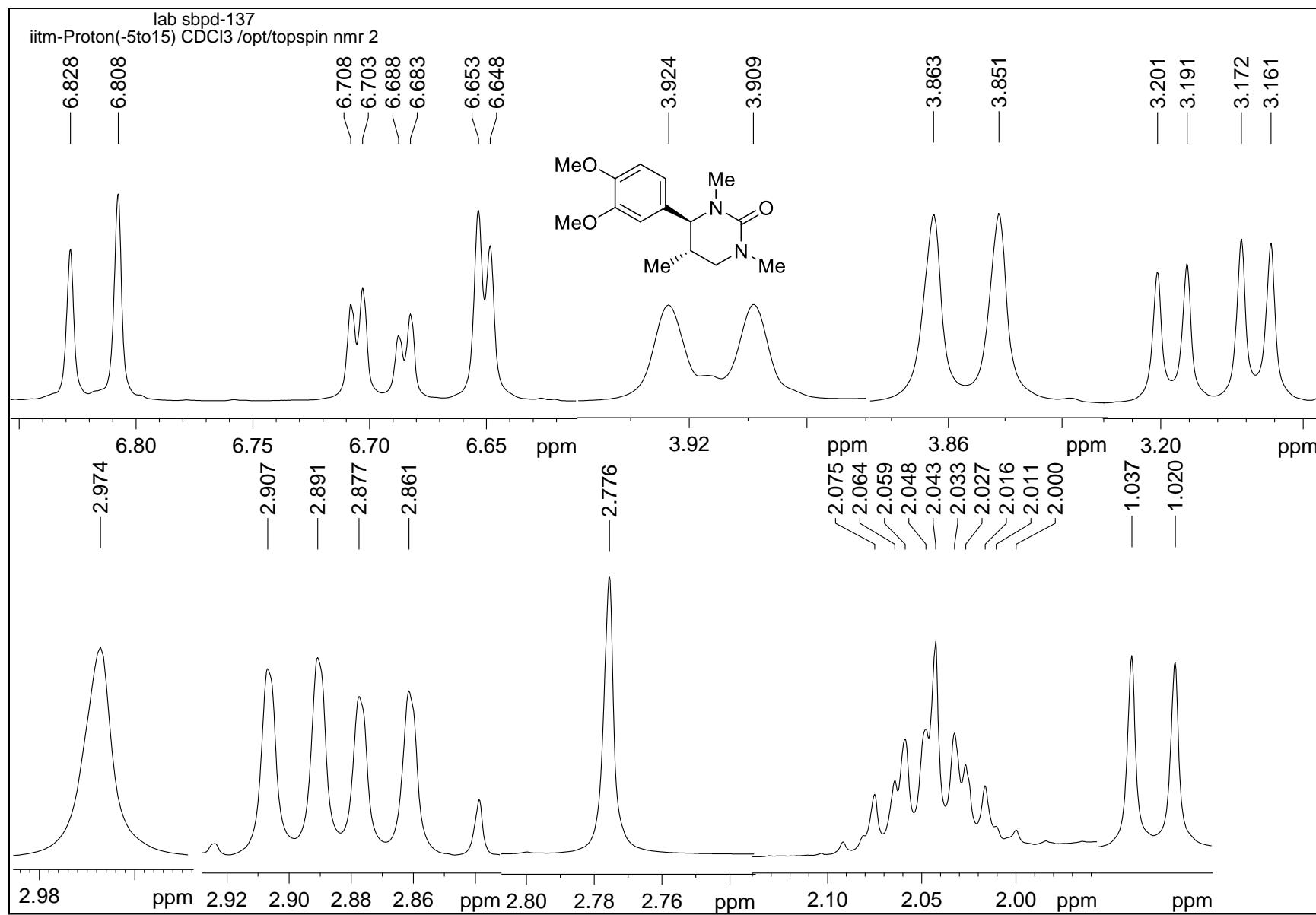


Figure 34 Expanded ¹H NMR spectrum of compound 15a

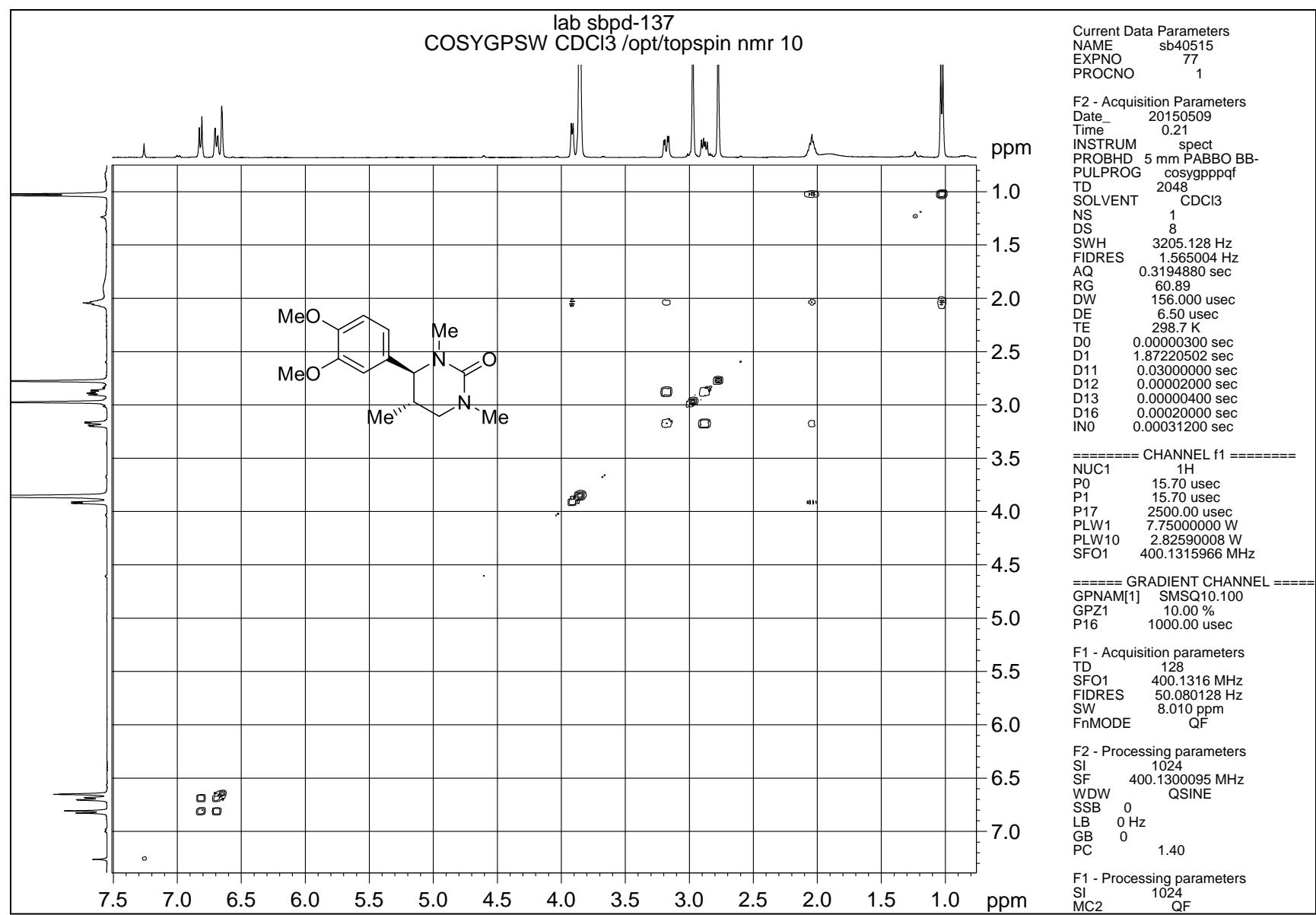


Figure 35 ¹H-¹H COSY NMR spectrum of compound 15a

lab sbpd-137
iitm_carbonshort CDCl₃ /opt/topspin nmr 2

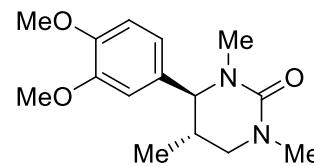
— 157.039
V 149.482
V 148.567

— 134.159

— 119.148
V 111.121
V 109.346

77.474
V 77.156
V 76.838
— 68.438
V 56.088
V 56.028
V 51.437
V 36.189
V 34.801
V 34.771

— 17.122



Current Data Parameters
NAME sb40515
EXPNO 61
PROCNO 1

F2 - Acquisition Parameters
Date 20150508
Time 3.07
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg30
TD 16540
SOLVENT CDCl₃
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 1.453353 Hz
AQ 0.3440320 sec
RG 200.34
DW 20.800 usec
DE 6.50 usec
TE 298.4 K
D1 1.0000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 15.00 usec
PLW1 76.0000000 W
SFO1 100.6228289 MHz

===== CHANNEL f2 =====
CPDPRGf2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 8.5000000 W
PLW12 0.0850000 W
PLW13 0.06885000 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127577 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

200 180 160 140 120 100 80 60 40 20 0 ppm

Figure 36 ¹³C NMR spectrum of compound 15a

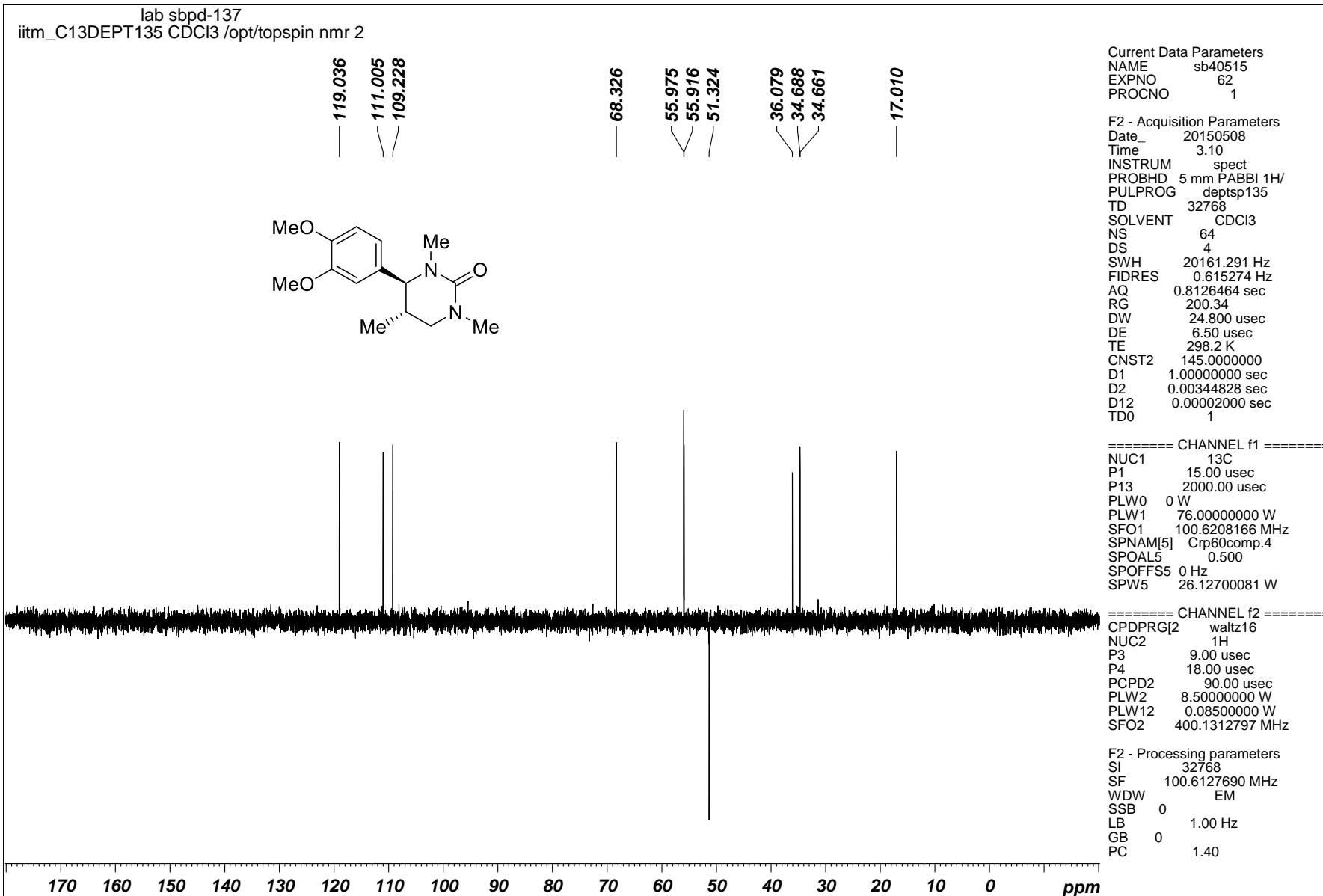


Figure 37 DEPT-135 NMR spectrum of compound 15a

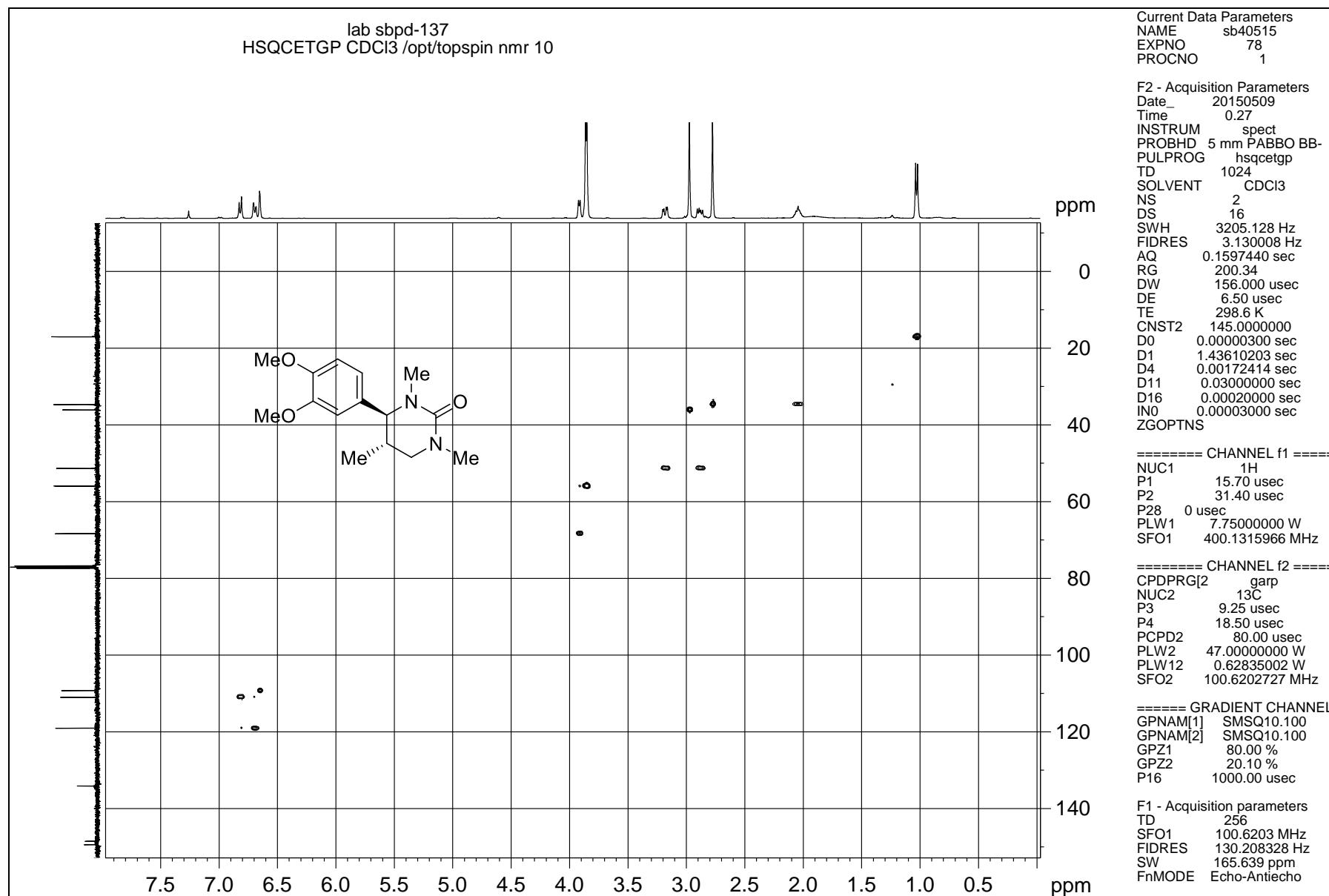
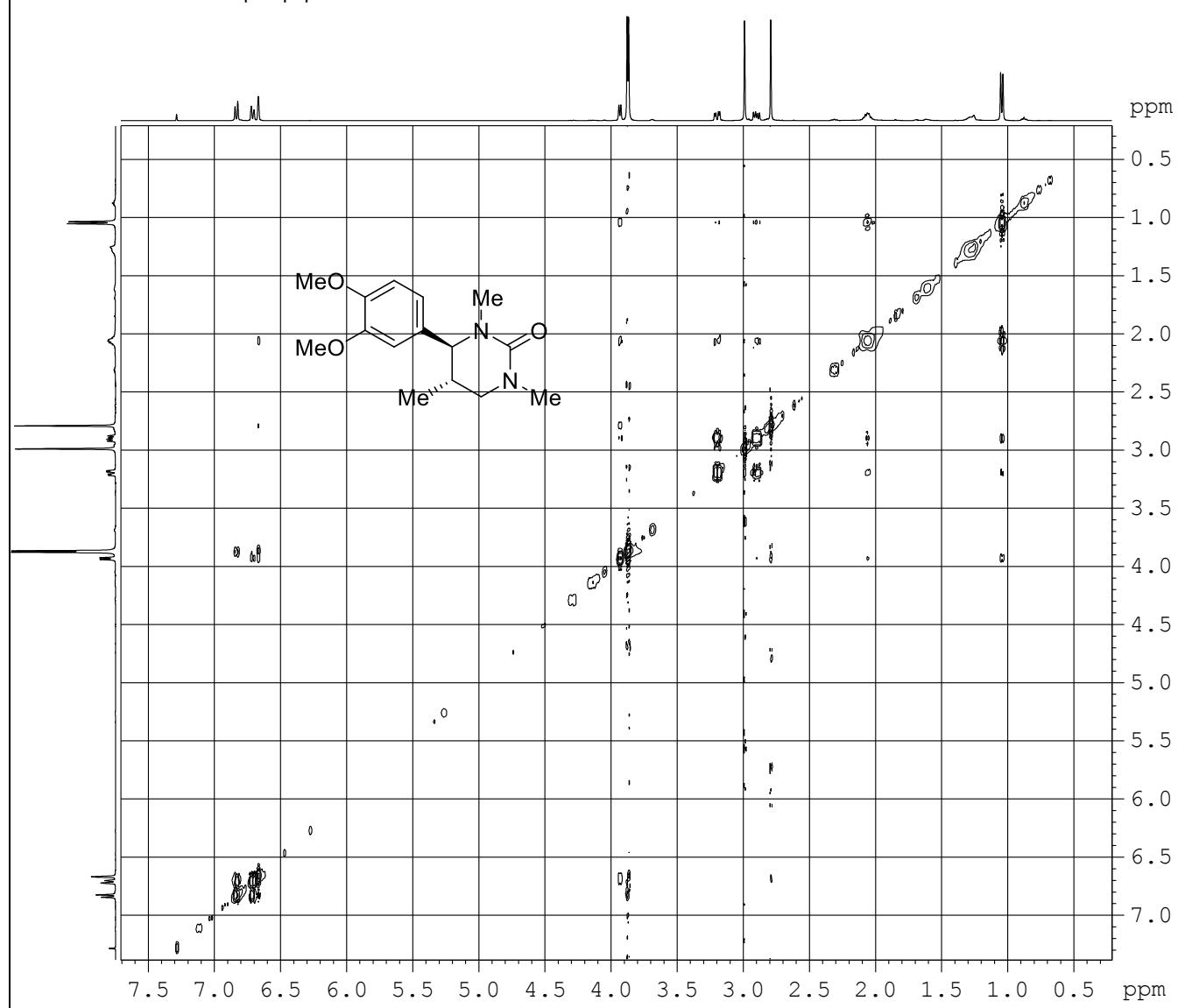


Figure 38 ¹H-¹³C HSQC NMR spectrum of compound 15a

lab sbPd-137
NOESYPHSW CDCl₃ /opt/topspin nmr 15



Current Data Parameters
 NAME pd
 EXPNO 209
 PROCNO 1

F2 - Acquisition Parameters
 Date 20200323
 Time 11.23
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG noesygpphp
 TD 2048
 SOLVENT CDCl₃
 NS 4
 DS 32
 SWH 2873.563 Hz
 FIDRES 1.403107 Hz
 AQ 0.3563520 sec
 RG 60.89
 DW 174.000 used
 DE 6.50 used
 TE 297.3 K
 D0 0.00015401 sec
 D1 1.89964795 sec
 D8 0.30000001 sec
 D11 0.03000000 sec
 D12 0.00002000 sec
 D16 0.00002000 sec
 IN0 0.00034800 sec

===== CHANNEL f1 =====
 SFO1 400.1315182 MHz
 NUC1 1H
 P1 15.70 used
 P2 31.40 used
 P17 2500.00 used
 PLW1 7.7500000 W
 PLW10 2.1226008 W

===== GRADIENT CHANNEL =====
 GPNAME[1] SMSQ10.100
 GPZ1 40.00 %
 P16 1000.00 used

F1 - Acquisition parameters
 TD 256
 SFO1 400.1315 MHz
 FIDRES 22.449713 Hz
 SW 7.182 ppm
 FnMODE States-TPPI

F2 - Processing parameters
 SI 1024
 SF 400.1300000 MHz
 WDW QSINE
 SSB 2
 LB 0 Hz
 GB 0
 PC 1.00

F1 - Processing parameters
 SI 1024
 MC2 States-TPPI
 SF 400.1300000 MHz

Figure 39 ¹H-¹H 2D-NOESY spectrum of compound 15a

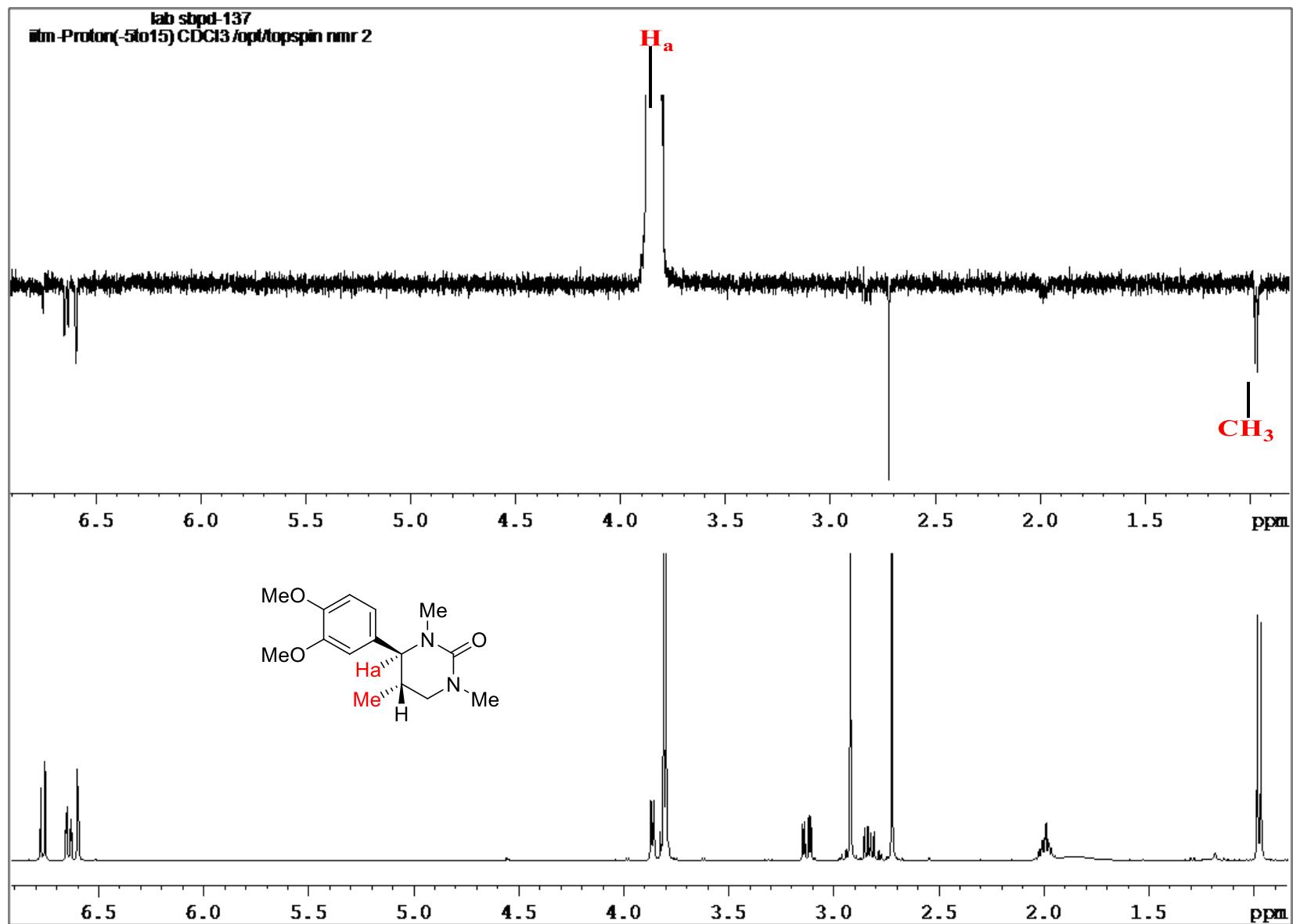


Figure 40 ¹H-¹H 1D-NOE NMR spectrum of compound 15a

lab sbpd-137
itm-Proton(-5to15) CDCl₃ /opt/topspin nmr 2

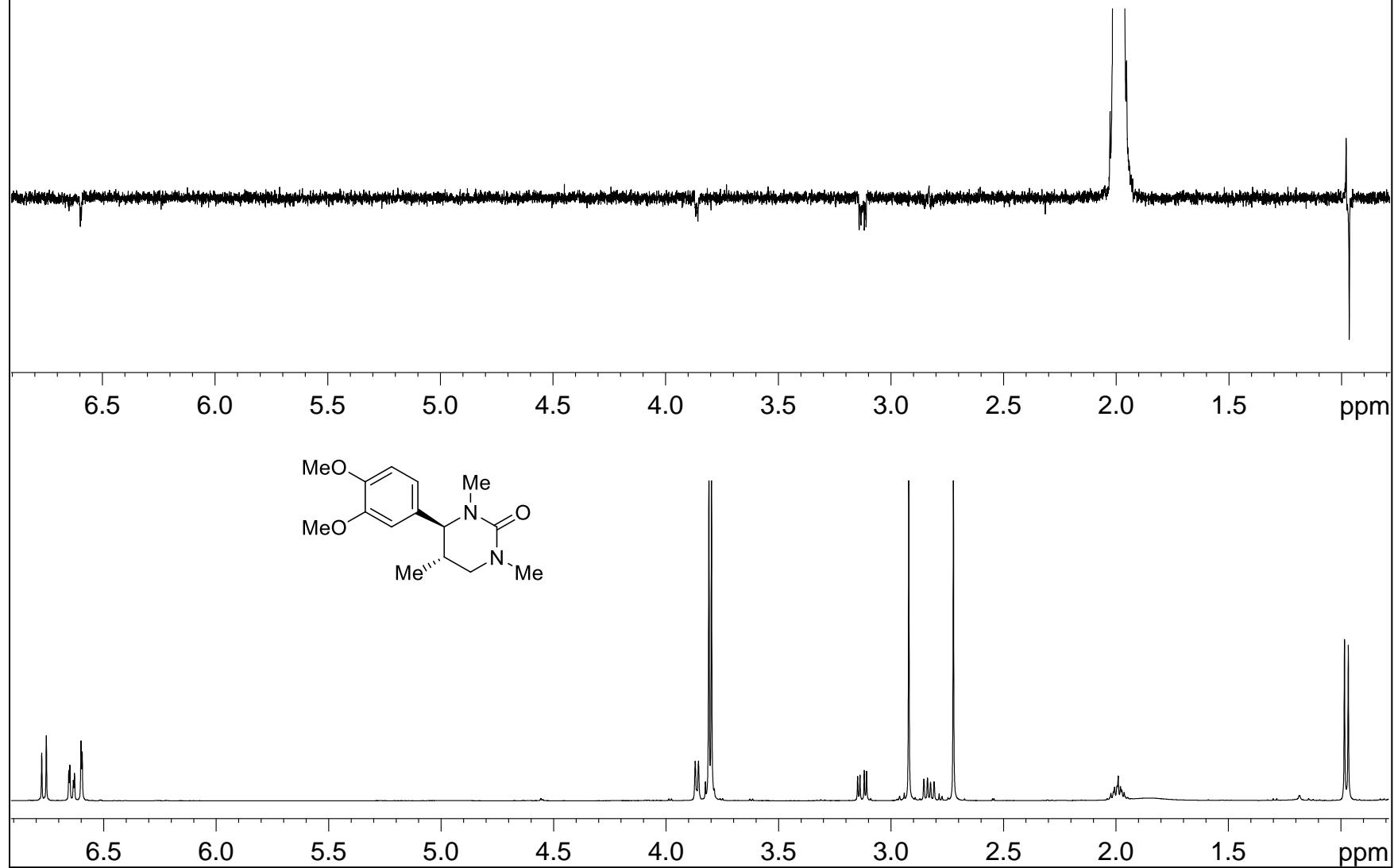
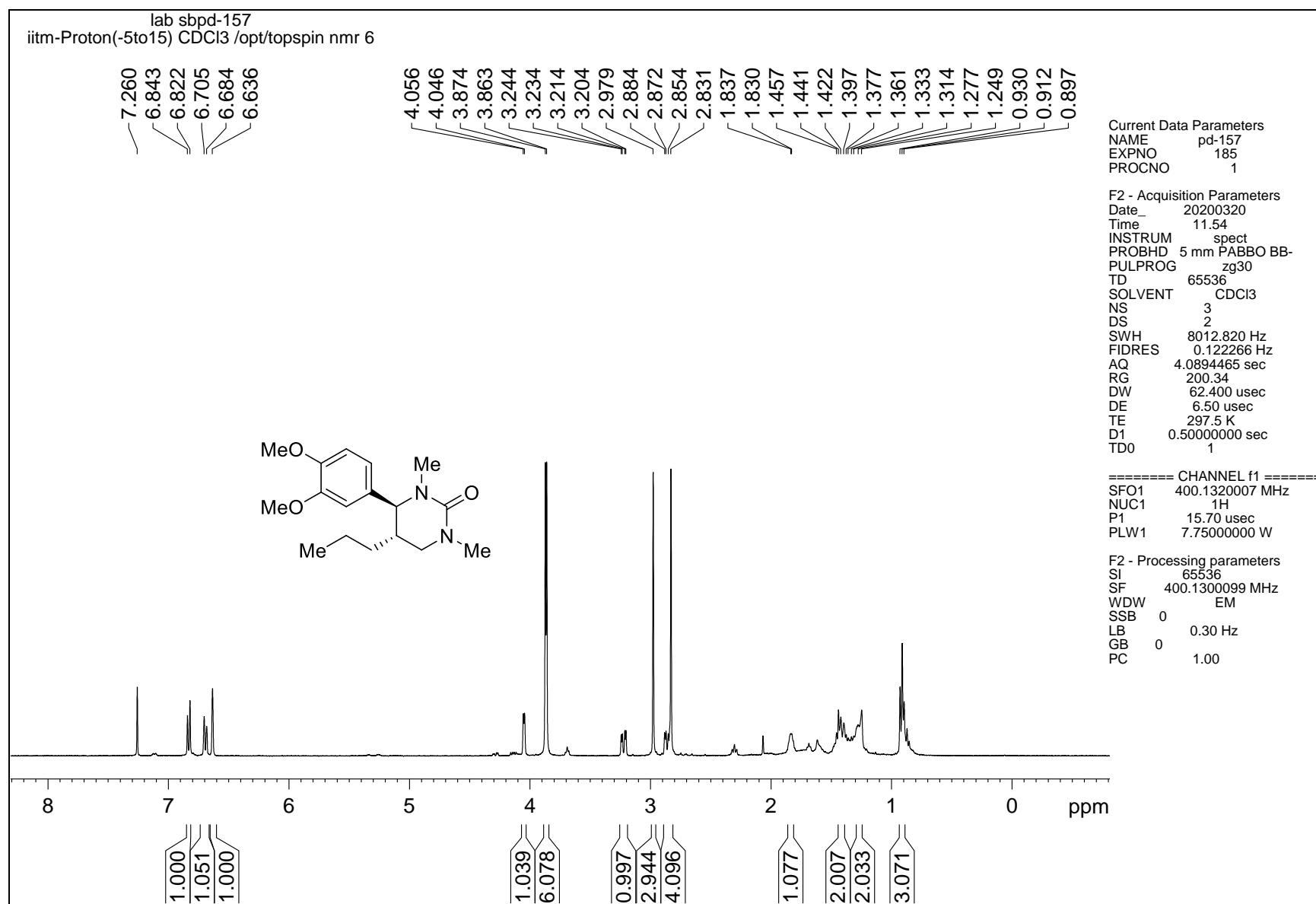


Figure 41 ¹H-¹H 1D-NOE NMR spectrum of compound 15a



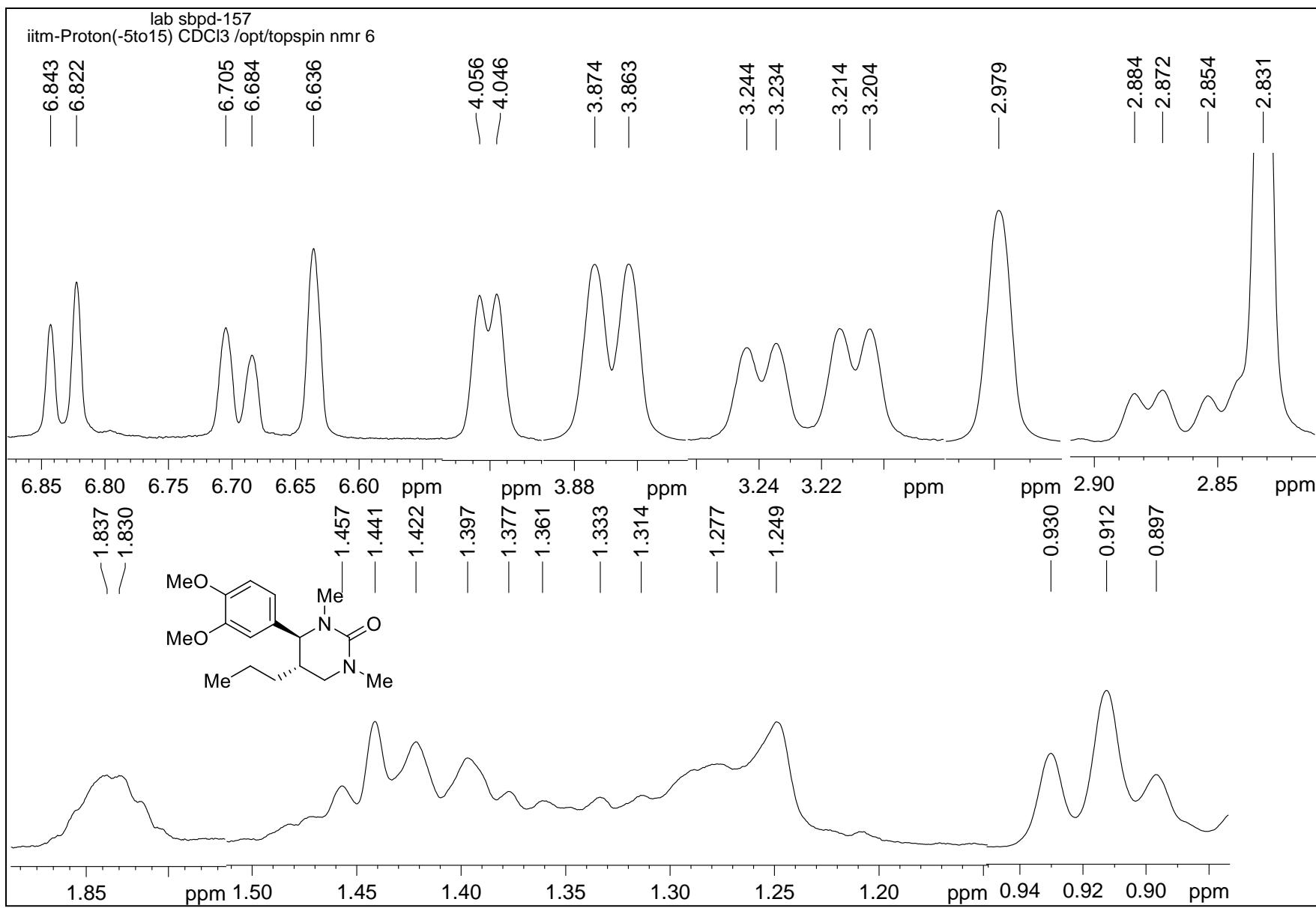
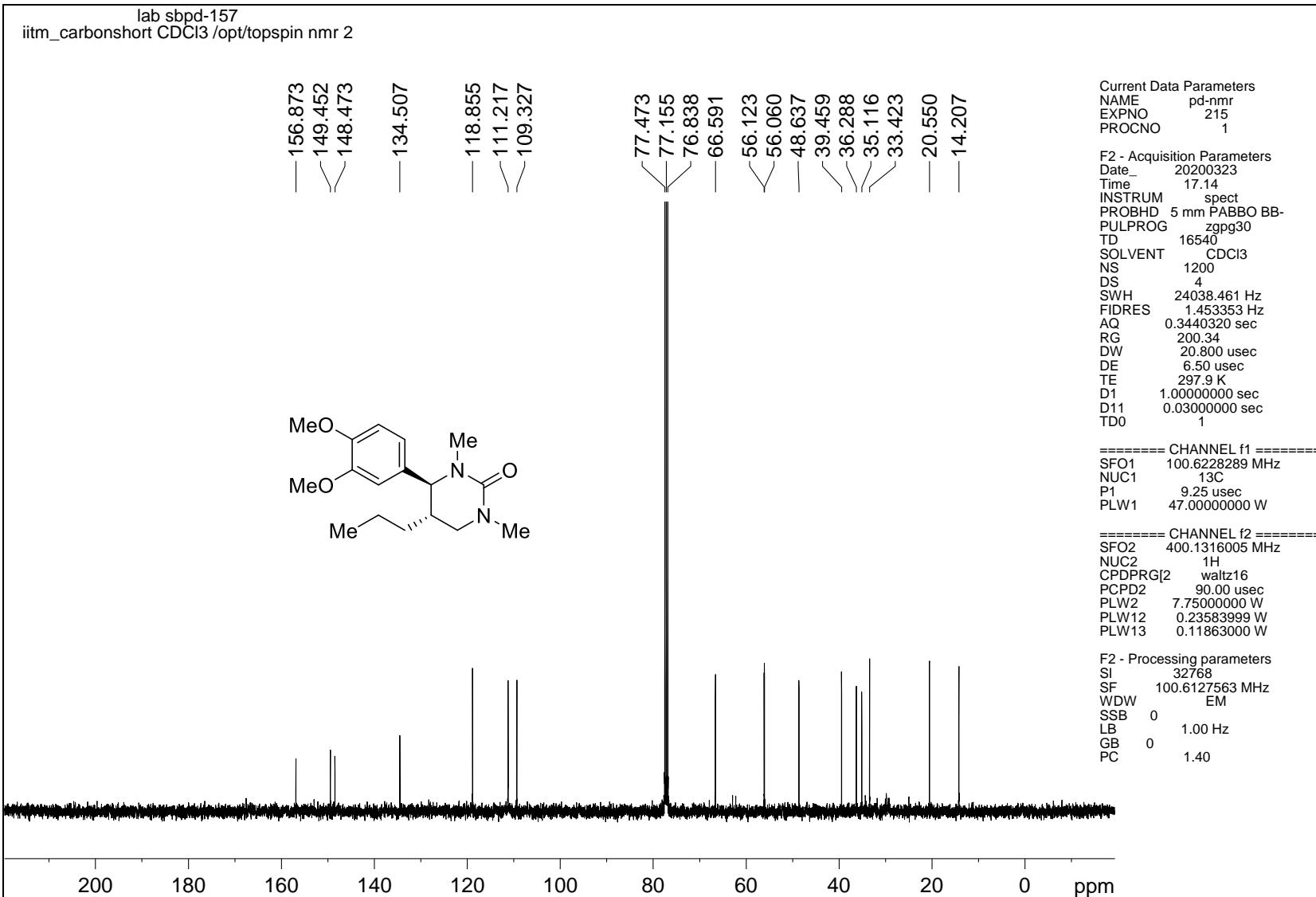


Figure 43 Expanded ¹H NMR spectrum of compound 16a



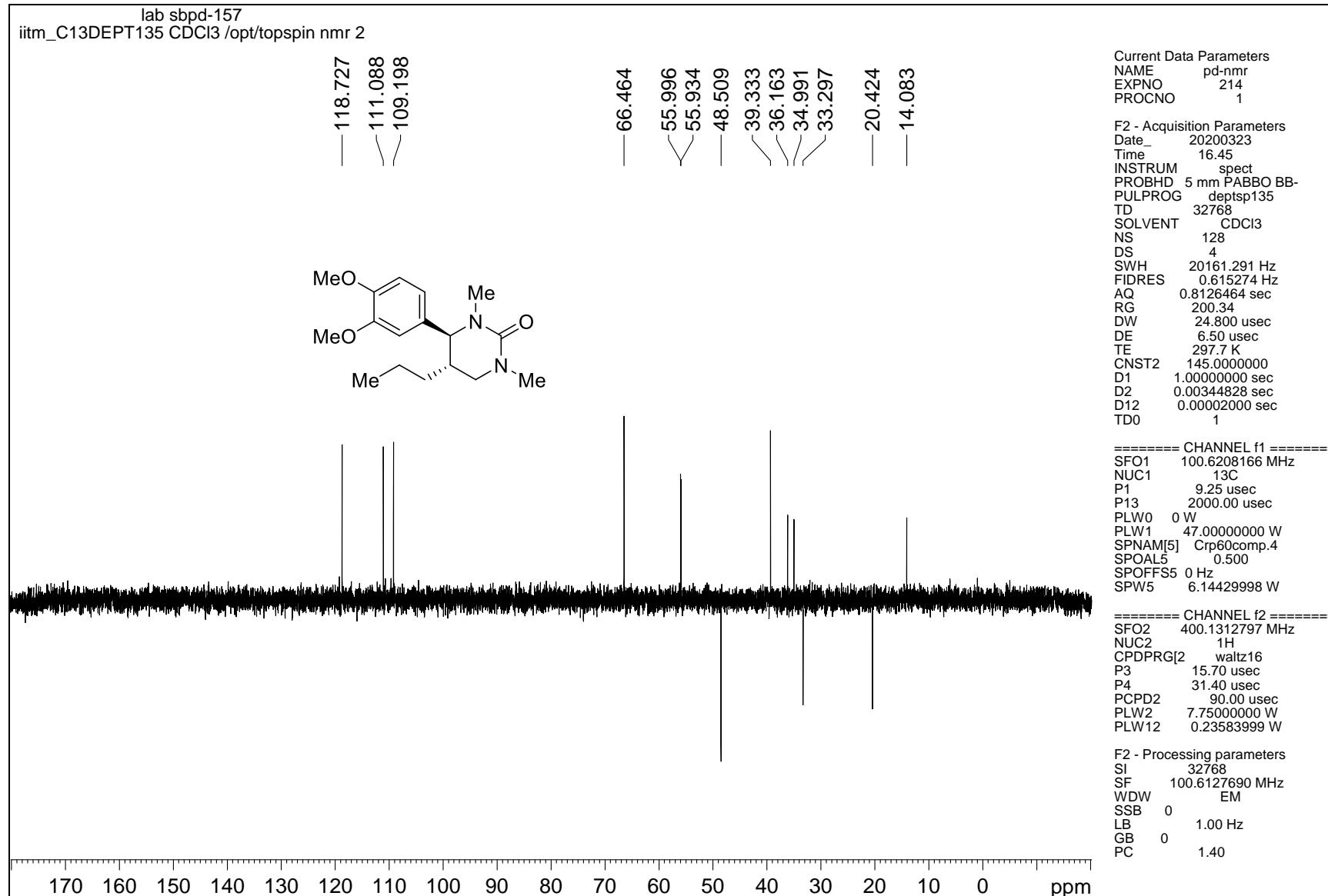
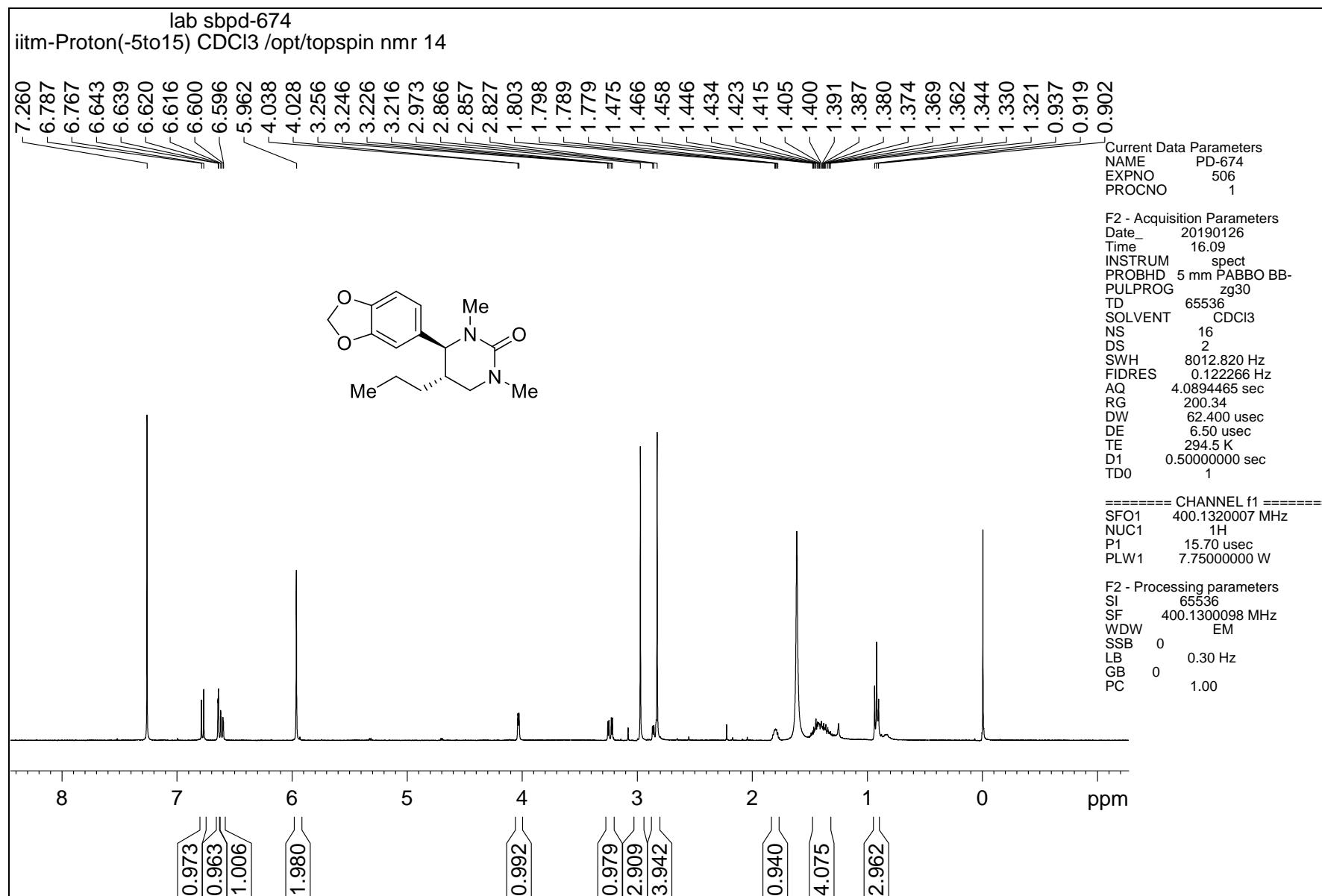


Figure 45 DEPT-135 NMR spectrum of compound 16a



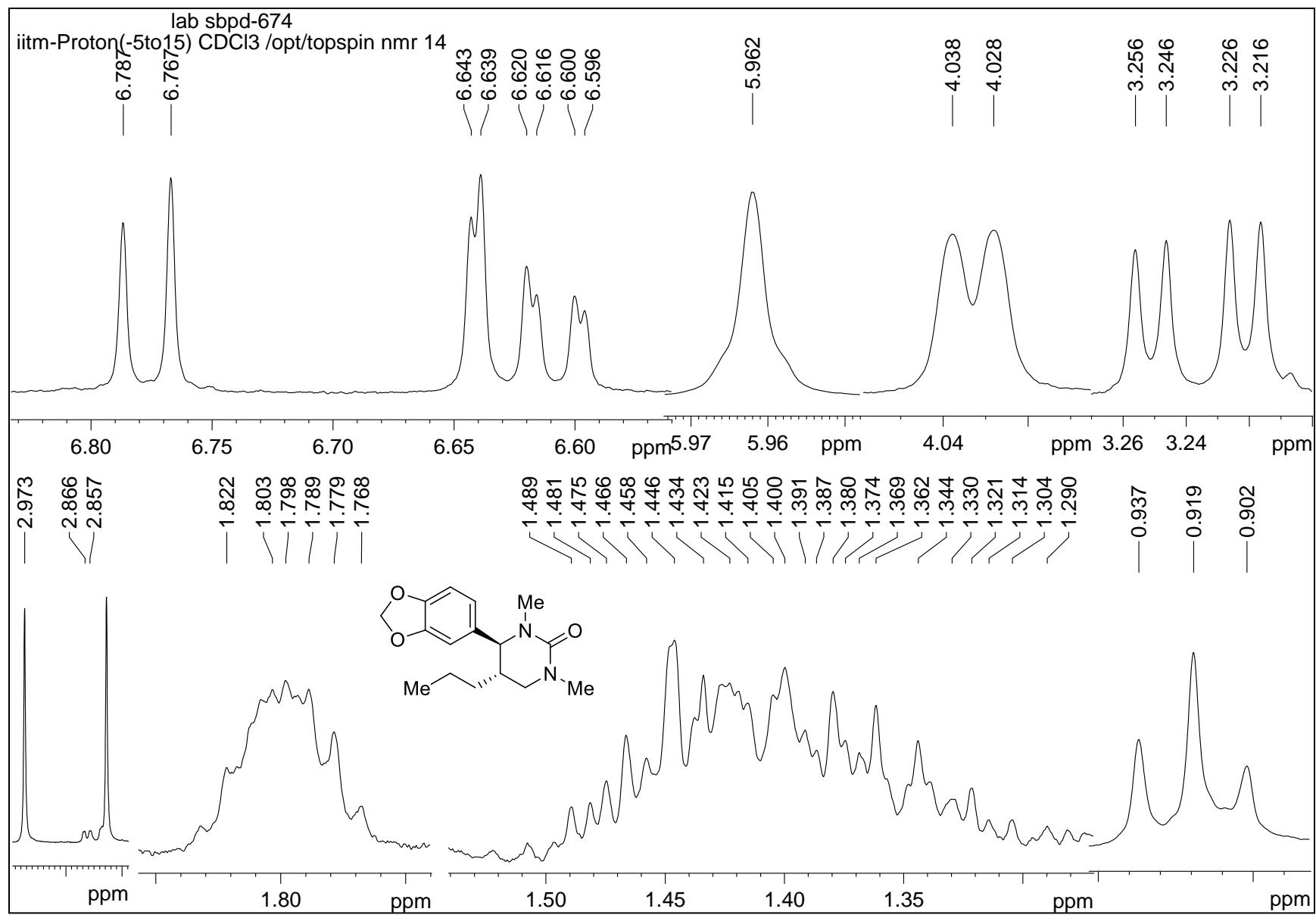


Figure 47 Expanded ¹H NMR spectrum of compound 17a

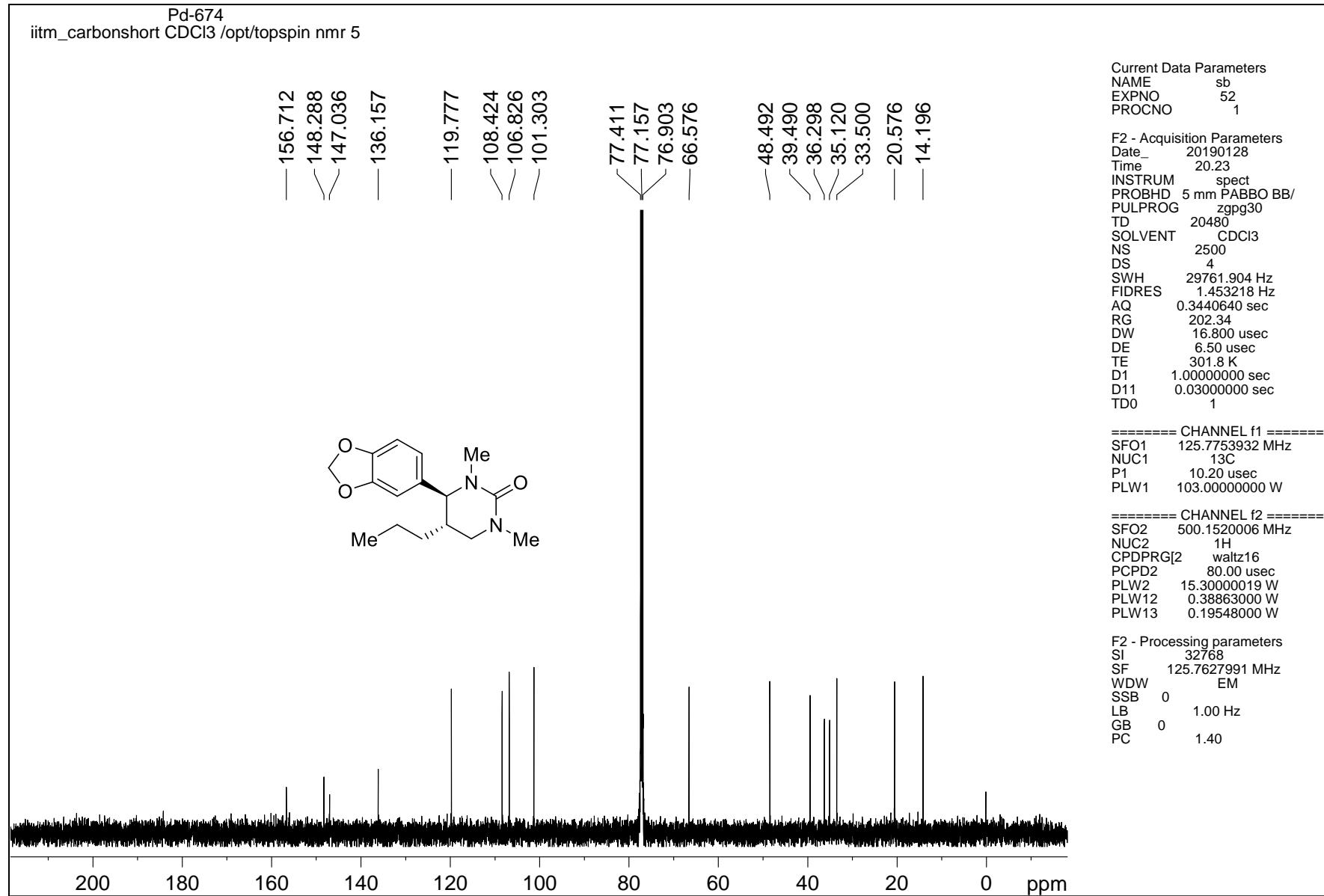


Figure 48 ¹³C NMR spectrum of compound 17a

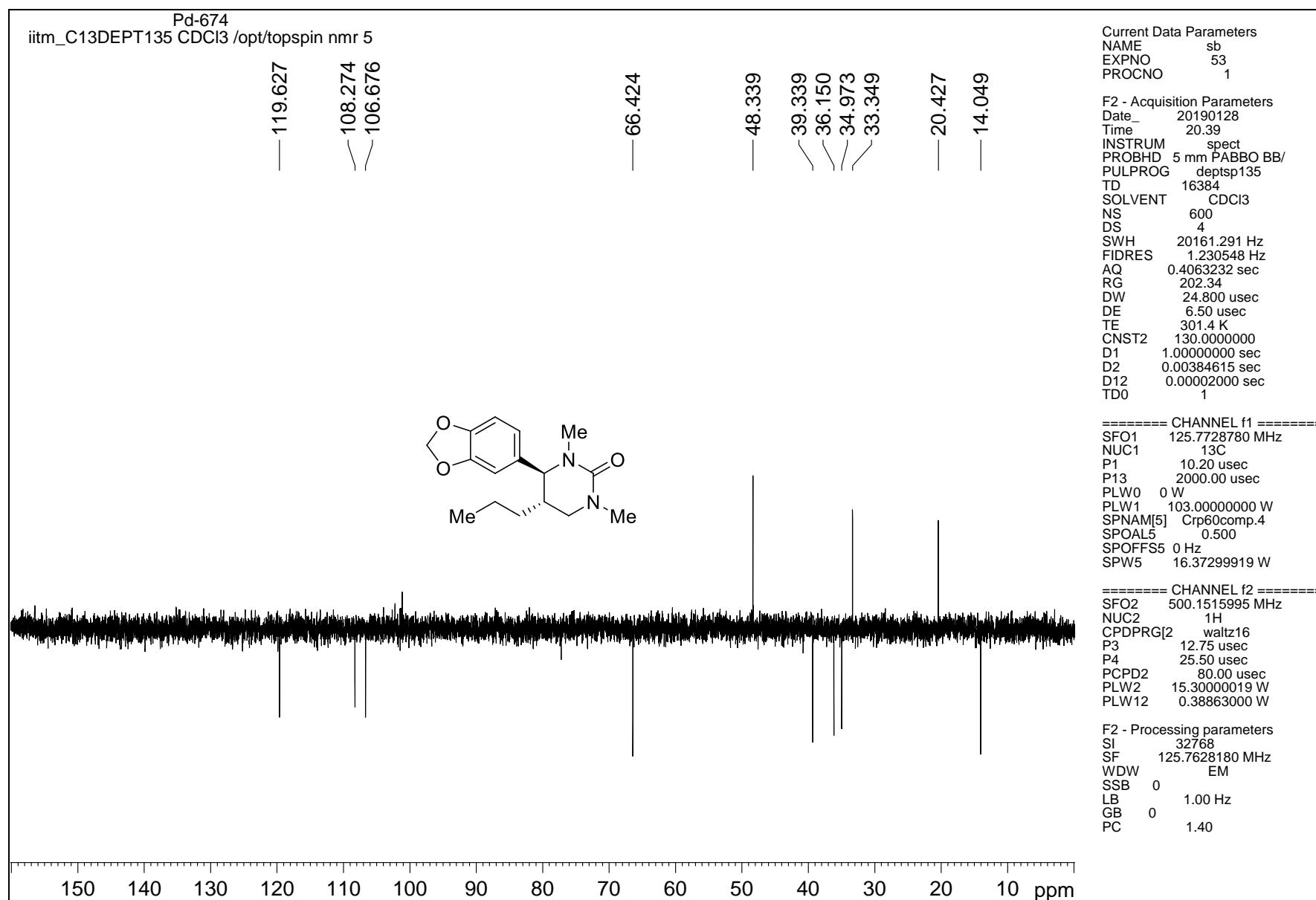


Figure 49 DEPT-135 NMR spectrum of compound 17a

lab sbpd-686
itm-Proton(-5to15) CDCl₃ /opt/topspin nmr 14

7.260
7.191
7.171
6.869
6.849

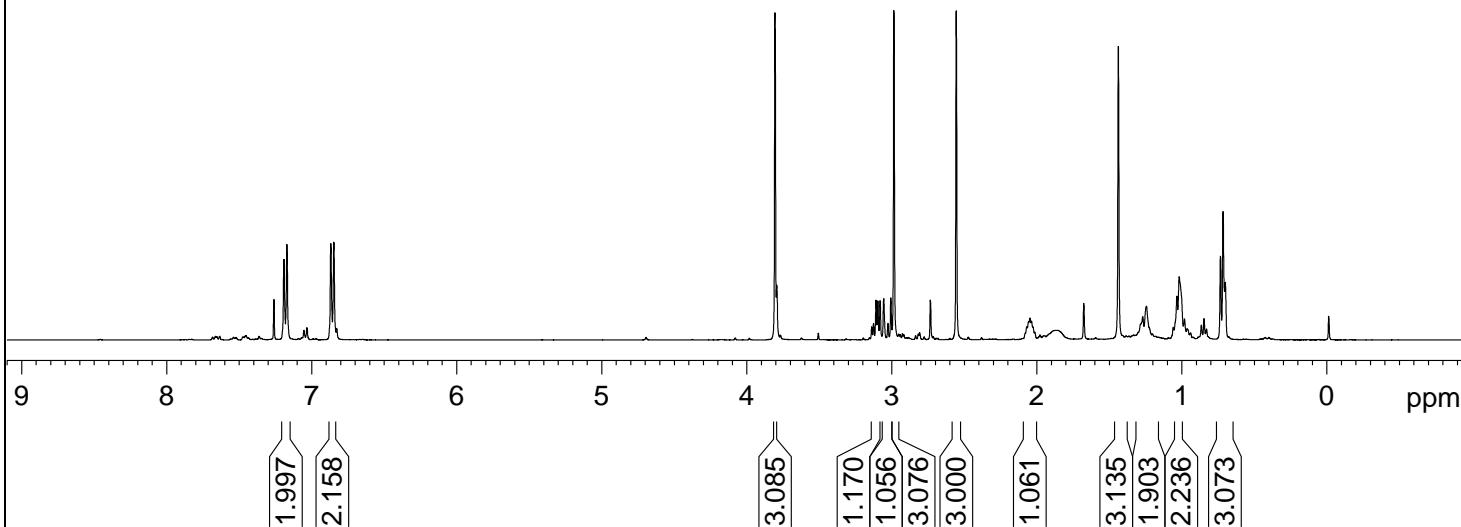
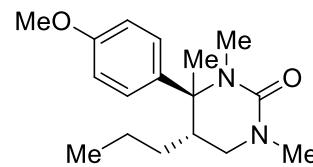
3.807
3.140
3.127
3.110
3.097
3.082
3.056
3.028
3.008
2.987
2.557
2.071
2.059
2.049
2.038
2.028
2.015
1.439
1.284
1.271
1.245
1.060
1.035
1.019
0.983
0.867
0.849

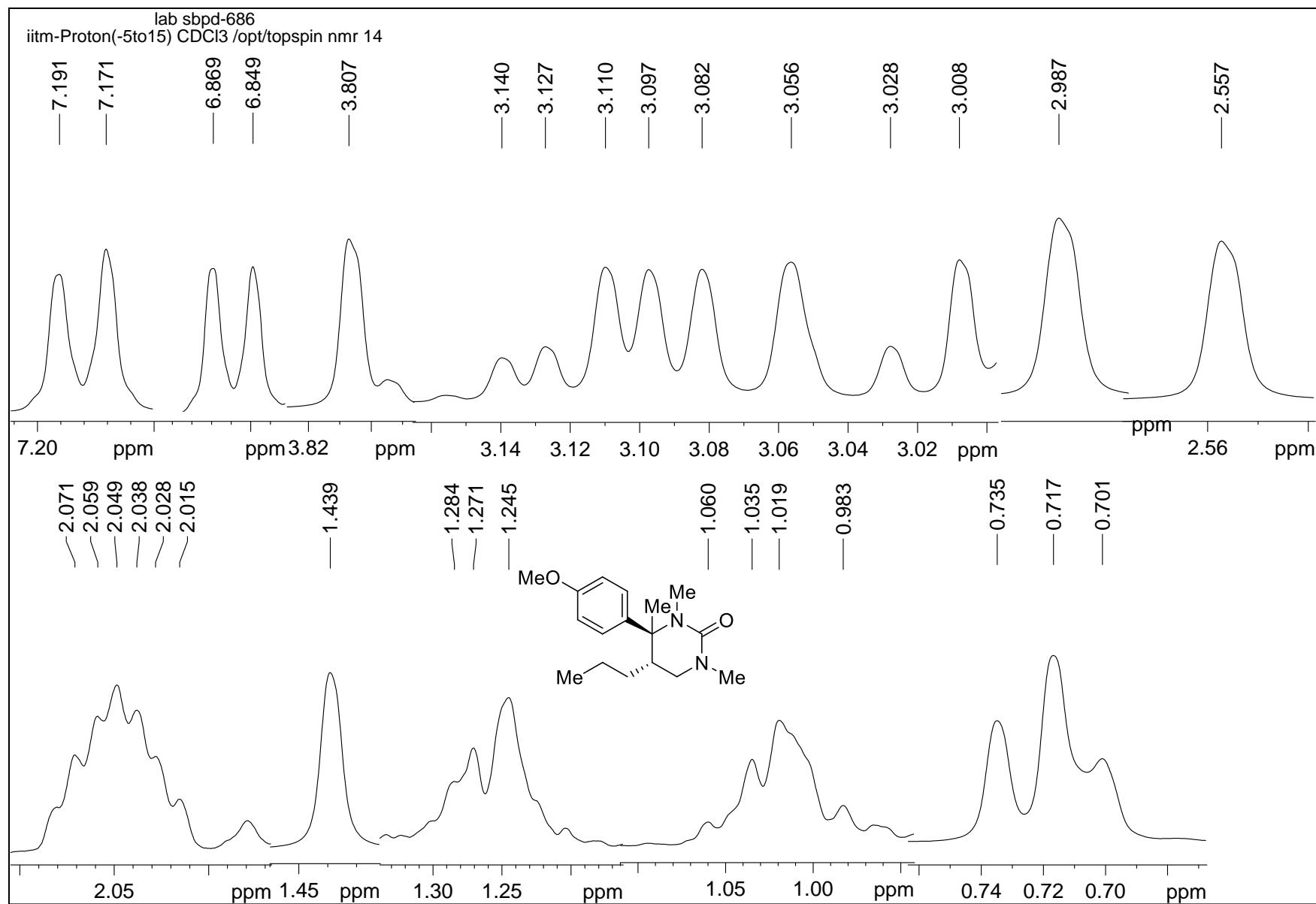
Current Data Parameters
NAME pd-706
EXPNO 354
PROCNO 1

F2 - Acquisition Parameters
Date 20190225
Time 14.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 138.85
DW 62.400 usec
DE 6.50 usec
TE 296.4 K
D1 0.5000000 sec
TD0 1

===== CHANNEL f1 =====
SF01 400.1320007 MHz
NUC1 1H
P1 15.70 usec
PLW1 7.7500000 W

F2 - Processing parameters
SI 65536
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





lab sbpd-686
itm_carbonshort CDCl₃ /opt/topspin nmr 14

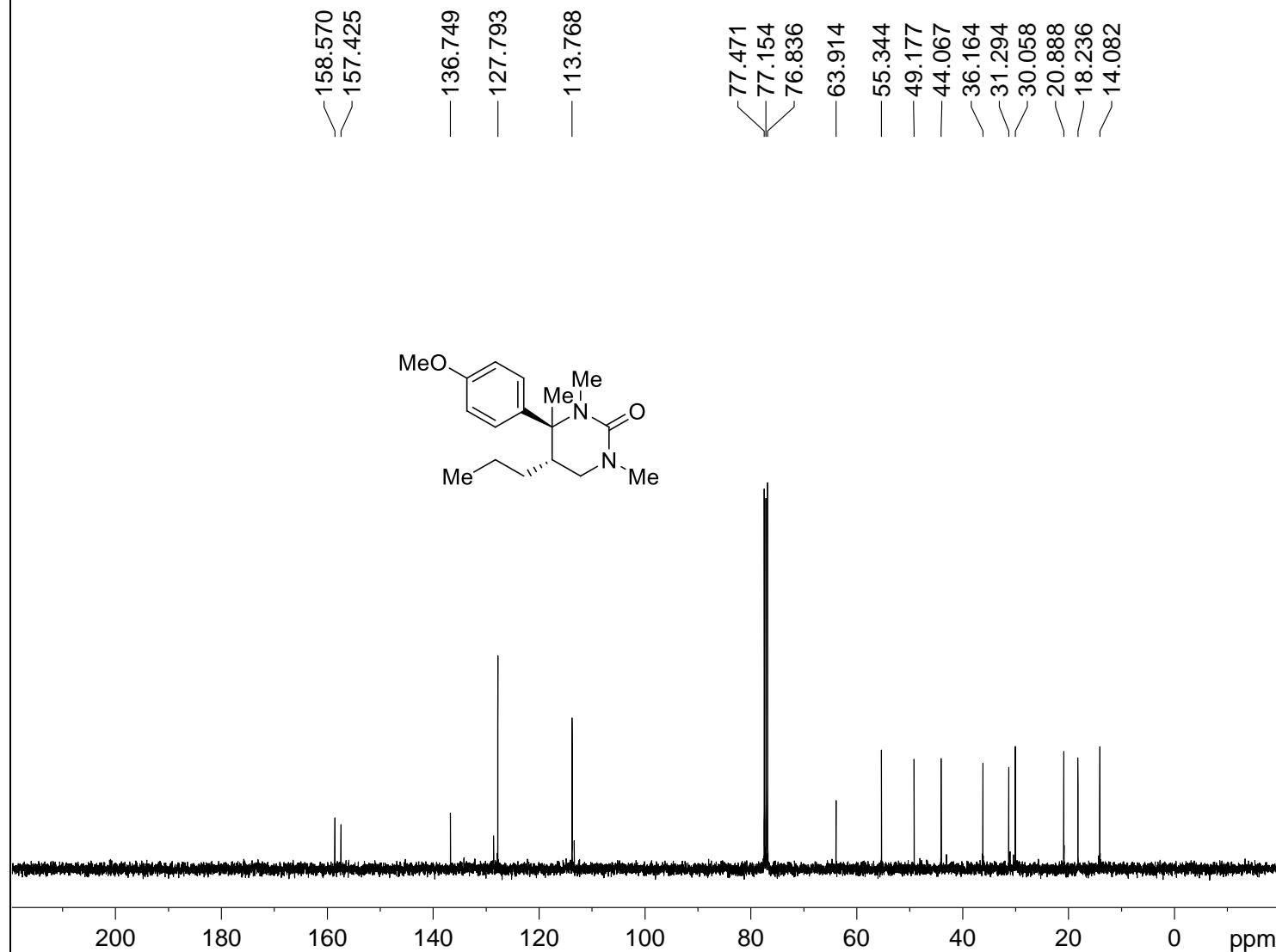


Figure 52 ¹³C NMR spectrum of compound 18a

lab sbpd-686
itm_C13DEPT135 CDCl₃ /opt/topspin nmr 14

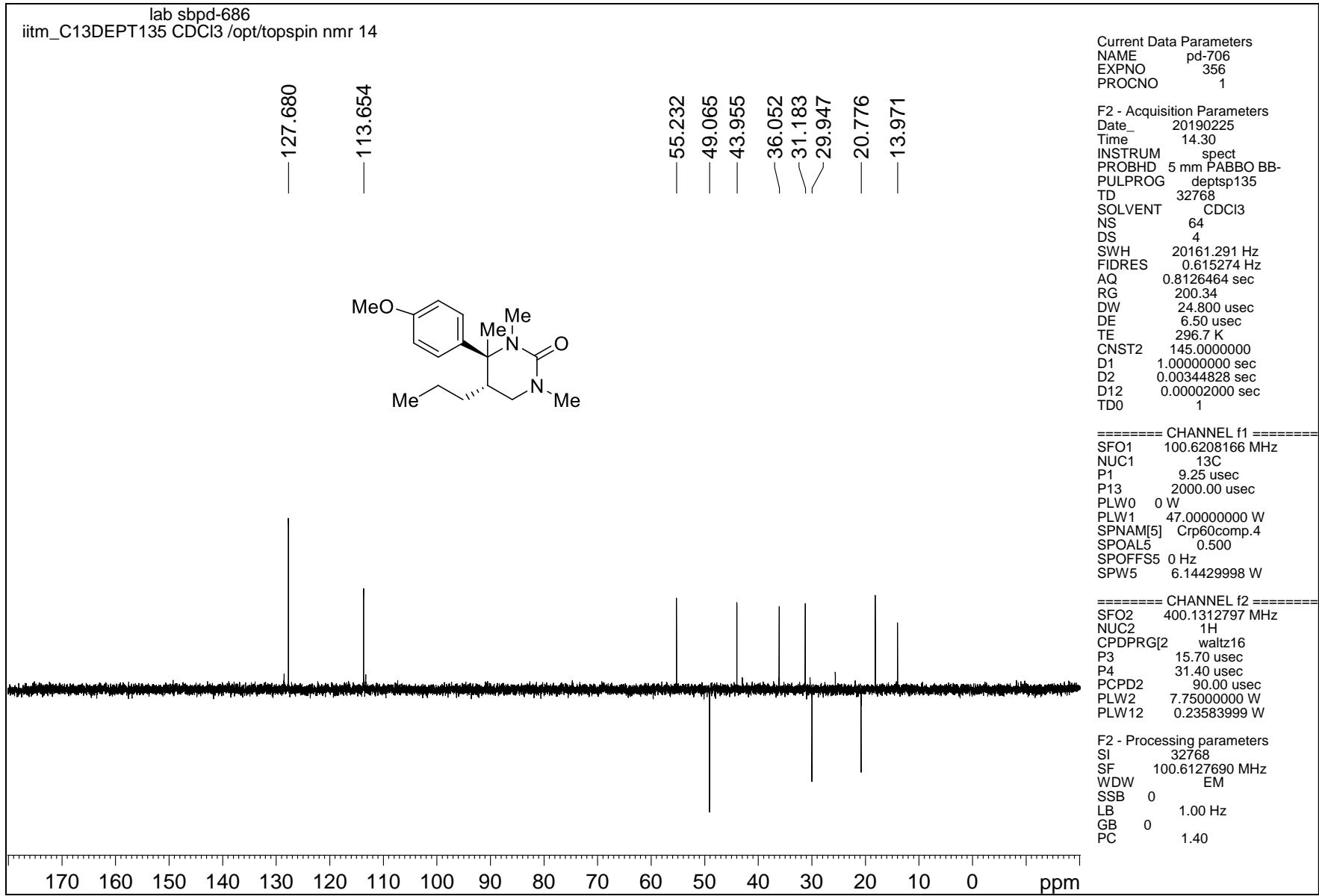
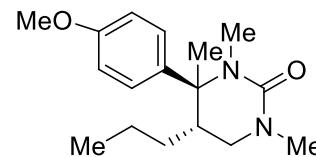


Figure 53 DEPT-135 NMR spectrum of compound 18a

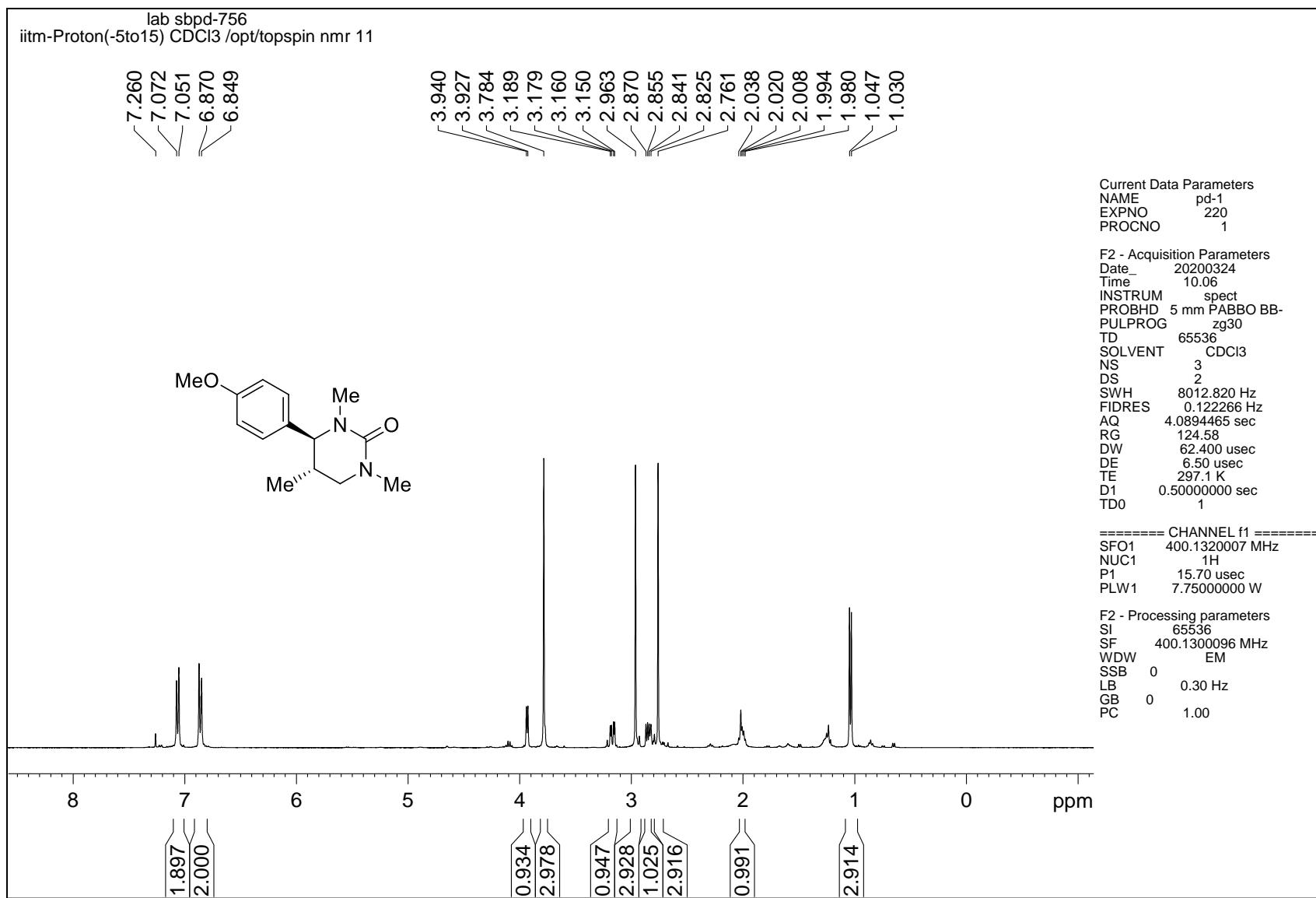


Figure 54 ¹H NMR spectrum of compound 19a

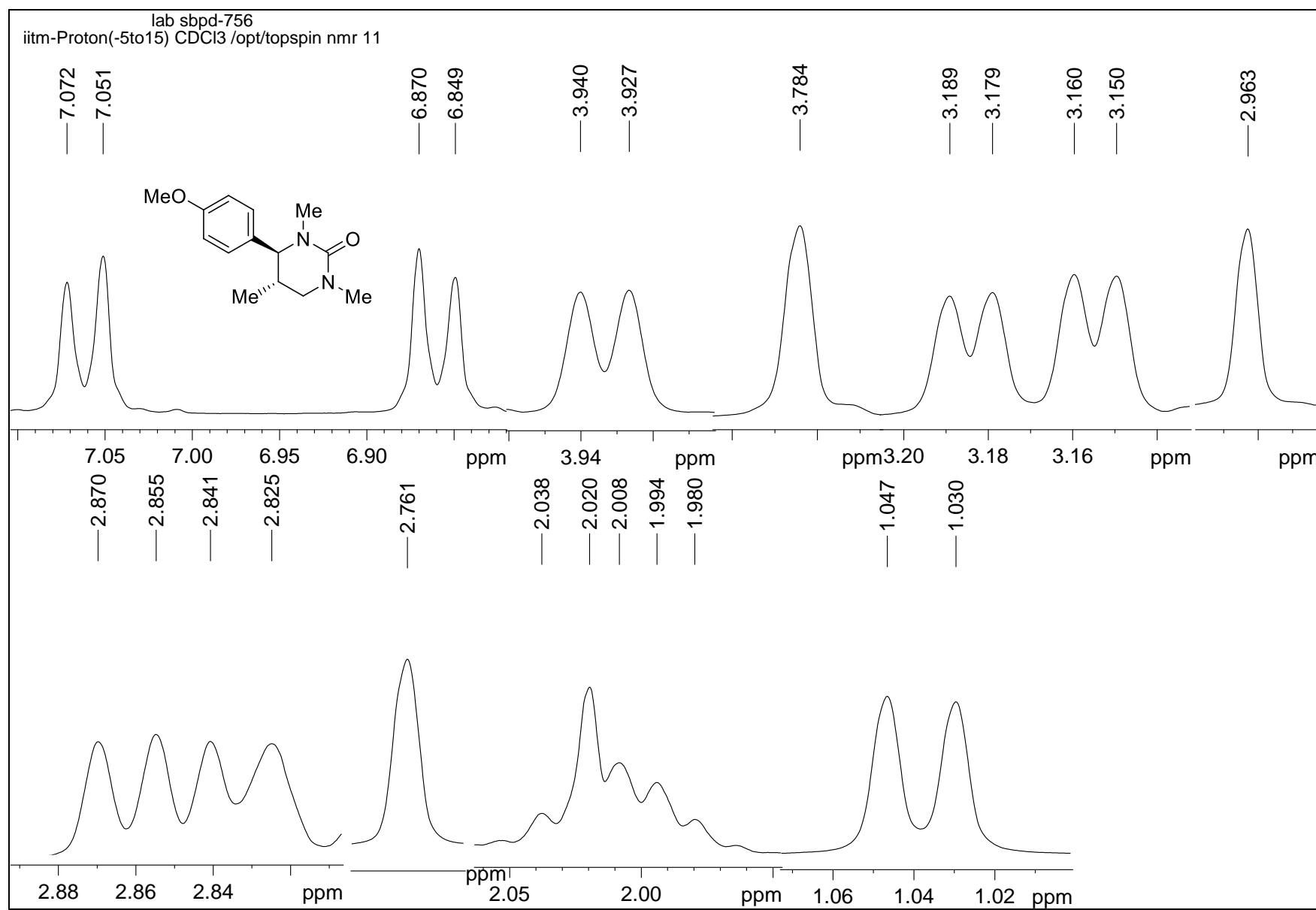


Figure 55 Expanded ^1H NMR spectrum of compound 19a

lab sbpd-756
itm_carbonshort CDCl₃ /opt/topspin nmr 1

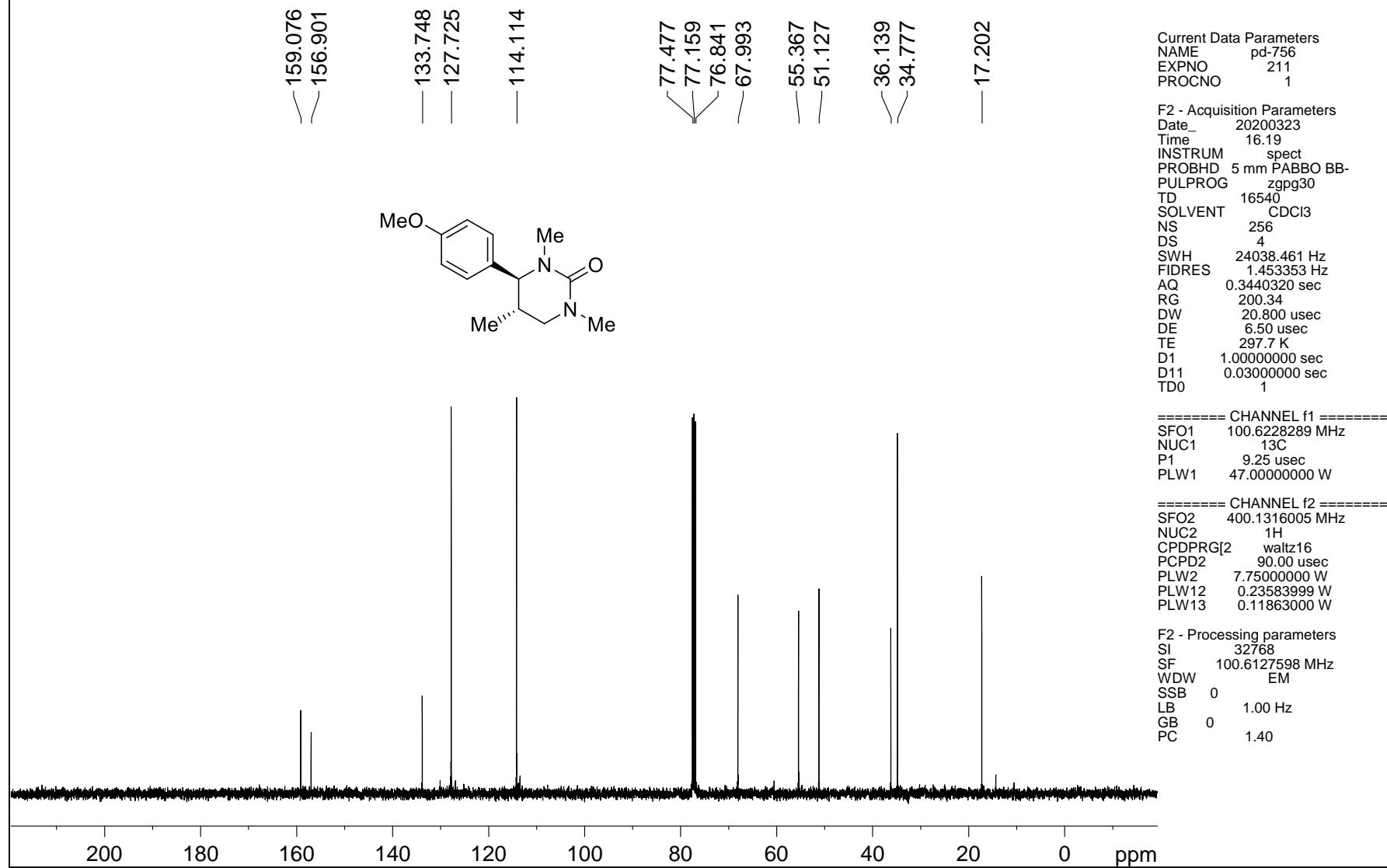


Figure 56 ¹³C NMR spectrum of compound 19a

lab sbpd-756
itm_C13DEPT135 CDCl₃ /opt/topspin nmr 1

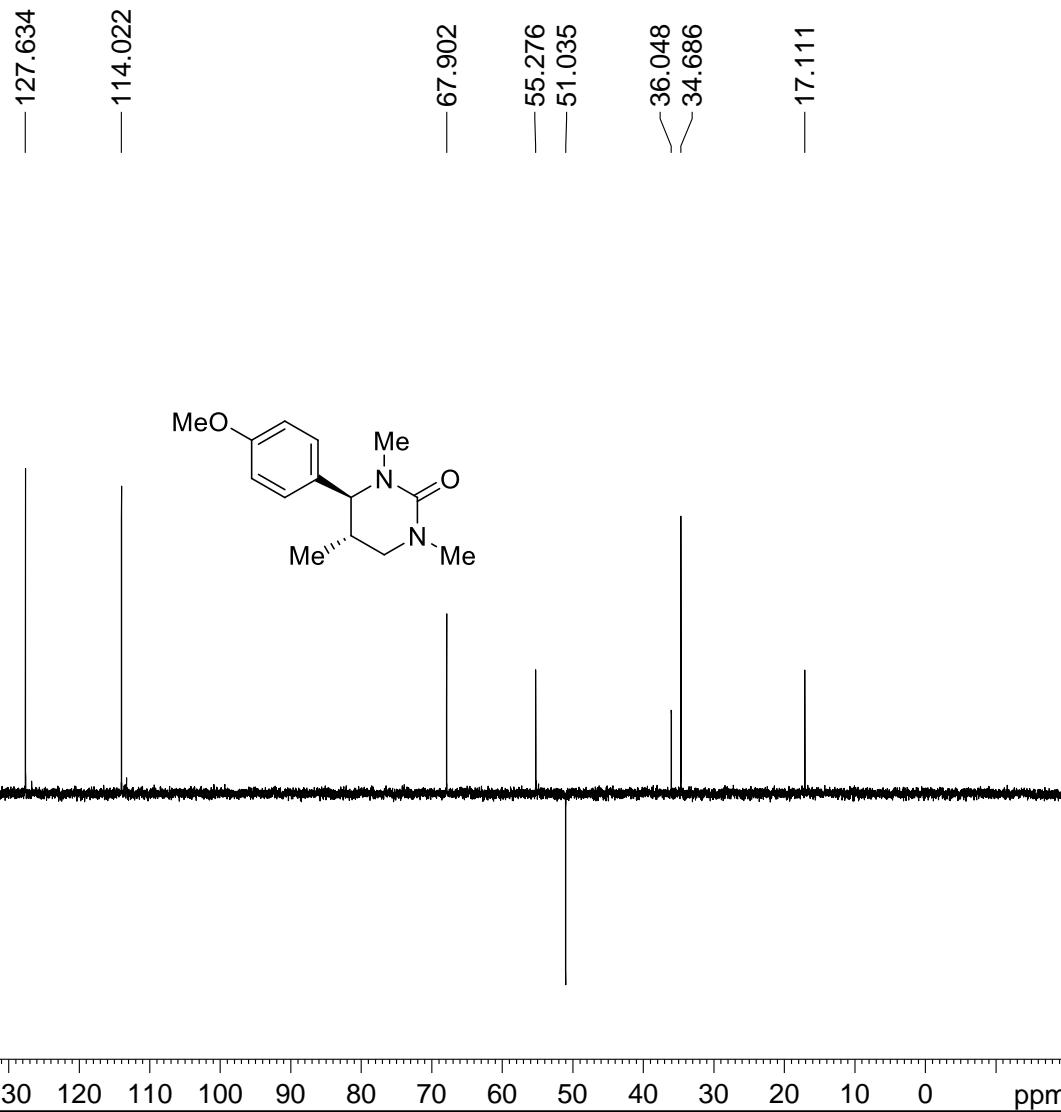


Figure 57 DEPT-135 NMR spectrum of compound 19a

lab sbpd-792
iitm-Proton(-5to15) CDCl₃ /opt/topspin nmr 4

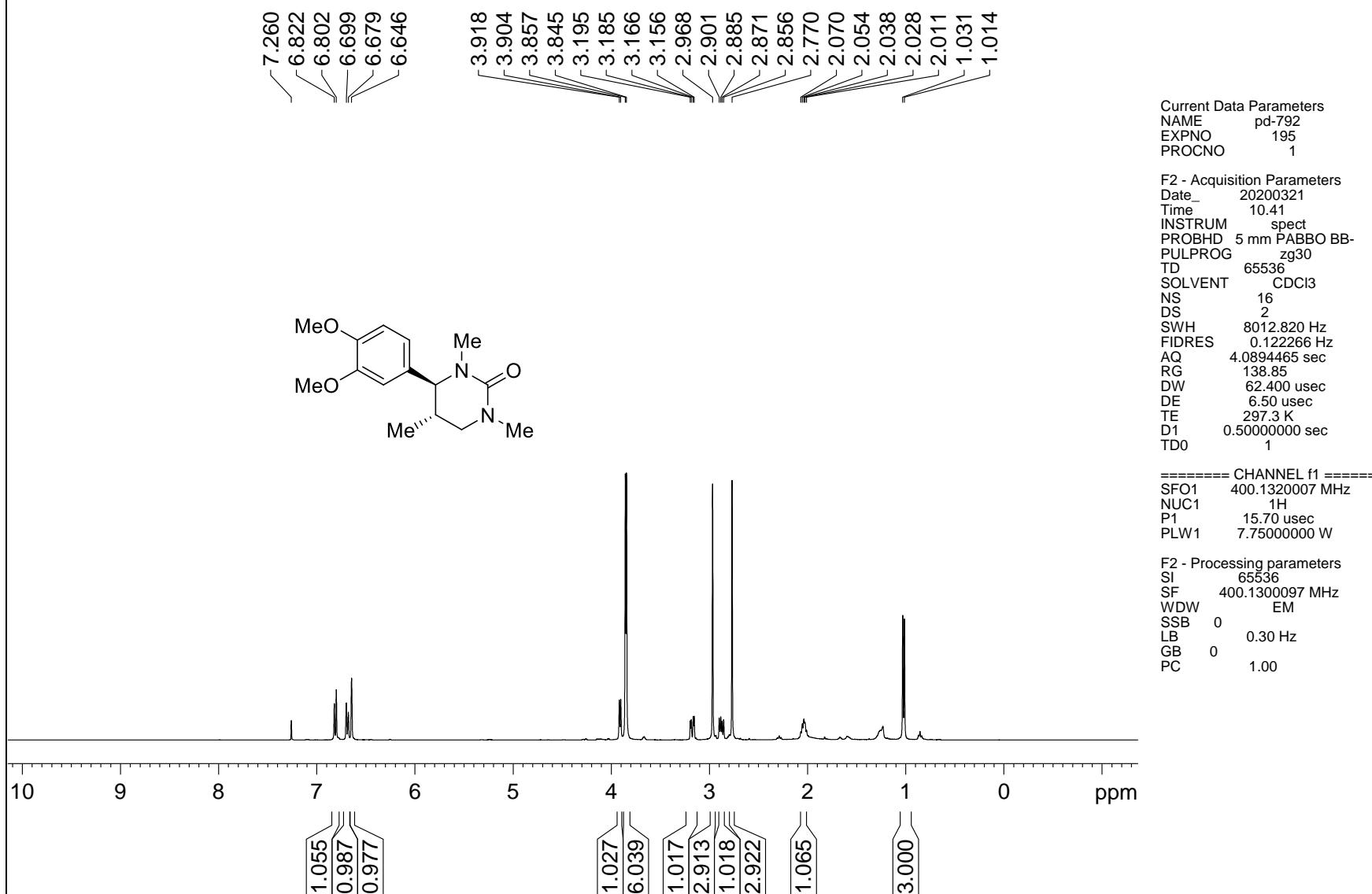
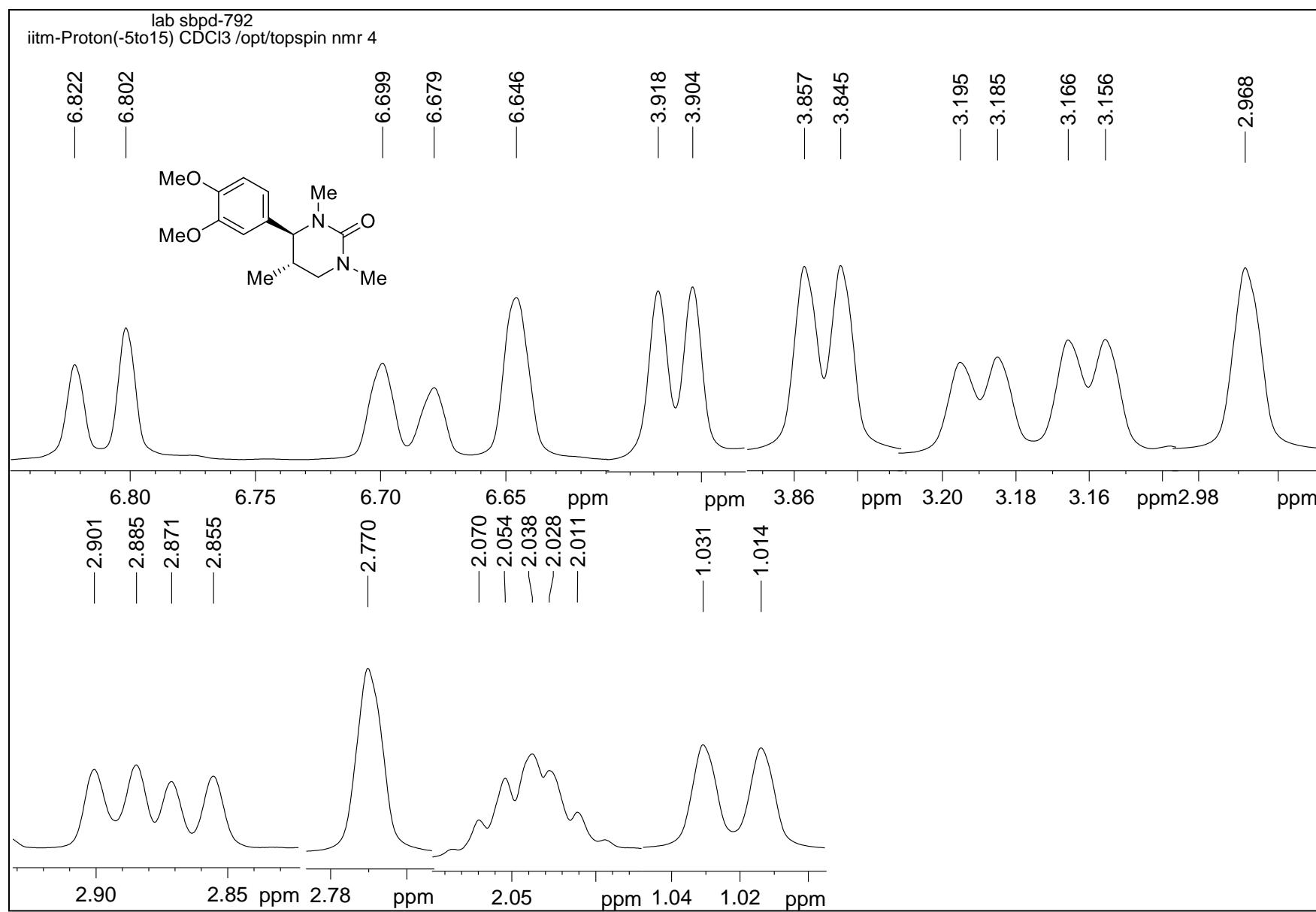


Figure 58 ¹H NMR spectrum of compound 15a from 20



lab sbpd-792
iitm_carbonshort CDCl₃ /opt/topspin nmr 4

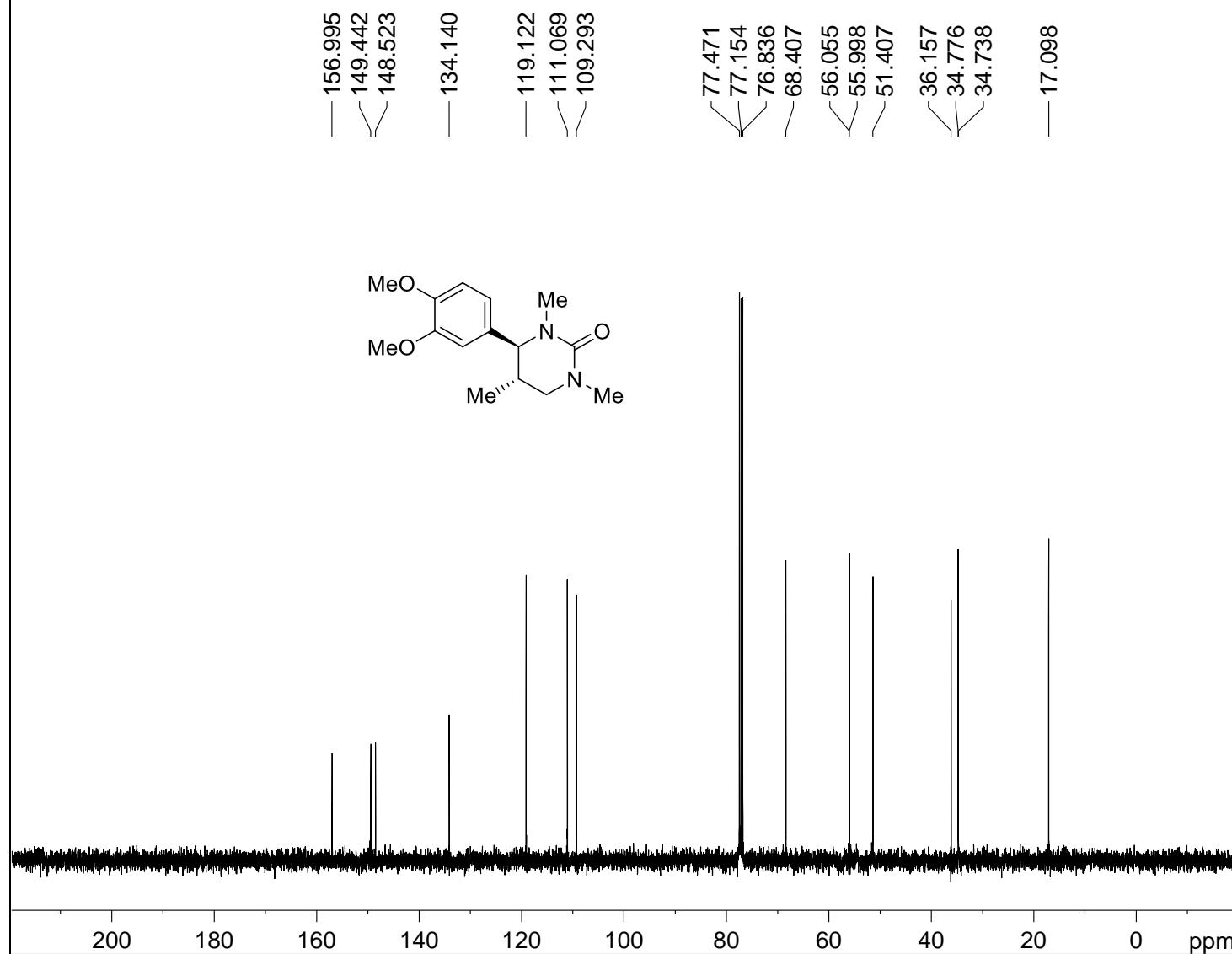


Figure 60 ¹³C NMR spectrum of compound 15a from 20

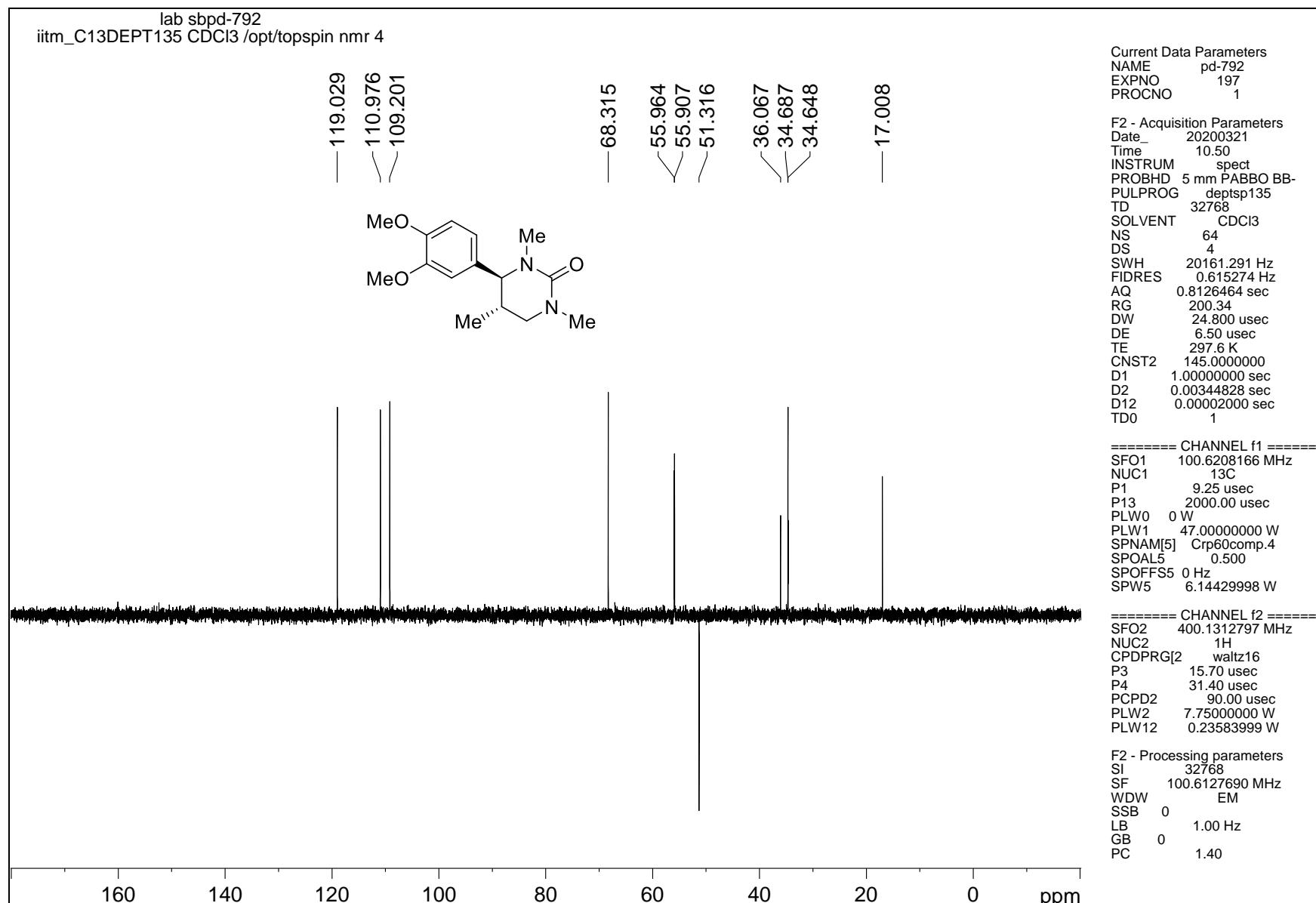


Figure 61 DEPT-135 NMR spectrum of compound 15a from 20

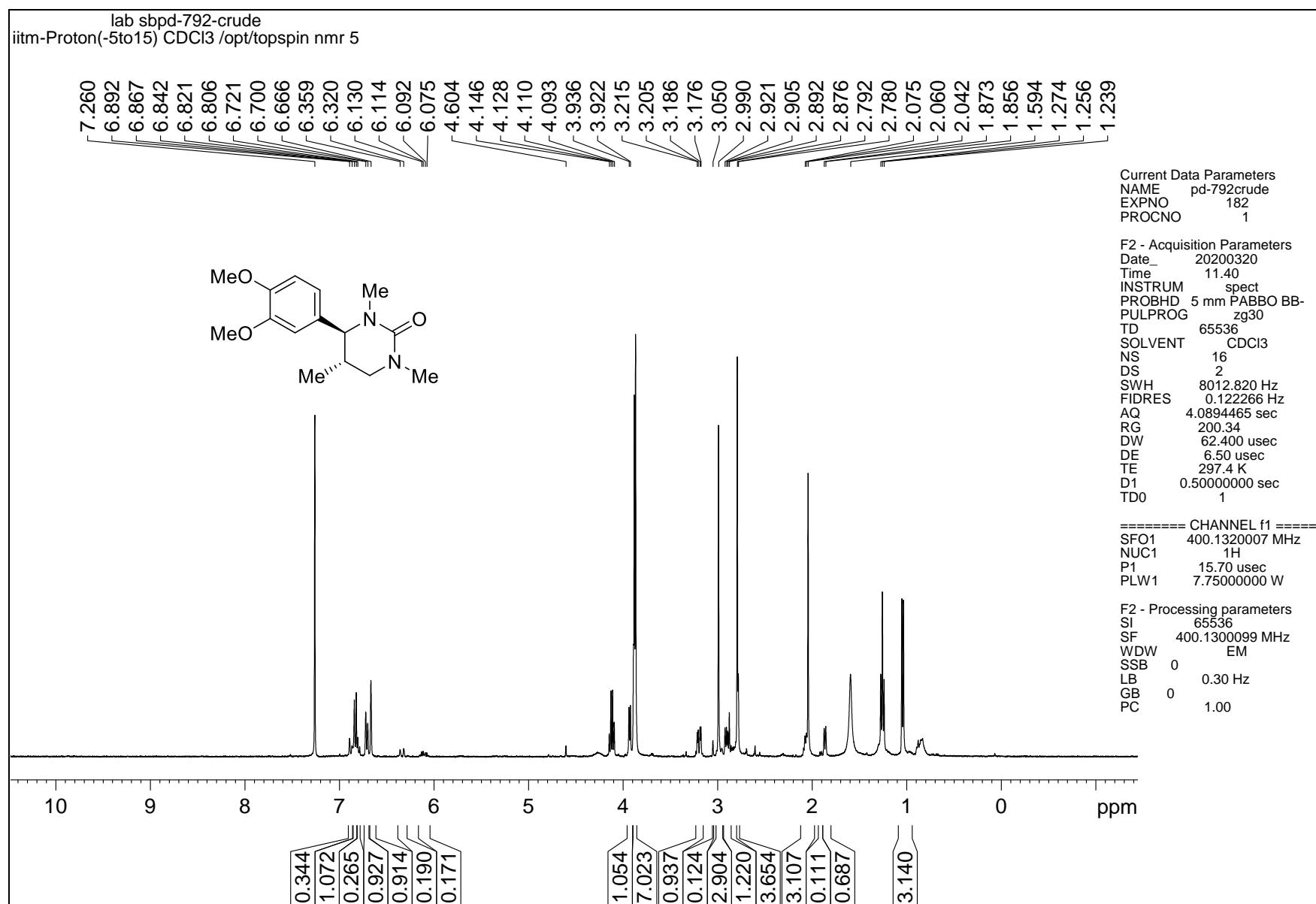


Figure 62 ¹H NMR spectrum of crude compound 15a

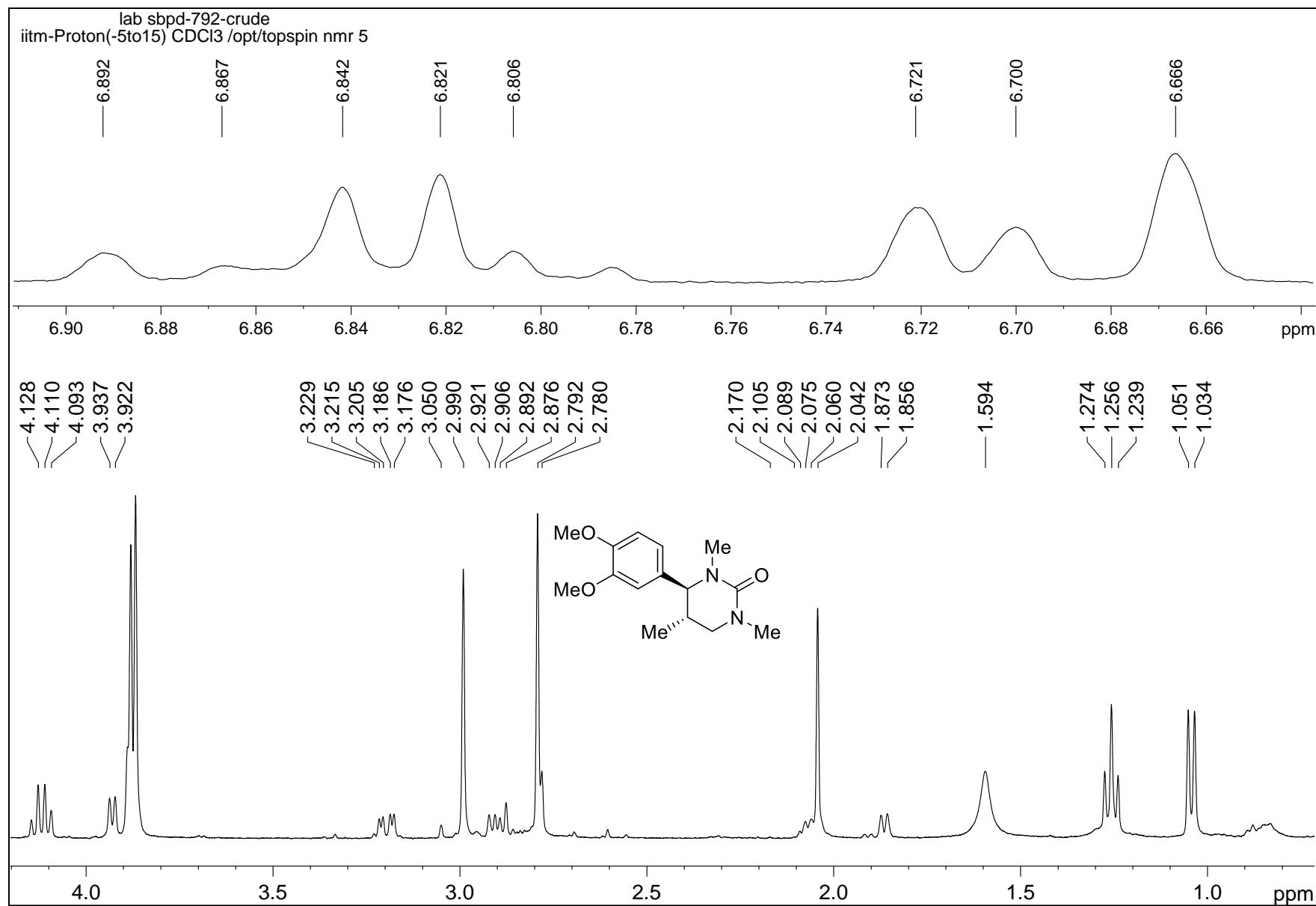
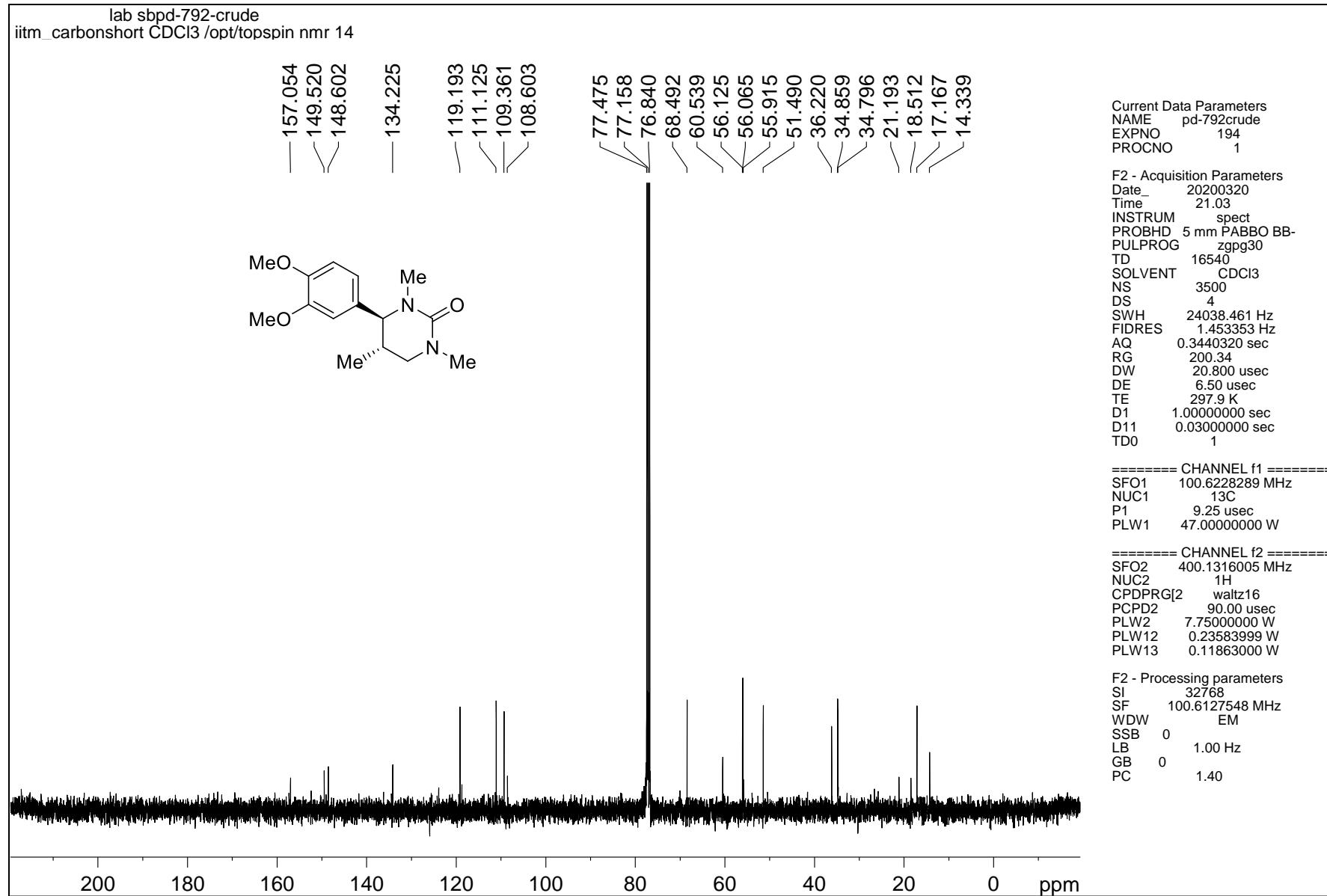


Figure 63 Expanded ¹H NMR spectrum of crude compound 15a



lab sbpd-792-crude
itm_C13DEPT135 CDCl₃ /opt/topspin nmr 4

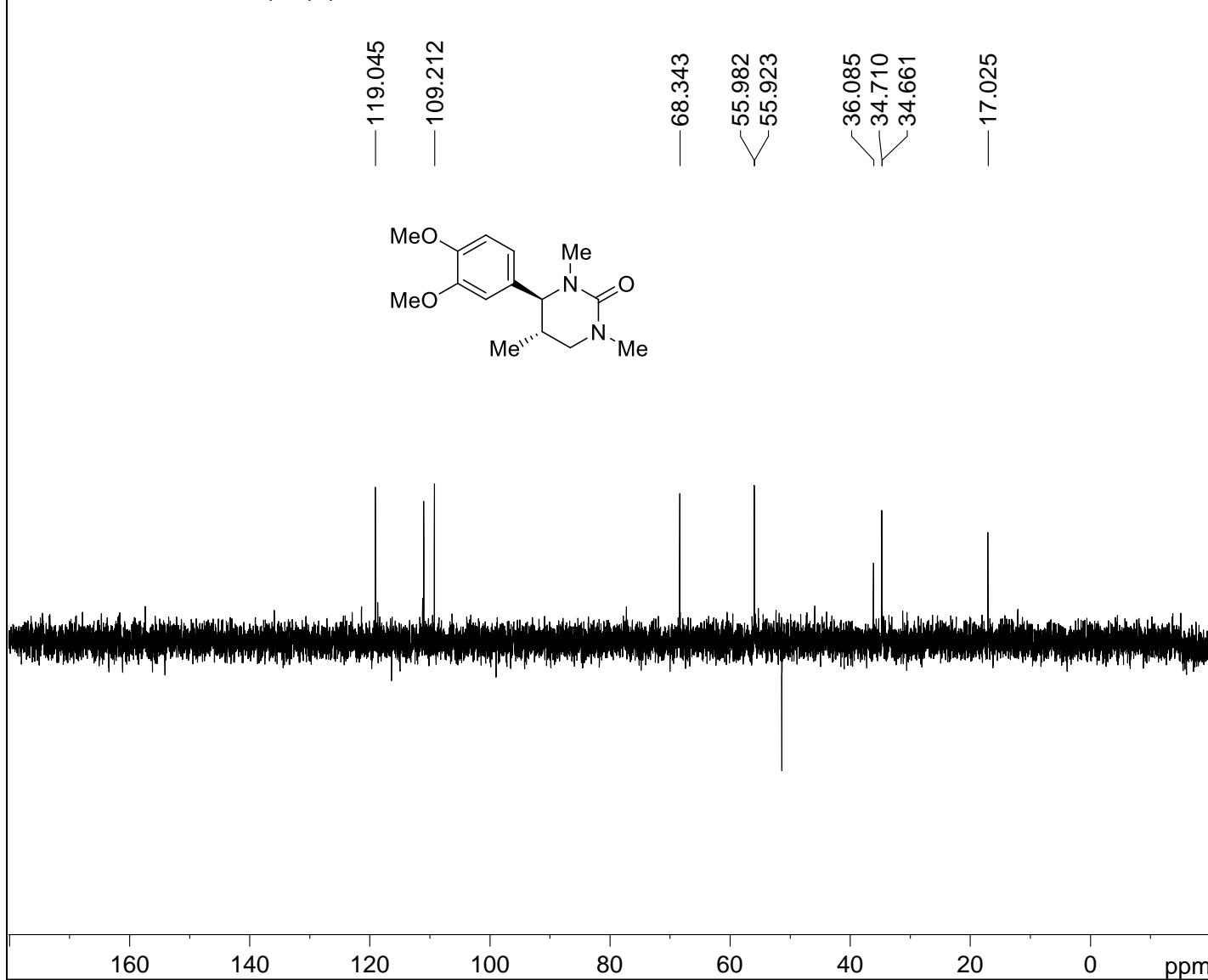
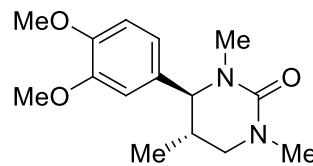
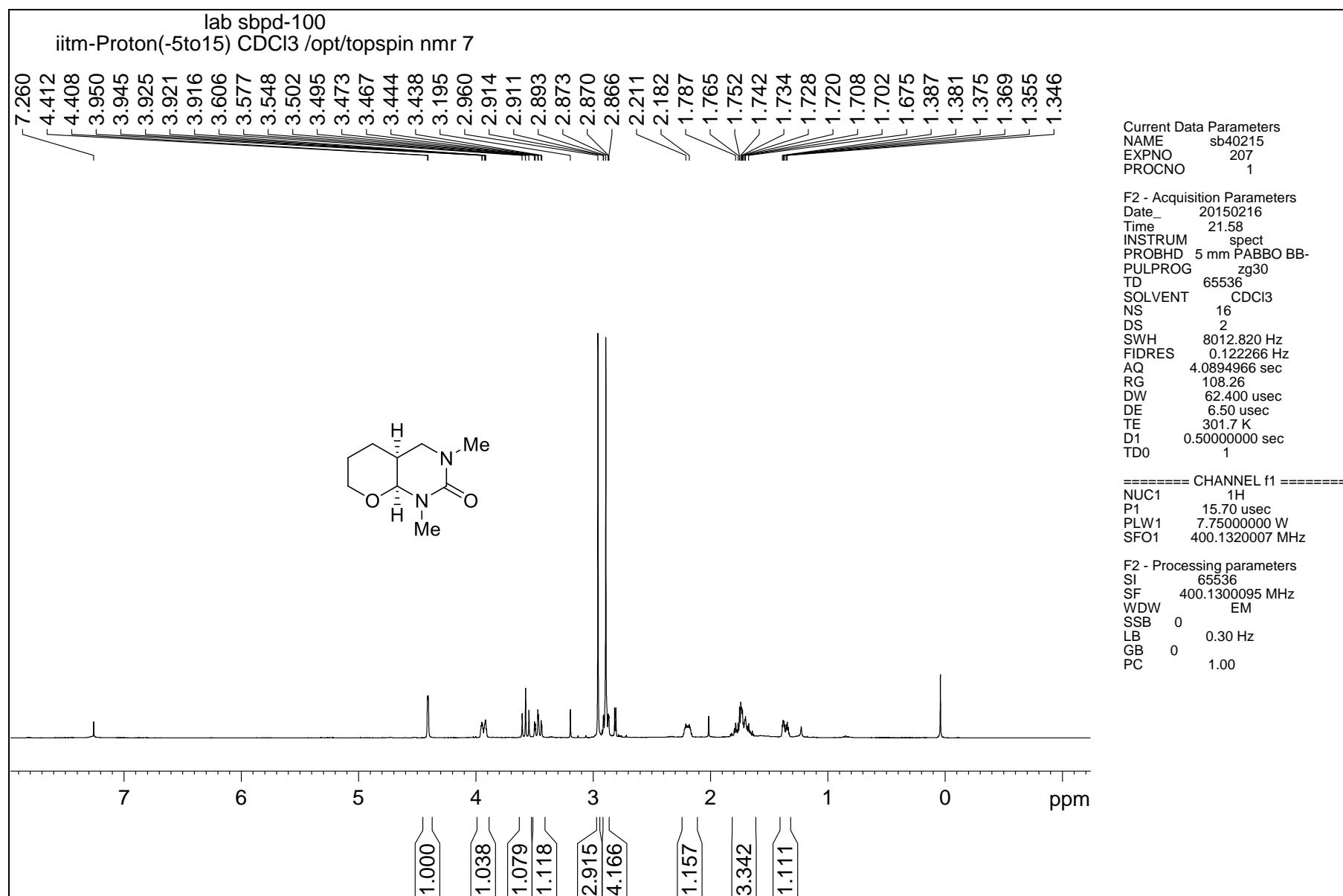


Figure 65 DEPT-135 NMR spectrum of crude compound 15a



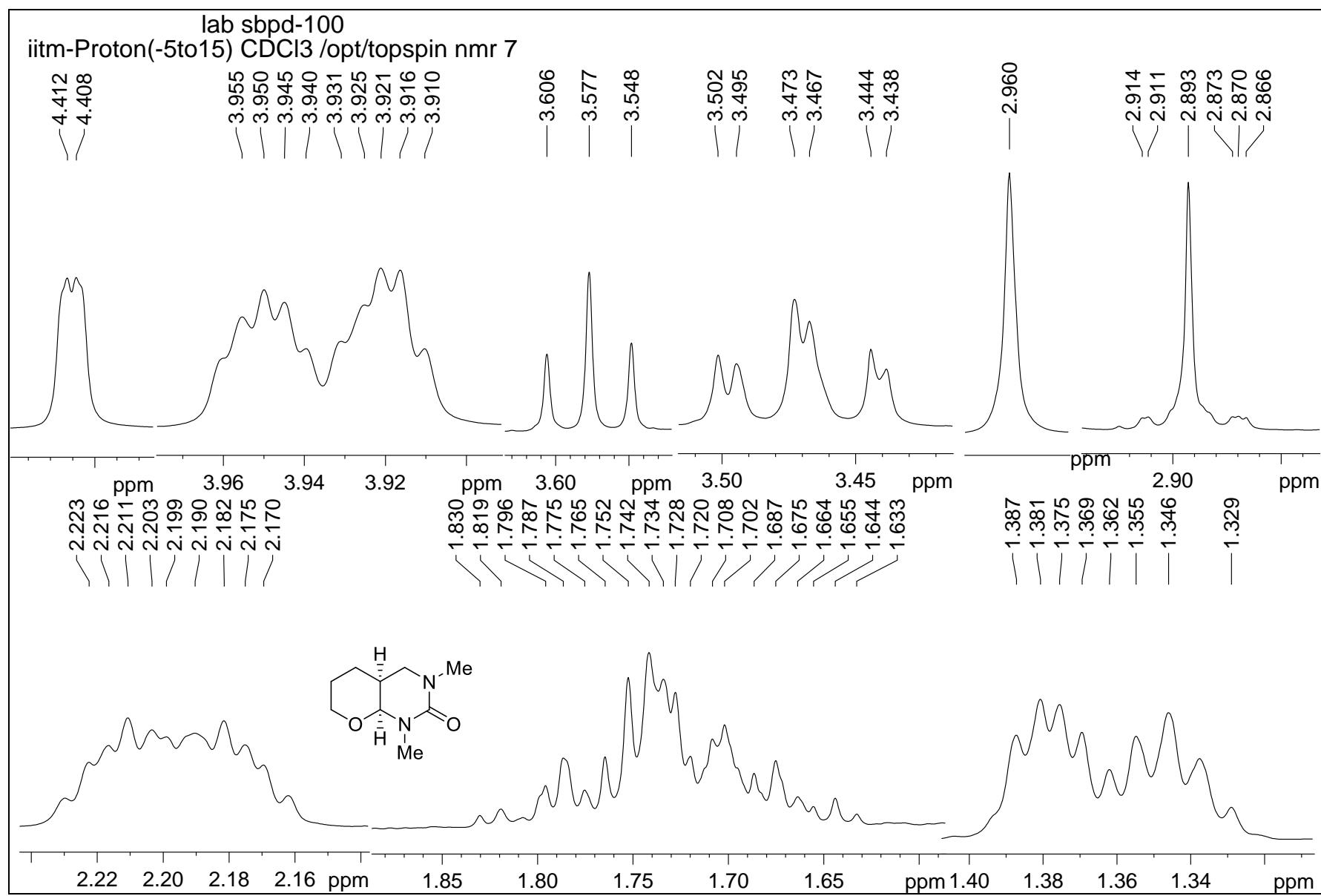


Figure 67 Expanded ¹H NMR spectrum of compound 21a

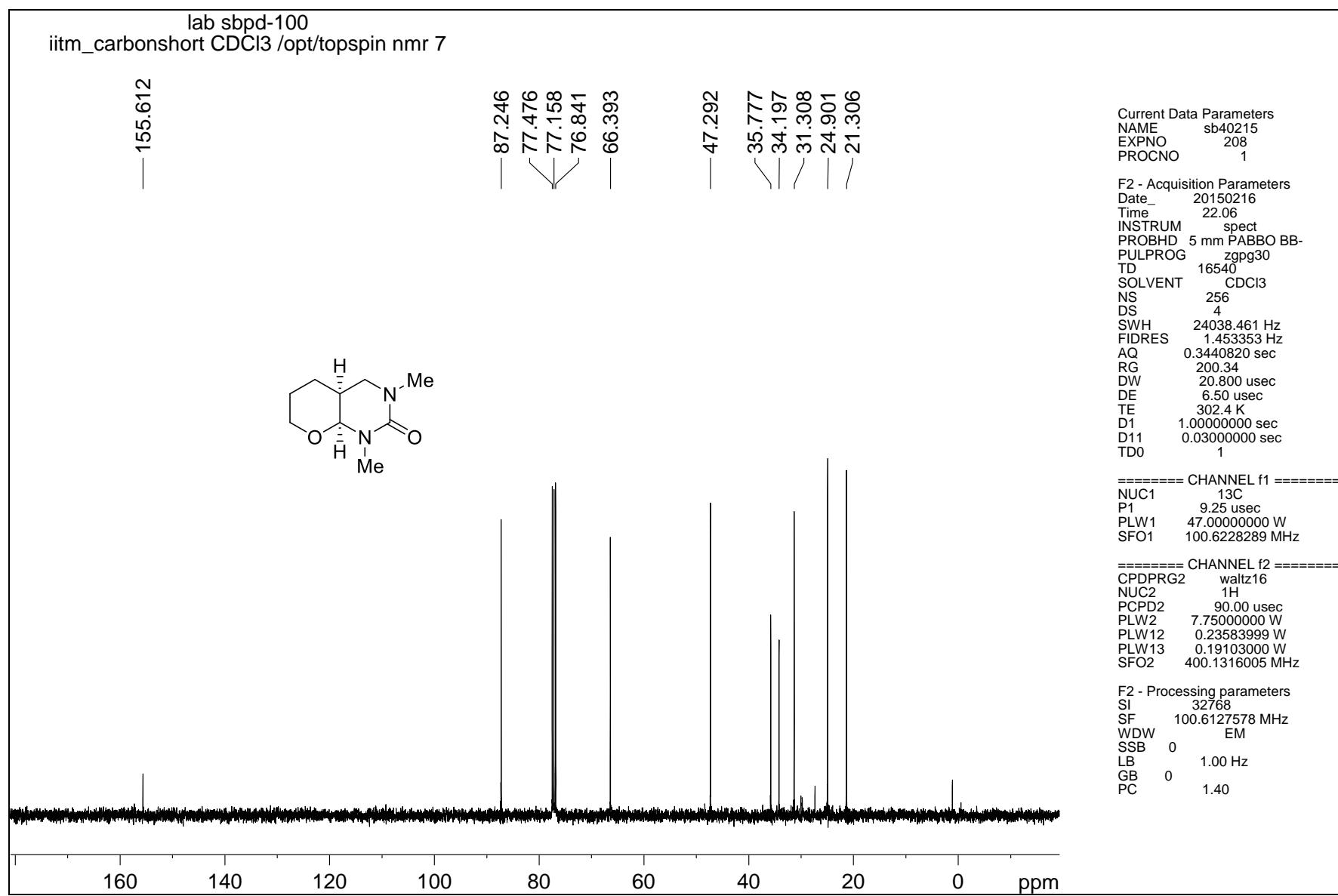
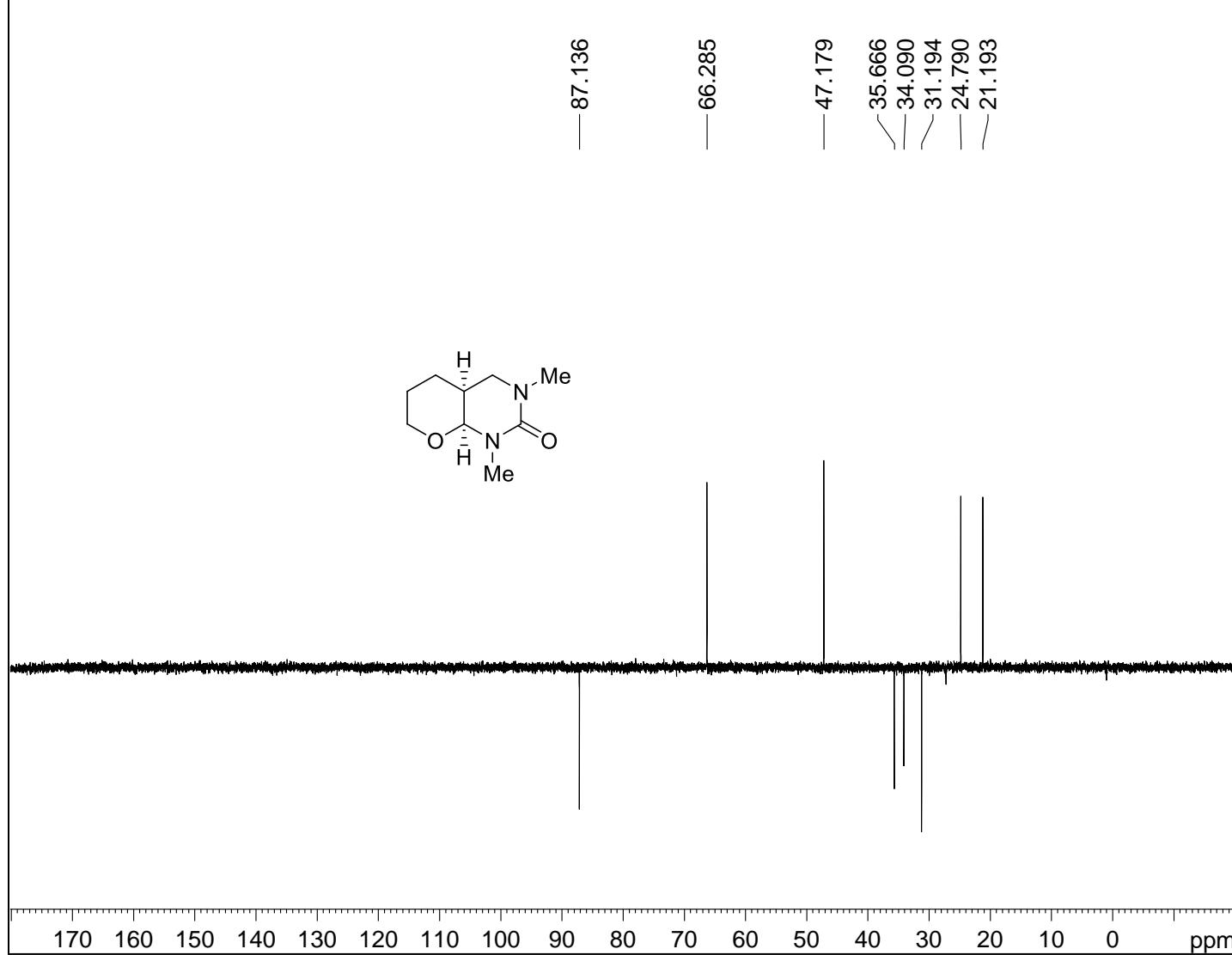
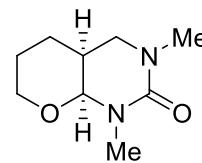


Figure 68 ¹³C NMR spectrum of compound 21a

lab sbpd-100
iitm_C13DEPT135 CDCl3 /opt/topspin nmr 7



Current Data Parameters
NAME sb40215
EXPNO 209
PROCNO 1

F2 - Acquisition Parameters
Date 20150216
Time 22.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG deptsp135
TD 32768
SOLVENT CDCl3
NS 64
DS 4
SWH 20161.291 Hz
FIDRES 0.615274 Hz
AQ 0.8126464 sec
RG 200.34
DW 24.800 usec
DE 6.50 usec
TE 302.1 K
CNST2 145.000000
D1 1.0000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 ¹³C
P1 9.25 usec
P13 2000.00 usec
PLW0 0 W
PLW1 47.0000000 W
SFO1 100.6208166 MHz
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.1442998 W

===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 ¹H
P3 15.70 usec
P4 31.40 usec
PCPD2 90.00 usec
PLW2 7.7500000 W
PLW12 0.23583999 W
SFO2 400.1312797 MHz

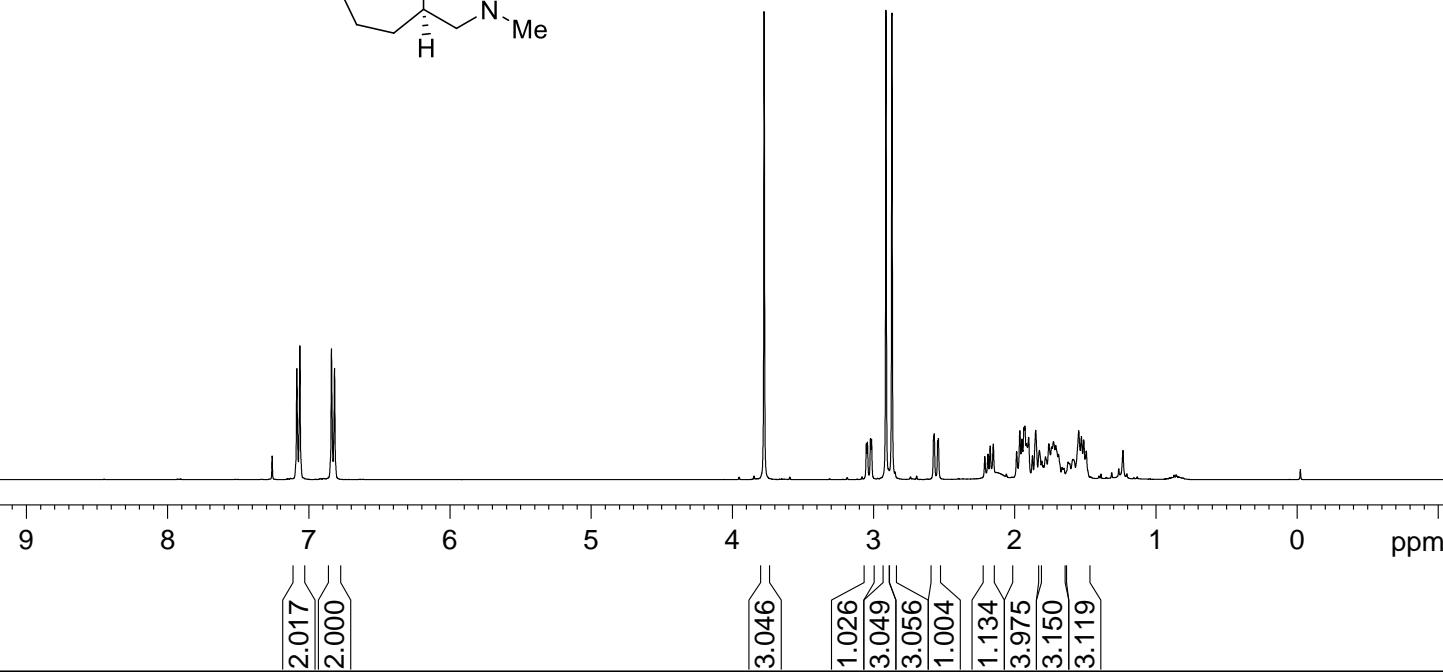
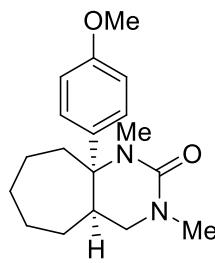
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure 69 DEPT-135 NMR spectrum of compound 21a

lab sbpd-714
itm-Proton(-5to15) CDCl₃ /opt/topspin nmr 6

7.260
7.085
7.063
6.839
6.818

3.775
3.051
3.043
3.021
3.014
2.912
2.870
2.574
2.570
2.545
2.541
2.211
2.189
2.173
2.151
1.986
1.949
1.934
1.927
1.916
1.901
1.873
1.851
1.825
1.807
1.781
1.757
1.738
1.725
1.709
1.689



Current Data Parameters
NAME pd-714
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190301
Time 17.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 79.8
DW 62.400 usec
DE 6.50 usec
TE 296.1 K
D1 0.5000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 400.1320007 MHz
NUC1 1H
P1 15.70 usec
PLW1 7.7500000 W

F2 - Processing parameters
SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure 70 ¹H NMR spectrum of compound 22a

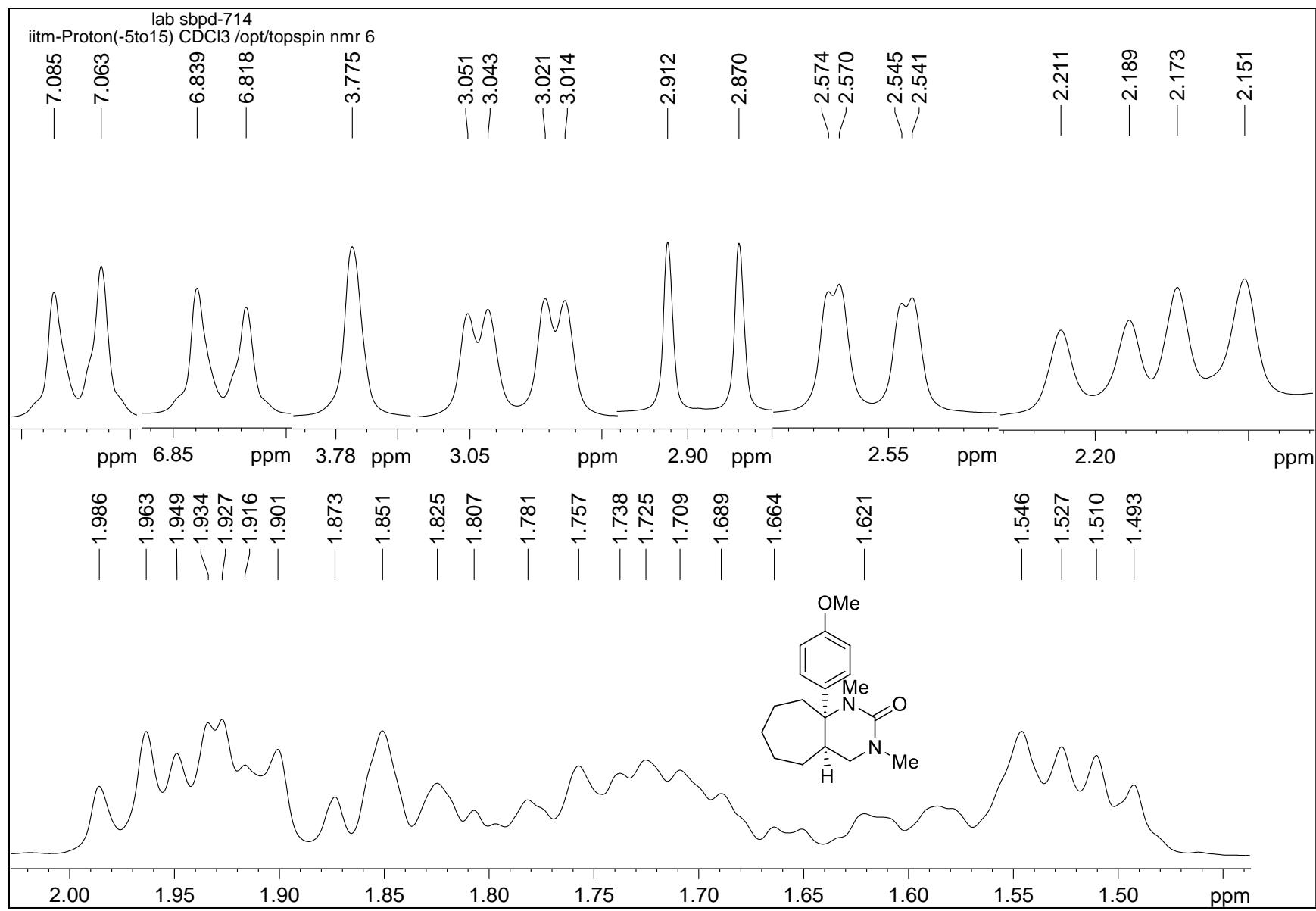


Figure 71 Expanded¹H NMR spectrum of compound 22a

lab sbpd-714
itm_carbonshort CDCl₃ /opt/topspin nmr 6

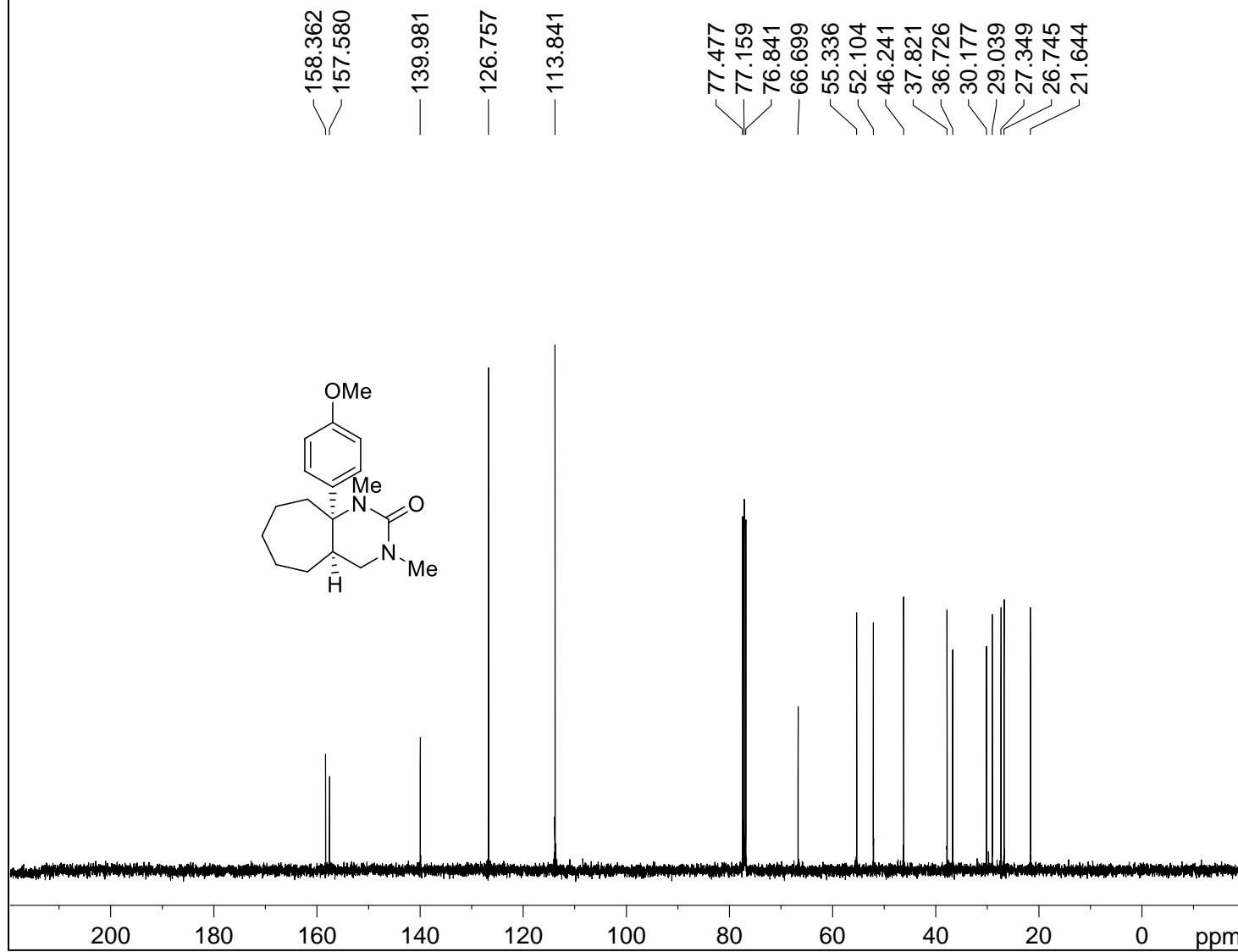


Figure 72 ¹³C NMR spectrum of compound 22a

lab sbpd-714
iitm_C13DEPT135 CDCl3 /opt/topspin nmr 6

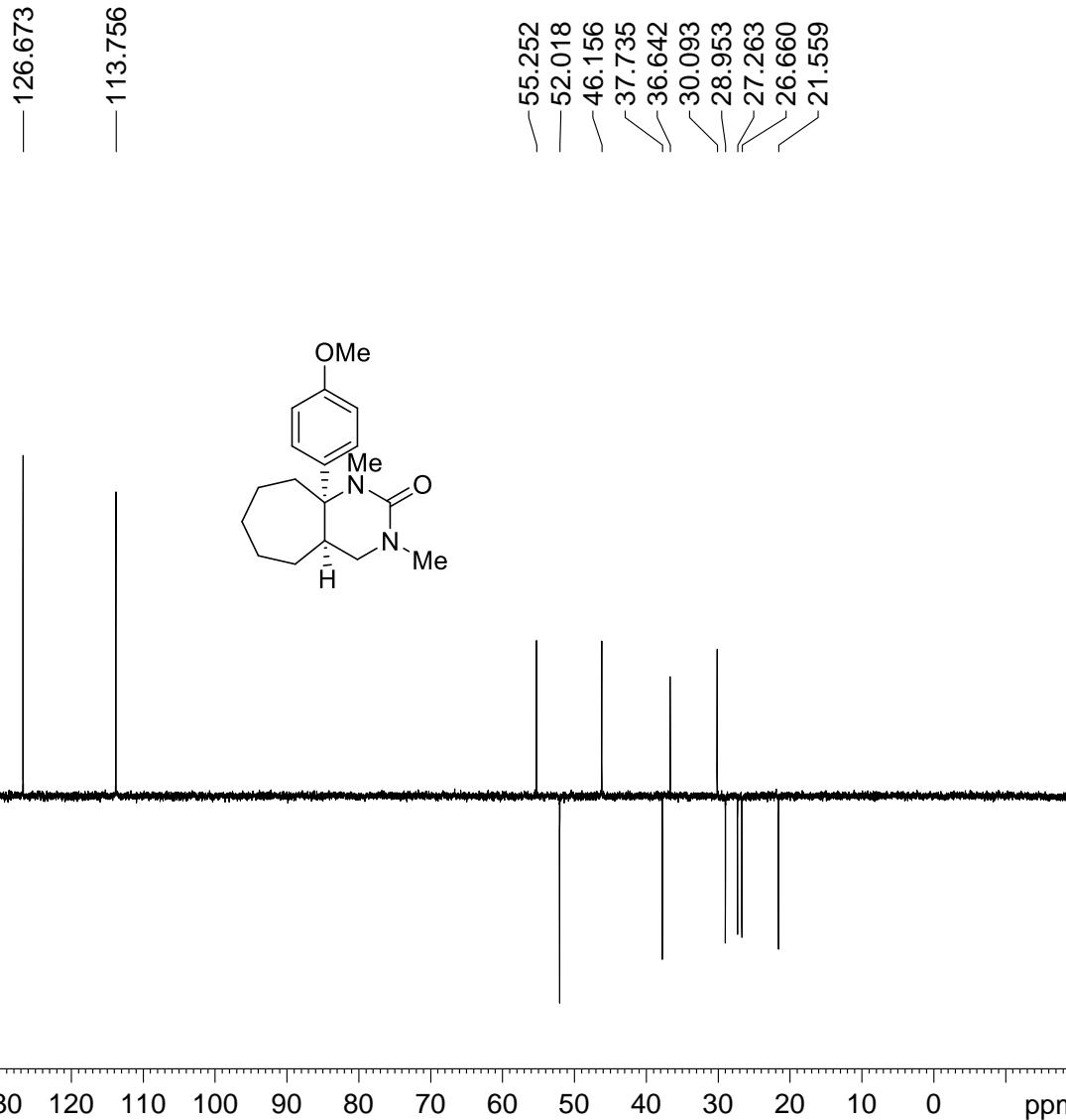
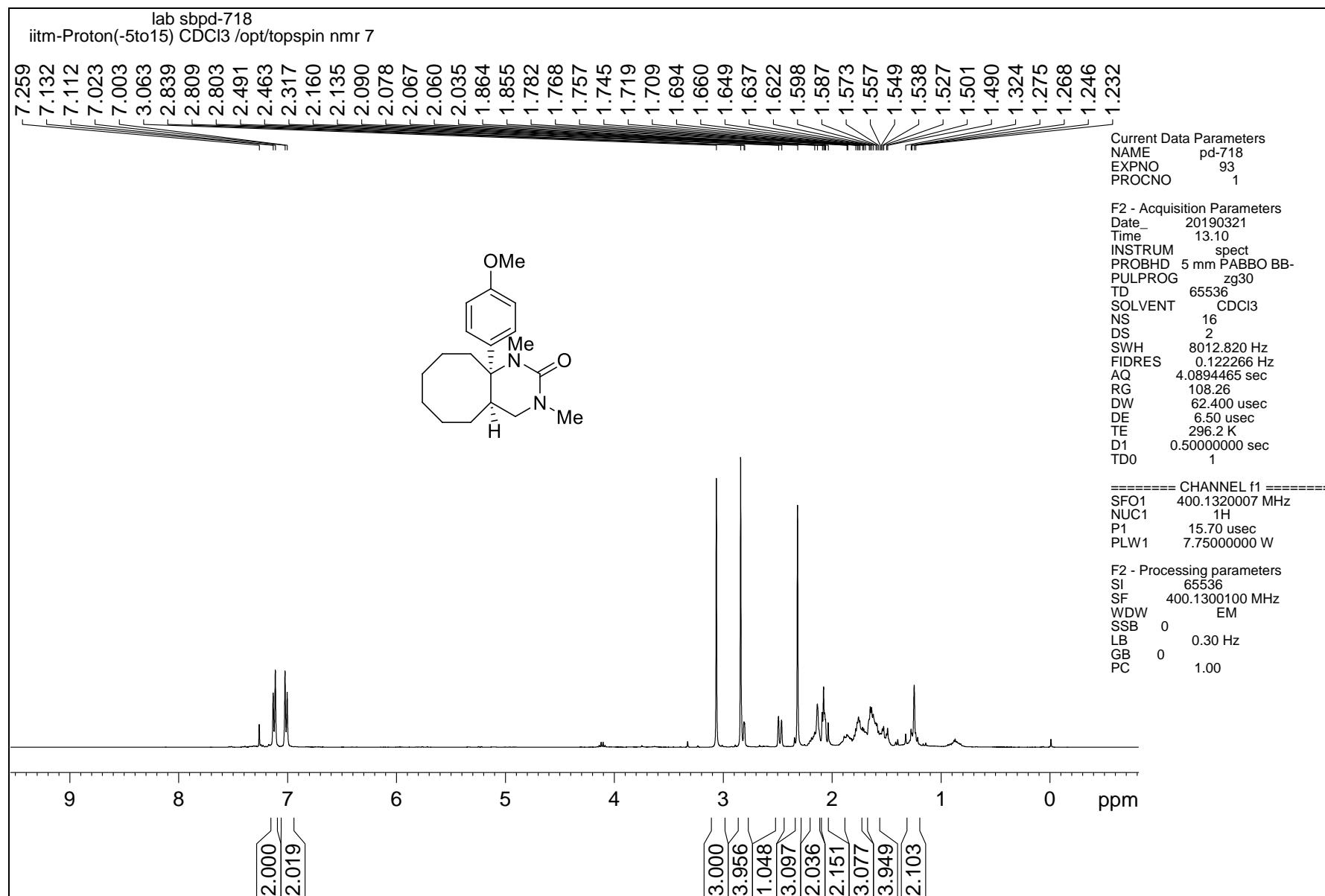


Figure 73 DEPT-135 NMR spectrum of compound 22a



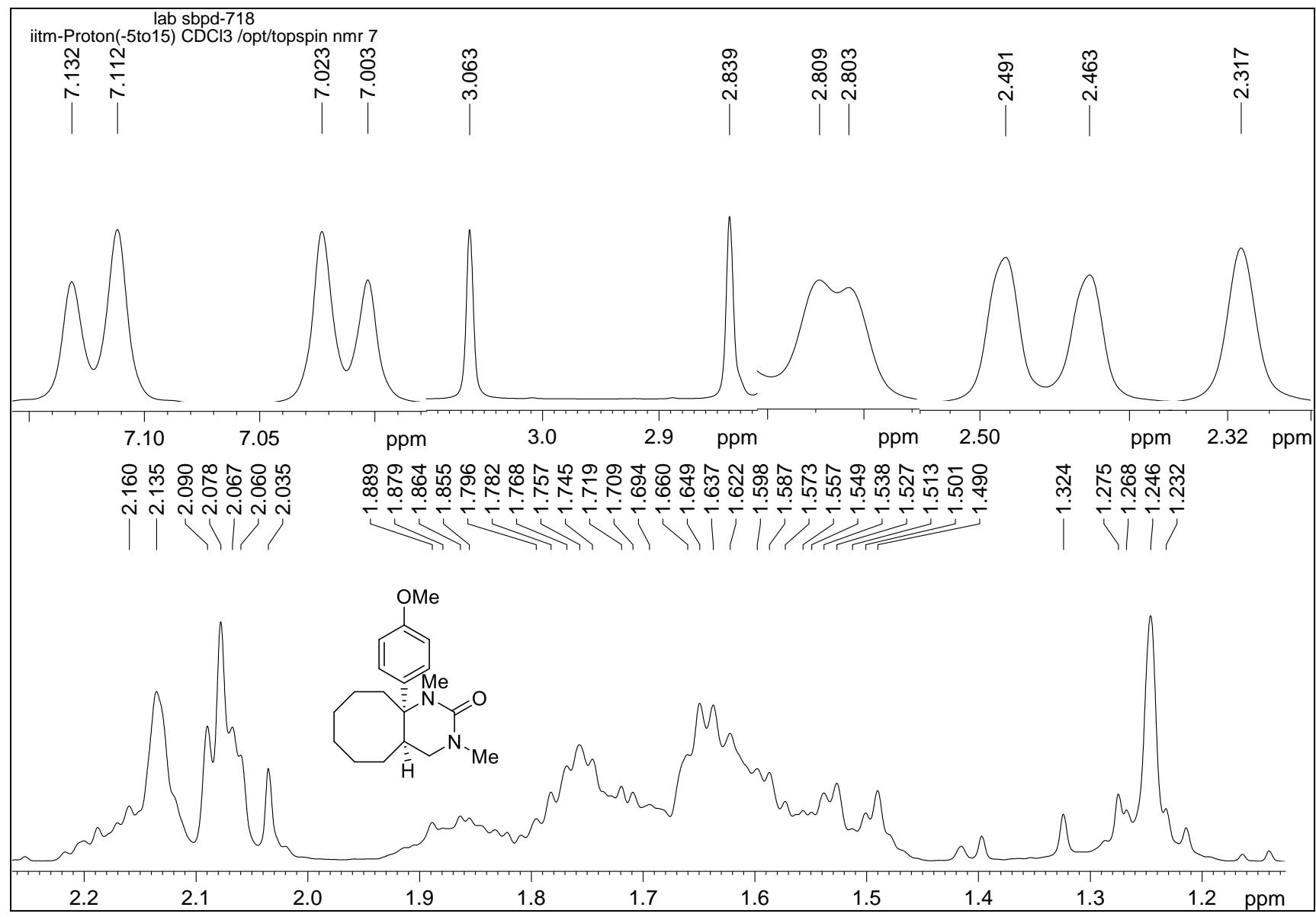


Figure 75 Expanded ¹H NMR spectrum of compound 23a

lab sbpd-718
iitm_carbonshort CDCl₃ /opt/topspin nmr 7

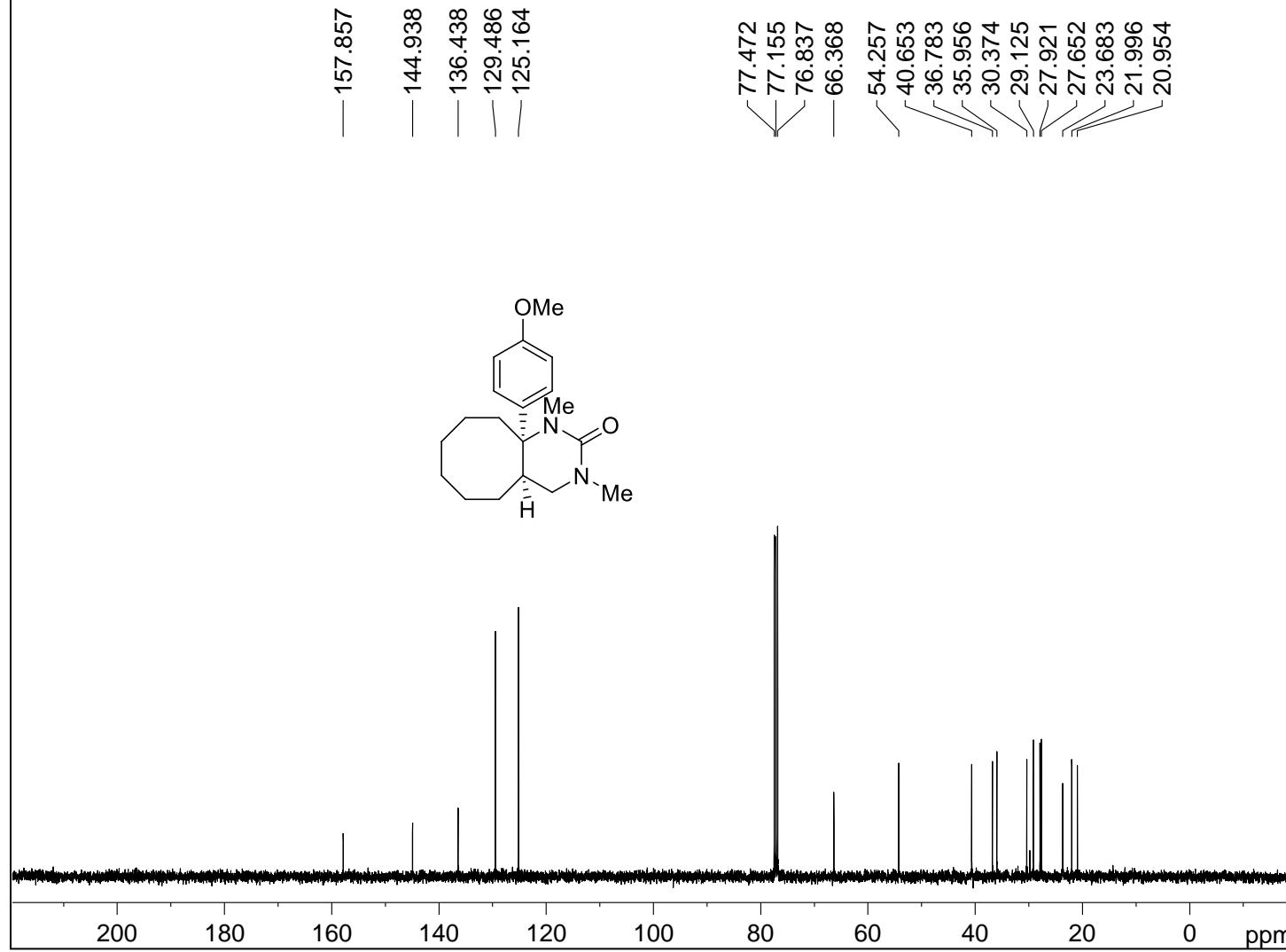
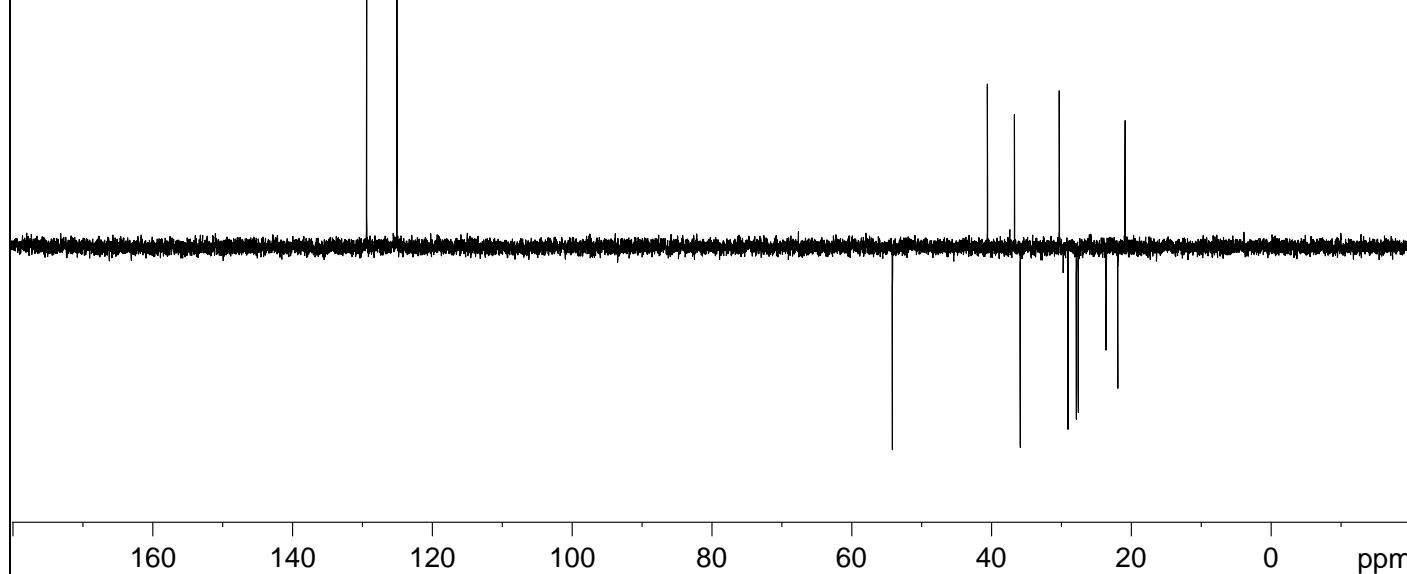
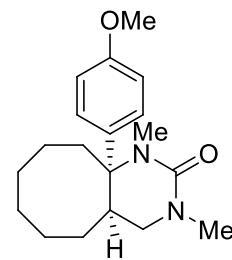


Figure 76 ¹³C NMR spectrum of compound 23a

lab sbpd-718
iitm_C13DEPT135 CDCl₃ /opt/topspin nmr 7

—129.381
—125.059

—54.153
—40.547
—36.678
—35.850
—30.269
—29.019
—27.815
—27.546
—23.576
—21.890
—20.849



Current Data Parameters
NAME pd-718
EXPNO 95
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190321
Time 13.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG deptspp135
TD 32768
SOLVENT CDCl₃
NS 64
DS 4
SWH 20161.291 Hz
FIDRES 0.615274 Hz
AQ 0.8126464 sec
RG 200.34
DW 24.800 usec
DE 6.50 usec
TE 296.4 K
CNST2 145.0000000
D1 1.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

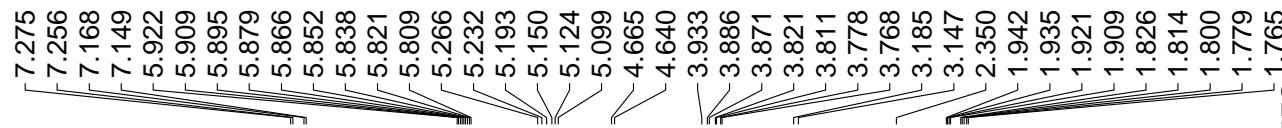
===== CHANNEL f1 =====
SFO1 100.6208166 MHz
NUC1 13C
P1 9.25 usec
P13 2000.00 usec
PLW0 0 W
PLW1 47.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.14429998 W

===== CHANNEL f2 =====
SFO2 400.1312797 MHz
NUC2 1H
CPDPRG[2] waltz16
P3 15.70 usec
P4 31.40 usec
PCPD2 90.00 usec
PLW2 7.75000000 W
PLW12 0.23583999 W

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure 77 DEPT-135 NMR spectrum of compound 23a

lab sbpd-798
iitm-Proton(-5to15) CDCl₃ /opt/topspin nmr 4

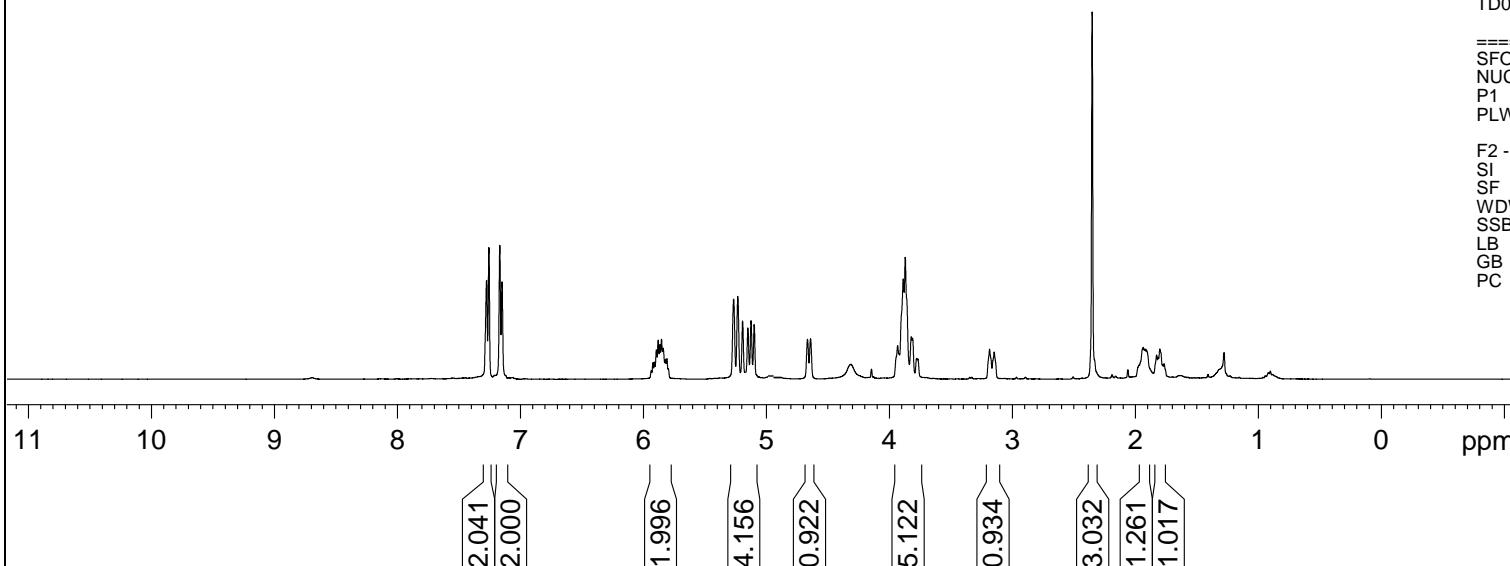


Current Data Parameters
NAME pd-798
EXPNO 45
PROCNO 1

F2 - Acquisition Parameters
Date_ 20200425
Time 18.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 153.13
DW 62.400 usec
DE 6.50 usec
TE 297.3 K
D1 0.5000000 sec
TDO 1

===== CHANNEL f1 =====
SFO1 400.1320007 MHz
NUC1 1H
P1 15.70 usec
PLW1 7.75000000 W

F2 - Processing parameters
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



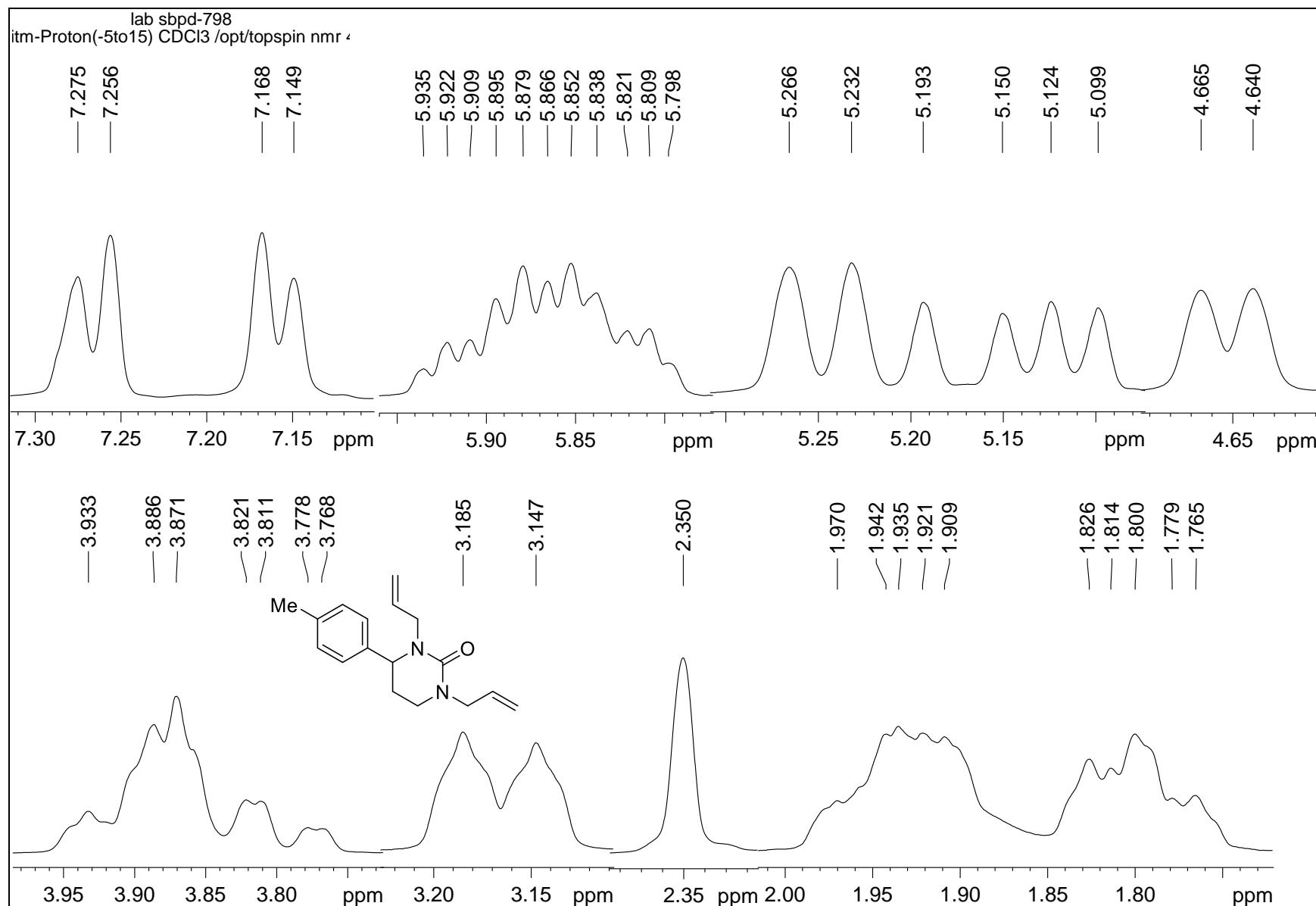


Figure 79 Expanded ¹H NMR spectrum of compound 24

lab sbpd-798
iitm_carbonshort CDCl₃ /opt/topspin nmr 4

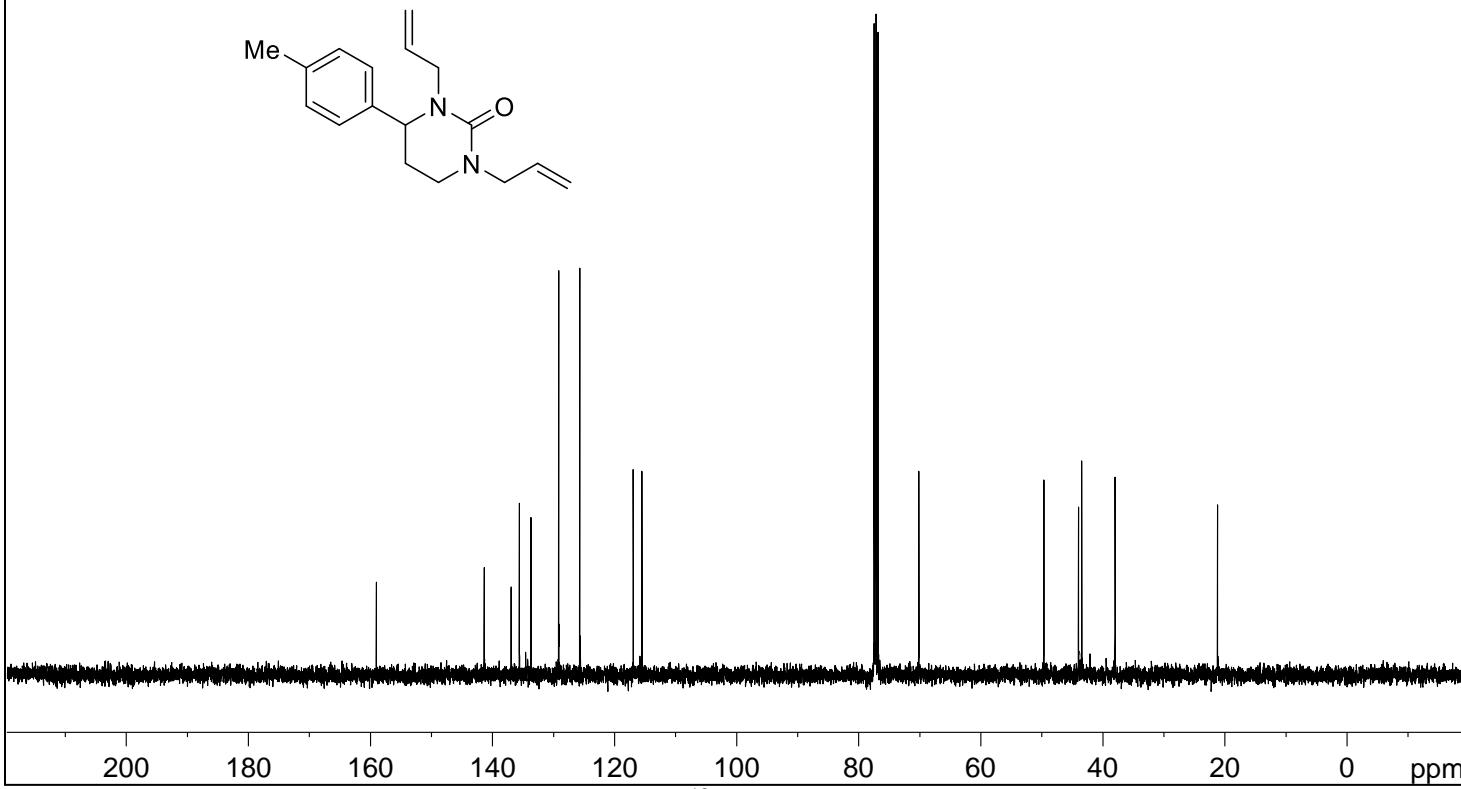
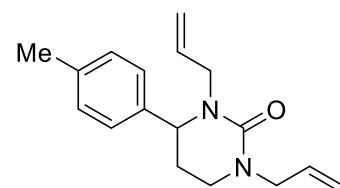
— 159.026

141.356
136.948
135.596
133.693
129.155
125.704
116.943
115.529

77.476
77.158
76.840
70.147

49.657
43.982
43.462
37.990

— 21.202



Current Data Parameters
NAME pd-798
EXPNO 46
PROCNO 1

F2 - Acquisition Parameters
Date 20200425
Time 18.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 16540
SOLVENT CDCl₃
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 1.453353 Hz
AQ 0.3440320 sec
RG 200.34
DW 20.800 usec
DE 6.50 usec
TE 297.4 K
D1 1.0000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228289 MHz
NUC1 ¹³C
P1 9.25 usec
PLW1 47.00000000 W

===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 ¹H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 7.75000000 W
PLW12 0.23583999 W
PLW13 0.11863000 W

F2 - Processing parameters
SI 32768
SF 100.6127584 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

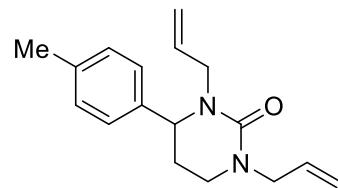
Figure 80 ¹³C NMR spectrum of compound 24

lab sbpd-798
iitm_C13DEPT135 CDCl₃ /opt/topspin nmr 4

135.491
133.589
129.051
125.600
116.838
115.424

49.554
43.878
43.358
37.885

21.098



Current Data Parameters
NAME pd-798
EXPNO 47
PROCNO 1

F2 - Acquisition Parameters
Date 20200425
Time 18.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG deptsps135
TD 32768
SOLVENT CDCl₃
NS 64
DS 4
SWH 20161.291 Hz
FIDRES 0.615274 Hz
AQ 0.8126464 sec
RG 200.34
DW 24.800 usec
DE 6.50 usec
TE 297.5 K
CNST2 145.0000000
D1 1.0000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TDO 1

===== CHANNEL f1 =====
SFO1 100.6208166 MHz
NUC1 ¹³C
P1 9.25 usec
P13 2000.00 usec
PLW0 0 W
PLW1 47.00000000 W
SPNAM[5] Crp60comp.4
SPOALS5 0.500
SPOFFS5 0 Hz
SPW5 6.14429998 W

===== CHANNEL f2 =====
SFO2 400.1312797 MHz
NUC2 ¹H
CPDPRG[2] waltz16
P3 15.70 usec
P4 31.40 usec
PCPD2 90.00 usec
PLW2 7.75000000 W
PLW12 0.23583999 W

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

160 140 120 100 80 60 40 20 0 ppm

Figure 81 DEPT-135 NMR spectrum of compound 24

lab sbpd-798-1
PROTON CDCl₃ /opt/topspin nmr 3

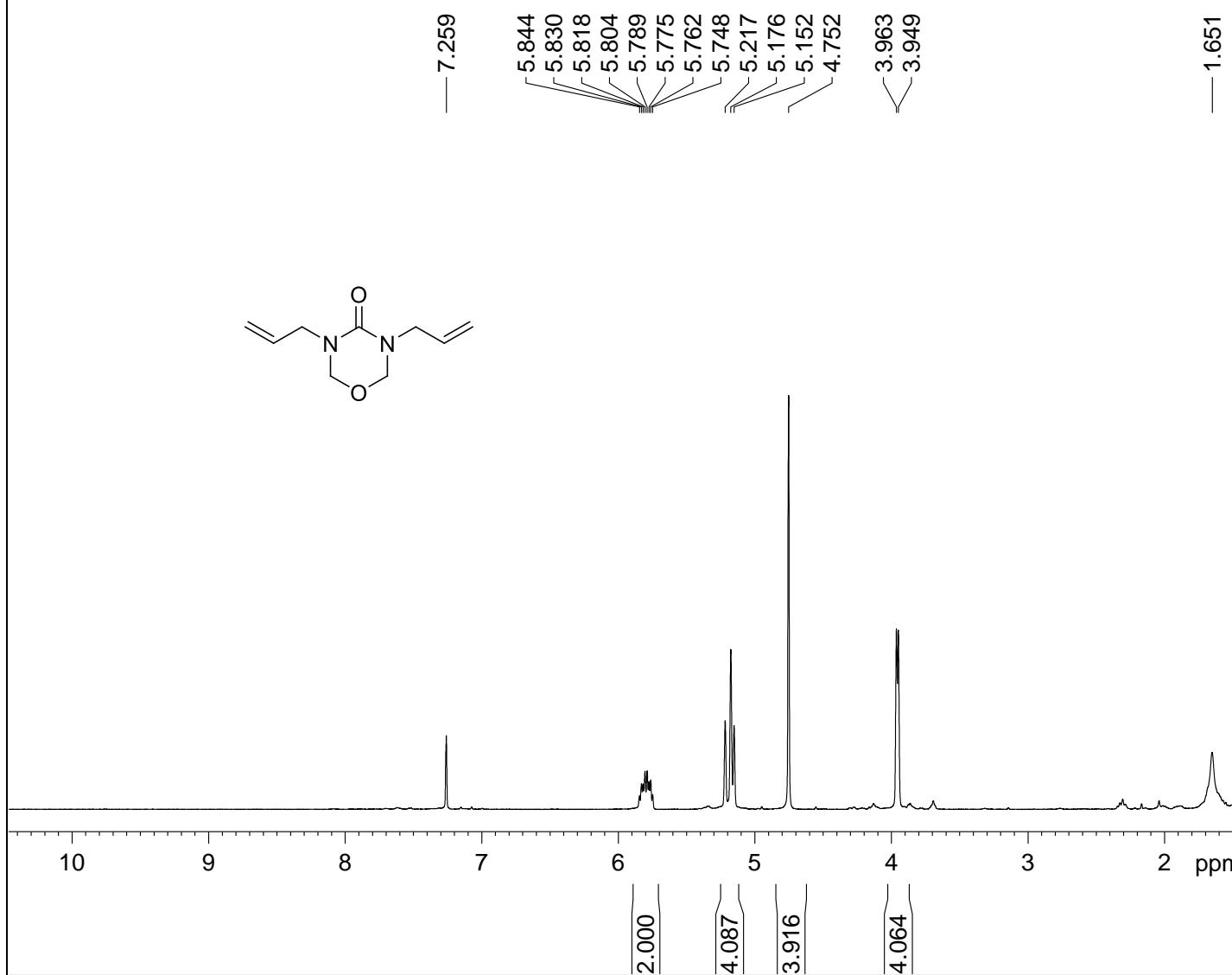
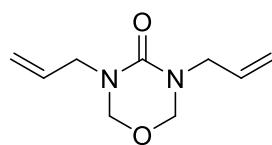


Figure 82 ¹H NMR spectrum of compound 25

Current Data Parameters
NAME PD-798-1
EXPNO 77
PROCNO 1

F2 - Acquisition Parameters
Date 20200430
Time 12.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 200.34
DW 62.400 usec
DE 6.50 usec
TE 297.3 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 400.1324710 MHz
NUC1 1H
P1 15.70 usec
PLW1 7.7500000 W

F2 - Processing parameters
SI 65536
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

lab sbpd-798-2
iitm-Proton(-5to15) CDCl₃ /opt/topspin nmr 10

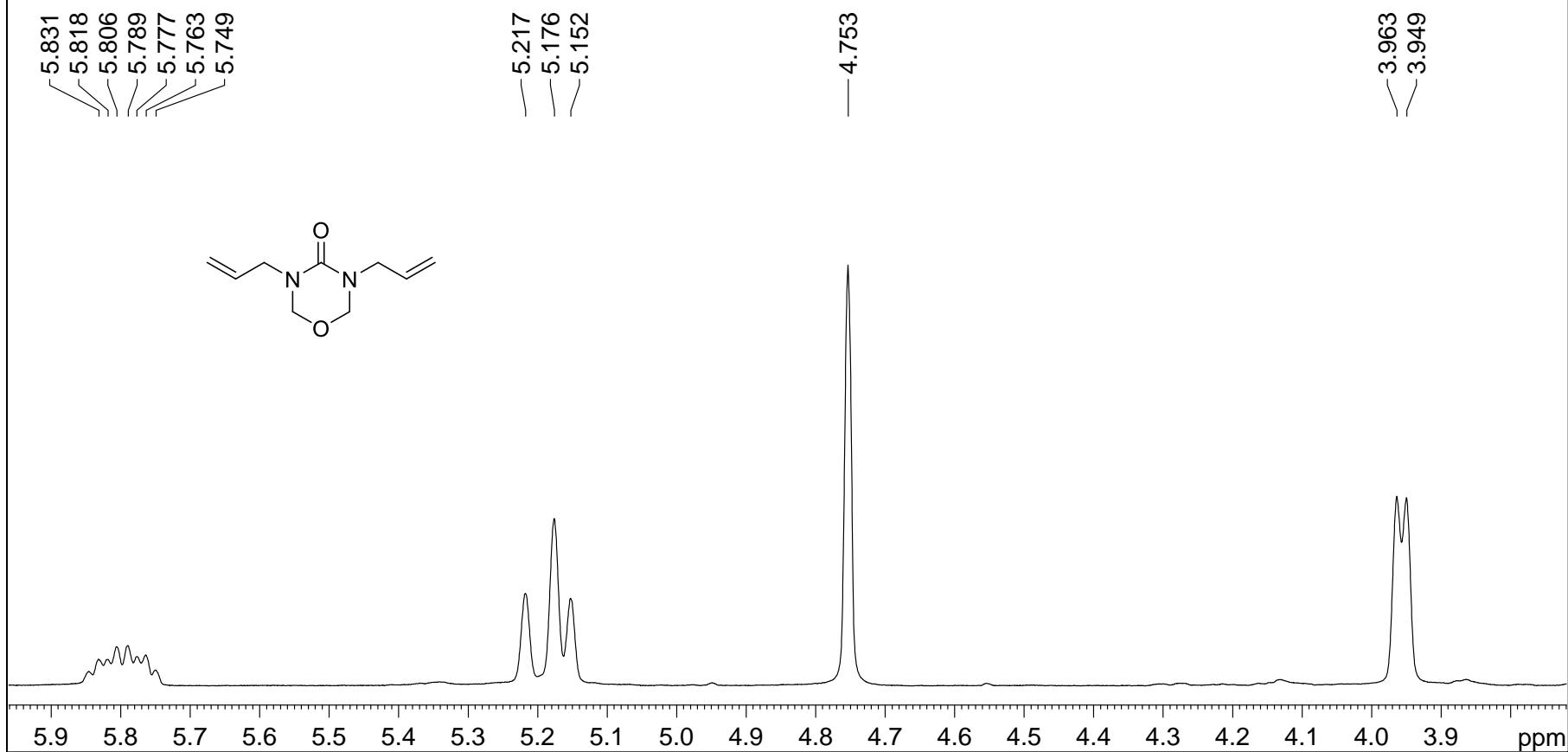


Figure 83 Expanded ¹H NMR spectrum of compound 25

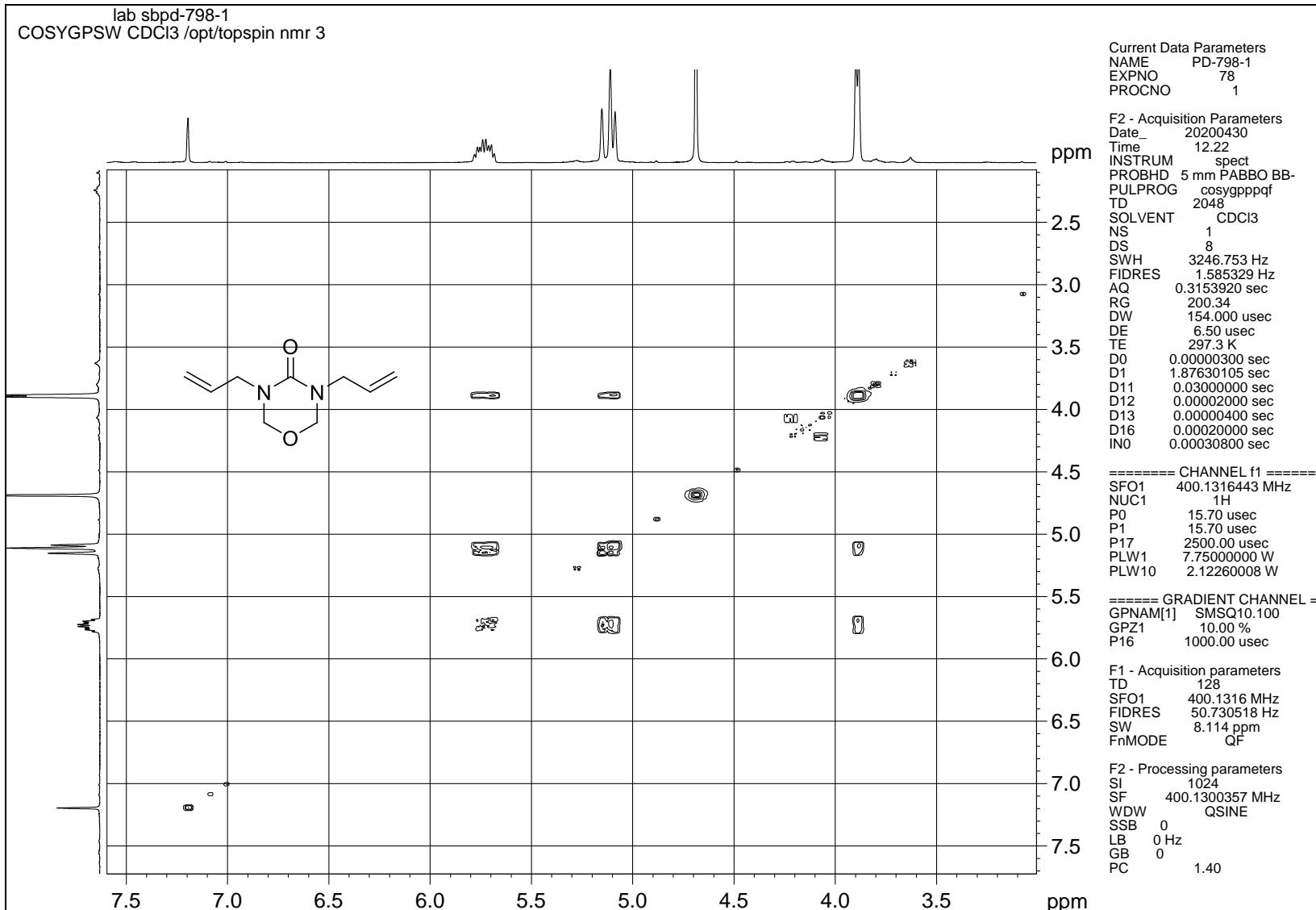


Figure 84 ¹H-¹H COSY NMR spectrum of compound 25

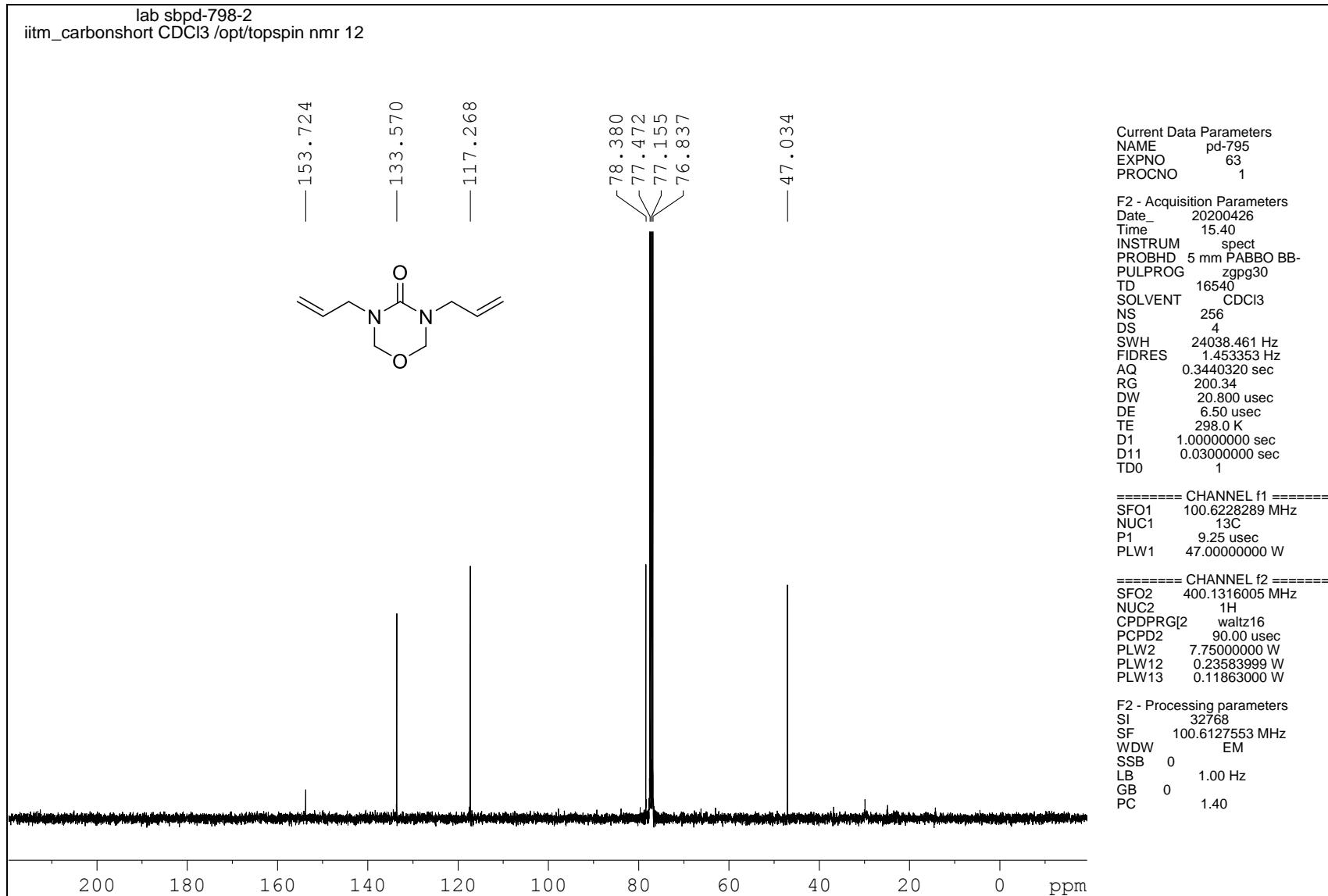


Figure 85 ¹³C NMR spectrum of compound 25

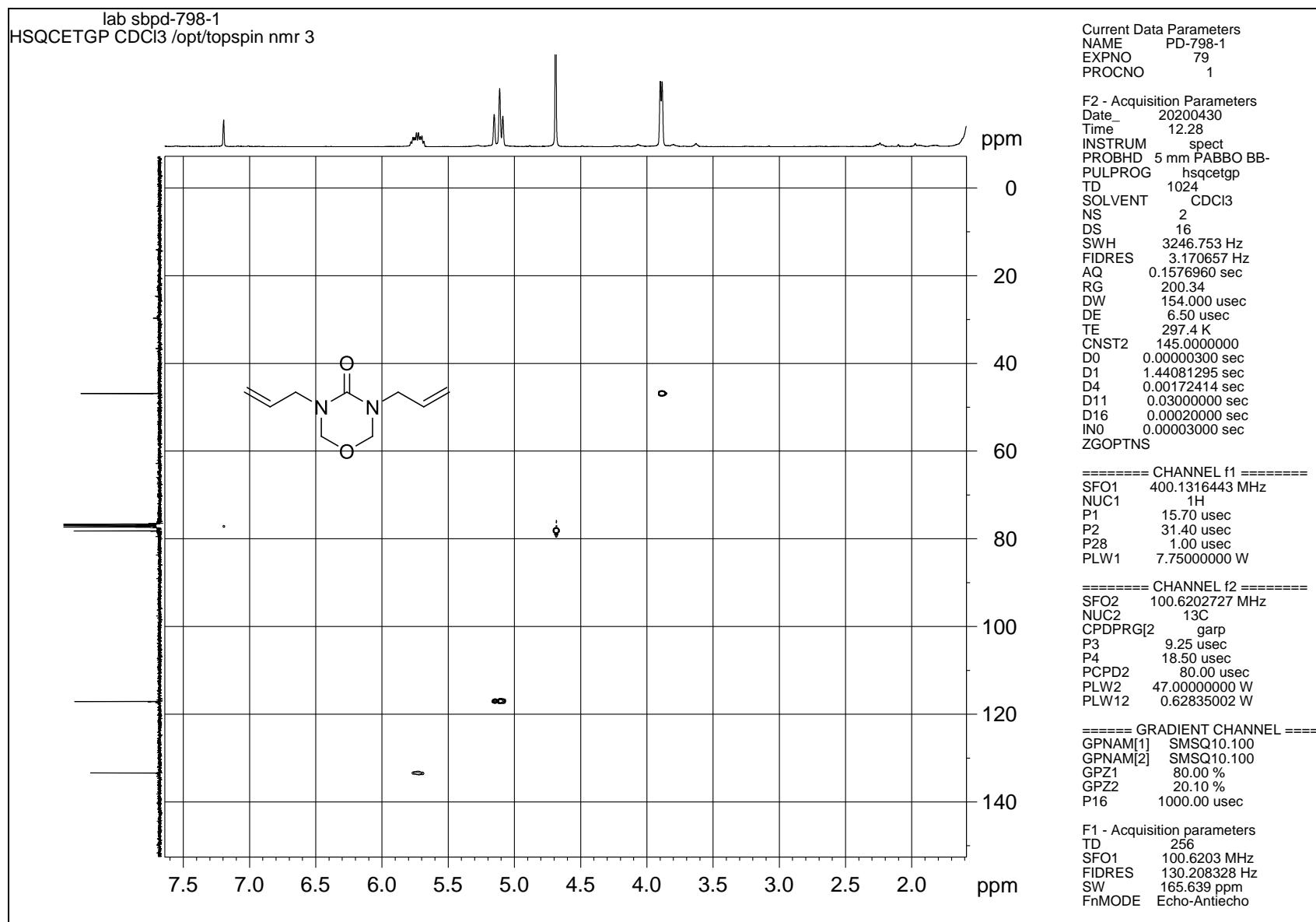
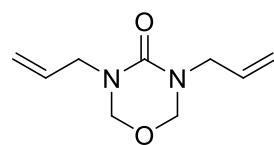


Figure 86 ¹H-¹³C HSQC NMR spectrum of compound 25

lab sbpd-798-2
itm_C13DEPT135 CDCl₃ /opt/topspin nmr 10



Current Data Parameters
NAME pd-798-2
EXPNO 56
PROCNO 1

F2 - Acquisition Parameters
Date 20200426
Time 13.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG deptsp135
TD 32768
SOLVENT CDCl₃
NS 250
DS 4
SWH 20161.291 Hz
FIDRES 0.615274 Hz
AQ 0.8126464 sec
RG 200.34
DW 24.800 usec
DE 6.50 usec
TE 298.0 K
CNST2 145.0000000
D1 1.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6208166 MHz
NUC1 ¹³C
P1 9.25 usec
P13 2000.00 usec
PLW0 0 W
PLW1 47.00000000 W
SPNAM[5] Crp60comp.4
SPOALS5 0.500
SPOFFS5 0 Hz
SPW5 6.14429998 W

===== CHANNEL f2 =====
SFO2 400.1312797 MHz
NUC2 ¹H
CPDPKG[2] waltz16
P3 15.70 usec
P4 31.40 usec
PCPD2 90.00 usec
PLW2 7.75000000 W
PLW12 0.23583999 W

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure 87 DEPT-135 NMR spectrum of compound 25

lab sbpd-795
itm-Proton(-5to15) CDCl₃ /opt/topspin nmr 13

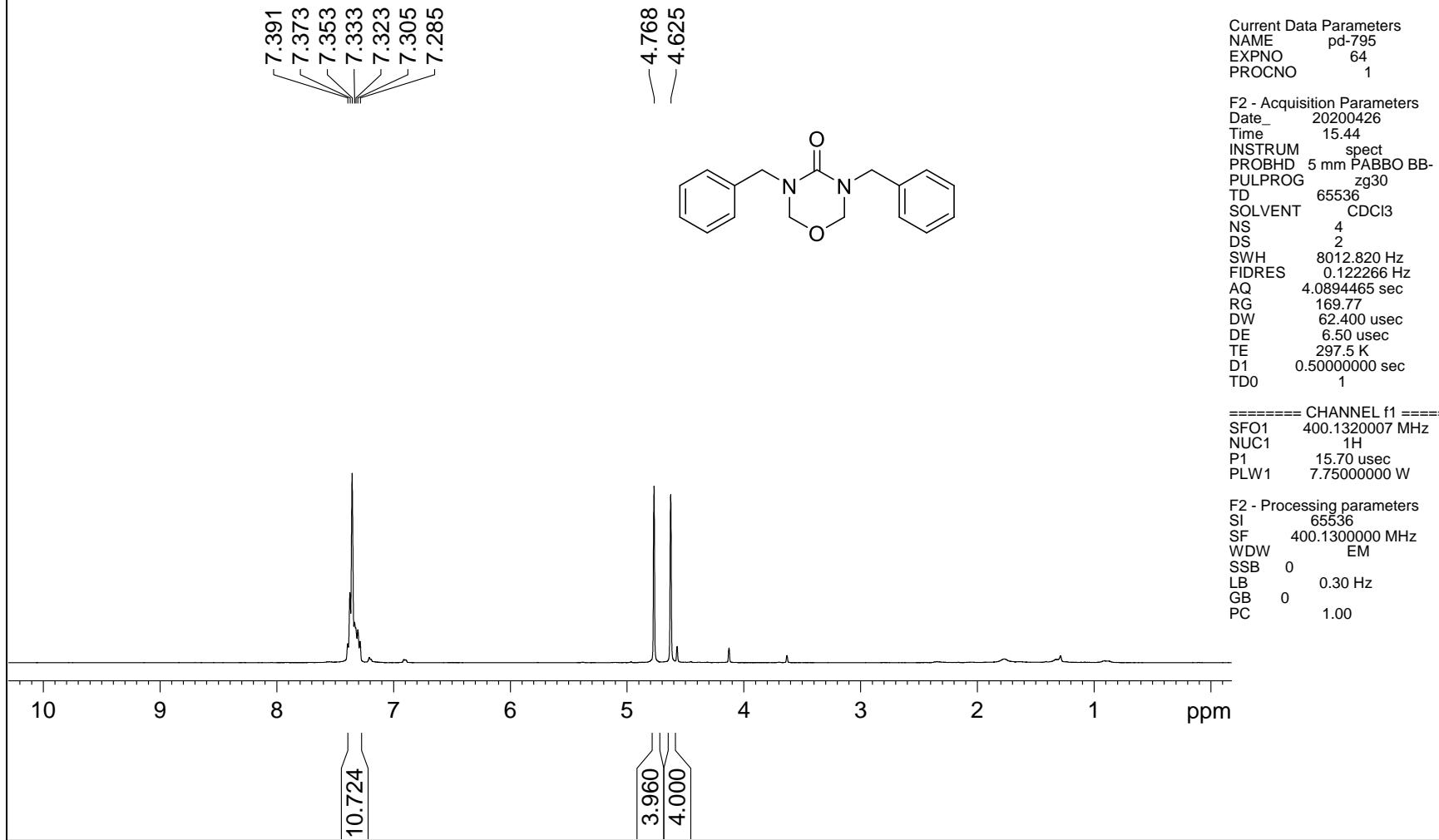


Figure 88 ¹H NMR spectrum of compound 27

lab sbpd-795
COSYGPSW CDCl₃ /opt/topspin nmr 1

Current Data Parameters
NAME pd-795
EXPNO 72
PROCNO 1

F2 - Acquisition Parameters
Date 20200430
Time 11.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG cosypppqf
TD 2048
SOLVENT CDCl₃
NS 1
DS 8
SWH 3184.713 Hz
FIDRES 1.555036 Hz
AQ 0.3215360 sec
RG 60.89
DW 157.000 usec
DE 6.50 usec
TE 297.3 K
D0 0.00000300 sec
D1 1.87015700 sec
D11 0.03000000 sec
D12 0.00002000 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00031400 sec

===== CHANNEL f1 =====
SFO1 400.1317019 MHz
NUC1 1H
P0 15.70 usec
P1 15.70 usec
P17 2500.00 usec
PLW1 7.7500000 W
PLW10 2.12260008 W

===== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPZ1 10.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 128
SFO1 400.1317 MHz
FIDRES 49.761147 Hz
SW 7.959 ppm
FnMODE QF

F2 - Processing parameters
SI 1024
SF 400.1300000 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0
PC 1.40

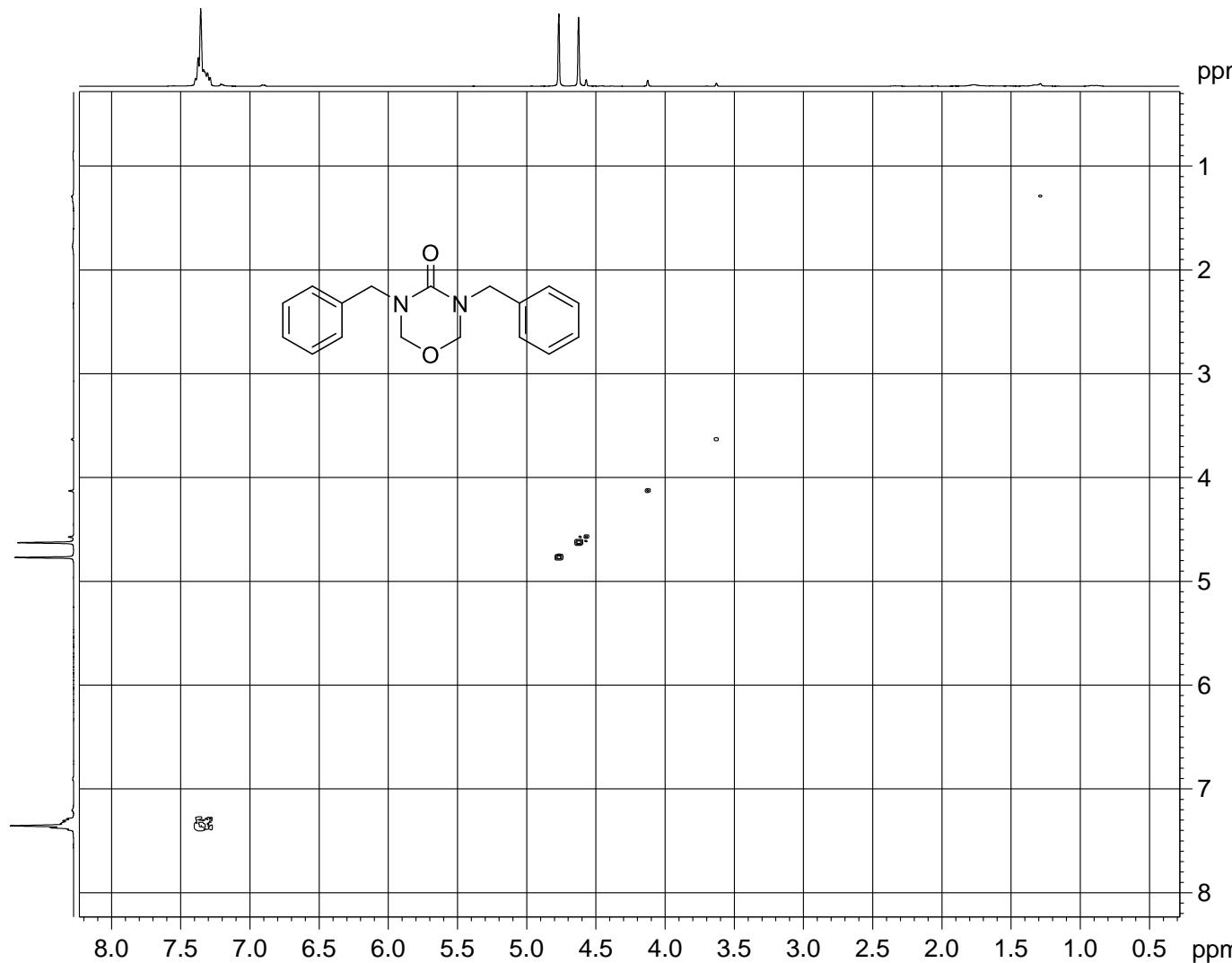


Figure 89 ¹H-¹H COSY NMR spectrum of compound 27

lab sbpd-795
itm_carbonshort CDCl₃ /opt/topspin nmr 13

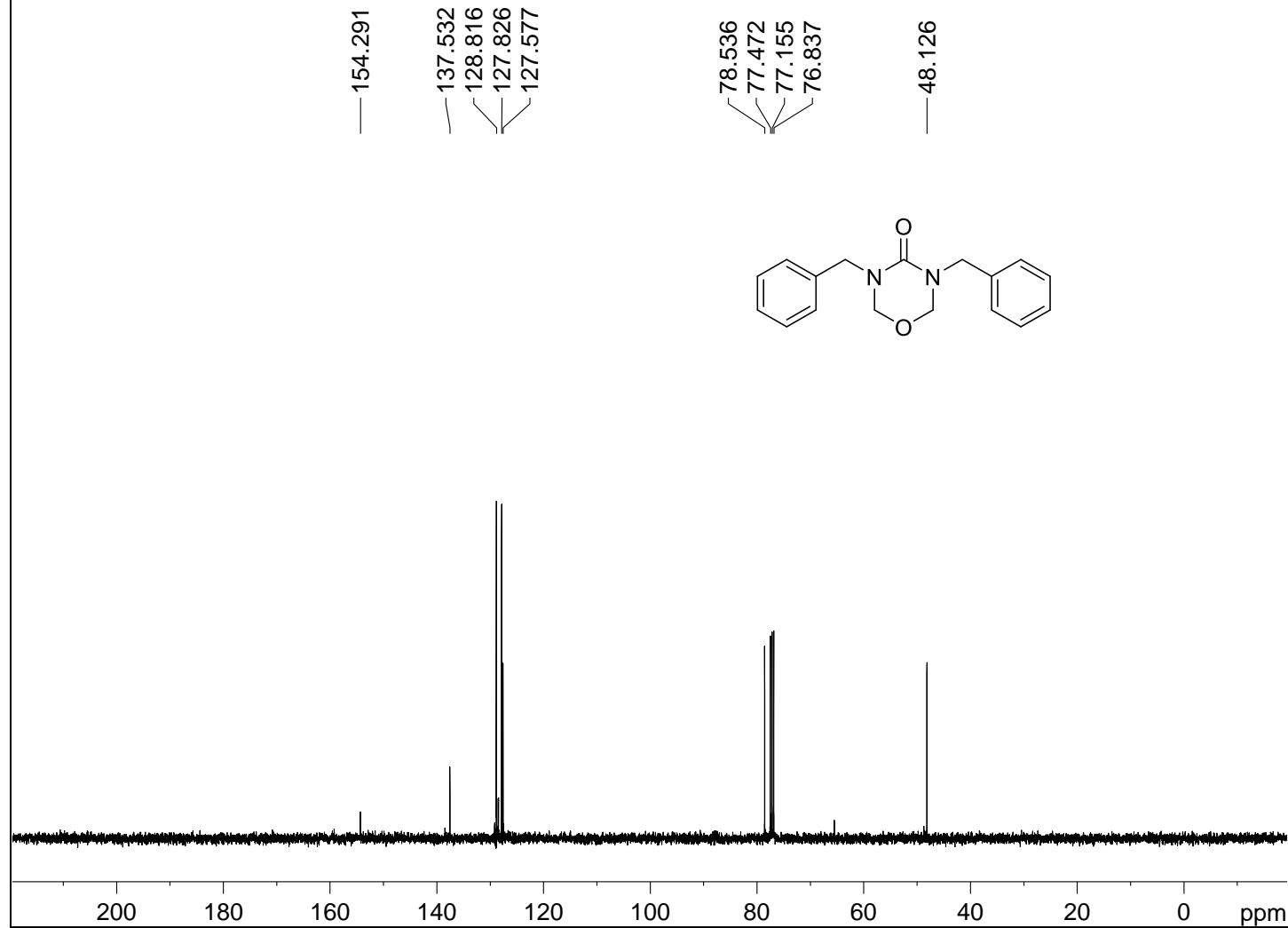


Figure 90 ¹³C NMR spectrum of compound 27

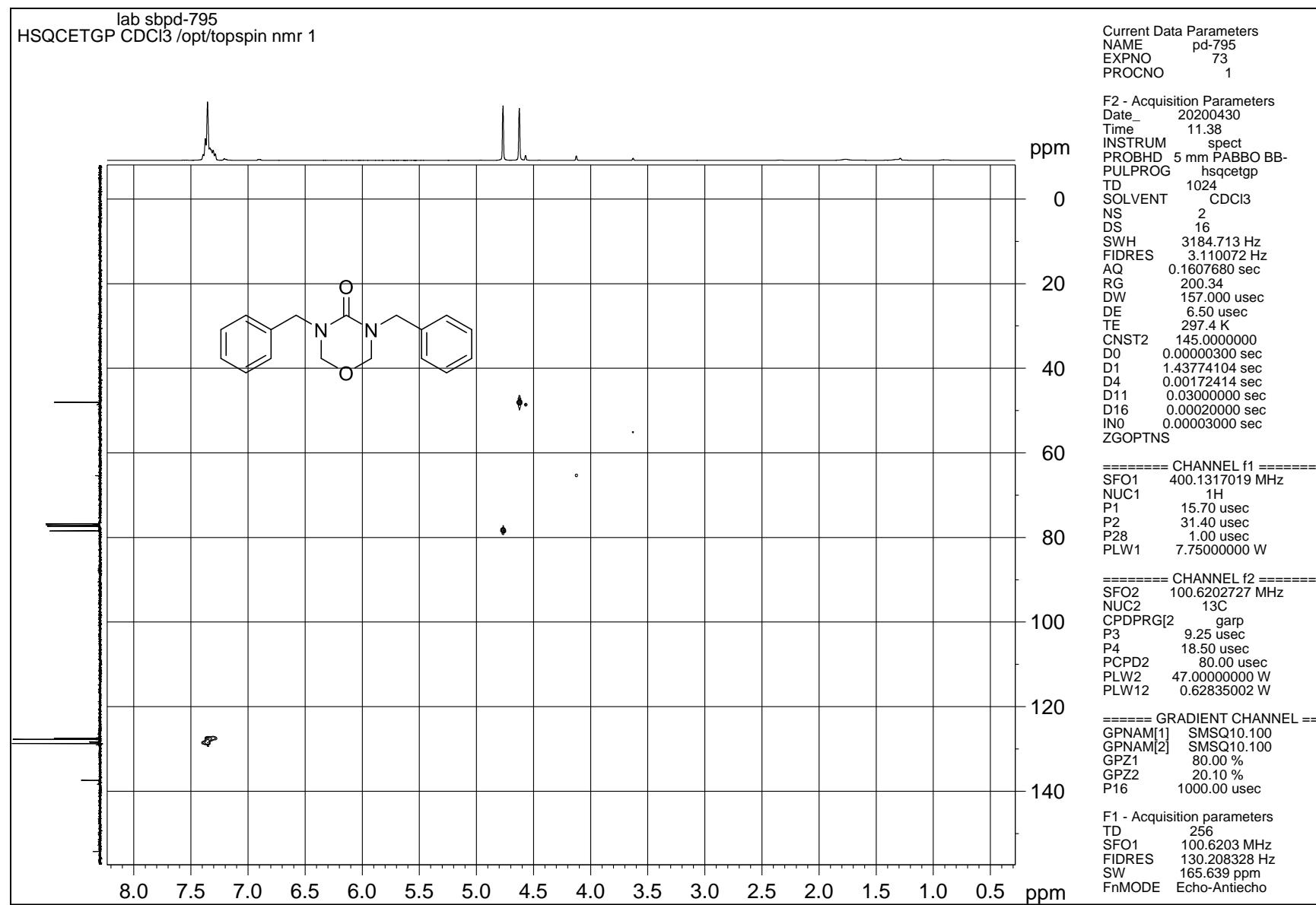


Figure 91 ¹H-¹³C HSQC NMR spectrum of compound 27

lab sbpd-795
itm_C13DEPT135 CDCl₃ /opt/topspin nmr 13

Current Data Parameters
NAME pd-795
EXPNO 66
PROCNO 1

F2 - Acquisition Parameters
Date 20200426
Time 15.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG deptsp135
TD 32768
SOLVENT CDCl₃
NS 32
DS 4
SWH 20161.291 Hz
FIDRES 0.615274 Hz
AQ 0.8126464 sec
RG 200.34
DW 24.800 usec
DE 6.50 usec
TE 297.9 K
CNST2 145.0000000
D1 1.0000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6208166 MHz
NUC1 ¹³C
P1 9.25 usec
P13 2000.00 usec
PLW0 0 W
PLW1 47.00000000 W
SPNAM[5] Crp60comp.4
SPOALS5 0.500
SPOFFS5 0 Hz
SPW5 6.14429998 W

===== CHANNEL f2 =====
SFO2 400.1312797 MHz
NUC2 ¹H
CPDPRG[2] waltz16
P3 15.70 usec
P4 31.40 usec
PCPD2 90.00 usec
PLW2 7.75000000 W
PLW12 0.23583999 W

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

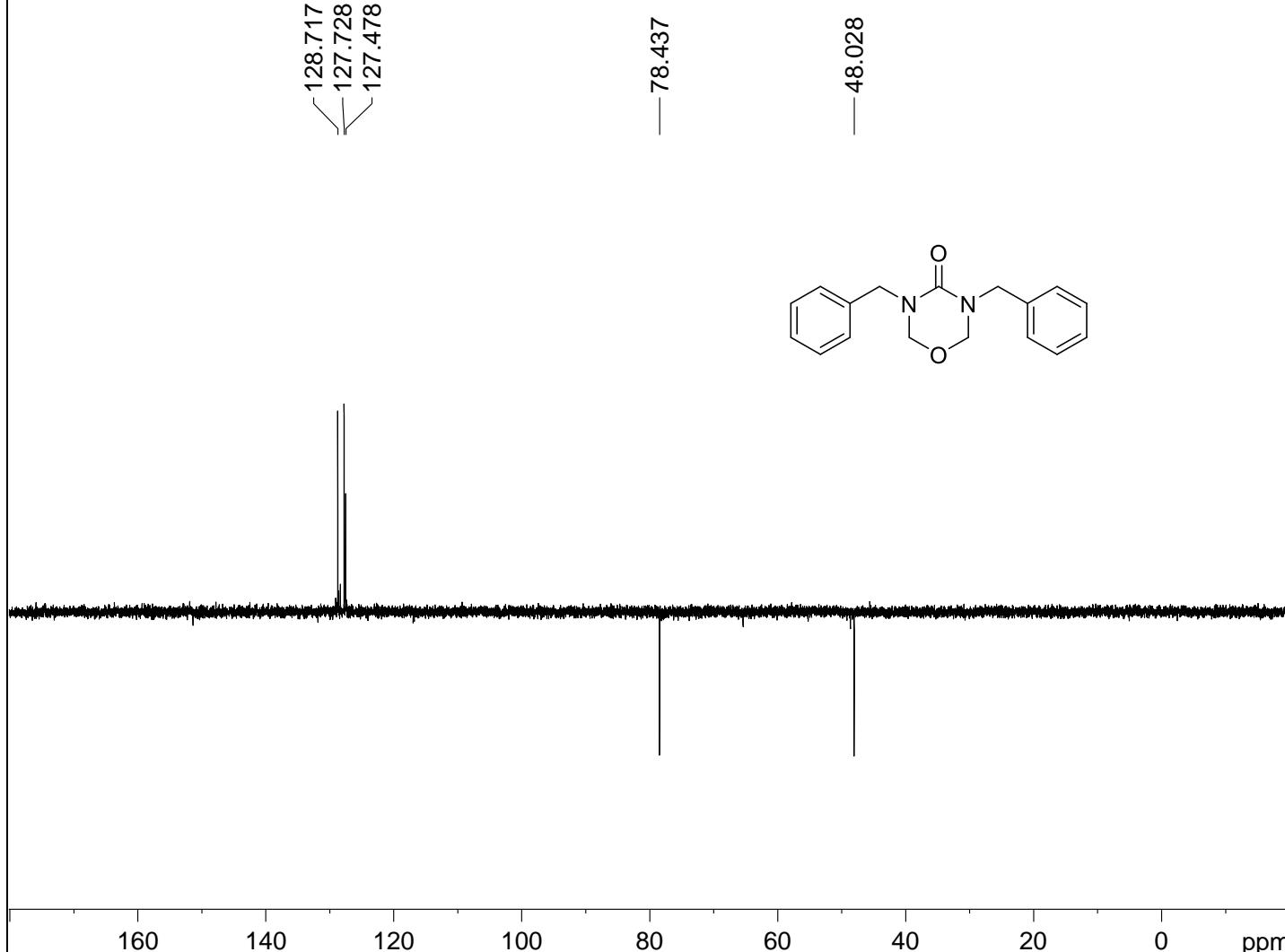


Figure 92 DEPT-135 NMR spectrum of compound 27

lab sbpd-742
iitm-Proton(-5to15) CDCl₃ /opt/topspin nmr 5

7.177
7.158
7.055
7.035

3.687
3.665
2.888
2.859
2.689
2.683
2.661
2.655
2.478
2.455
2.251
2.091
2.084
2.055
2.031
2.025
1.885
1.848
1.837
1.817
1.806
1.559
1.554
1.526
1.521

Current Data Parameters
NAME pd-742
EXPNO 176
PROCNO 1

F2 - Acquisition Parameters
Date_ 20200319
Time 22.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 3
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 88.51
DW 62.400 usec
DE 6.50 usec
TE 297.6 K
D1 0.5000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 400.1320007 MHz
NUC1 1H
P1 15.70 usec
PLW1 7.7500000 W

F2 - Processing parameters
SI 65536
SF 400.1300382 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

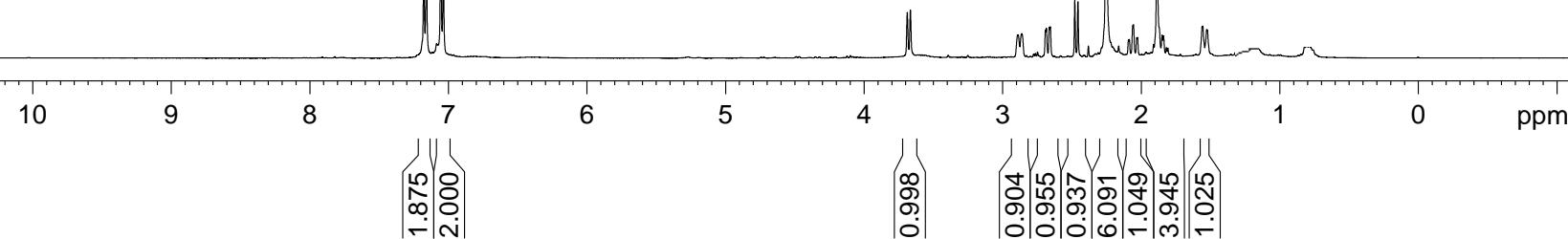


Figure 93 ¹H NMR spectrum of compound 8b

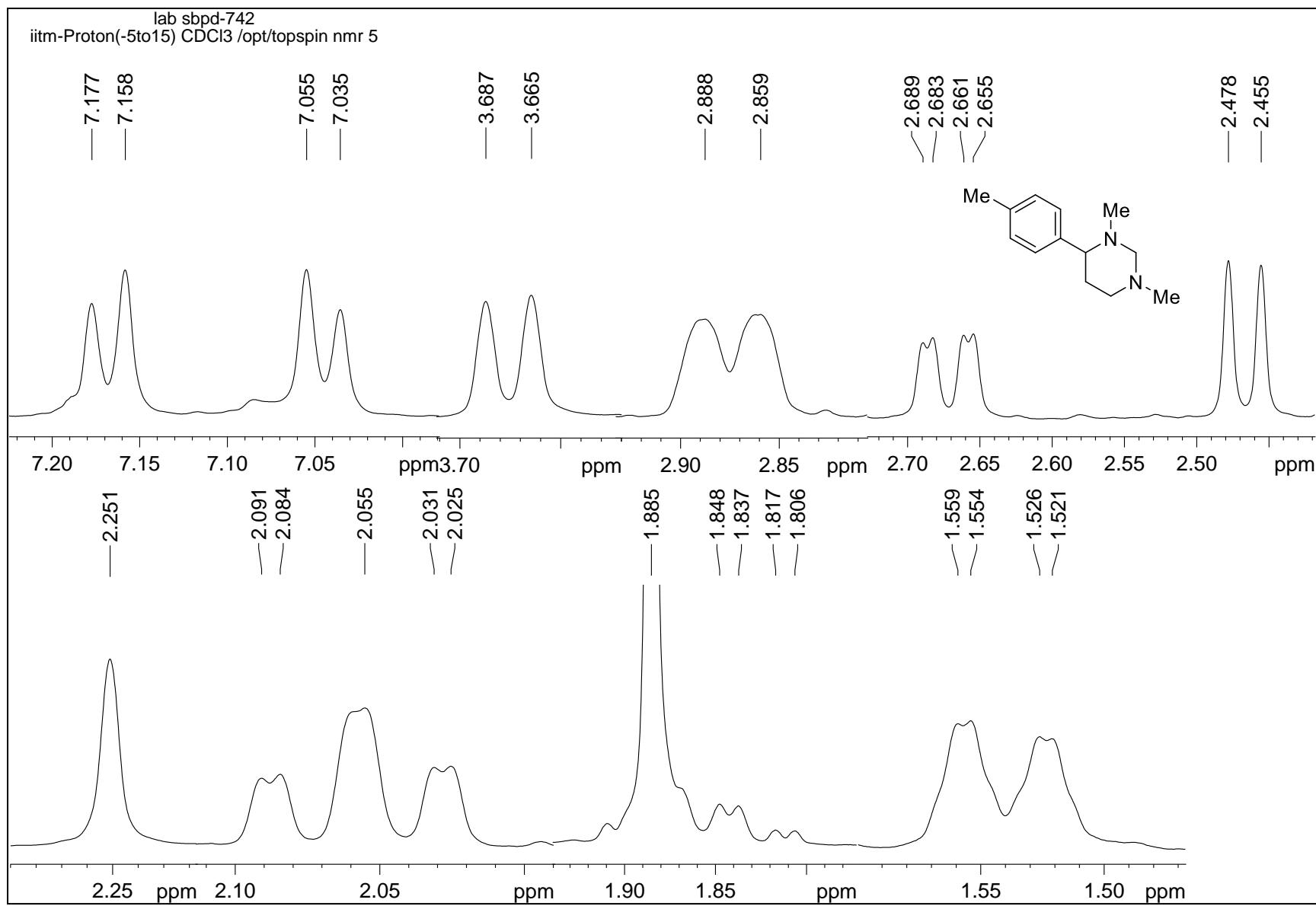
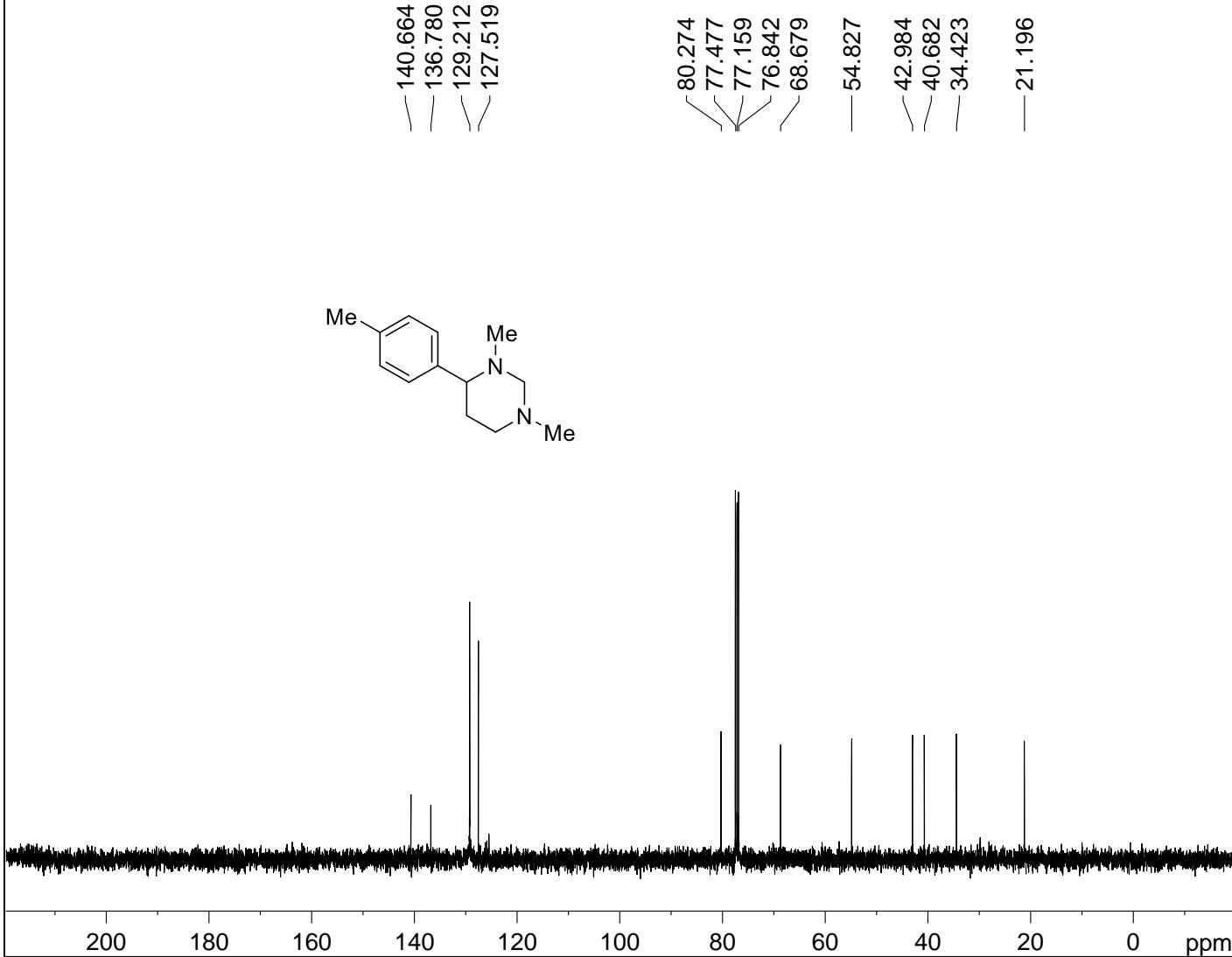
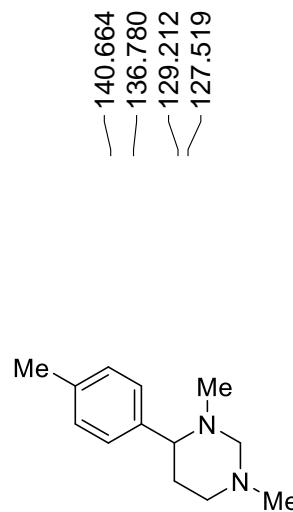


Figure 94 Expanded ¹H NMR spectrum of compound 8b

lab sbpd-742
itm_carbonshort CDCl₃ /opt/topspin nmr 7



Current Data Parameters
NAME pd
EXPNO 228
PROCNO 1

F2 - Acquisition Parameters
Date_ 20200330
Time 10.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 16540
SOLVENT CDCl₃
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 1.453353 Hz
AQ 0.3440320 sec
RG 200.34
DW 20.800 usec
DE 6.50 usec
TE 297.2 K
D1 1.0000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228289 MHz
NUC1 ¹³C
P1 9.25 usec
PLW1 47.00000000 W

===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 ¹H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 7.75000000 W
PLW12 0.23583999 W
PLW13 0.11863000 W

F2 - Processing parameters
SI 32768
SF 100.6127599 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure 95 ¹³C NMR spectrum of compound 8b

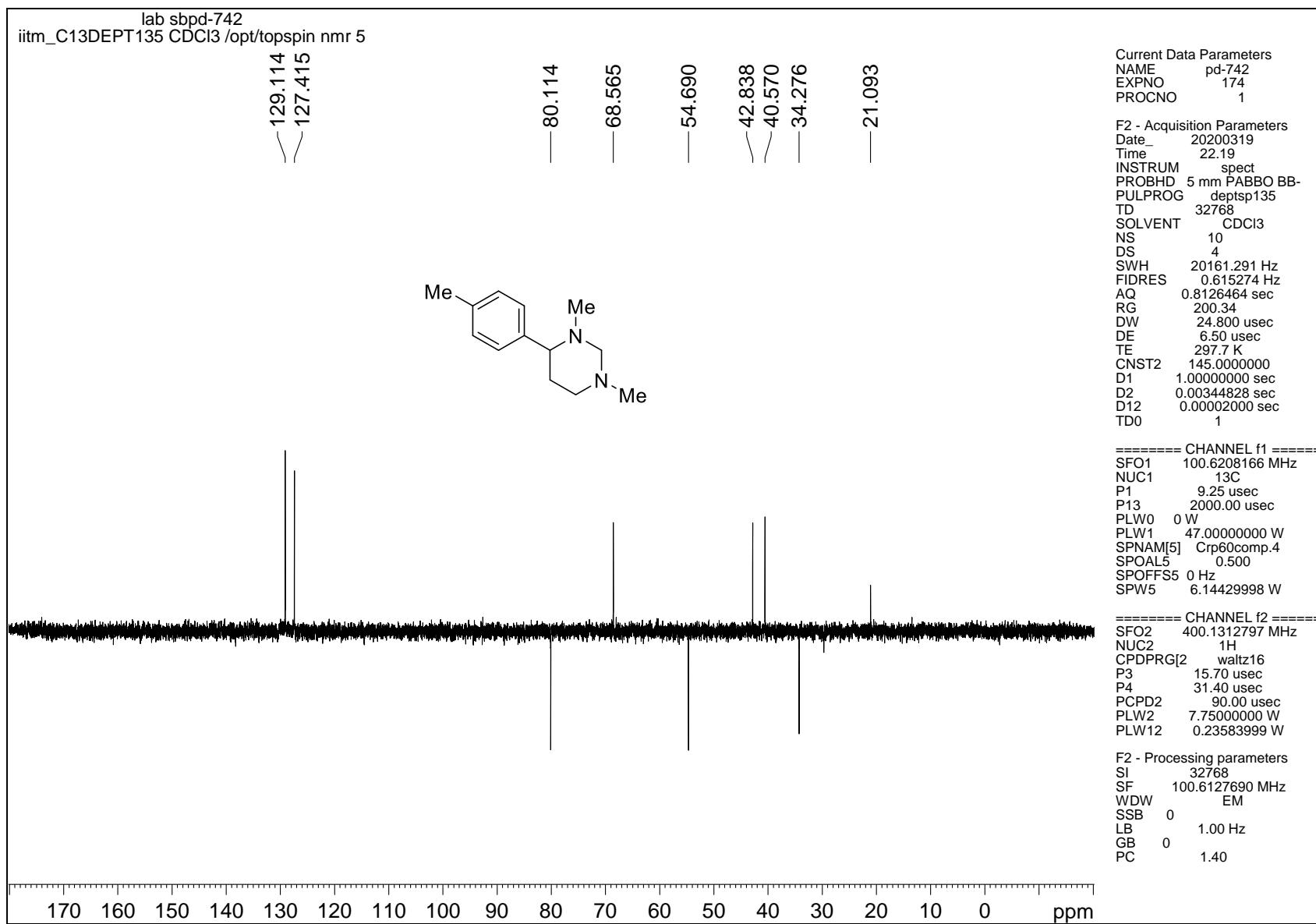


Figure 96 DEPT-135 NMR spectrum of compound 8b

lab sbpd-749
itm-Proton(-5to15) MeOD /opt/topspin nmr 15

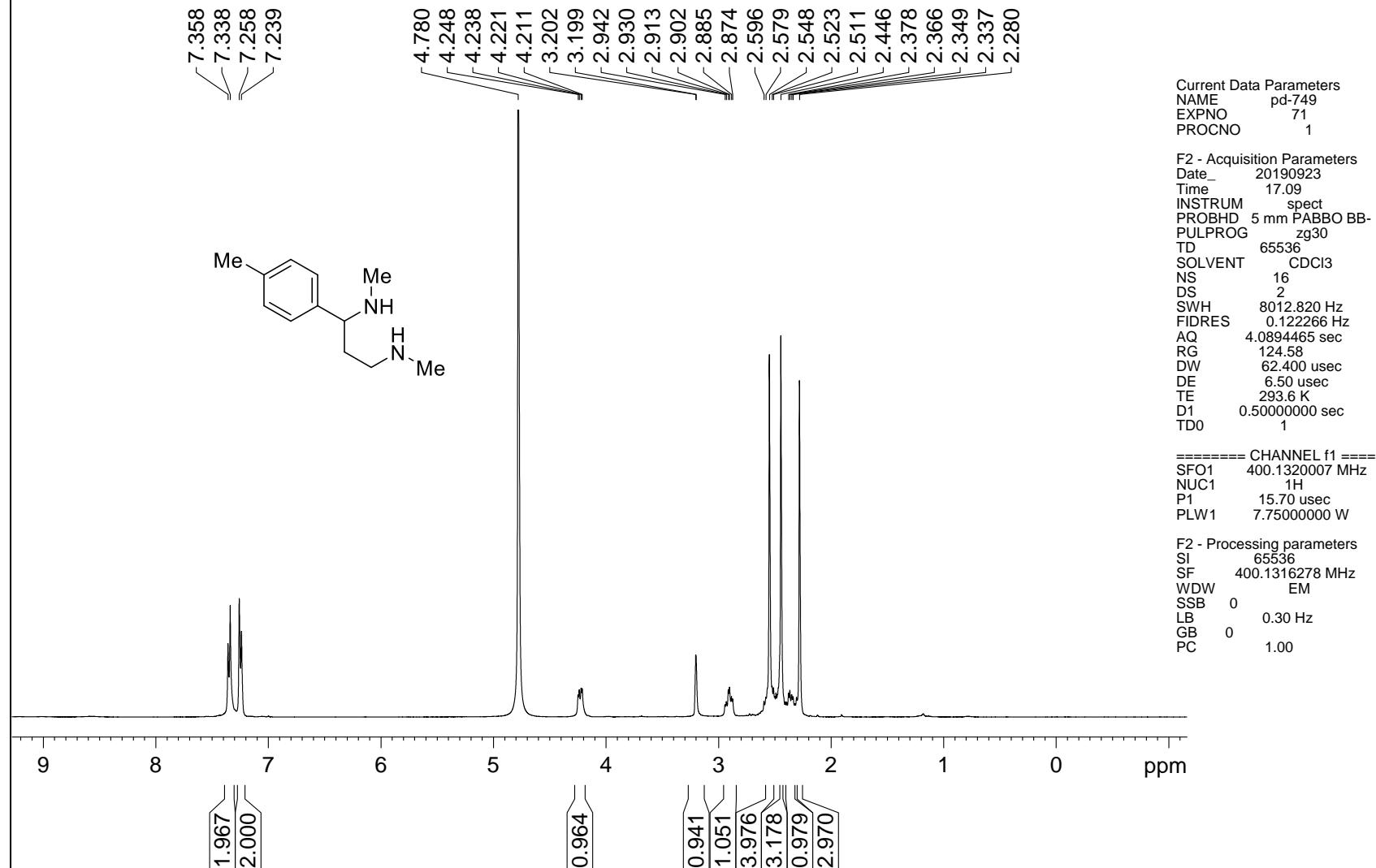


Figure 97 ^1H NMR spectrum of compound 8c

lab sbpd-749
iitm-Proton(-5to15) MeOD /opt/topspin nmr 15

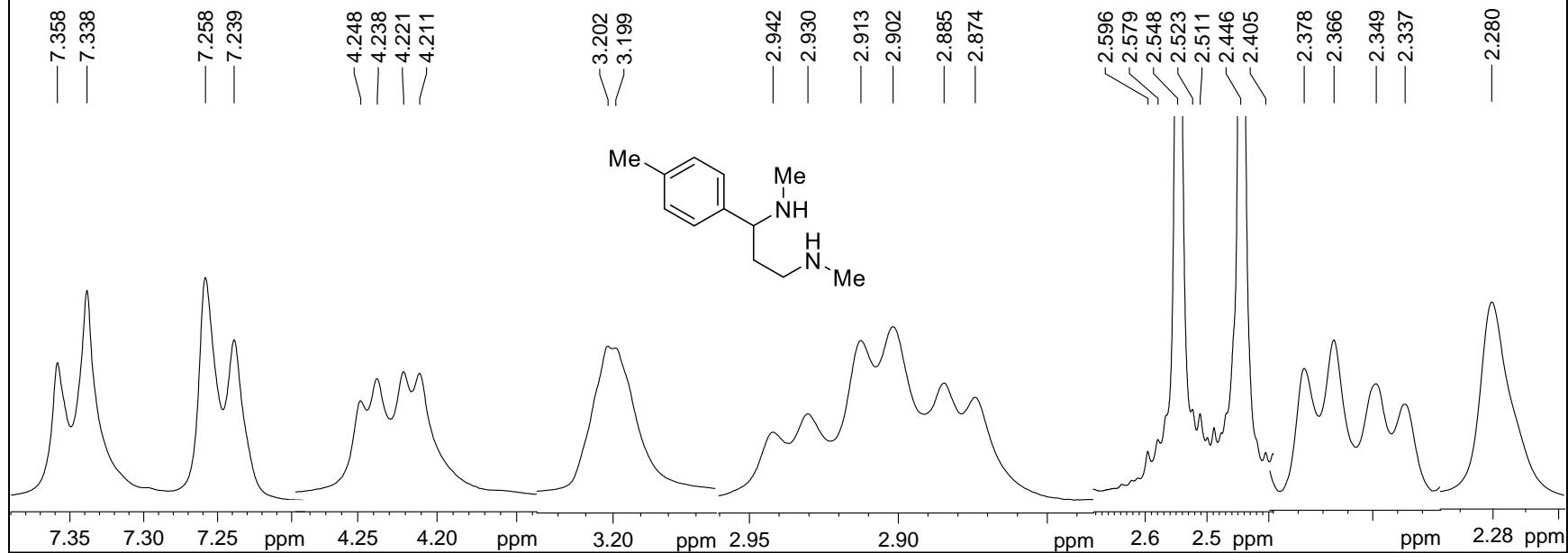


Figure 98 Expanded ¹H NMR spectrum of compound 8c

lab sbpd-749
iitm_carbonshort MeOD /opt/topspin nmr 16

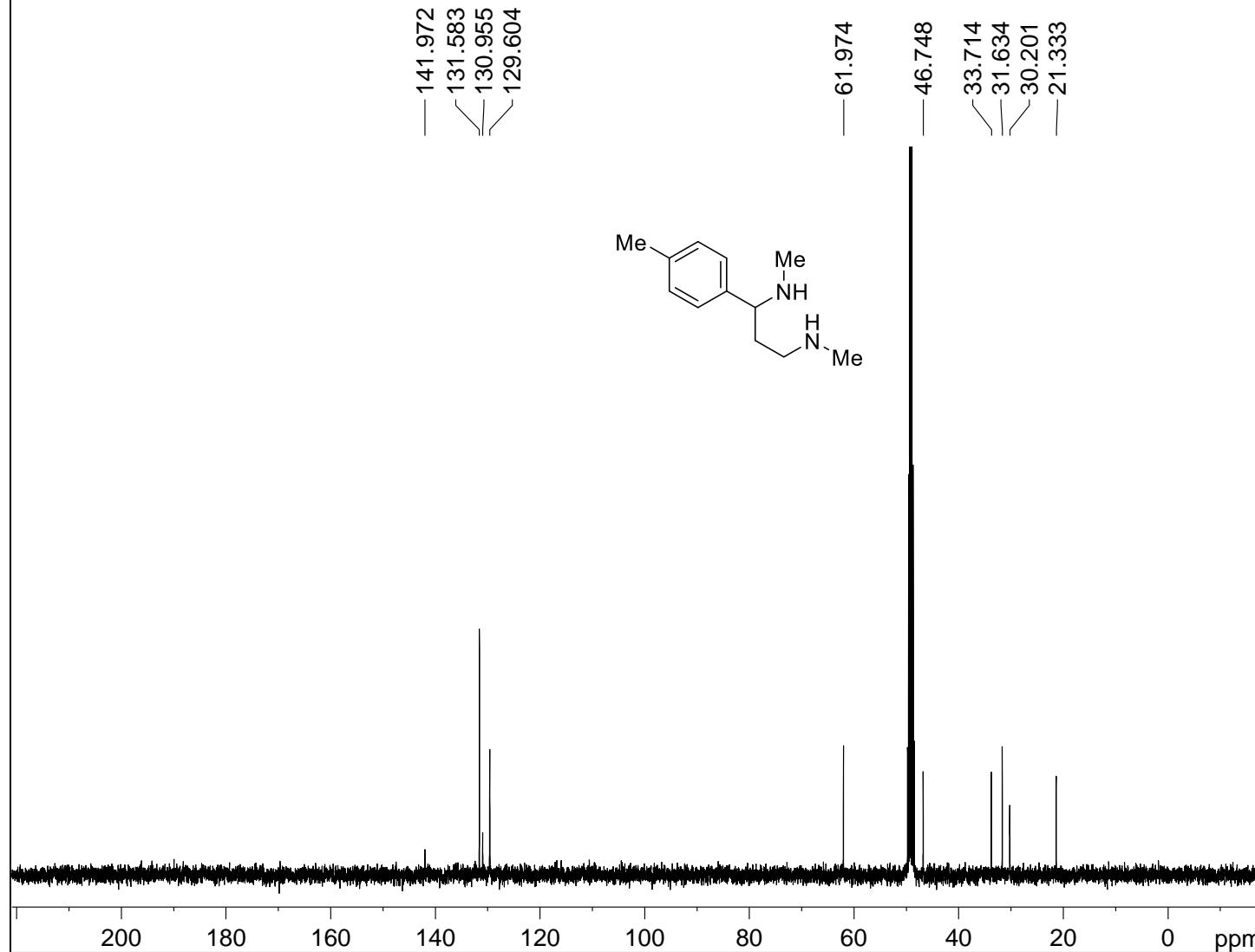


Figure 99 ^{13}C NMR spectrum of compound 8c

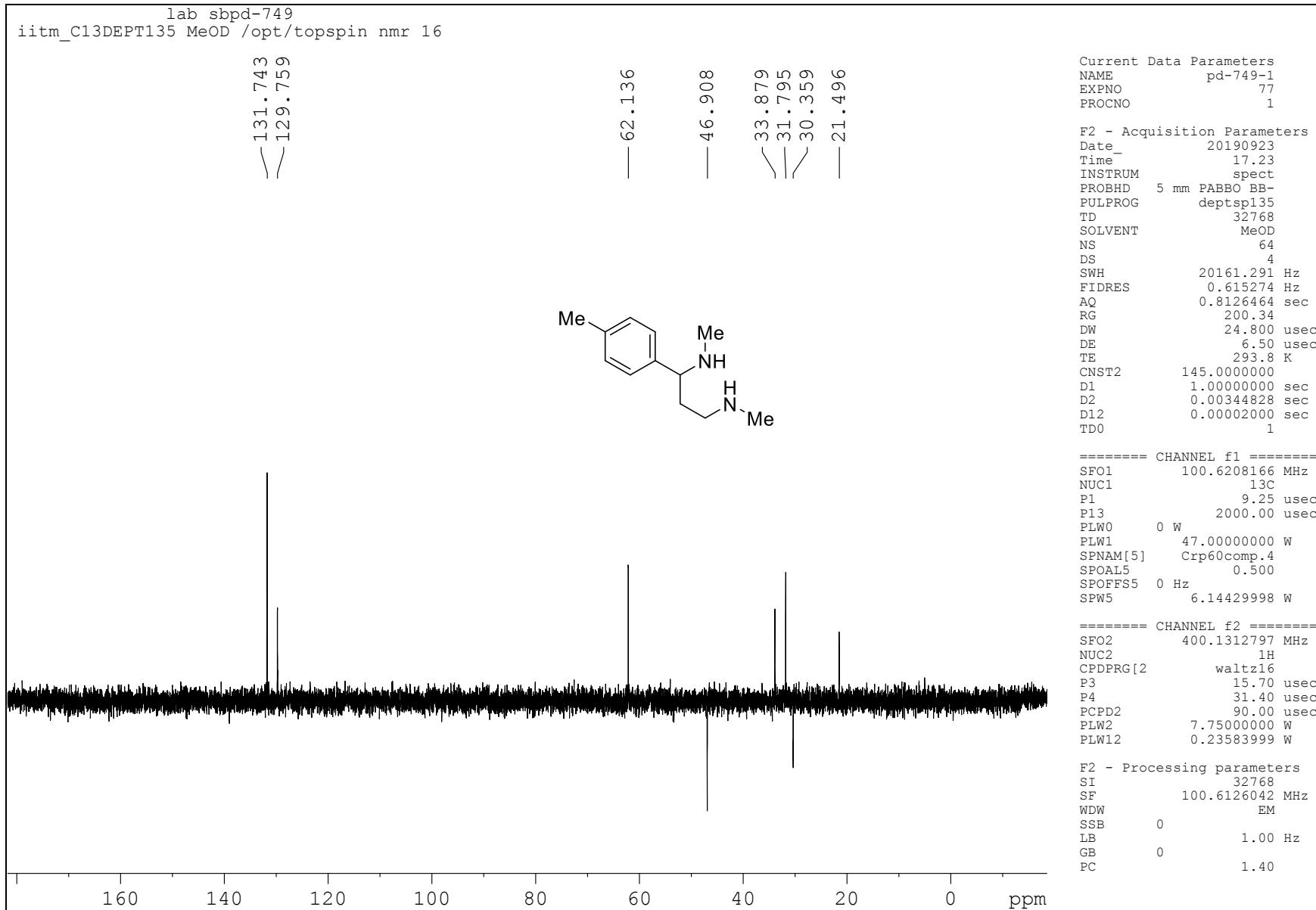


Figure 100 DEPT-135 NMR spectrum of compound 8c