Supplementary Material (ESI) for Green Chemistry

Electronic Supplementary Information:

Solvent-free microwave-assisted Meyers' lactamization

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Contents

1. General information

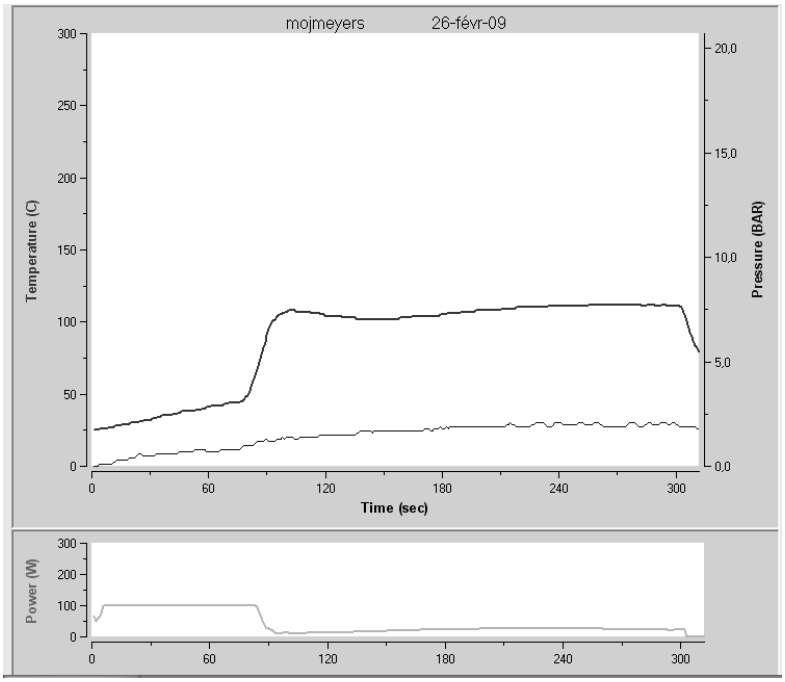
^{1}H NMR and ^{13}C NMR were recorded at room temperature on a Bruker™ DPX 300 at 300 and 100 MHz, respectively. Chemical shifts are in parts per million (ppm). The assignments were made using one dimensional (1D) ^{1}H and ^{13}C spectra or two-dimensional (2D) HSQC and COSY spectra. Mass spectra were recorded with a LCMS-MS triple-quadrupole system (Varian 1200ws). HPLC analysis were performed using a C18 TSK-GEL Super ODS 2 μm particle size column, dimensions 50 * 4.6 mm with a gradient starting from 100% H_2O / 0.1% formic acid and reaching 20% H_2O /80% CH_3CN / 0.08% formic acid within 10 min at a flow rate of 1 mL/min Melting points were determined on a Büchi B-540 apparatus and are uncorrected. Reactions were performed using a Discover™ microwave from CEM™. All commercial reagents and solvents were used without further purification. Specific rotations were measured on a Jasco™ DIP-370 polarimeter using a 10 cm cell.

Crystal data were collected with a Bruker™ Smart Apex-II CCD diffractometer using graphite monochromated MoKα radiation (λ = 0.71073 Å) at 298 K. Cell parameters were retrieved using APEX2[1] software and refined with SAINT[2] on all observed reflections. Data reduction was performed with the SAINT software and corrected for Lorentz and polarization effects. Absorption corrections were applied with the program SADABS[3]. The structure was solved by charge flipping methods using SUPERFLIP[4] program and refined by full-matrix least-squares methods on F using CRYSTALS software[5]. All non-hydrogen atomic positions were located in difference Fourier maps and refined anisotropically. The hydrogen atoms were placed in their geometrically generated positions. Colorless crystals were isolated in rectangular shape at room temperature.

[1] *APEX2 V2008.2-0*. Bruker AXS Inc., Madison, Wisconsin, USA. (**2008**)
[2] *SAINT V7.34A*. Bruker AXS Inc., Madison, Wisconsin, USA. (**2008**)
[3] SADABS V2004/1 - Bruker Nonius area detector scaling and absorption correction. Bruker AXS Inc., Madison, Wisconsin, USA. (**2004**)
[4] Palatinus, L. & van der Lee, A. (**2008**). *J. Appl. Cryst.* **41**, 975.
[5] Betteridge, P.W., Carruthers, J.R., Cooper, R.I., Prout, K. & Watkin, D.J. (**2003**). J. Appl. Cryst. **36**, 1487.

2. General Procedure for Meyers' lactamization using solvent-free microwave conditions.

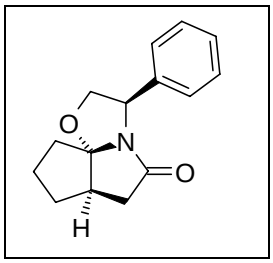
The amino-alcohol (0.32 mmol, 1eq.) and the ketoacid (0.32 mmol, 1 eq.) were mixed in a capped 10-mL microwave-vessel. The mixture was heated at 110 °C at least 5 min (average effective ramp time =1 min). The power was set at 100 W and the pressure was set at 15 bar (average effective pressure = 3 bar). After completion of the reaction, the conversion was directly determined from LCMS analysis. The crude product was dissolved in DCM (10 mL). The organic layer was washed with 1N HCl (5 mL) and $NaHCO_3$ sat. (5mL), and dried over $MgSO_4$). Solvent was removed under reduced pressure. If required flash chromatography was used for purification or separation of diastereoisomers.



Typical T °C = f(t) and Power W = f(t) curves for microwave-assisted Meyers' lactamization

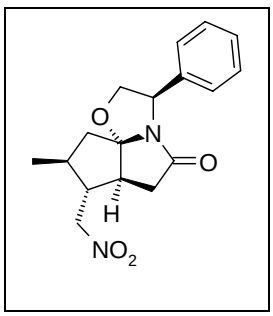
3. Characterization of compounds.

(3R,5aR,8aS)-3-Phenyl-hexahydro-1-oxa-3a-aza-cyclopenta[*c*]pentalen-4-one (1):
Synthesized from 2-oxocyclopentanacetic acid (rac) and R-phenylglycinol



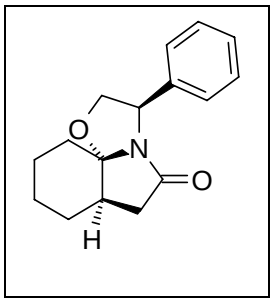
Yield (409 mg, 94%); off-white crystals; mp= 44–45 °C; $[\alpha]_D$ -163 ° (*c*2, CH_2Cl_2); Purity: 100%; δ_H (300 MHz, $CDCl_3$) 7.35-7.23 (m, 5 H), 5.13 (t, *J* = 7.8 Hz, 1H), 4.57 (t, *J* = 8.7 Hz, 1H), 3.95 (t, *J* = 7.8 Hz, 1H), 2.88 (dd, *J* = 17.1, 10.2 Hz, 1H), 2.74-2.64 (m, 1H), 2.45 (dd, *J* = 17.1, 6.6 Hz, 1H), 2.05-1.87 (m, 2H), 1.81-1.64 (m, 3H), 1.58-1.48 (m, 1H). δ_C (50 MHz, $CDCl_3$) 24.6, 32.3, 36.6, 40.7, 41.4, 57.9, 73.5, 110.9 (Cq), 125.6, 127.4, 128.8, 139.7 (Cq), 180.3 (Cq); rt(LCMS) = 5.22 min; $(M+H^+)$ = 244.

(3R,5aS,6R,7S,8aS)-7-Methyl-6-nitromethyl-3-phenyl-hexahydro-1-oxa-3a-aza-cyclopenta[*c*] pentalen-4-one (2):
Synthesized from (1 S-(1b,2a,3b))-(+)-3-methyl-2-(nitromethyl)) 5-oxocyclopentaneacetic acid and R-phenylglycinol



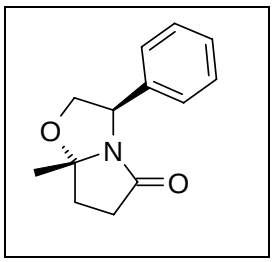
Yield (98 mg, 97%); white crystals; mp= 152 -154 °C; $[\alpha]_D$ -146 ° (*c* 0.5, CH_2Cl_2); Purity: 100%; δ_H (300 MHz, $CDCl_3$) 7.38-7.23 (m, 5 H), 5.10 (t, *J* = 7.8 Hz, 1H), 4.64 (dd, *J* = 13.3, 4.4 Hz, 1H), 4.60 (t, *J* = 9 Hz, 1H), 4.36 (dd, *J* = 12.6, 9.6 Hz, 1H), 3.94 (dd, *J* = 9, 7.8 Hz, 1H), 2.90 (dd, *J* = 19.8, 12.6 Hz, 1H), 2.66-2.57 (m, 2H), 2.22-2.09 (m, 2H), 2.03-1.95 (m, 1H), 1.63 (t, *J* = 12.6 Hz, 1H), 1.11 (d, *J* = 6.6 Hz, 3H); δ_C (50 MHz, $CDCl_3$) 17.3, 38.3, 40.4, 44.8, 48.6, 52.0, 58.4, 73.6, 78.2, 108.0 (Cq), 125.8, 127.7, 128.8, 139.4 (Cq), 179.3 (Cq); rt(LCMS) = 5.53 min; $(M+H^+)$ = 317.

(3R,5aR,9aS)-3-Phenyl-octahydro-1-oxa-3a-aza-cyclopenta[*c*]inden-4-one (3):
Synthesized from 2-oxocyclohexanacetic acid (rac) and R-phenylglycinol



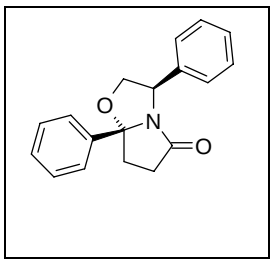
Yield (51 mg, 84%); white crystals; mp= 55-57 °C; $[\alpha]_D$ -108 ° (*c* 0.5, CH_2Cl_2); Purity: 100%; δ_H (300 MHz, $CDCl_3$) 7.34-7.25(m, 5 H), 5.13 (t, *J* = 7.2 Hz,1H), 4.59 (dd, *J* = 9.6, 8.4 Hz, 1H), 4.14 (dd, *J* = 9.0, 6.6 Hz, 1H), 2.71 (dd, *J* = 18.0, 10.5 Hz, 1H), 2.58-2.48 (m, 2H), 1.87-1.51 (m, 8H). δ_C (50 MHz, $CDCl_3$) 19.8, 20.8, 25.6, 31.4, 39.2, 41.3, 73.9, 100.3 (Cq), 125.6, 126.8, 127.0, 127.4, 140.0 (Cq), 177.1 (Cq); rt(LCMS) = 5.09 min; $(M+H^+)$ = 258.

(3R,7aS)-7a-Methyl-3-phenyl-tetrahydro-pyrrolo[2,1-*b*]oxazol-5-one (4):
Synthesized from levulinic acid and R-phenylglycinol



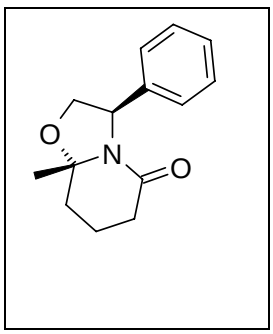
Yield (88 mg, 94%); white crystals; mp= 136-138 °C; $[\alpha]_D$ -175° (*c* 4, CH_2Cl_2); Purity: 100%; δ_H (300 MHz, $CDCl_3$) 7.37-7.24 (m, 5 H), 5.17 (t, *J* = 7.5 Hz, 1H), 4.61 (t, *J* = 8.7 Hz, 1H), 4.10 (dd, *J* = 8.7, 6.9 Hz, 1H), 2.88 (dt, *J* = 17.1, 9.9 Hz, 1H), 2.58 (ddd, *J* = 17.1, 7.8, 5.4 Hz, 1H), 2.31-2.25 (m, 2H), 1.45 (s, 3H); δ_C (50 MHz, $CDCl_3$) 24.3, 32.9, 34.1, 57.4, 73.3, 100.5 (Cq), 125.5, 127.4, 128.7, 139.9 (Cq), 178.8 (Cq); rt(LCMS) = 4.58 min; $(M+H^+)$ = 218.

(3R,7aR)-3,7a-Diphenyl-tetrahydro-pyrrolo[2,1-*b*]oxazol-5-one (5):
Synthesized from 3-Benzoylproponic acid and R-phenylglycinol



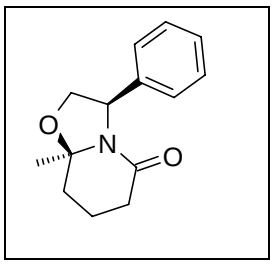
Yield (21 mg, 69%); white crystals; mp= 71-73 °C; $[\alpha]_D$ -101 ° (*c* 2, CH_2Cl_2); Purity:100%; purification by flash-chromatography (AcOEt/petroleum ether 30/70); δ_H (300 MHz, $CDCl_3$) 7.49-7.07 (m, 10H), 5.17 (t, *J* = 8.1 Hz, 1H), 4.66 (dd, *J* = 9.0, 8.1 Hz, 1H), 3.89 (t, *J* = 9.0 Hz,1H), 3.04-2.91 (m, 1H), 2.71-2.56 (m, 2H), 2.39-2.29 (m, 1H); δ_C (50 MHz, $CDCl_3$) 33.2, 36.0, 59.2, 73.9, 102.8 (Cq), 125.1, 126.8, 127.4, 128.3, 128.4, 128.6, 138.9 (Cq), 142.03 (Cq), 179.9 (Cq); rt(LCMS) =5.95 min; $(M+H^+)$ =280.

(3R,8aS)-8a-Methyl-3-phenyl-hexahydro-oxazolo[3,2-*a*]pyridin-5-one (6a)
Synthesized from 4-acetylbutyric acid and R-phenylglycinol



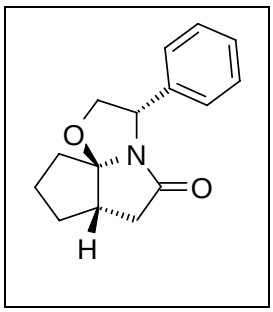
Yield (60 mg, 68%); white crystals; mp= 61-63 °C; $[\alpha]_D$ -131° (*c* 0.5, CH_2Cl_2); Purity: 100%; purification by flash-chromatography (AcOEt/petroleum ether 20/80); δ_H (300 MHz, $CDCl_3$) 7.36-7.19 (m, 5 H), 5.37 (t, *J* = 8.1 Hz, 1H), 4.51 (t, *J* = 8.7 Hz, 1H), 3.96 (dd, *J* = 9.0, 8.1 Hz, 1H), 2.61 (ddd, *J* = 17.5, 7.4, 2.7 Hz, 1H), 2.45 (ddd, *J* = 17.5, 10.2, 7.4 Hz, 1H), 2.24-2.17 (m, 1H), 2.04-1.85 (m, 2H), 1.70 (ddd, *J* = 17.1, 12.3, 4.5 1H), 1.45 (s, 3H); δ_C (50 MHz, $CDCl_3$) 17.3, 23.7, 30.8, 35.2, 58.6, 69.6, 93.9(Cq), 125.5, 127.2, 128.6, 140.0(Cq), 169.7(Cq); rt(LCMS) =4.69 min; $(M+H^+)$ =232.4.

(3R,8aR)-8a-Methyl-3-phenyl-hexahydro-oxazolo[3,2-*a*]pyridin-5-one (6b)
Synthesized from 4-acetylbutyric acid and R-phenylglycinol



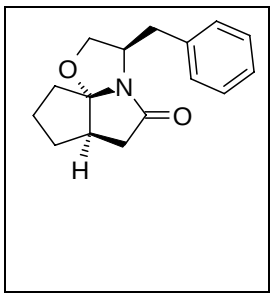
Yield (14 mg, 17%); white crystals; mp= 53-55 °C; $[\alpha]_D$ -141 ° (*c* 0.5, CH_2Cl_2); Purity: 100%; purification by flash-chromatography (AcOEt/petroleum ether 20/80); δ_H (300 MHz, $CDCl_3$) 7.31-7.30 (m, 5 H), 4.96 (dd, *J* = 7.2, 1.8 Hz,1H), 4.49 (dd, *J* = 9.3, 7.2 Hz, 1H), 3.97 (dd, *J* = 9.3, 1.8 Hz, 1H), 2.39-2.26 (m, 3H), 2.01-1.99 (m, 3H), 1.45 (s, 3H); δ_C (50 MHz, $CDCl_3$) 17.3, 23.1, 30.0, 34.3, 59.1, 71.3, 93.5(Cq), 126.3, 127.5, 128.6, 141.7 (Cq), 167.6 (Cq); rt(LCMS) = 4.34 min; $(M+H^+)$ =232.4.

(3S,5aS,8aR)-3-Phenyl-hexahydro-1-oxa-3a-aza-cyclopenta[*c*]pentalen-4-one (7)
Synthesized from 2-oxocyclopentanacetic acid (rac) and S- phenylglycinol



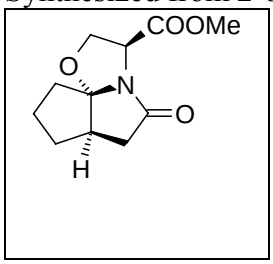
Yield (118 mg, 98%); white crystals; mp= 39-41 °C; $[\alpha]_D$ +117 ° (*c* 0.5, CH_2Cl_2); Purity 100 %; δ_H (300 MHz, $CDCl_3$) 7.36-7.24 (m, 5 H), 5.17 (t, *J* = 7.8 Hz, 1H), 4.63 (t, *J* = 8.4 Hz, 1H), 3.99 (t, *J* = 8.4Hz, 1H), 2.90 (dd, *J* = 17.4, 10.5 Hz, 1H), 2.79-2.69 (m, 1H), 2.50 (dd, *J* = 17.4, 6.3 Hz, 1H), 2.08-1.92 (m, 2H), 1.86-1.70 (m, 3H), 1.66-1.54(m, 1H); δ_C (50 MHz, $CDCl_3$) 24.5, 32.4, 36.7, 40.7, 41.4, 57.9, 73.5, 110.8(Cq), 125.6, 127.4, 128.7, 139.8(Cq), 180.3(Cq); rt(LCMS) = 5.16 min; $(M+H^+)$ = 244.

(3R,5aR,8aS)-3-Benzyl-hexahydro-1-oxa-3a-aza-cyclopenta[*c*]pentalen-4-one (8)
Synthesized from 2-oxocyclopentanacetic acid (rac) and R-phenylalaninol



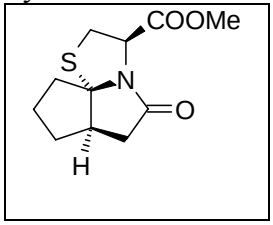
Yield (41 mg; 95%); white crystals; mp= 26-28 °C; $[\alpha]_D$ -103° (*c* 0.5, CH_2Cl_2); Purity 100 %; δ_H (300 MHz, $CDCl_3$) 7.34-7.23 (m, 5 H), 4.40-430(m, 1H), 4.04 (dd, *J* = 8.7, 6.9 Hz, 1H), 3.77 (dd, *J* = 8.7, 6.3 Hz, 1H), 3.11 (dd, *J* = 13.5, 5.4 Hz, 1H), 2.82-2.72 (m, 2H), 2.65-2.55 (m, 1H), 2.37 (dd, *J* = 17.1, 6.9 Hz, 1H), 2.00-1.92 (m, 1H), 1.85-1.67 (m, 4H), 1.55-1.47 (m, 1H); δ_C (50 MHz, $CDCl_3$) 24.6, 32.1, 37.6, 39.8, 40.4, 42.0, 55.9, 71.6, 110.4 (Cq), 126.7, 128.5, 129.3, 137.0 (Cq), 179.8 (Cq); rt(LCMS) = 5.55 min; $(M+H^+)$ = 258.

(3S,5aR,8aS)-4-Oxo-octahydro-1-oxa-3a-aza-cyclopenta[*c*]pentalene-3-carboxylic acid methyl ester (9)
Synthesized from 2-oxocyclopentanacetic acid (rac) and L-serine methyl ester



Yield (95 mg, 85%); brown oil; $[\alpha]_D$ +97 °(*c* 0.5, CH_2Cl_2); Purity 100 %; δ_H (300 MHz, $CDCl_3$) 4.70 (dd, *J* = 8.7, 6.9 Hz, 1H), 4.38 (t, *J* = 8.7 Hz, 1H), 4.10 (dd, *J* = 8.7, 6.9 Hz, 1H), 3.79 (s, 3H), 2.83 (dd, *J* = 17.1, 10.2 Hz, 1H), 2.72-2.62 (m, 1H), 2.45 (dd, *J* = 17.1, 6.3 Hz, 1H), 2.16-1.95 (m, 2H), 1.89-1.68 (m, 3H), 1.61-153 (m, 1H); δ_C (50 MHz, $CDCl_3$) 24.5, 32.3, 36.1, 40.1, 41.3, 52.6, 55.6, 69.2, 111.4(Cq), 170.8(Cq), 179.7(Cq); rt(LCMS) =3.76 min; $(M+H^+)$ =226.

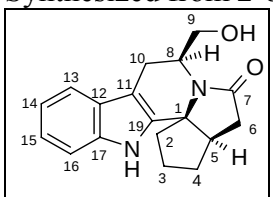
(3R,5aR,8aS)-4-Oxo-octahydro-1-thia-3a-aza-cyclopenta[*c*]pentalene-3-carboxylic acid methyl ester (10)
Synthesized from 2-oxocyclopentanacetic acid (rac) and L-cysteine methyl ester



Yield (110 mg, 91%); brown oil; $[\alpha]_D$ -124 °(*c* 0.5, CH_2Cl_2); Purity 100 %; δ_H (300 MHz, $CDCl_3$) 5.01 (dd, *J* = 8.4, 6.0 Hz, 1H), 3.76 (s, 3H), 3.57 (dd, *J* = 11.7, 6.0 Hz, 1H), 3.47 (dd, *J* = 11.7, 8.4 Hz, 1H), 2.90-2.80 (m, 2H), 2.47-2.30 (m, 2H), 2.05-.1.92 (m, 1H), 1.86-1.72 (m, 3H), 1.64-1.54 (m, 1H); δ_C (50 MHz, $CDCl_3$) 25.1, 33.8, 36.2, 39.7, 41.6, 43.1, 52.8, 58.3, 87.0(Cq), 170.8(Cq), 177.0(Cq); rt(LCMS) = 4.37 min; $(M+H^+)$ = 242.

(1R,5S,11bR)-5-Hydroxymethyl-1,11b-cyclohexyl-1,2,5,6,11,11b-hexahydro-indolizino[8,7-*b*]indol-3-one (11)

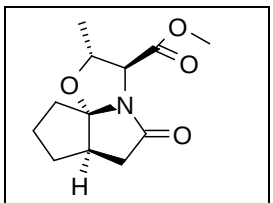
Synthesized from 2-oxocyclopentanacetic acid (rac) and L-tryptophanol



Yield (122 mg, 83%); white crystals; mp= 144-146 °C; $[\alpha]_D$ -87 °(*c* 0.5, CH_2Cl_2); Purity 100 %; δ_H (300 MHz, $CDCl_3$) 11.01 (br s, 1H, NH), 7.37 (d, *J* = 7.8 Hz, 1H, $C_{(16)}H$), 7.31 (d, *J* = 7.8 Hz, 1H, $C_{(13)}H$), 7.06(dd, *J* = 7.2, 3 Hz, 1H, $C_{(15)}H$), 6.97 (dd, *J* = 7.2, 1.2 Hz, 1H, $C_{(14)}H$), 5.05 (dd, *J* = 7.5, 5.1 Hz, 1H, $C_{(9)}H$), 4.14-3.99 (m, 2H, $C_{(9)}H$, $C_{(10)}H$), 3.55-3.47 (m, 1H, $C_{(8)}H$), 3.35 (br s, 1H, OH), 2.89-2.68(m, 3H, $C_{(5)}H$, $C_{(6)}H$, $C_{(10)}H$), 2.57 (dd, *J* = 18, 10.8 Hz, 1H, $C_{(2)}H$), 2.27-2.07 (m, 3H, $C_{(2)}H$, $C_{(6)}H$, $C_{(3)}H$), 1.88-1.58 (m, 3H, $C_{(3)}H$, $C_{(4)}H$); δ_C (50 MHz, $CDCl_3$) 24.0 ($C_{(3)}$), 24.9 ($C_{(4)}$), 34.8 ($C_{(2)}$), 38.9 ($C_{(6)}$), 39.5 ($C_{(10)}$), 41.3 ($C_{(5)}$), 56.6 ($C_{(8)}$), 62.2 ($C_{(9)}$), 73.9 ($C_{(1)}$), 107.8 ($C_{(11)}$), 111.6 ($C_{(13)}$), 118.1 ($C_{(16)}$), 119.1 ($C_{(14)}$), 121.5 ($C_{(15)}$), 126.7 ($C_{(12)}$), 136.8 ($C_{(19)}$), 138.1 ($C_{(17)}$), 176.0 ($C_{(7)}$); rt(LCMS) =5.95 min; ($M+H^+$) =297.

(2R,3S,5aR,8aS)-2-Methyl-4-oxo-octahydro-1-oxa-3a-aza-cyclopenta[c]pentalene-3-carboxylic acid methyl ester (12)

Synthesized from 2-oxocyclopentanacetic acid (rac) and L-threonine methyl ester

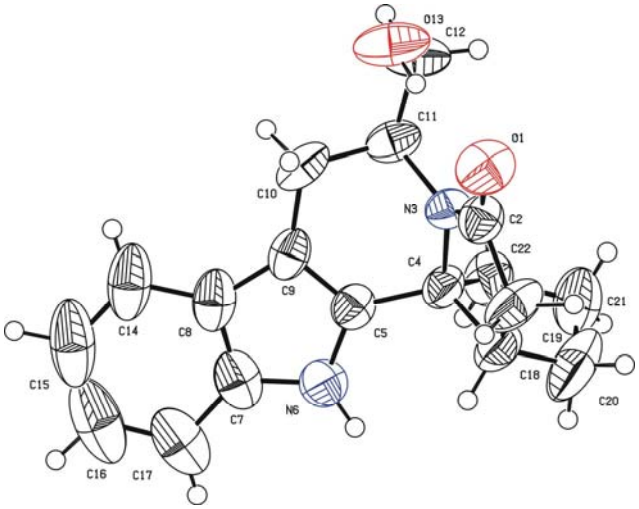


Yield (117 mg, 93%); white crystals; mp= 89-91 °C; $[\alpha]_D$ +79 °(*c* 0.5, CH_2Cl_2); Purity 100 %; δ_H (300 MHz, $CDCl_3$) 4.40-4.32 (m, 1H), 4.12 (d, *J* = 8.1 Hz, 1H), 3.77 (s, 3H), 2.82 (dd, *J* = 17.4, 10.5 Hz, 1H), 2.71-2.62 (m, 1H), 2.42 (dd, *J* = 17.4, 6.3 Hz, 1H), 2.23-2.16 (m, 1H), 2.01-1.81 (m, 1H), 1.84-176 (m, 2H), 1.72-1.63 (m, 1H), 1.56-150 (m, 1H), 1.45 (d, *J* = 6.0 Hz, 1H); δ_C (50 MHz, $CDCl_3$) 19.7, 24.4, 29.7, 32.2, 36.2, 40.1, 41.5, 52.6, 62.4, 78.5, 111.1(Cq), 170.6(Cq), 179.5(Cq); rt(LCMS) =4.27 min; ($M+H^+$) =240.

4. X-ray diffraction data for 11 and 12 and CCDC codes.

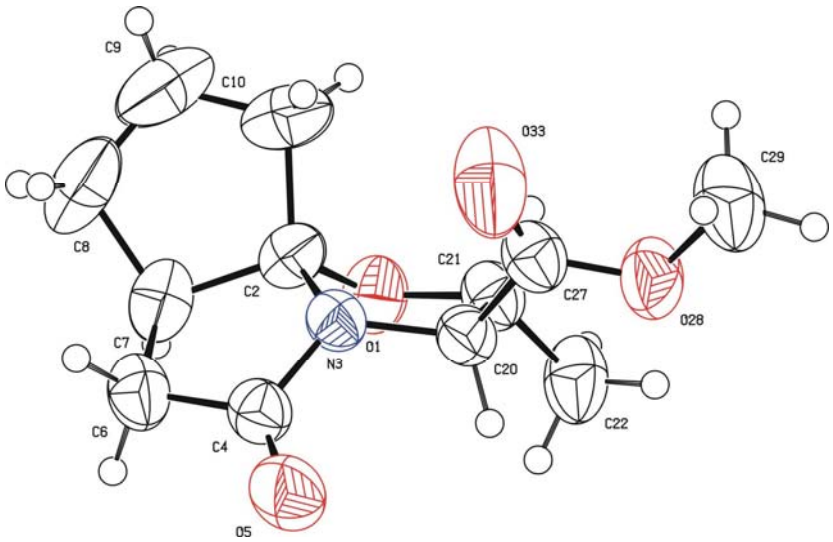
(1R,5S,11bR)-5-Hydroxymethyl-1,11b-cyclohexyl-1,2,5,6,11,11b-hexahydro-indolizino[8,7-*b*]indol-3-one (11)

CCDC 749287
Unit cell parameters:
a 8.4078(8) b 8.3744(8) c 11.4399(10) beta 98.698(4)space group P21



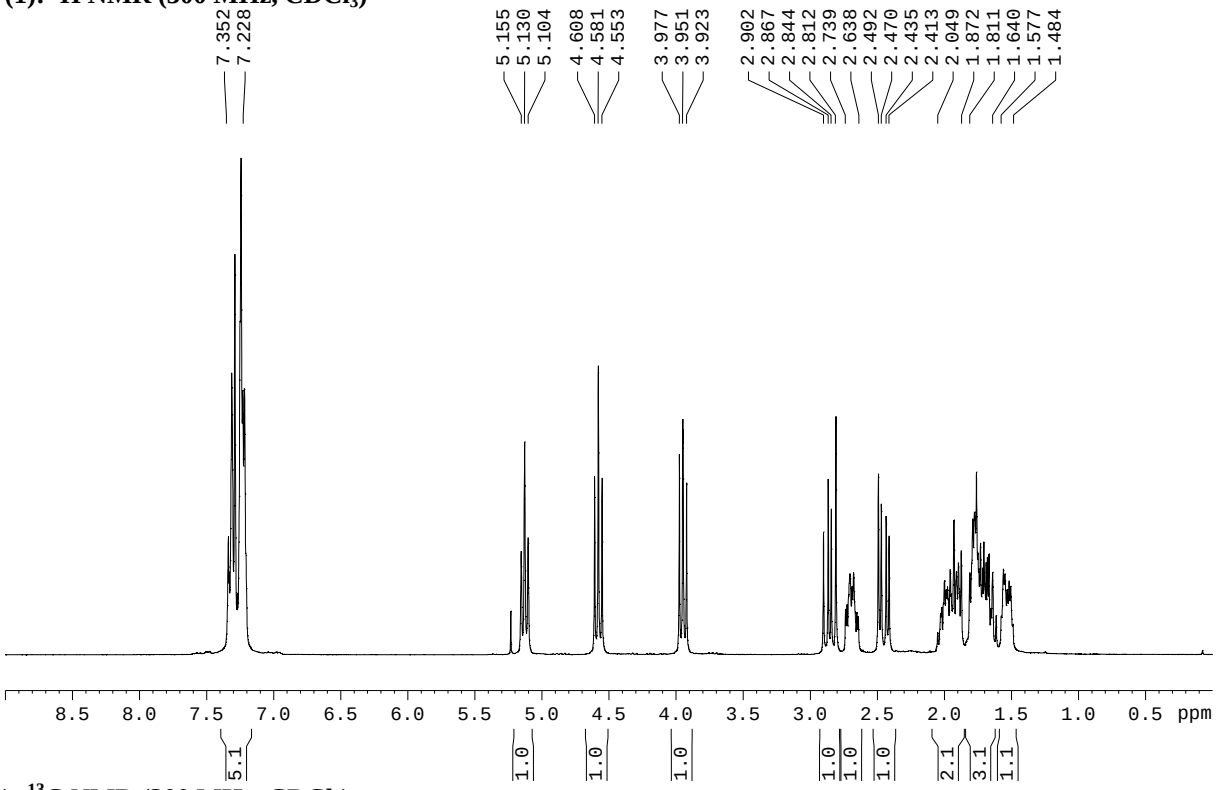
(2R,3S,5aR,8aS)-2-Methyl-4-oxo-octahydro-1-oxa-3a-aza-cyclopenta[c]pentalene-3-carboxylic acid methyl ester (12)

CCDC 749288
Unit cell parameters: a 17.5352(8) b 7.6758(4) c 10.0025(4) beta 113.780(3)
space group C2

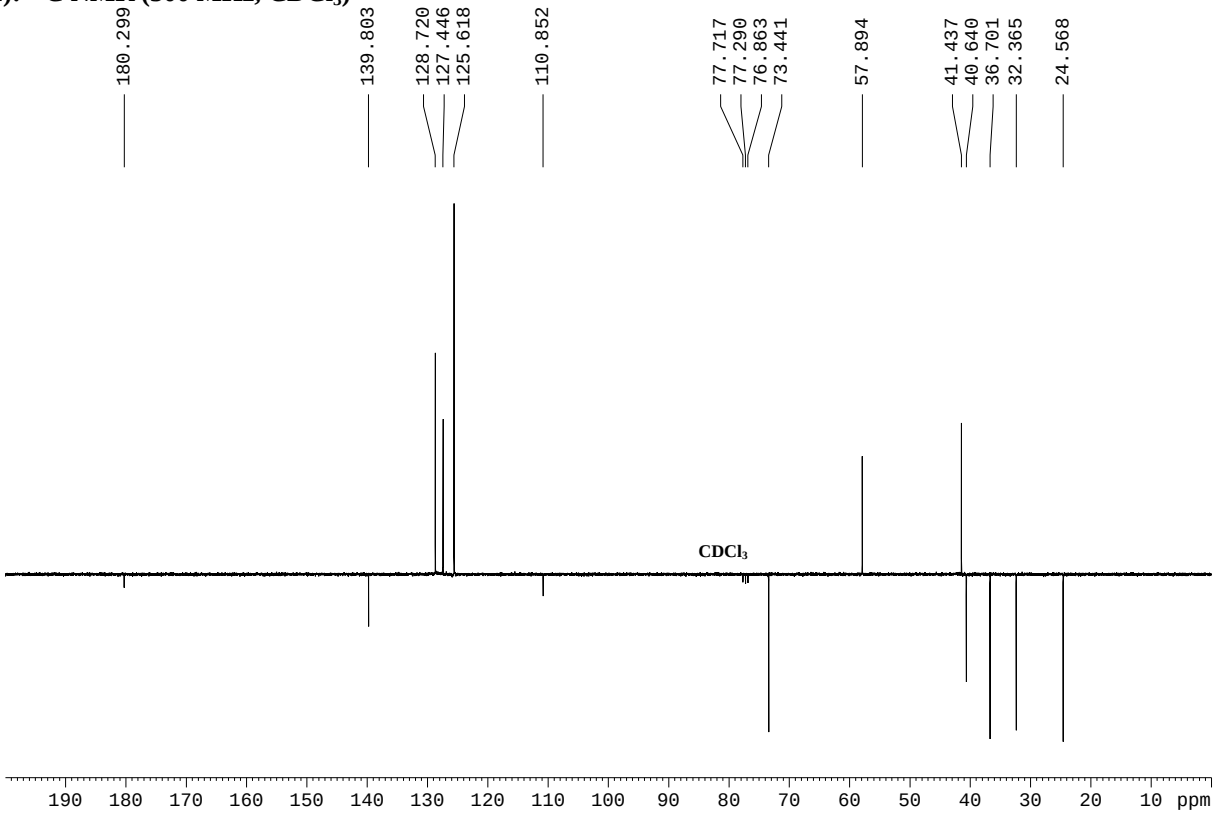


5. NMR Spectra

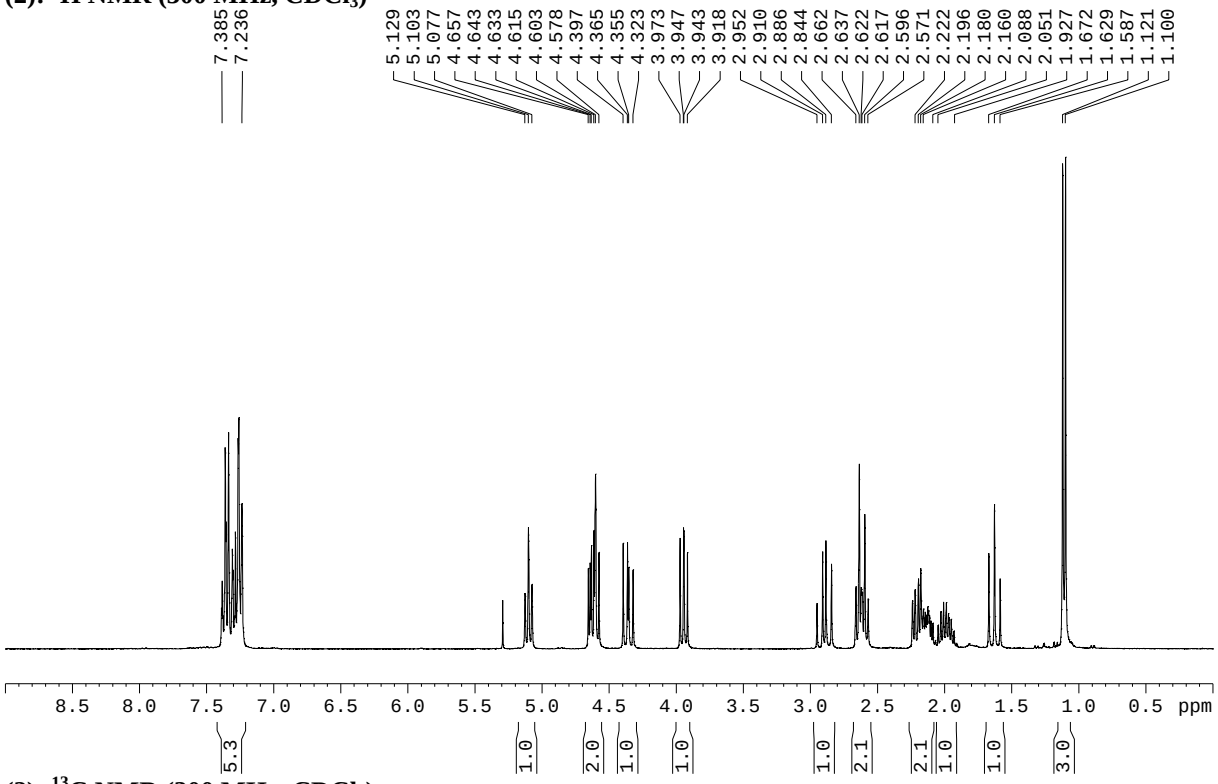
(1): ^{1}H NMR (300 MHz, $CDCl_3$)



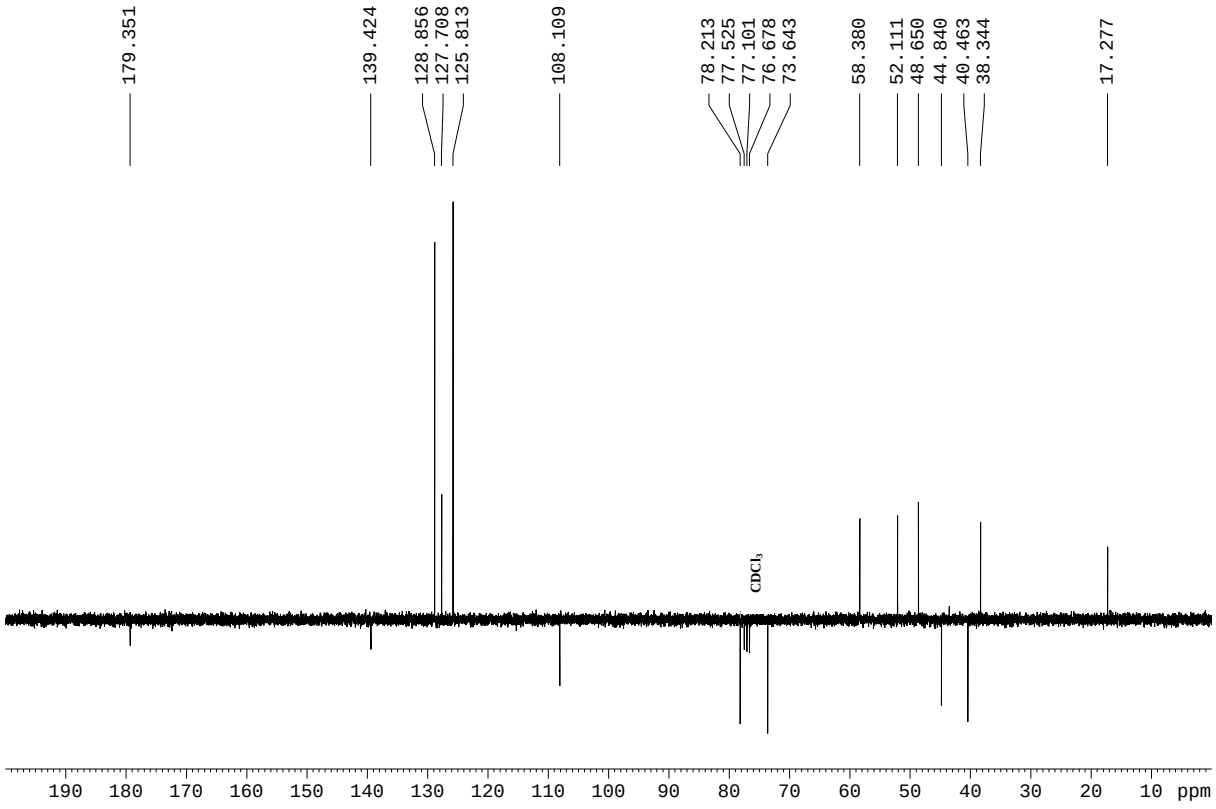
(1): ^{13}C NMR (300 MHz, $CDCl_3$)



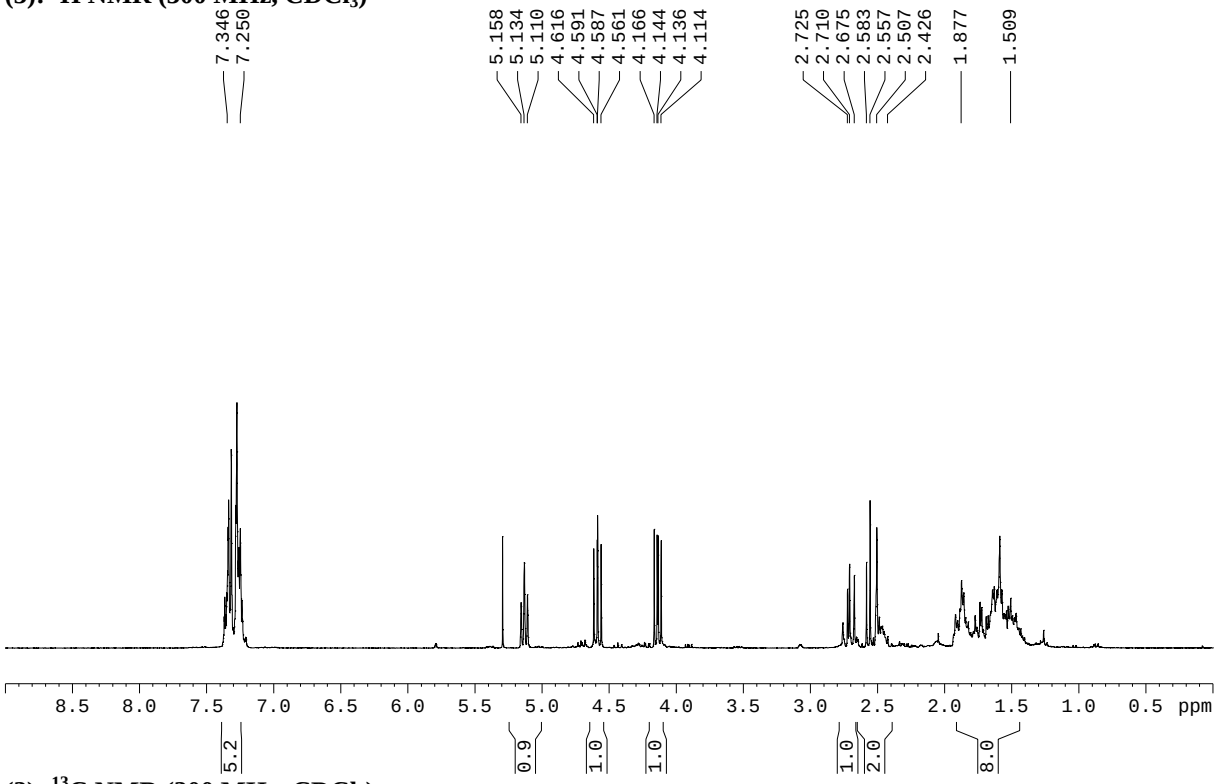
(2): ^{1}H NMR (300 MHz, $CDCl_3$)



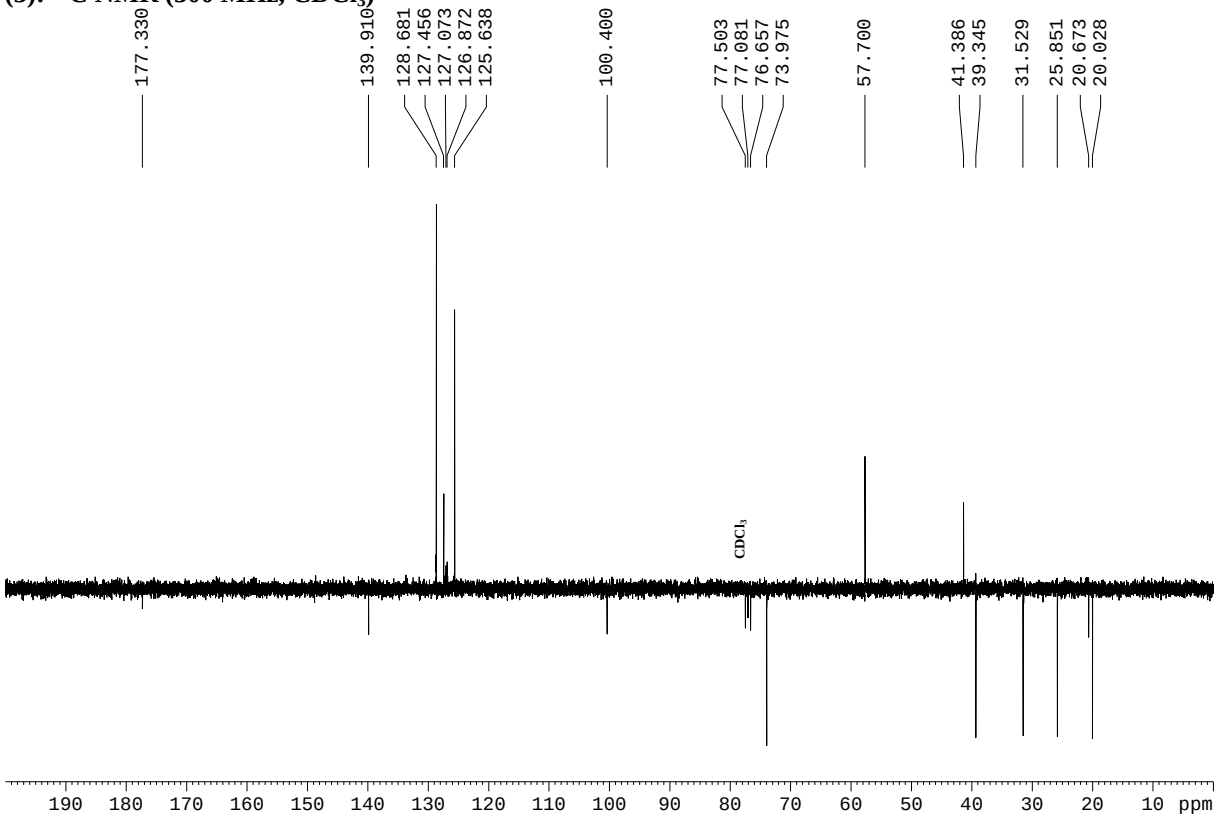
(2): ^{13}C NMR (300 MHz, $CDCl_3$)



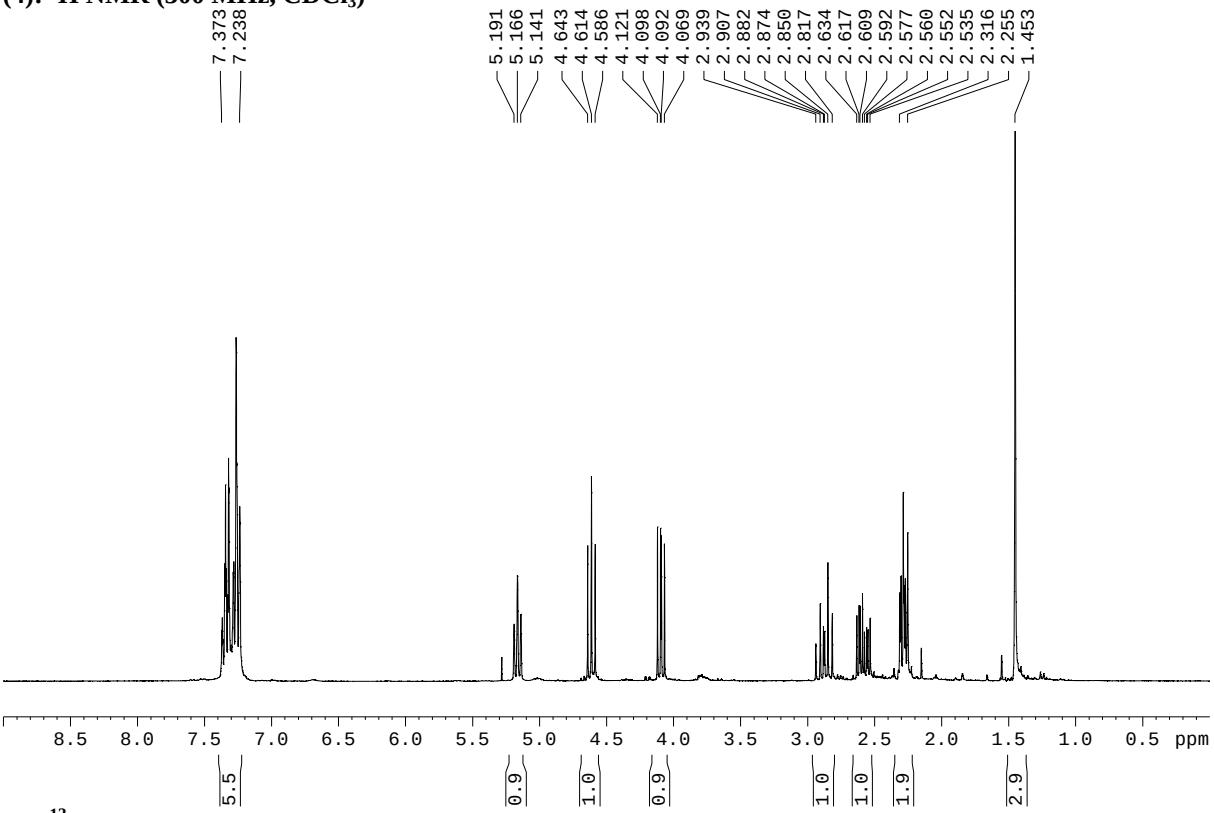
(3): ^{1}H NMR (300 MHz, $CDCl_3$)



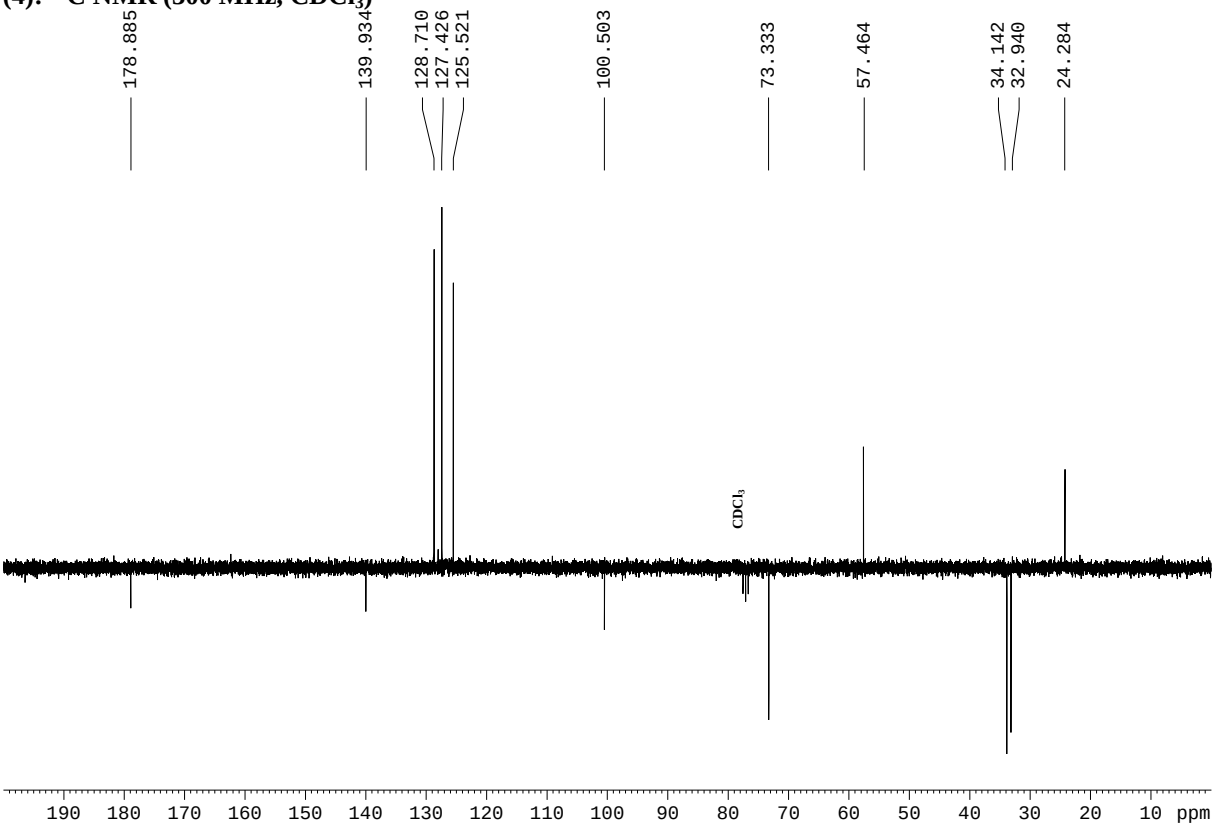
(3): ^{13}C NMR (300 MHz, $CDCl_3$)



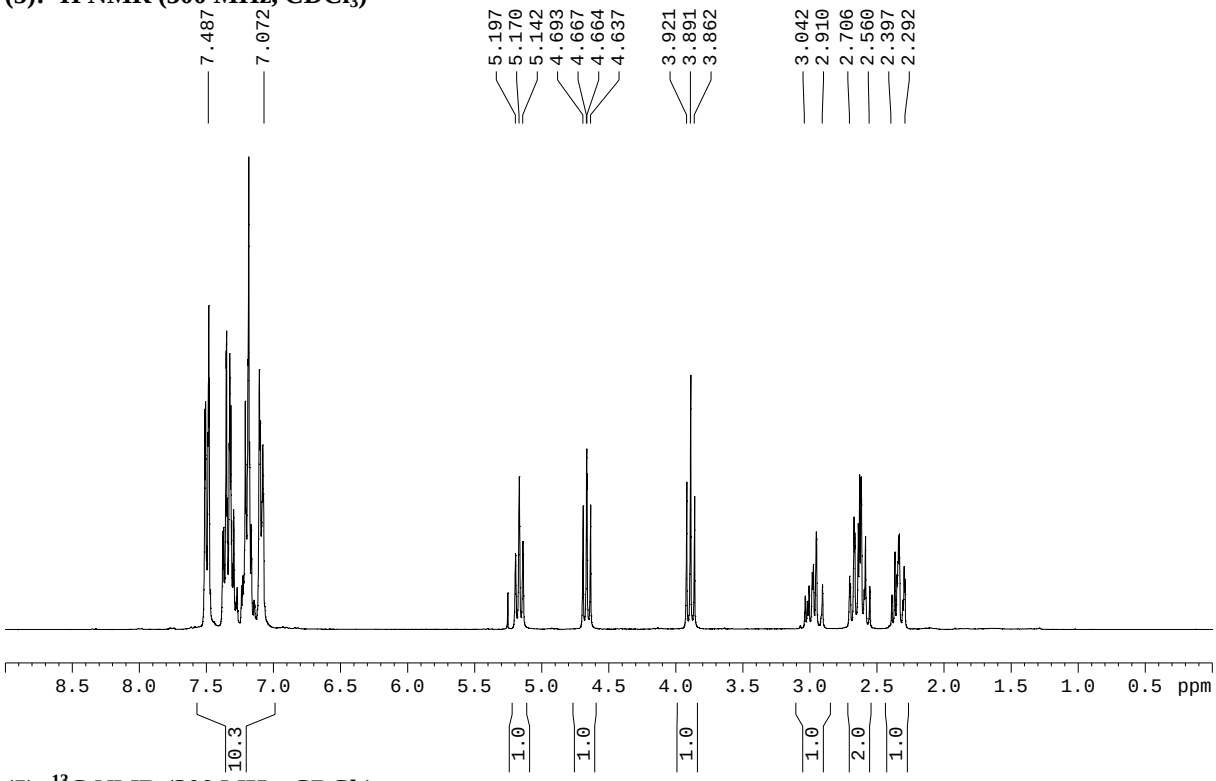
(4): ^{1}H NMR (300 MHz, $CDCl_3$)



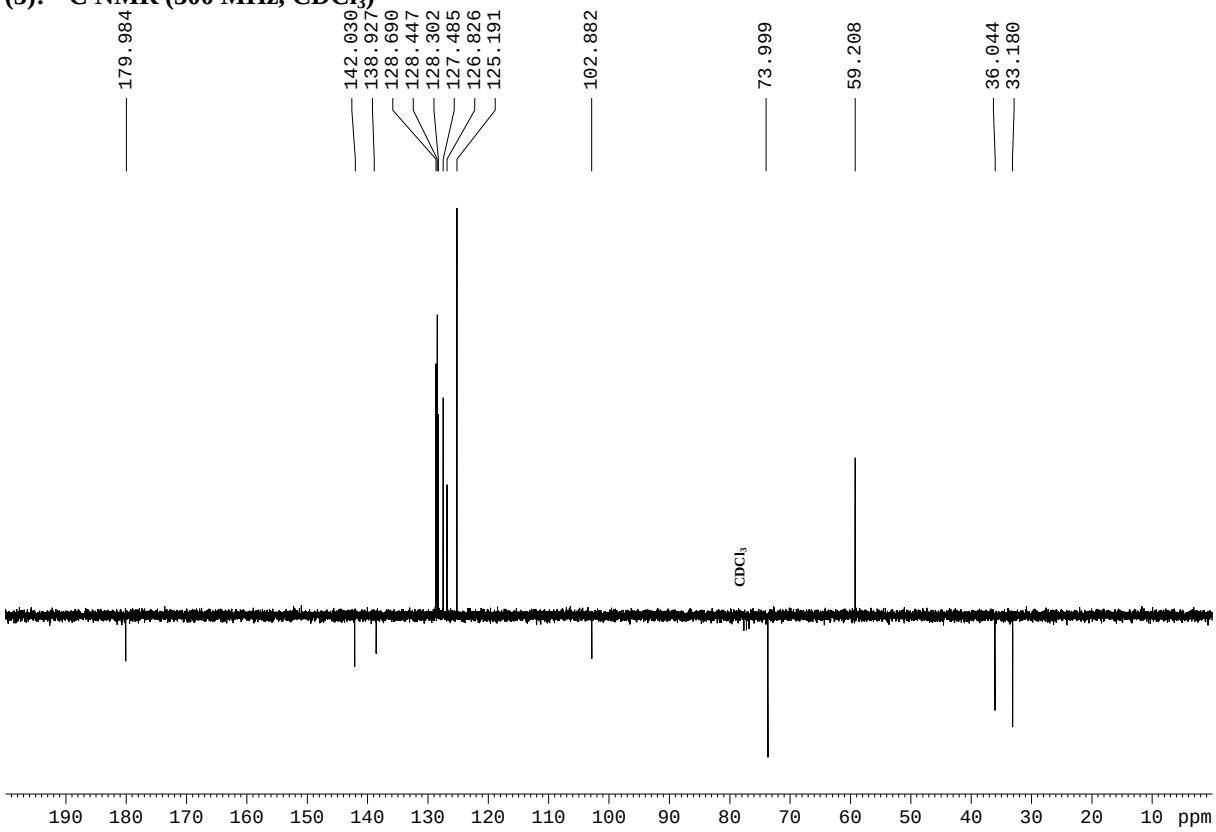
(4): ^{13}C NMR (300 MHz, $CDCl_3$)



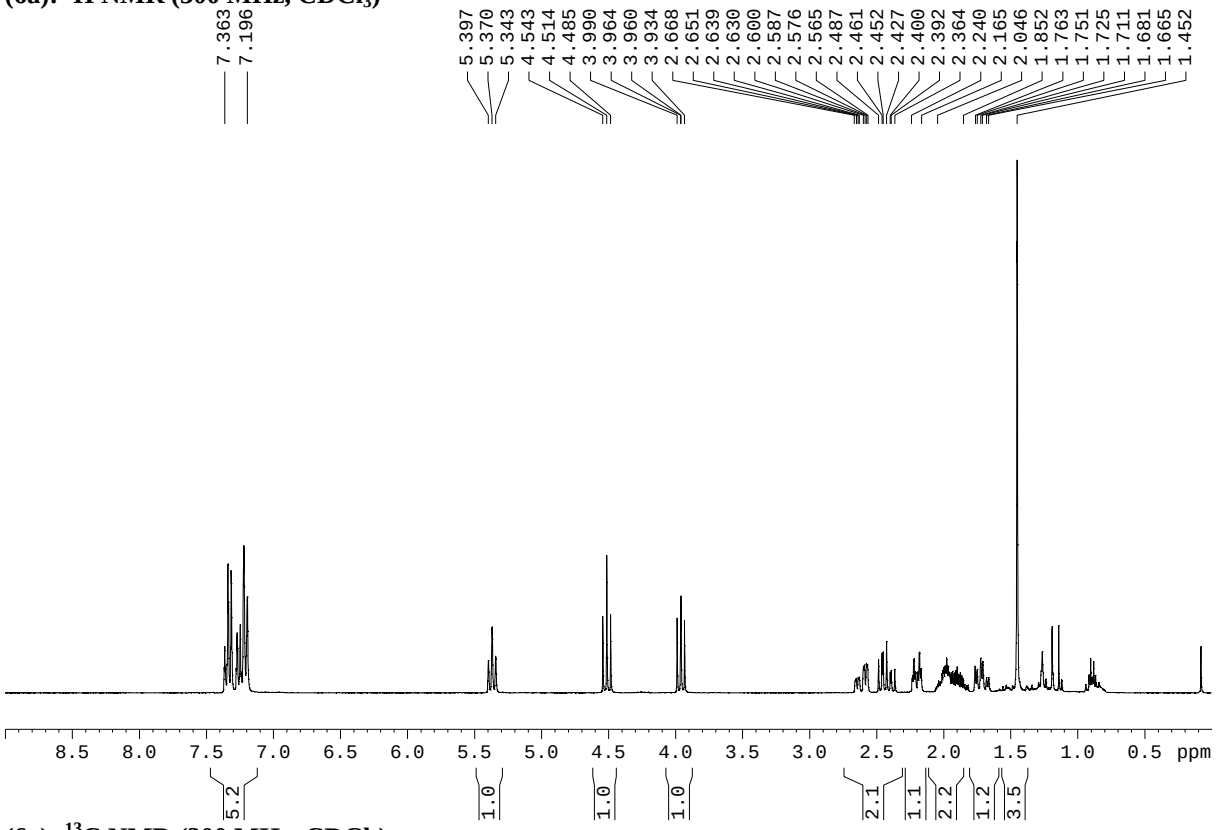
(5): ^{1}H NMR (300 MHz, $CDCl_3$)



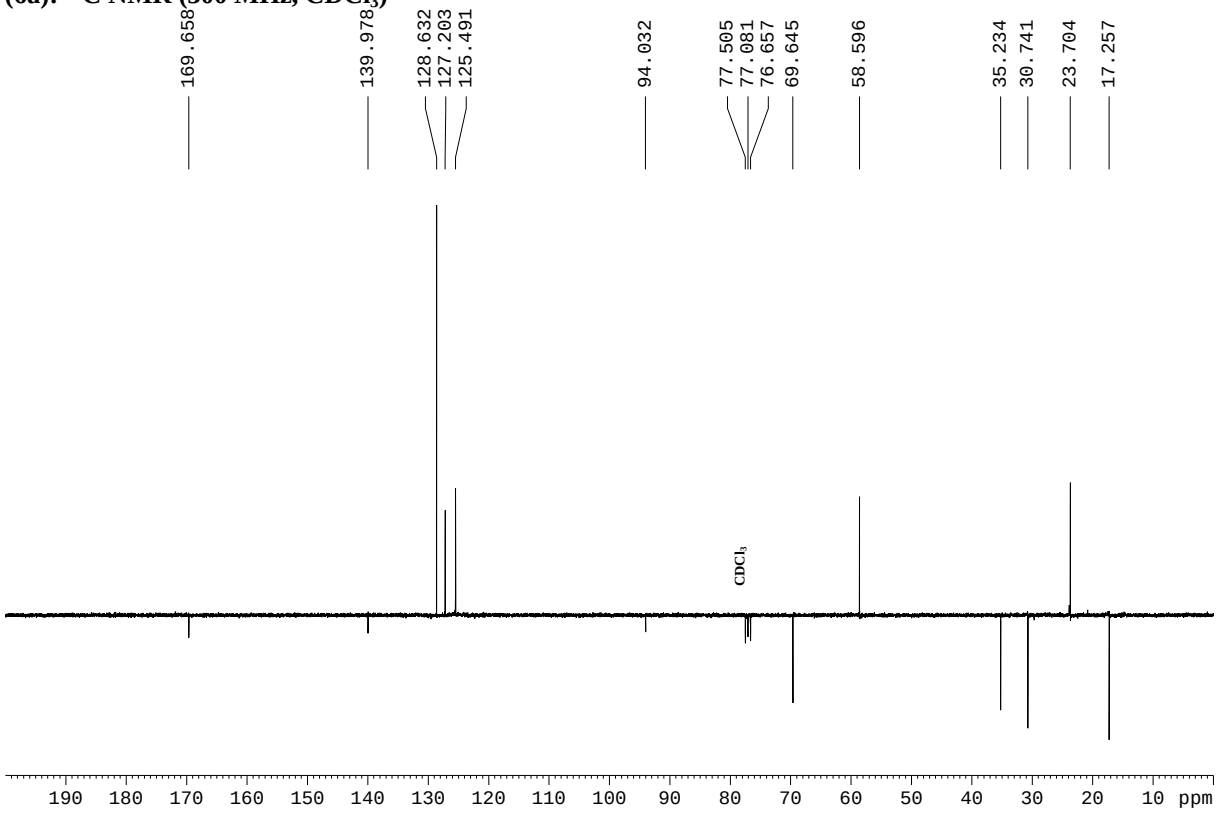
(5): ^{13}C NMR (300 MHz, $CDCl_3$)



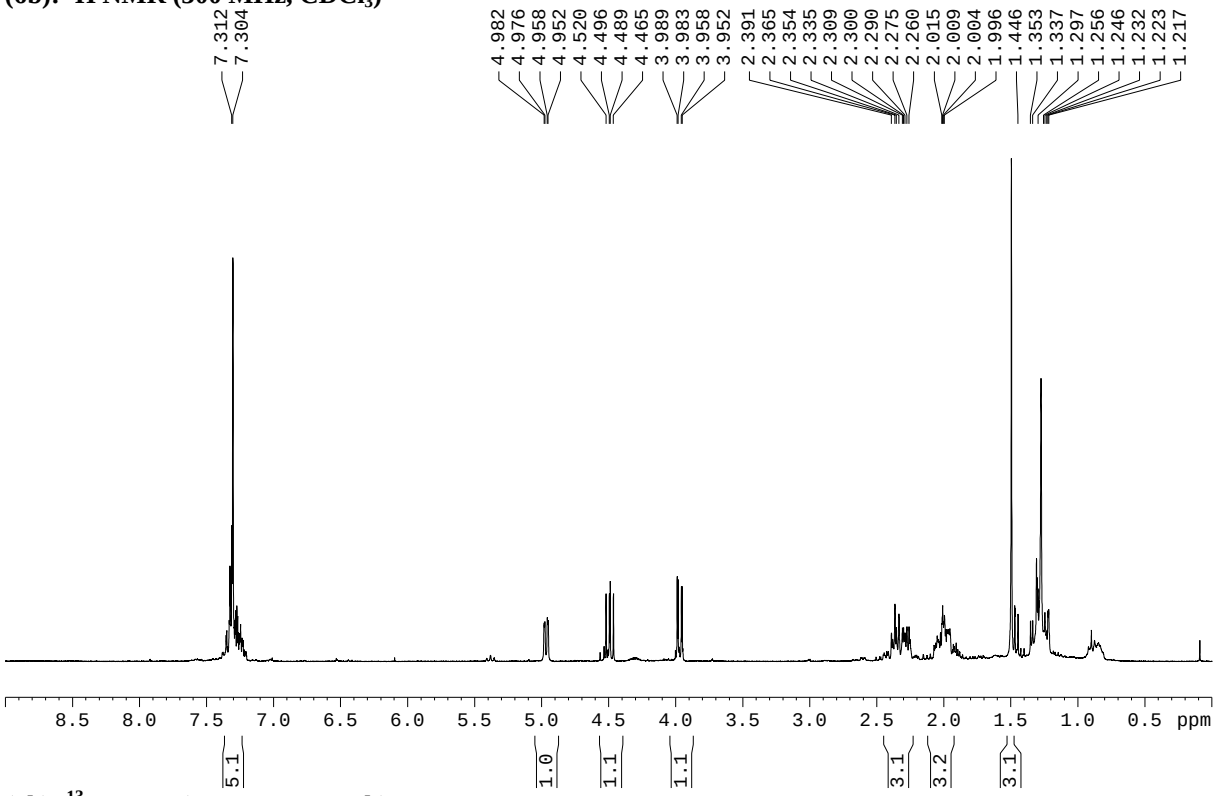
(6a): ^{1}H NMR (300 MHz, $CDCl_3$)



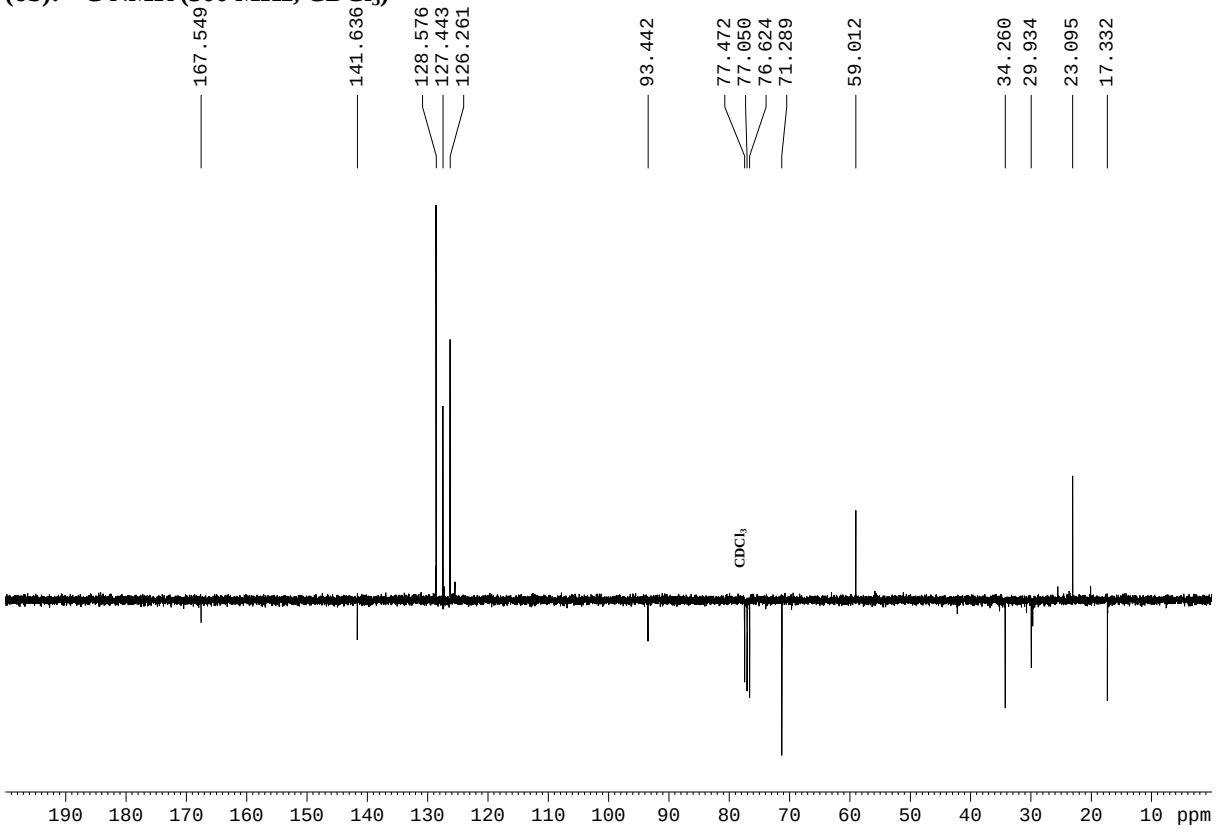
(6a): ^{13}C NMR (300 MHz, $CDCl_3$)



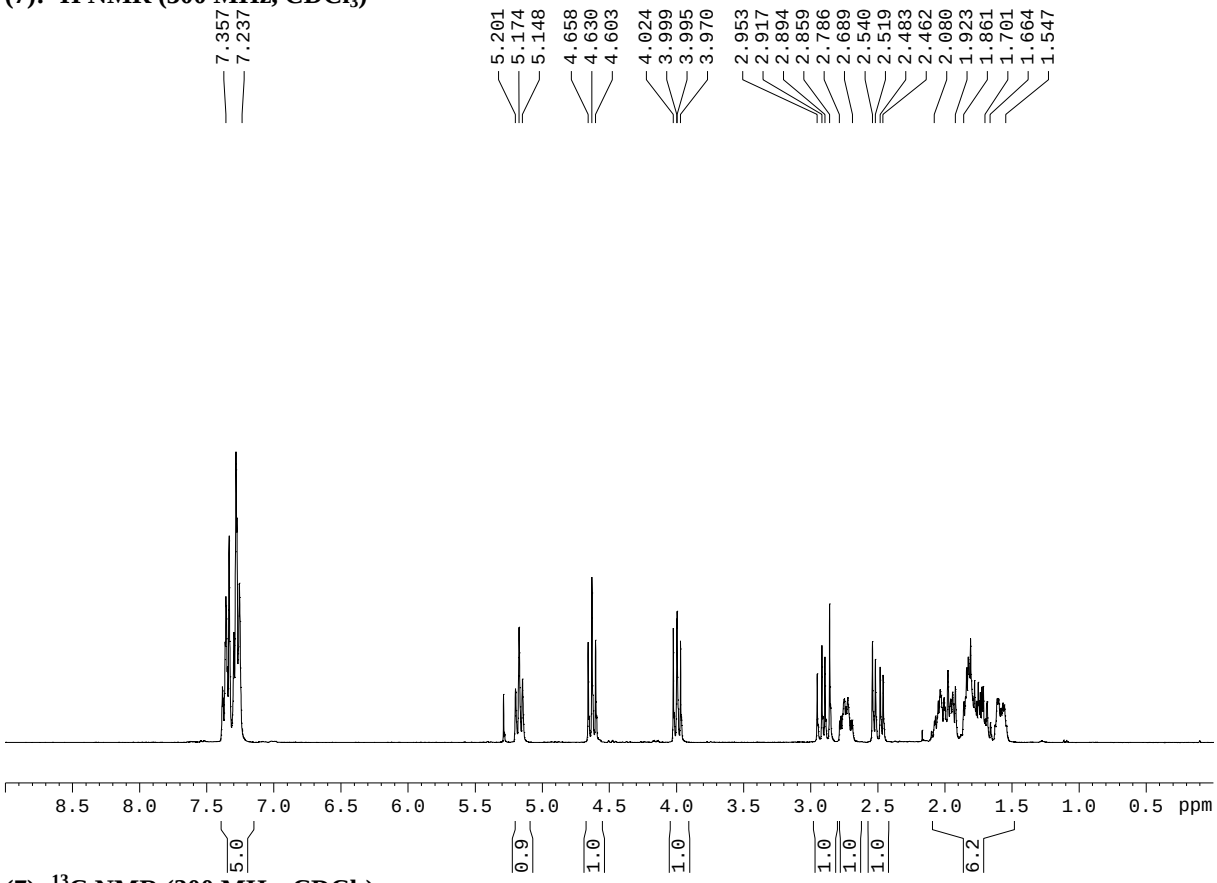
(6b): ^{1}H NMR (300 MHz, $CDCl_3$)



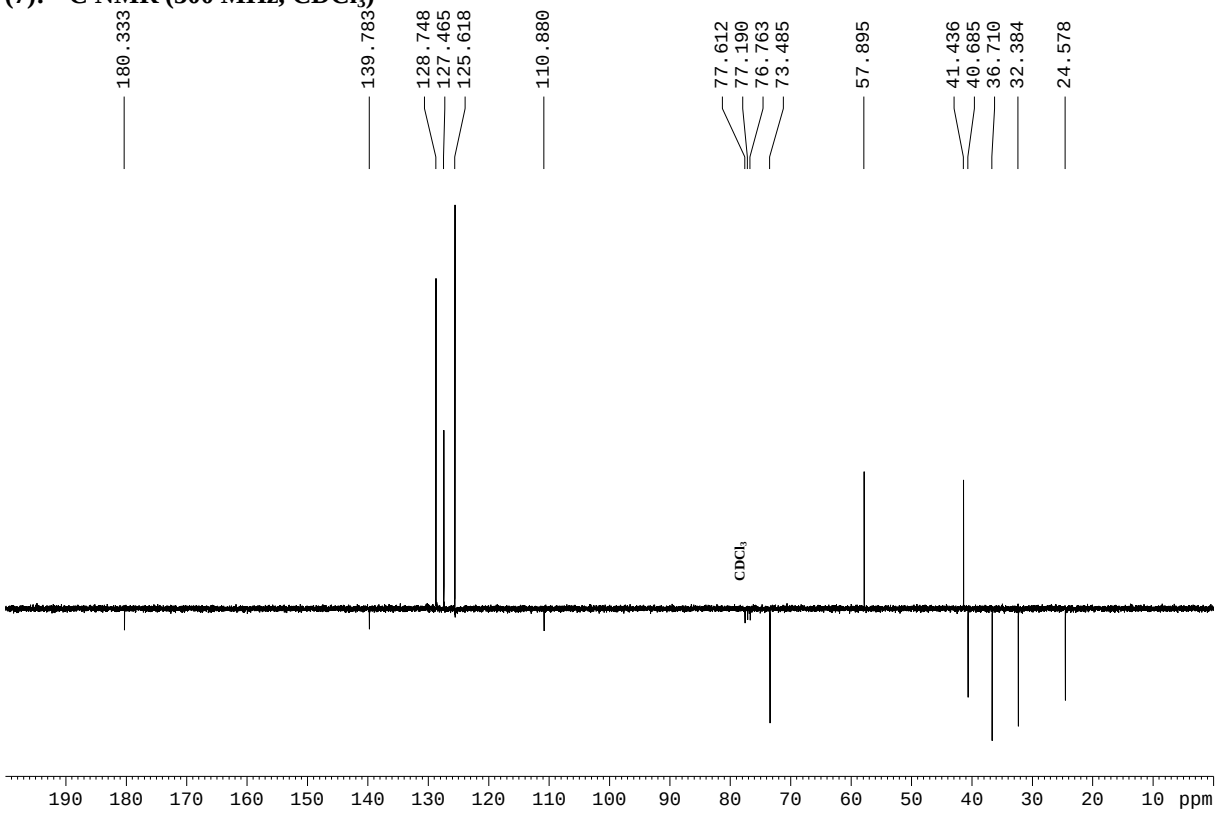
(6b): ^{13}C NMR (300 MHz, $CDCl_3$)



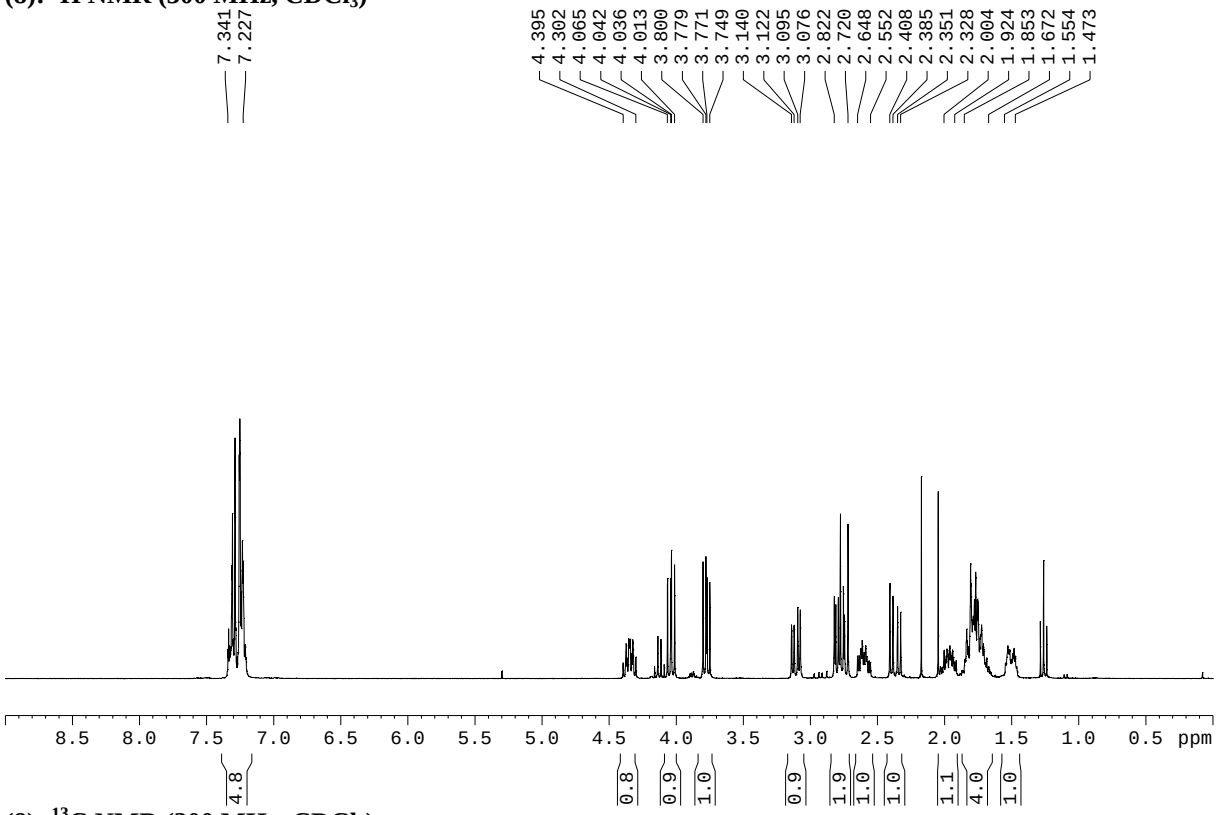
(7): ^{1}H NMR (300 MHz, $CDCl_3$)



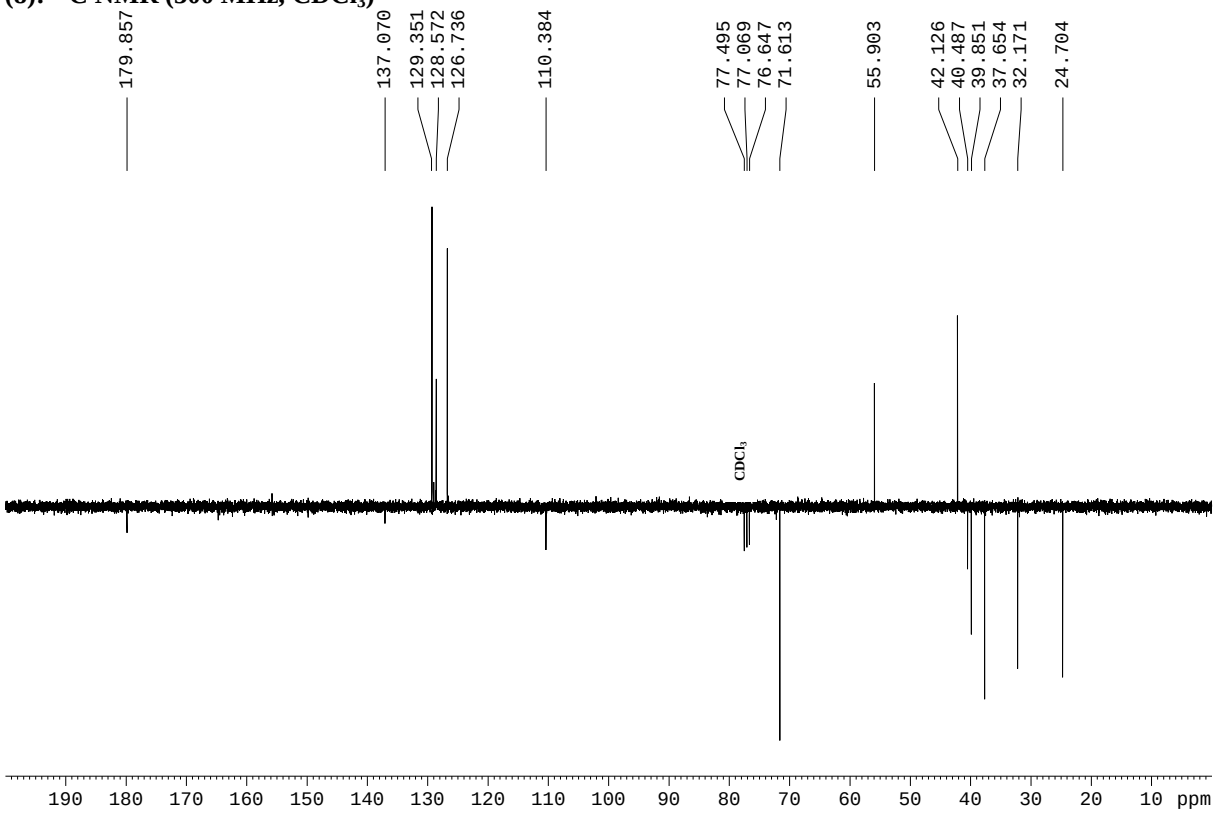
(7): ^{13}C NMR (300 MHz, $CDCl_3$)



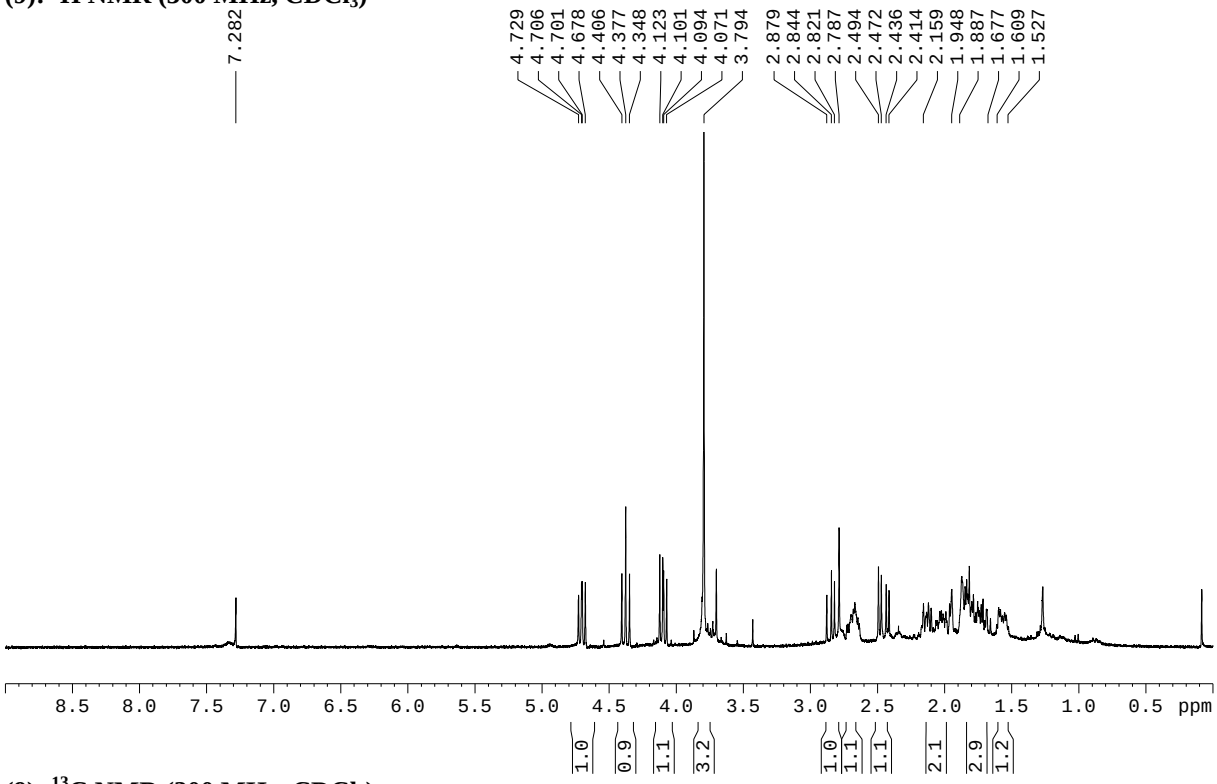
(8): ^{1}H NMR (300 MHz, $CDCl_3$)



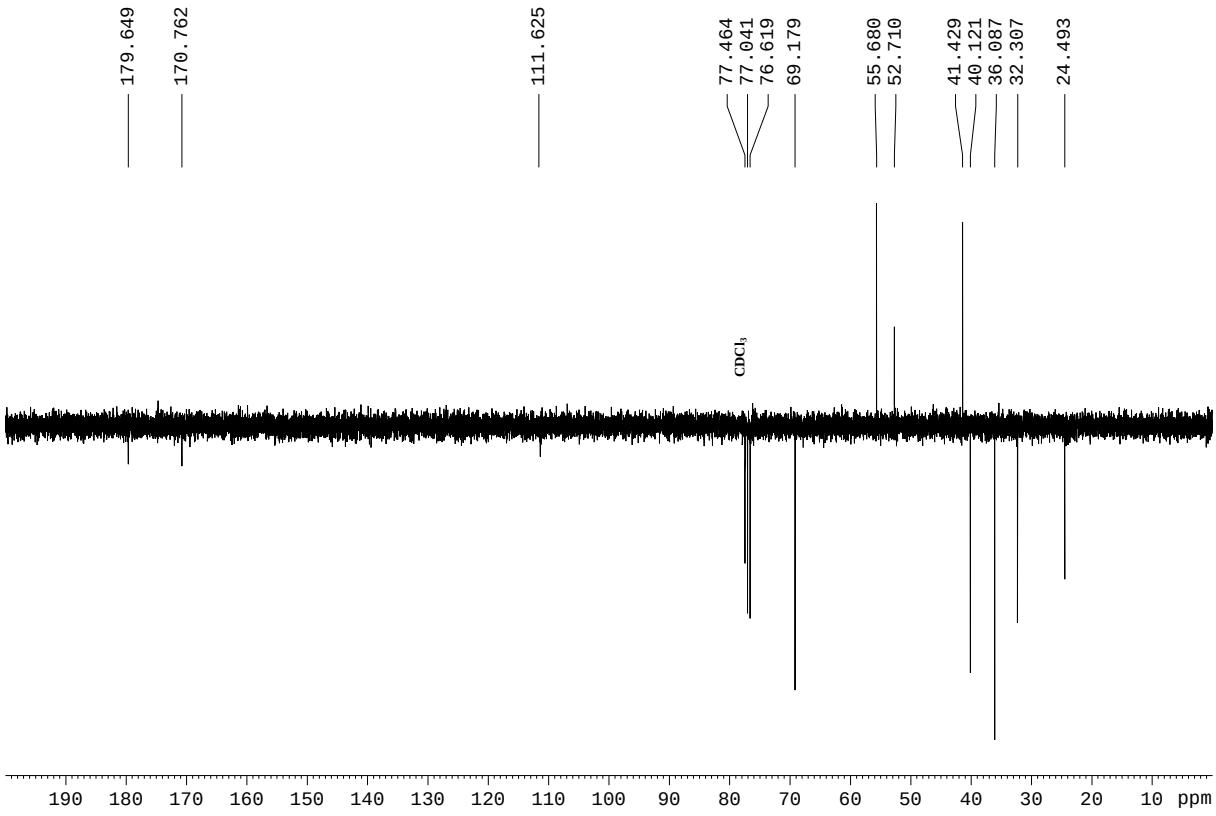
(8): ^{13}C NMR (300 MHz, $CDCl_3$)



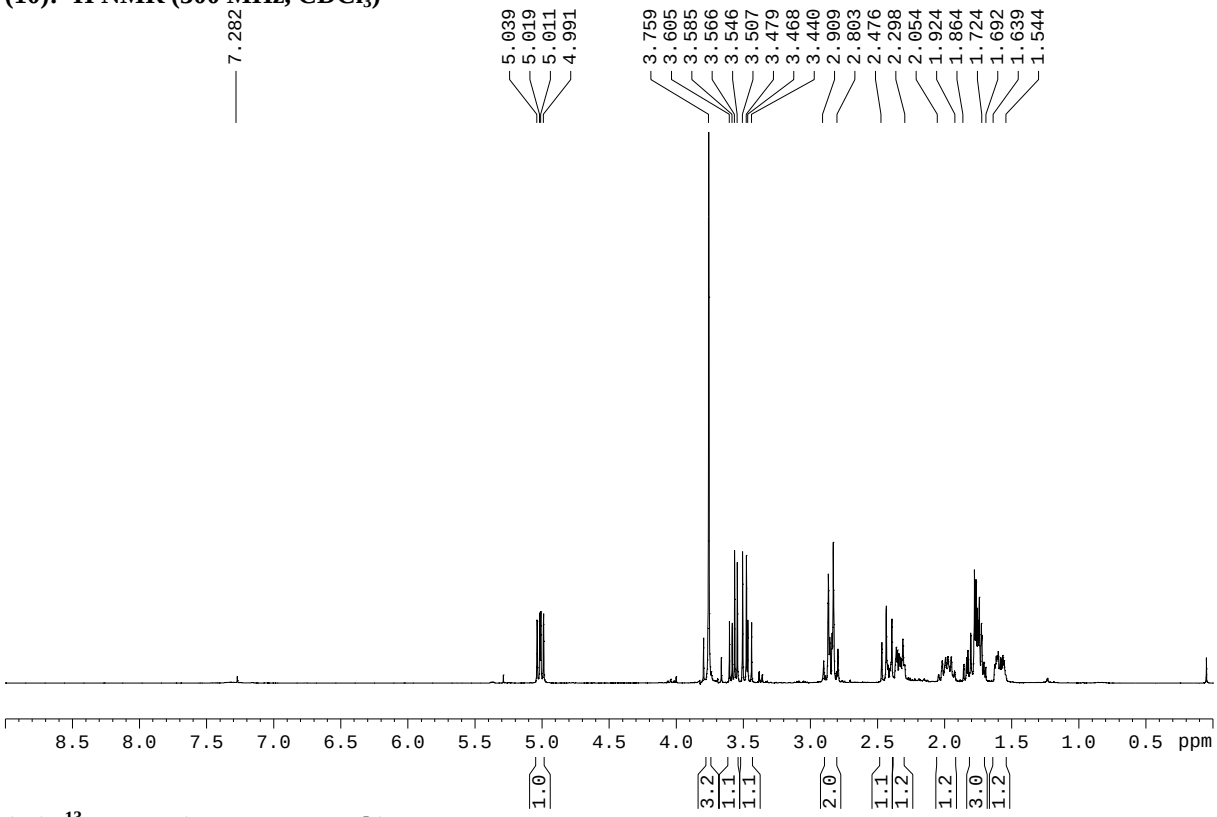
(9): ^{1}H NMR (300 MHz, $CDCl_3$)



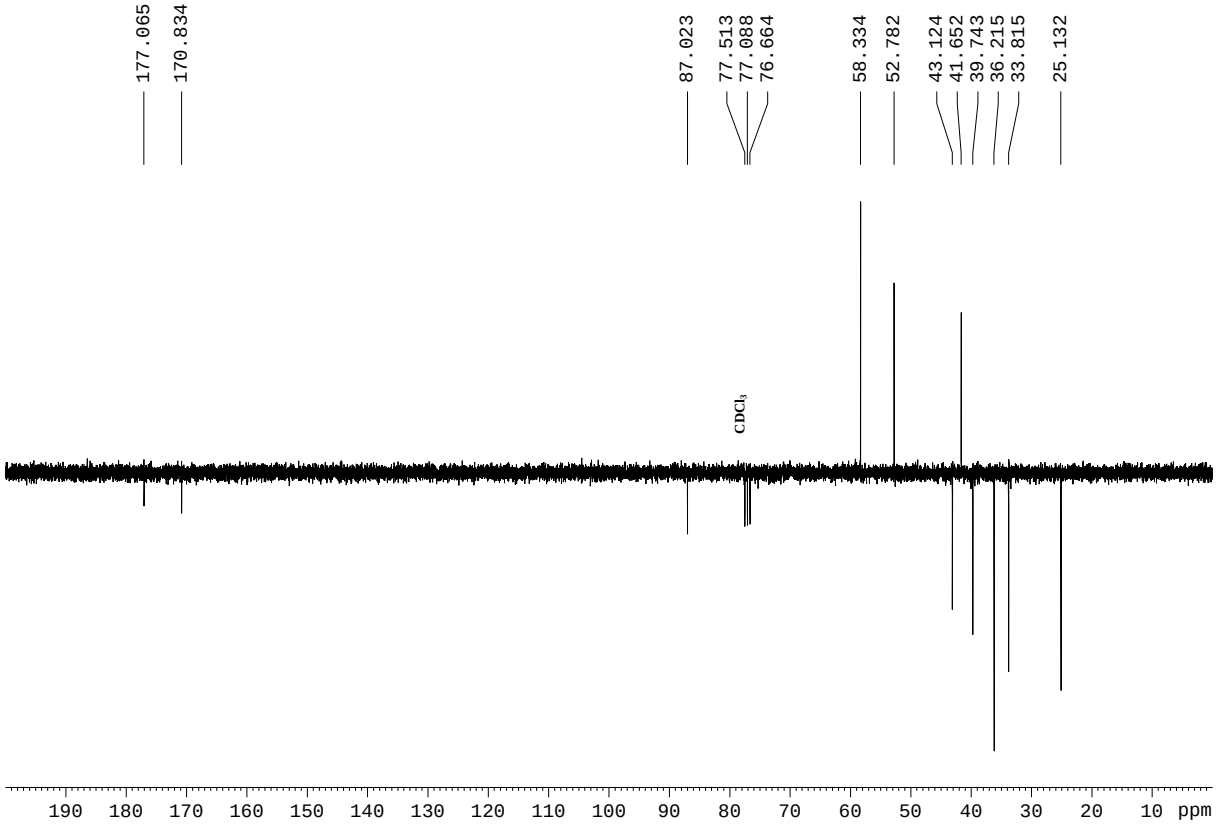
(9): ^{13}C NMR (300 MHz, $CDCl_3$)



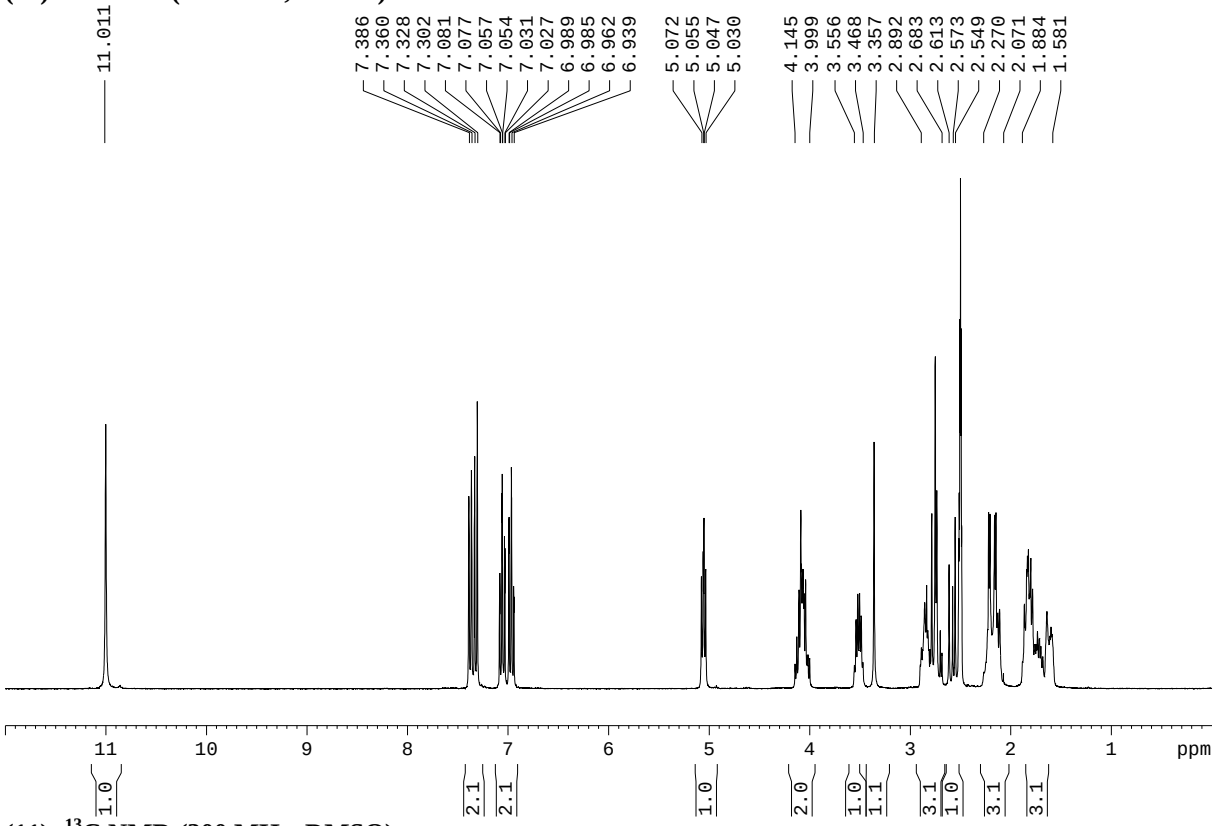
(10): ^{1}H NMR (300 MHz, $CDCl_3$)



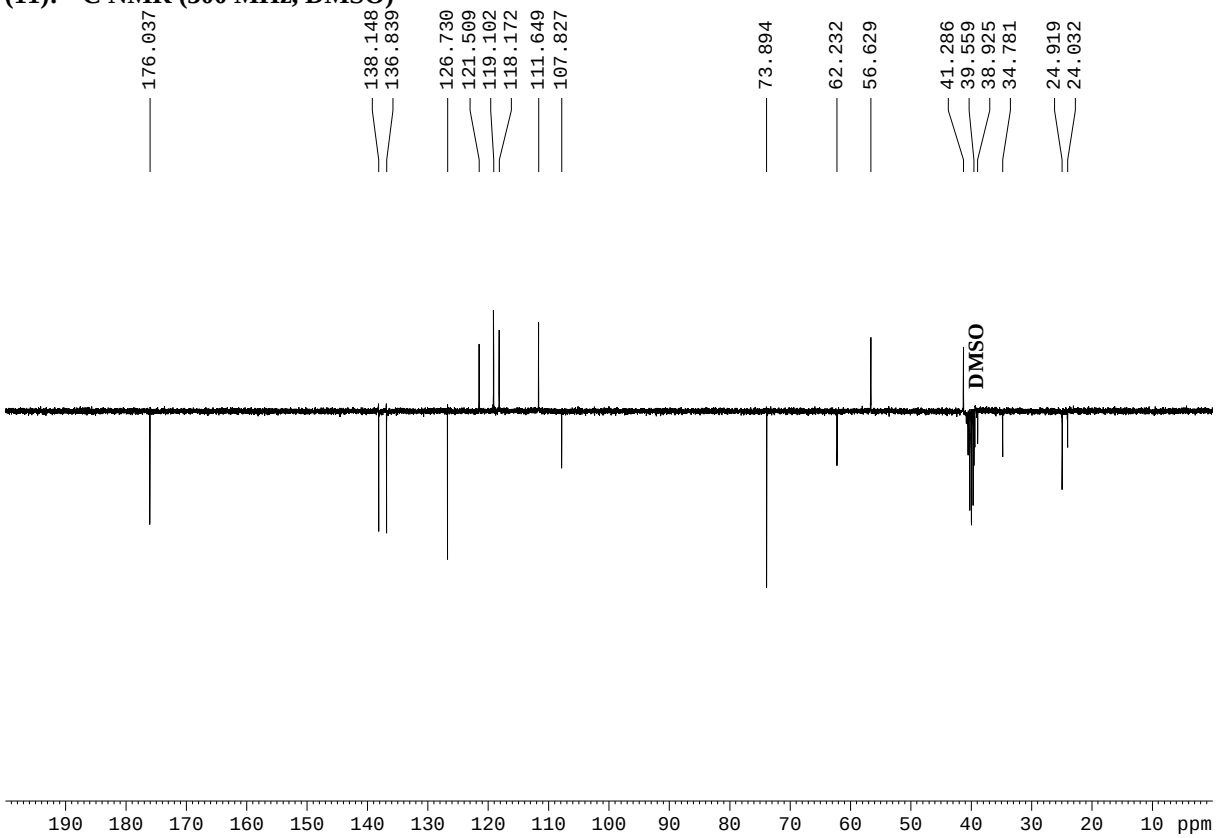
(10): ^{13}C NMR (300 MHz, $CDCl_3$)



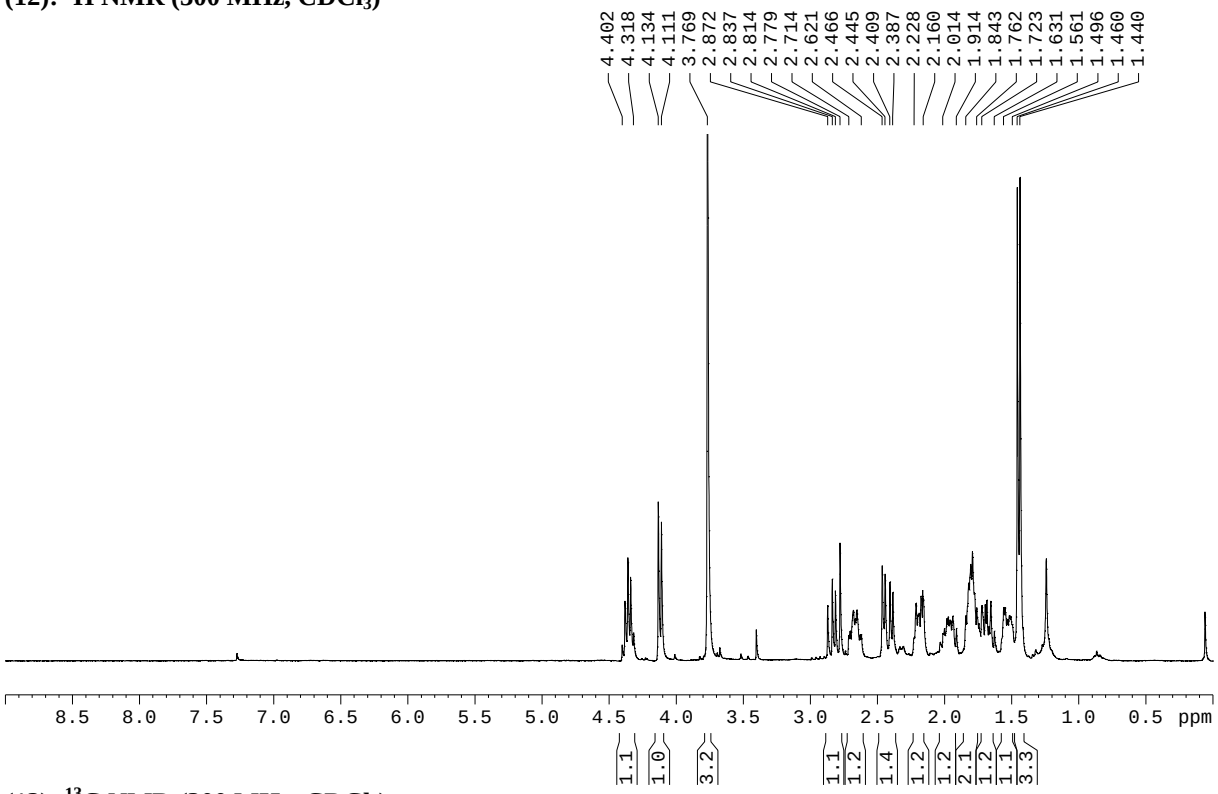
(11): ^{1}H NMR (300 MHz, DMSO)



(11): ^{13}C NMR (300 MHz, DMSO)



(12): ^{1}H NMR (300 MHz, $CDCl_3$)



(12): ^{13}C NMR (300 MHz, $CDCl_3$)