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Rapid Synthesis of Hexahydropyrrolo[3,4-b]pyrrole-Fused Quinolines via A Consecutive [3+2] Cycloaddition and Reduction/Intramolecular Lactamization Cascade

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General Methods

^{1}H NMR (400 MHz) and ^{13}C NMR (101 MHz) spectra were recorded on 400-MR automated spectrometer. Chemical shifts are reported in parts per million (ppm) on the δ scale from an internal standard (TMS). Analytical thin-layer chromatography (TLC) was performed using 0.25 mm silica gel-coated Kiselgel 60 F_{254} plates. Flash chromatography was performed using the indicated solvent and silica gel 60 (Merck, 230-400 mesh). High-resolution mass spectra (HRMS) were recorded in ESI mode using TOF mass spectrometer. IR spectra were recorded using a Bruker spectrophotometer. All materials were purchased from commercial sources and used without further purification.

A representative procedure for the synthesis of 1-(3-methoxypropyl)-1H-pyrrole-2,5-dione (3b)

To the stirred solution of 3-methoxypropylamine (200 mg, 0.0022 mmol) in acetic acid (5 mL) was added maleic anhydride (439 mg, 0.0044 mmol) and the reaction mixture was refluxed for 8 h. After completion of the reaction, solvent was evaporated under vacuum. The residue was neutralized by saturated $NaHCO_3$ solution and extracted with ethyl acetate (20 mL x 3). The combined organic layers were dried over $MgSO_4$ and concentrated *in vacuo*. The crude product was purified by flash column chromatography (15-30% ethyl acetate in hexanes) to afford 1-(3-methoxypropyl)-1H-pyrrole-2,5-dione **3b** (132 g, 60%)

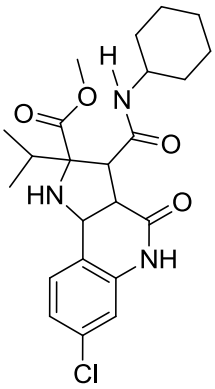
A representative procedure for the synthesis of 9a-benzyl-8-(3-methoxypropyl)-5,6a,6b,9a,10,10a-hexahydropyrrolo [3',4':4,5] pyrrolo[3,2-c]quinoline-6,7,9(8H)-trione (9a)

A mixture of L-phenylalanine methyl ester **1b** (0.1 g, 0.55 mmol), 2-nitrobenzaldehyde **2b** (0.092 g, 0.61 mmol) and 1-(3-methoxypropyl)-1H-pyrrole-2,5-dione **3b** (0.1 g, 0.61 mmol) in the presence of AcOH (3.1 µL, 0.05 mmol) in toluene (15 mL) was refluxed for 12 h. After completion of the reaction, solvent was evaporated under reduced pressure and the crude

product was purified by flash column chromatography (30-35% ethyl acetate in hexanes) to afford methyl 1-benzyl-5-(3-methoxypropyl)-3-(2-nitrophenyl)-4,6-dioxooctahydropyrrolo [3,4-c]pyrrole-1-carboxylate **6a** (0.241 g, 90%). To the stirred solution of methyl 1-benzyl-5-(3-methoxypropyl)-3-(2-nitrophenyl)-4,6-dioxooctahydropyrrolo[3,4-c]pyrrole-1-carboxylate **6a** (0.241 g, 0.5 mmol) in acetonitrile was added zinc (0.49 g, 7.5 mmol) and ammonium formate (0.23 g, 3.75 mmol) and the reaction was refluxed for 12 h. After completion of the reaction zinc powder was filtered through celite bed and the filterate was evaporated under reduced pressure. Dichloromethane was added to the crude product and the precipitated ammonium formate was filtered through celite bed. Filtrate was concentrated in vacuo and the crude product was purified by flash column chromatography (30-60% ethyl acetate in hexanes) to afford 9a-benzyl-8-(3-methoxypropyl)-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-c]quinoline-6,7,9(8H)-trione **9a** (0.142 g, 68%).

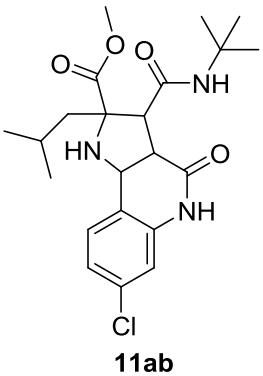
Characterization Data

methyl 7-chloro-3-(cyclohexylcarbamoyl)-2-isopropyl-4-oxo-2,3,3a,4,5,9b-hexahydro-1H-pyrrolo[3,2-c]quinoline-2-carboxylate (11a)



^{1}H NMR (400 MHz, DMSO-d_6) δ 10.14 (s, 1H), 7.87 (d, *J* = 7.8 Hz, 1H), 7.34 (d, *J* = 8.2 Hz, 1H), 6.97 (d, *J* = 8.2 Hz, 1H), 6.80 (s, 1H), 4.46 (s, 1H), 3.49 (s, 3H), 3.29 – 3.17 (m, 2H), 1.92 – 1.84 (m, 1H), 1.66 – 1.40 (m, 5H), 1.25 – 1.01 (m, 5H), 0.98 (d, *J* = 6.7 Hz, 3H), 0.93 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 171.9, 170.3, 168.5, 138.3, 132.0, 129.9, 122.5, 122.1, 114.4, 76.0, 55.3, 55.0, 51.6, 47.5, 46.8, 32.6, 32.5, 32.4, 25.6, 24.7, 18.5, 18.0; HRMS (ESI) calcd. for $C_{25}H_{31}ClN_3O_4$ 448.2003; found 448.1998.

Methyl 3-(tert-butylcarbamoyl)-7-chloro-2-isobutyl-4-oxo-2,3,3a,4,5,9b-hexahydro-1H-pyrrolo[3,2-c]quinoline-2-carboxylate (11ab)



11ab

^{1}H NMR (400 MHz, $CDCl_3$) *δ* 9.21 (s, 1H), 7.38 (d, *J* = 8.2 Hz, 1H), 6.96 (d, *J* = 8.2 Hz, 1H), 6.78 (s, 1H), 5.78 (s, 1H), 4.48 (s, 1H), 3.69 (s, 3H), 3.60 – 3.49 (m, 1H), 3.37 (s, 1H), 3.19 (d, *J* = 8.5 Hz, 1H), 1.89 (tt, *J* = 12.8, 6.5 Hz, 1H), 1.77 (dd, *J* = 14.1, 7.0 Hz, 1H), 1.56 (dd, *J* = 14.1, 5.0 Hz, 1H), 1.18 (s, 9H), 1.02 (d, *J* = 6.6 Hz, 3H), 0.85 (d, *J* = 6.6 Hz,

3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.9, 170.2, 170.0, 136.3, 133.8, 130.3, 123.4, 119.8, 115.4, 73.0, 61.5, 55.6, 52.1, 51.2, 47.2, 44.2, 28.3, 24.5, 24.4, 22.8; HRMS (ESI) calcd. for $C_{22}H_{31}ClN_3O_4$ 436.2003; found 436.2009.

9a-benzyl-8-(3-methoxypropyl)-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-*c*]quinoline-6,7,9(8*H*)-trione (9a)

^{1}H NMR (300 MHz, $CDCl_3$) δ 9.13 (s, 1H), 7.37 (d, *J* = 7.5 Hz, 1H), 7.31 – 7.18 (m, 5H), 7.14 (t, *J* = 7.5 Hz, 1H), 7.00 (t, *J* = 7.5 Hz, 1H), 6.78 (d, *J* = 7.8 Hz, 1H), 4.67 (d, *J* = 7.2 Hz, 1H), 3.53 (d, *J* = 10.2 Hz, 1H), 3.30 (d, *J* = 13.4 Hz, 1H), 3.22 (dd, *J* = 10.2, 7.4 Hz, 1H), 3.16 (s, 3H), 3.05 (d, *J* = 13.4 Hz, 1H), 2.98 – 2.79 (m, 5H), 1.14 – 0.97 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 179.6, 174.2, 168.4, 135.6, 134.4, 130.2, 129.4, 129.0, 127.7, 126.8, 124.0, 122.5, 116.1, 70.4, 69.7, 58.5, 58.1, 49.9, 48.0, 41.0, 35.9, 26.8.; HRMS (EI) calcd. for $C_{24}H_{25}N_3O_4$ 419.1845; found 419.1854.; IR (cm^{-1}, neat) 2923, 2856, 1700, 1205.

9a-benzyl-8-methyl-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-*c*]quinoline-6,7,9(8*H*)-trione (9b)

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.63 (s, 1H), 7.36 (d, *J* = 7.5 Hz, 1H), 7.33 – 7.19 (m, 6H), 7.13 (t, *J* = 7.5 Hz, 1H), 7.02 (t, *J* = 7.8 Hz, 1H), 6.75 (d, *J* = 7.5 Hz, 1H), 4.62 (d, *J* = 6.9 Hz, 1H), 3.47 (d, *J* = 9.9 Hz, 1H), 3.22 (d, *J* =

13.6 Hz, 1H), 3.11 – 3.05 (m, 2H), 2.23 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 179.8, 174.4, 168.2, 135.5, 134.4, 130.1, 129.4, 129.1, 127.9, 126.8, 124.0, 122.4, 116.0, 70.4, 58.1, 49.9, 48.4, 40.6, 24.4.; HRMS (EI) calcd. for $C_{21}H_{19}N_3O_3$ 361.1426; found 361.1434.; IR (cm^{-1}, neat) 3318, 1698, 1378, 757.

9a-benzyl-8-phenyl-5,6a,6b,9a,10,10a-hexahydropyrrolo [3',4':4,5]pyrrolo[3,2-c]quinoline-6,7,9(8H)-trione (9c)

^{1}H NMR (600 MHz, Acetone-d_6) δ 9.21 (s, 1H), 7.55 (d, J = 7.6 Hz, 1H), 7.42 (d, J = 7.4 Hz, 2H), 7.35 (t, J = 7.5 Hz, 2H), 7.30 (t, J = 7.2 Hz, 1H), 7.24 – 7.18 (m, 4H), 6.97 (t, J = 7.5 Hz, 1H), 6.90 (d, J = 7.9 Hz, 1H), 6.26 (d, J = 7.3 Hz, 2H), 4.78 (d, J = 7.2 Hz, 1H), 3.68 (d, J = 10.1 Hz, 1H), 3.44 (d, J = 13.1 Hz, 1H), 3.30 – 3.18 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 178.9, 173.5, 168.4, 135.8, 134.3, 131.1, 130.1, 129.3, 128.9, 128.7, 128.6, 127.7, 127.1, 126.3, 124.1, 122.6, 116.3, 70.9, 58.2, 50.1, 48.1, 41.3.; HRMS (EI) calcd. for $C_{26}H_{21}N_3O_3$ 423.1583; found 423.1591.; IR (cm^{-1}, neat) 3322, 1708, 1679.

9a-methyl-8-phenyl-5,6a,6b,9a,10,10a-hexahydropyrrolo [3',4':4,5] pyrrolo[3,2-*c*]quinoline-6,7,9(8*H*)-trione (9d)

^{1}H NMR (300 MHz, $CDCl_3$) δ 9.10 (s, 1H), 7.44 (d, *J* = 7.1 Hz, 1H), 7.21 – 7.13 (m, 5H), 7.01 (t, *J* = 7.1 Hz, 1H), 6.80 (d, *J* = 8.1 Hz, 1H), 6.41 (d, *J* = 4.6 Hz, 2H), 4.74 (d, *J* = 7.1 Hz, 1H), 3.64 – 3.44 (m, 2H), 1.63 (s, 3H).; ^{13}C NMR (75 MHz, $CDCl_3$) δ 179.0, 173.3, 167.6, 137.8, 135.8, 131.2, 129.6, 128.9, 128.8, 127.4, 126.3, 124.5, 122.6, 116.1, 66.2, 58.6, 53.5, 48.8, 23.0**.;** HRMS (EI) calcd. for $C_{20}H_{17}N_3O_3$ 347.1270; found 347.1265**.**; IR (cm^{-1}, neat) 3774, 3710, 1706, 1668.

9a-benzyl-8-(3-phenylpropyl)-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-*c*]quinoline-6,7,9(8*H*)-trione (9e)

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.78 (s, 1H), 7.38 – 7.11 (m, 10H), 7.04 – 6.95 (m, 2H), 6.93 – 6.83 (m, 2H), 6.71 – 6.60 (m, 1H), 4.62 (d, *J* = 7.3 Hz, 1H), 3.50 (d, *J* = 10.2 Hz, 1H), 3.22 (d, *J* = 13.5 Hz, 1H), 3.16 (dd, *J* = 10.2, 7.3 Hz, 1H), 3.03 (d, *J* = 13.5 Hz, 1H), 2.89 – 2.76 (m, 2H), 2.28 – 2.12 (m, 2H), 1.10 – 0.96 (m, 2H).; ^{13}C NMR (75 MHz, $CDCl_3$) δ 179.6, 174.1, 168.1, 141.1, 135.5, 134.3, 130.2, 129.5, 129.0, 128.4, 128.3, 127.8, 126.8, 126.0, 124.1, 122.5, 115.9, 70.4, 58.1, 50.0, 48.1, 41.0, 38.5, 32.8, 27.9**.;** HRMS

(EI) calcd. for $C_{29}H_{27}N_3O_3$ 465.2052; found 465.2053; IR (cm^{-1}, neat) 1693, 1595, 1396.

8-(3-methoxypropyl)-9a-methyl-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-*c*]quinoline-6,7,9(8*H*)-trione (9f)

^{1}H NMR (300 MHz, $CDCl_3$) δ 9.27 (s, 1H), 7.35 (d, *J* = 7.6 Hz, 1H), 7.12 (t, *J* = 7.5 Hz, 1H), 6.96 (t, *J* = 7.5 Hz, 1H), 6.81 (d, *J* = 7.7 Hz, 1H), 4.71 (d, *J* = 7.3 Hz, 1H), 3.50 (dd, *J* = 10.1, 7.5 Hz, 1H), 3.34 (d, *J* = 10.2 Hz, 1H), 3.17 (s, 1H), 3.09 (t, *J* = 5.9 Hz, 1H), 2.99 (t, *J* = 7.4 Hz, 1H), 1.55 (s, 1H), 1.08 (dq, *J* = 12.5, 6.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 179.9, 174.3, 168.6, 135.8, 129.3, 127.1, 124.0, 122.5, 116.1, 70.1, 66.3, 58.6, 58.5, 53.5, 48.5, 36.4, 26.8, 22.5; HRMS (EI) calcd. for $C_{18}H_{21}N_3O_4$ 343.1532; found 343.1523; IR (cm^{-1}, neat) 3324, 1698, 1396, 759.

8,9a-dimethyl-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-*c*]quinoline-6,7,9(8*H*)-trione (9g)

^{1}H NMR (300 MHz, CD_3CN) δ 8.27 (s, 1H), 7.34 (d, *J* = 7.7 Hz, 1H), 7.15 – 7.07 (m, 1H), 6.93 (td, *J* = 7.5, 1.1 Hz, 1H), 6.72 (d, *J* = 7.9 Hz, 1H), 4.61 (d, *J* = 6.7 Hz, 1H), 3.31 – 3.15 (m, 2H), 2.19 (s, 3H), 1.44 (s, 3H); ^{13}C NMR (75 MHz, CD_3CN) δ 180.6, 175.8, 168.5, 17.45, 129.7, 128.0, 124.2, 123.8,

115.9, 67.6, 59.6, 54.1, 49.8, 24.4, 21.6; HRMS (EI) calcd. for $C_{15}H_{15}N_3O_3$ 285.1113; found; 285.1113; IR (cm^{-1}, neat) 3318, 1697, 1380, 757.

9a-benzyl-8-(2-methylpropyl)-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-*c*]quinoline-6,7,9(8*H*)-trione (9h)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (s, 1H), 7.34 (d, *J* = 7.7 Hz, 1H), 7.35-7.23 (m, 3H), 7.20 (d, *J* = 7.7 Hz, 2H), 7.14 (t, *J* = 7.6 Hz, 1H), 7.01 (t, *J* = 7.4 Hz, 1H), 6.64 (d, *J* = 7.9 Hz, 1H), 4.64 (d, *J* = 7.1 Hz, 1H), 3.51 (d, *J* = 10.0 Hz, 1H), 3.30 (d, *J* = 13.4 Hz, 1H), 3.22 (dd, *J* = 10.0, 7.2 Hz, 1H), 3.00 (d, *J* = 13.5 Hz, 1H), 2.52 (dd, *J* = 13.1, 6.5 Hz, 1H), 2.40 (dd, *J* = 13.0, 8.1 Hz, 1H), 1.51 – 1.39 (m, 1H), 0.52 (d, *J* = 6.7 Hz, 3H), 0.34 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 179.4, 174.3, 167.3, 135.1, 134.1, 130.1, 129.3, 128.8, 127.6, 126.6, 123.9, 122.3, 115.4, 70.0, 57.7, 49.4, 47.8, 45.6, 41.2, 26.7, 19.9, 19.7; HRMS (EI) calcd. for $C_{24}H_{25}N_3O_3$ 403.1896; found 403.1893; IR (cm^{-1}, neat) 3320, 1698, 1394, 757.

9a-methyl-8-(2-methylpropyl)-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-*c*]quinoline-6,7,9(8*H*)-trione (9i)

^{1}H NMR (300 MHz, $CDCl_3$) δ 9.10 (s, 1H), 7.34 (d, *J* = 7.5 Hz, 1H), 7.13 (t, *J* = 7.5 Hz, 1H), 6.97 (t, *J* = 7.5 Hz, 1H), 6.81 (d, *J* = 7.8 Hz, 1H), 4.71 (d, *J* = 7.0 Hz, 1H), 3.58 – 3.40 (m, 1H), 3.34 (d, *J* = 10.0 Hz, 1H), 2.61 (d, *J* =

7.0 Hz, 2H), 1.70 – 1.51 (m, 1H), 1.56 (s, 3H), 0.64 (d, *J* = 3.7 Hz, 3H), 0.62 (d, *J* = 3.7 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 180.3, 174.7, 168.5, 135.7, 129.4, 127.0, 124.0, 122.3, 116.1, 66.0, 58.5, 53.2, 48.4, 45.7, 27.0, 22.8, 20.3, 20.1.; HRMS (EI) calcd. for $C_{18}H_{21}N_3O_3$ 327.1583; found 327.1579.; IR (cm^{-1}, neat) 3322, 1698, 1363, 757.

9a-benzyl-8-pentyl-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-*c*]quinoline-6,7,9(8*H*)-trione (9j)

^{1}H NMR (300 MHz, $CDCl_3$); δ 8.34 (s, 1H), 7.35 (d, *J* = 7.5 Hz, 1H), 7.32 – 7.18 (m, 5H), 7.13 (t, *J* = 7.5 Hz, 1H), 7.00 (t, *J* = 7.5 Hz, 1H), 6.69 (d, *J* = 7.7 Hz, 1H), 4.65 (d, *J* = 7.3 Hz, 1H), 3.50 (d, *J* = 10.1 Hz, 1H), 3.27 (d, *J* = 13.4 Hz, 1H), 3.17 (dd, *J* = 10.1, 7.3 Hz, 1H), 3.02 (d, *J* = 13.4 Hz, 1H), 2.81 – 2.66 (m, 2H), 1.17 – 1.01 (m, 2H), 0.92 – 0.63 (m, 4H), 0.75 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 179.5, 174.1, 167.9, 135.5, 134.4, 130.2, 129.5, 129.0, 127.8, 126.9, 124.1, 122.6, 115.8, 70.3, 58.1, 49.9, 48.1, 41.1, 38.8, 28.8, 26.3, 22.3, 14.0; HRMS (EI) calcd. for $C_{25}H_{27}N_3O_3$ 417.2052; found 417.2050; IR (cm^{-1}, neat) 3336, 1697, 1398, 759.

9a-methyl-8-pentyl-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-*c*]quinoline-6,7,9(8*H*)-trione (9k)

^{1}H NMR (300 MHz, $CDCl_3$) δ 9.04 (s, 1H), 7.35 (d, *J* = 7.5 Hz, 1H), 7.12 (t, *J* = 7.5 Hz, 1H), 6.97 (t, *J* = 7.5 Hz, 1H), 6.79 (d, *J* = 7.8 Hz, 1H), 4.71 (d, *J* = 7.4 Hz, 1H), 3.49 (dd, *J* = 10.2, 7.4 Hz, 1H), 3.33 (d, *J* = 10.2 Hz, 1H), 2.93 – 2.82 (m, 2H), 1.56 (s, 3H), 1.20 – 1.06 (m, 2H), 1.06 – 0.91 (m, 2H), 0.78 (t, *J* = 7.2 Hz, 3H), 0.83 – 0.68 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 179.9, 174.3, 168.6, 135.8, 129.4, 127.0, 124.0, 122.5, 116.1, 66.2, 58.6, 53.4, 48.4, 38.9, 28.9, 26.2, 22.6, 22.3, 14.0; HRMS (EI) calcd. for $C_{19}H_{23}N_3O_3$ 341.1739; found 341.1734.; IR (cm^{-1}, neat) 2933, 1700, 1367, 759.

9a-methyl-8-phenyl-6b,9a,10,10a-tetrahydro-6*H*-chromeno[4,3-*b*]pyrrolo[3,4-*d*]pyrrole-6,7,9(6a*H*,8*H*)-trione (9l)

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.49 (d, *J* = 6.5 Hz, 1H), 7.40 – 7.23 (m, 4H), 7.18 (d, *J* = 6.5 Hz, 1H), 7.06 (d, *J* = 7.5 Hz, 1H), 6.50 (s, 2H), 4.80 (d, *J* = 5.8 Hz, 1H), 3.75 (t, *J* = 8.2 Hz, 1H), 3.52 (d, *J* = 9.6 Hz, 1H), 1.68 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 178.4, 173.0, 165.1, 150.2, 130.9, 130.6, 128.9, 128.9, 127.5, 126.1, 125.8, 121.7, 117.7, 66.1, 57.3, 53.7, 47.0, 23.0;

HRMS (EI) calcd. for $C_{20}H_{16}N_2O_4$ 348.1110; found 348.1100; IR (cm^{-1}, neat) 3334, 1760, 1710.

9a-benzyl-8-[2-(cyclohex-1-en-1-yl)ethyl]-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-*c*]quinoline-6,7,9(8*H*)-trione (9m)

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.25 (s, 1H), 7.36 (d, *J* = 7.5 Hz, 1H), 7.33 – 7.19 (m, 5H), 7.15 (t, *J* = 7.5 Hz, 1H), 7.00 (t, *J* = 7.5 Hz, 1H), 6.69 (d, *J* = 7.8 Hz, 1H), 5.07 (s, 1H), 4.62 (d, *J* = 7.2 Hz, 1H), 3.47 (d, *J* = 10.1 Hz, 1H), 3.25 – 3.01 (m, 3H), 2.94 – 2.79 (m, 2H), 1.95 – 1.63 (m, 5H), 1.58 – 1.32 (m, 5H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ* 179.2, 173.7, 167.7, 135.3, 135.1, 134.2, 133.9, 130.1, 129.3, 128.9, 127.6, 126.7, 125.0, 123.9, 122.8, 122.4, 115.7, 70.0, 57.9, 49.8, 48.0, 40.7, 37.0, 34.3, 27.9, 25.1, 22.6, 22.1; HRMS (EI) calcd. for $C_{28}H_{29}N_3O_3$ 455.2209; found 455.2213; IR (cm^{-1}, neat) 2917, 1693, 755.

8-(3,3-diphenylpropyl)-9a-methyl-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-*c*]quinoline-6,7,9(8*H*)-trione (9n)

^{1}H NMR (300 MHz, $CDCl_3$) δ 9.18 (s, 1H), 7.34 (d, *J* = 7.6 Hz, 1H), 7.30 – 7.05 (m, 10H), 6.88 – 6.73 (m, 1H), 6.73 – 6.61 (m, 1H), 4.67 (d, *J* = 7.4 Hz, 1H), 3.67 (t, *J* = 7.8 Hz, 1H), 3.48 (dd, *J* = 10.2, 7.5 Hz, 1H), 3.25 (d, *J* = 10.2 Hz, 1H), 2.98 – 2.93 (m, 2H), 1.47 (s, 3H), 1.44 – 1.30 (m, 2H); ^{13}C

NMR (75 MHz, $CDCl_3$) δ 179.6, 174.1, 168.4, 143.8, 143.7, 135.6, 129.7, 128.5, 128.5, 127.8, 127.8, 127.0, 126.5, 126.4, 124.1, 122.2, 116.0, 66.3, 58.6, 53.4, 49.2, 48.3, 38.1, 31.6, 22.5; HRMS (EI) calcd. for $C_{29}H_{27}N_3O_3$ 469.2052; found 469.2048; IR (cm^{-1}, neat) 2927, 1693, 1365, 698.

8-(4-methoxybenzyl)-9a-methyl-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-*c*]quinoline-6,7,9(8*H*)-trione (9o)

^1H NMR (300 MHz, $CDCl_3$) δ 8.96 (s, 1H), 7.33 (d, *J* = 7.6 Hz, 1H), 7.17 – 7.00 (m, 3H), 6.93 (t, *J* = 7.6 Hz, 1H), 6.76 (d, *J* = 7.8 Hz, 1H), 6.69 (d, *J* = 8.6 Hz, 2H), 4.70 (d, *J* = 7.2 Hz, 1H), 3.98 – 3.79 (m, 2H), 3.73 (s, 3H), 3.48 (dd, *J* = 10.1, 7.2 Hz, 1H), 3.31 (d, *J* = 10.1 Hz, 1H), 1.52 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 179.6, 174.2, 168.3, 159.2, 135.6, 130.3, 129.4, 127.3, 126.8, 124.0, 122.3, 116.0, 114.0, 66.2, 58.5, 55.3, 53.3, 48.5, 41.7, 22.5; HRMS (EI) calcd. for $C_{22}H_{21}N_3O_4$ 391.1532; found 391.1541; IR (cm^{-1}, neat) 2919, 1695, 757.

9a-methyl-8-(thiophen-2-ylmethyl)-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-*c*]quinoline-6,7,9(8*H*)-trione (9p)

^1H NMR (300 MHz, $CDCl_3$) δ 8.87 (s, 1H), 7.34 (d, *J* = 7.6 Hz, 1H), 7.16 – 7.04 (m, 2H), 6.93 (td, *J* = 7.6, 0.9 Hz, 1H), 6.80 (d, *J* = 3.5 Hz, 2H), 6.76 (dd, *J* = 7.8, 0.9 Hz, 1H), 4.72 (d, *J* = 7.1 Hz, 1H), 4.15 – 4.06 (m, 2H), 3.49

(dd, *J* = 10.1, 7.1 Hz, 1H), 3.33 (d, *J* = 10.1 Hz, 1H), 1.54 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 179.1, 173.8, 168.2, 136.4, 135.6, 129.4, 128.1, 126.9, 126.9, 126.1, 124.0, 122.2, 116.0, 66.4, 58.6, 53.4, 48.6, 36.3, 22.4; HRMS (EI) calcd. for $C_{19}H_{17}N_3O_3S$ 367.0991; found 367.0998; IR (cm^{-1}, neat) 1700, 1390, 757.

9a-methyl-8-(3-phenylpropyl)-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-*c*]quinoline-6,7,9(8*H*)-trione (9q)

^{1}H NMR (300 MHz, $CDCl_3$) δ 9.45 (s, 1H), 7.37 – 7.29 (m, 1H), 7.24 – 7.11 (m, 4H), 7.02 (d, *J* = 8.0 Hz, 1H), 6.84 (dd, *J* = 5.5, 3.5 Hz, 2H), 6.80 – 6.71 (m, 1H), 4.67 (d, *J* = 7.4 Hz, 1H), 3.50 (dd, *J* = 10.1, 7.4 Hz, 1H), 3.32 (d, *J* = 10.1 Hz, 1H), 2.96 (t, *J* = 7.8 Hz, 2H), 2.33 (t, *J* = 7.8 Hz, 2H), 1.52 (s, 3H), 1.16 – 0.97 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 179.8, 174.2, 168.6, 141.0, 135.7, 129.4, 128.3, 128.3, 127.0, 126.0, 124.0, 122.4, 116.1, 66.2, 58.5, 53.4, 48.4, 38.6, 33.0, 27.9, 22.5; HRMS (EI) calcd. for $C_{23}H_{23}N_3O_3$ 389.1739; found 389.1739; IR (cm^{-1}, neat) 3322, 2927,1698, 1708, 759.

8-[2-(cyclohex-1-en-1-yl)ethyl]-9a-methyl-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-*c*]quinoline-6,7,9(8*H*)-trione (9r)

^{1}H NMR (300 MHz, $CDCl_3$) δ 9.19 (s, 1H), 7.35 (d, *J* = 7.6 Hz, 1H), 7.12 (t, *J* = 7.6 Hz, 1H), 6.97 (t, *J* = 7.6 Hz, 1H), 6.81 (d, *J* = 7.9 Hz, 1H), 5.15 (s,

1H), 4.71 (d, *J* = 7.2 Hz, 1H), 3.49 (dd, *J* = 10.2, 7.2 Hz, 1H), 3.31 (d, *J* = 10.2 Hz, 1H), 2.94 (t, *J* = 7.6 Hz, 2H), 1.84 – 1.74 (m, 4H), 1.53 (s, 3H), 1.52 – 1.37 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 179.9, 174.2, 168.6, 135.8, 134.2, 129.4, 127.0, 124.0, 123.1, 122.5, 116.1, 66.2, 58.6, 53.3, 48.5, 37.0, 34.4, 28.0, 25.2, 22.8, 22.7, 22.2; HRMS (EI) calcd. for $C_{22}H_{25}N_3O_3$ 379.1896; found 379.1899; IR (cm^{-1}, neat) 3318, 3058, 1695.

9a-benzyl-8-(3-methylbutyl)-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-*c*]quinoline-6,7,9(8*H*)-trione (9s)

^{1}H NMR (300 MHz, $CDCl_3$) δ 9.29 (s, 1H), 7.35 (d, *J* = 7.5 Hz, 1H), 7.27 – 7.15 (m, 5H), 7.10 (t, *J* = 7.5 Hz, 1H), 6.97 (t, *J* = 7.5 Hz, 1H), 6.78 (d, *J* = 7.8 Hz, 1H), 4.65 (d, *J* = 7.4 Hz, 1H), 3.52 (d, *J* = 10.2 Hz, 1H), 3.28 (d, *J* = 13.5 Hz, 1H), 3.22 (dd, *J* = 10.2, 7.4 Hz, 1H), 3.02 (d, *J* = 13.5 Hz, 1H), 2.76 (t, *J* = 7.9 Hz, 2H), 1.16 – 0.95 (m, 1H), 0.67 (d, *J* = 6.2 Hz, 3H), 0.65 (d, *J* = 6.2 Hz, 3H), 0.62 – 0.45 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 179.6, 174.1, 168.5, 135.6, 134.4, 130.2, 129.4, 128.9, 127.7, 126.8, 124.1, 122.6, 116.1, 70.4, 58.0, 49.9, 47.9, 41.1, 37.2, 34.9, 25.7, 22.3, 22.2; HRMS (EI) calcd. for $C_{25}H_{27}N_3O_3$ 417.2052; found 417.2050; IR (cm^{-1}, neat) 3315, 2954, 1691, 1365.

9a-benzyl-8-(2-methoxyethyl)-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-*c*]quinoline-6,7,9(8*H*)-trione (9t)

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.89 (s, 1H), 7.35 (d, *J* = 7.6 Hz, 1H), 7.32 – 7.23 (m, 3H), 7.23 – 7.18 (m, 2H), 7.14 (t, *J* = 7.6 Hz, 1H), 7.00 (t, *J* = 7.6 Hz, 1H), 6.76 (d, *J* = 7.8 Hz, 1H), 4.63 (d, *J* = 7.2 Hz, 1H), 3.50 (d, *J* = 10.1 Hz, 1H), 3.25 (d, *J* = 13.6 Hz, 1H), 3.19 – 3.05 (m, 2H), 3.09 (s, 3H), 3.06 – 2.84 (m, 2H), 2.77 (t, *J* = 6.6 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 179.6, 174.1, 168.2, 135.7, 134.4, 130.2, 129.4, 129.0, 127.8, 126.9, 124.0, 122.5, 116.1, 70.3, 67.7, 58.6, 58.1, 49.9, 48.2, 40.9, 37.6; HRMS (EI) calcd. for $C_{23}H_{23}N_3O_4$ 405.1689; found 405.1685; IR (cm^{-1}, neat) 3320, 1687, 1342.

9a-benzyl-2,3-dimethoxy-8-(3-methoxypropyl)-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-c]quinoline-6,7,9(8H)-trione (9u)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.96 (s, 1H), 7.25-7.17 (m, 5H), 6.91 (s, 1H), 6.32 (s, 1H), 4.60 (d, *J* = 6.8 Hz, 1H), 3.84 (s, 3H), 3.76 (s, 3H), 3.50 (d, *J* = 10.2 Hz, 1H), 3.29 (d, *J* = 13.3 Hz, 1H), 3.03 (d, *J* = 13.4 Hz, 1H), 2.93 – 2.81 (m, 5H), 1.14 – 0.99 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 179.3, 173.9, 168.2, 149.6, 145.5, 134.1, 129.9, 128.9, 128.8, 127.7, 113.4, 109.7, 100.2, 70.7, 69.4, 58.3, 58.2, 56.3, 56.0, 48.7, 40.7, 35.8, 27.0; HRMS (ESI) calcd. for $C_{26}H_{30}N_3O_6$ 480.2135; found 480.2138.

9a-((1H-indol-3-yl)methyl)-3-chloro-8-cyclohexyl-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-c]quinoline-6,7,9(8H)-trione (9v)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.71 (s, 1H), 8.42 (s, 1H), 7.64 (d, *J* = 7.7 Hz, 1H), 7.35 (t, *J* = 7.4 Hz, 2H), 7.24 – 7.07 (m, 3H), 6.96 (d, *J* = 8.1 Hz, 1H), 6.73 (s, 1H), 4.52 (d, *J* = 7.1 Hz, 1H), 3.53 (d, *J* = 10.4 Hz, 1H), 3.46 – 3.33 (m, 2H), 3.29 (d, *J* = 14.5 Hz, 1H), 3.19 – 3.08 (m, 1H), 1.63 – 1.37 (m, 5H), 1.10 – 0.92 (m, 3H), 0.52 (d, *J* = 11.7 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 179.7, 174.2, 168.2, 136.7, 135.9, 134.8, 128.8, 127.5, 123.9, 123.7, 122.5, 121.0, 120.2, 118.5, 115.6, 111.4, 108.2, 70.5, 58.0, 51.7, 51.0, 48.3, 30.6, 27.6, 27.3, 25.5, 24.7; HRMS (ESI) calcd. for $C_{28}H_{28}ClN_4O_3$ 503.1850; found 503.1850.

8-cycloheptyl-2,3-dimethoxy-9a-(2-(methylthio)ethyl)-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-c]quinoline-6,7,9(8H)-trione (9w)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.62 (s, 1H), 6.84 (s, 1H), 6.34 (s, 1H), 4.66 (d, *J* = 6.2 Hz, 1H), 3.83 (s, 3H), 3.80 (s, 3H), 3.71 – 3.58 (m, 1H), 3.50 – 3.39 (m, 1H), 2.14 – 2.00 (m, 4H), 1.66 – 1.50 (m, 5H), 1.49 – 1.34 (m, 5H), 1.25 (s, 3H), 1.23 – 1.12 (m, 1H), 0.93 – 0.82 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 179.0, 173.6, 168.1, 150.0, 145.8, 128.9, 113.5, 109.7, 100.3, 68.8,

57.6, 56.4, 56.1, 53.5, 50.6, 48.6, 34.6, 30.9, 30.5, 29.2, 28.6, 27.4, 25.3, 25.2, 15.6; HRMS (ESI) calcd. for $C_{25}H_{34}N_3O_5S$ 488.2219; found 488.2222.

8-(2-(cyclohex-1-en-1-yl)ethyl)-9a-(4-hydroxybenzyl)-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-c]quinoline-6,7,9(8H)-trione (9x)

^{1}H NMR (400 MHz, Acetone-d_6) δ 9.12 (s, 1H), 8.31 (s, 1H), 7.44 (d, J = 7.6 Hz, 1H), 7.16 (d, J = 8.3 Hz, 2H), 7.11 (d, J = 7.5 Hz, 1H), 6.92 (t, J = 7.5 Hz, 1H), 6.82 (d, J = 7.9 Hz, 1H), 6.77 (d, J = 8.3 Hz, 2H), 5.06 (s, 1H), 4.66 (d, J = 7.2 Hz, 1H), 3.48 – 3.42 (m, 1H), 3.17 (d, J = 13.5 Hz, 1H), 3.05 (dd, J = 10.0, 7.4 Hz, 1H), 3.00 (d, J = 13.4 Hz, 1H), 2.78 (t, J = 7.8 Hz, 2H), 1.84 (s, 2H), 1.74 (s, 2H), 1.53-1.41 (m, 4H), 1.37-1.29 (m, 2H), 0.89 (t, J = 7.3 Hz, 1H); HRMS (ESI) calcd. for $C_{28}H_{30}N_3O_4$ 472.2236; found 472.2243.

9a-isobutyl-2-methoxy-8-propyl-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-c]quinoline-6,7,9(8H)-trione (9y)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (s, 1H), 6.93 (s, 1H), 6.71 (dd, J = 8.6, 2.4 Hz, 1H), 6.60 (d, J = 8.7 Hz, 1H), 4.67 (d, J = 7.0 Hz, 1H), 3.76 (s, 3H), 3.49 – 3.33 (m, 2H), 2.95 – 2.75 (m, 2H), 1.95 (dd, J = 13.7, 6.0 Hz, 1H), 1.78 – 1.61 (m, 3H), 0.96 (d, J = 6.4 Hz, 3H), 0.92 (d, J = 6.4 Hz, 3H), 0.69 (t, J = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 179.7, 174.4, 167.6, 156.3, 128.7, 123.6, 116.9, 115.8, 111.1, 69.5, 57.6, 55.7, 50.8, 48.6, 43.9,

40.2, 24.9, 24.2, 23.8, 20.0, 11.1; HRMS (ESI) calcd. for $C_{21}H_{28}N_3O_4$ 386.2080; found 386.2083.

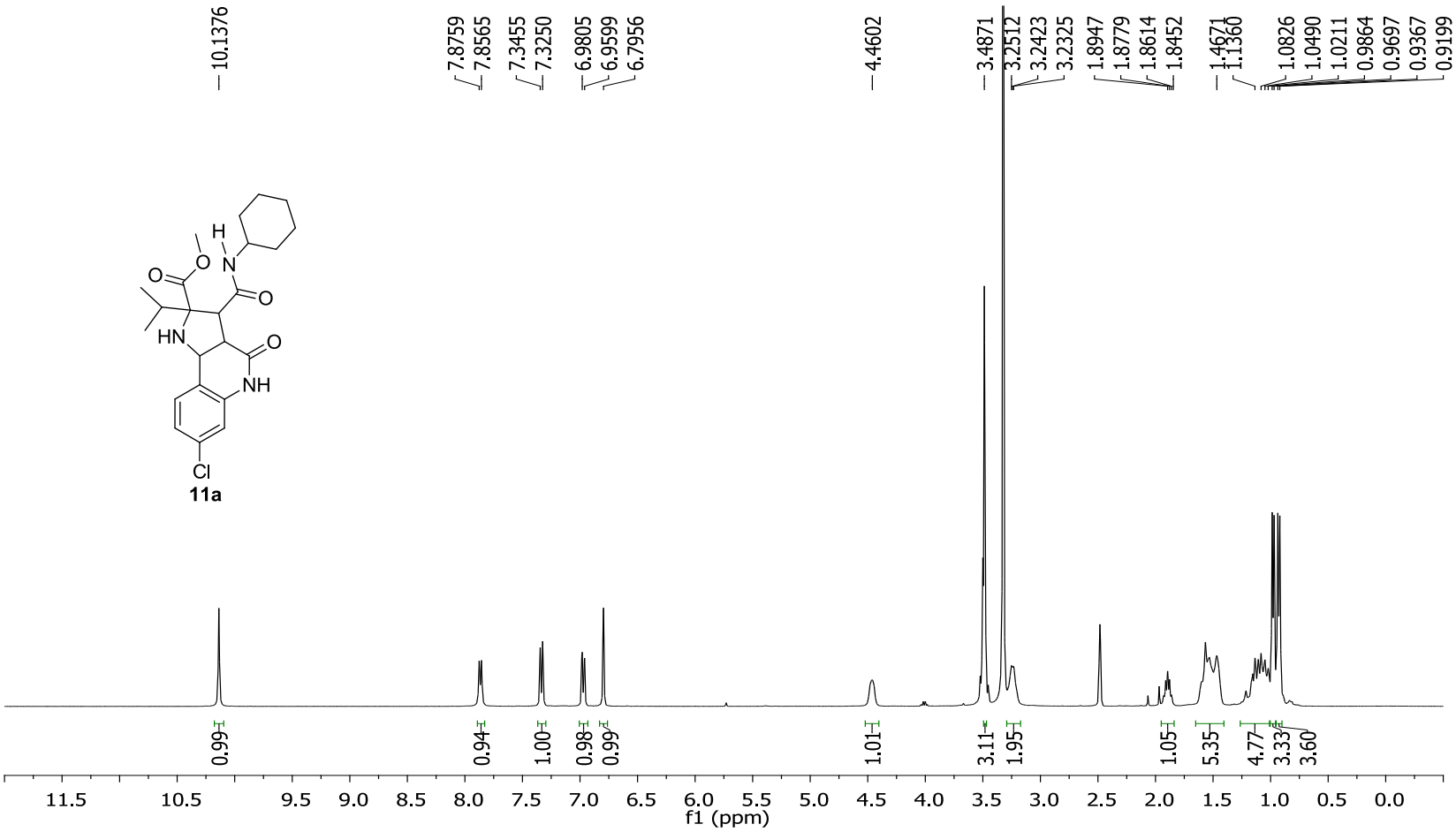
2-methoxy-8-methyl-9a-phenyl-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-c]quinoline-6,7,9(8H)-trione (9z)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38 (s, 1H), 7.48 (d, J = 7.2 Hz, 2H), 7.41-7.32 (m, 3H), 6.95 (s, 1H), 6.72 (s, 2H), 4.86 (d, J = 7.0 Hz, 1H), 3.79 (s, 3H), 3.68 (d, J = 10.1 Hz, 1H), 3.54 (dd, J = 10.1, 7.1 Hz, 1H), 2.43 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.7, 174.1, 167.2, 156.2, 138.5, 129.0, 128.6, 125.5, 123.1, 117.1, 115.9, 110.9, 71.8, 58.3, 55.8, 54.7, 48.3, 24.7; HRMS (ESI) calcd. for $C_{21}H_{20}N_3O_4$ 378.1454; found 378.1454.

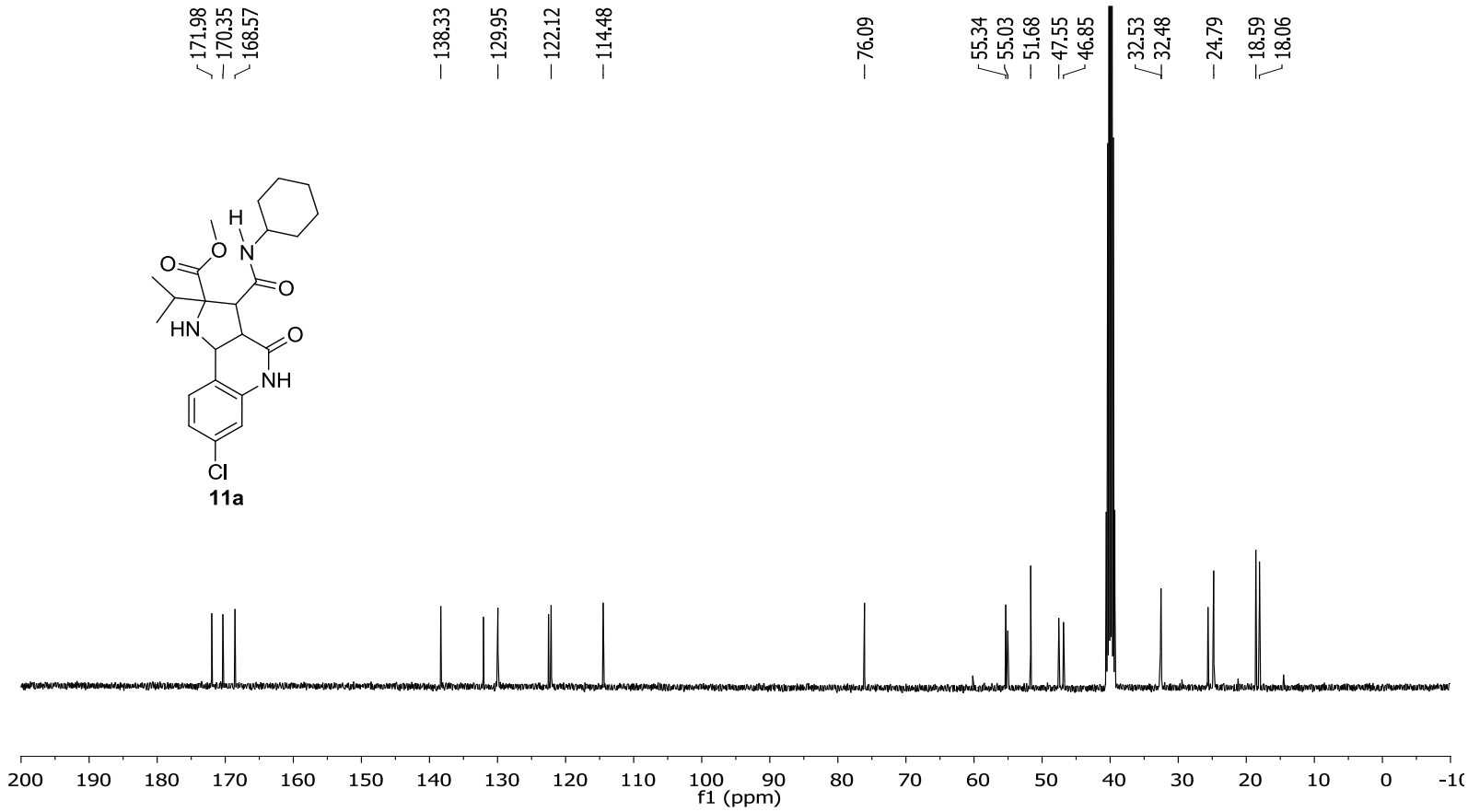
9a-isopropyl-2-methoxy-8-propyl-5,6a,6b,9a,10,10a-hexahydropyrrolo[3',4':4,5]pyrrolo[3,2-c]quinoline-6,7,9(8H)-trione (9aa)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 (s, 1H), 6.86 (s, 1H), 6.68 (q, J = 8.7 Hz, 2H), 4.69 (d, J = 6.2 Hz, 1H), 3.76 (s, 3H), 3.29 (q, J = 10.1 Hz, 2H), 2.92 – 2.72 (m, 2H), 2.24 (dt, J = 13.6, 6.9 Hz, 1H), 1.05 (d, J = 6.8 Hz, 3H), 1.0-0.93 (m, 2H), 0.90 (d, J = 6.7 Hz, 3H), 0.68 (t, J = 7.4 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 180.0, 174.4, 167.4, 156.3, 128.6, 123.8, 116.8, 115.5,

111.0, 72.8, 57.8, 55.7, 48.3, 47.6, 40.1, 31.7, 20.1, 17.6, 16.4, 11.1; HRMS (ESI) calcd. for $C_{20}H_{26}N_3O_4$ 372.1923; found 372.1917.



^{1}H NMR spectrum (400 MHz) of compound **11a** in DMSO-d_6

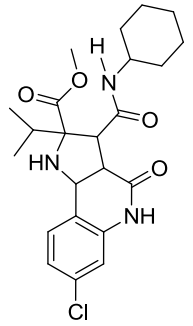


^{13}C NMR spectrum (101 MHz) of compound 11**a** in DMSO-d_6

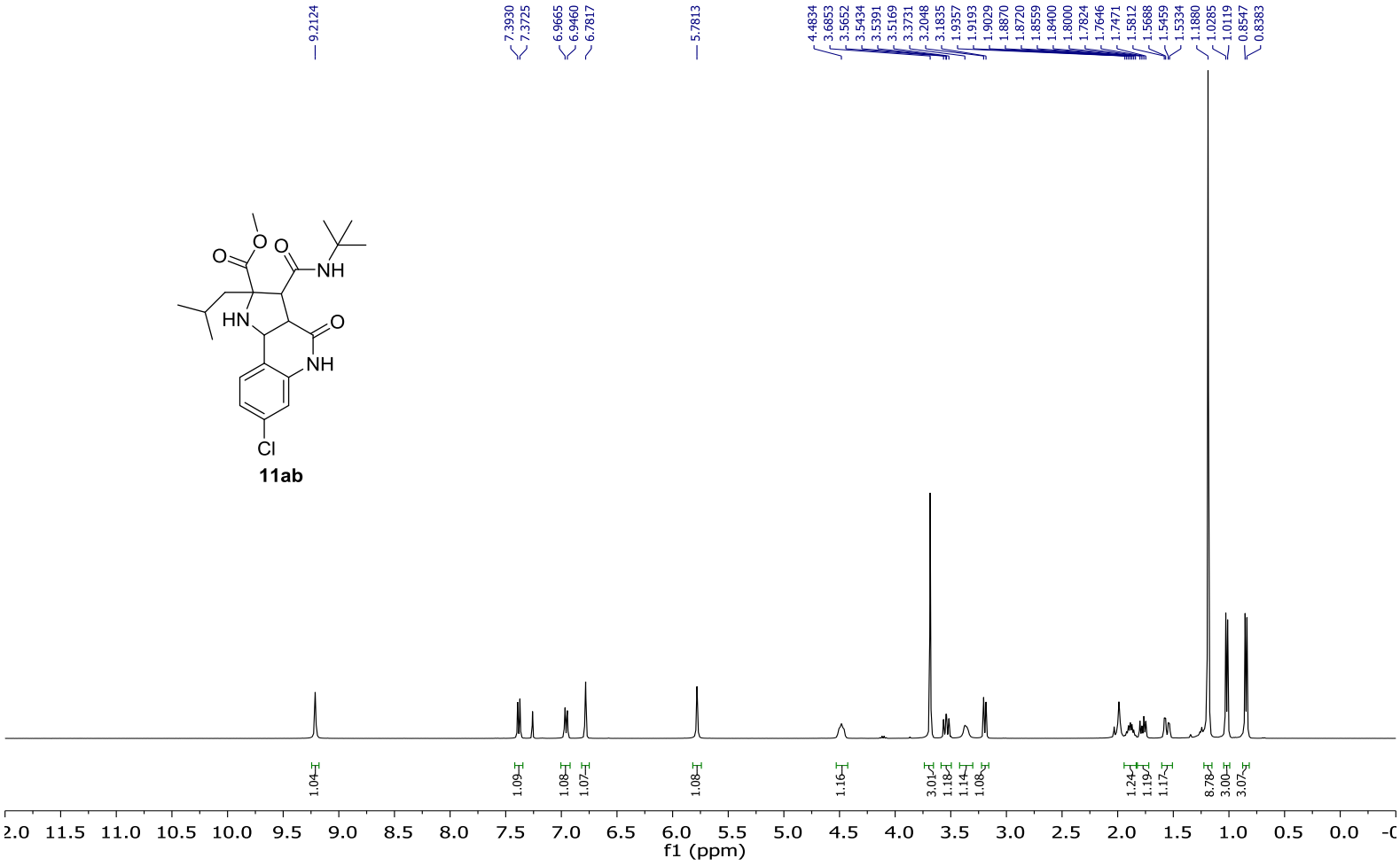
Display Report

Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdb	e¯ Conf	N-Rule	Adduct
448.1998	1	C23H31ClN3O4	448.1998	-0.1	29.4	1	100.00	9.5	even	ok	M+H

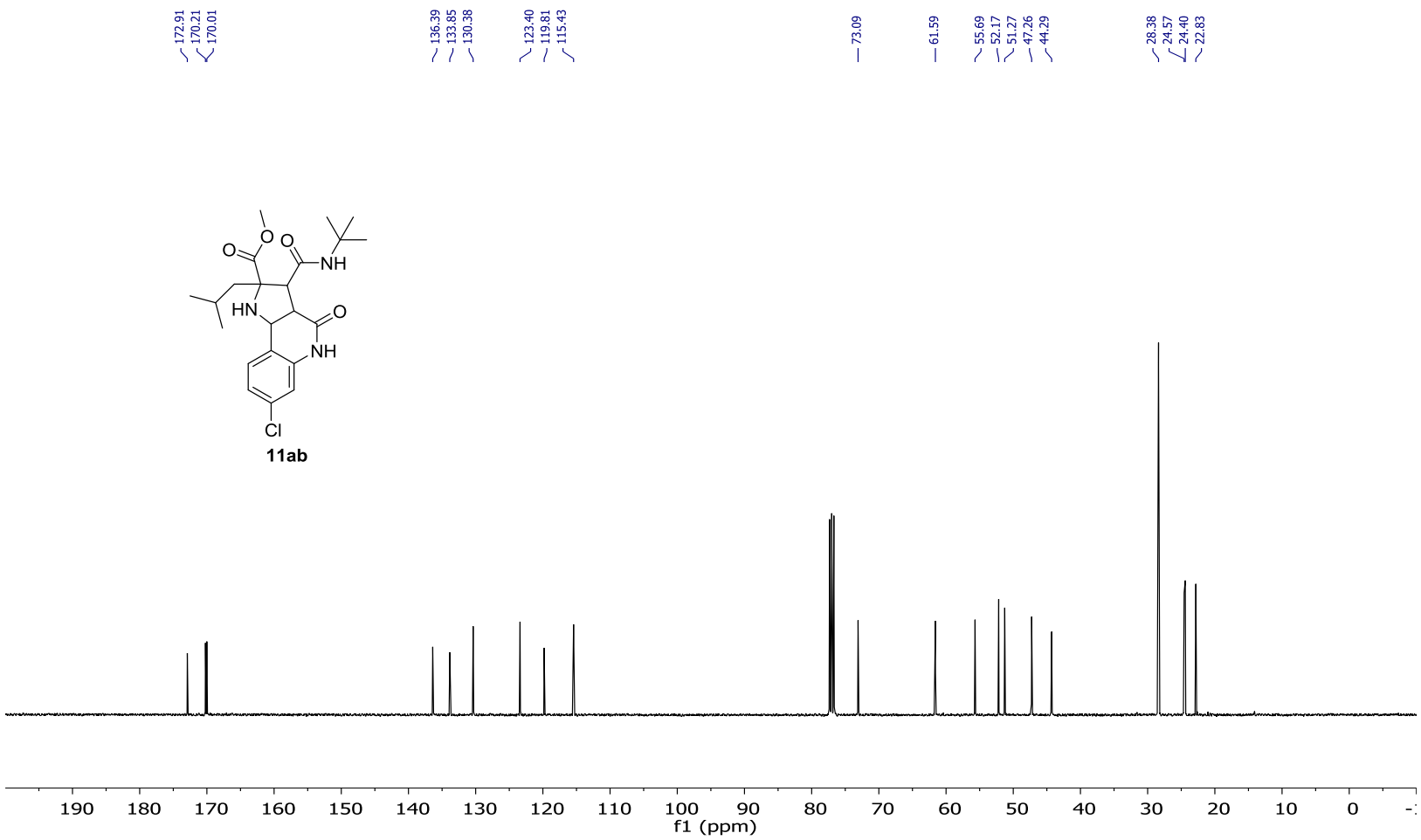
HRMS of compound **11a**



11a



^{1}H NMR (400 MHz) of compound **11ab** in $CDCl_3$



^{13}C NMR (101 MHz) of compound **11ab** in $CDCl_3$

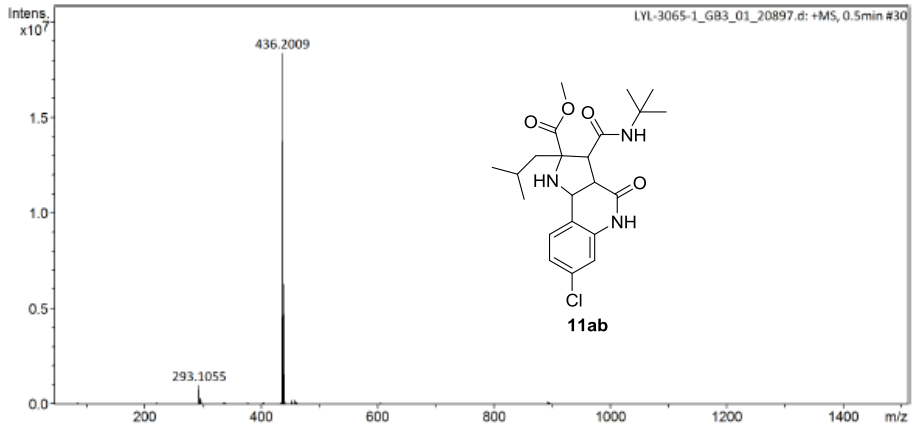
Display Report

Analysis Info

Analysis Name	D:\Data\nctu service\data\2020\20200619\LYL-3065-1_GB3_01_20897.d	Acquisition Date	6/19/2020 12:01:43 PM
Method	Small molecule.m	Operator	NCTU
Sample Name	LYL-3065-1	Instrument	impact HD 1819696.00164
Comment			

Acquisition Parameter

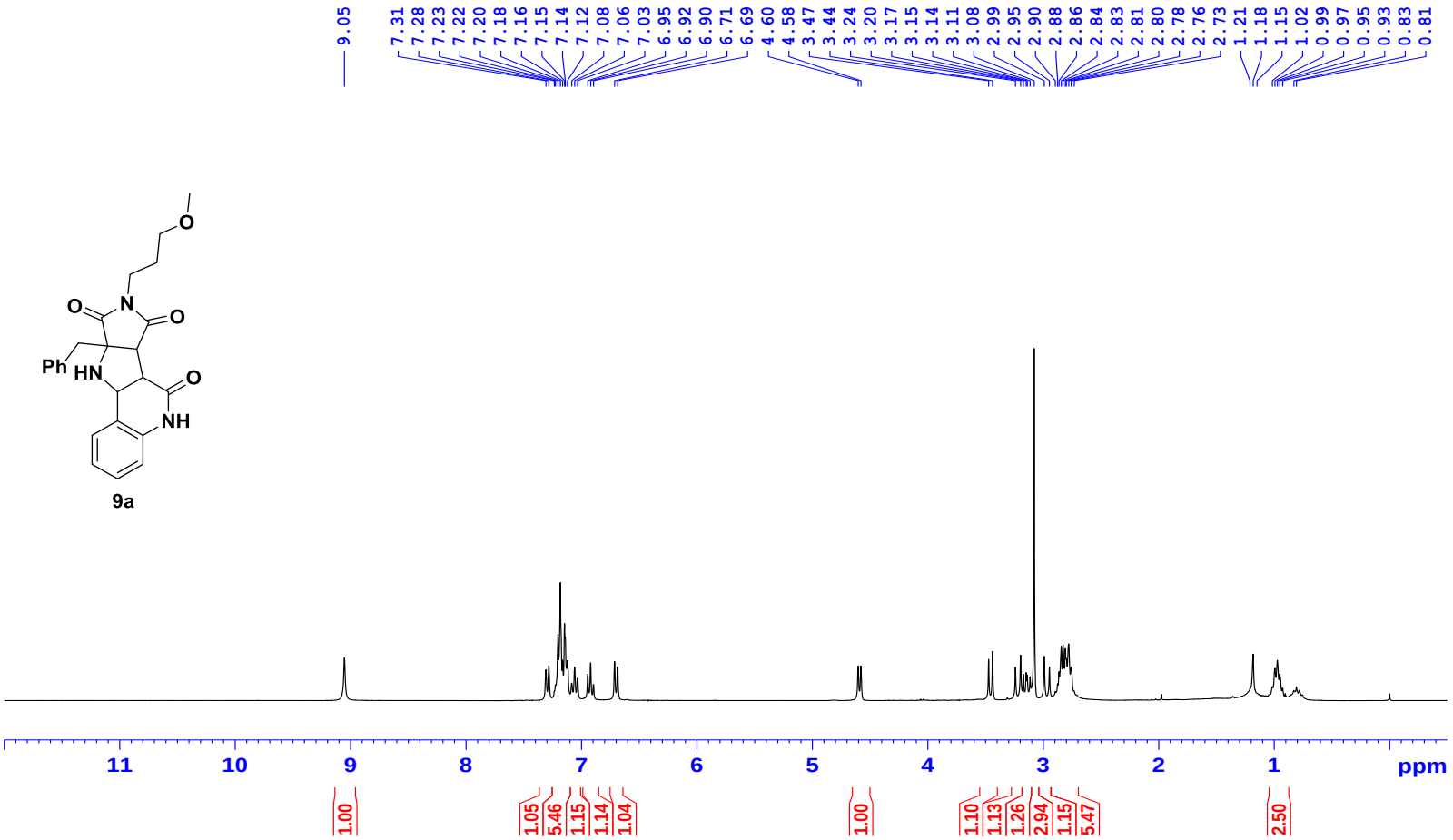
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



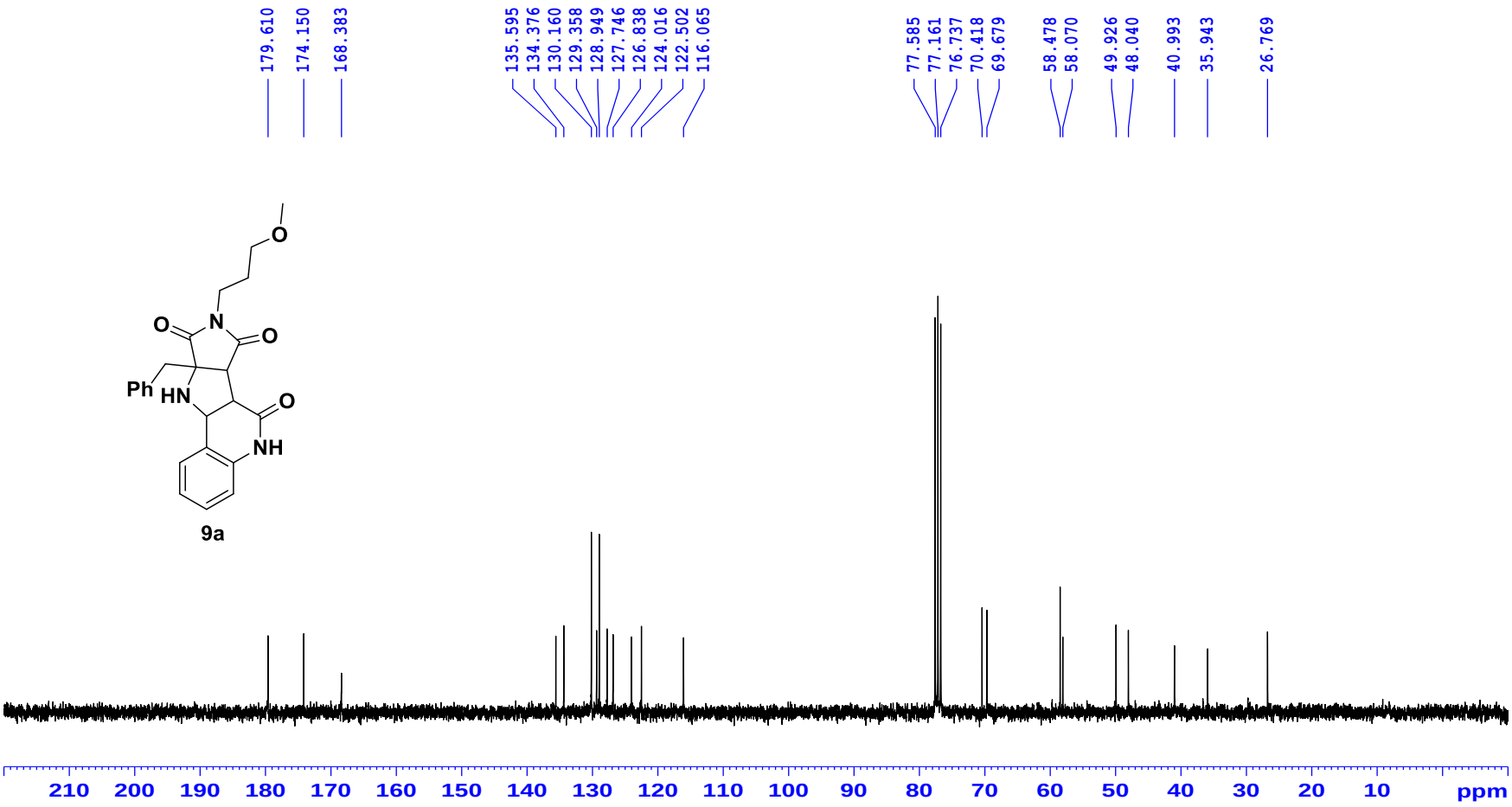
Display Report

Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdb	e⁻ Conf	N-Rule	Adduct
436.2009	1	C22H31ClN3O4	436.1998	-2.5	10.8	1	100.00	8.5	even	ok	M+H

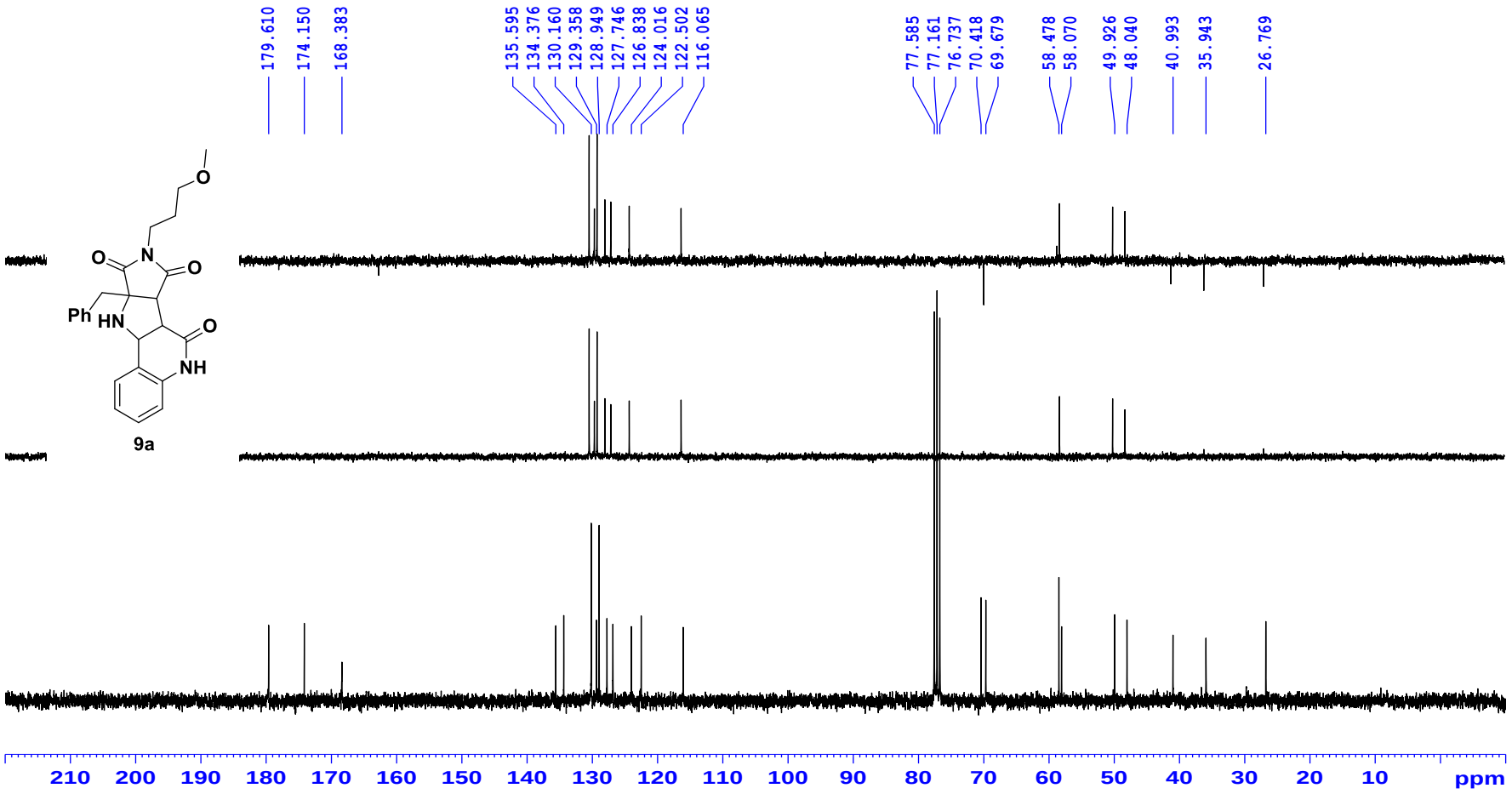
HRMS (ESI) of compound **11ab**



^{1}H NMR spectrum (400 MHz) of compound **9a** in $CDCl_3$



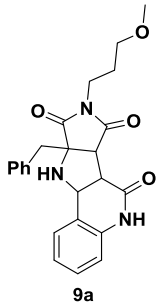
^{13}C NMR spectrum (101 MHz) of compound **9a** in $CDCl_3$



DEPT spectrum (101 MHz) of compound **9a** in $CDCl_3$

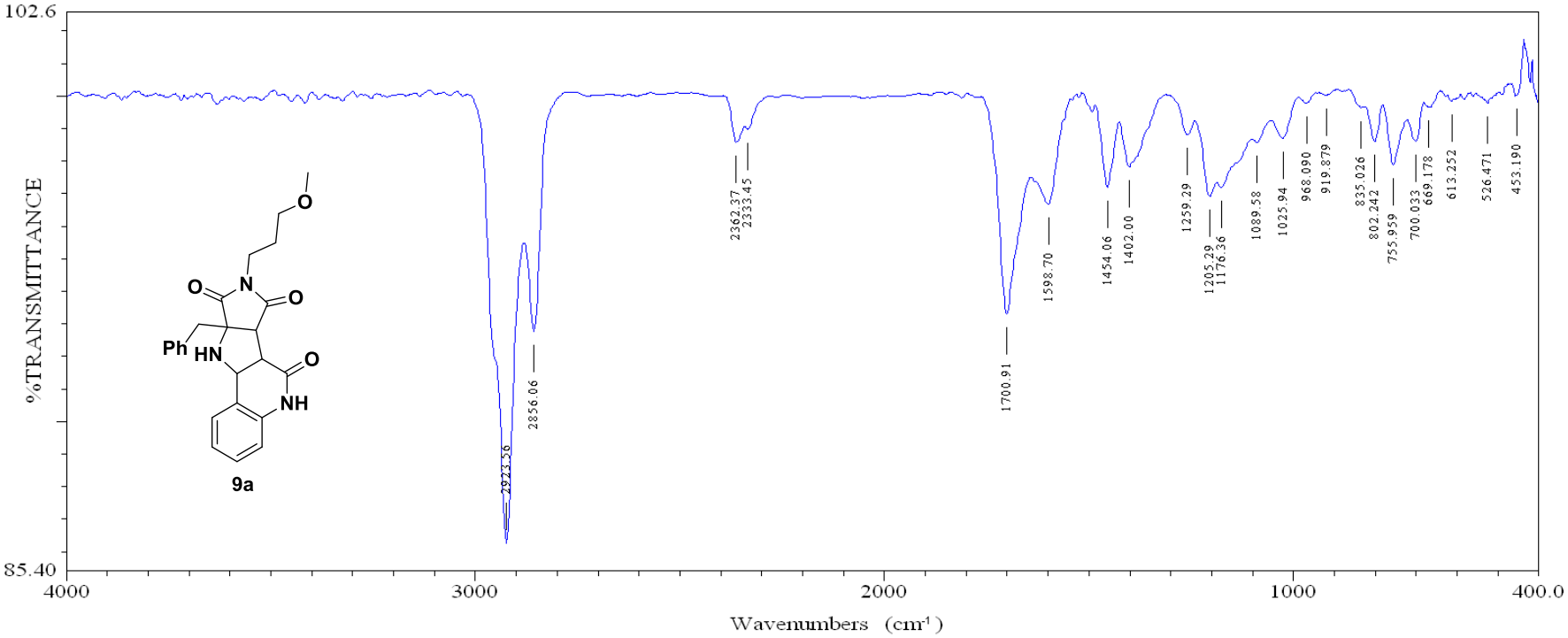
```
LIST: hei2532(leeut-el03)-c4                    29-Sep-11 REG    : 08:24.1      #9
                                                          Start  : 16:39:04   3358
Samp:
Mode: EI +VE +LMR  ESCAN (EXP) UP μR NRM                  Inlet  :
Oper:
Limt: (  0)     .     .     .
    : (466) C25.H44.N3.O5
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/1616>1782  (CMASS : converted; CMASS : converted; CMASS : con

                  16                          (mmu)
     Mass    Intensity      %RA    Flags     Delta   R+D  Composition
  419.1854        76835    100.00    #         -0.9  14.C  C24.H25.N3.O4
```

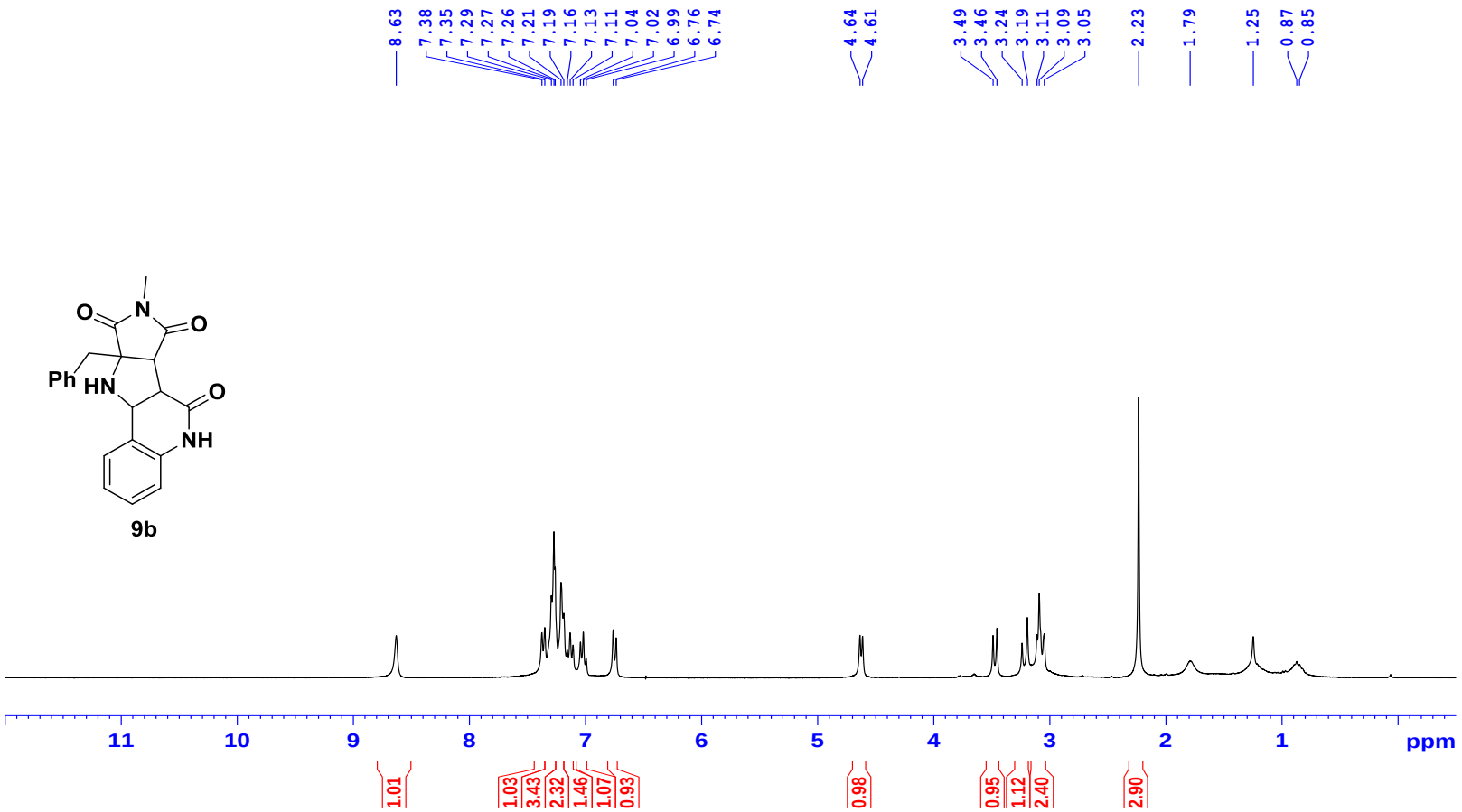


9a

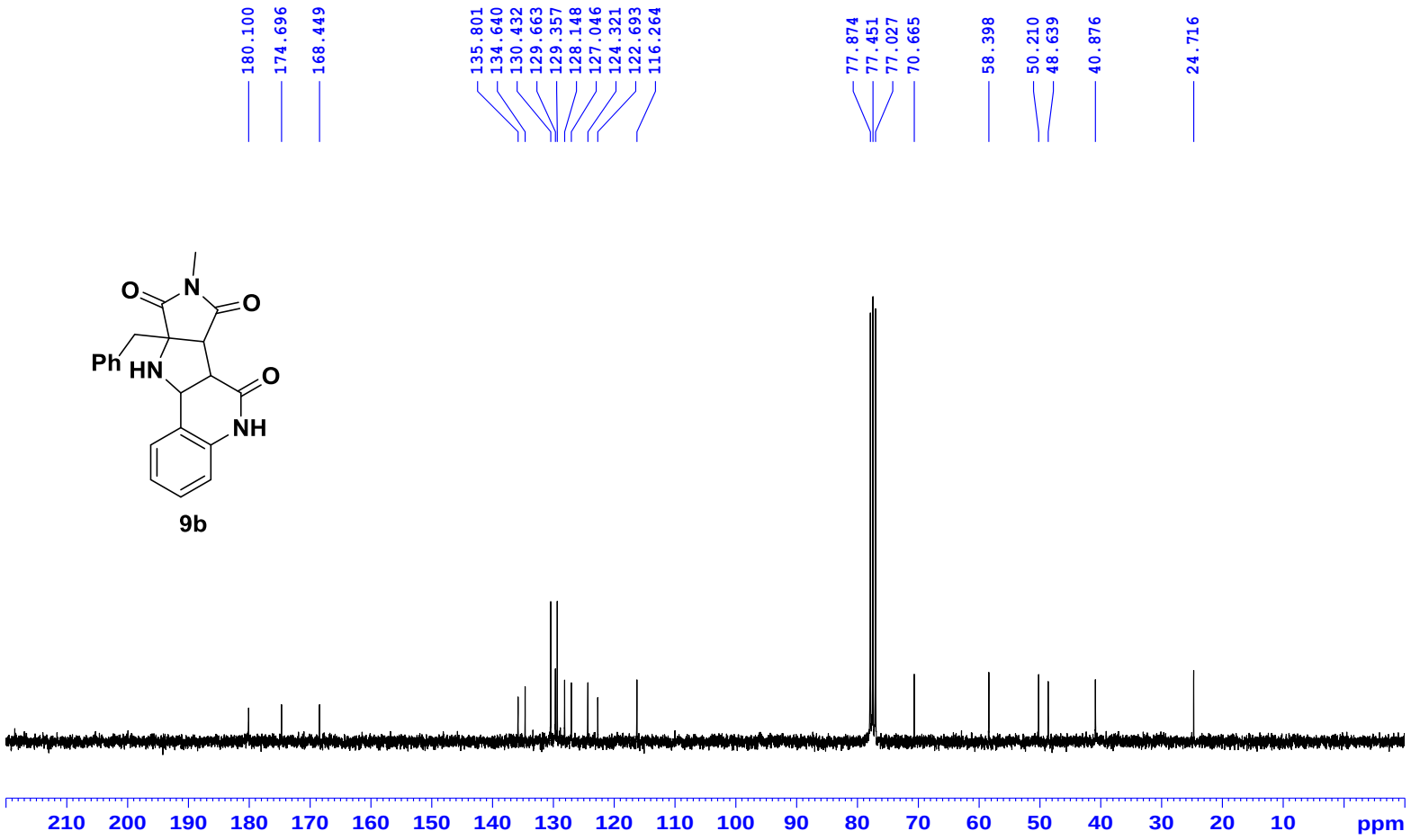
HRMS of compound **9a**



IR of compound **9a**



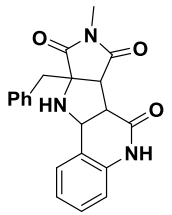
^{1}H NMR spectrum (400 MHz) of compound **9b** in $CDCl_3$



^{13}C NMR spectrum (101 MHz) of compound **9b** in $CDCl_3$

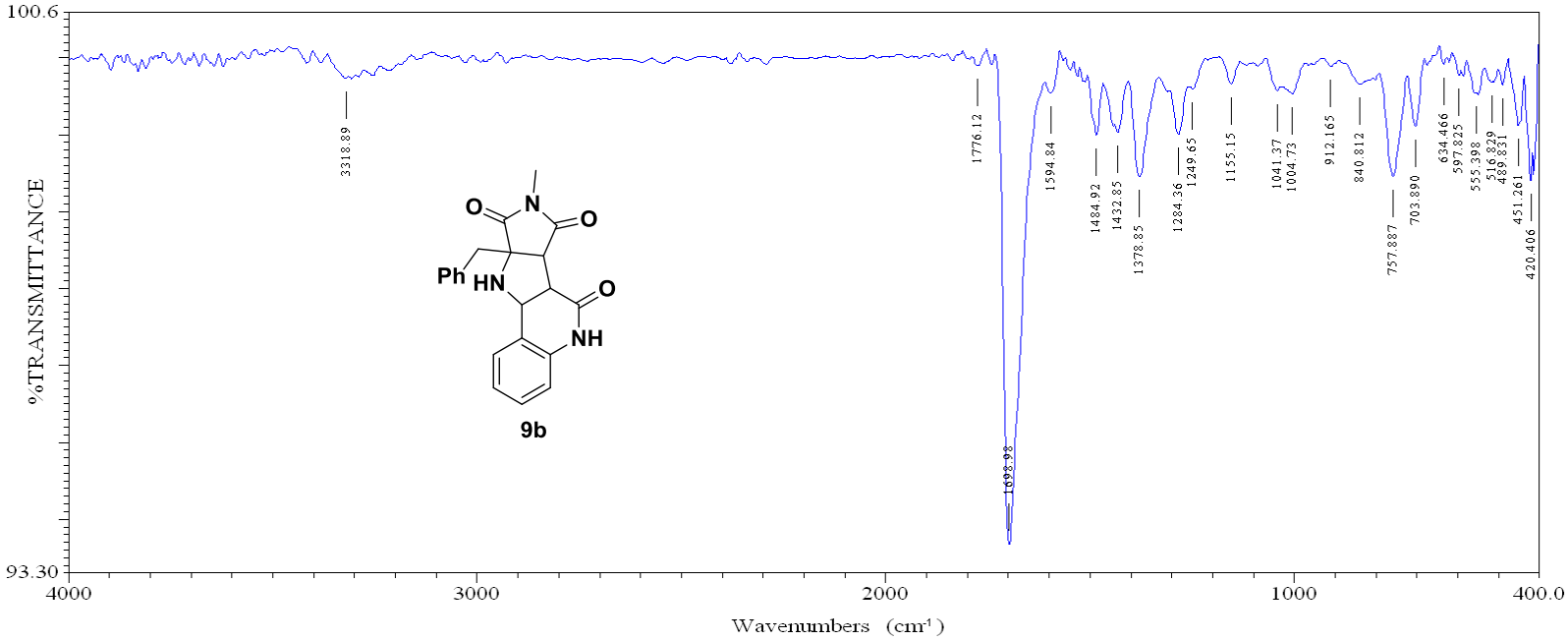
```
LIST: hei2111(leeutel02)-c3              07-Jun-11 REG    : 04:14.3     #9
Samp:                                              Start  : 16:16:35  2209
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                              Inlet  :
Limt: (  0)    .    .    .
    : (388) C21.H30.N3.O4
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/792>901  (CMASS : converted |CMASS : converted |CMASS : conve
```

Mass	Intensity	%RA	Flags	(mmu) Delta	R+D	Composition
361.1434	49466	100.00	#	-0.8	14.0	C21.H19.N3.O3

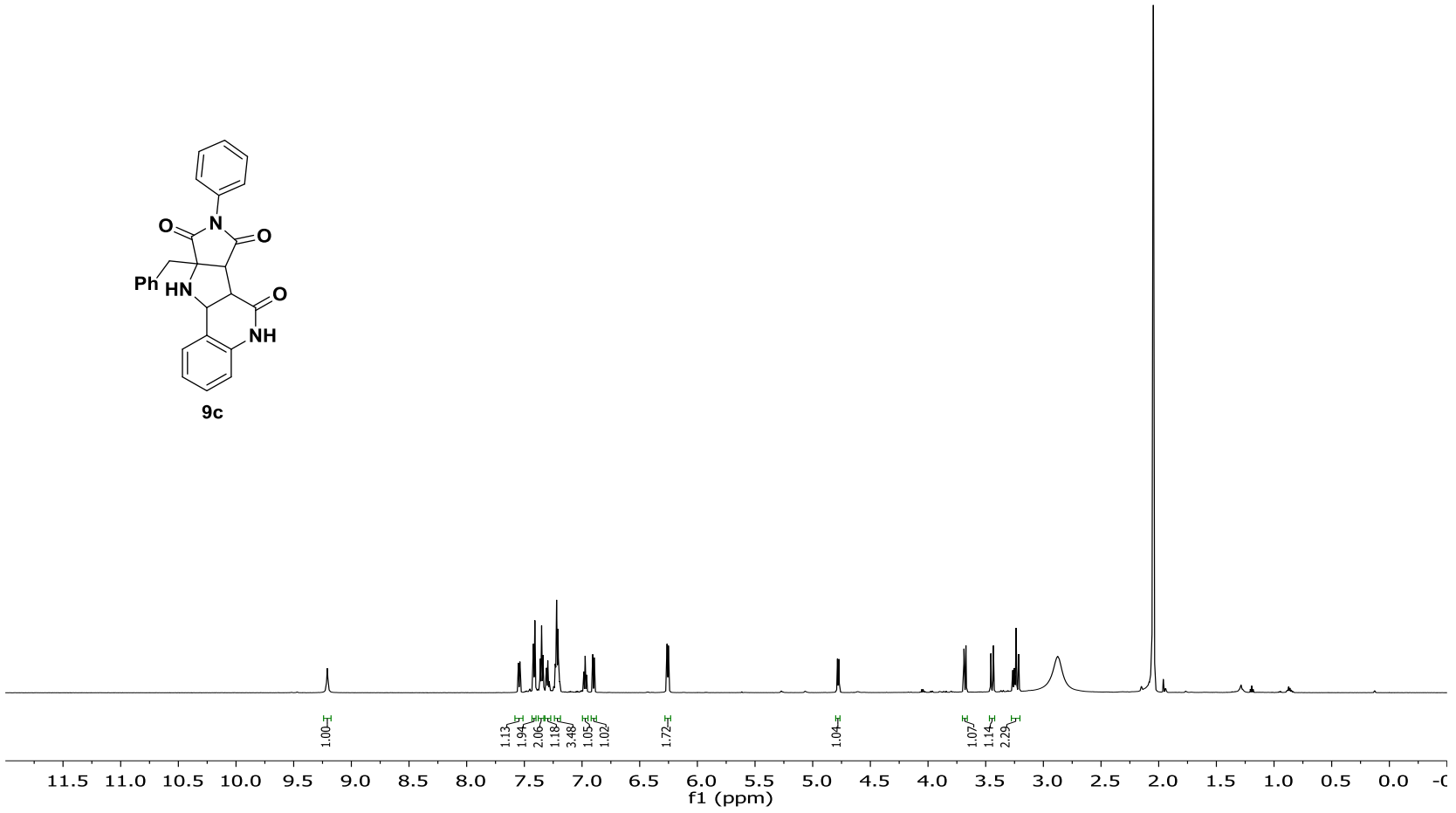


9b

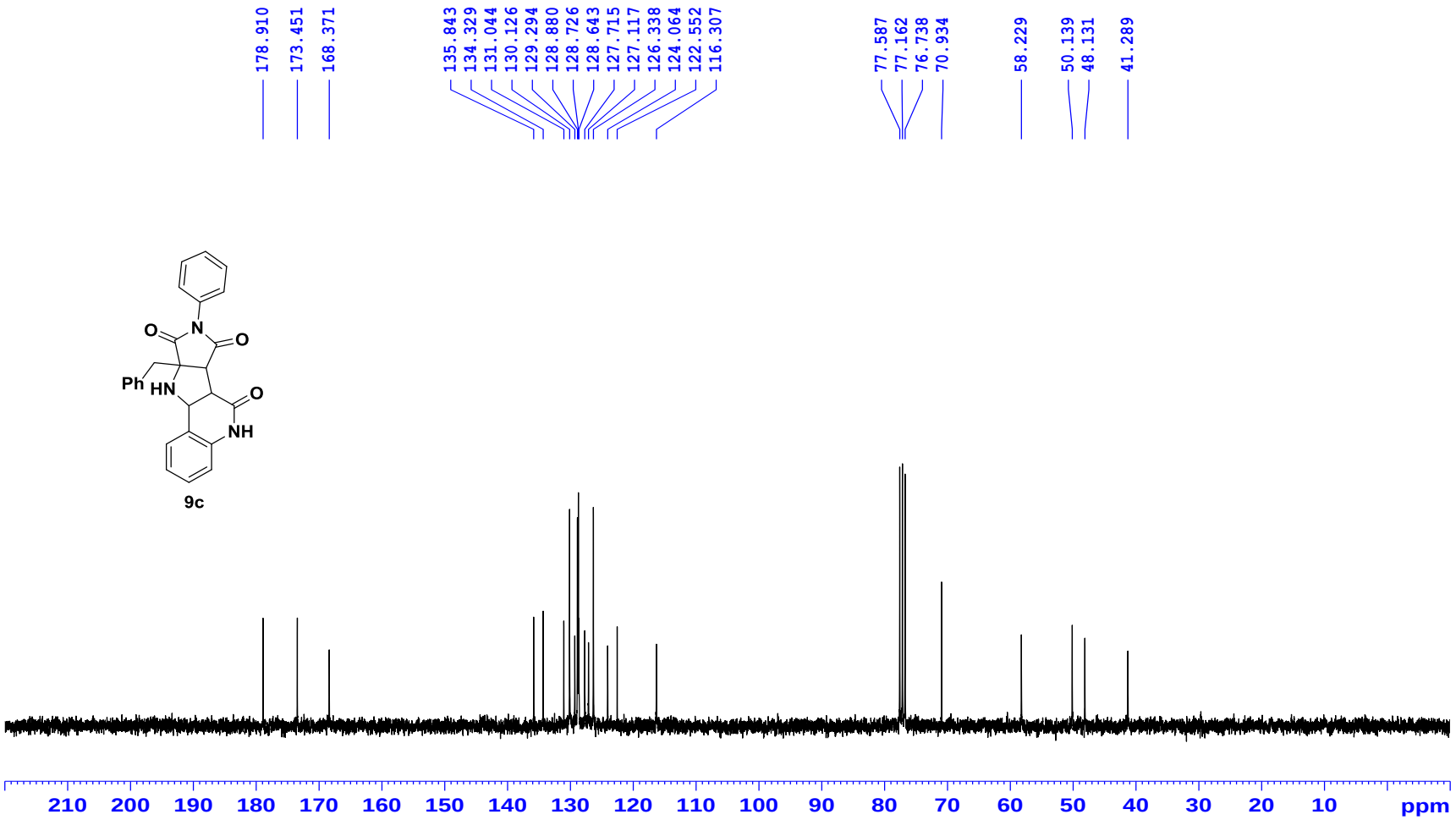
HRMS (EI) of compound **9b**



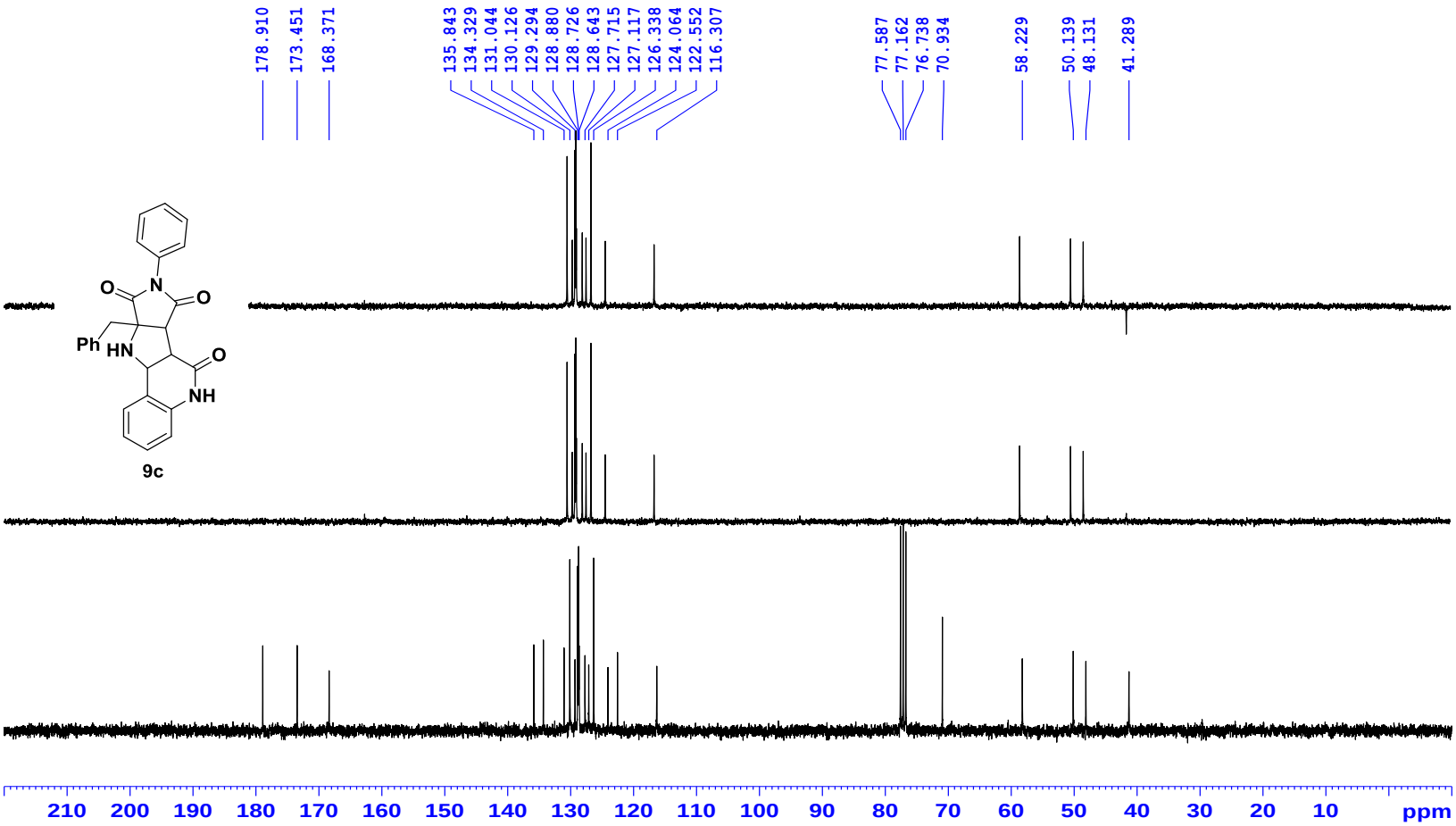
IR of compound **9b**



^{1}H NMR spectrum (400 MHz) of compound **9c** in $CDCl_3$



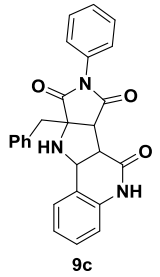
^{13}C NMR spectrum (101 MHz) of compound **9c** in $CDCl_3$



DEPT of compound **9c** in $CDCl_3$

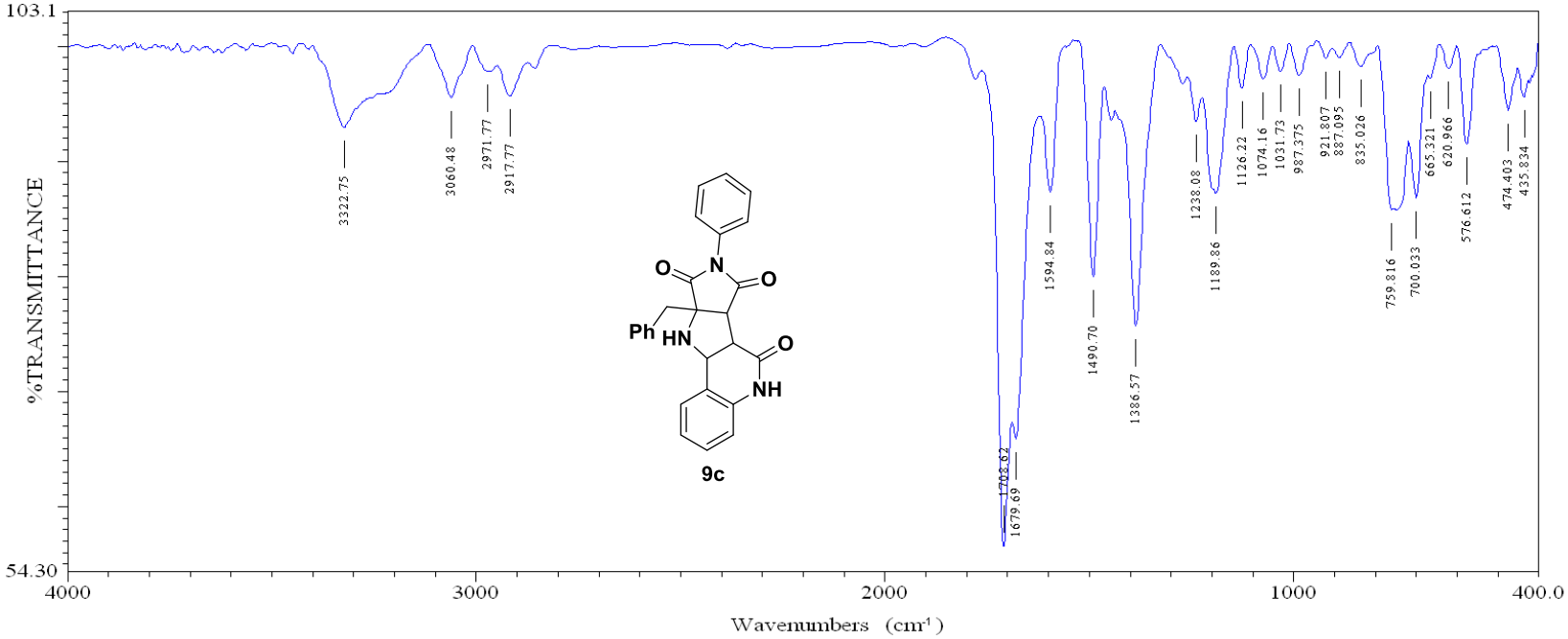
```
LIST: hei2112(leeutel01)-c3          07-Jun-11 REG    : 07:07.4     #9
Samp:                                          Start  : 16:30:22  8816
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                          Inlet  :
Limt: (  0)     .     .     .
    : (468) C29.H30.N3.O3
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/1369>1752  (CMASS : converted |CMASS : converted |CMASS : con

                  25                        (mmu)
    Mass  Intensity      %RA   Flags Delta  R+D  Composition
423.1591        4528   35.68   #      -0.8 18.0  C26.H21.N3.O3
```

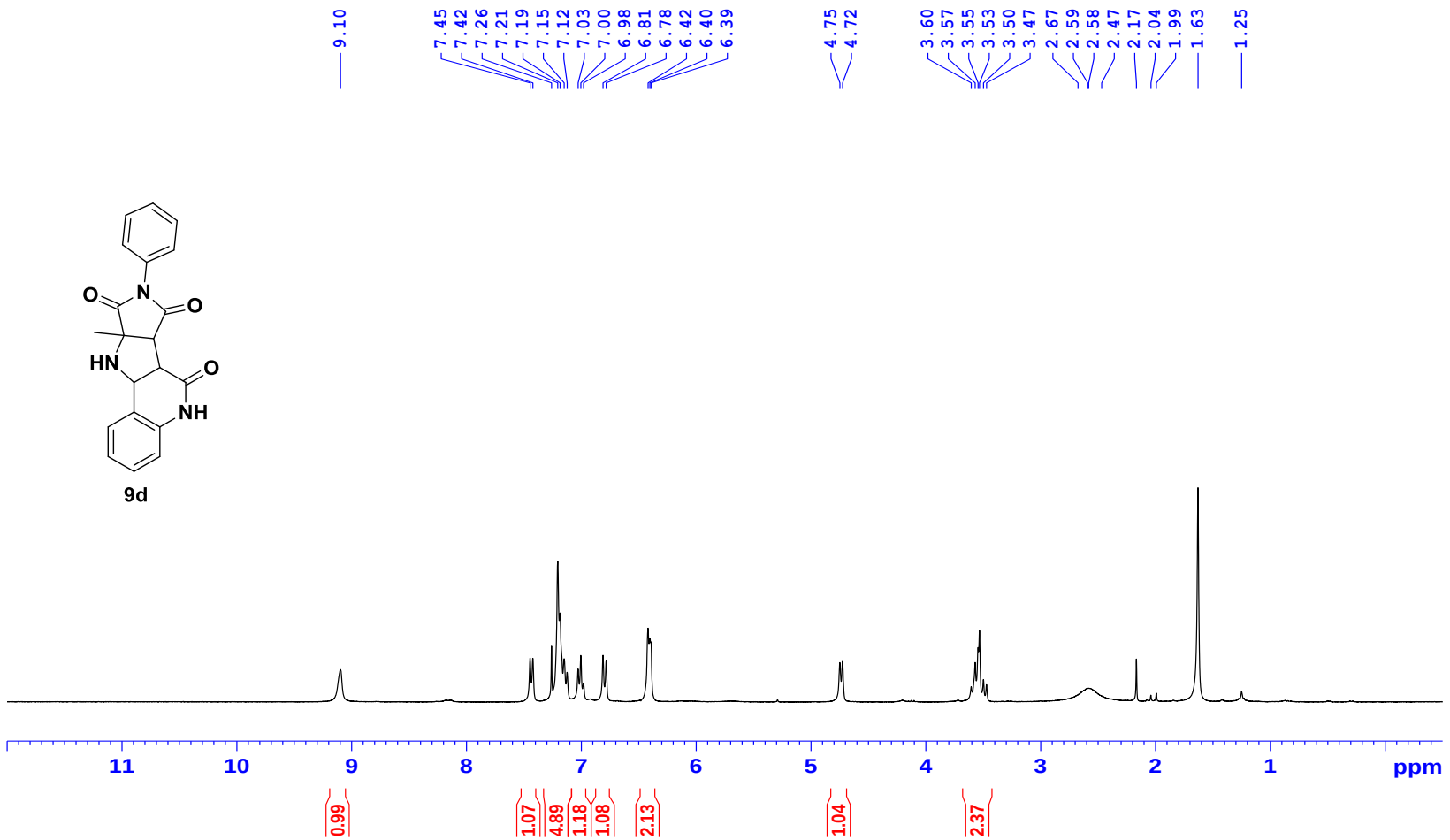


9c

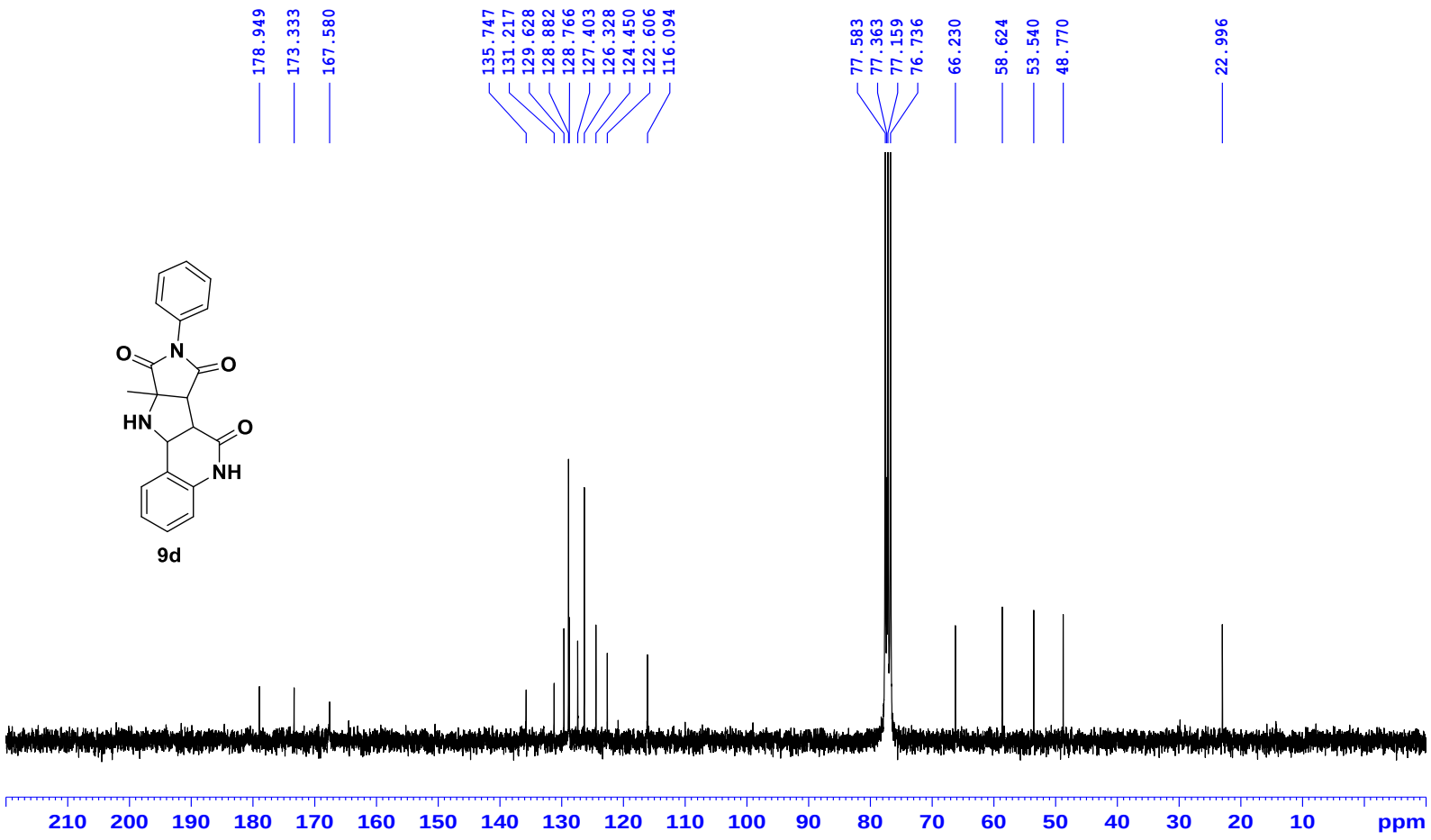
HRMS (EI) of compound **9c**



IR of compound **9c**



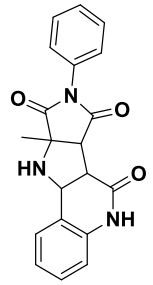
^{1}H NMR spectrum (400 MHz) of compound **9d** in $CDCl_3$



^{13}C NMR spectrum (101 MHz) of compound **9d** in $CDCl_3$

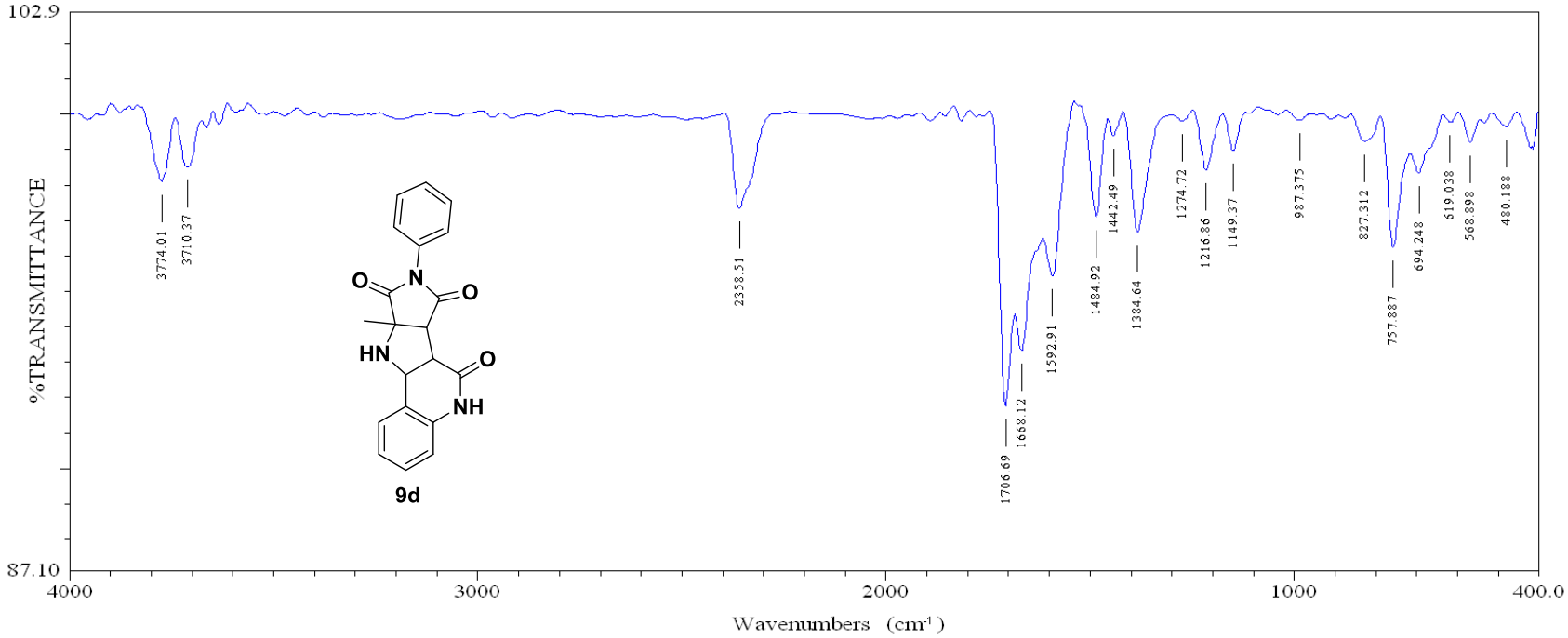
```
LIST: hei2110(leeutel04)-c2              07-Jun-11 REG    : 02:38.3      #9
Samp:                                              Start  : 15:40:28   6177
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                              Inlet  :
Limt: (  0)     .     .     .
    : (388) C21.H30.N3.O4
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/488>636  (CMASS : converted |CMASS : converted |CMASS : conve

                69                          (mmu)
    Mass  Intensity      %RA   Flags Delta  R+D  Composition
347.1265      34708   100.00    #       0.5 14.0  C20.H17.N3.O3
```

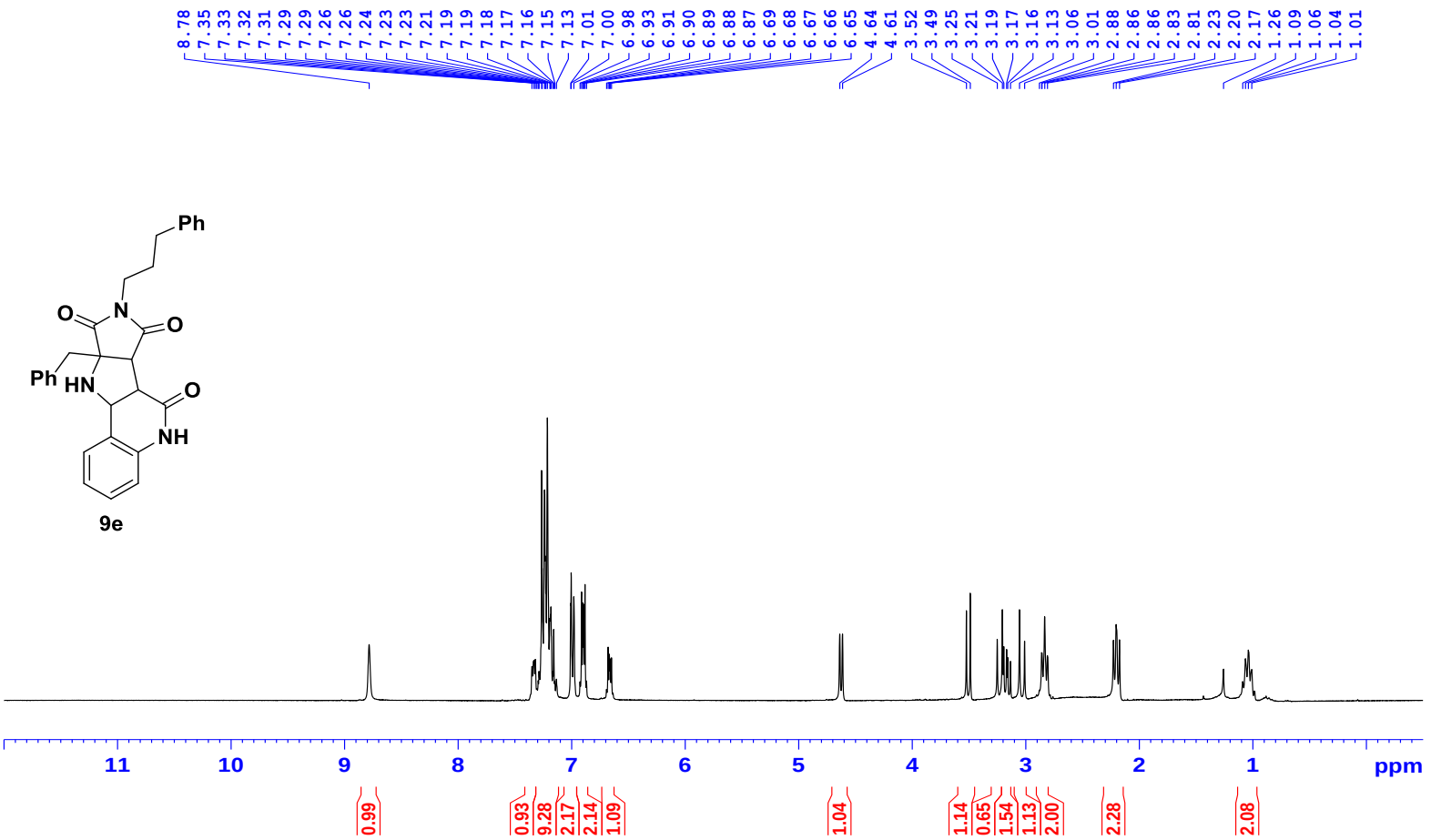


9d

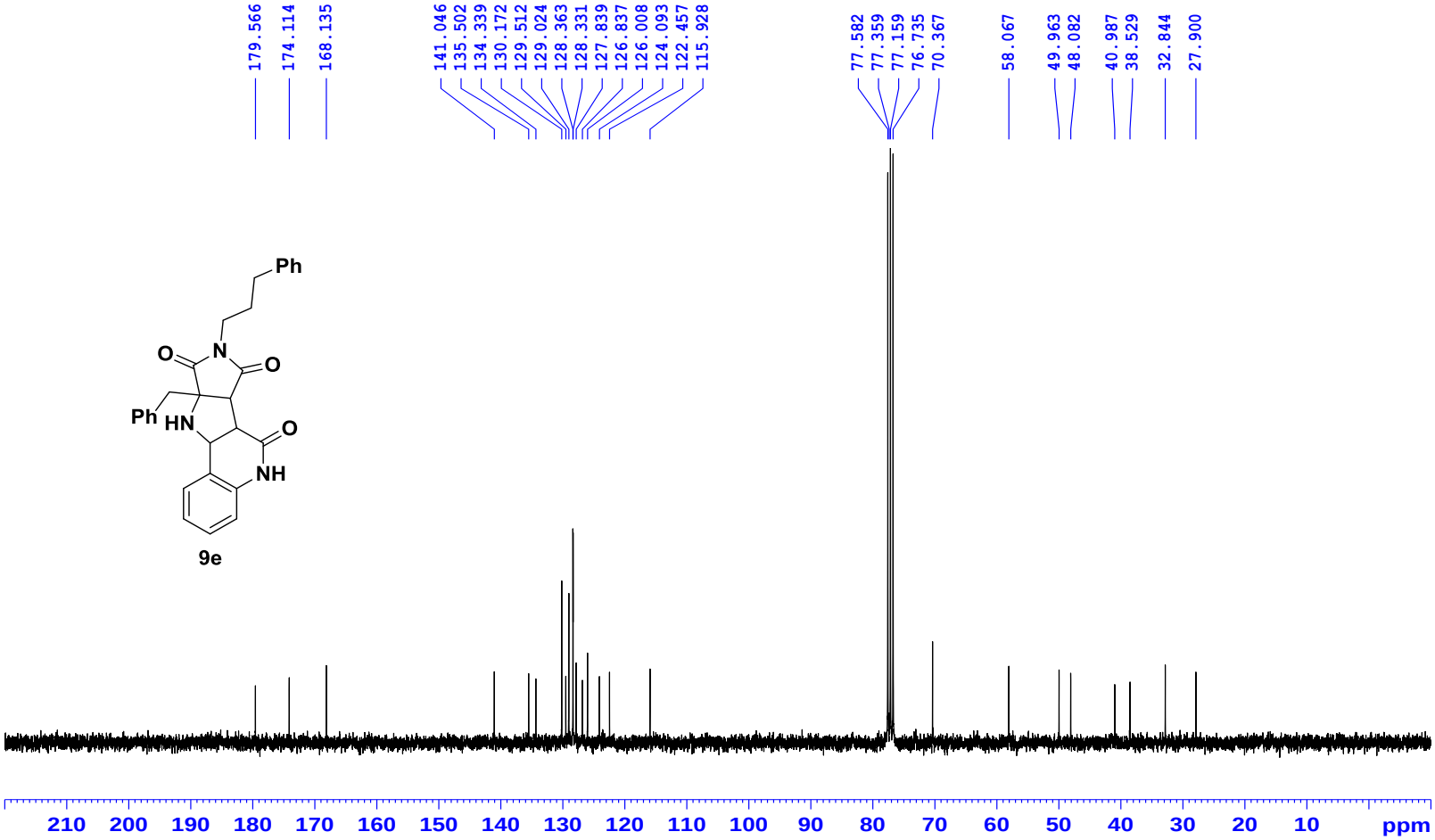
HRMS (EI) of compound **9d**



IR of compound **9d**



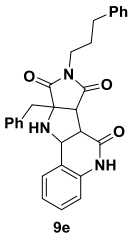
^{1}H NMR spectrum (400 MHz) of compound **9e** in $CDCl_3$



^{13}C NMR spectrum (101 MHz) of compound **9e** in $CDCl_3$

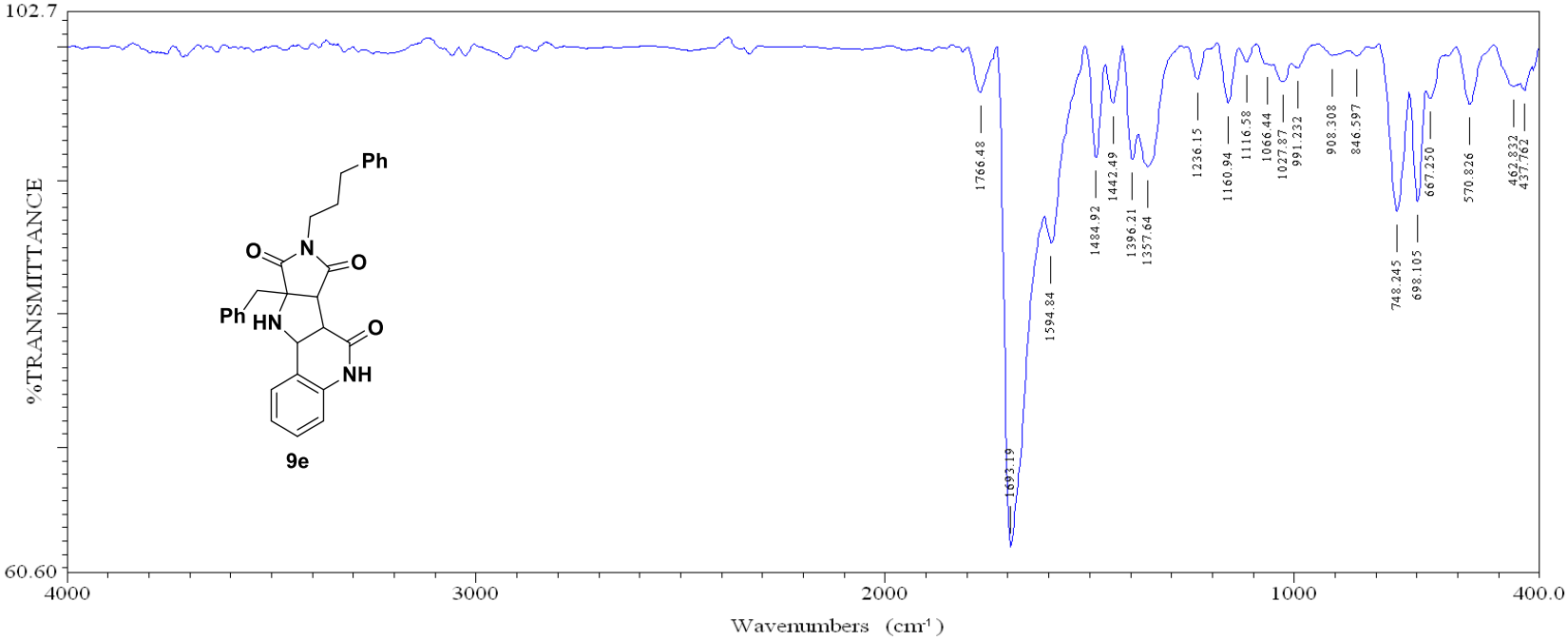
[Elemental Composition]
Data : 201111049 Date : 22-Oct-2011 17:25
Sample: leeut-El05
Note : -
Inlet : Direct Ion Mode : EI+
RT : 1.18 min Scan#: 59
Elements : C 29/0, H 27/0, O 5/0, N 3/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.1 - 36.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
465.2053	99.5	+0.1 / +0.0	18.0	C 29 H 27 O 3 N 3

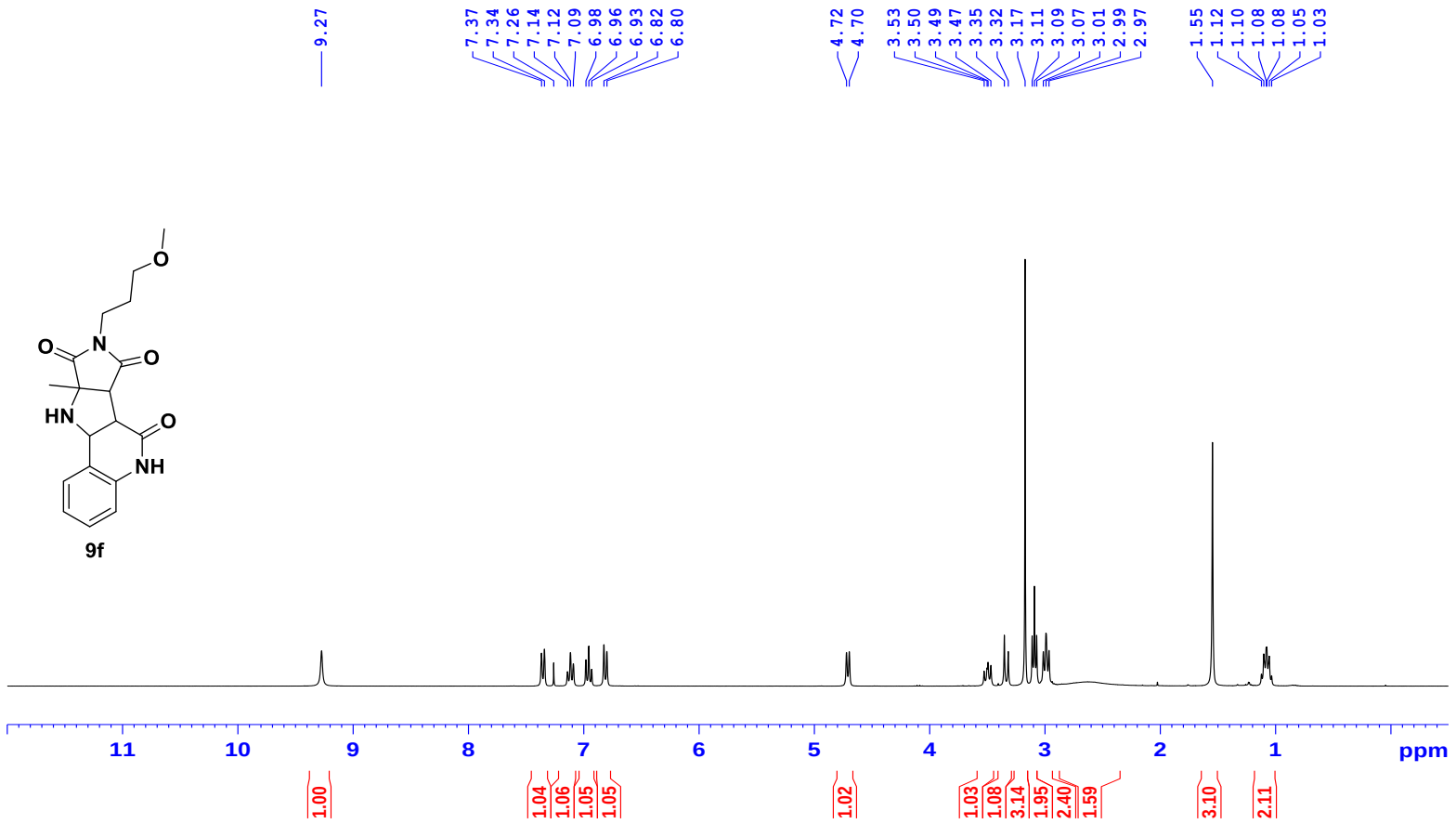


9e

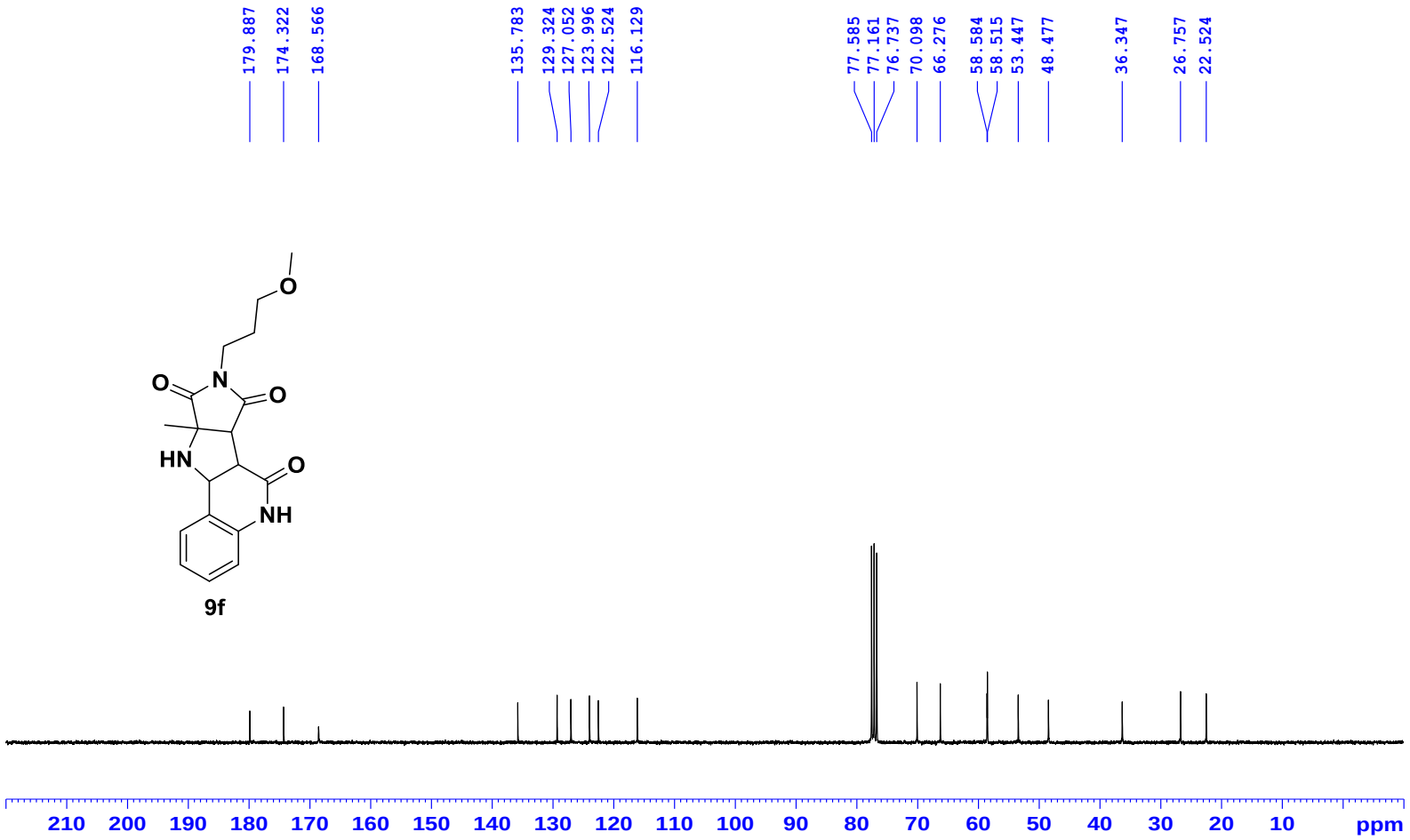
HRMS (EI) of compound **9e**



IR of compound **9e**



^{1}H NMR spectrum (400 MHz) of compound **9f** in $CDCl_3$



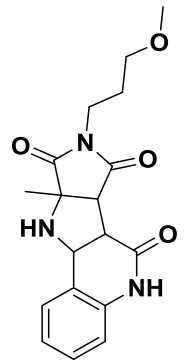
^{13}C NMR spectrum (101 MHz) of compound **9f** in $CDCl_3$

[Elemental Composition]
Data : 1000512-013
Sample: Leeut-EL06
Note : 343.38
Inlet : Direct
RT : 0.95 min
Elements : C 18/0, H 36/0, N 3/0, O 4/0
Mass Tolerance : 10ppm, 3mmu if m/z < 300, 20mmu if m/z > 2000
Unsaturation (U.S.) : -0.5 - 200.0

Date : 12-May-2011 12:07

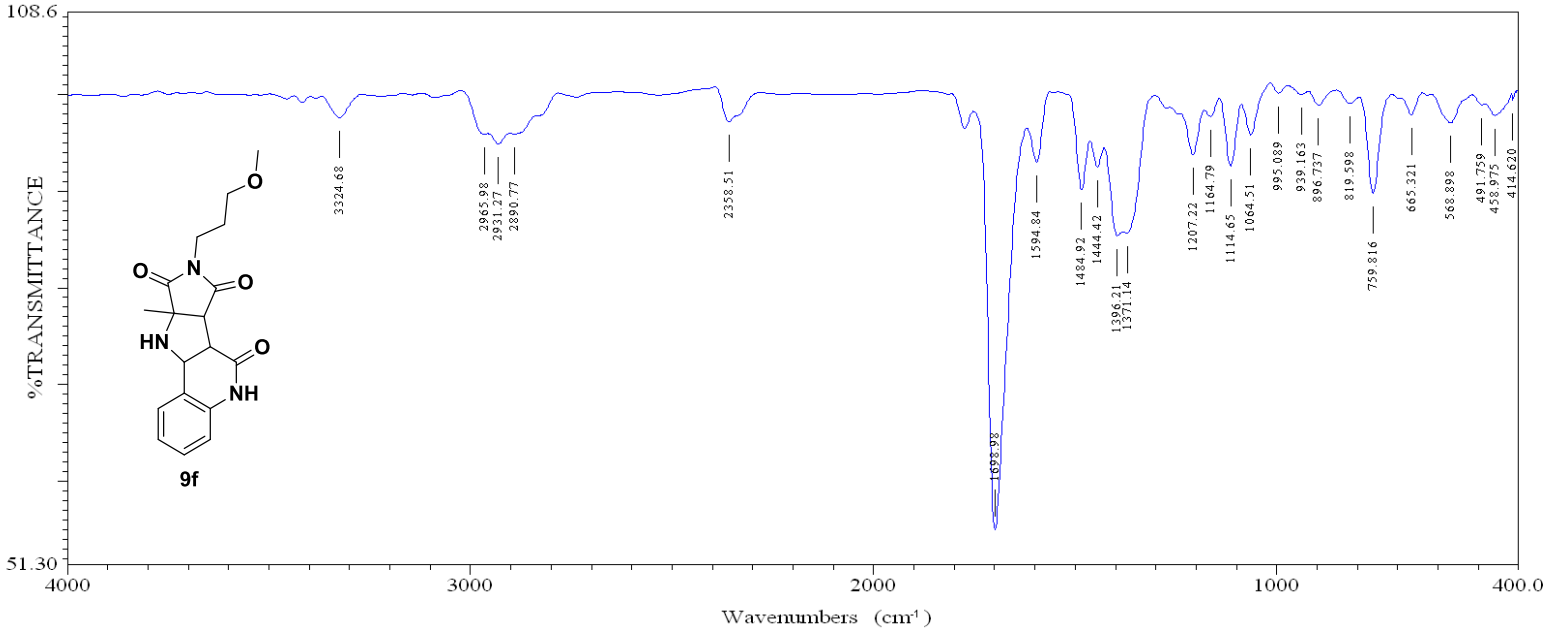
Ion Mode : EI+
Scan#: 58

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
343.1523	100.0	-2.8 / -1.0	10.0	C 18 H 21 N 3 O 4

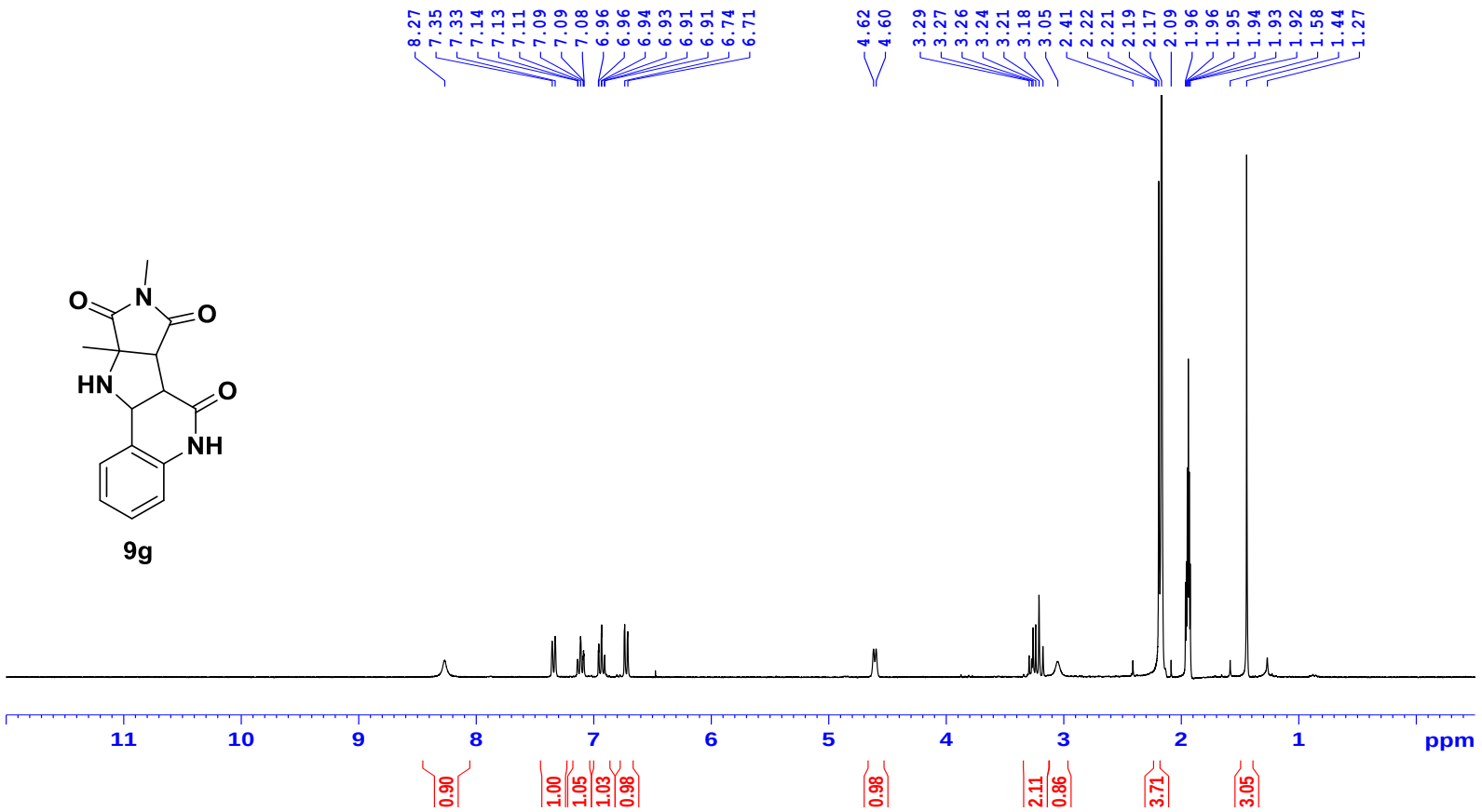


9f

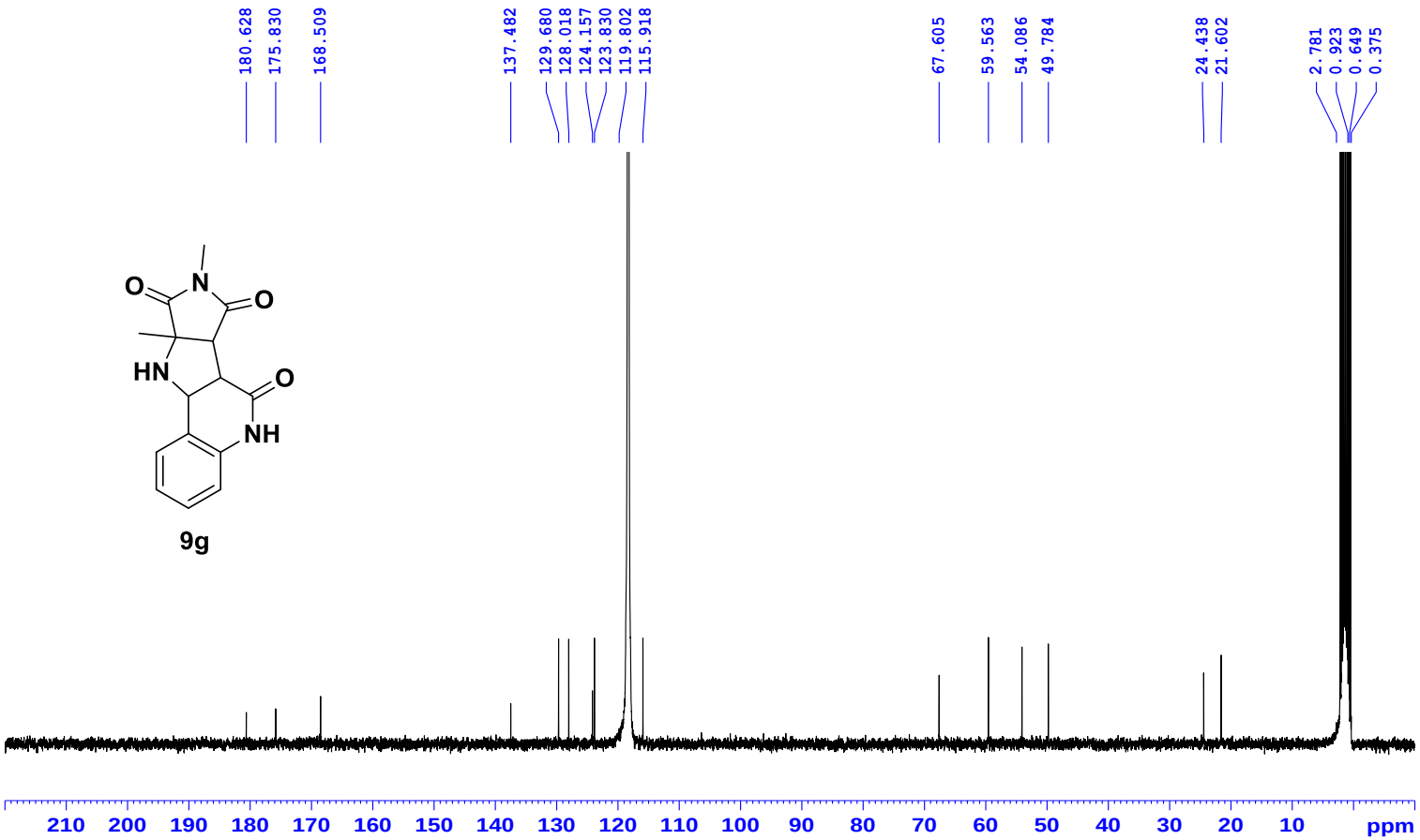
HRMS (EI) of compound **9f**



IR of compound **9f**



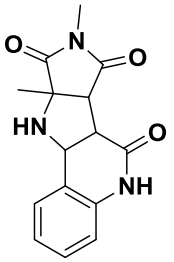
^{1}H NMR spectrum (400 MHz) of compound **9g** in CD_3CN



^{13}C NMR spectrum (101 MHz) of compound **9g** in CD_3CN

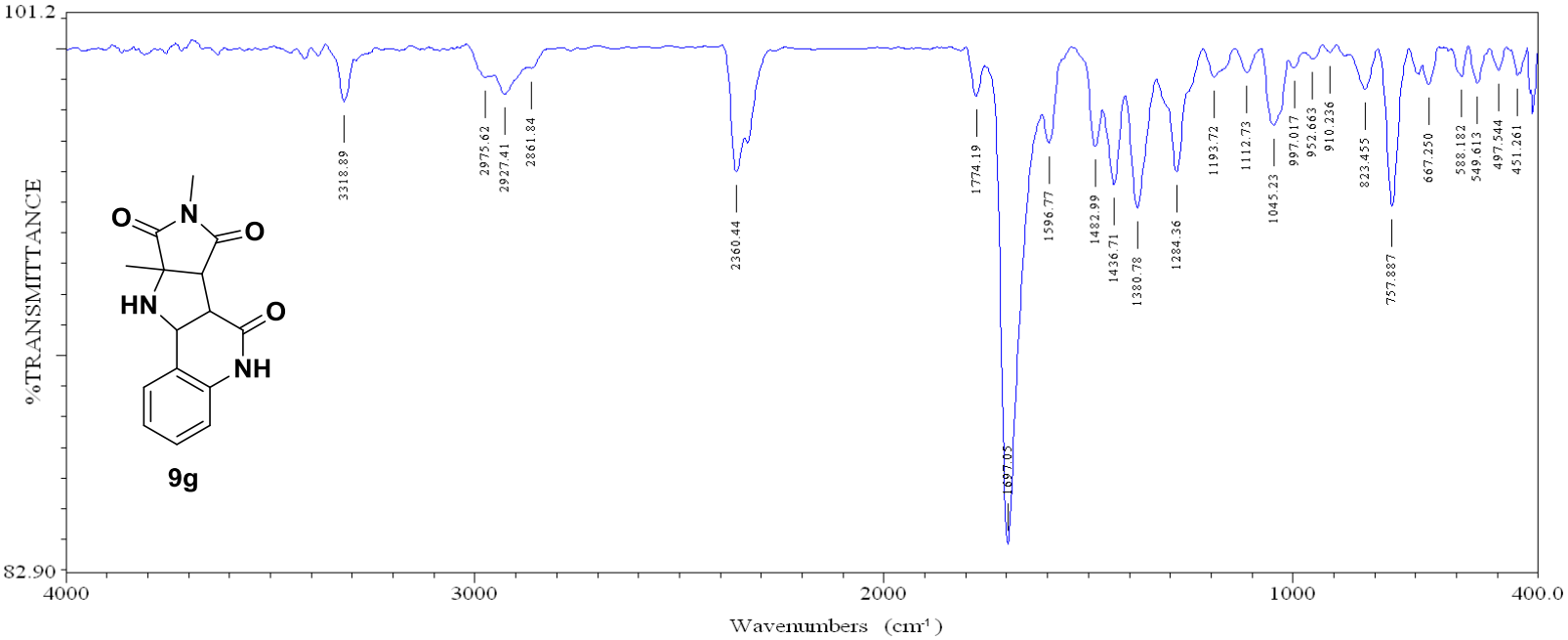
[Elemental Composition]
Data : 1001027-012 Date : 27-Oct-2011 10:33
Sample: leeut-FL07
Note : 285.11
Inlet : Direct Ion Mode : EI+
RT : 0.86 min Scan#: 26
Elements : C 15/0, H 40/0, N 3/0, O 3/0
Mass Tolerance : 10ppm, 5mmu if m/z < 500, 50mmu if m/z > 5000
Unsaturation (U.S.) : -0.5 - 200.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
285.1113	80.3	-0.2 / +0.0	10.0	C 15 H 15 N 3 O 3

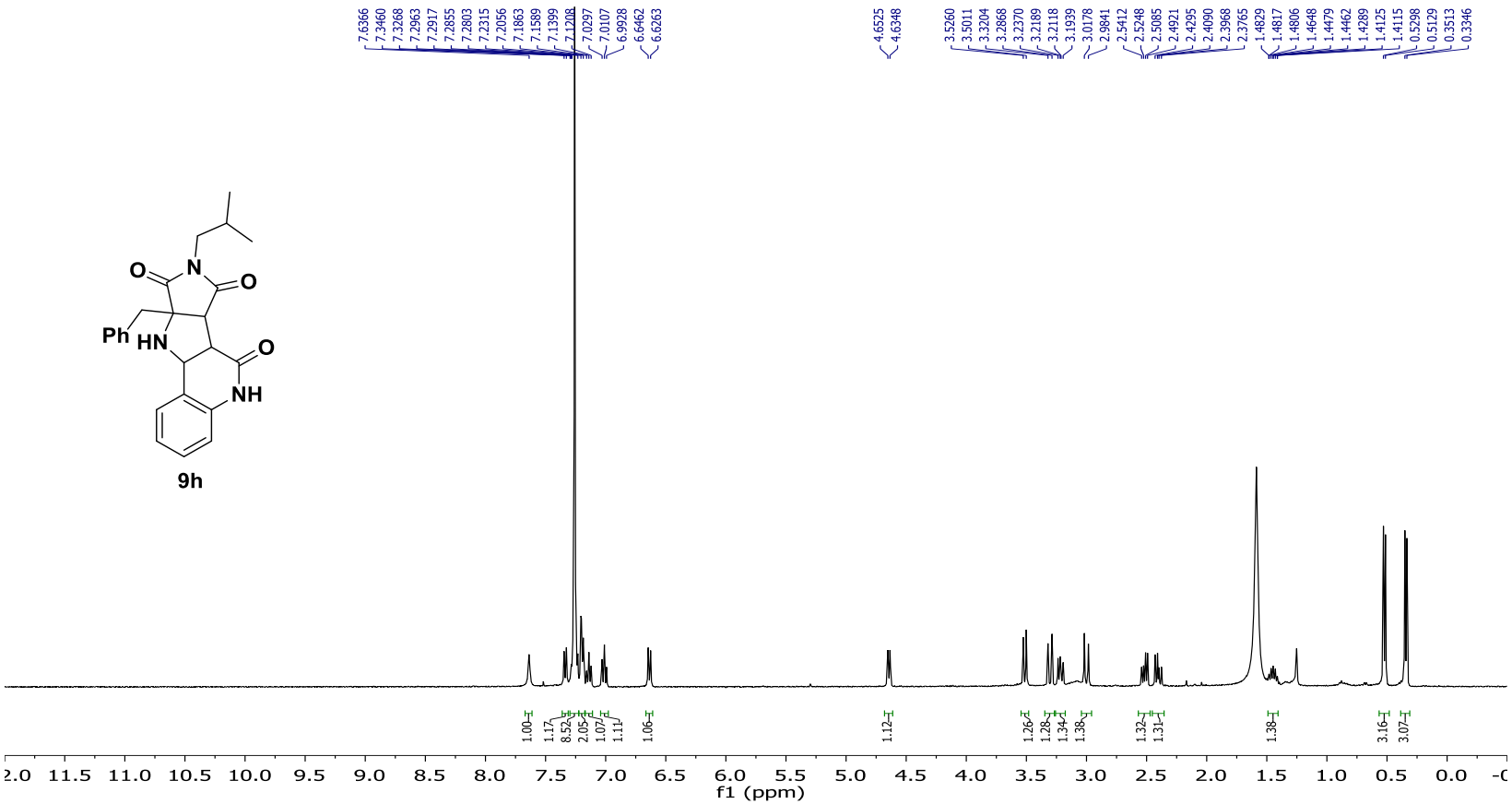


9g

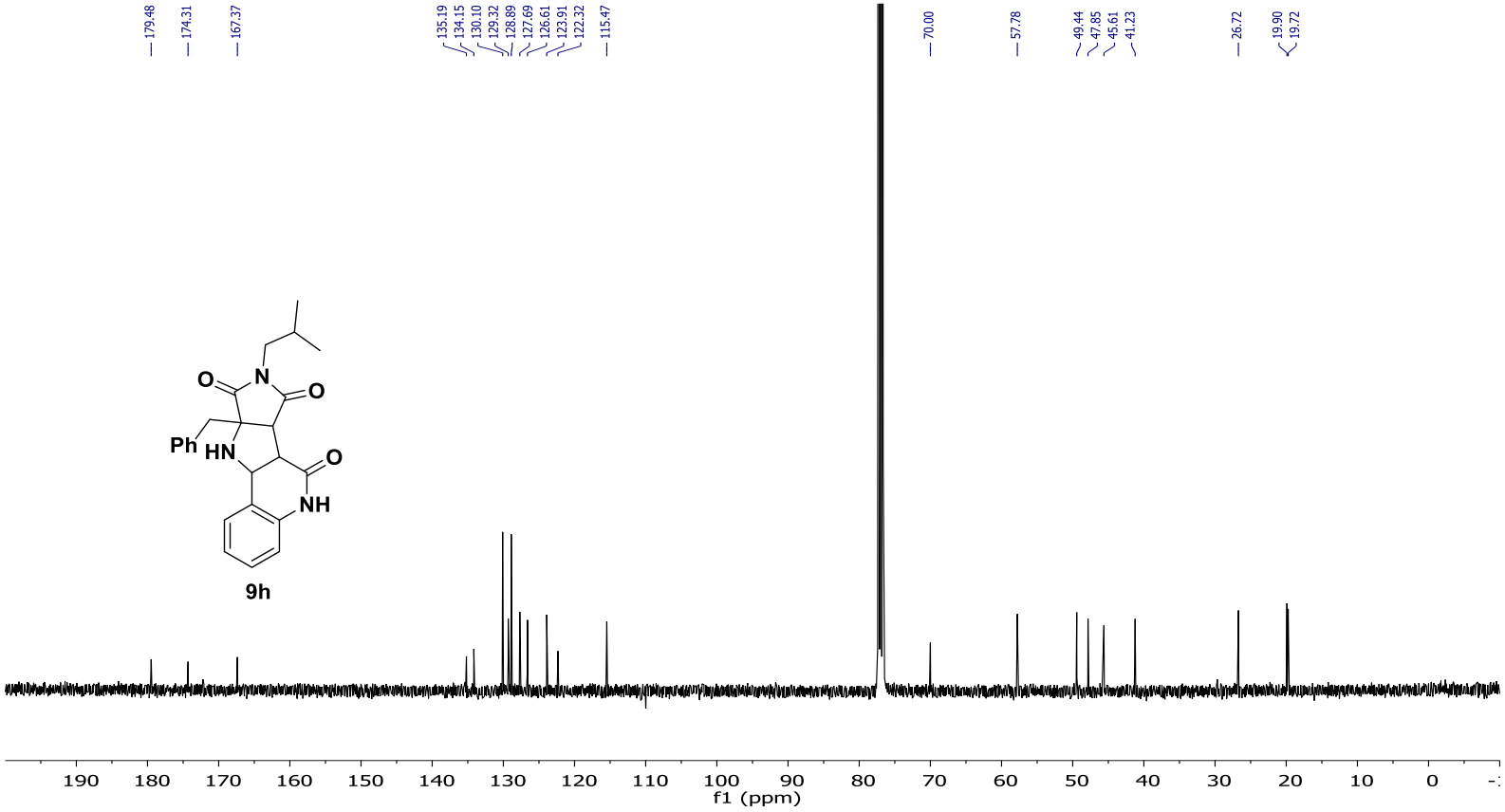
HRMS (EI) of compound **9g**



IR of compound **9g**



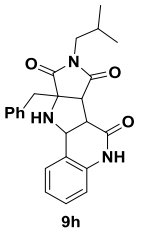
^{1}H NMR spectrum (400 MHz) of compound **9h** in $CDCl_3$



^{13}C NMR spectrum (101 MHz) of compound **9h** in $CDCl_3$

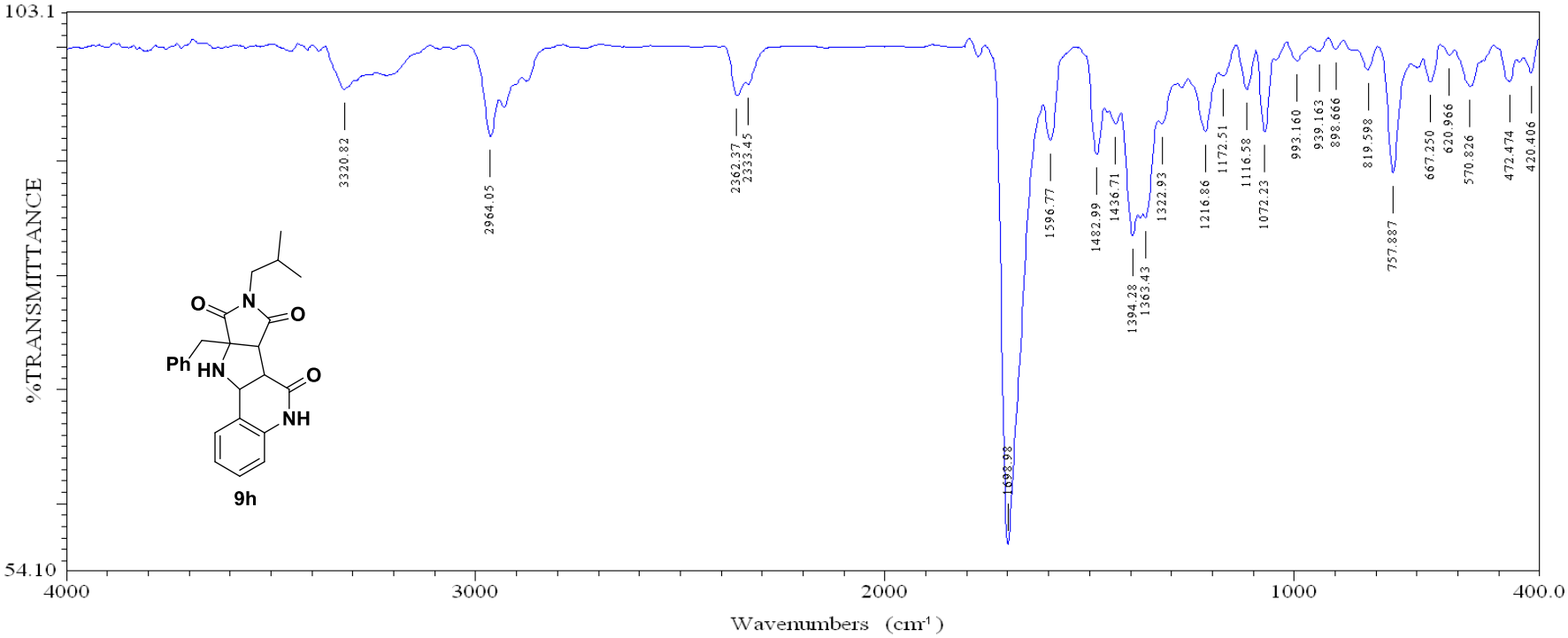
```
[ Elemental Composition ]
Data  : 201111050                          Date : 22-Oct-2011 17:30
Sample: leeut-El08
Note : -
Inlet : Direct                             Ion Mode : EI+
RT : 0.87 min                              Scan#: 43
Elements : C 24/0, H 25/0, O 5/0, N 3/0
Mass Tolerance       : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.1 - 36.0

Observed m/z Int%   Err[ppm / mmu]    U.S.  Composition
  403.1893    33.4     -0.7 /  -0.3   14.0  C 24 H 25 O 3 N 3
```

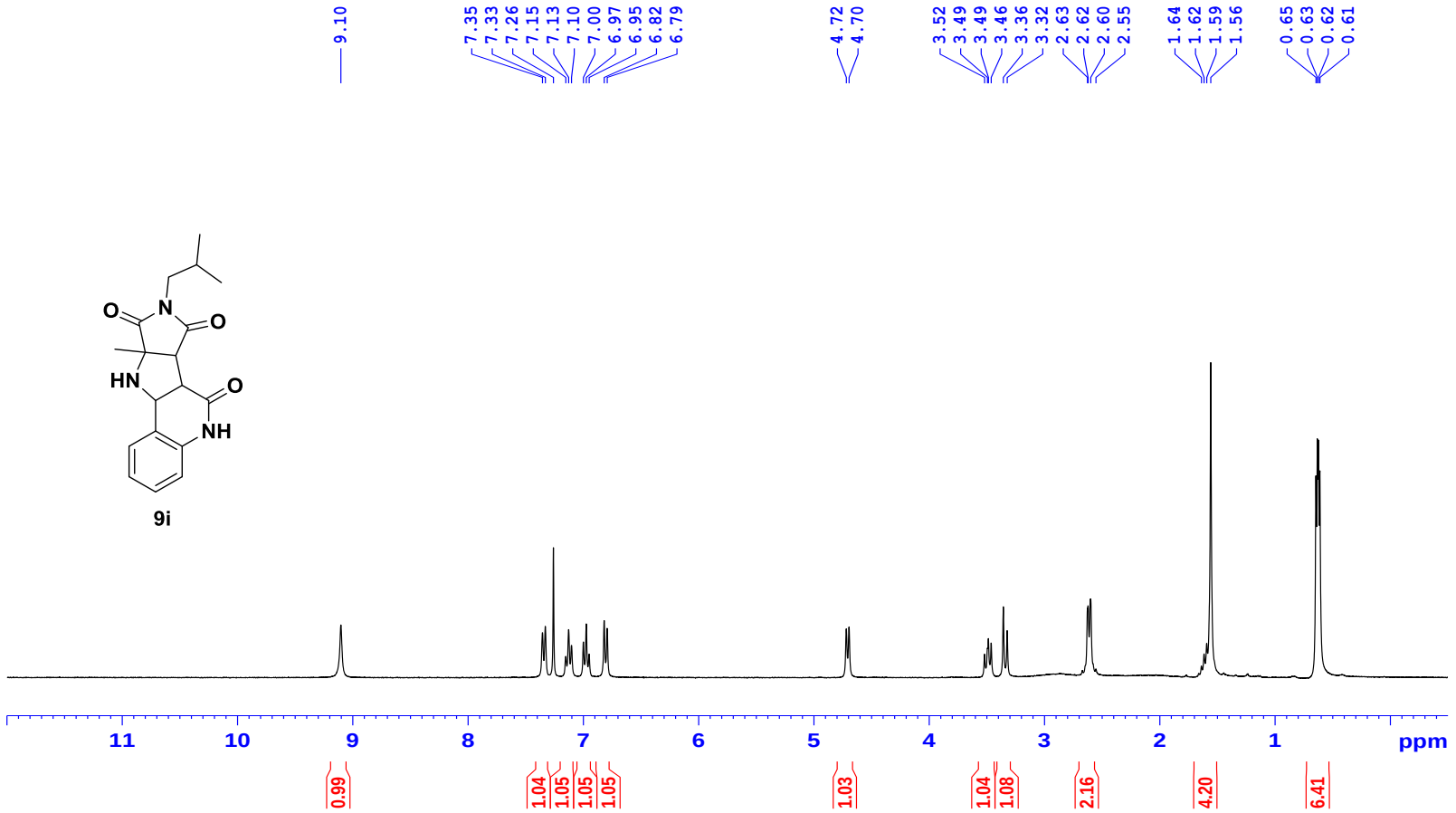


9h

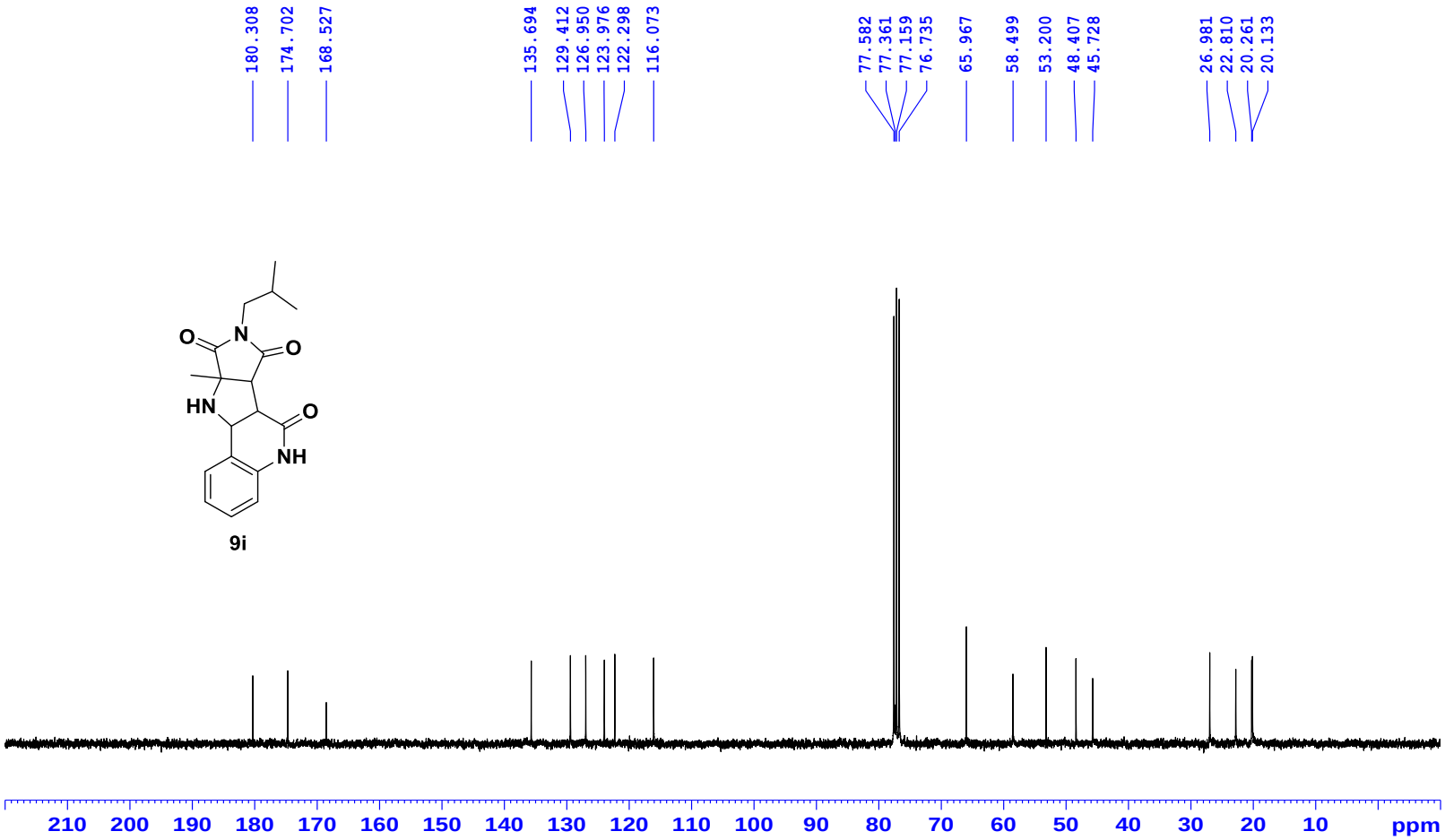
HRMS (EI) of compound **9h**



IR of compound **9h**



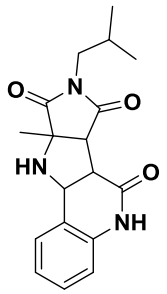
^{1}H NMR spectrum (400 MHz) of compound **9i** in $CDCl_3$



^{13}C NMR spectrum (101 MHz) of compound **9i** in $CDCl_3$

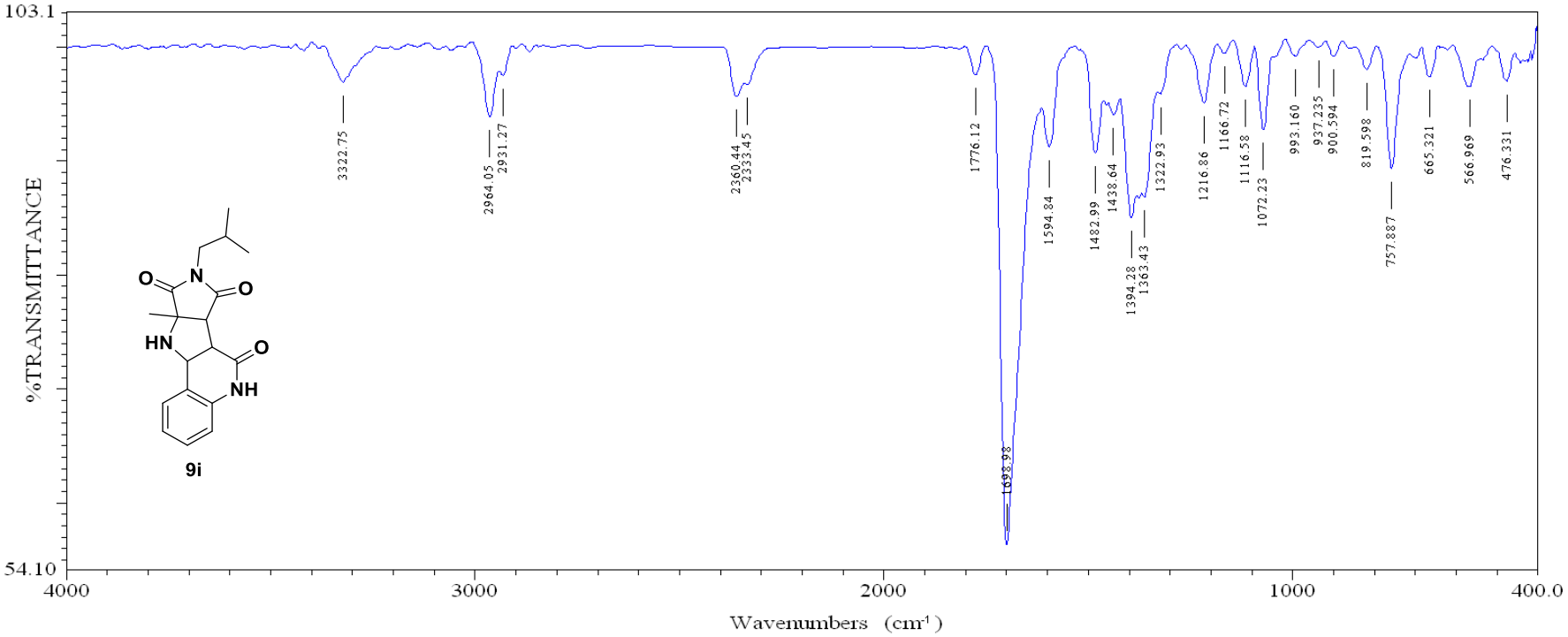
```
LIST: hei2109(leeutel9)-c2                    07-Jun-11 REG    : 03:13.9      #9
                                                        Start  : 14:50:29    753
Samp:
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
                                                        Inlet  :
Oper:
Limt: (  0)     .     .     .
    : (377) C19.H43.N3.O4
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/590>724  (CMASS : converted; CMASS : converted |CMASS : conve
```

Mass	Intensity	%RA	Flags	(mmu) Delta	R+D	Composition
327.1579	3598620	100.00	#	0.4	10.0	C18.H21.N3.O3

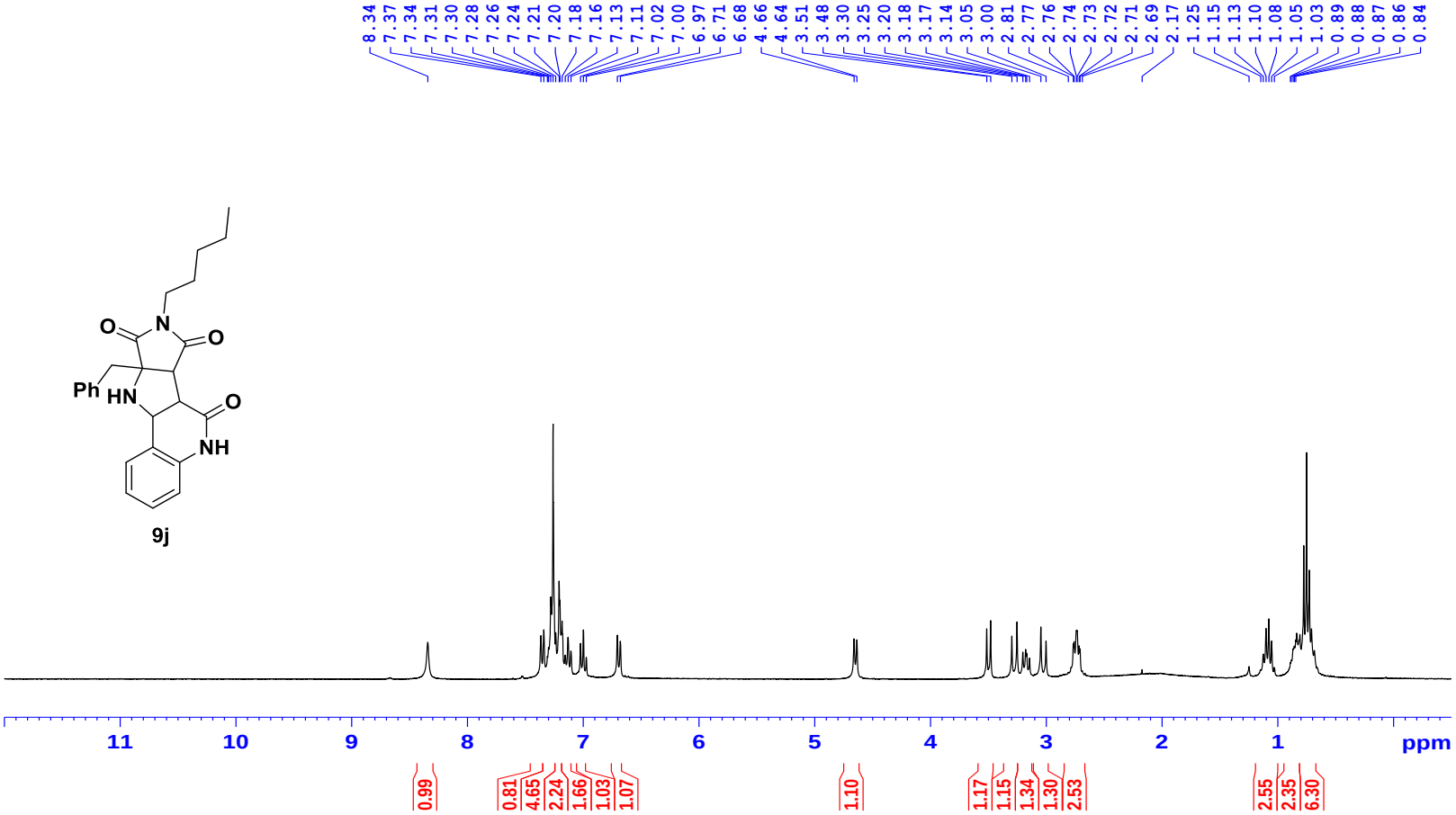


9i

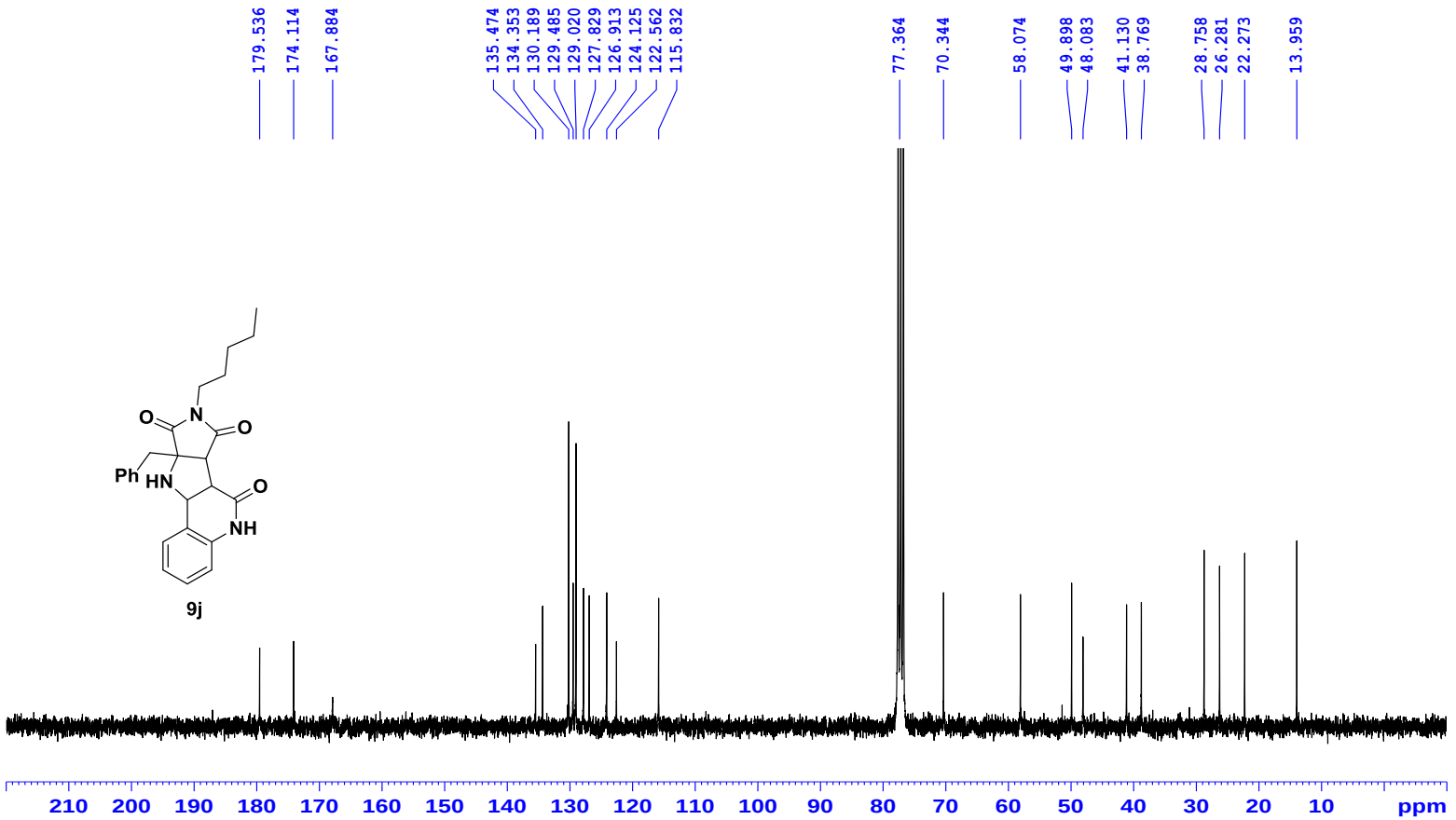
HRMS (EI) of compound **9i**



IR of compound **9i**



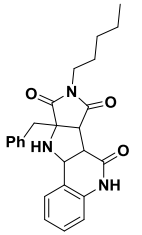
^{1}H NMR spectrum (400 MHz) of compound **9j** in $CDCl_3$



^{13}C NMR spectrum (101 MHz) of compound **9j** in $CDCl_3$

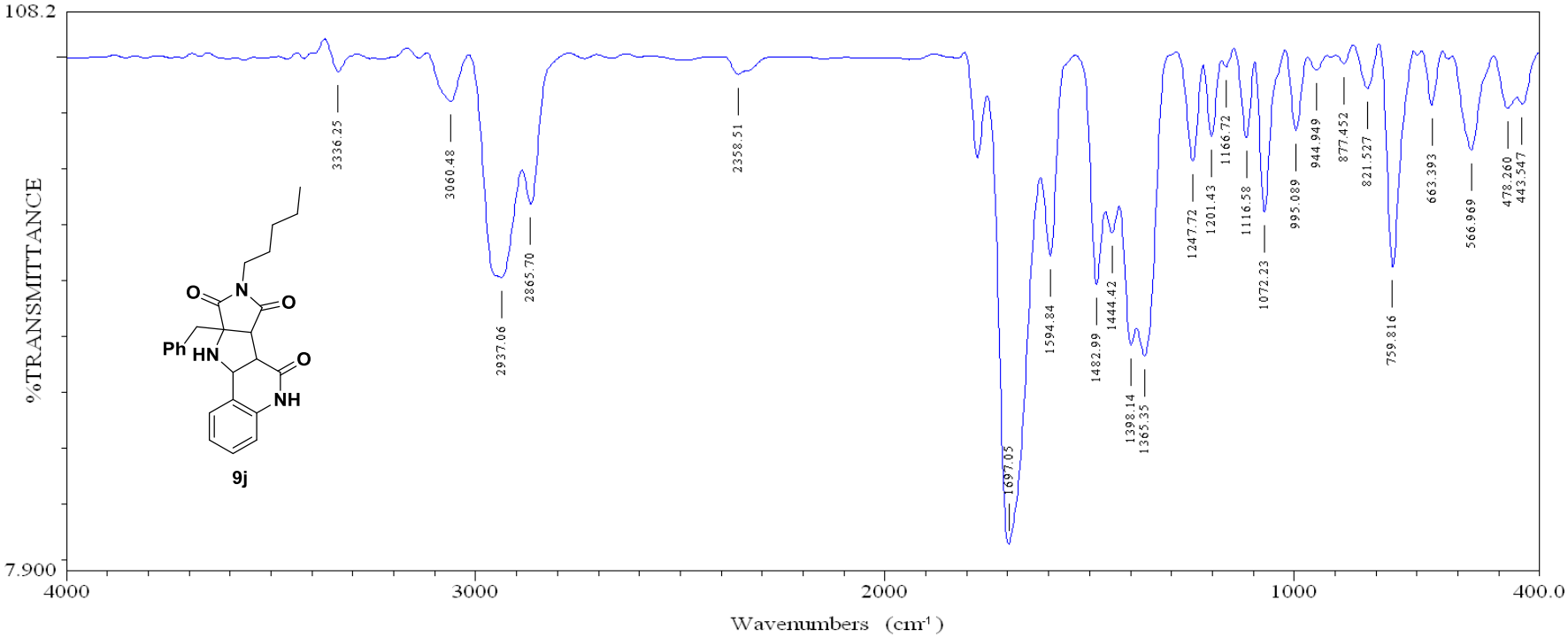
[Elemental Composition]
Data : 201111051 Date : 22-Oct-2011 17:38
Sample: leeut-El10
Note : -
Inlet : Direct Ion Mode : EI+
RT : 0.86 min Scan#: 39
Elements : C 25/0, H 27/0, O 5/0, N 3/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.1 - 36.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
417.2050	100.0	-0.5 / -0.2	14.0	C 25 H 27 O 3 N 3

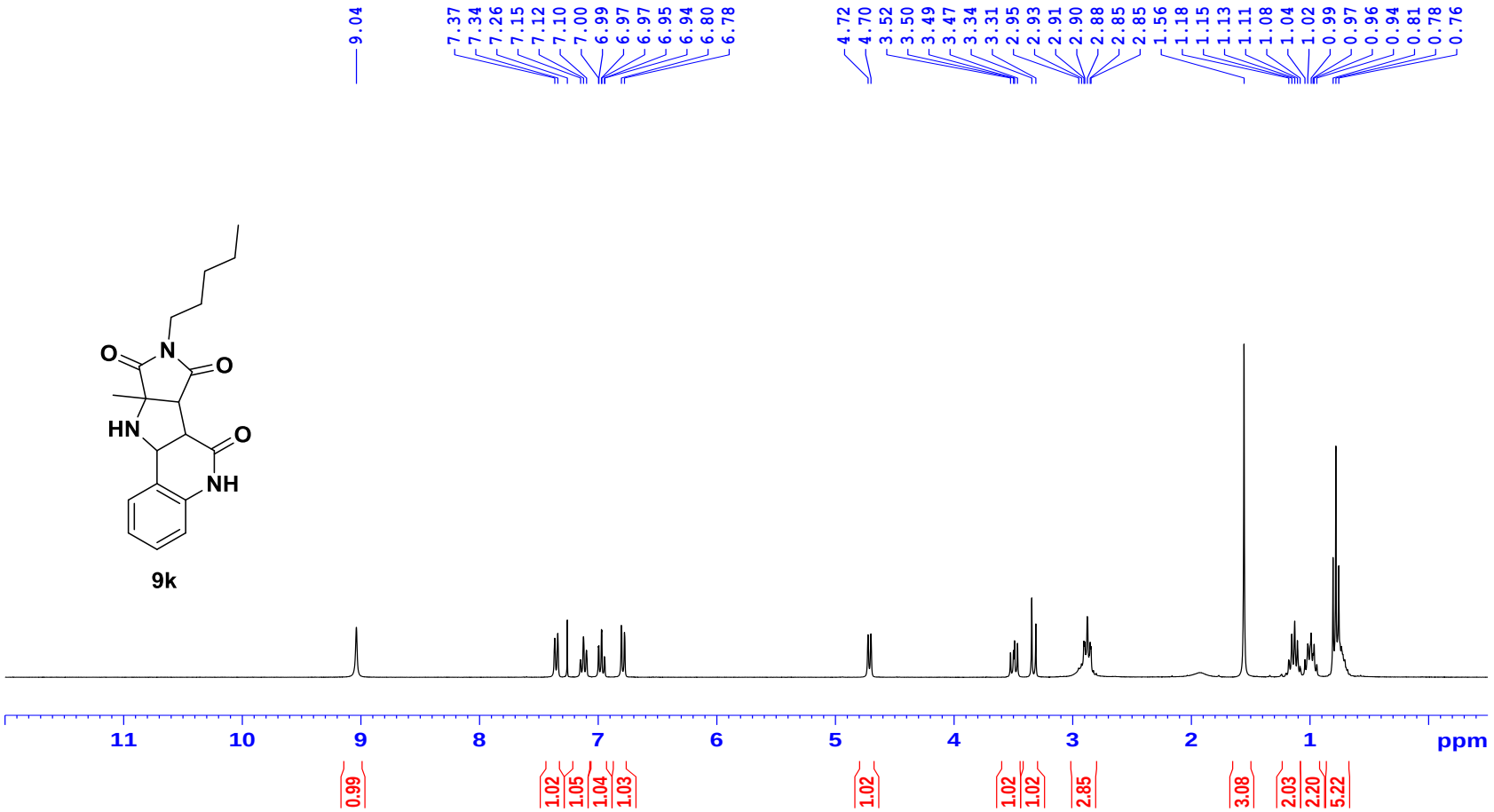


9j

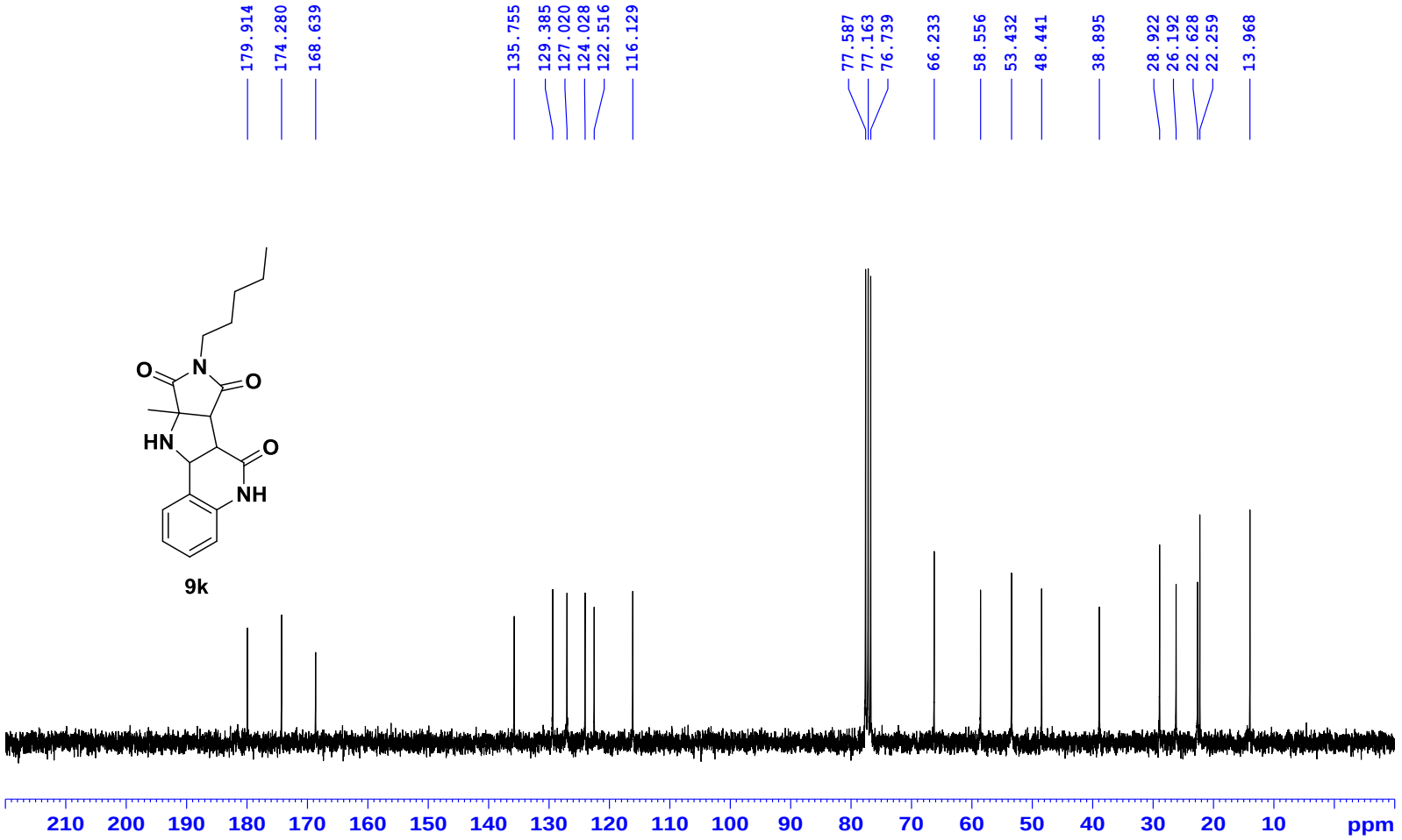
HRMS (EI) of compound **9j**



IR of compound **9j**



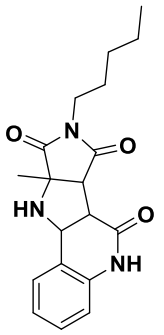
^{1}H NMR spectrum (400 MHz) of compound **9k** in $CDCl_3$



^{13}C NMR spectrum (101 MHz) of compound **9k** in $CDCl_3$

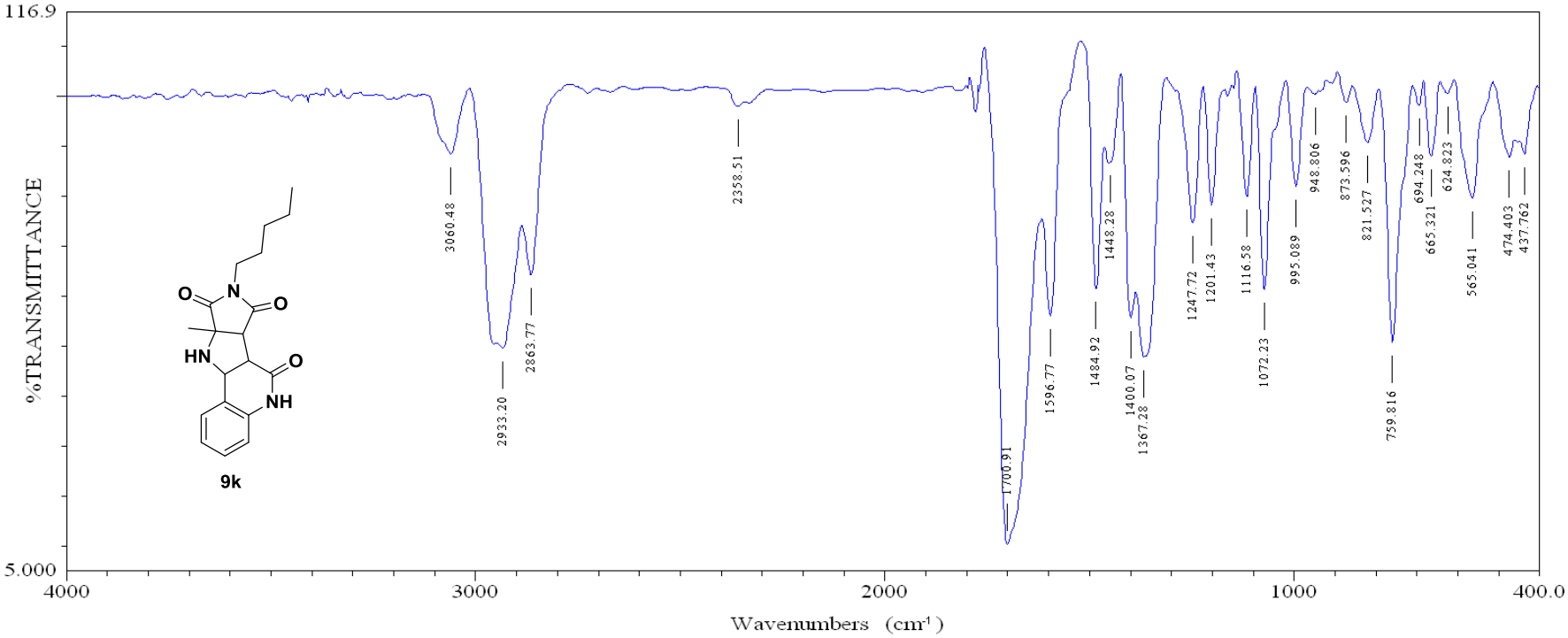
```
LIST: hei2108(leeutel11)-c2                    07-Jun-11 REG    : 06:56.8      #9
                                                         Start  : 13:57:02   1706
Samp:
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM                 Inlet  :
Oper:
Limt: (  0)     .     .     .
    : (389) C20.H45.O3.N4
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/1244>1522  (CMASS : converted; CMASS : converted |CMASS : con

                14648                          (mmu)
    Mass    Intensity      %RA   Flags Delta  R+D  Composition
341.1734        14822    50.59   #       0.6 10.0  C19.H23.O3.N3
```

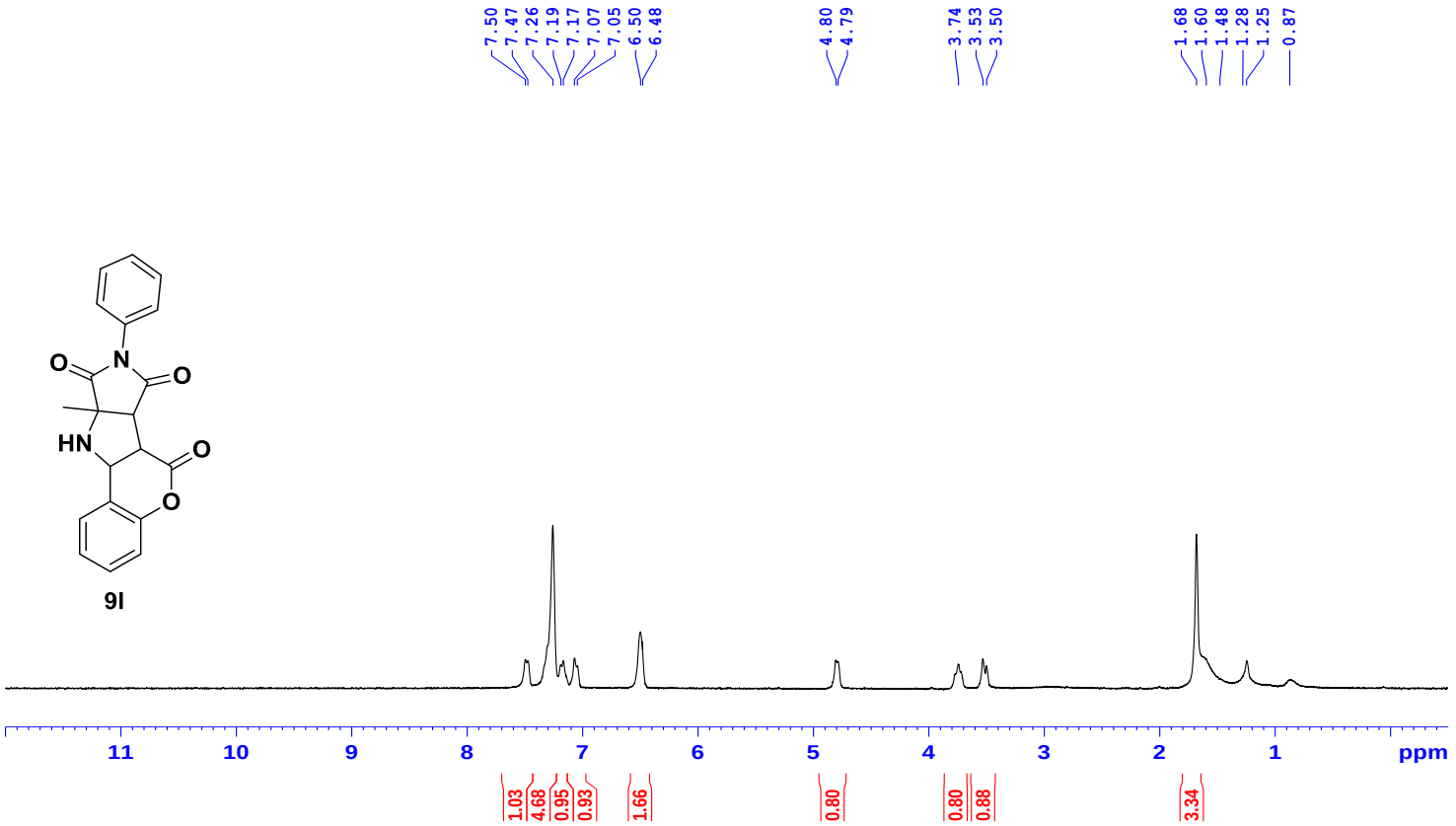


9k

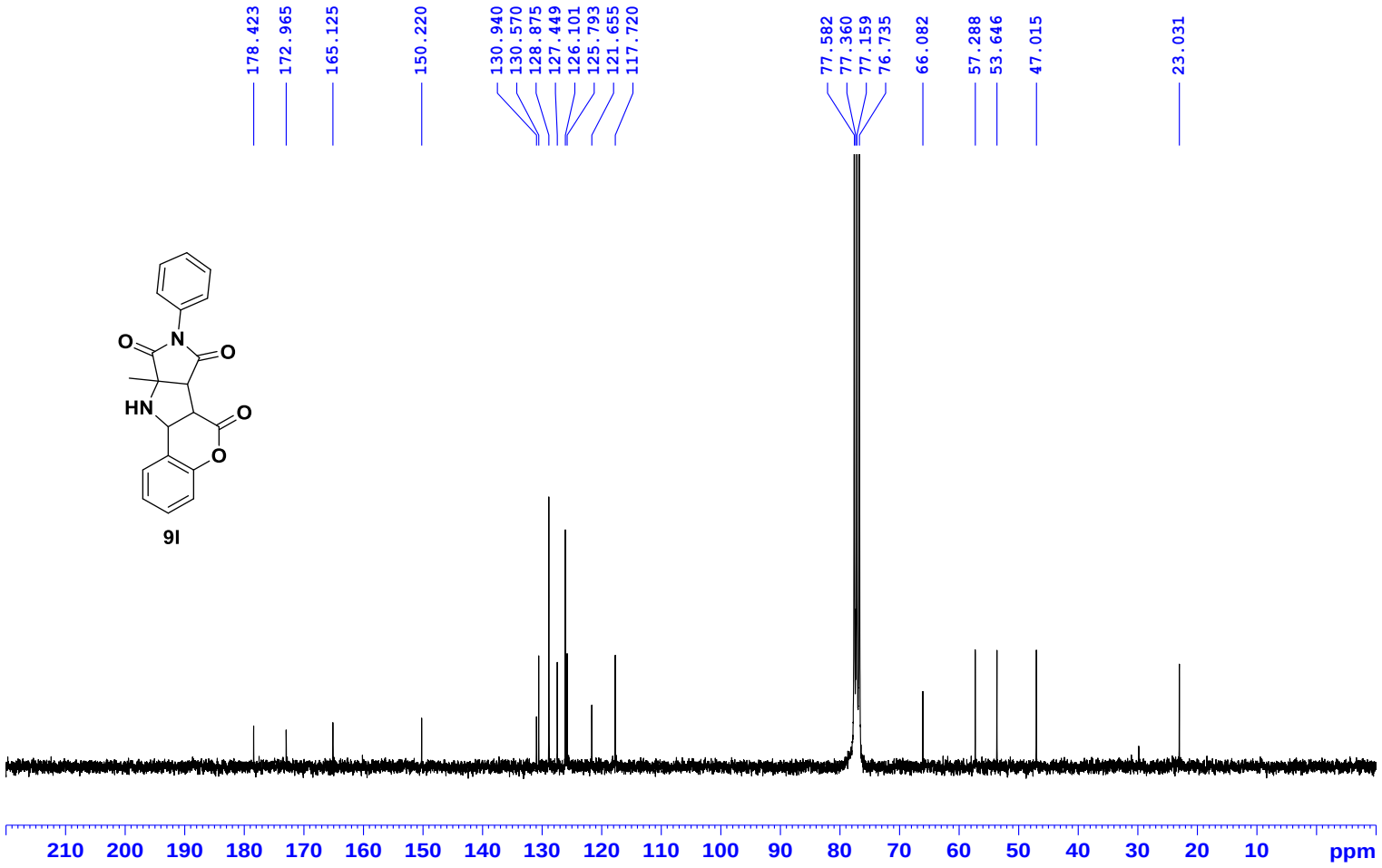
HRMS (EI) of compound **9k**



IR of compound **9k**



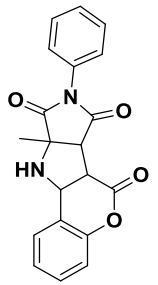
^{1}H NMR spectrum (400 MHz) of compound **9l** in $CDCl_3$



^{13}C NMR spectrum (101 MHz) of compound **9l** in $CDCl_3$

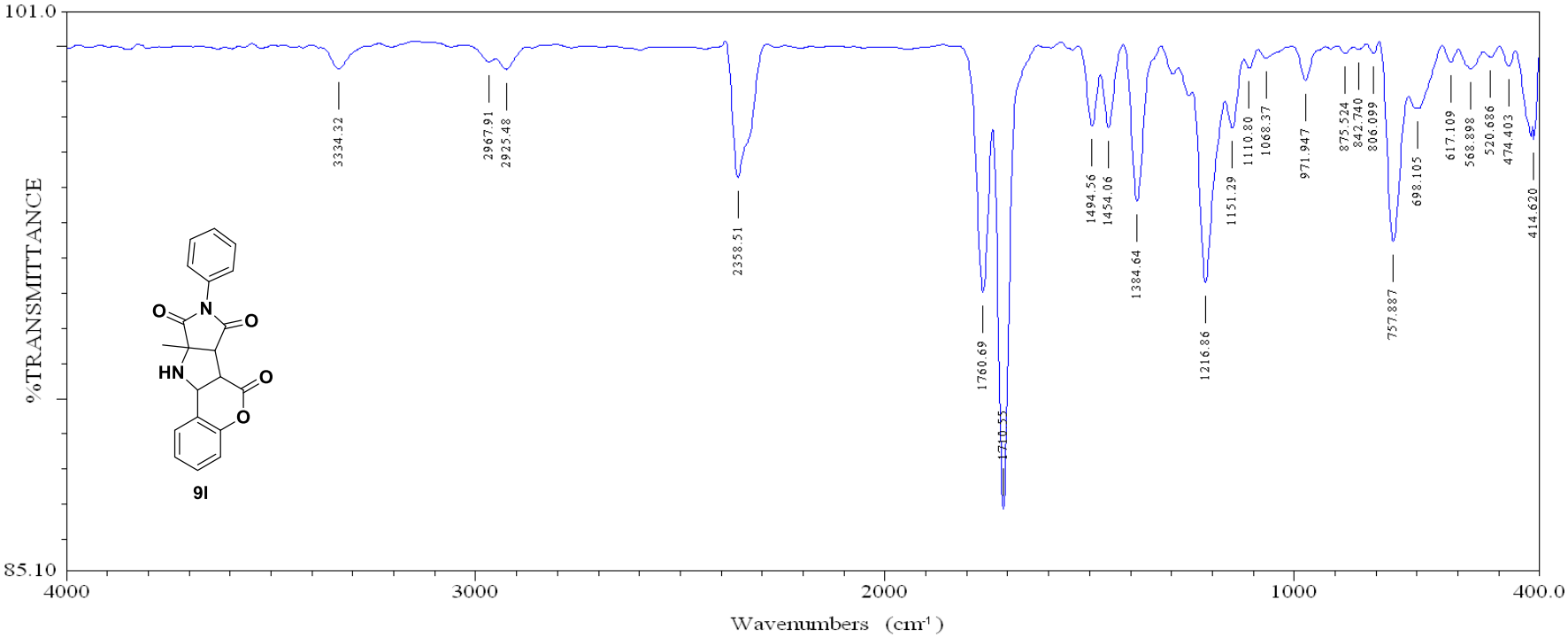
```
LIST: hei2107(leeutel12)-c3                    07-Jun-11 REG   : 04:17.7      #9
                                                         Start : 14:21:16   5332
Samp:
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM                 Inlet :
Oper:
Limt: (  0)     .     .     .
    : (477) C28.H33.N2.O5
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/796>1145  (CMASS : converted |CMASS : converted |CMASS : conv

                    0                          (mmu)
     Mass  Intensity      %RA   Flags Delta  R+D  Composition
 348.1100     124575   100.00    #       1.0 14.0  C20.H16.N2.O4
```

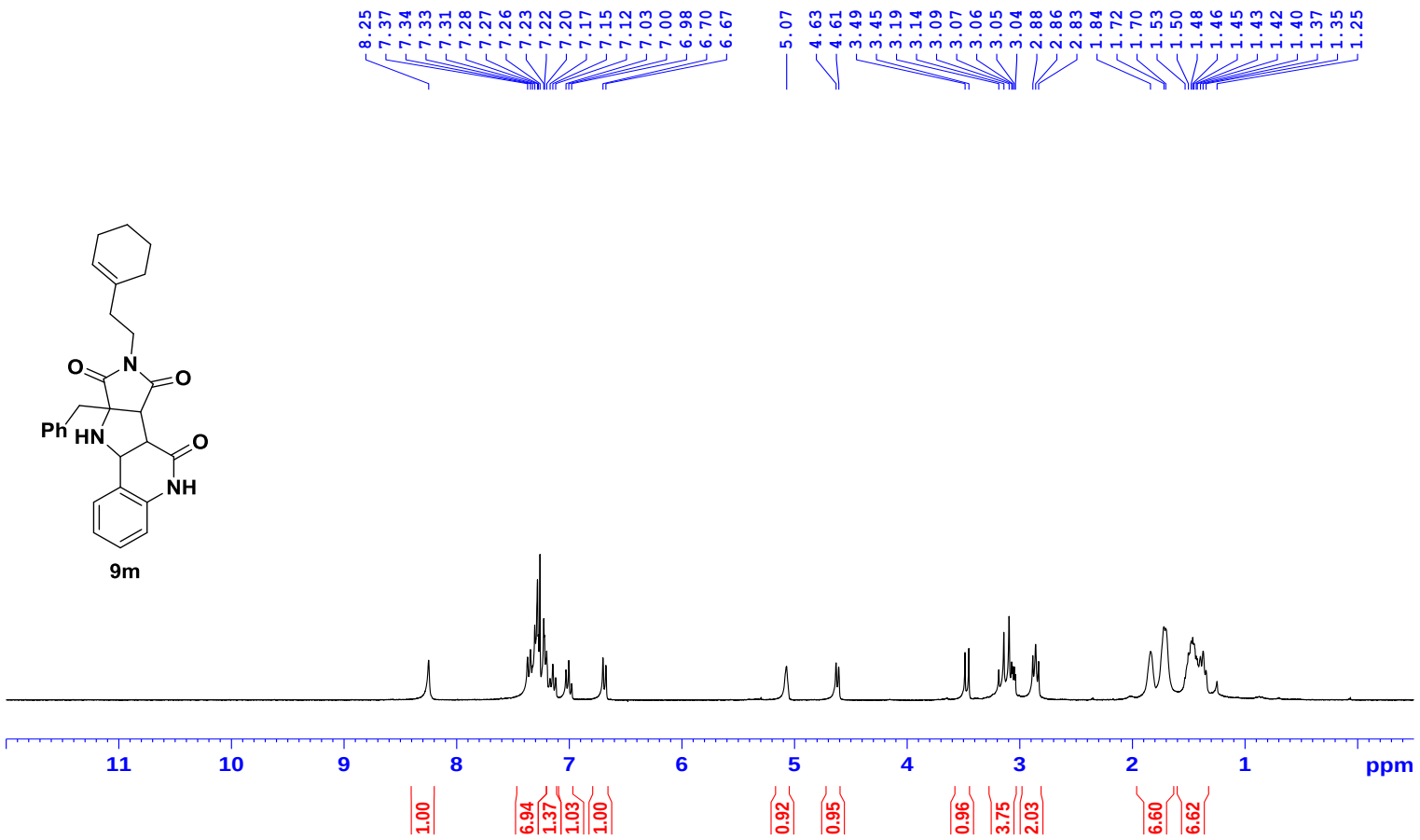


9l

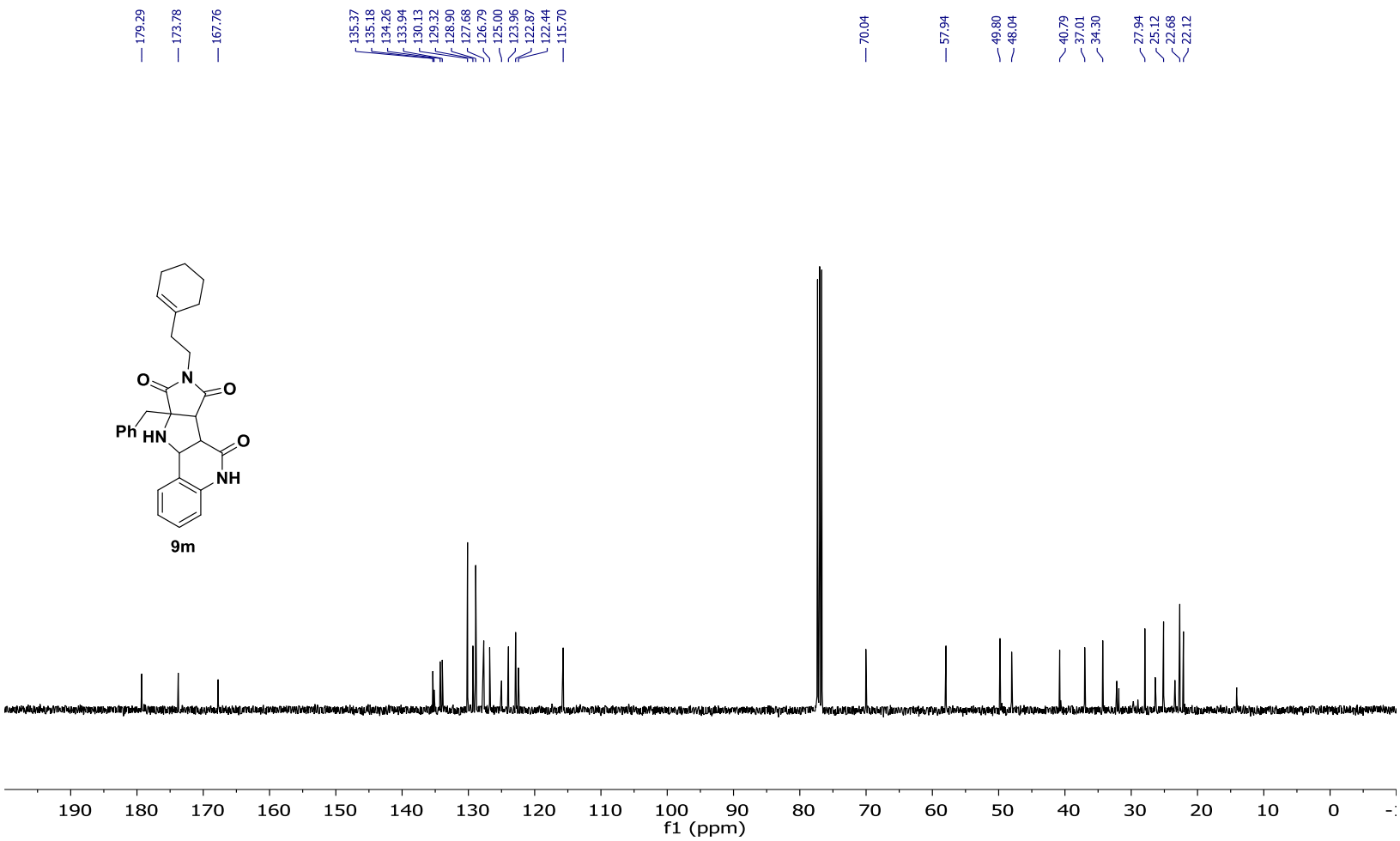
HRMS (EI) of compound **9l**



IR of compound **9l**



^{1}H NMR spectrum (400 MHz) of compound **9m** in $CDCl_3$



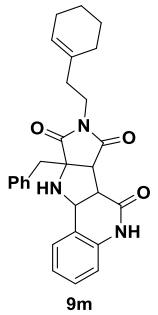
^{13}C NMR spectrum (101 MHz) of compound **9m** in $CDCl_3$

[Elemental Composition]
Data : 1000512-012
Sample: Leeut-EL13
Note : 455.55
Inlet : Direct
RT : 0.81 min
Elements : C 28/0, H 36/0, N 3/0, O 3/0
Mass Tolerance : 10ppm, 3mmu if m/z < 300, 20mmu if m/z > 2000
Unsaturation (U.S.) : -0.5 - 200.0

Date : 12-May-2011 11:54

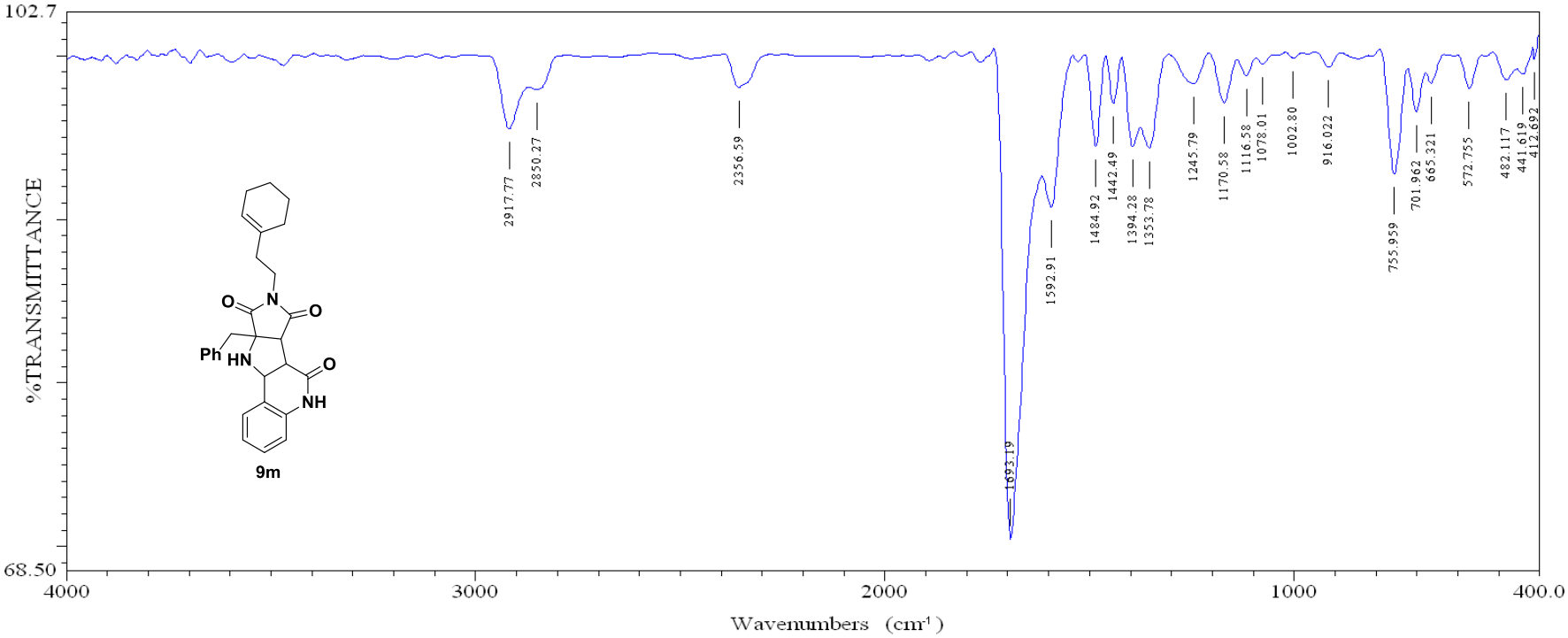
Ion Mode : EI+
Scan#: 56

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
455.2213	100.0	+0.9 / +0.4	16.0	C 28 H 29 N 3 O 3

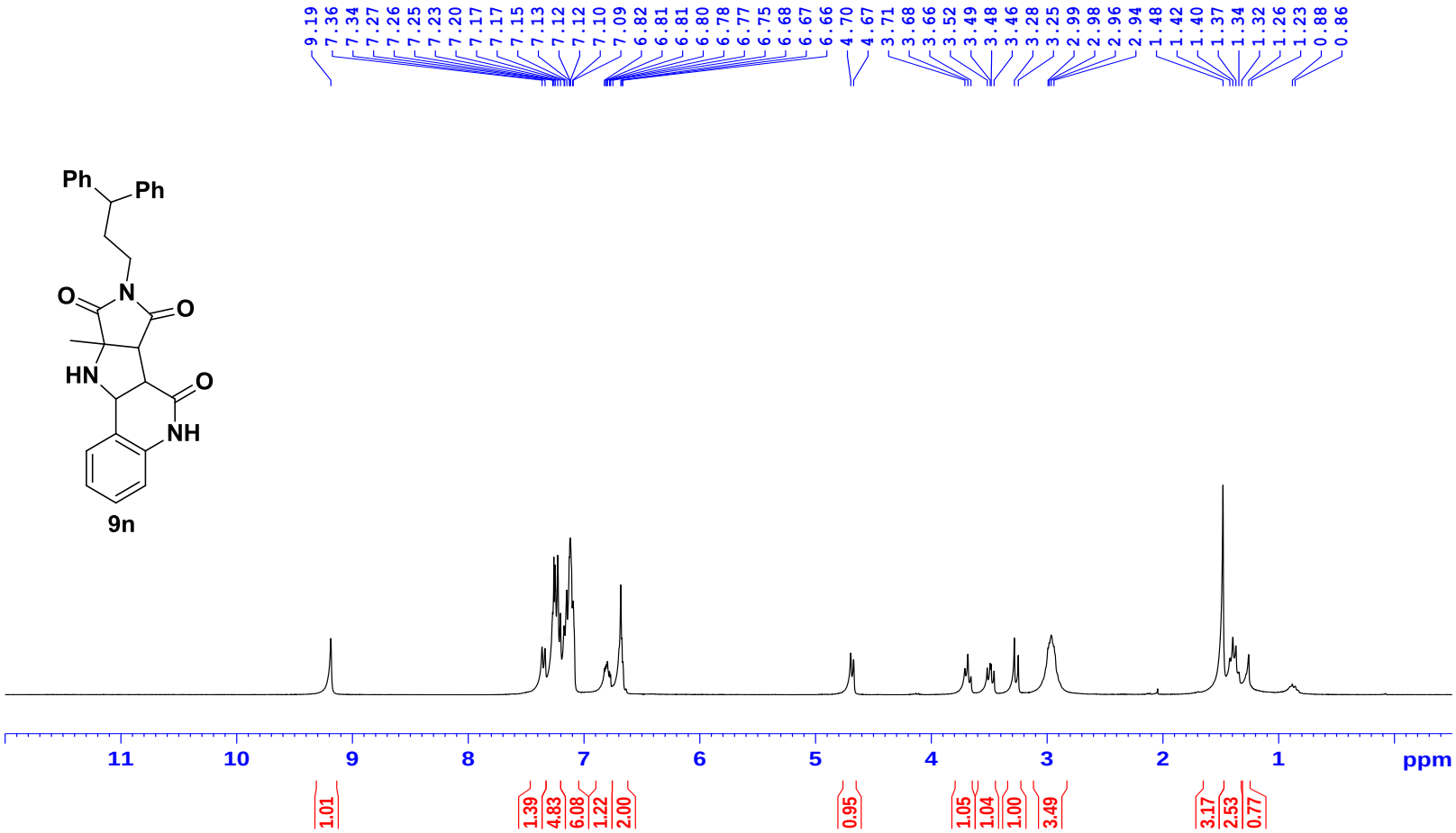


9m

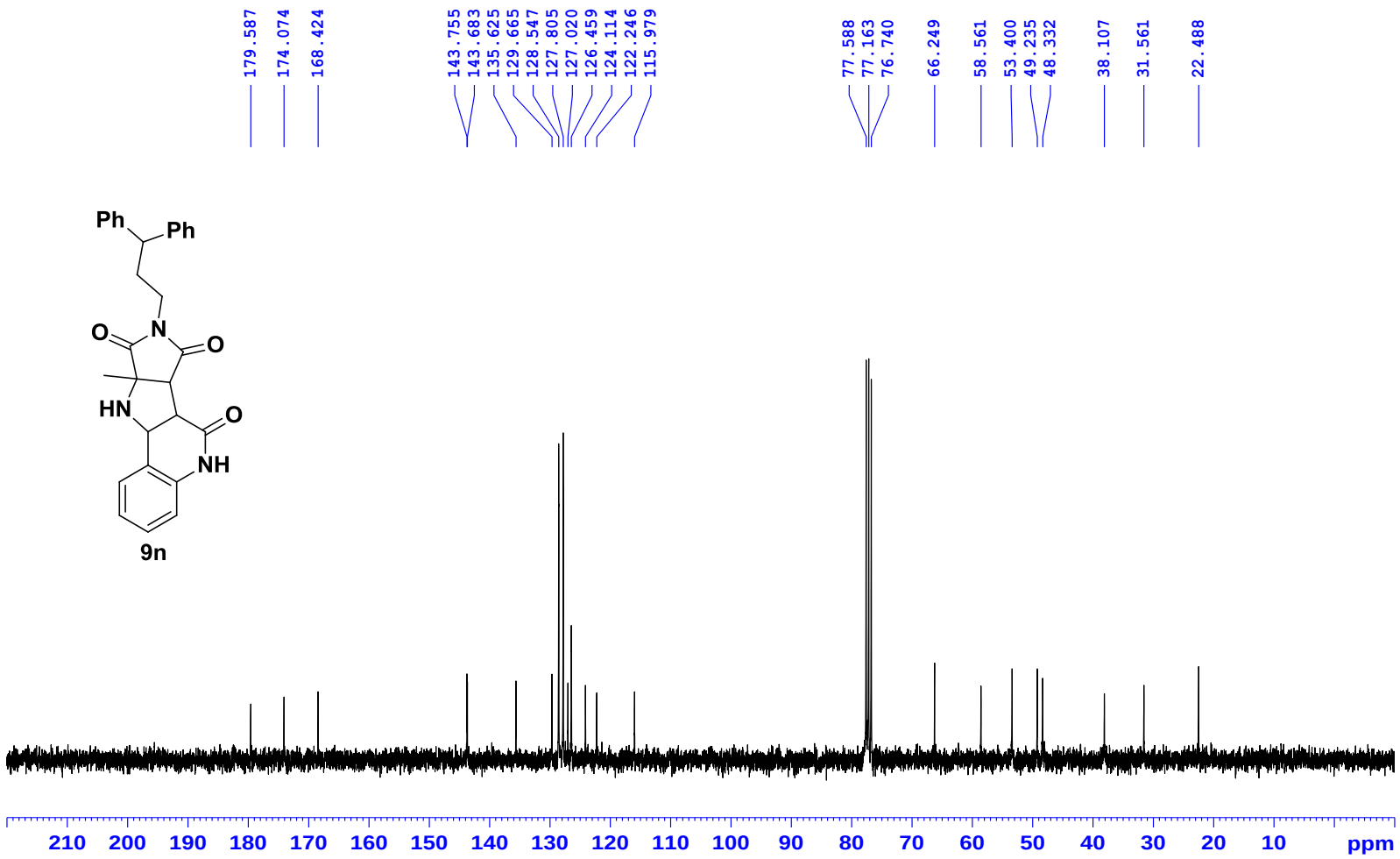
HRMS (EI) of compound **9m**



IR spectrum of compound **9m**



^{1}H NMR spectrum (400 MHz) of compound **9n** in $CDCl_3$



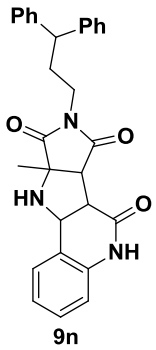
^{13}C NMR spectrum (101 MHz) of compound **9n** in $CDCl_3$

[Elemental Composition]
Data : 1000512-011
Sample: Leeut-EL14
Note : 465.54
Inlet : Direct
RT : 0.89 min
Elements : C 29/0, H 36/0, N 3/0, O 3/0
Mass Tolerance : 10ppm, 3mmu if m/z < 300, 20mmu if m/z > 2000
Unsaturation (U.S.) : -0.5 - 200.0

Date : 12-May-2011 11:49

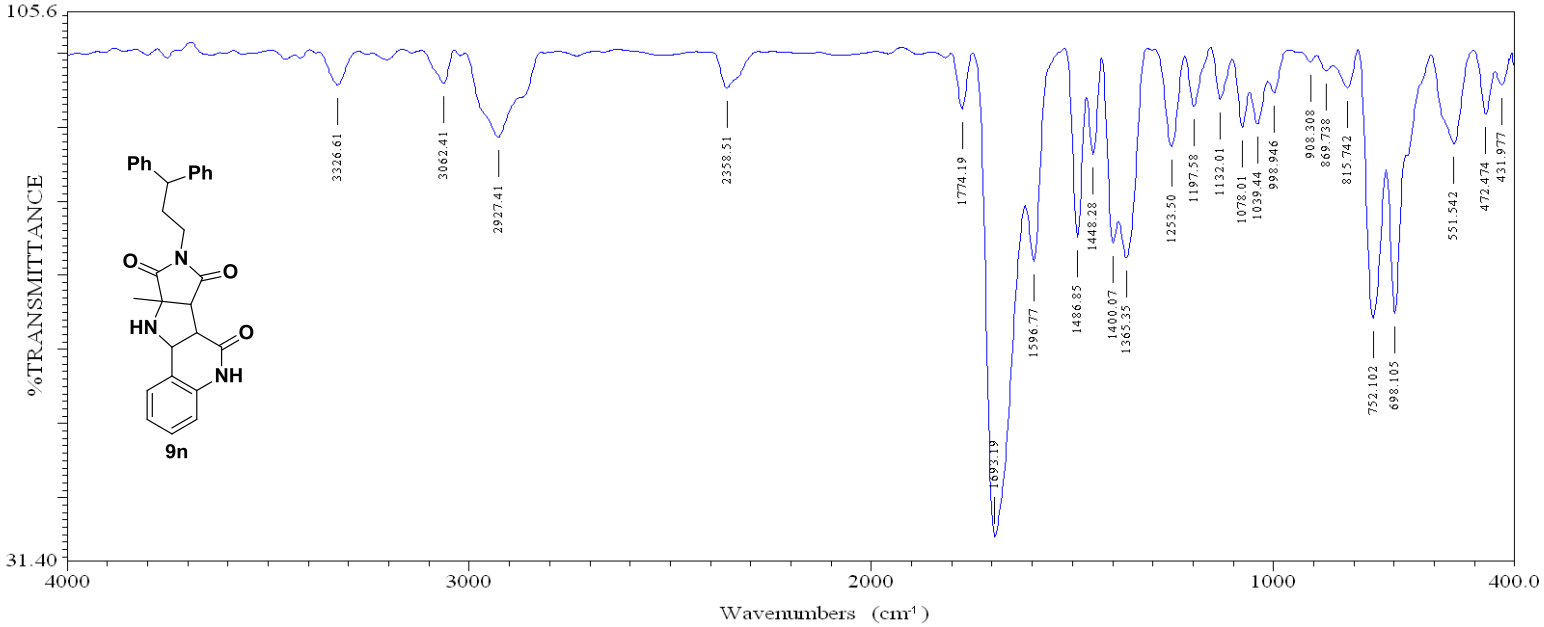
Ion Mode : EI+
Scan#: 61

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
465.2048	100.0	-0.9 / -0.4	18.0	C 29 H 27 N 3 O 3

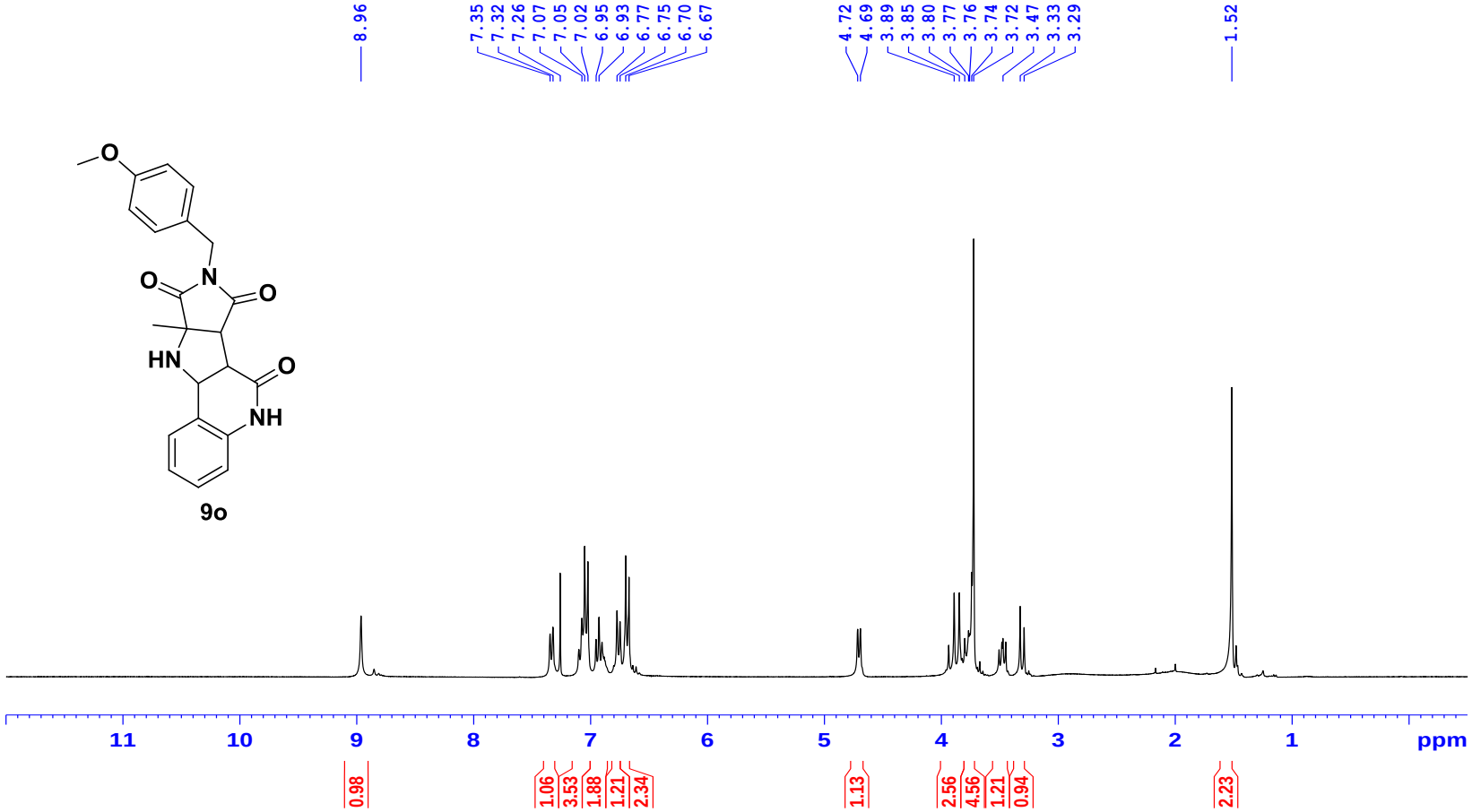


9n

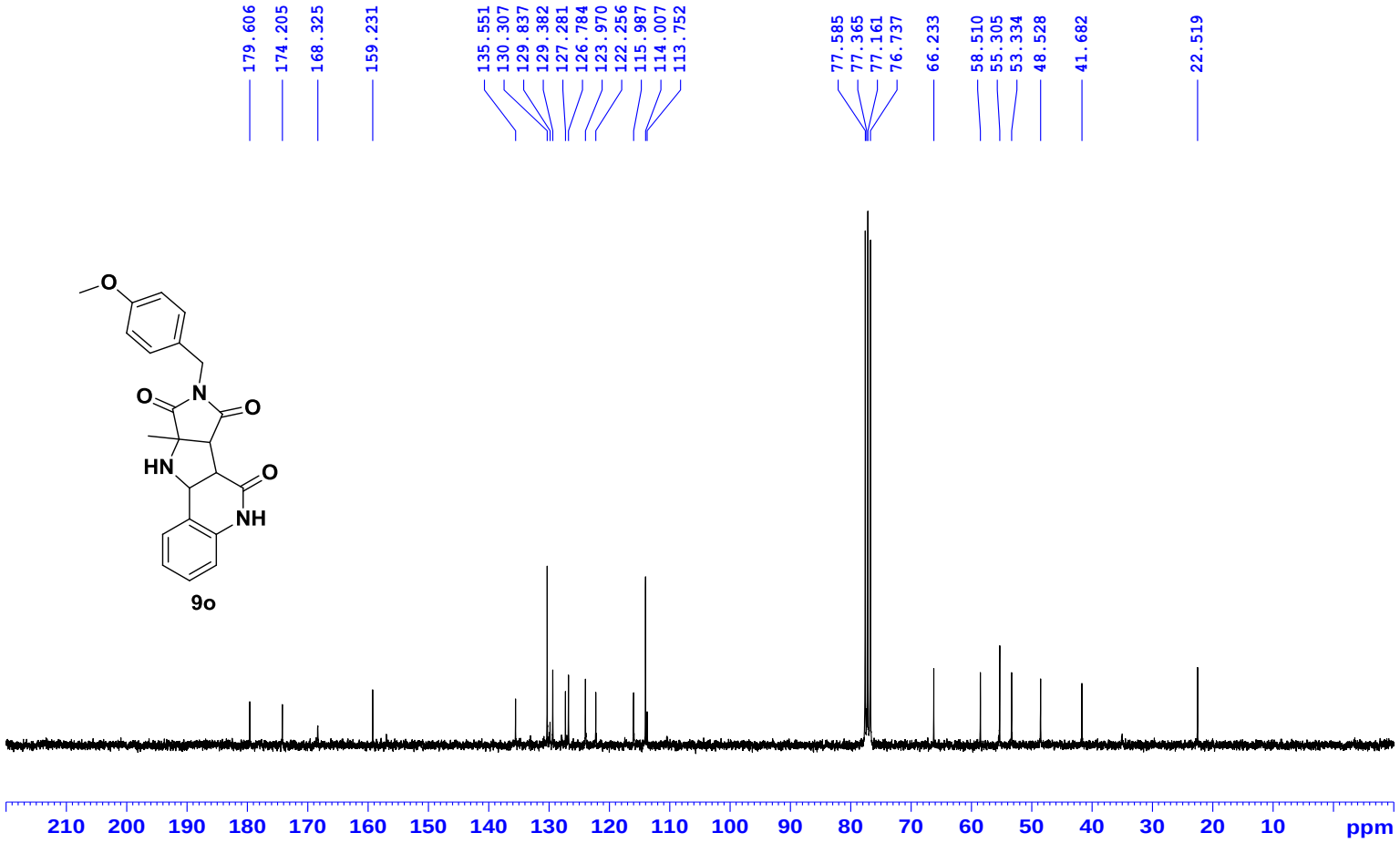
HRMS (EI) of compound **9n**



IR spectrum of compound of **9n**



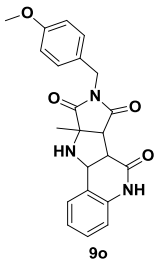
^{1}H NMR spectrum (400 MHz) of compound **9o** in $CDCl_3$



^{13}C NMR spectrum (101 MHz) of compound **9o** in $CDCl_3$

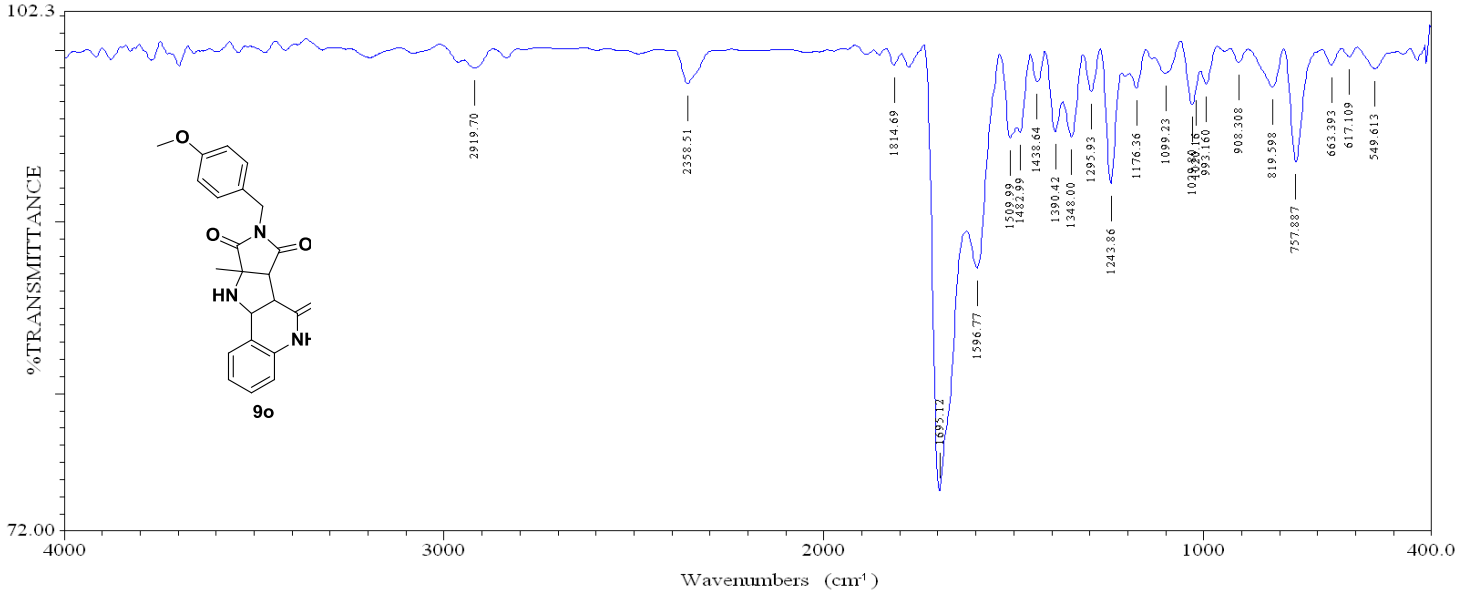
LIST: hei2532(leeut-el15)-c3 29-Sep-11 REG : 10:29.3 #9
Start : 16:17:02 3545
Samp:
Mode: EI +VE +LMR ESCAN (EXP) UP HR NRM Inlet :
Oper:
Limt: (0) . . .
: (442) C23.H44.N3.O5
Peak: 1000.00 mmu R+D: -2.0 > 60.0
Data: +/2012>2456 (CMASS : converted; CMASS : converted |CMASS : con

Mass	Intensity	%RA	Flags	(mmu) Delta	R+D	Composition
391.1541	22334	49.96	#	-0.9	14.0	C22.H21.N3.O4

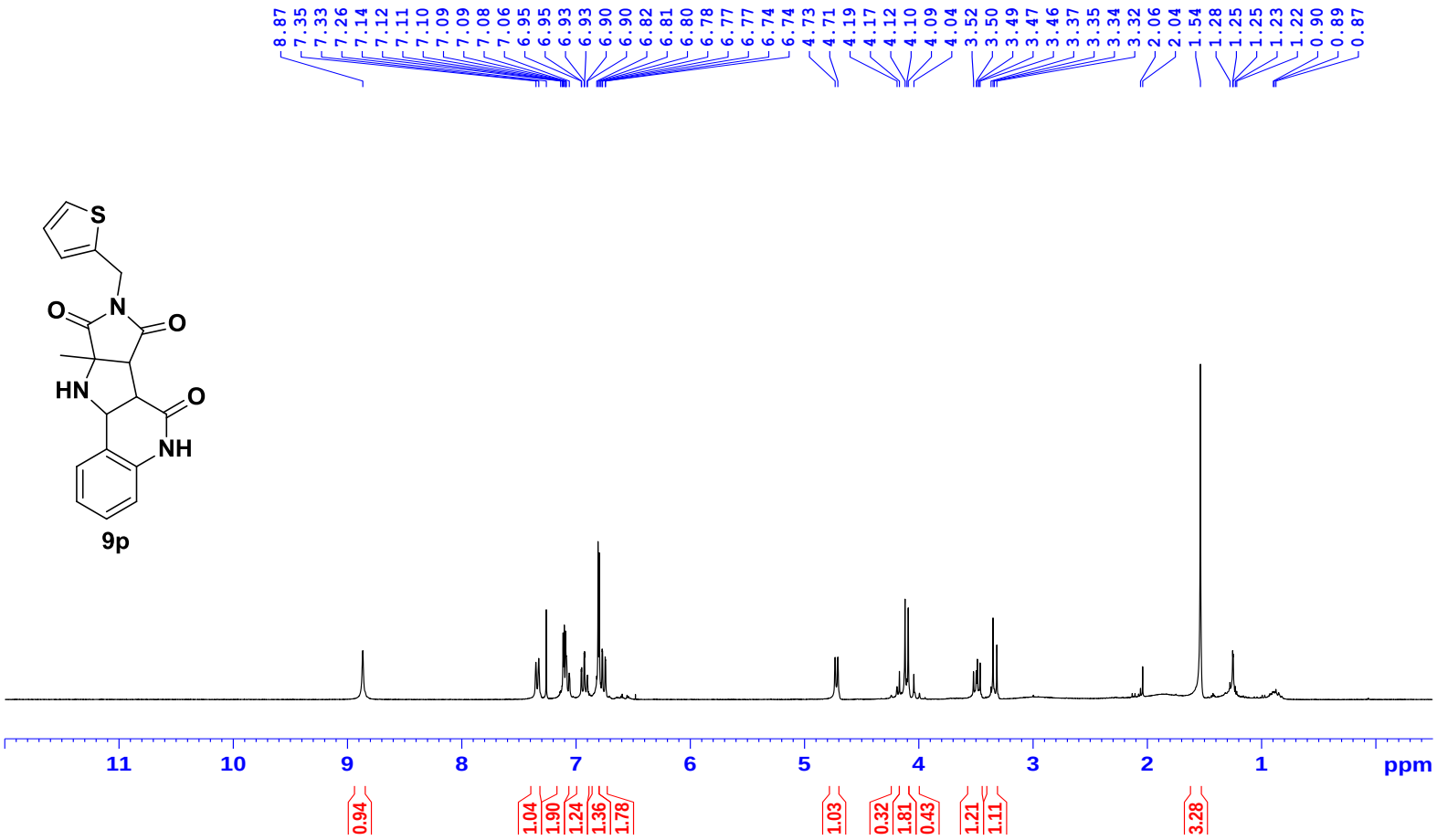


9o

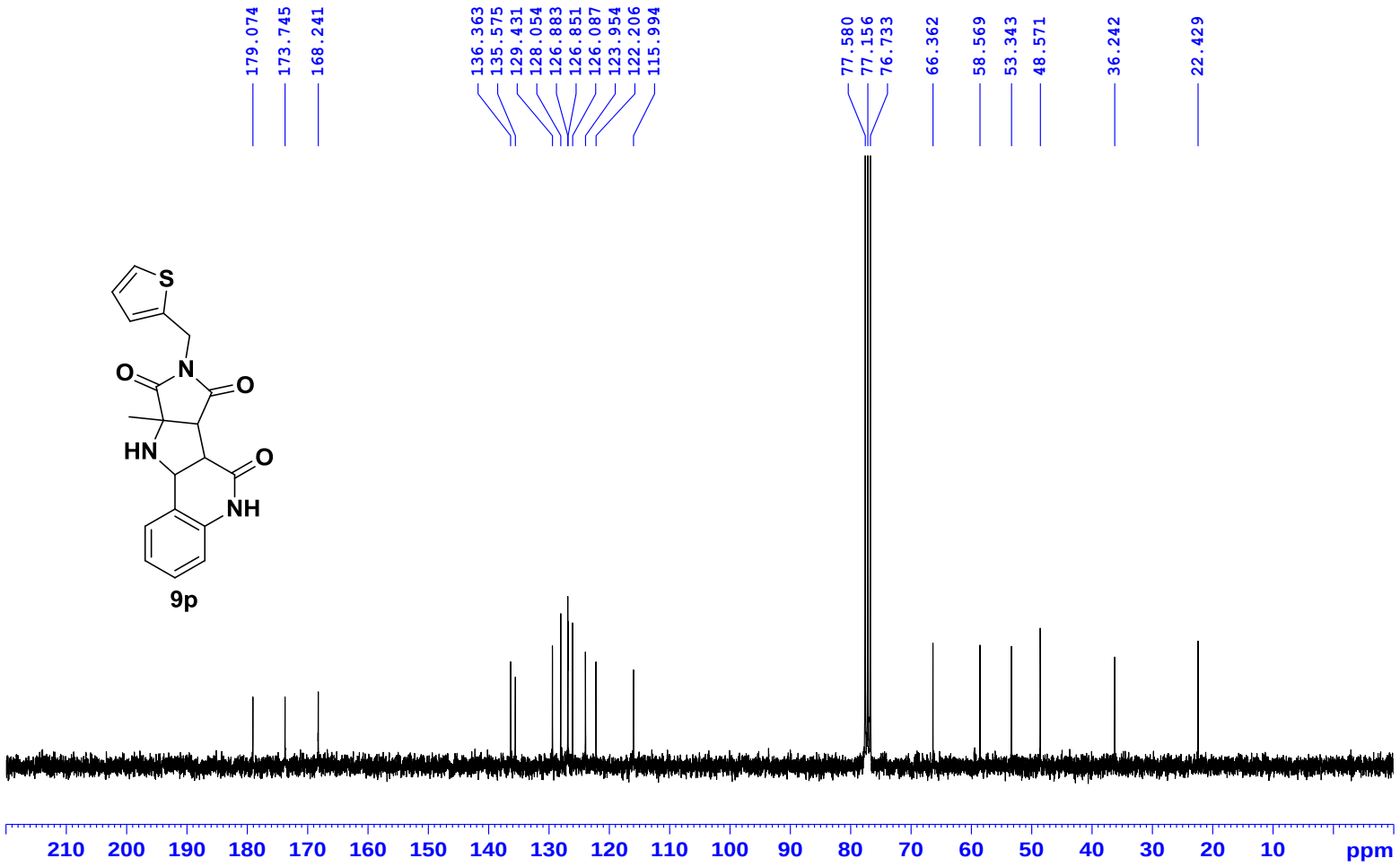
HRMS (EI) of compound **9o**



IR spectrum of compound of **9o**



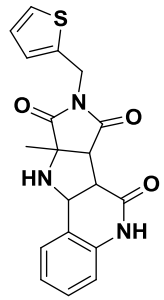
^{1}H NMR spectrum (400 MHz) of compound **9p** in $CDCl_3$



^{13}C NMR spectrum (101 MHz) of compound **9p** in $CDCl_3$

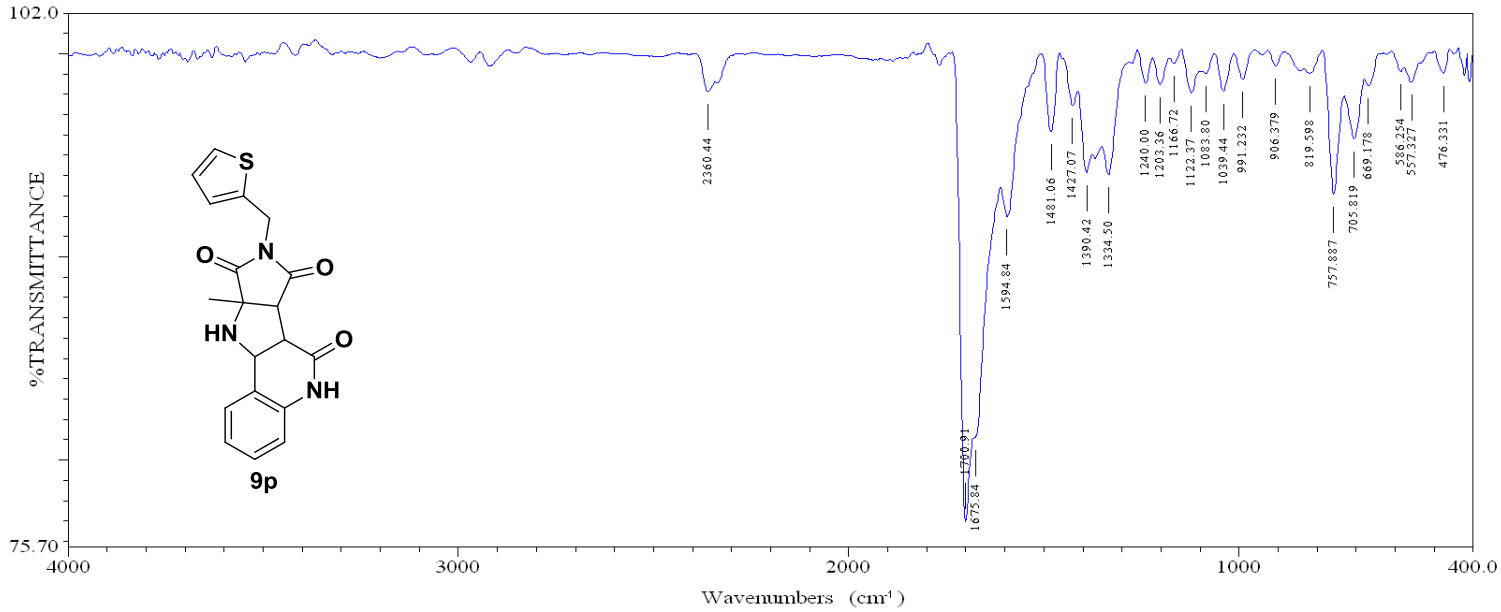
LIST: hei2530(leeutel16)-c2 29-Sep-11 REG : 02:40.3 #9
Samp: Start : 15:40:54 2365
Mode: EI +VE +LMR ESCAN (EXP) UP HR NRM Inlet :
Oper:
Limt: (0) . . . .
: (394) C19.H44.S.N3.O3
Peak: 1000.00 mmu R+D: -2.0 > 60.0
Data: +/463>901 (CMASS : converted |CMASS : converted |CMASS : conve

Mass	Intensity	%RA	Flags	(mmu) Delta	R+D	Composition
367.0998	5086	72.54	#?	-0.7	13.0	C19.H17.S.N3.O3

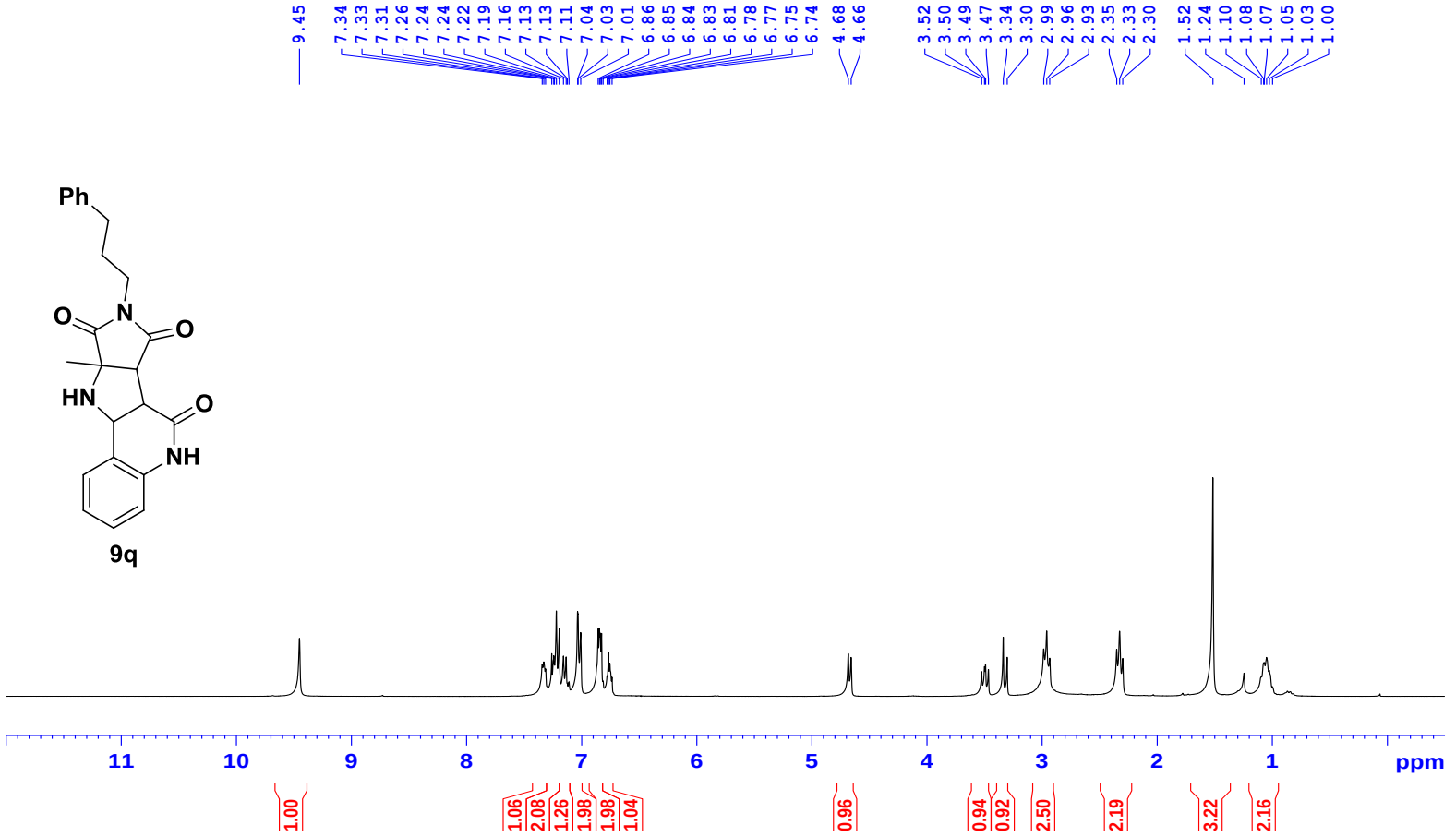


9p

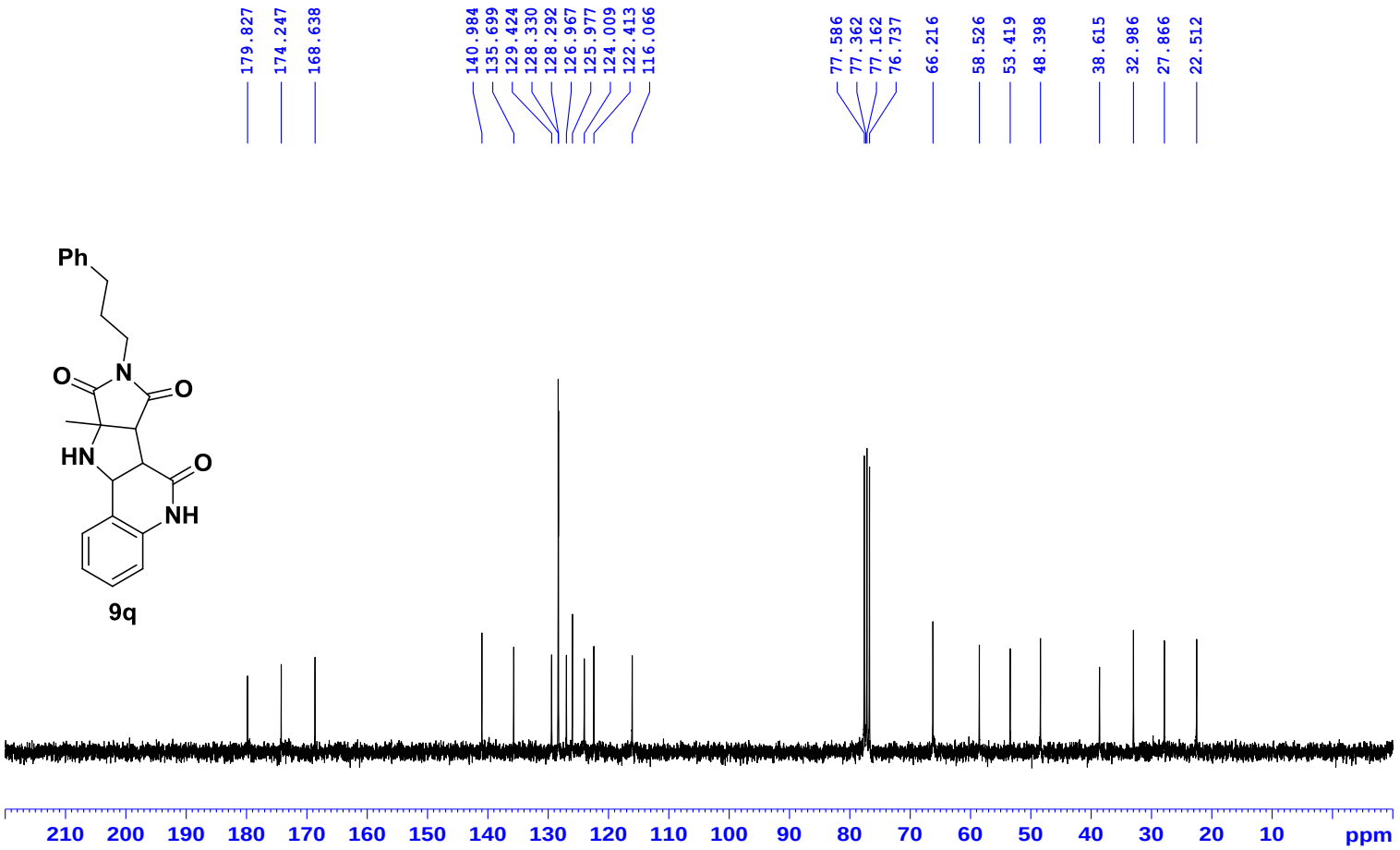
HRMS (EI) of compound **9p**



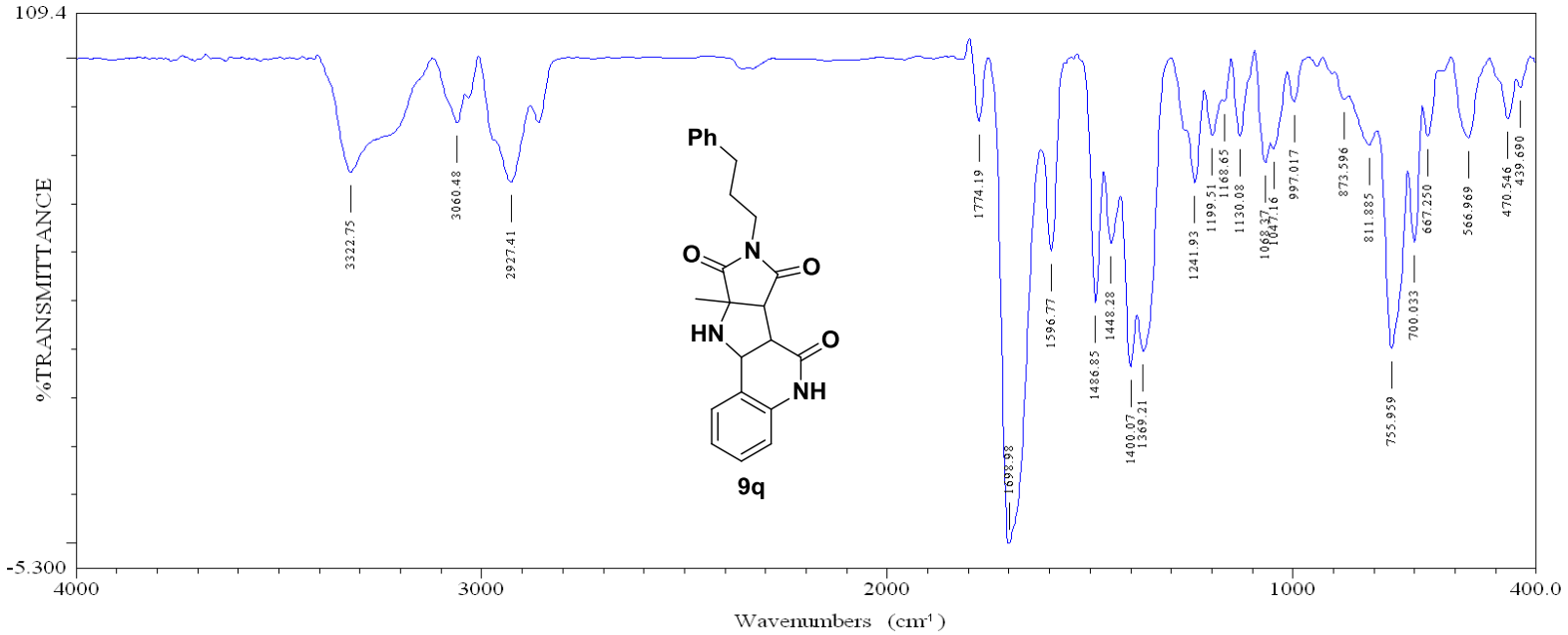
IR spectrum of **9p**



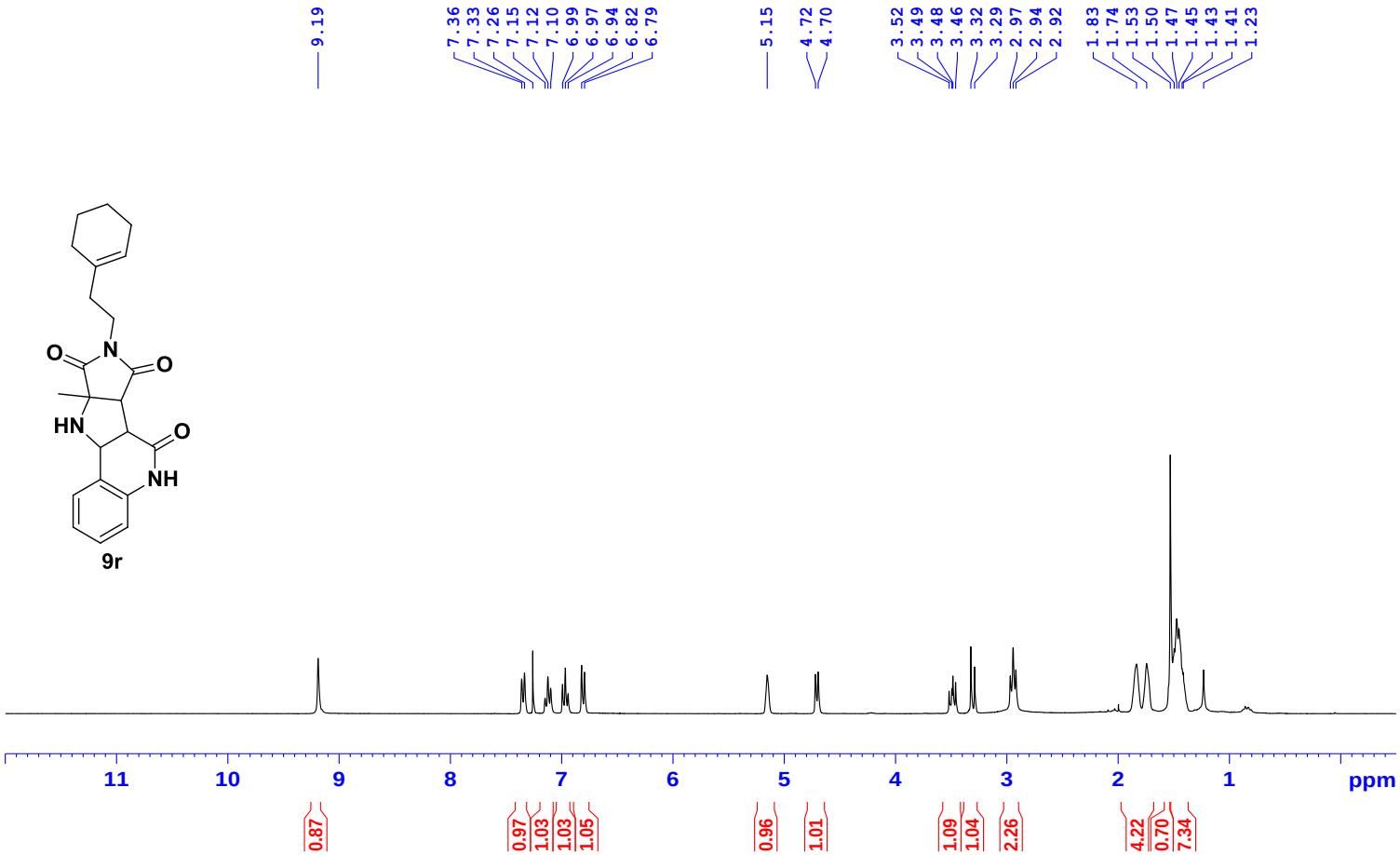
^{1}H NMR spectrum (400 MHz) of compound **9q** in $CDCl_3$



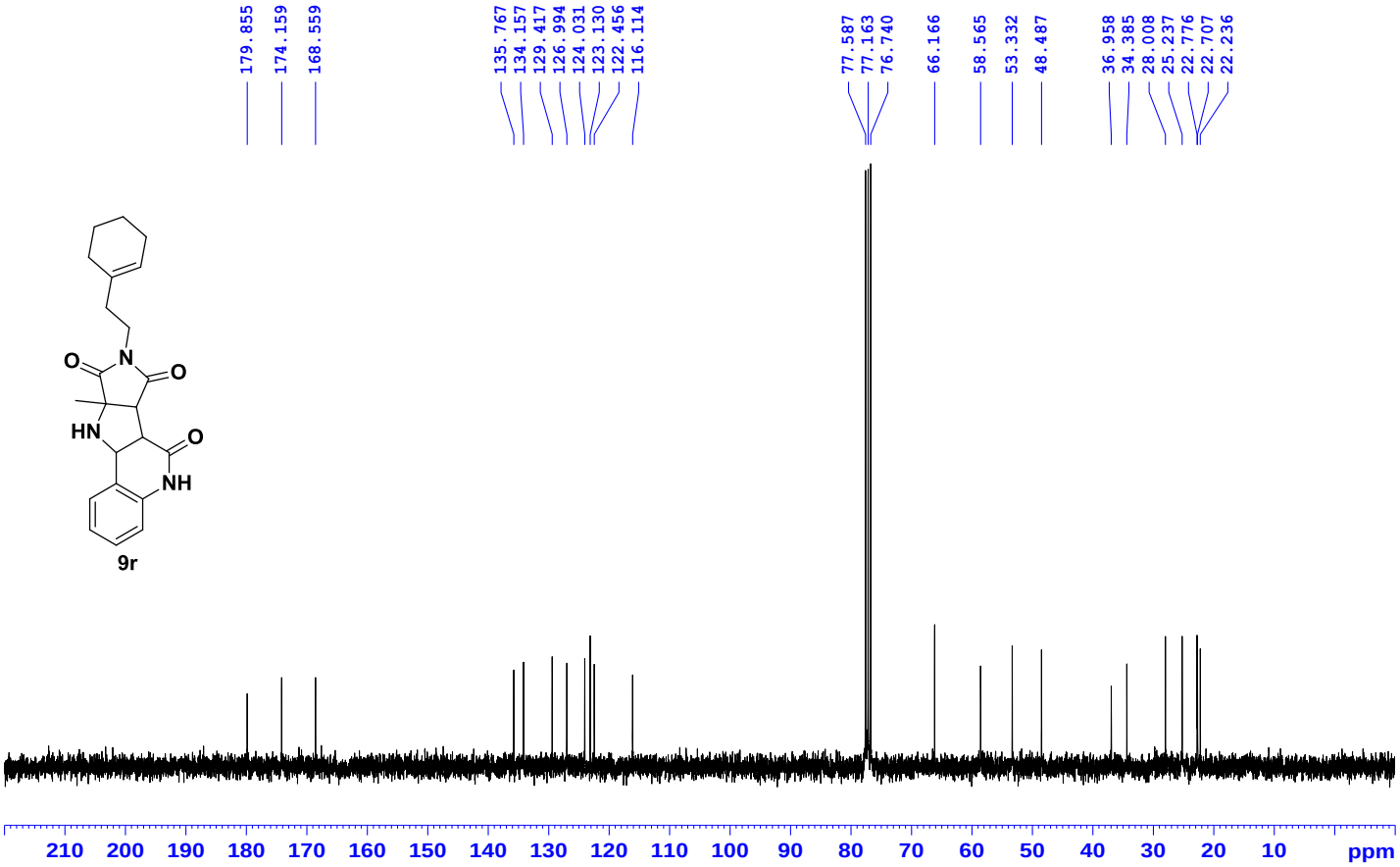
^{13}C NMR spectrum (101 MHz) of compound **9q** in $CDCl_3$



IR spectrum of **9q**



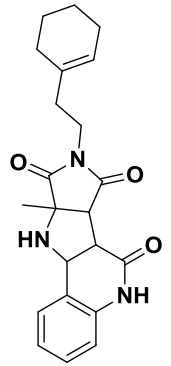
^{1}H NMR spectrum (400 MHz) of compound **9r** in $CDCl_3$



^{13}C NMR spectrum (101 MHz) of compound **9r** in $CDCl_3$

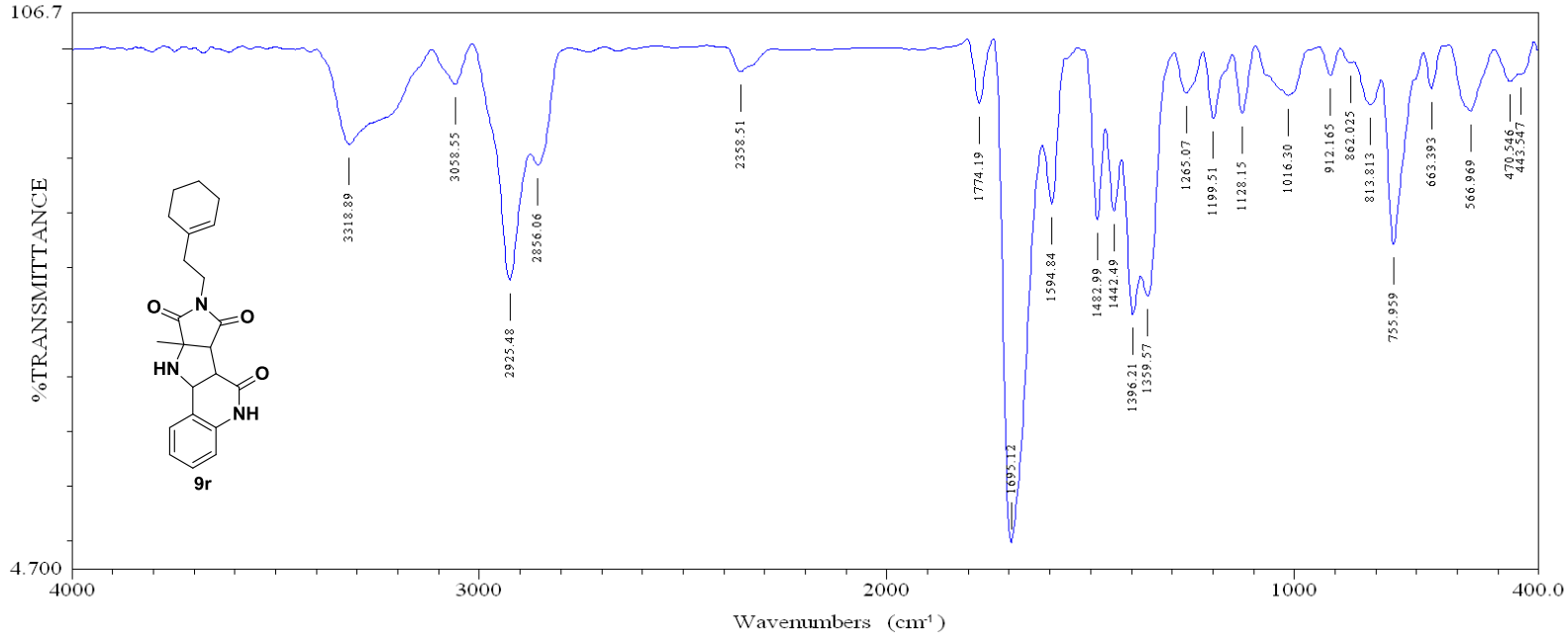
[Elemental Composition]
Data : 201111053 Date : 22-Oct-2011 17:49
Sample: leeut-El18
Note : -
Inlet : Direct Ion Mode : EI+
RT : 0.78 min Scan#: 32
Elements : C 23/0, H 27/0, O 5/0, N 3/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.1 - 36.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
379.1899	60.7	+0.8 / +0.3	12.0	C 22 H 25 O 3 N 3

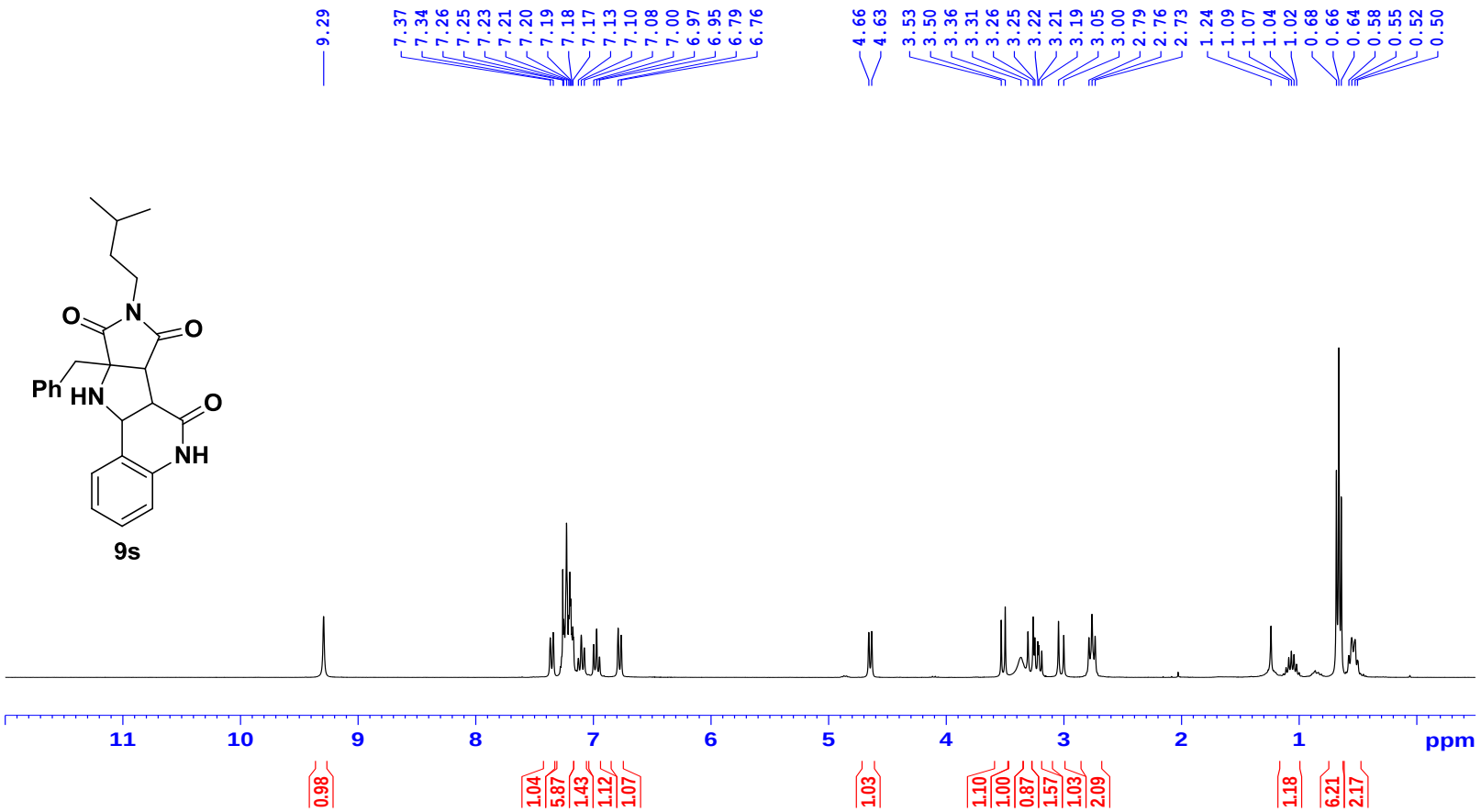


9r

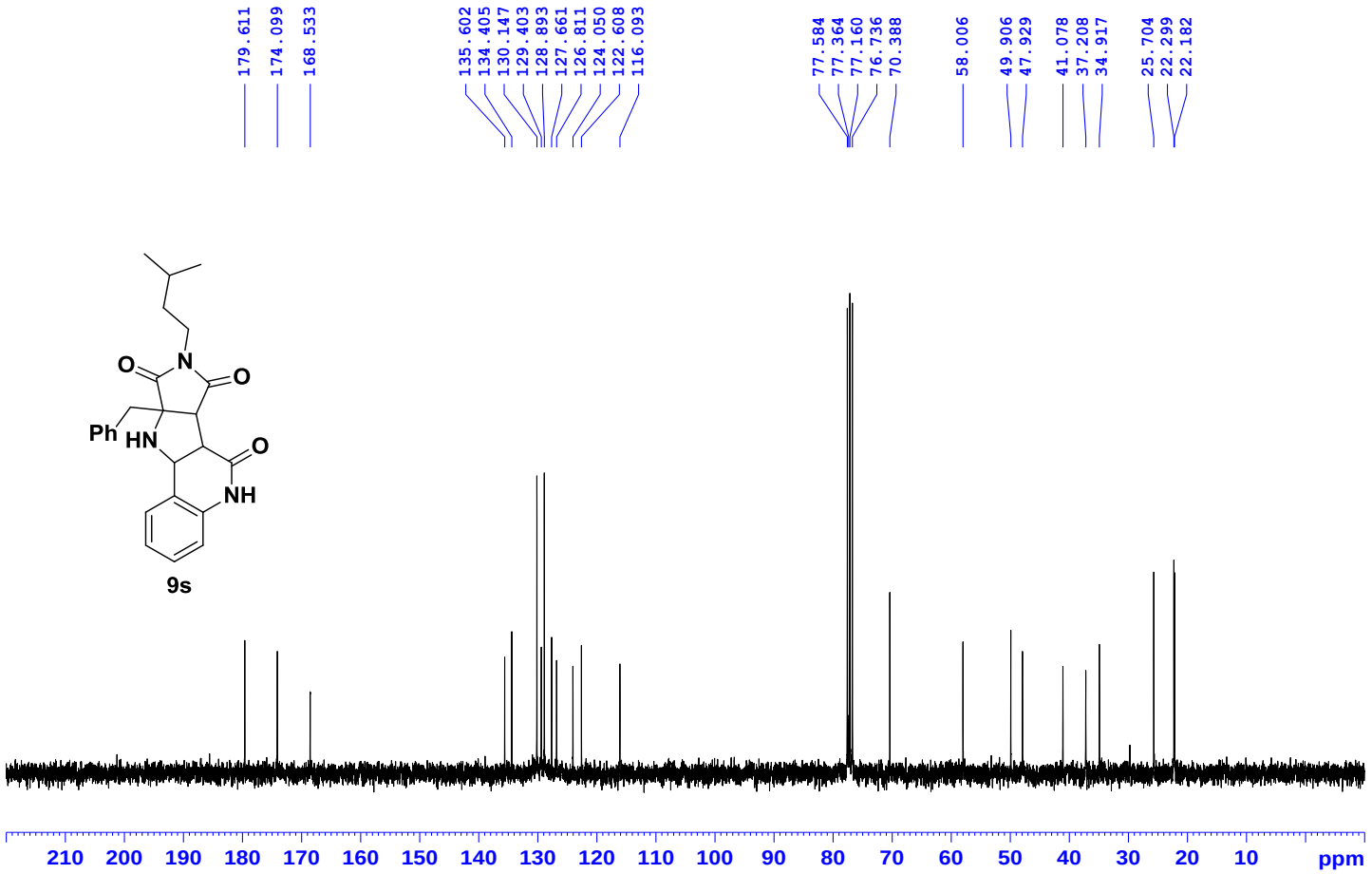
HRMS (EI) of compound **9r**



IR spectrum of **9r**



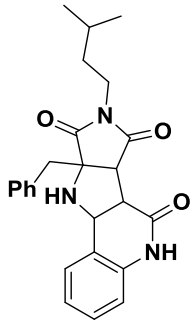
^{1}H NMR spectrum (400 MHz) of compound **9s** in $CDCl_3$



^{13}C NMR spectrum (101 MHz) of compound **9s** in $CDCl_3$

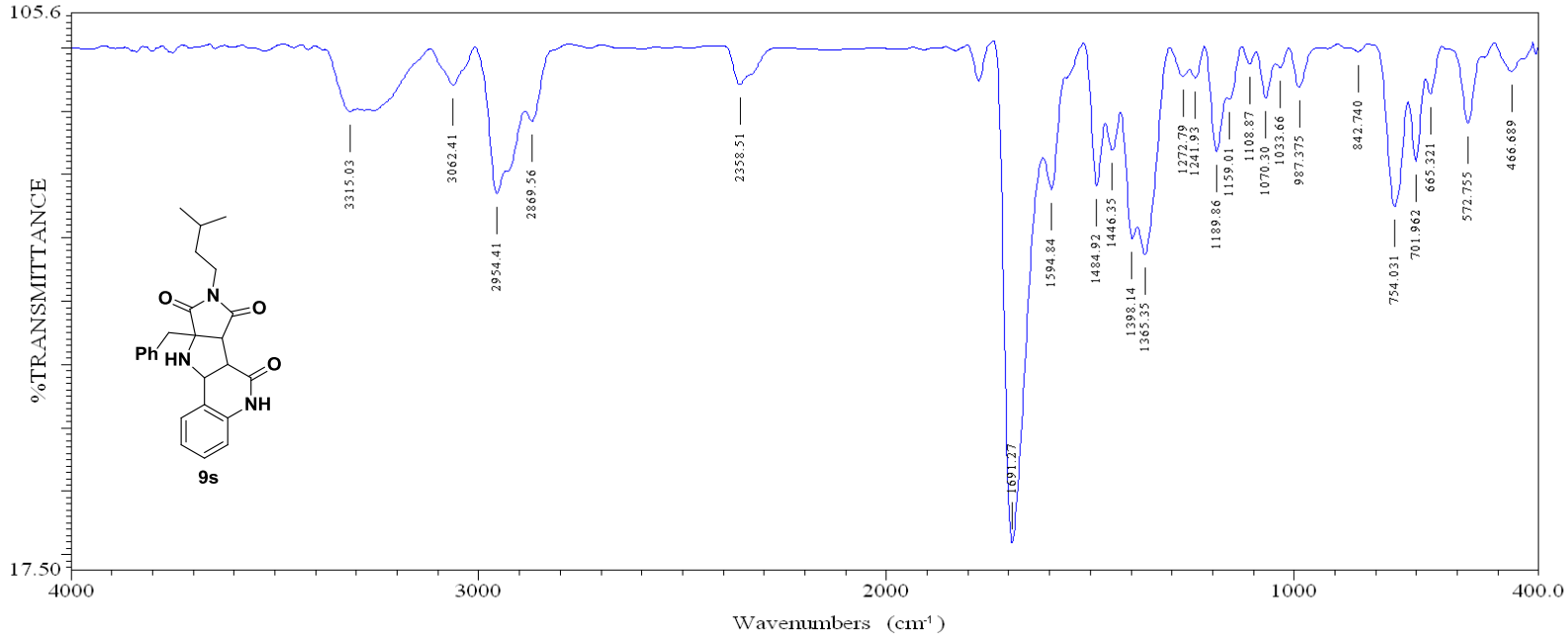
[Elemental Composition]
Data : 201111054 Date : 22-Oct-2011 17:54
Sample: leeut-El19
Note : -
Inlet : Direct Ion Mode : EI+
RT : 0.70 min Scan#: 32
Elements : C 25/0, H 27/0, O 5/0, N 3/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.1 - 36.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
417.2050	36.6	-0.5 / -0.2	14.0	C 25 H 27 O 3 N 3

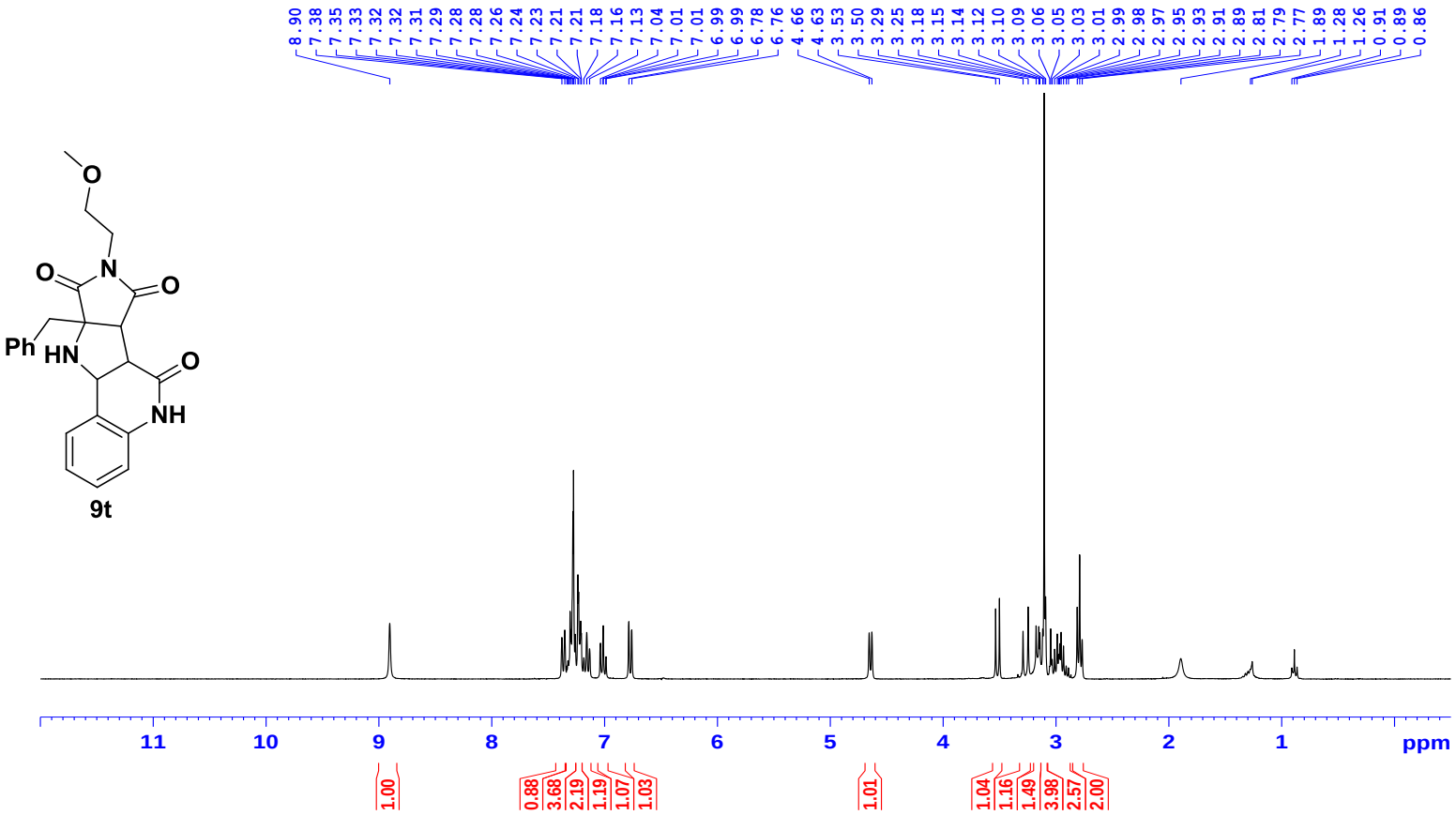


9s

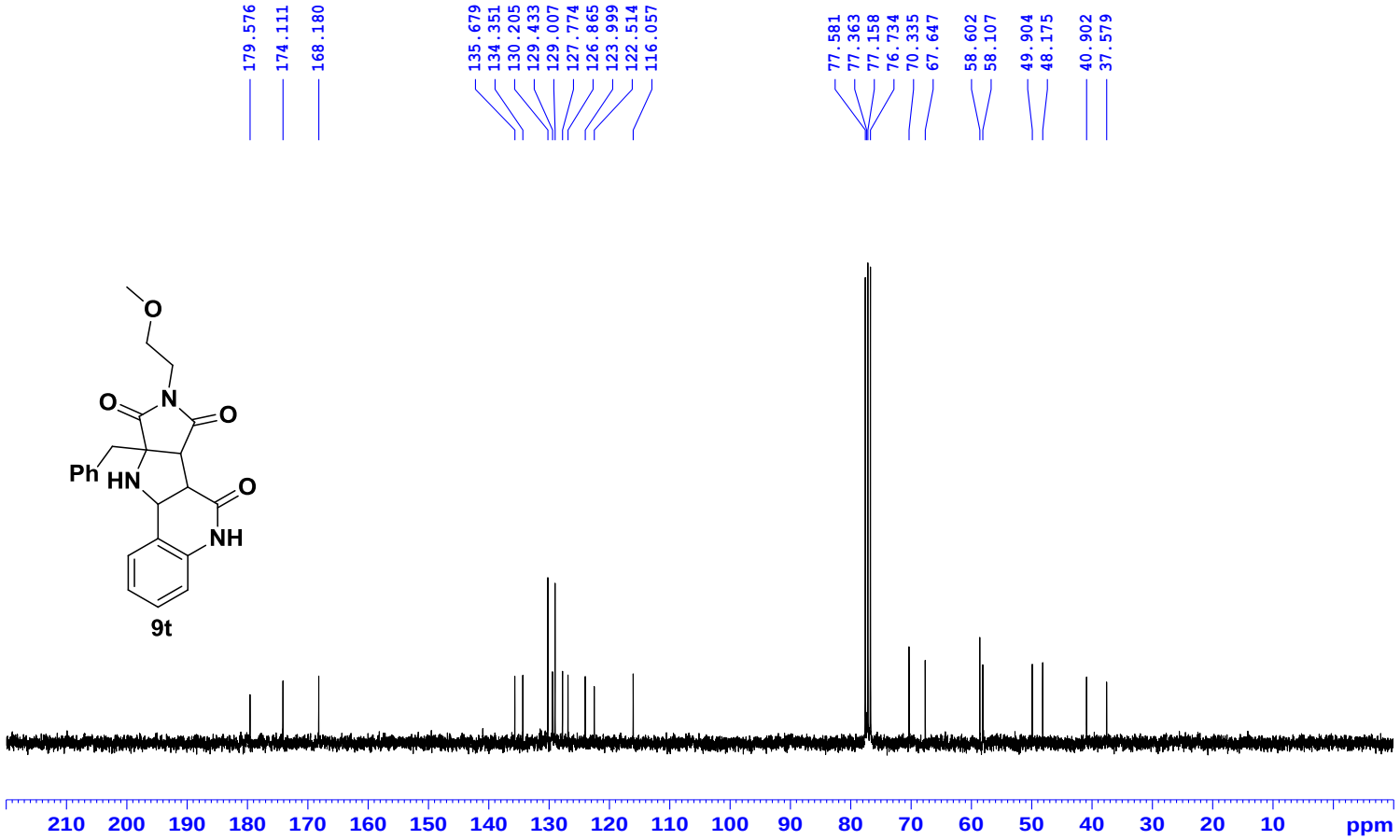
HRMS (EI) of compound **9s**



IR spectrum of compound **9s**



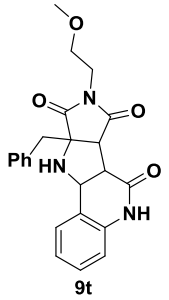
^{1}H NMR spectrum (400 MHz) of compound **9t** in $CDCl_3$



^{13}C NMR spectrum (101 MHz) of compound **9t** in $CDCl_3$

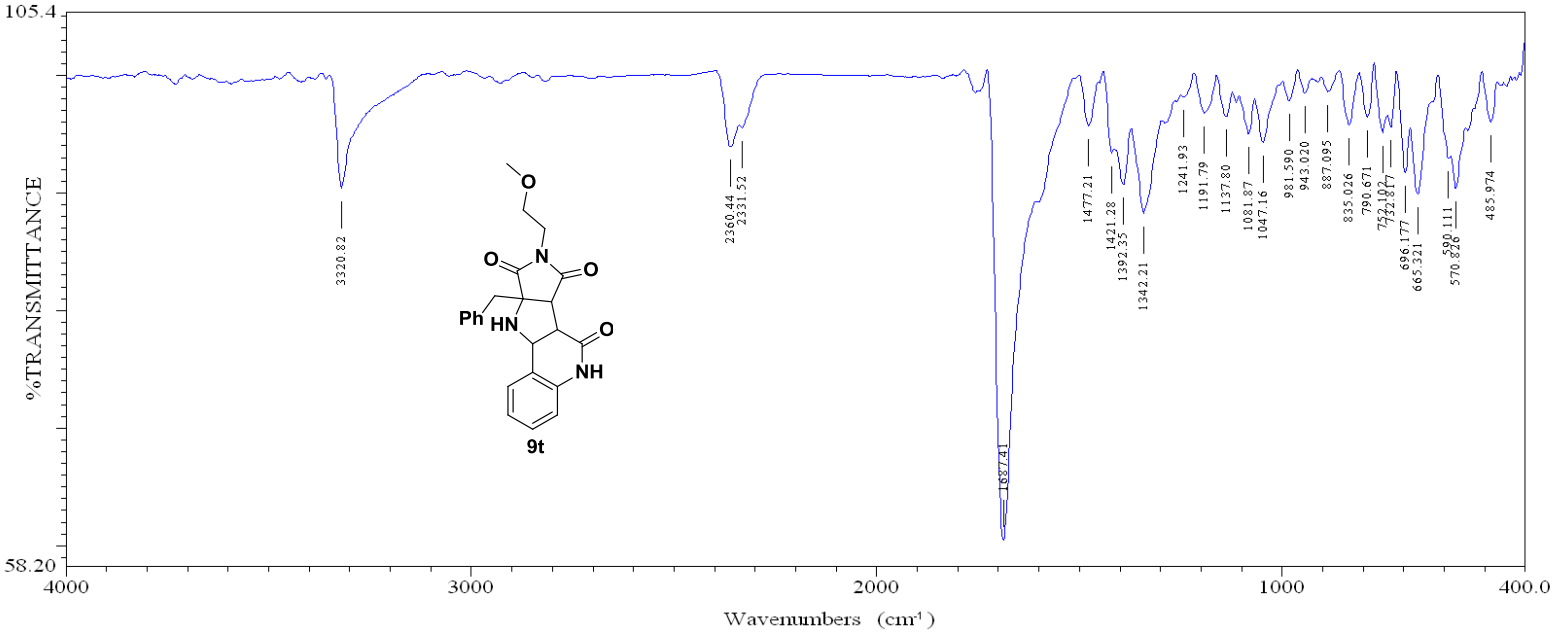
[Elemental Composition]
Data : 201111055 Date : 22-Oct-2011 17:58
Sample: leeut-El20
Note : -
Inlet : Direct Ion Mode : EI+
RT : 0.85 min Scan#: 42
Elements : C 23/0, H 27/0, O 5/0, N 3/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.1 - 36.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
405.1685	100.0	-0.9 / -0.4	14.0	C 23 H 23 O 4 N 3

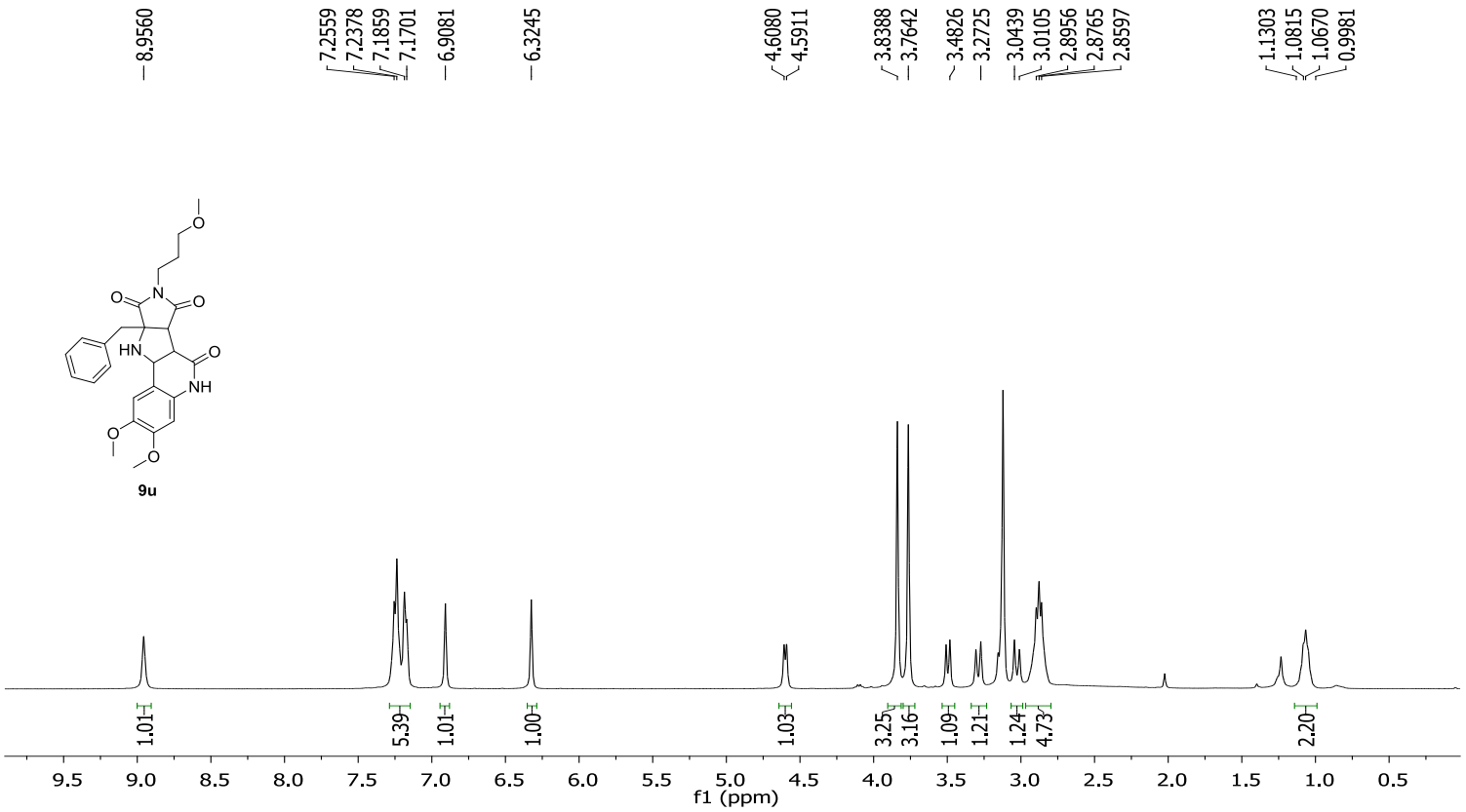


9t

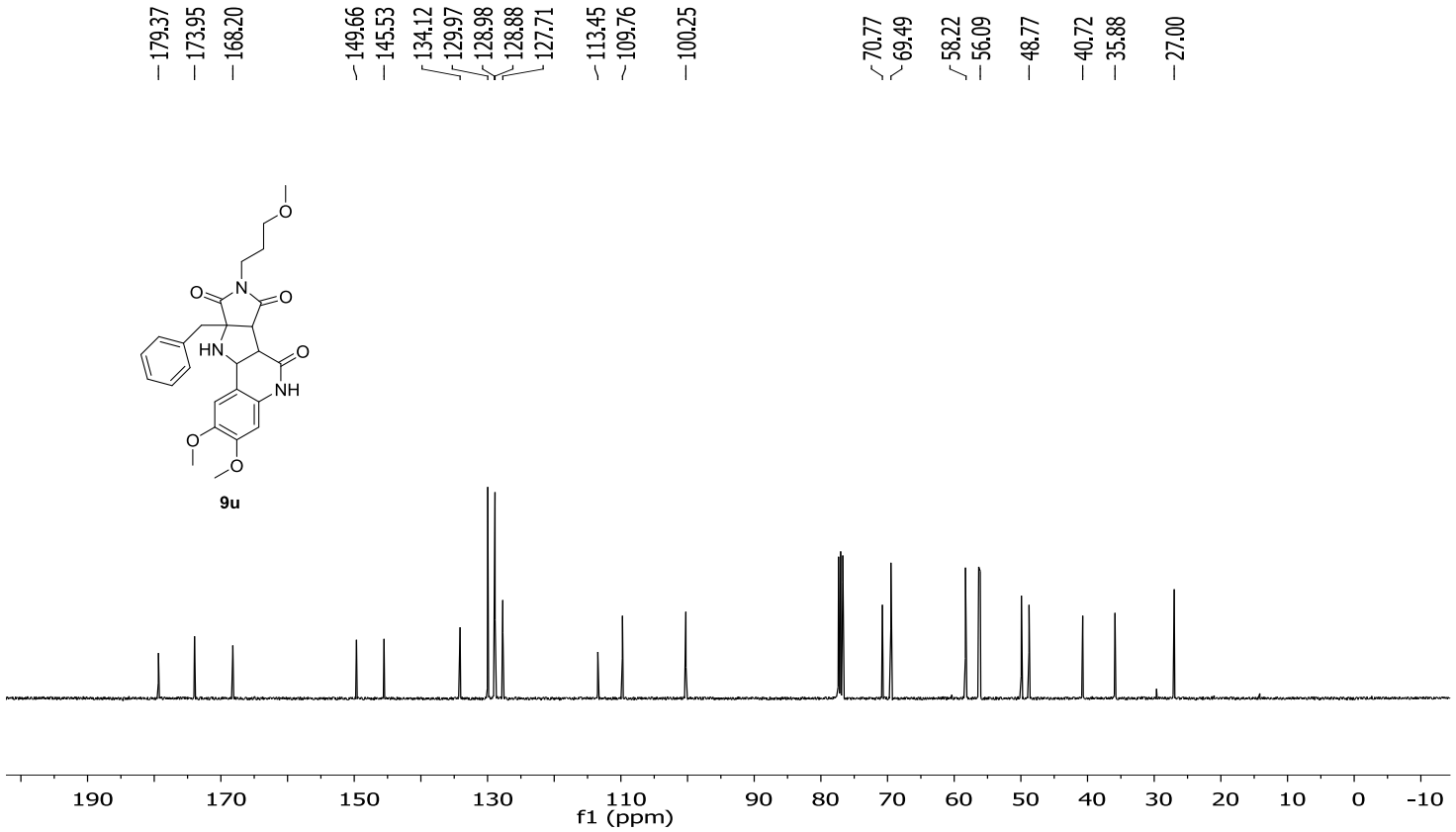
HRMS (EI) of compound **9t**



IR spectrum of compound **9t**



^{1}H NMR of compound **9u** in $CDCl_3$

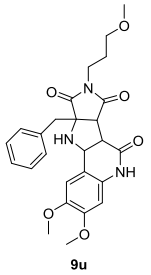


^{13}C NMR of compound **9u** in $CDCl_3$

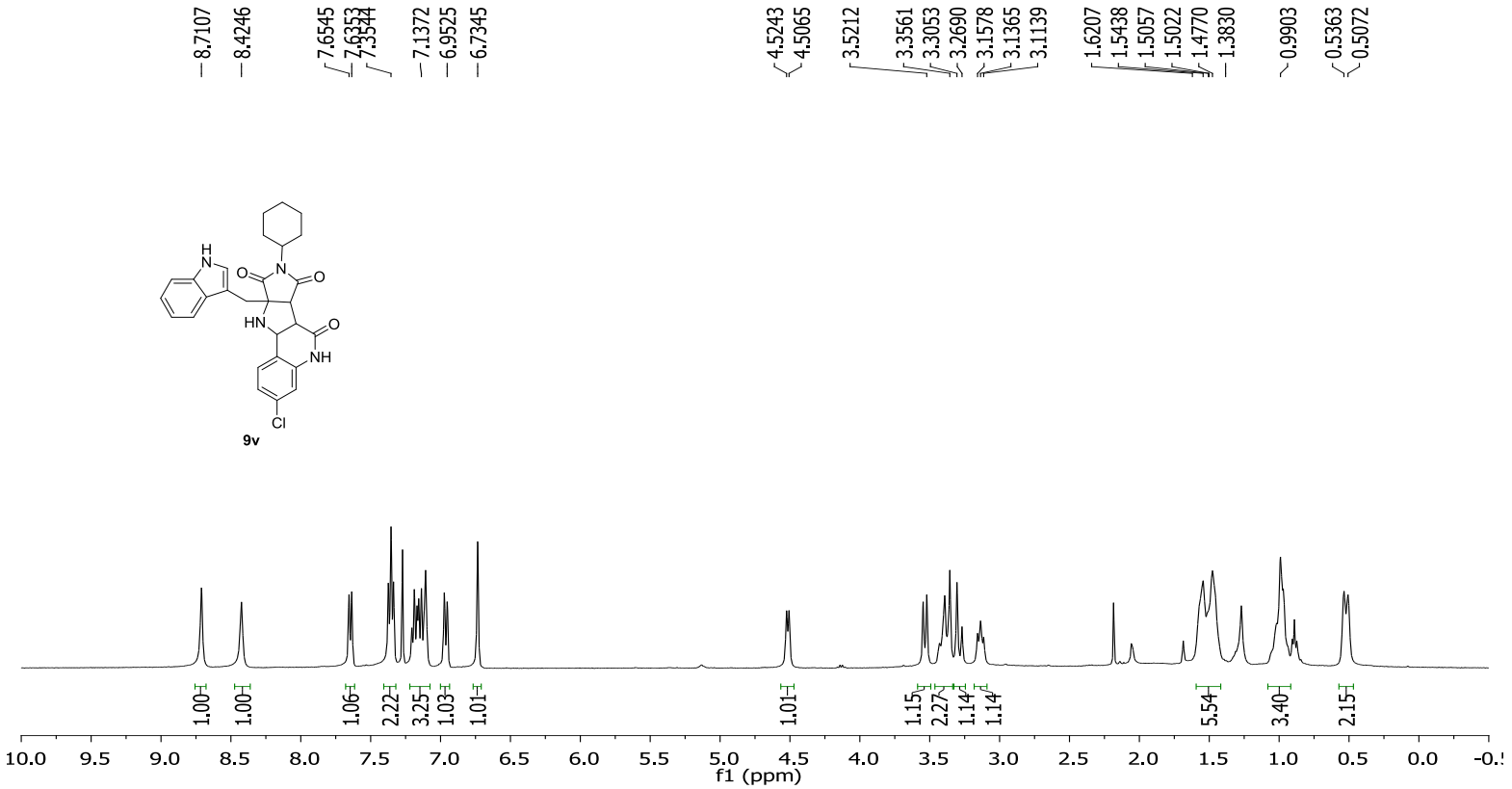
Display Report

Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdb	e¯ Conf	N-Rule	Adduct
480.2138	1	C26H30N3O6	480.2129	-1.9	11.1	1	100.00	13.5	even	ok	M+H

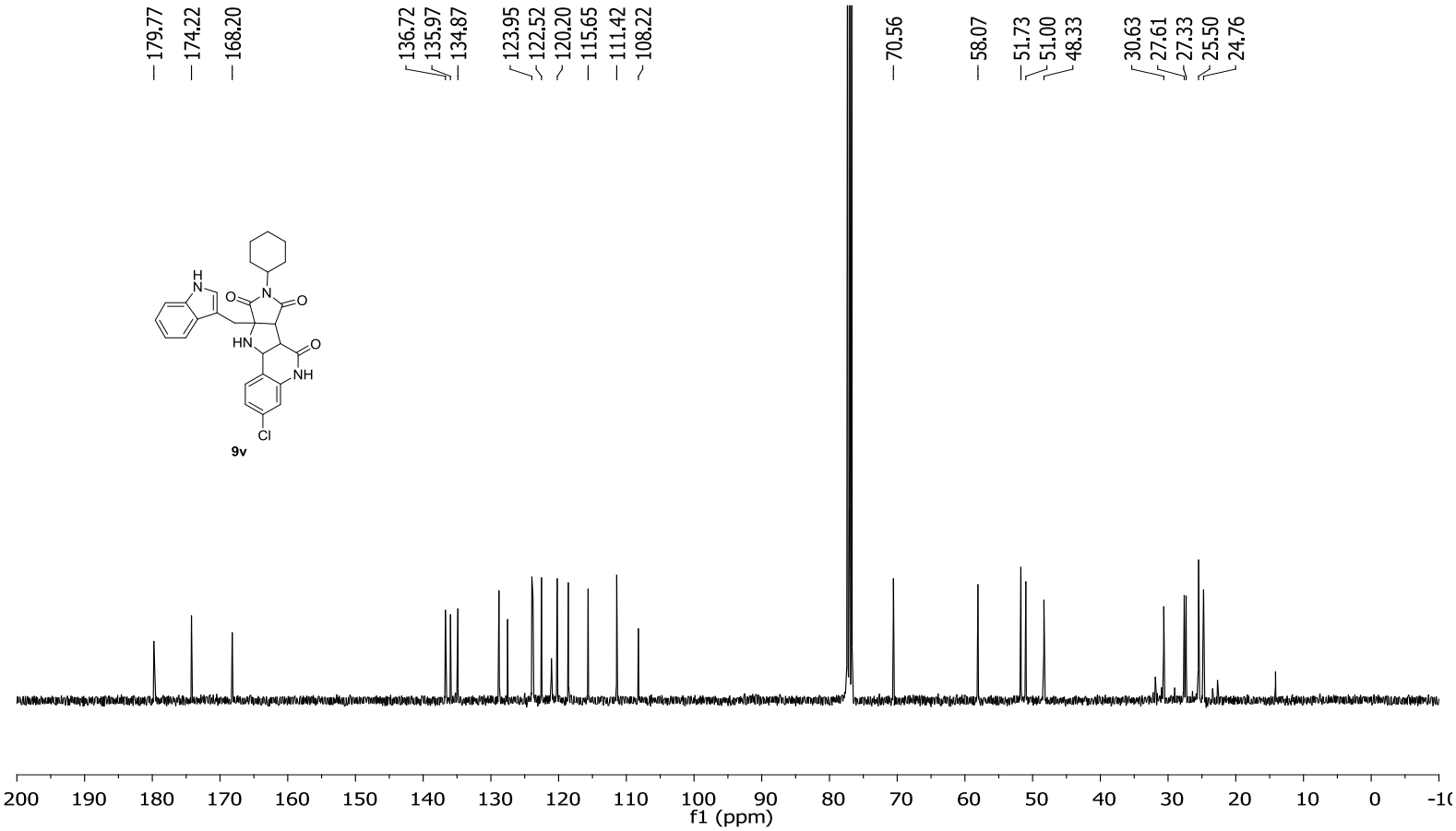
HRMS (ESI) of compound of **9u**



9u



^{1}H NMR of compound **9v** in $CDCl_3$

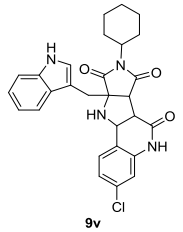


^{13}C NMR of compound **9v** in $CDCl_3$

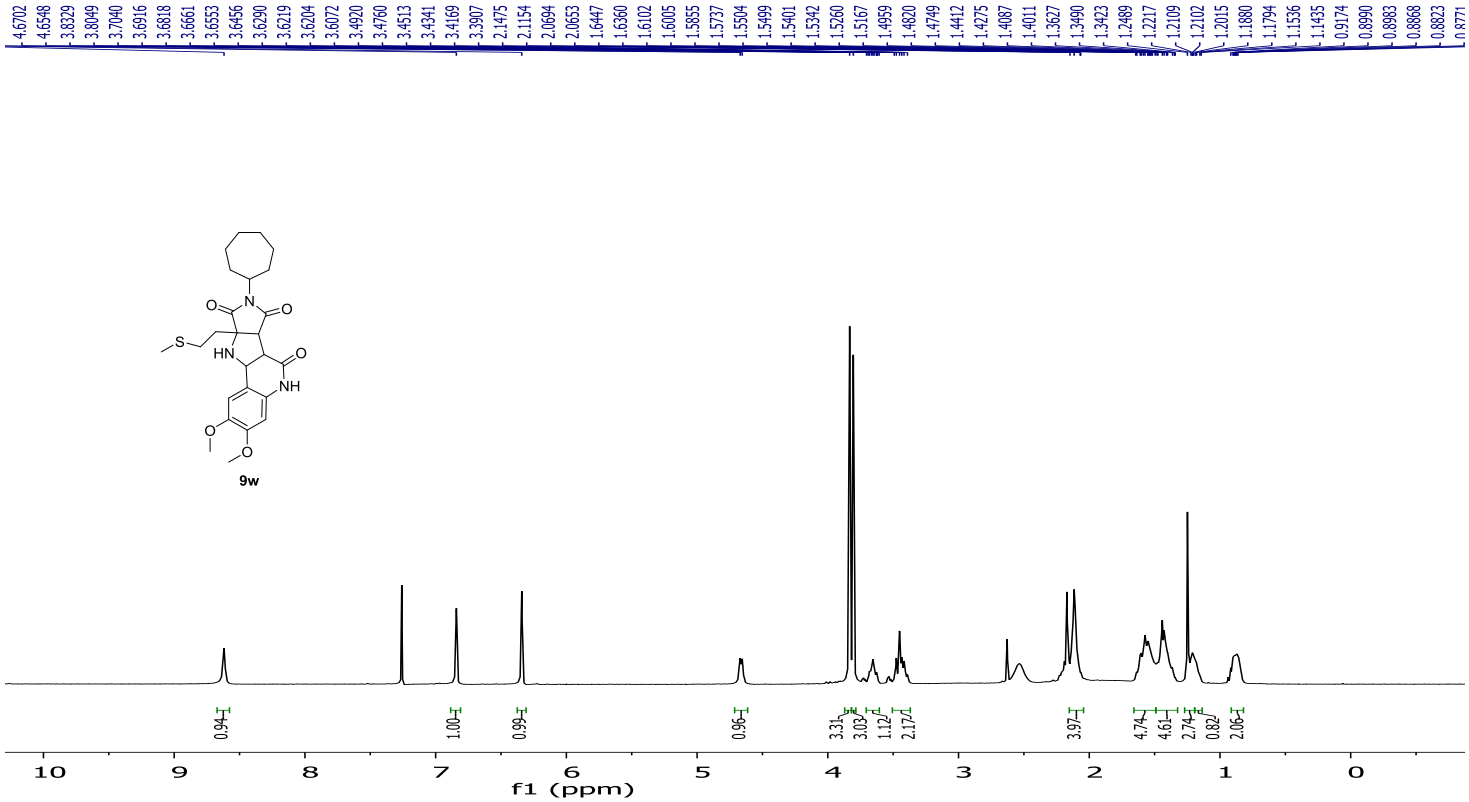
Display Report

Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdb	e¯ Conf	N-Rule	Adduct	Adduct
503.1850	1	C28H28ClN4O3	503.1844	1.1	7.9	1	100.00	16.5	even	ok	M+H	M+H

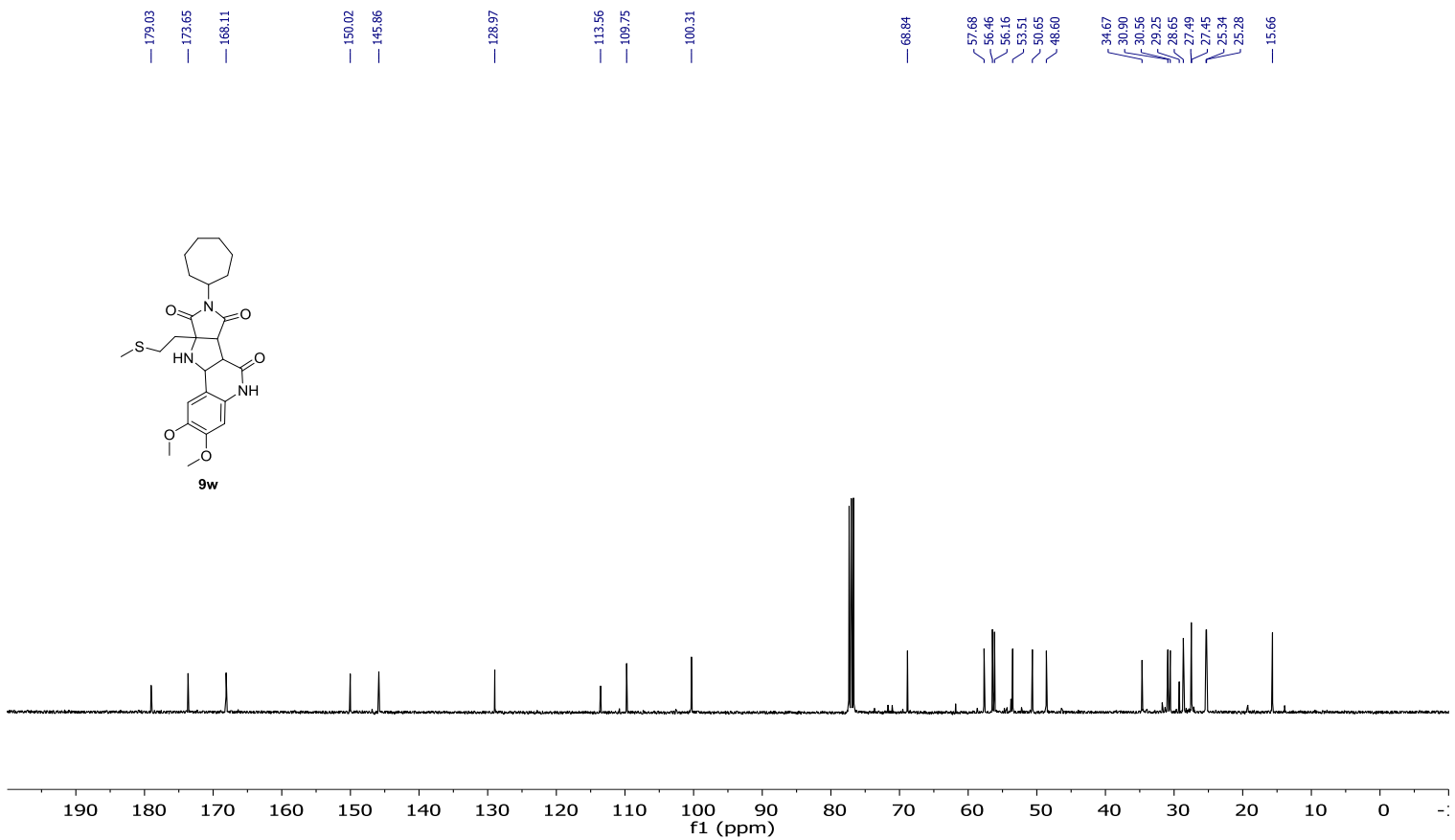
HRMS (ESI) of compound **9v**



9v



^{1}H NMR of compound **9w** in $CDCl_3$

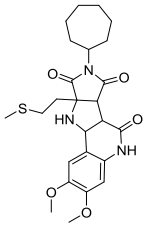


^{13}C NMR of compound **9w** in $CDCl_3$

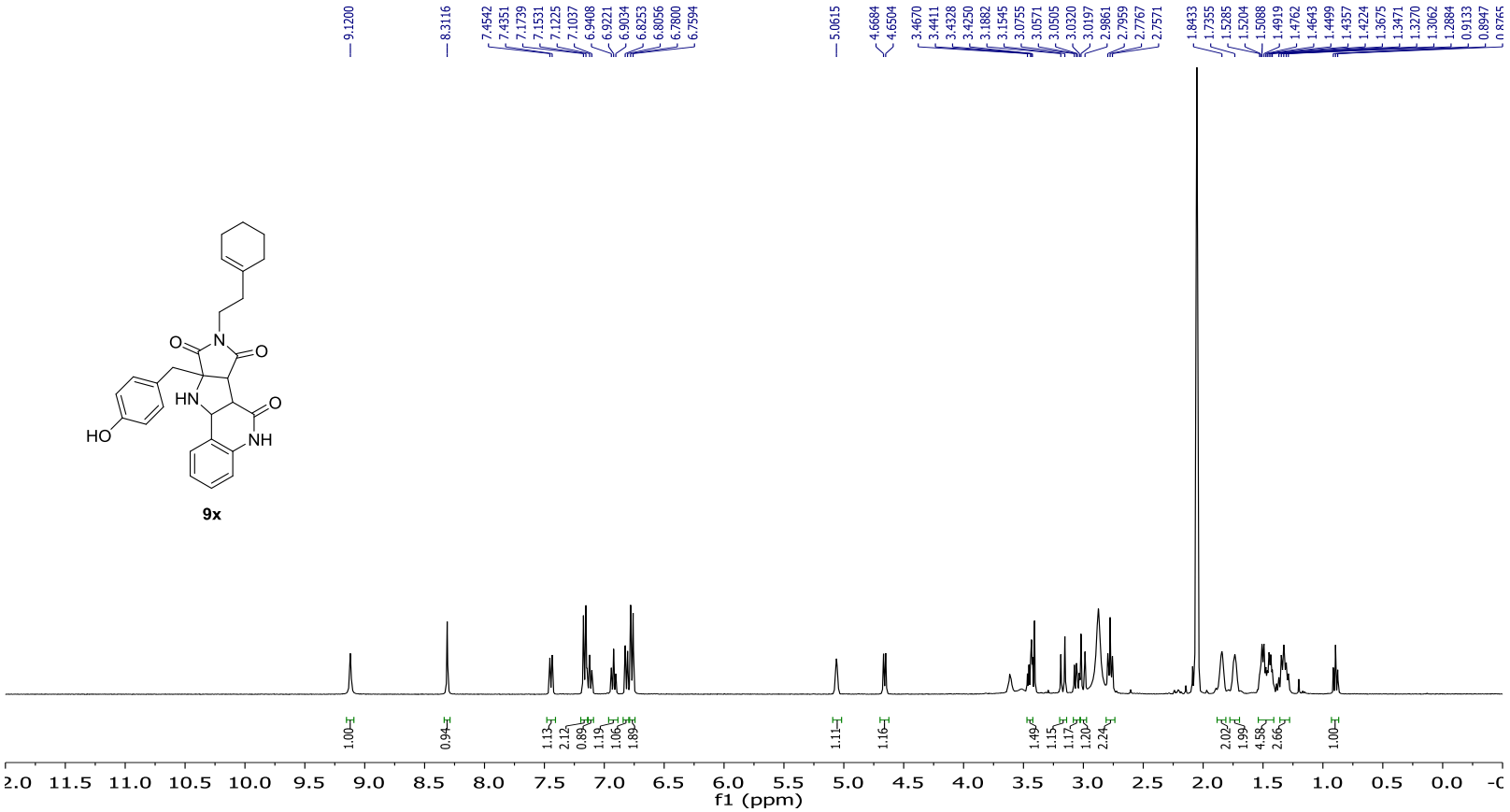
Display Report

Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdb	e¯ Conf	N-Rule	Adduct
488.2222	1	C25H34N3O5S	488.2214	-1.8	28.2	1	100.00	10.5	even	ok	M+H

HRMS (ESI) of compound **9w**



9w



9x

^{1}H NMR (400 MHz) of compound **9x** in Acetone-d_6

^{13}C NMR (101 MHz) of compound **9x** in

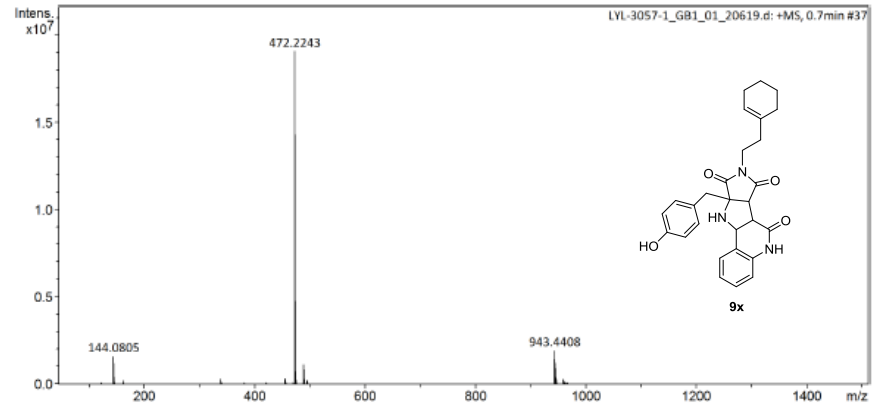
Display Report

Analysis Info

Analysis Name	D:\Data\nctu service\data\2020\20200526\LYL-3057-1_GB1_01_20619.d	Acquisition Date	5/26/2020 12:06:20 PM
Method	Small molecule.m	Operator	NCTU
Sample Name	LYL-3057-1	Instrument	impact HD 1819696.00164
Comment			

Acquisition Parameter

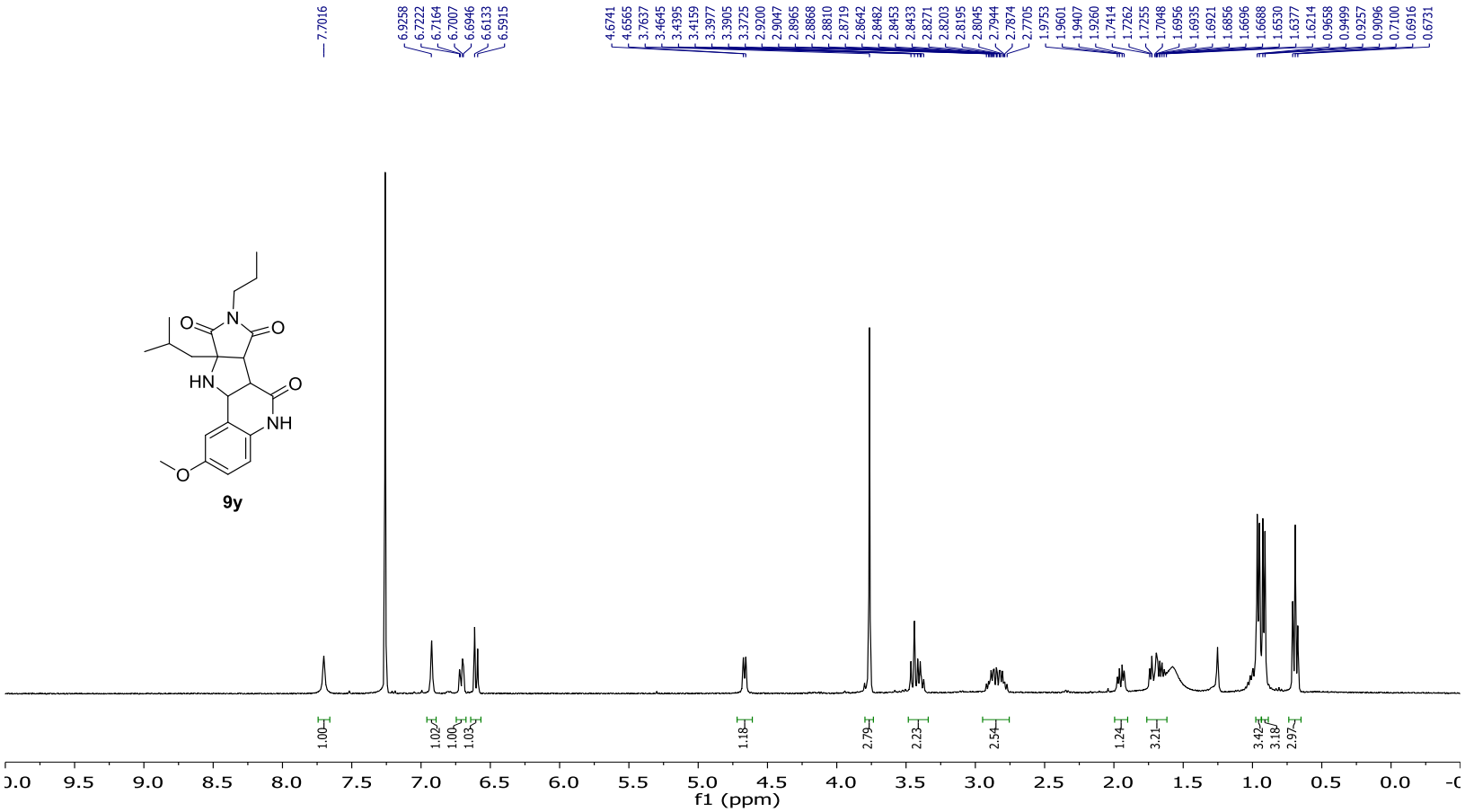
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



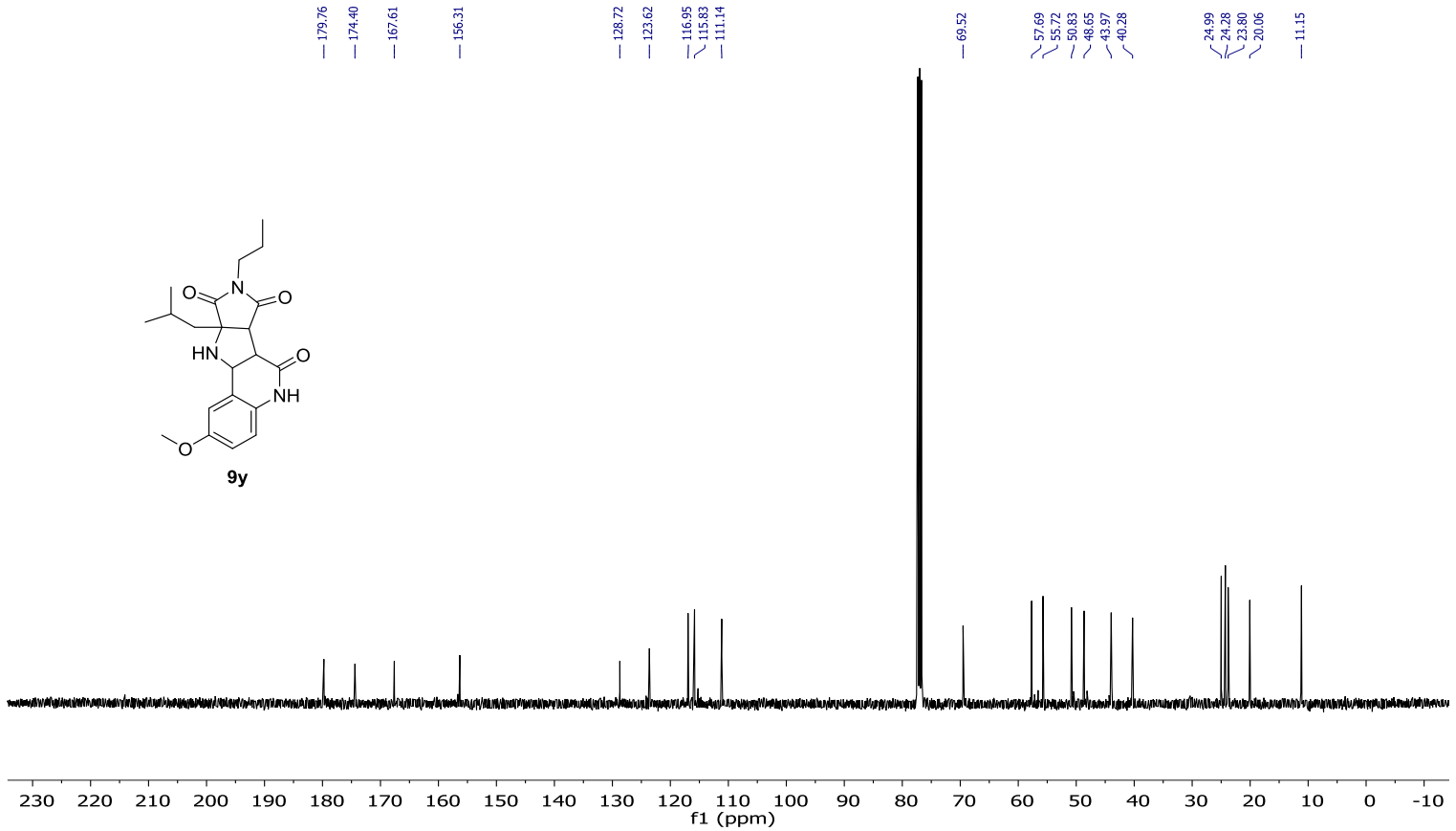
Display Report

Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdb	e⁻ Conf	N-Rule	Adduct
472.2243	1	C28H30N3O4	472.2231	-2.7	8.1	1	100.00	15.5	even	ok	M+H

HRMS (ESI) of compound **9x**



^{1}H NMR (400 MHz) of compound **9y** in $CDCl_3$



^{13}C NMR (101 MHz) of compound **9y** in $CDCl_3$

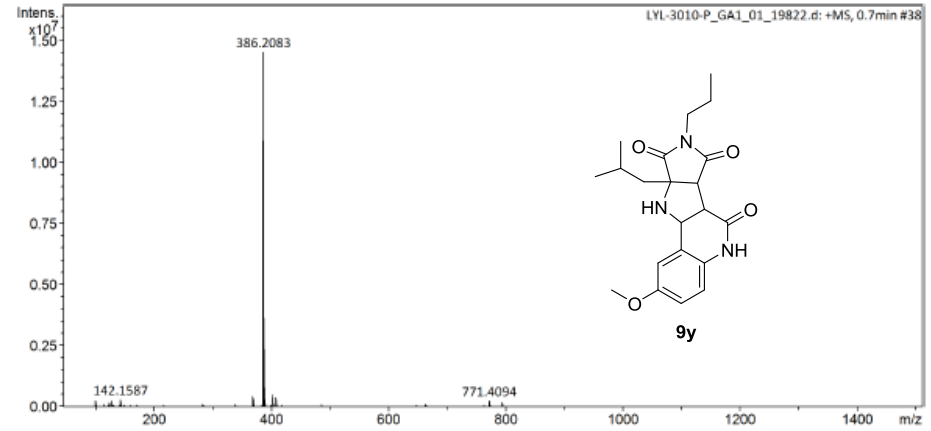
Display Report

Analysis Info

Analysis Name	D:\Data\nctu service\data\2020\20200221\LYL-3010-P_GA1_01_19822.d	Acquisition Date	2/21/2020 12:53:44 PM
Method	Small molecule.m	Operator	NCTU
Sample Name	LYL-3010-P	Instrument	impact HD 1819696.00164
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C

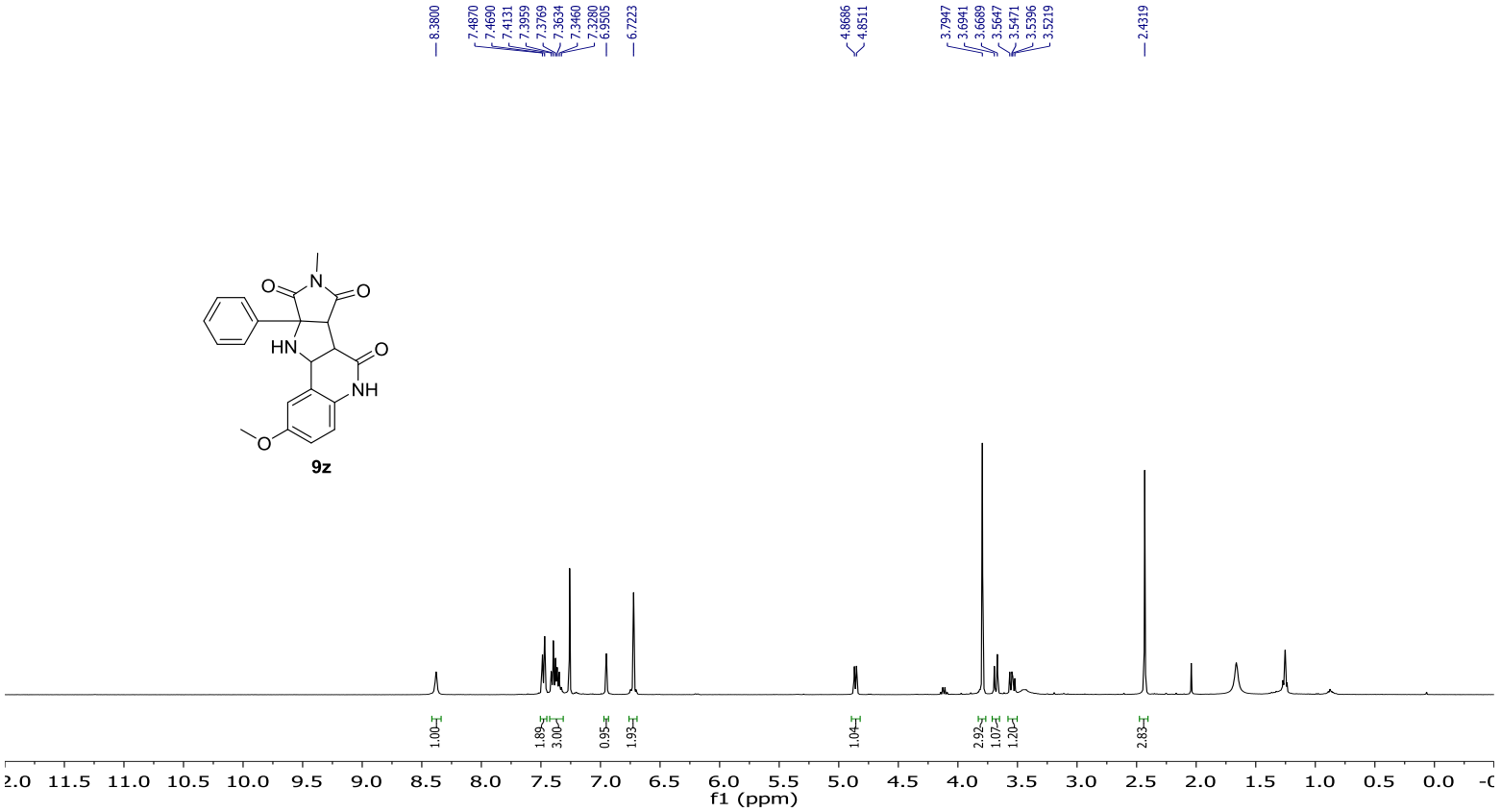


9y

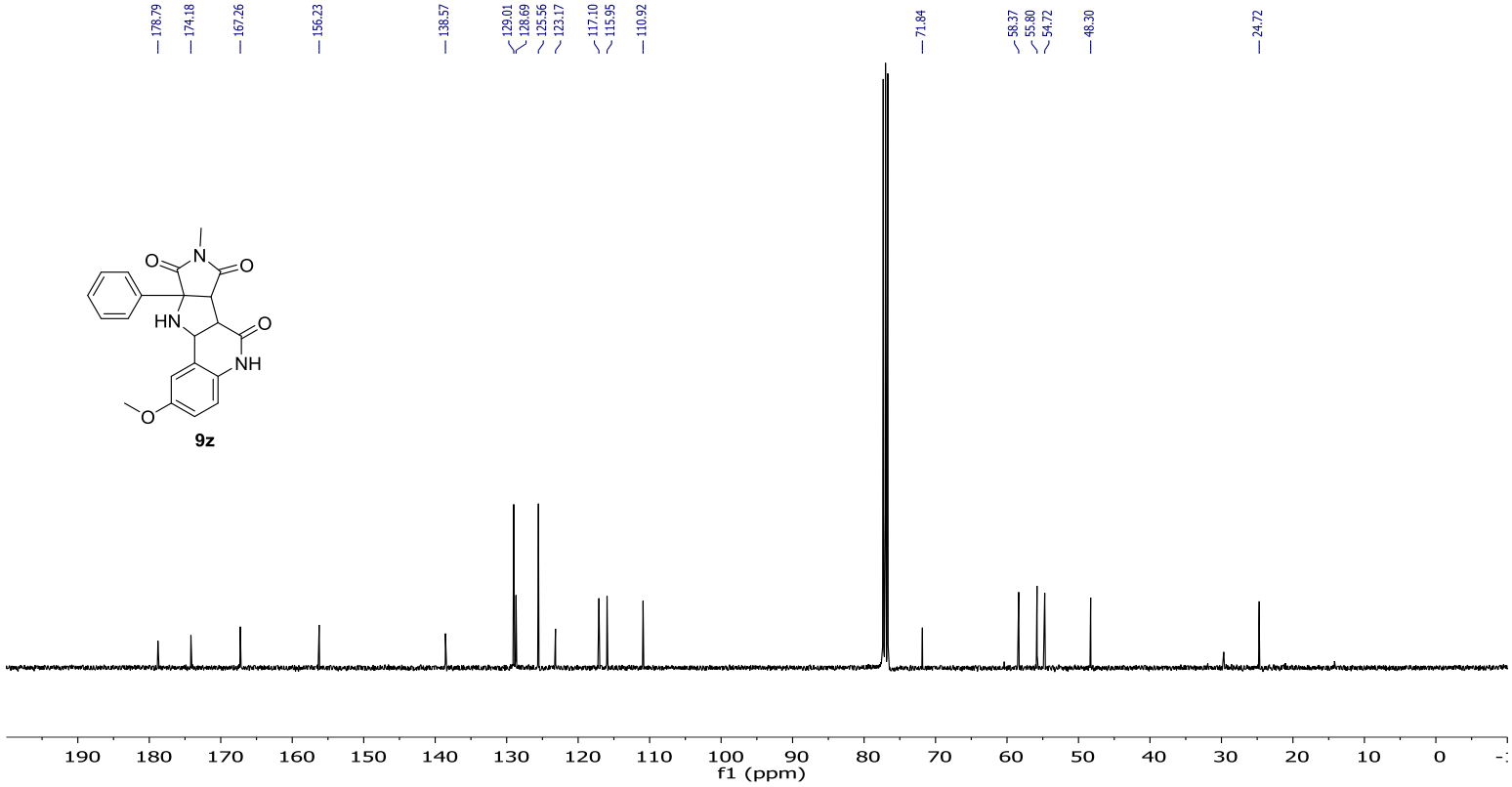
Display Report

Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdb	e¯ Conf	N-Rule	Adduct
386.2083	1	C21H28N3O4	386.2074	-2.1	13.8	1	100.00	9.5	even	ok	M+H

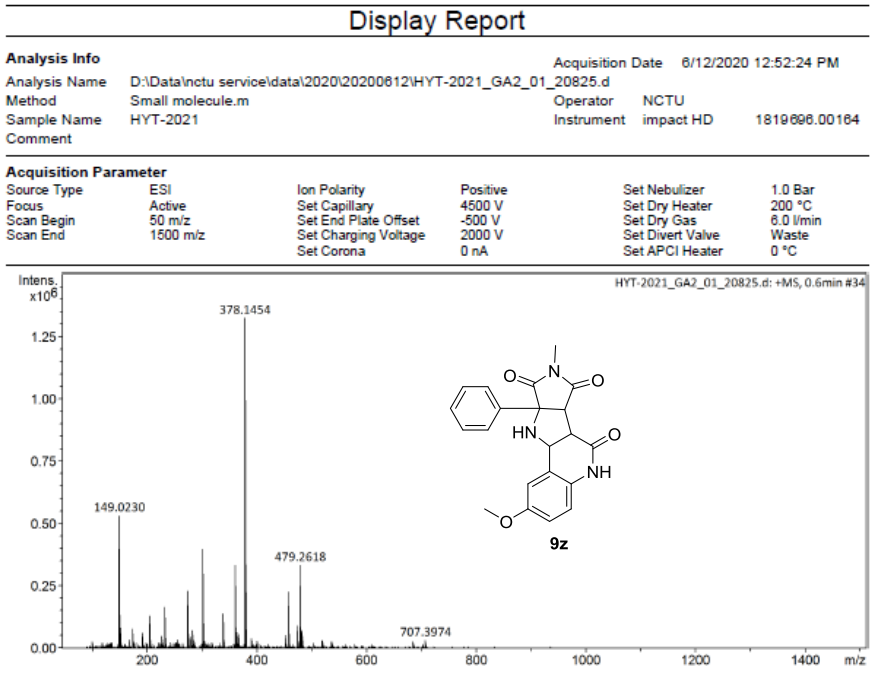
HRMS (ESI) of compound **9y**



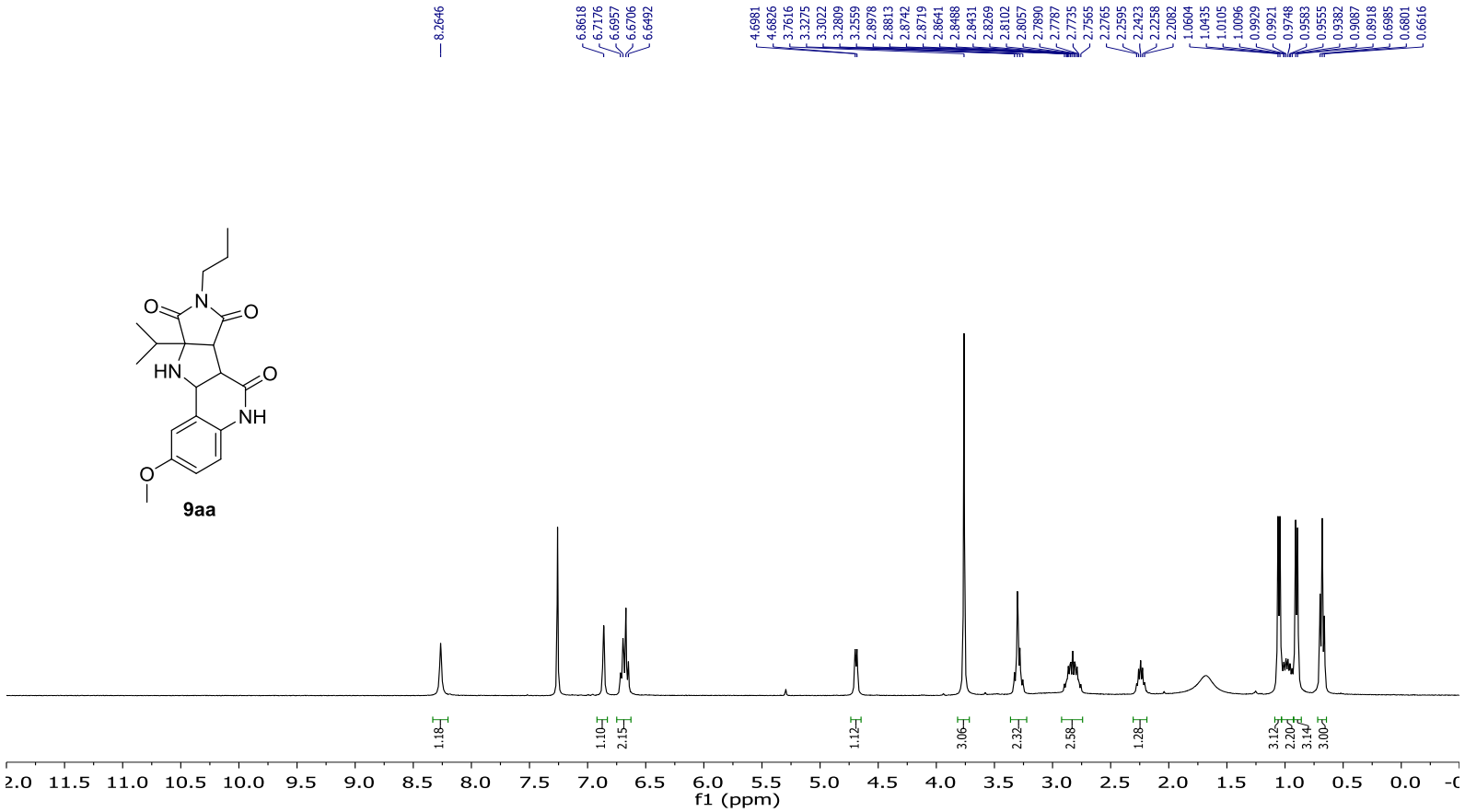
^{1}H NMR (400 MHz) of compound **9z** in $CDCl_3$



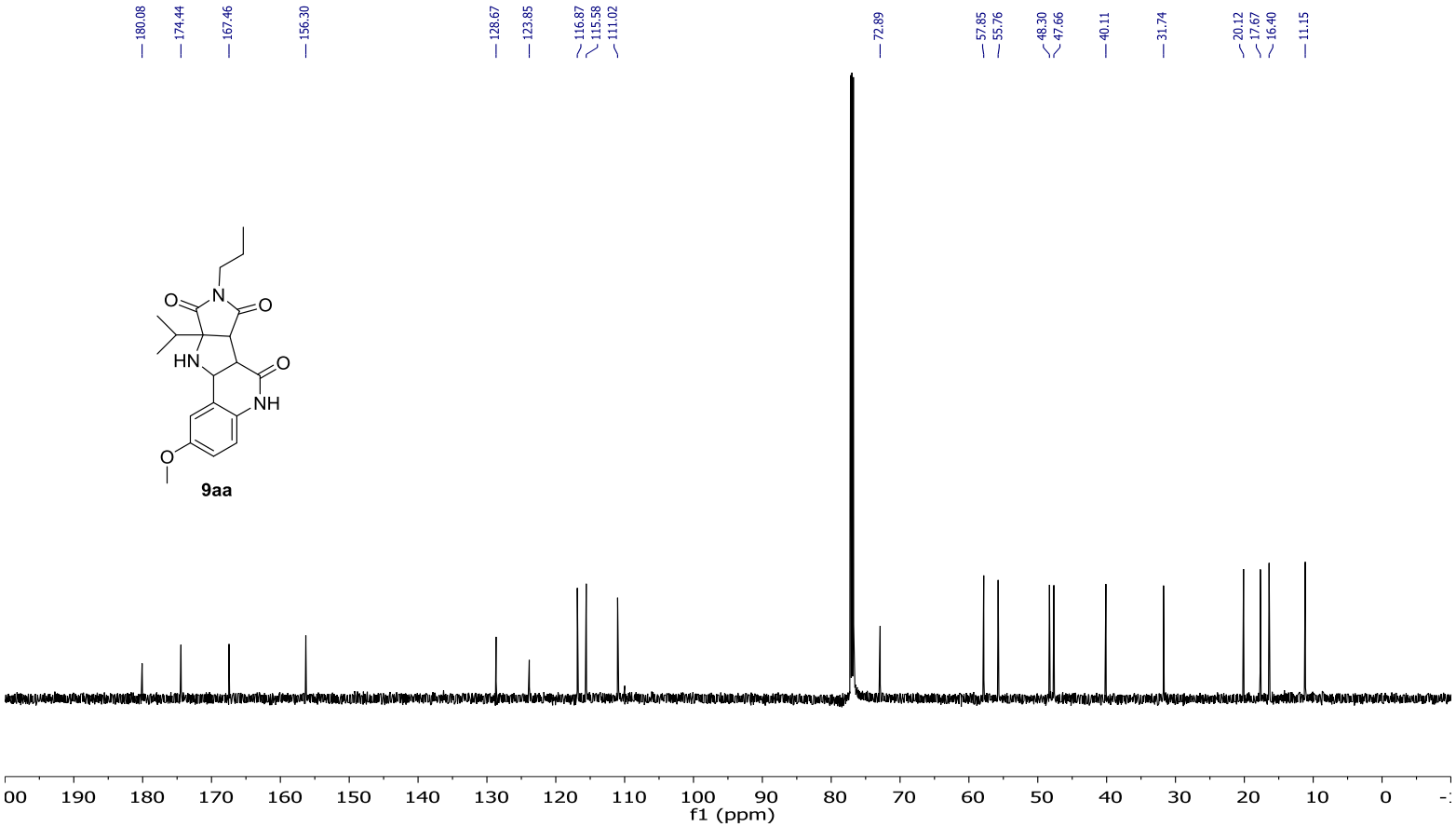
^{13}C NMR (101 MHz) of compound **9z** in $CDCl_3$



HRMS (ESI) of compound **9z**



^{1}H NMR (400 MHz) of compound **9aa** in $CDCl_3$



^{13}C NMR (101 MHz) of compound **9aa** in $CDCl_3$

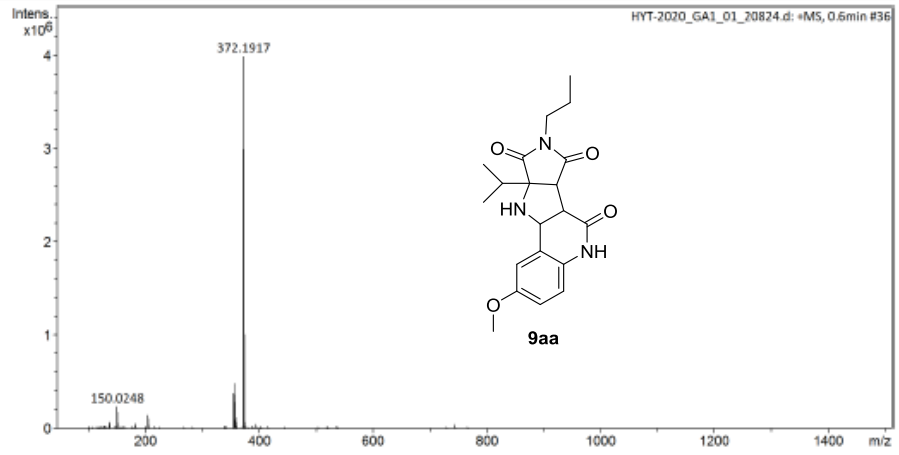
Display Report

Analysis Info

Analysis Name	D:\Data\nctu service\data\2020\20200612\HYT-2020_GA1_01_20824.d	Acquisition Date	6/12/2020 12:48:07 PM
Method	Small molecule.m	Operator	NCTU
Sample Name	HYT-2020	Instrument	impact HD 1819696.00164
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C

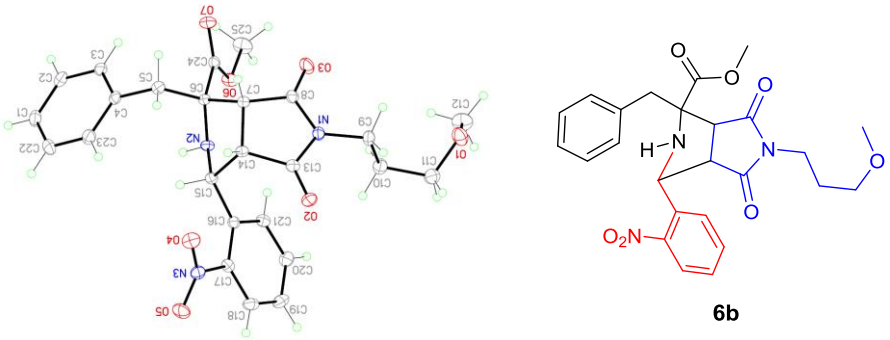


Display Report

Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdb	e⁻ Conf	N-Rule	Adduct
372.1917	1	C20H26N3O4	372.1918	-0.3	20.8	1	100.00	9.5	even	ok	M+H

HRMS (ESI) of compound **9aa**

X-ray crystallographic data of compound 6b



ORTEP diagram of compound 6b. Atomic displacement ellipsoids are drawn at the 50% probability level

CCDC No. 1985032

Table 1. Crystal data and structure refinement for 191240lt_0m.

Identification code	191240lt_0m	
Empirical formula	C25 H27 N3 O7	
Formula weight	481.49	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 10.5539(6) Å	α= 90°.
	b = 21.1479(12) Å	β= 108.937(3)°.
	c = 10.7227(6) Å	γ = 90°.
Volume	2263.7(2) $Å^3$	
Z	4	
Density (calculated)	1.413 Mg/m^3	
Absorption coefficient	0.104 mm^{-1}	
F(000)	1016	
Crystal size	0.10 x 0.10 x 0.08 mm^3	
Theta range for data collection	1.926 to 26.394°.	
Index ranges	-12<=h<=13, -26<=k<=26, -13<=l<=12	
Reflections collected	18444	
Independent reflections	4640 [R(int) = 0.0332]	
Completeness to theta = 25.242°	99.9 %	
Absorption correction	Semi-empirical from equivalents	

Max. and min. transmission	0.7454 and 0.6931
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4640 / 0 / 318
Goodness-of-fit on F^2	1.060
Final R indices [I>2sigma(I)]	R1 = 0.0402, wR2 = 0.1008
R indices (all data)	R1 = 0.0526, wR2 = 0.1080
Extinction coefficient	n/a
Largest diff. peak and hole	0.507 and -0.240 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 191240lt_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	2937(1)	660(1)	6068(1)	25(1)
O(2)	1090(1)	1486(1)	886(1)	20(1)
O(3)	5311(1)	1875(1)	3626(1)	23(1)
O(4)	1043(1)	1563(1)	-2506(1)	23(1)
O(5)	112(1)	755(1)	-3676(1)	25(1)
O(6)	7063(1)	1223(1)	2286(1)	18(1)
O(7)	7513(1)	2260(1)	2315(1)	19(1)
N(1)	3118(1)	1637(1)	2489(1)	15(1)
N(2)	4861(1)	1272(1)	263(1)	13(1)
N(3)	847(1)	994(1)	-2649(1)	17(1)
C(1)	7626(2)	1570(1)	-2824(2)	20(1)
C(2)	8363(2)	1757(1)	-1555(2)	18(1)
C(3)	7717(2)	2010(1)	-726(2)	16(1)
C(4)	6332(2)	2084(1)	-1156(2)	14(1)
C(5)	5605(2)	2359(1)	-274(2)	14(1)
C(6)	5437(2)	1886(1)	768(2)	12(1)
C(7)	4336(2)	2145(1)	1290(2)	13(1)
C(8)	4373(2)	1877(1)	2614(2)	15(1)
C(9)	2720(2)	1399(1)	3590(2)	17(1)
C(10)	2869(2)	685(1)	3740(2)	21(1)
C(11)	2282(2)	431(1)	4763(2)	26(1)
C(12)	4102(2)	312(1)	6737(2)	29(1)
C(13)	2247(2)	1653(1)	1204(2)	14(1)
C(14)	3006(2)	1898(1)	317(2)	12(1)
C(15)	3436(2)	1362(1)	-487(2)	12(1)
C(16)	2684(2)	740(1)	-554(2)	13(1)
C(17)	1491(2)	574(1)	-1536(2)	14(1)
C(18)	822(2)	10(1)	-1511(2)	19(1)
C(19)	1333(2)	-412(1)	-492(2)	20(1)
C(20)	2530(2)	-271(1)	484(2)	18(1)
C(21)	3194(2)	292(1)	441(2)	16(1)
C(22)	6249(2)	1643(1)	-3267(2)	23(1)
C(23)	5605(2)	1898(1)	-2436(2)	20(1)

C(24)	6783(2)	1820(1)	1875(2)	13(1)
C(25)	8281(2)	1153(1)	3386(2)	25(1)

Table 3. Bond lengths [Å] and angles [°] for 191240lt_0m.

O(1)-C(12)	1.413(2)
O(1)-C(11)	1.429(2)
O(2)-C(13)	1.2095(19)
O(3)-C(8)	1.208(2)
O(4)-N(3)	1.2211(18)
O(5)-N(3)	1.2317(18)
O(6)-C(24)	1.3384(19)
O(6)-C(25)	1.4425(19)
O(7)-C(24)	1.2023(19)
N(1)-C(8)	1.384(2)
N(1)-C(13)	1.386(2)
N(1)-C(9)	1.4644(19)
N(2)-C(6)	1.4623(19)
N(2)-C(15)	1.4694(19)
N(2)-H(11)	0.9502
N(3)-C(17)	1.466(2)
C(1)-C(22)	1.383(3)
C(1)-C(2)	1.388(2)
C(1)-H(1)	0.9500
C(2)-C(3)	1.391(2)
C(2)-H(17)	0.9500
C(3)-C(4)	1.392(2)
C(3)-H(14)	0.9500
C(4)-C(23)	1.395(2)
C(4)-C(5)	1.514(2)
C(5)-C(6)	1.551(2)
C(5)-H(18)	0.9900
C(5)-H(13)	0.9900
C(6)-C(24)	1.533(2)
C(6)-C(7)	1.544(2)
C(7)-C(8)	1.517(2)
C(7)-C(14)	1.544(2)
C(7)-H(5)	1.0000
C(9)-C(10)	1.520(2)
C(9)-H(23)	0.9900
C(9)-H(24)	0.9900

C(10)-C(11)	1.522(2)
C(10)-H(25)	0.9900
C(10)-H(4)	0.9900
C(11)-H(27)	0.9900
C(11)-H(26)	0.9900
C(12)-H(28)	0.9800
C(12)-H(2)	0.9800
C(12)-H(3)	0.9800
C(13)-C(14)	1.520(2)
C(14)-C(15)	1.577(2)
C(14)-H(22)	1.0000
C(15)-C(16)	1.527(2)
C(15)-H(6)	1.0000
C(16)-C(21)	1.397(2)
C(16)-C(17)	1.399(2)
C(17)-C(18)	1.390(2)
C(18)-C(19)	1.378(2)
C(18)-H(10)	0.9500
C(19)-C(20)	1.387(2)
C(19)-H(9)	0.9500
C(20)-C(21)	1.390(2)
C(20)-H(8)	0.9500
C(21)-H(7)	0.9500
C(22)-C(23)	1.393(2)
C(22)-H(15)	0.9500
C(23)-H(16)	0.9500
C(25)-H(20)	0.9800
C(25)-H(21)	0.9800
C(25)-H(19)	0.9800

C(12)-O(1)-C(11)	112.69(14)
C(24)-O(6)-C(25)	114.17(13)
C(8)-N(1)-C(13)	113.11(13)
C(8)-N(1)-C(9)	124.32(13)
C(13)-N(1)-C(9)	122.56(13)
C(6)-N(2)-C(15)	108.42(11)
C(6)-N(2)-H(11)	112.8
C(15)-N(2)-H(11)	114.3

O(4)-N(3)-O(5)	123.22(14)
O(4)-N(3)-C(17)	118.74(13)
O(5)-N(3)-C(17)	118.03(14)
C(22)-C(1)-C(2)	119.80(15)
C(22)-C(1)-H(1)	120.1
C(2)-C(1)-H(1)	120.1
C(1)-C(2)-C(3)	120.07(16)
C(1)-C(2)-H(17)	120.0
C(3)-C(2)-H(17)	120.0
C(2)-C(3)-C(4)	120.78(15)
C(2)-C(3)-H(14)	119.6
C(4)-C(3)-H(14)	119.6
C(3)-C(4)-C(23)	118.51(15)
C(3)-C(4)-C(5)	121.79(14)
C(23)-C(4)-C(5)	119.69(14)
C(4)-C(5)-C(6)	113.71(12)
C(4)-C(5)-H(18)	108.8
C(6)-C(5)-H(18)	108.8
C(4)-C(5)-H(13)	108.8
C(6)-C(5)-H(13)	108.8
H(18)-C(5)-H(13)	107.7
N(2)-C(6)-C(24)	112.03(12)
N(2)-C(6)-C(7)	100.28(12)
C(24)-C(6)-C(7)	111.64(12)
N(2)-C(6)-C(5)	115.87(12)
C(24)-C(6)-C(5)	108.79(12)
C(7)-C(6)-C(5)	107.94(12)
C(8)-C(7)-C(14)	104.31(12)
C(8)-C(7)-C(6)	114.35(13)
C(14)-C(7)-C(6)	105.12(12)
C(8)-C(7)-H(5)	110.9
C(14)-C(7)-H(5)	110.9
C(6)-C(7)-H(5)	110.9
O(3)-C(8)-N(1)	124.40(14)
O(3)-C(8)-C(7)	127.31(15)
N(1)-C(8)-C(7)	108.27(13)
N(1)-C(9)-C(10)	112.33(13)
N(1)-C(9)-H(23)	109.1

C(10)-C(9)-H(23)	109.1
N(1)-C(9)-H(24)	109.1
C(10)-C(9)-H(24)	109.1
H(23)-C(9)-H(24)	107.9
C(9)-C(10)-C(11)	112.04(14)
C(9)-C(10)-H(25)	109.2
C(11)-C(10)-H(25)	109.2
C(9)-C(10)-H(4)	109.2
C(11)-C(10)-H(4)	109.2
H(25)-C(10)-H(4)	107.9
O(1)-C(11)-C(10)	113.86(14)
O(1)-C(11)-H(27)	108.8
C(10)-C(11)-H(27)	108.8
O(1)-C(11)-H(26)	108.8
C(10)-C(11)-H(26)	108.8
H(27)-C(11)-H(26)	107.7
O(1)-C(12)-H(28)	109.5
O(1)-C(12)-H(2)	109.5
H(28)-C(12)-H(2)	109.5
O(1)-C(12)-H(3)	109.5
H(28)-C(12)-H(3)	109.5
H(2)-C(12)-H(3)	109.5
O(2)-C(13)-N(1)	123.84(14)
O(2)-C(13)-C(14)	127.72(14)
N(1)-C(13)-C(14)	108.44(13)
C(13)-C(14)-C(7)	103.93(12)
C(13)-C(14)-C(15)	113.60(12)
C(7)-C(14)-C(15)	104.67(12)
C(13)-C(14)-H(22)	111.4
C(7)-C(14)-H(22)	111.4
C(15)-C(14)-H(22)	111.4
N(2)-C(15)-C(16)	109.75(12)
N(2)-C(15)-C(14)	102.65(11)
C(16)-C(15)-C(14)	114.07(12)
N(2)-C(15)-H(6)	110.0
C(16)-C(15)-H(6)	110.0
C(14)-C(15)-H(6)	110.0
C(21)-C(16)-C(17)	115.76(14)

C(21)-C(16)-C(15)	118.86(14)
C(17)-C(16)-C(15)	125.37(14)
C(18)-C(17)-C(16)	122.63(15)
C(18)-C(17)-N(3)	115.58(14)
C(16)-C(17)-N(3)	121.77(14)
C(19)-C(18)-C(17)	120.05(15)
C(19)-C(18)-H(10)	120.0
C(17)-C(18)-H(10)	120.0
C(18)-C(19)-C(20)	118.99(15)
C(18)-C(19)-H(9)	120.5
C(20)-C(19)-H(9)	120.5
C(19)-C(20)-C(21)	120.35(15)
C(19)-C(20)-H(8)	119.8
C(21)-C(20)-H(8)	119.8
C(20)-C(21)-C(16)	122.15(15)
C(20)-C(21)-H(7)	118.9
C(16)-C(21)-H(7)	118.9
C(1)-C(22)-C(23)	120.02(16)
C(1)-C(22)-H(15)	120.0
C(23)-C(22)-H(15)	120.0
C(22)-C(23)-C(4)	120.81(16)
C(22)-C(23)-H(16)	119.6
C(4)-C(23)-H(16)	119.6
O(7)-C(24)-O(6)	123.73(14)
O(7)-C(24)-C(6)	123.23(14)
O(6)-C(24)-C(6)	113.04(13)
O(6)-C(25)-H(20)	109.5
O(6)-C(25)-H(21)	109.5
H(20)-C(25)-H(21)	109.5
O(6)-C(25)-H(19)	109.5
H(20)-C(25)-H(19)	109.5
H(21)-C(25)-H(19)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 191240lt_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

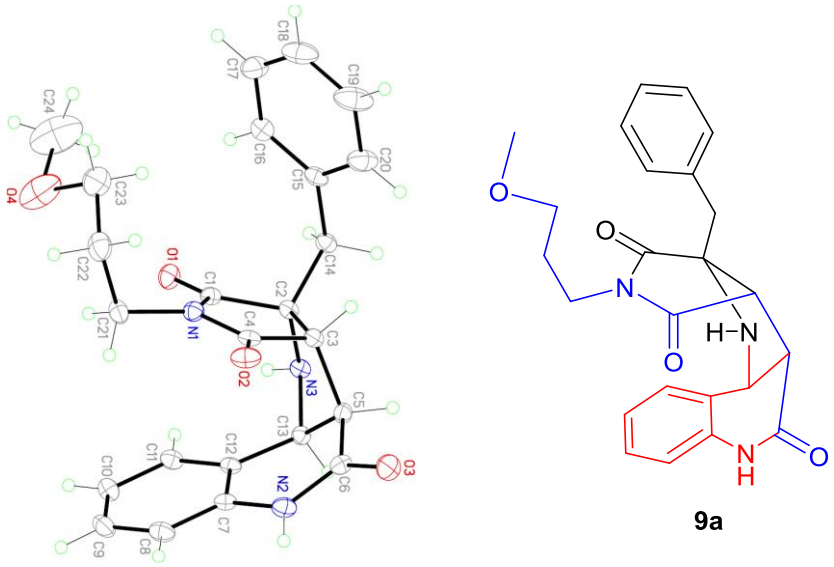
	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	23(1)	34(1)	18(1)	2(1)	5(1)	4(1)
O(2)	13(1)	25(1)	22(1)	-2(1)	6(1)	-4(1)
O(3)	16(1)	40(1)	12(1)	-1(1)	2(1)	-1(1)
O(4)	20(1)	19(1)	26(1)	3(1)	4(1)	2(1)
O(5)	18(1)	36(1)	16(1)	-2(1)	-2(1)	-2(1)
O(6)	16(1)	16(1)	16(1)	2(1)	-1(1)	2(1)
O(7)	15(1)	19(1)	20(1)	-2(1)	2(1)	-4(1)
N(1)	14(1)	17(1)	13(1)	0(1)	6(1)	0(1)
N(2)	11(1)	13(1)	14(1)	-2(1)	2(1)	-1(1)
N(3)	11(1)	22(1)	18(1)	0(1)	4(1)	0(1)
C(1)	24(1)	22(1)	19(1)	2(1)	12(1)	-1(1)
C(2)	14(1)	19(1)	21(1)	6(1)	7(1)	0(1)
C(3)	16(1)	16(1)	14(1)	2(1)	4(1)	-3(1)
C(4)	16(1)	12(1)	14(1)	4(1)	5(1)	-1(1)
C(5)	13(1)	14(1)	15(1)	2(1)	3(1)	1(1)
C(6)	11(1)	12(1)	12(1)	0(1)	3(1)	-1(1)
C(7)	13(1)	14(1)	13(1)	-1(1)	4(1)	-1(1)
C(8)	14(1)	16(1)	15(1)	-3(1)	6(1)	0(1)
C(9)	18(1)	21(1)	16(1)	1(1)	9(1)	0(1)
C(10)	23(1)	22(1)	21(1)	1(1)	10(1)	-1(1)
C(11)	27(1)	29(1)	19(1)	3(1)	5(1)	-8(1)
C(12)	22(1)	30(1)	30(1)	2(1)	2(1)	3(1)
C(13)	14(1)	12(1)	16(1)	-1(1)	5(1)	2(1)
C(14)	11(1)	12(1)	14(1)	0(1)	3(1)	0(1)
C(15)	10(1)	14(1)	12(1)	0(1)	2(1)	1(1)
C(16)	12(1)	13(1)	13(1)	-1(1)	5(1)	0(1)
C(17)	13(1)	16(1)	13(1)	0(1)	4(1)	2(1)
C(18)	14(1)	20(1)	21(1)	-5(1)	3(1)	-3(1)
C(19)	19(1)	15(1)	28(1)	-1(1)	9(1)	-4(1)
C(20)	20(1)	16(1)	20(1)	3(1)	7(1)	1(1)
C(21)	15(1)	16(1)	15(1)	-1(1)	3(1)	0(1)
C(22)	24(1)	31(1)	12(1)	0(1)	6(1)	-4(1)
C(23)	15(1)	27(1)	15(1)	4(1)	3(1)	-1(1)

C(24)	12(1)	17(1)	12(1)	0(1)	5(1)	1(1)
C(25)	20(1)	27(1)	20(1)	4(1)	-4(1)	5(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 191240lt_0m.

	x	y	z	U(eq)
H(1)	8066	1393	-3387	24
H(17)	9310	1712	-1252	21
H(14)	8228	2134	143	19
H(18)	4708	2504	-829	17
H(13)	6106	2733	187	17
H(5)	4349	2618	1311	16
H(23)	1775	1515	3447	21
H(24)	3279	1603	4415	21
H(25)	3831	573	4004	26
H(4)	2411	482	2879	26
H(27)	1322	548	4497	31
H(26)	2336	-37	4770	31
H(28)	3871	-136	6760	44
H(2)	4484	471	7640	44
H(3)	4760	358	6275	44
H(22)	2499	2242	-276	15
H(6)	3334	1515	-1397	15
H(10)	13	-84	-2197	22
H(9)	871	-794	-459	24
H(8)	2899	-561	1185	22
H(7)	4022	375	1111	19
H(15)	5742	1519	-4138	27
H(16)	4658	1945	-2746	23
H(20)	9036	1323	3149	37
H(21)	8436	704	3610	37
H(19)	8198	1385	4147	37
H(11)	5354	1057	-208	80

X-ray crystallographic data of compound 9a



ORTEP diagram of compound 9a. Atomic displacement ellipsoids are drawn at the 50% probability level

CCDC No. 844607

Table 6. Crystal data and structure refinement for 110206lt_0m.

Identification code	110206lt_0m	
Empirical formula	C24 H25 N3 O4	
Formula weight	419.47	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 8.5285(2) Å	α= 74.4790(10)°.
	b = 10.0990(2) Å	β= 80.0180(10)°.
	c = 12.5940(3) Å	γ = 87.2130(10)°.
Volume	1029.33(4) $Å^3$	
Z	2	
Density (calculated)	1.353 Mg/m^3	
Absorption coefficient	0.093 mm^{-1}	
F(000)	444	
Crystal size	0.15 x 0.12 x 0.12 mm^3	
Theta range for data collection	1.70 to 26.40°.	

Index ranges	-10<=h<=10, -12<=k<=12, -15<=l<=15
Reflections collected	17022
Independent reflections	4203 [R(int) = 0.0280]
Completeness to theta = 26.40°	99.5 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9486 and 0.9067
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4203 / 67 / 298
Goodness-of-fit on F^2	1.047
Final R indices [I>2sigma(I)]	R1 = 0.0627, wR2 = 0.1758
R indices (all data)	R1 = 0.0796, wR2 = 0.1978
Largest diff. peak and hole	1.820 and -0.469 e.$Å^{-3}$

Table 7. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 110206lt_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	1054(3)	1485(2)	3203(2)	14(1)
C(2)	-90(3)	821(2)	2681(2)	13(1)
C(3)	816(3)	903(2)	1496(2)	14(1)
C(4)	2378(3)	1603(2)	1422(2)	15(1)
C(5)	994(3)	-617(2)	1442(2)	15(1)
C(6)	2407(3)	-899(2)	622(2)	16(1)
C(7)	3964(3)	-1392(2)	2142(2)	15(1)
C(8)	5495(3)	-1431(2)	2398(2)	20(1)
C(9)	5702(3)	-1486(2)	3476(2)	21(1)
C(10)	4387(3)	-1483(3)	4299(2)	21(1)
C(11)	2861(3)	-1443(2)	4042(2)	17(1)
C(12)	2631(3)	-1403(2)	2967(2)	14(1)
C(13)	979(3)	-1403(2)	2666(2)	14(1)
C(14)	-1739(3)	1512(2)	2753(2)	16(1)
C(15)	-1738(3)	3057(2)	2269(2)	17(1)
C(16)	-1649(3)	3944(3)	2933(2)	24(1)
C(17)	-1682(3)	5363(3)	2488(3)	32(1)
C(18)	-1791(3)	5905(3)	1379(3)	33(1)
C(19)	-1868(4)	5047(3)	712(2)	36(1)
C(20)	-1853(3)	3626(3)	1149(2)	27(1)
N(1)	2396(2)	1937(2)	2417(2)	16(1)
N(2)	3759(2)	-1329(2)	1043(2)	18(1)
O(1)	853(2)	1574(2)	4158(1)	19(1)
O(2)	3457(2)	1853(2)	631(1)	22(1)
O(3)	2322(2)	-750(2)	-365(1)	24(1)
N(3)	-272(2)	-656(2)	3226(2)	14(1)
C(21)	3790(30)	2454(15)	2760(16)	19(1)
C(22)	3937(17)	4012(14)	2359(9)	26(1)
C(23)	2704(11)	4713(9)	2977(8)	35(1)
O(4)	2920(9)	4298(7)	4173(6)	54(1)
C(24)	1436(13)	4731(11)	4687(9)	75(2)
C(21')	3840(30)	2502(12)	2580(13)	19(1)
C(22')	4050(14)	4030(11)	2057(6)	26(1)

C(23')	2743(8)	4895(7)	2464(6)	35(1)
O(4')	2475(7)	4679(6)	3638(6)	54(1)
C(24')	3791(11)	4952(9)	4166(7)	75(2)

Table 8. Bond lengths [Å] and angles [°] for 110206lt_0m.

C(1)-O(1)	1.212(3)
C(1)-N(1)	1.383(3)
C(1)-C(2)	1.534(3)
C(2)-N(3)	1.470(3)
C(2)-C(14)	1.538(3)
C(2)-C(3)	1.540(3)
C(3)-C(4)	1.515(3)
C(3)-C(5)	1.554(3)
C(3)-H(3)	1.0000
C(4)-O(2)	1.214(3)
C(4)-N(1)	1.384(3)
C(5)-C(6)	1.514(3)
C(5)-C(13)	1.531(3)
C(5)-H(5)	1.0000
C(6)-O(3)	1.226(3)
C(6)-N(2)	1.357(3)
C(7)-C(8)	1.395(3)
C(7)-C(12)	1.400(3)
C(7)-N(2)	1.408(3)
C(8)-C(9)	1.386(4)
C(8)-H(8)	0.9500
C(9)-C(10)	1.389(4)
C(9)-H(9)	0.9500
C(10)-C(11)	1.390(3)
C(10)-H(10)	0.9500
C(11)-C(12)	1.391(3)
C(11)-H(11)	0.9500
C(12)-C(13)	1.521(3)
C(13)-N(3)	1.467(3)
C(13)-H(13)	1.0000
C(14)-C(15)	1.516(3)
C(14)-H(14A)	0.9900
C(14)-H(14B)	0.9900
C(15)-C(16)	1.391(4)
C(15)-C(20)	1.392(3)
C(16)-C(17)	1.393(4)

C(16)-H(16)	0.9500
C(17)-C(18)	1.375(4)
C(17)-H(17)	0.9500
C(18)-C(19)	1.369(4)
C(18)-H(18)	0.9500
C(19)-C(20)	1.393(4)
C(19)-H(19)	0.9500
C(20)-H(20)	0.9500
N(1)-C(21')	1.45(2)
N(1)-C(21)	1.50(3)
N(2)-H(2)	0.8800
N(3)-H(3A)	0.8922
C(21)-C(22)	1.523(11)
C(21)-H(21A)	0.9900
C(21)-H(21B)	0.9900
C(22)-C(23)	1.466(11)
C(22)-H(22A)	0.9900
C(22)-H(22B)	0.9900
C(23)-O(4)	1.494(10)
C(23)-H(23A)	0.9900
C(23)-H(23B)	0.9900
O(4)-C(24)	1.423(11)
C(24)-H(24A)	0.9800
C(24)-H(24B)	0.9800
C(24)-H(24C)	0.9800
C(21')-C(22')	1.512(9)
C(21')-H(21C)	0.9900
C(21')-H(21D)	0.9900
C(22')-C(23')	1.488(9)
C(22')-H(22C)	0.9900
C(22')-H(22D)	0.9900
C(23')-O(4')	1.415(8)
C(23')-H(23C)	0.9900
C(23')-H(23D)	0.9900
O(4')-C(24')	1.472(11)
C(24')-H(24D)	0.9800
C(24')-H(24E)	0.9800
C(24')-H(24F)	0.9800

O(1)-C(1)-N(1)	124.6(2)
O(1)-C(1)-C(2)	126.2(2)
N(1)-C(1)-C(2)	109.15(18)
N(3)-C(2)-C(1)	111.24(17)
N(3)-C(2)-C(14)	109.12(17)
C(1)-C(2)-C(14)	111.45(18)
N(3)-C(2)-C(3)	104.89(17)
C(1)-C(2)-C(3)	103.73(17)
C(14)-C(2)-C(3)	116.17(18)
C(4)-C(3)-C(2)	105.29(17)
C(4)-C(3)-C(5)	114.17(18)
C(2)-C(3)-C(5)	104.47(17)
C(4)-C(3)-H(3)	110.9
C(2)-C(3)-H(3)	110.9
C(5)-C(3)-H(3)	110.9
O(2)-C(4)-N(1)	124.1(2)
O(2)-C(4)-C(3)	126.9(2)
N(1)-C(4)-C(3)	109.00(19)
C(6)-C(5)-C(13)	115.59(19)
C(6)-C(5)-C(3)	114.78(18)
C(13)-C(5)-C(3)	102.86(17)
C(6)-C(5)-H(5)	107.7
C(13)-C(5)-H(5)	107.7
C(3)-C(5)-H(5)	107.7
O(3)-C(6)-N(2)	122.2(2)
O(3)-C(6)-C(5)	121.5(2)
N(2)-C(6)-C(5)	116.24(19)
C(8)-C(7)-C(12)	120.4(2)
C(8)-C(7)-N(2)	119.8(2)
C(12)-C(7)-N(2)	119.9(2)
C(9)-C(8)-C(7)	120.0(2)
C(9)-C(8)-H(8)	120.0
C(7)-C(8)-H(8)	120.0
C(8)-C(9)-C(10)	120.1(2)
C(8)-C(9)-H(9)	120.0
C(10)-C(9)-H(9)	120.0
C(9)-C(10)-C(11)	119.9(2)

C(9)-C(10)-H(10)	120.1
C(11)-C(10)-H(10)	120.1
C(10)-C(11)-C(12)	120.8(2)
C(10)-C(11)-H(11)	119.6
C(12)-C(11)-H(11)	119.6
C(11)-C(12)-C(7)	118.9(2)
C(11)-C(12)-C(13)	122.2(2)
C(7)-C(12)-C(13)	118.9(2)
N(3)-C(13)-C(12)	116.74(18)
N(3)-C(13)-C(5)	100.74(17)
C(12)-C(13)-C(5)	110.29(18)
N(3)-C(13)-H(13)	109.6
C(12)-C(13)-H(13)	109.6
C(5)-C(13)-H(13)	109.6
C(15)-C(14)-C(2)	115.34(19)
C(15)-C(14)-H(14A)	108.4
C(2)-C(14)-H(14A)	108.4
C(15)-C(14)-H(14B)	108.4
C(2)-C(14)-H(14B)	108.4
H(14A)-C(14)-H(14B)	107.5
C(16)-C(15)-C(20)	118.2(2)
C(16)-C(15)-C(14)	121.1(2)
C(20)-C(15)-C(14)	120.7(2)
C(15)-C(16)-C(17)	120.8(3)
C(15)-C(16)-H(16)	119.6
C(17)-C(16)-H(16)	119.6
C(18)-C(17)-C(16)	120.1(3)
C(18)-C(17)-H(17)	119.9
C(16)-C(17)-H(17)	119.9
C(19)-C(18)-C(17)	119.9(3)
C(19)-C(18)-H(18)	120.1
C(17)-C(18)-H(18)	120.1
C(18)-C(19)-C(20)	120.6(3)
C(18)-C(19)-H(19)	119.7
C(20)-C(19)-H(19)	119.7
C(15)-C(20)-C(19)	120.4(3)
C(15)-C(20)-H(20)	119.8
C(19)-C(20)-H(20)	119.8

C(1)-N(1)-C(4)	112.73(19)
C(1)-N(1)-C(21')	127.8(7)
C(4)-N(1)-C(21')	118.8(7)
C(1)-N(1)-C(21)	119.7(8)
C(4)-N(1)-C(21)	126.4(8)
C(21')-N(1)-C(21)	8.2(13)
C(6)-N(2)-C(7)	124.3(2)
C(6)-N(2)-H(2)	117.8
C(7)-N(2)-H(2)	117.8
C(13)-N(3)-C(2)	108.73(17)
C(13)-N(3)-H(3A)	110.4
C(2)-N(3)-H(3A)	111.7
N(1)-C(21)-C(22)	112.1(16)
N(1)-C(21)-H(21A)	109.2
C(22)-C(21)-H(21A)	109.2
N(1)-C(21)-H(21B)	109.2
C(22)-C(21)-H(21B)	109.2
H(21A)-C(21)-H(21B)	107.9
C(23)-C(22)-C(21)	112.6(12)
C(23)-C(22)-H(22A)	109.1
C(21)-C(22)-H(22A)	109.1
C(23)-C(22)-H(22B)	109.1
C(21)-C(22)-H(22B)	109.1
H(22A)-C(22)-H(22B)	107.8
C(22)-C(23)-O(4)	107.5(7)
C(22)-C(23)-H(23A)	110.2
O(4)-C(23)-H(23A)	110.2
C(22)-C(23)-H(23B)	110.2
O(4)-C(23)-H(23B)	110.2
H(23A)-C(23)-H(23B)	108.5
C(24)-O(4)-C(23)	100.3(8)
N(1)-C(21')-C(22')	114.5(13)
N(1)-C(21')-H(21C)	108.6
C(22')-C(21')-H(21C)	108.6
N(1)-C(21')-H(21D)	108.6
C(22')-C(21')-H(21D)	108.6
H(21C)-C(21')-H(21D)	107.6
C(23')-C(22')-C(21')	114.6(10)

C(23')-C(22')-H(22C)	108.6
C(21')-C(22')-H(22C)	108.6
C(23')-C(22')-H(22D)	108.6
C(21')-C(22')-H(22D)	108.6
H(22C)-C(22')-H(22D)	107.6
O(4')-C(23')-C(22')	113.3(6)
O(4')-C(23')-H(23C)	108.9
C(22')-C(23')-H(23C)	108.9
O(4')-C(23')-H(23D)	108.9
C(22')-C(23')-H(23D)	108.9
H(23C)-C(23')-H(23D)	107.7
C(23')-O(4')-C(24')	118.2(6)
O(4')-C(24')-H(24D)	109.5
O(4')-C(24')-H(24E)	109.5
H(24D)-C(24')-H(24E)	109.5
O(4')-C(24')-H(24F)	109.5
H(24D)-C(24')-H(24F)	109.5
H(24E)-C(24')-H(24F)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 9. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 110206lt_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	16(1)	12(1)	15(1)	-3(1)	-3(1)	2(1)
C(2)	12(1)	14(1)	11(1)	-2(1)	-1(1)	1(1)
C(3)	14(1)	16(1)	12(1)	-4(1)	-2(1)	3(1)
C(4)	15(1)	11(1)	16(1)	-1(1)	-1(1)	4(1)
C(5)	15(1)	16(1)	14(1)	-6(1)	-3(1)	0(1)
C(6)	19(1)	14(1)	15(1)	-4(1)	-1(1)	0(1)
C(7)	17(1)	11(1)	17(1)	-3(1)	-2(1)	3(1)
C(8)	15(1)	17(1)	23(1)	-4(1)	1(1)	2(1)
C(9)	15(1)	20(1)	28(1)	-3(1)	-7(1)	2(1)
C(10)	21(1)	21(1)	21(1)	-5(1)	-9(1)	4(1)
C(11)	16(1)	17(1)	17(1)	-4(1)	-1(1)	3(1)
C(12)	14(1)	10(1)	16(1)	-1(1)	-1(1)	1(1)
C(13)	13(1)	14(1)	15(1)	-4(1)	-2(1)	0(1)
C(14)	14(1)	18(1)	14(1)	-4(1)	-1(1)	2(1)
C(15)	11(1)	19(1)	20(1)	-4(1)	0(1)	3(1)
C(16)	25(1)	22(1)	29(1)	-8(1)	-11(1)	5(1)
C(17)	29(2)	20(1)	51(2)	-14(1)	-12(1)	4(1)
C(18)	30(2)	17(1)	43(2)	2(1)	2(1)	4(1)
C(19)	46(2)	27(2)	24(1)	4(1)	3(1)	14(1)
C(20)	36(2)	25(1)	20(1)	-7(1)	-3(1)	11(1)
N(1)	15(1)	14(1)	17(1)	-4(1)	-2(1)	-1(1)
N(2)	18(1)	19(1)	15(1)	-5(1)	2(1)	4(1)
O(1)	23(1)	22(1)	13(1)	-4(1)	-4(1)	-1(1)
O(2)	20(1)	18(1)	22(1)	-3(1)	7(1)	2(1)
O(3)	27(1)	30(1)	15(1)	-10(1)	-3(1)	4(1)
N(3)	14(1)	15(1)	12(1)	-2(1)	-1(1)	1(1)
C(21)	16(2)	20(1)	20(4)	-3(2)	-6(3)	-1(1)
C(22)	26(2)	24(1)	29(4)	-2(2)	-8(3)	-7(1)
C(23)	34(2)	30(2)	44(3)	-11(3)	-9(3)	-2(2)
O(4)	57(2)	41(2)	64(3)	-26(2)	10(2)	-11(2)
C(24)	89(3)	54(3)	75(3)	-26(2)	17(3)	-4(2)
C(21')	16(2)	20(1)	20(4)	-3(2)	-6(3)	-1(1)
C(22')	26(2)	24(1)	29(4)	-2(2)	-8(3)	-7(1)

C(23')	34(2)	30(2)	44(3)	-11(3)	-9(3)	-2(2)
O(4')	57(2)	41(2)	64(3)	-26(2)	10(2)	-11(2)
C(24')	89(3)	54(3)	75(3)	-26(2)	17(3)	-4(2)

Table 10. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 110206lt_0m.

	x	y	z	U(eq)
H(3)	199	1446	917	17
H(5)	9	-872	1213	18
H(8)	6396	-1420	1834	24
H(9)	6745	-1525	3653	25
H(10)	4529	-1508	5036	25
H(11)	1965	-1443	4607	21
H(13)	631	-2371	2787	17
H(14A)	-2407	1096	2359	19
H(14B)	-2245	1303	3549	19
H(16)	-1565	3578	3698	29
H(17)	-1628	5957	2951	38
H(18)	-1814	6873	1076	39
H(19)	-1931	5425	-56	43
H(20)	-1923	3042	680	33
H(2)	4562	-1586	601	21
H(3A)	-225	-845	3954	17
H(21A)	4781	2034	2448	22
H(21B)	3687	2166	3585	22
H(22A)	4997	4286	2445	32
H(22B)	3858	4312	1555	32
H(23A)	1633	4443	2907	42
H(23B)	2810	5721	2674	42
H(24A)	1102	5590	4201	112
H(24B)	1551	4879	5407	112
H(24C)	632	4021	4806	112
H(21C)	4768	2017	2263	22
H(21D)	3845	2316	3393	22
H(22C)	5065	4317	2207	32
H(22D)	4134	4207	1238	32
H(23C)	3006	5874	2106	42
H(23D)	1750	4695	2230	42
H(24D)	4676	4322	4047	112

H(24E)	3424	4812	4970	112
H(24F)	4151	5902	3833	112