Efficient Synthesis of Trisimidazole and Glutaric Acid-Bearing Porphyrins: Active-Site Models of Bacterial Nitric Oxide Reductase (NOR)

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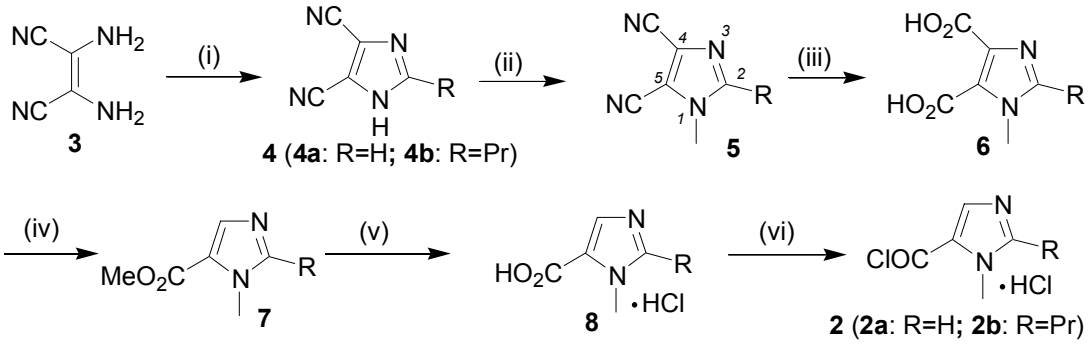
Supporting Information

General method:

All reactions were performed in oven-dried flasks. Air and moisture-sensitive compounds were introduced *via* syringes through a rubber septum under N_2 atmosphere. All reagents were used as supplied commercially unless otherwise noted. THF were distilled from sodium metal-benzophenone before use. Dichloromethane was distilled over calcium hydride. Flash column chromatography was performed on E. Merck silica gel 60 (230–400 mesh ASTM). Nuclear magnetic resonance spectra were recorded in deuteriochloroform ($CDCl_3$) unless otherwise indicated, with tetramethylsilane (TMS) as internal standard at ambient temperature on a Varian XL-400 or XL-500 Spectrometer.

Preparation of Imidazolecarboxylic Acid Chlorides 2:

Scheme S1:



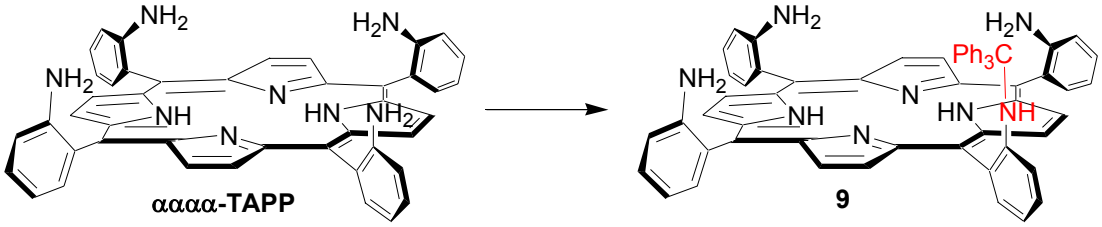
Reaction conditions: (i) $(CH_3O)_3CR$, xylene, 130°C, 83–88%; (ii) R=H: $(CH_3O)_2SO_2$, $NaHCO_3$; R=Pr: $(CH_3O)_3CCH_3$, 84–90%; (iii) (a) 2.0 N NaOH, (b) HCl, 75–90%; (iv) (a) Ac_2O, 95–100 °C; (b) $SOCl_2$, reflux, then CH_3OH, 60–69%; (v) HCl, reflux, 80–93%; (vi) Oxalyl Chloride, CH_3CN, 72–85%.

5-imidazolecarboxylic acid chlorides **2** were prepared following Scheme S1. Synthesis of 5-imidazolecarboxylic acid methyl esters **7** followed a literature procedure with modification as shown in the reaction conditions of Scheme S1.[1]

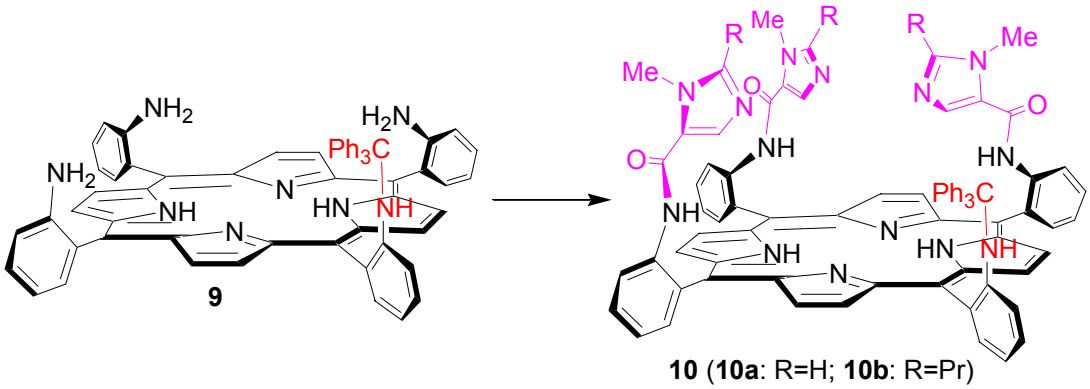
General procedure for transformation of 5-imidazolecarboxylic acid methyl esters 7 to acids 8: To 5-imidazolecarboxylic acid methyl esters **7b** (1.60 g, 8.8 mmol) was added concentrated hydrochloride acid (40 mL). The mixture was stirred and refluxed for 3 h. After removal of hydrochloride acid under vacuum, the solid was washed with CH_3CN/Et_2O (v/v 1/1) to give product 5-imidazolecarboxylic acid **8b** (1.67 g, 93%). ^{1}H NMR (500 MHz, DMSO-d6): the carboxylic acid proton was not assigned, δ 8.23 (s, 1H), 3.91 (s, 3H), 2.98 (t, J = 7.5 Hz, 2H), 1.71 (m, 2 H), 0.91 (t, J = 7.5 Hz, 3H); ^{13}C NMR (DMSO-d6): 159.56, 150.76, 124.90, 124.50, 33.25, 25.92, 19.74, 13.27; MS (ESI): m/e = 169.8 $[M+H]^+$ for $C_8H_{12}N_2O_2$; HRMS (ESI) for $C_8H_{13}N_2O_2$ $[M+H]^+$: calcd. 169.0977, found 169.0981.

Characterization of 5-imidazolecarboxylic acid 8a (prepared similarly to **8b**): ^{1}H NMR (500 MHz, DMSO-d6): the carboxylic acid proton was not assigned, δ 7.87 (s, 1H), 7.58 (s, 1H), 3.82 (s, 3H); ^{13}C NMR (DMSO-d6): 161.94, 143.81, 137.20, 124.11, 34.30; MS (ESI): m/e = 126.4 $[M]^+$ for $C_5H_6N_2O_2$; HRMS (ESI) for $C_5H_7N_2O_2$ $[M+H]^+$: calcd. 127.0508, found 127.0502.

General procedure for transformation of 5-imidazolecarboxylic acids 8 to acid chlorides 2: 5-imidazolecarboxylic acid hydrochloride **8b** (1.2 g, 5.9 mmol) was stirred in dry CH_3CN (10 mL). Oxalyl chloride (4.0 mL) was added followed by 2 drops of DMF. The reaction was stirred at room temperature for 4 h. After removal of most solvent CH_3CN, dry Et_2O (5 mL) was added. The mixture was stirred for further 10 min. Product 5-imidazolecarboxylic acid chloride hydrochloride **2b** (0.95 g, 72%) was collected by filtration. The product was used for next reaction without further purification.



Preparation of Compound 9: Trityl bromide (185 mg, 0.57 mmol) was added to a solution of αααα-TAPP (350 mg, 0.52 mmol) in dry CH_2Cl_2 (100 mL) at 0°C followed by triethylamine (0.2 mL, 1.56 mmol). The mixture was stirred at 0 °C for 2 h then concentrated under vacuum. The residue was purified by chromatography on silica gel with eluent EtOAc/CH_2Cl_2=5/95 to give compound **9** (298 mg, 63%) as a solid. The compound is capable of atropisomerization at room temperature thus should be stored in a freezer. ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.05 (s, 4H), 8.98-9.01 (m, 3H), 8.01 (dd, *J* = 1.5, 7.5 Hz, 2H), 7.92 (dd, *J* = 1.5, 7.5 Hz, 1H), 7.64–7.71 (m, 4H), 7.31 (t, *J* = 8 Hz, 1H), 7.22–7.29 (m, 6H), 7.13–7.20 (m, 7H), 6.96–7.04(m, 10H), 6.77 (d, *J* = 8.5 Hz, 1H), 5.28 (s, 1H), 3.61 (s, br, 6H), –2.59 (s, 2H); ^{13}C NMR ($CDCl_3$): δ 146.88, 145.46, 145.13, 135.41, 134.94, 134.81, 129.79, 128.68, 128.26, 127.91, 127.77, 126.90, 126.47, 117.68, 117.62, 116.55, 116.26, 116.00, 115.93, 115.75, 115.35, 115.30, 71.59; MS (ESI): m/e = 917.3 $[M+H]^+$for $C_{63}H_{49}N_8$. HRMS (ESI) for $C_{63}H_{49}N_8$ $[M+H]^+$: calcd.917.4056, found 917.4061.



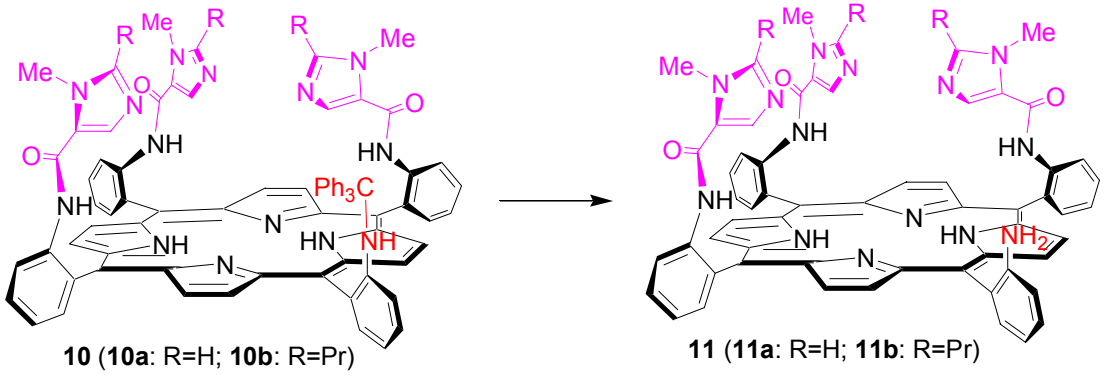
Preparation of Compound 10a:

1-methyl-5-imidazolecarboxylic acid hydrochloride **8a** (790 mg, 4.86 mmol) was stirred in dry acetonitrile (20 mL). Oxalyl chloride (3 mL) was added followed by 2 drops of DMF. The suspension was stirred at room temperature for 4 h. After removal of one half of the solvent under vacuum, dry diethyl ether (20 mL) was added. The

suspension was stirred for further 10 min. 1-methyl-5-imidazolecarboxylic acid chloride hydrochloride **2a** (~ 790 mg) was collected by filtration. The product was used without further purification.

The above 1-methyl-5-imidazolecarboxylic acid chloride hydrochloride **2a** (~ 790 mg) was stirred in dry acetonitrile (20 mL) at room temperature. *N*, *N*-Diethylaniline (1.1 mL) was added. A solution of compound **9** (430 mg, 0.47 mmol) in dry THF (20 mL) was added dropwise over a period of 1 h followed by further stirring for 30 min. Trace amount of mono- and bisimidazole-substituted porphyrins remained. The mixture was concentrated and washed with saturated $NaHCO_3$ water solution. After drying over Na_2SO_4 and concentrated. The residue was purified by chromatography on silica gel with eluent CH_3OH/CH_2Cl_2 (40% saturated with NH_3 gas) = 5/95 to give compound **10a** (520 mg, 90%) as a solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.87–8.98 (m, 8H), 8.46–8.49 (m, 3H), 8.31 (s, 1H), 8.10 (d, *J* = 7.5 Hz, 1H), 8.06–8.07 (d, *J* = 7.5 Hz, 3H), 7.84–7.89 (m, 3H), 7.58–7.62 (m, 3H), 7.36 (d, *J* = 7.5 Hz, 1H), 7.21 (t, *J* = 7.5 Hz, 1H), 7.05–7.09 (m, 8H), 6.84–6.90 (m, 12H), 67.00 (d, *J* = 8.5 Hz, 1H), 5.87 (s, 2H), 5.60 (s, 1H), 5.21 (s, 1H), 3.57 (s, 3H), 3.27 (s, 6H), –2.58 (s, 2H); ^{13}C NMR ($CDCl_3$): δ 159.06, 159.00, 144.97, 141.35, 141.21, 138.60, 138.13, 135.58, 135.47, 133.27, 132.90, 132.18, 131.93, 129.75, 128.52, 128.35, 127.78, 127.41, 126.60, 125.75, 125.67, 124.05, 123.55, 117.02, 116.69, 116.54, 115.35, 114.95, 71.55, 33.71, 33.36; MS (ESI): m/e for $C_{78}H_{61}N_{14}O_3$ $[M+H]^+$ calcd. 1241.5, found 1241.5. HRMS (ESI) for $C_{78}H_{61}N_{14}O_3$ $[M+H]^+$: calcd. 1241.5051, found 1241.5081.

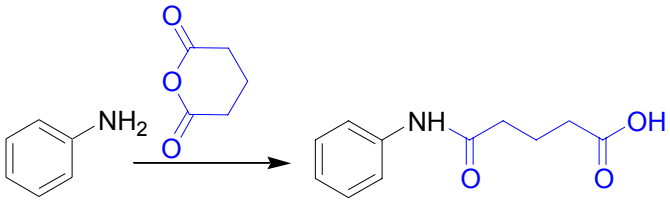
Characterization of 10b (prepared similarly to 10a): A solid; ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.83-8.98 (m, 8H), 8.48 (d, *J* = 7.0 Hz, 1H), 8.35 (d, *J* = 8.0 Hz, 2H), 8.20–8.34 (m, 2H), 8.13 (d, *J* = 7.5 Hz, 1H), 7.99 (d, *J* = 7.5 Hz, 2H), 7.82–7.85 (m, 3H), 7.53–7.61 (m, 3H), 7.35 (d, *J* = 7.0 Hz, 1H), 7.18 (t, *J* = 7.5 Hz, 1H), 6.85–7.00 (m, 17H), 6.64 (d, *J* = 8.5 Hz, 1H), 6.13 (s, 2H), 5.27 (s, 1H), 5.15 (s, 1H), 3.67 (s, 3H), 3.34 (s, 6H), 2.41 (t, *J* = 7.0 Hz, 2H), 1.95–1.99 (m, 4H), 1.47–1.51 (m, 2H), 1.16–1.28 (m, 4H), 0.79 (t, *J* = 7.5 Hz, 3H), 0.61(t, *J* = 7.5 Hz, 6H), –2.61 (s, 2H); ^{13}C NMR ($CDCl_3$): δ 159.77, 159.45, 152.52, 145.17, 138.82, 138.14, 135.88, 135.76, 135.23, 129.54, 128.56, 128.29, 127.82, 127.55, 126.57, 125.48, 124.06, 116.75, 116.59, 116.38, 115.23, 71.53, 53.50, 31.69, 28.43, 20.83, 20.26, 13.85, 13.70; MS (ESI): m/e for $C_{87}H_{79}N_{14}O_3$ $[M+H]^+$ 1367.8; HRMS (ESI) for $C_{87}H_{79}N_{14}O_3$ $[M+H]^+$: calcd. 1367.6460, found 1364.6467.



10 (**10a**: R=H; **10b**: R=Pr) **11** (**11a**: R=H; **11b**: R=Pr)

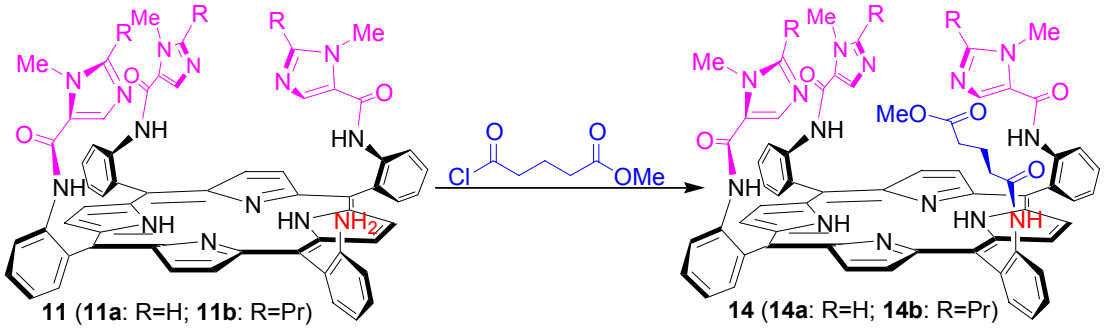
Preparation of Compound 11a: To a solution of compound **10a** (100 mg, 0.08 mmol) in dichloromethane (50 mL) was added trifluoroacetic acid (100 μL). The solution was stirred at room temperature for 30 min. After washed with saturated $NaHCO_3$ water solution and dried over Na_2SO_4, the dichloromethane solution was concentrated. The residue was purified by chromatography on silica gel with eluent CH_3OH/CH_2Cl_2 (40% saturated with NH_3 gas) = 5/95 to give compound **11a** (75 mg, 94%) as a solid. ^1H NMR (500 MHz, $CDCl_3$): δ 8.86–8.94 (m, 8H), 8.56 (d, J = 7.5 Hz, 2H), 8.44 (d, J = 7.5 Hz, 1H), 8.12–8.15 (m, 3H), 8.07 (s, 1H), 7.95 (s, 2H), 7.82–7.86 (m, 4H), 7.55–7.60 (m, 4H), 7.12 (t, J = 7.0 Hz, 1H), 7.07 (d, J = 8.0 Hz, 1H), 6.95 (s, 2H), 6.91 (s, 1H), 5.50 (s, 1H), 5.39 (s, 2H), 3.57 (s, 3H), 3.49 (s, 6H), 3.29 (s, br, 2H, NH_2), -2.56 (s, 2H); ^{13}C NMR ($CDCl_3$): δ 158.99, 158.41, 147.44, 141.24, 138.37, 138.27, 135.45, 135.24, 134.43, 133.07, 132.18, 132.00, 131.09, 129.97, 129.84, 129.71, 125.97, 125.82, 125.63, 124.03, 123.78, 123.60, 122.20, 117.82, 117.15, 115.37, 114.81, 114.59, 33.74, 33.60; MS (ESI): m/e for $C_{59}H_{47}N_{14}O_3$ $[M+H]^+$ 999.4; HRMS (ESI) for $C_{59}H_{47}N_{14}O_3$ $[M+H]^+$: calcd. 999.3956, found 999.3908.

Characterization of 11b (prepared similarly to 11a): A solid; ^1H NMR (500 MHz, $CDCl_3$): δ 8.81–8.94 (m, 8H), 8.57 (d, J = 8.0 Hz, 2H), 8.39 (d, J = 8.0 Hz, 1H), 8.20 (s, 1H), 8.09–8.14 (m, 3H), 8.05 (s, 2H), 7.81–7.85 (m, 4H), 7.55–7.60 (m, 4H), 7.09–7.13 (m, 2H), 5.47 (s, 2H), 5.35 (s, 1H), 3.54 (s, 5H, CH_3+NH_2), 3.52 (s, 6H), 2.19–2.24 (m, 6H), 1.27–1.33 (m, 6H), 0.63–0.68 (m, 9H), –2.55 (s, 2H); ^{13}C NMR ($CDCl_3$): δ 159.54, 159.02, 152.56, 152.46, 147.60, 138.63, 138.48, 135.54, 135.39, 134.45, 133.34, 132.13, 130.94, 130.42, 129.93, 129.75, 129.58, 126.14, 125.67, 125.38, 124.04, 123.96, 123.53, 122.10, 117.74, 117.10, 115.44, 114.98, 114.67, 31.77, 31.64, 28.44, 20.49, 20.46, 13.68; MS (ESI): m/e for $C_{68}H_{65}N_{14}O_3$ $[M+H]^+$ 1125.5; HRMS (ESI) for $C_{68}H_{65}N_{14}O_3$ $[M+H]^+$: calcd. 1125.5364, found 1125.5347.



12 **13**

Preparation of Compound 13: Pure phenylamine (200 µL, 2.2 mmol) was stirred in dry CH2Cl2 (30 mL). Glutaric anhydride (280 mg, 2.5 mmol) was added followed by NEt_3 (0.9 mL). The reaction mixture was stirred at room temperature for 4 h. The mixture was concentrated and purified by column chromatography on silica gel with eluent CH_3OH/CH_2Cl_2 = 5/95 to give compound **13** (400 mg, 88%) as a solid. ^{1}H NMR (500 MHz, DMSO-d6): the carboxylic acid proton was not assigned, δ 9.89 (s, 1H), 7.56 (d, *J* = 8.0 Hz, 2H), 7.26 (t, *J* = 8.0 Hz, 2H), 7.00 (t, *J* = 8.0 Hz, 1H), 2.33 (t, *J* = 7.5 Hz, 2H), 2.27 (t, *J* = 7.5 Hz, 2H), 1.79 (m, 2H); ^{13}C NMR (DMSO-d6): 170.81, 139.31, 128.71, 123.04, 119.10, 35.45, 20.56; MS (ESI): m/e = 207.9 for $C_{11}H_{14}NO_3$ $[M+H]^+$; HRMS (ESI) for $C_{11}H_{13}NO_3Na$ $[M+Na]^+$: calcd. 230.0793, found 230.0782.

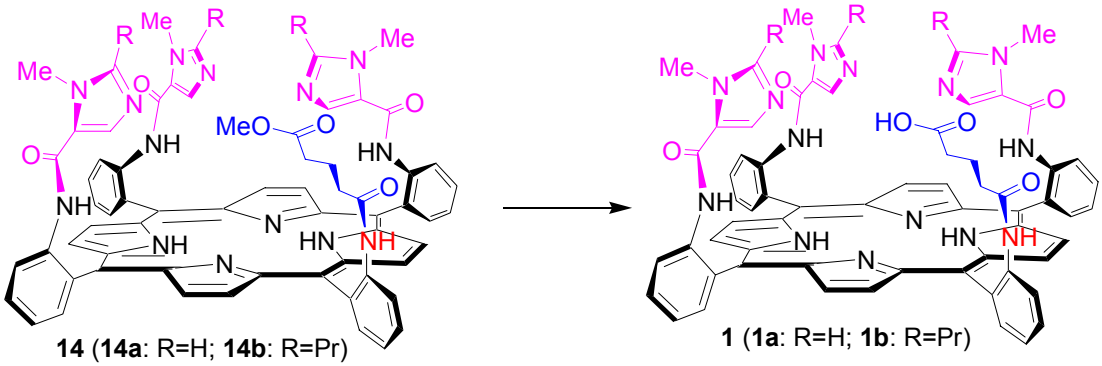


11 (**11a**: R=H; **11b**: R=Pr) **14** (**14a**: R=H; **14b**: R=Pr)

Preparation of Compound 14a: To a stirred solution of **11a** (200 mg, 0.2 mmol) in dry THF (30 mL), was added *N*, *N*-diethylaniline (0.16 mL, 1.0 mmol)) followed by methyl 5-chloro-5-oxovalerate (138 mL, 1.0 mmol). The mixture was stirred at room temperature for 1 h. After removal of THF solvent, the residue was re-dissolved in CH_2Cl_2 (60 mL). The solution was washed with saturated $NaHCO_3$ water solution. CH_2Cl_2 layer was dried over Na_2SO_4 and then concentrated. The residue was purified by chromatography on silica gel with eluent CH_3OH/CH_2Cl_2 (40% saturated with NH_3 gas) = 8/95 to give compound **14a** (195 mg, 86%) as a solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.85–8.91 (m, 8H), 8.54 (d, *J* = 8.0 Hz, 2H), 8.37 (d, *J* = 8.0 Hz, 1H), 8.32 (d, *J* = 8.5 Hz, 1H), 8.25 (s, 1H), 8.09-8.12 (m, 3H), 7.99 (s, 1H), 7.91–7.94 (m, 3H),

7.83–7.86 (m, 3H), 7.78 (t, J = 7.5 Hz, 1H), 7.56–7.61 (m, 3H), 7.49 (t, J = 7.0 Hz, 1H), 7.02 (s, 2H), 6.94 (s, 1H), 5.61 (s, 1H), 5.16 (s, 2H), 3.66 (s, 6H), 3.58 (s, 3H), 2.79 (s, 3H), 1.49–1.55 (m, 4H), 1.20 (t, J = 7.5 Hz, 2H), –2.55 (s, 2H); ^{13}C NMR ($CDCl_3$): δ 173.03, 171.80, 159.09, 158.51, 141.35, 141.22, 138.46, 138.20, 135.42, 135.20, 135.02, 133.45, 133.38, 132.18, 131.79, 131.61, 129.86, 129.71, 129.55, 125.78, 125.68, 124.79, 124.22, 124.12, 123.83, 122.47, 116.31, 115.22, 115.00, 50.83, 34.90, 33.76, 32.18, 23.89, 20.23; MS (ESI): m/e 1127.5 for $C_{65}H_{55}N_{14}O_6$ $[M+H]^+$; HRMS (ESI) for $C_{65}H_{55}N_{14}O_6$ $[M+H]^+$ calcd. 1127.4429, found 1127.4425.

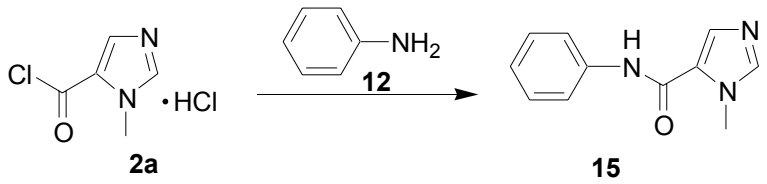
Characterization of 14b (prepared similarly to 14a): A solid; ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.85–8.91 (m, 8H), 8.57–8.59 (d, J = 8.0 Hz, 2H), 8.45 (s, 1H), 8.37 (s, 1H), 8.33 (d, J = 8.5 Hz, 1H), 8.18 (d, J = 8.5 Hz, 1H), 8.00–8.08 (m, 4H), 7.99 (d, 7.5 Hz, 2H), 7.77–7.85 (m, 4H), 7.50–7.59 (m, 4H), 5.50 (s, 1H), 5.01 (s, 2H), 3.66 (s, 6H), 3.57 (s, 3H), 2.37 (s, 3H), 2.29 (t, J = 8.0 Hz, 4H), 2.19 (t, J = 7.5 Hz, 2H), 1.57 (t, J = 6.5 Hz, 2H), 1.34–1.38 (m, 4H), 1.26 (t, J = 7.5 Hz, 2H), 1.15 (t, J = 7.5 Hz, 2H), 0.92 (t, J = 7.5 Hz, 2H), 0.73 (t, J = 7.5 Hz, 6H), 0.65 (t, J = 7.5 Hz, 3H), –2.50 (s, 2H); ^{13}C NMR ($CDCl_3$): δ 172.49, 171.89, 159.67, 159.06, 152.68, 152.43, 138.69, 138.59, 138.33, 135.35, 134.77, 134.66, 133.88, 132.19, 130.84, 129.65, 129.45, 125.71, 125.54, 125.37, 124.73, 124.17, 123.62, 122.62, 116.39, 115.26, 115.09, 50.40, 34.79, 31.77, 28.51, 28.45, 20.58, 20.38, 20.05, 13.70, 13.63; MS (ESI): m/e 1253.6 for $C_{74}H_{73}N_{14}O_6$ $[M+H]^+$; HRMS (ESI) for $C_{74}H_{73}N_{14}O_6$ $[M+H]^+$ calcd. 1253.5838, found 1253.5844.



Preparation of Compound 1: Compound **14a** (85 mg, 0.075 mmol) was dissolved in a mixed solvent of CH_3OH/THF (20 mL, v/v=1/1). Potassium hydroxide (1.0 g) water solution (10 mL) was added. The mixture was stirred at room temperature for 2 h. After removal of organic solvent under vacuum, the mixture was extracted with

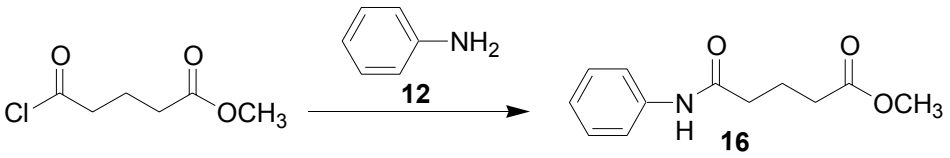
CH_2Cl_2 and washed with saturated $NaHCO_3$ solution. The organic extract was dried over Na_2SO_4 and concentrated. The residue was purified by chromatography on silica with eluent CH_3OH/CH_2Cl_2 (saturated with NH_3 gas)=20/80 to give compound **1a** (73 mg, 87%) as a solid. ^{1}H NMR (500 MHz, $CDCl_3$): the carboxylic acid proton is not assigned, δ 8.77–8.94 (m, 8H), 8.71 (d, *J* = 8.0 Hz, 1H), 8.38 (d, *J* = 5.5 Hz, 2H), 8.23 (d, *J* = 7.0 Hz, 1H), 8.14 (d, *J* = 8.0 Hz, 1H), 8.07 (s, 1H), 8.03 (d, *J* = 7.0 Hz, 1H), 7.95 (d, *J* = 5.5 Hz, 2H), 7.78-7.88 (m, 5H), 7.61 (t, *J* = 7.5 Hz, 1H), 7.52–7.57 (m, 3H), 7.30 (s, 1H), 6.96 (s, 2H), 6.75 (s, 1H), 5.58 (s, 2H), 4.21 (s, 1H), 3.64 (s, 6H), 3.53 (s, 3H), 1.61 (t, *J* = 6.5 Hz, 2H), 1.12 (t, *J* = 6.5 Hz, 2H), 0.84–0.87 (m, 4H), –2.59 (s, 2H); ^{13}C NMR ($CDCl_3$): δ 174.68, 172.15, 159.27, 157.35, 140.83, 140.27, 138.71, 138.59, 138.23, 136.14, 134.91, 134.34, 132.15, 131.40, 130.34, 130.12, 129.63, 129.50, 128.41, 126.25, 125.98, 125.43, 124.43, 123.96, 123.58, 120.27, 116.81, 113.22, 35.33, 33.73, 33.62, 32.32, 29.75, 20.29; MS (ESI): m/e 1113.4 for $C_{64}H_{53}N_{14}O_6$ $[M+H]^+$; HRMS (ESI) for $C_{64}H_{53}N_{14}O_6$ $[M+H]^+$ calcd. 1113.4273, found 1113.4279.

Characterization of 1b (prepared similarly to 1a): A solid; ^{1}H NMR (500 MHz, $CDCl_3$): the carboxylic acid proton is not assigned, δ 8.79–8.95 (m, 9H), 8.44 (s, 2H), 8.40 (s, 2H), 8.14 (d, *J* = 7.0 Hz, 1H), 8.07 (d, *J* = 7.5 Hz, 1H), 8.03 (d, *J* = 8.5 Hz, 1H), 7.95 (s, 2H), 7.79–7.88 (m, 5H), 7.55–7.59 (m, 3H), 7.51 (t, *J* = 7.5 Hz, 2H), 5.60 (s, 2H), 4.51 (s, 1H), 3.69 (s, 6H), 3.54 (s, 3H), 2.31–2.38 (m, 4H), 2.16 (t, *J* = 7.5 Hz, 2H), 1.60 (t, *J* = 6.5 Hz, 2H), 1.43–1.45 (m, 4H), 1.12–1.17 (m, 2H), 0.78–0.81 (m, 8H), 0.56 (t, *J* = 7.5 Hz, 3H), 0.46 (t, *J* = 6.0 Hz, 2H), –2.56 (s, 2H); ^{13}C NMR ($CDCl_3$): δ 174.67, 172.38, 159.88, 157.94, 152.41, 151.91, 138.81, 138.59, 138.41, 136.14, 135.00, 134.50, 131.33, 131.14, 129.95, 129.47, 127.52, 127.02, 125.91, 125.27, 124.70, 123.74, 123.38, 120.26, 116.99, 113.23, 35.65, 32.41, 31.86, 31.64, 28.58, 28.05, 20.74, 20.27, 20.11, 13.77, 13.52; MS (ESI): m/e 1239.6 for $C_{73}H_{71}N_{14}O_6$ $[M+H]^+$; HRMS (ESI) for $C_{73}H_{71}N_{14}O_6$ $[M+H]^+$ calcd. 1239.5681, found 1239.5709.



2a **15**

Preparation of Compound 15: Freshly prepared imidazole acid chloride hydrochloride **2a** (165 mg, 1.0 mmol) was stirred in dry CH_3CN (10 mL). Aniline (180 μL, 2.0 mmol) was added via a syringe followed by pyridine (240 μL. 3.0 mmol). The reaction was stirred for further 3 h under N_2 atmosphere at room temperature. After removal of solvent, the residue was dissolved in CH_2Cl_2 and washed with $NaHCO_3$ solution. The organic extract was concentrated and purified by chromatography on silica gel with eluent CH_3OH/CH_2Cl_2 =3/97 to give compound **15** (122 mg, 61%) as a solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.18 (s, 1H), 7.60 (s, 1H), 7.54 (d, *J* = 7.0 Hz, 2H), 7.52 (s, 1H), 7.33 (t, *J* = 7.0 Hz, 2H), 7.12 (t, *J* = 7.0 Hz, 1H), 3.93 (s, 3H); ^{13}C NMR ($CDCl_3$): δ 158.60, 142.22, 137.54, 132.14, 129.16, 124.69, 120.44, 34.30; MS (ESI): m/e 201.9 for $C_{11}H_{12}N_3O$ $[M+H]^+$; HRMS (ESI) for $C_{11}H_{12}N_3O$ $[M+H]^+$ calcd. 202.0980, found 202.0987.



Preparation of Compound 16: To a stirred solution of aniline **12** (0.30 mL, 3.25 mmol) in dry THF (20 mL) was added methyl 5-chloro-5-oxovalerate (0.20 mL, 1.45 mmol) via a syringe followed by pyridine (0.46 mL, 5.8 mmol). The reaction was stirred for further 2 h under N_2 atmosphere at room temperature. After removal of solvent, the residue was dissolved in CH_2Cl_2 and washed with $NaHCO_3$ solution. The organic extract was concentrated and purified by chromatography on silica gel with eluent CH_3OH/CH_2Cl_2 =3/97 to give compound **16** (262 mg, 82%) as a solid. A solid; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.75 (s, 1H), 7.48 (d, *J* = 8.0 Hz, 2H), 7.27 (t, *J* = 7.5 Hz, 2H), 7.06 (t, *J* = 7.5 Hz, 1H), 3.64 (s, 3H), 2.37–2.41 (m, 4H), 2.01 (p, *J* = 7.5 Hz, 2H); ^{13}C NMR ($CDCl_3$): δ 174.11, 170.94, 138.17, 129.21, 124.49, 120.12, 51.97, 36.58, 33.22, 21.02; MS (ESI): m/e 221.9 for $C_{12}H_{16}NO_3$ $[M+H]^+$; HRMS (ESI) for $C_{12}H_{16}NO_3$ $[M+H]^+$ calcd. 222.1130, found 222.1124.

Reference:

1. O'Connell, J. F.; Parquette, J.; Yelle, W. E.; Wang, W.; Rapoport, H. *Synthesis*, **1988**, 767.

Figure s1: ^{1}H NMR spectrum of compound **14a** in $CDCl_3$

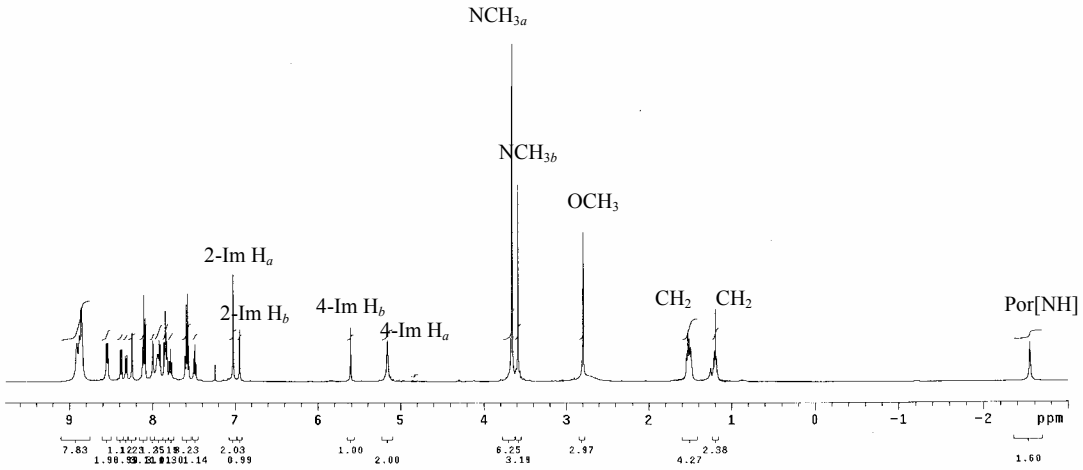
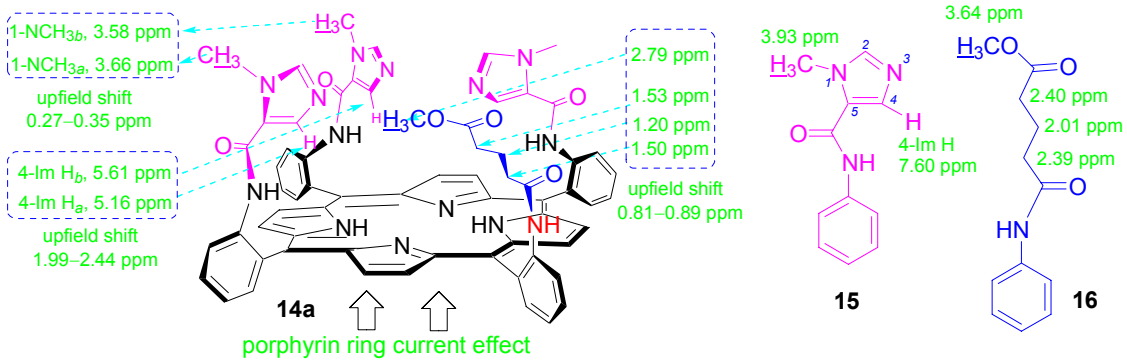
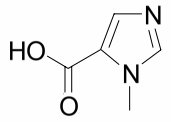


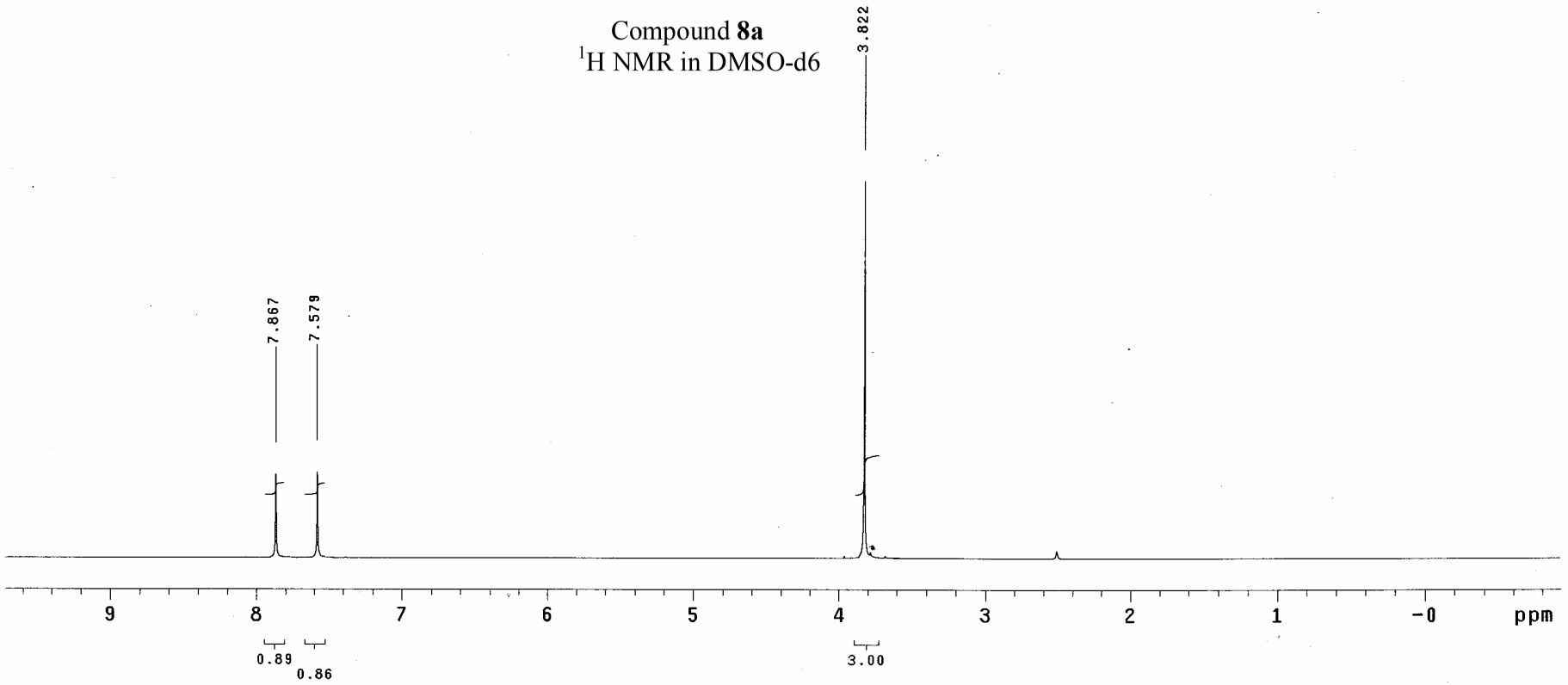
Figure s2: ^{1}H NMR chemical shifts for porphyrin compound **14a** and its non-porphyrin analogues **15**, **16** in $CDCl_3$

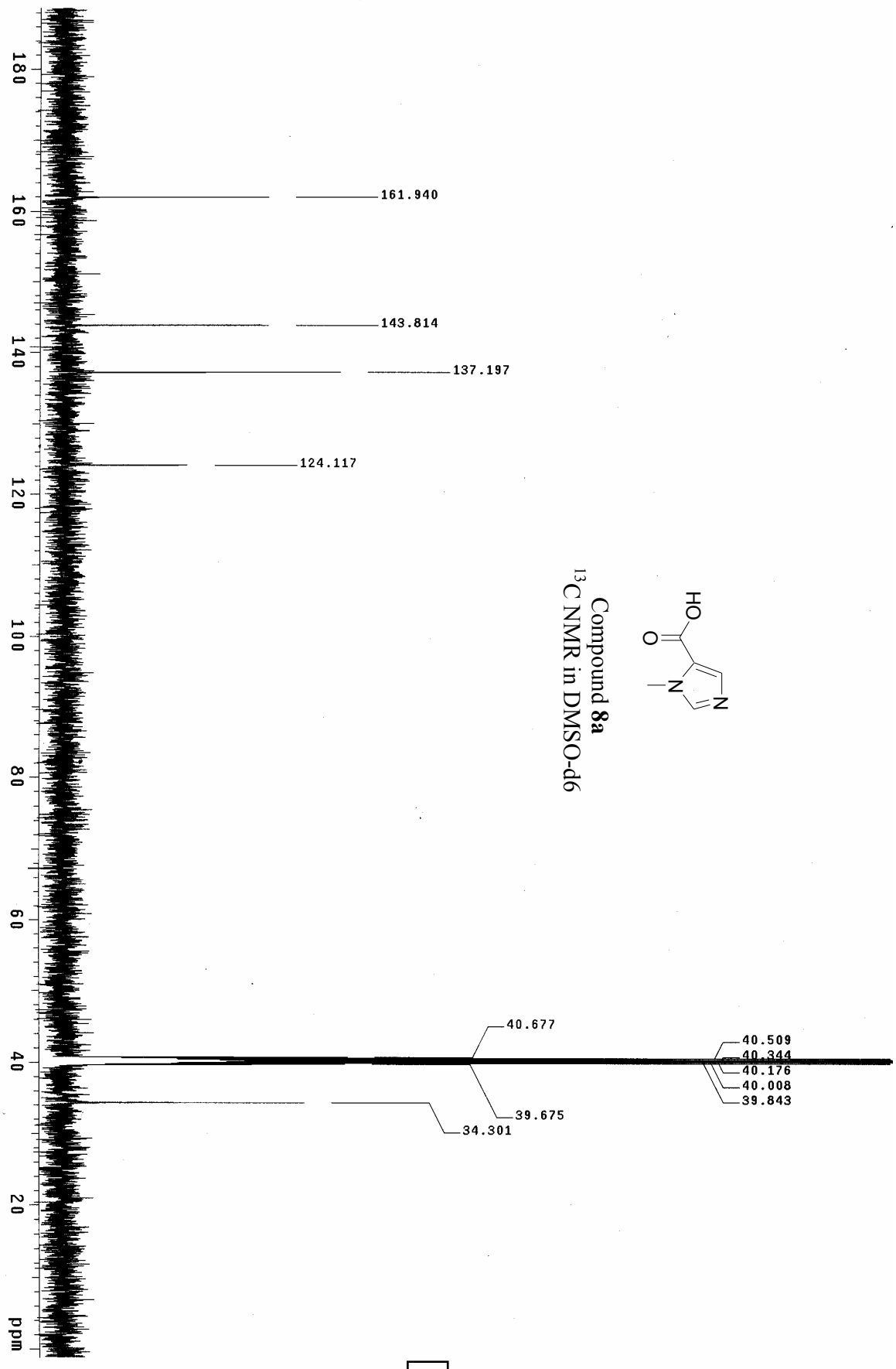


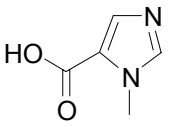
Interpretation of Figure s1 and s2: Compared to those of non-porphyrin analogues, the chemical shifts of substituents closer to the porphyrin ring center are strongly shifted upfield due to the ring current effect of the porphyrin ring. In compound **14a**, the diagnostic imidazole proton 4-Im H strongly shifts upfield 1.99–2.44 ppm when compared with its non-porphyrin analogue **15**, which is much more than that of imidazole 1-NCH_3 (0.27–0.35 ppm), suggesting that 4-Im H is close to the porphyrin center while 1-NMe is located at the porphyrin periphery. The upfield shift (0.81–0.89 ppm) of glutaric methyl ester group on compound **14a** compared to its analogue **16** implies that this glutaric moiety is suspended over the porphyrin plane.



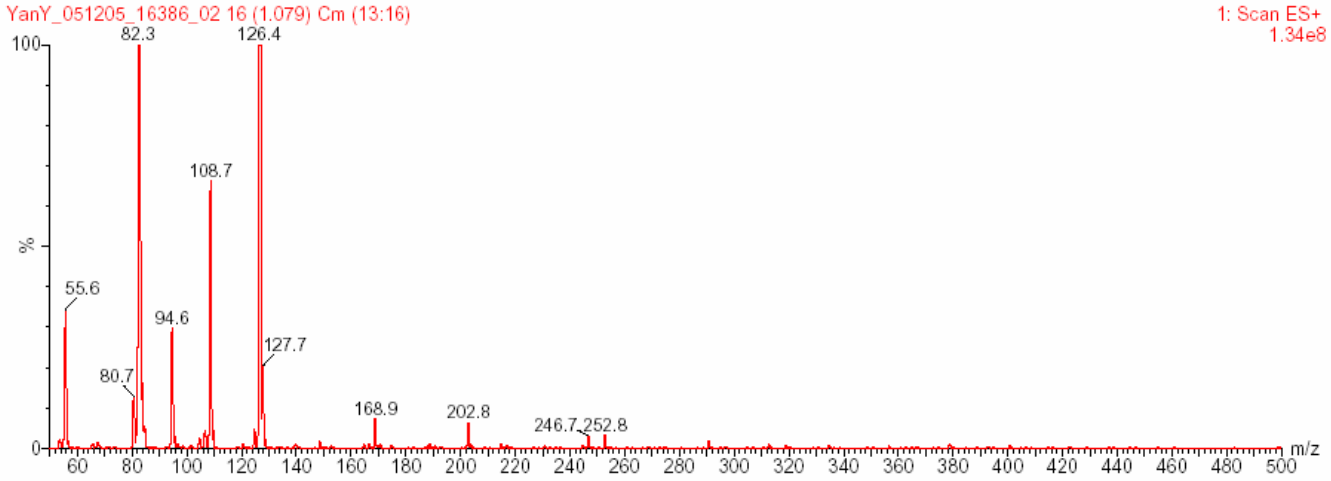
Compound **8a**
^{1}H NMR in DMSO-d6




Compound 8a
13C NMR in DMSO-d6
HO
O
N
N
161.940
143.814
137.197
124.117
40.677
40.509
40.344
40.176
40.008
39.843
39.675
34.301
180
160
140
120
100
80
60
40
20
ppm



Compound **8a**, Low resolution mass analysis



Compound **8a**, High resolution mass analysis

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 120.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
126 formula(e) evaluated with 1 results within limits (up to 140 closest results for each mass)

Yilong Yan, Yan-5-23
Cone V = 10
Coll V = 10.0
07-Dec-2005
17:06:36
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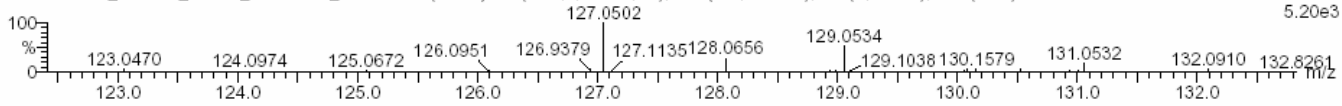
TOF MS ES+
5.20e3



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Maximum:		200.0	5.0	120.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
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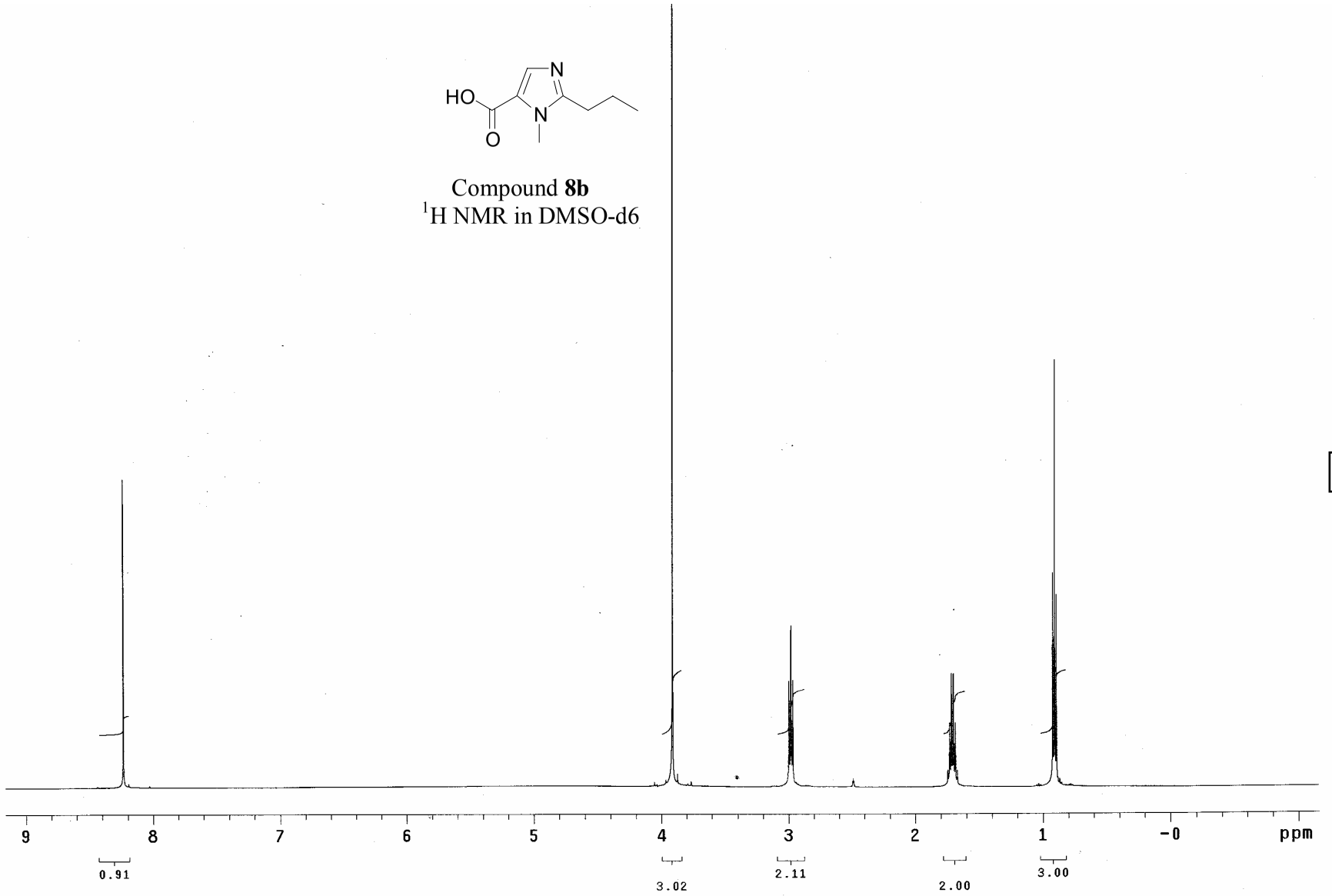
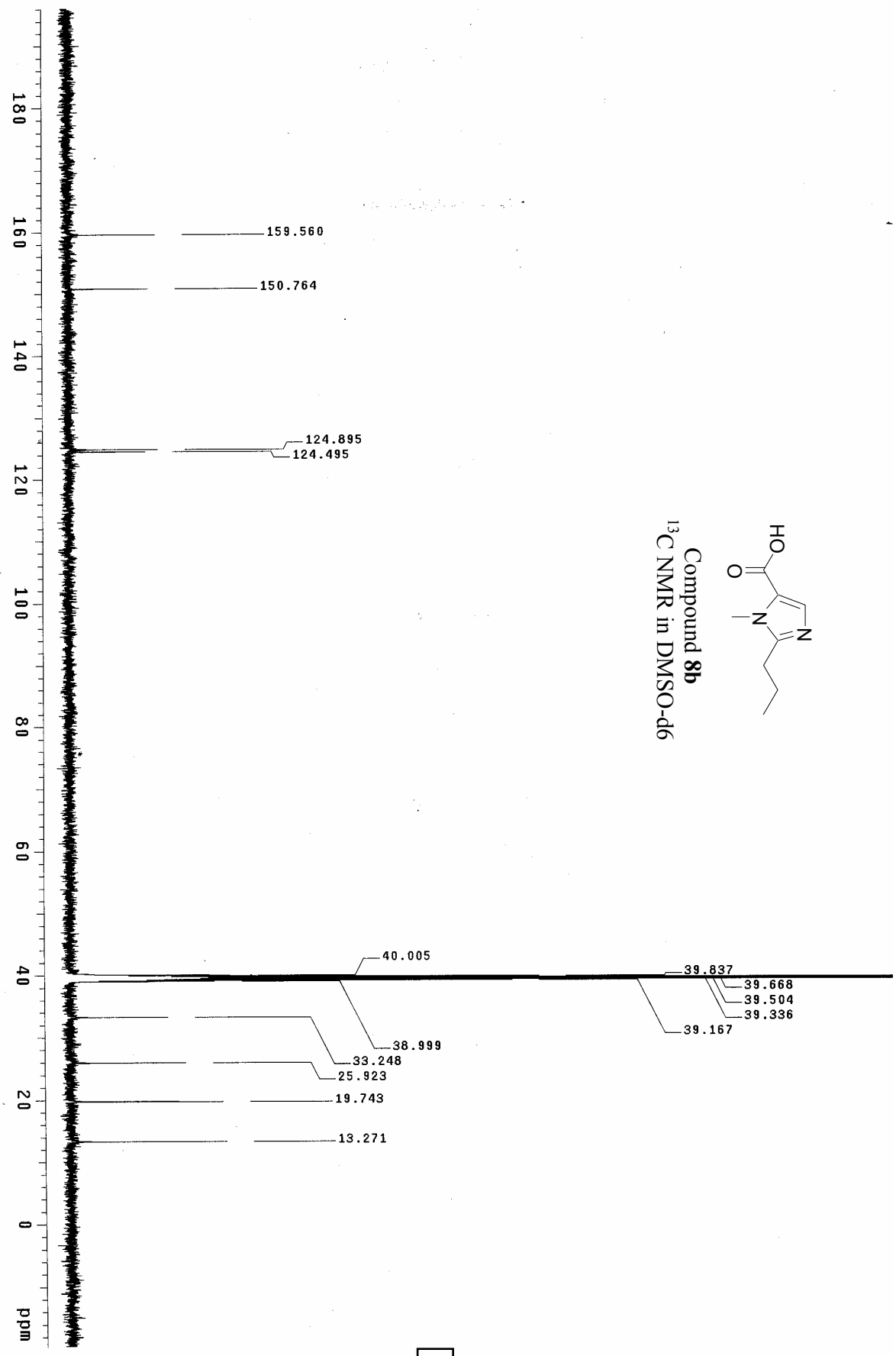
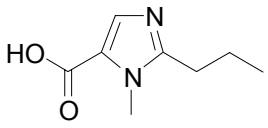
$[M+H]^+$


HO
O
N
N
Compound **8b**
^{1}H NMR in DMSO-d6
9
8
7
6
5
4
3
2
1
-0
ppm
0.91
3.02
2.11
2.00
3.00

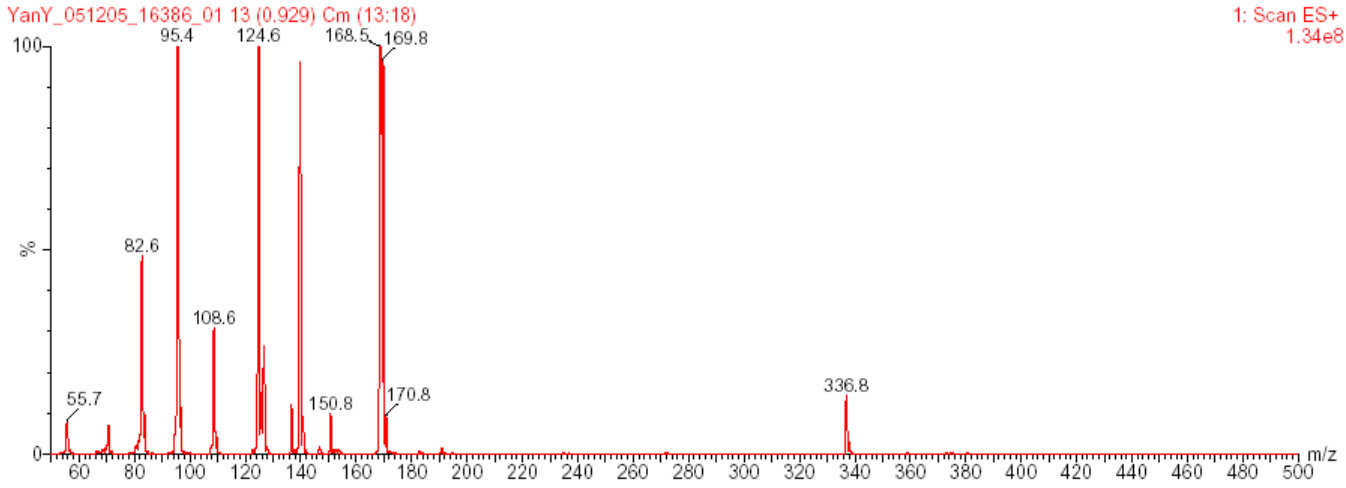
Compound **8b**

^{13}C NMR in DMSO-d6





Compound **8b**, Low resolution mass analysis



Compound **8b**, High resolution mass analysis

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 120.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
249 formula(e) evaluated with 1 results within limits (up to 140 closest results for each mass)

Yilong Yan, Yan-4-37
Cone V = 30
Coll V = 10.0
07-Dec-2005
16:55:32
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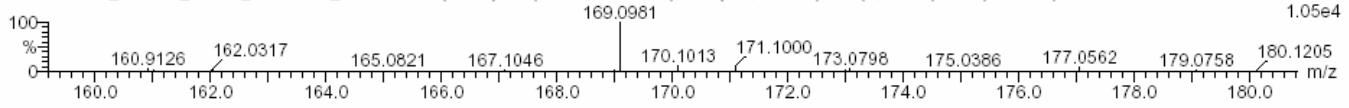
TOF MS ES+
1.05e4



Minimum:				-1.5		
Maximum:		200.0	5.0	120.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
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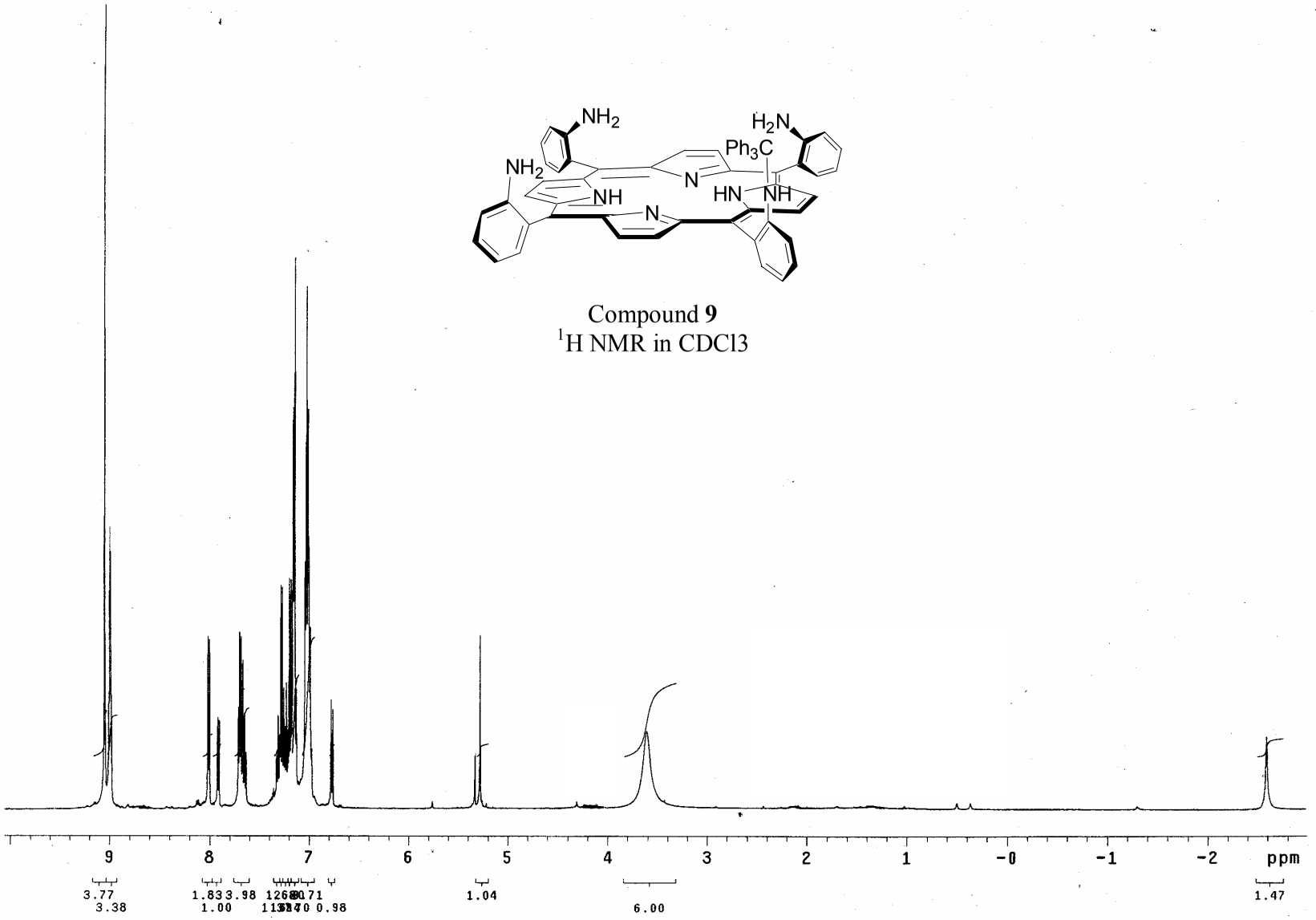
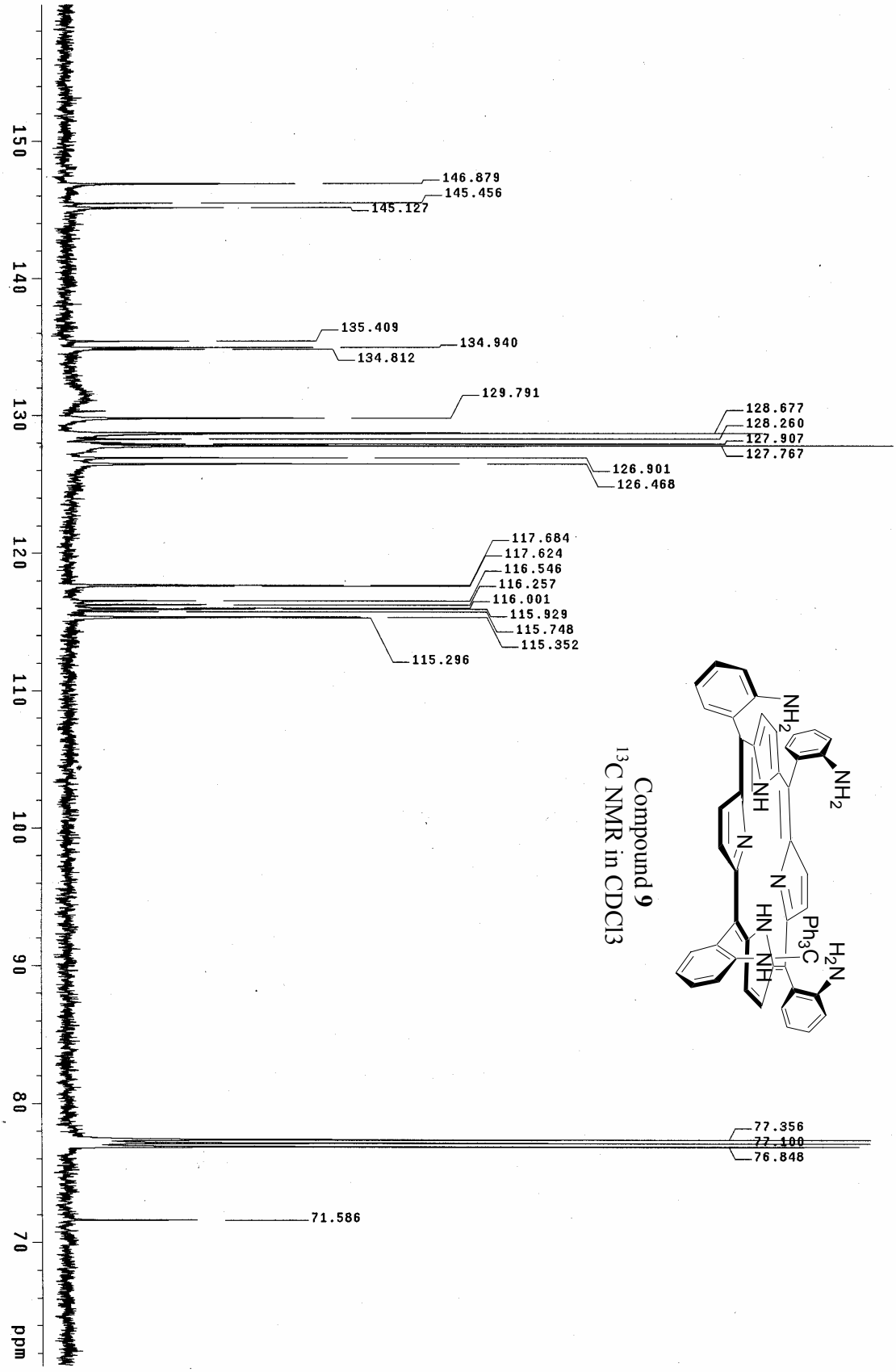
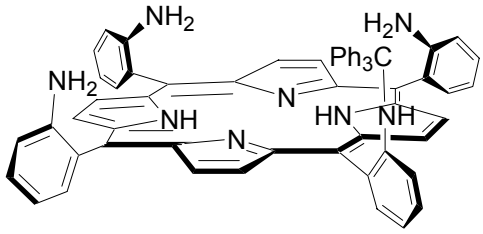
$[M+H]^+$

Compound **9**
^{1}H NMR in CDCl3

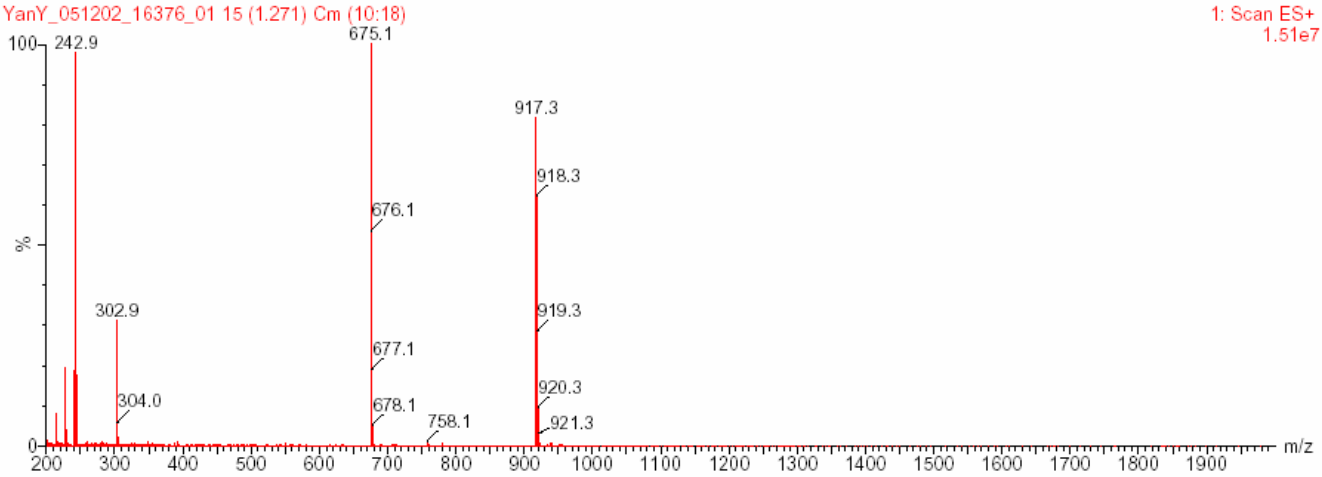




Compound **9**
^{13}C NMR in CDCl3



Compound **9**, Low resolution mass analysis



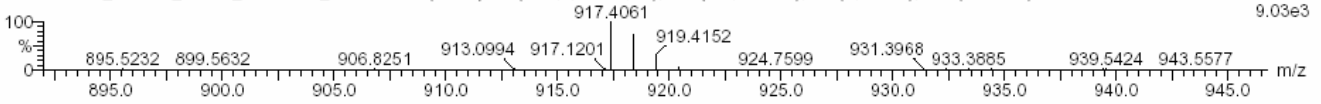
Compound **9**, High resolution mass analysis

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 120.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
2498 formula(e) evaluated with 25 results within limits (up to 140 closest results for each mass)

Yilong Yan, Yan-3-63 Cone V = 30 Coll V = 10.0 05-Dec-2005 16:43:18
YANYILONG_051205_16381_YAN-3-63_HRMS 141 (5.970) Cn (Cen,4, 80.00, Ar); Sm (SG, 2x3.00); Sb (2,20.00); Cm (136:142) TOF MS ES+ 9.03e3



Minimum:				-1.5		
Maximum:		200.0	5.0	120.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
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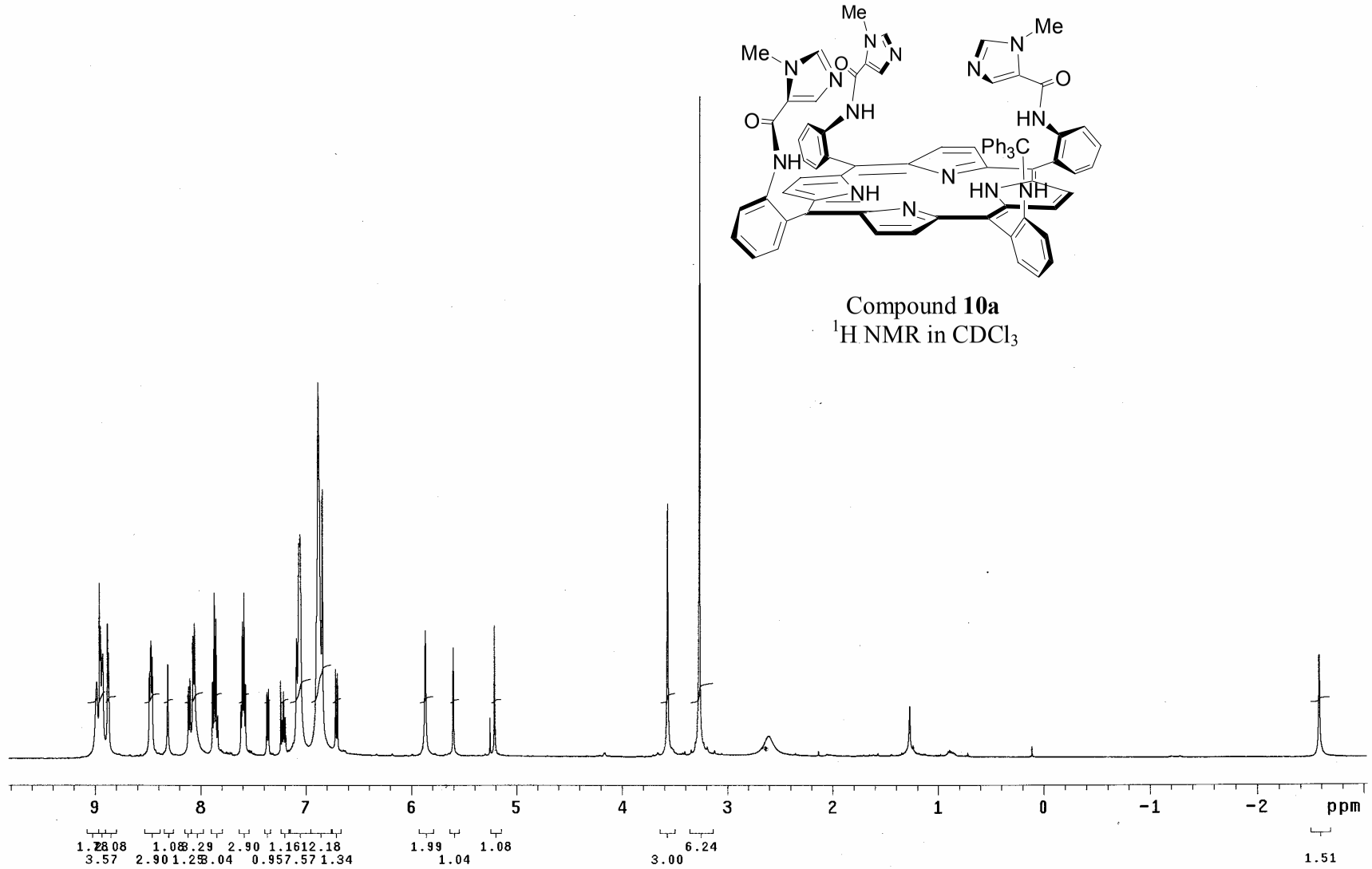
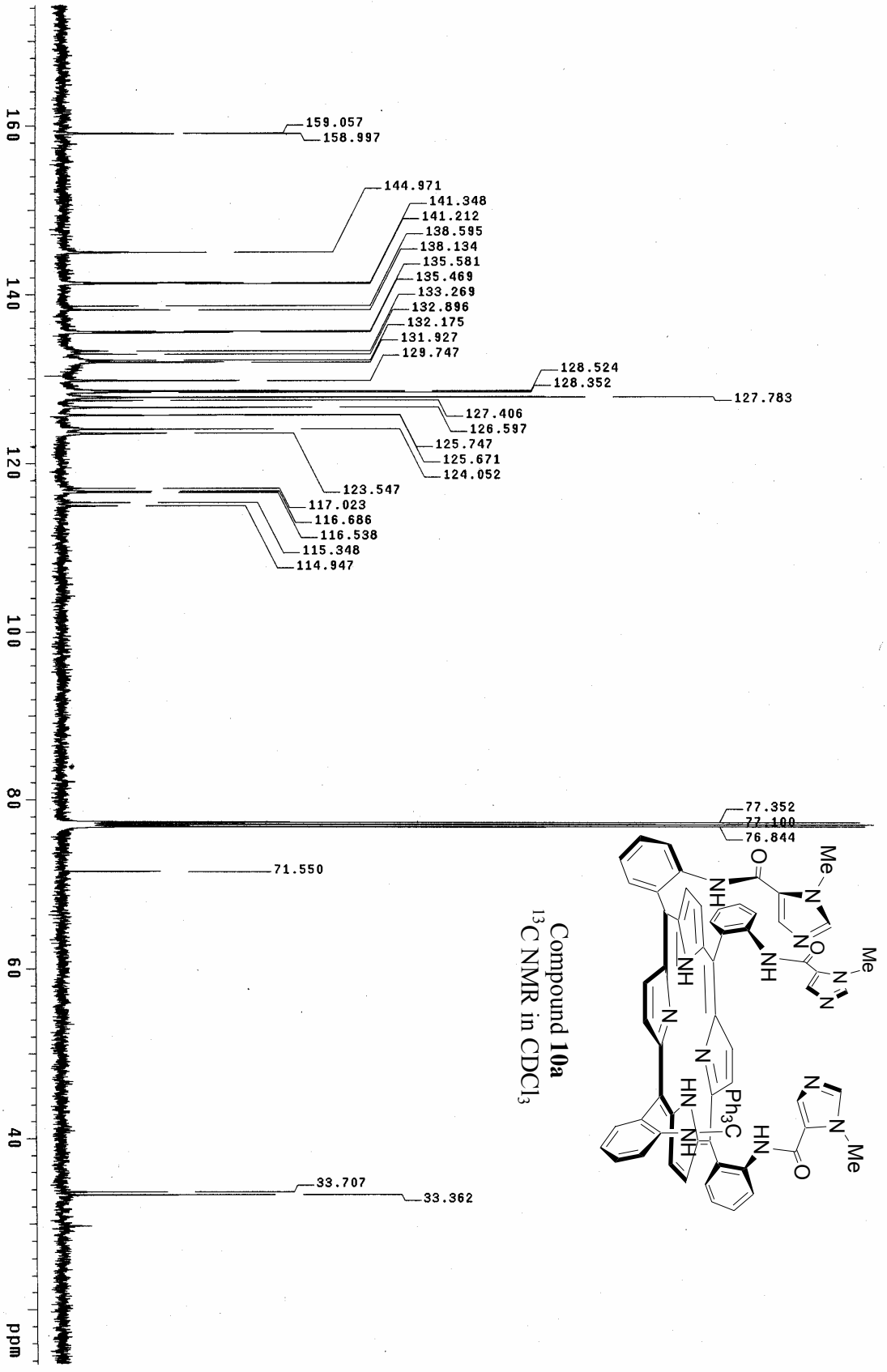
	917.4056	0.5	0.5	40.5	8	C61 H50 N8 Na
	917.4067	-0.6	-0.6	38.5	6	C62 H53 N4 O4
	917.4053	0.8	0.8	33.5	10	C61 H57 O8
	917.4053	0.8	0.8	39.0	9	C60 H51 N7 O3
	917.4070	-0.9	-0.9	40.0	5	C63 H52 N5 O Na
	917.4072	-1.1	-1.2	26.0	24	C48 H55 N9 O10
	917.4048	1.3	1.4	23.0	25	C46 H56 N9 O10 Na
	917.4075	-1.4	-1.5	27.5	23	C49 H54 N10 O7 Na
	917.4043	1.8	2.0	35.5	11	C60 H54 N4 O4 Na
	917.4080	-1.9	-2.1	43.5	4	C63 H49 N8
	917.4080	-1.9	-2.1	38.0	1	C64 H55 N O5
	917.4040	2.1	2.3	34.0	14	C59 H55 N3 O7
	917.4040	2.1	2.3	39.5	12	C58 H49 N10 O2
	917.4083	-2.2	-2.4	39.5	2	C65 H54 N2 O2 Na
	917.4088	-2.7	-3.0	27.0	22	C51 H56 N7 O8 Na
	917.4029	3.2	3.4	30.5	16	C59 H58 O8 Na
	917.4029	3.2	3.5	36.0	15	C58 H52 N7 O3 Na
	917.4094	-3.3	-3.6	43.0	3	C65 H51 N5 O
	917.4027	3.4	3.8	34.5	17	C57 H53 N6 O6
	917.4099	-3.8	-4.1	30.5	21	C51 H53 N10 O7
	917.4022	3.9	4.3	47.0	13	C71 H51 N
	917.4101	-4.0	-4.4	26.5	20	C53 H58 N4 O9 Na
	917.4016	4.5	4.9	31.0	19	C57 H56 N3 O7 Na
	917.4016	4.5	4.9	36.5	18	C56 H50 N10 O2 Na

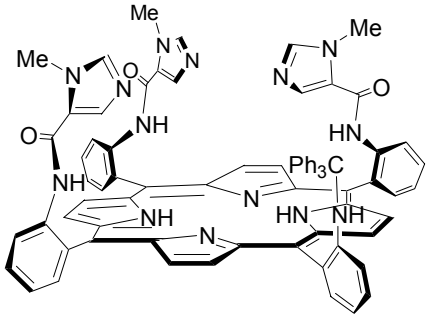
$[M+H]^+$



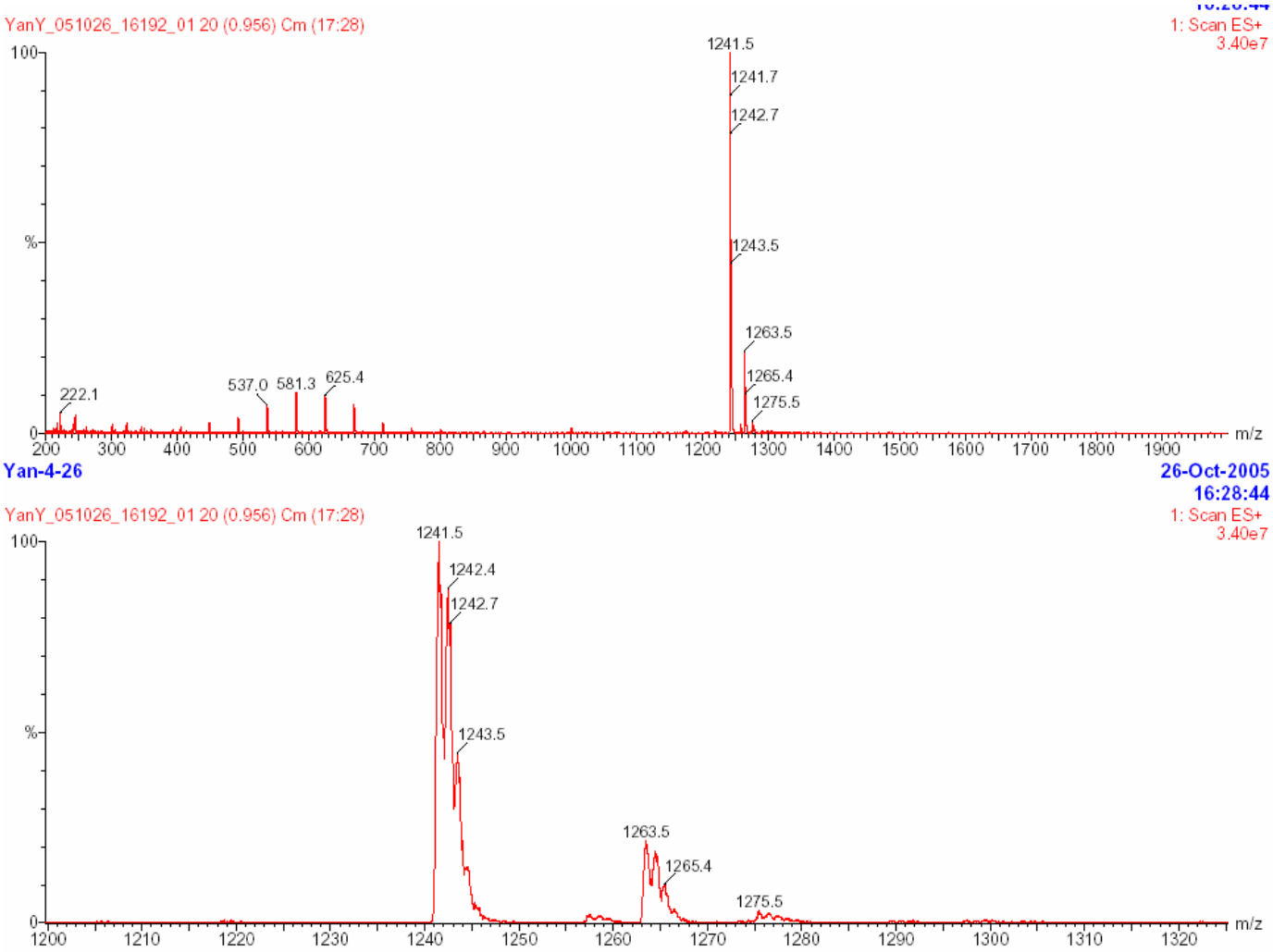
Compound **10a**
^{1}H NMR in $CDCl_3$

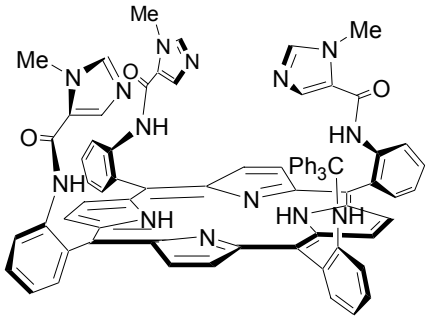


Compound **10a**
^{13}C NMR in $CDCl_3$



Compound **10a**, Low resolution mass analysis





Compound **10a**, High resolution mass analysis

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 120.0

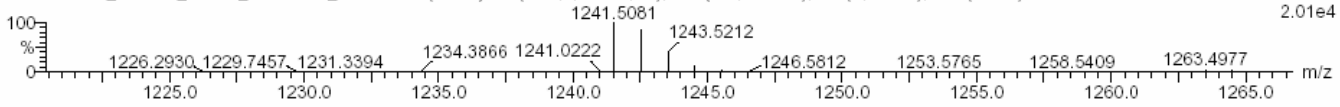
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

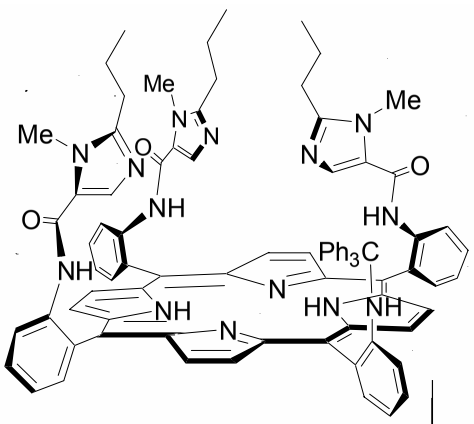
4929 formula(e) evaluated with 48 results within limits (up to 140 closest results for each mass)

Yilong Yan, Yan-4-26 | Cone V = 30 | Coll V = 10.0 | 05-Dec-2005 17:29:47 | TOF MS ES+ 2.01e4

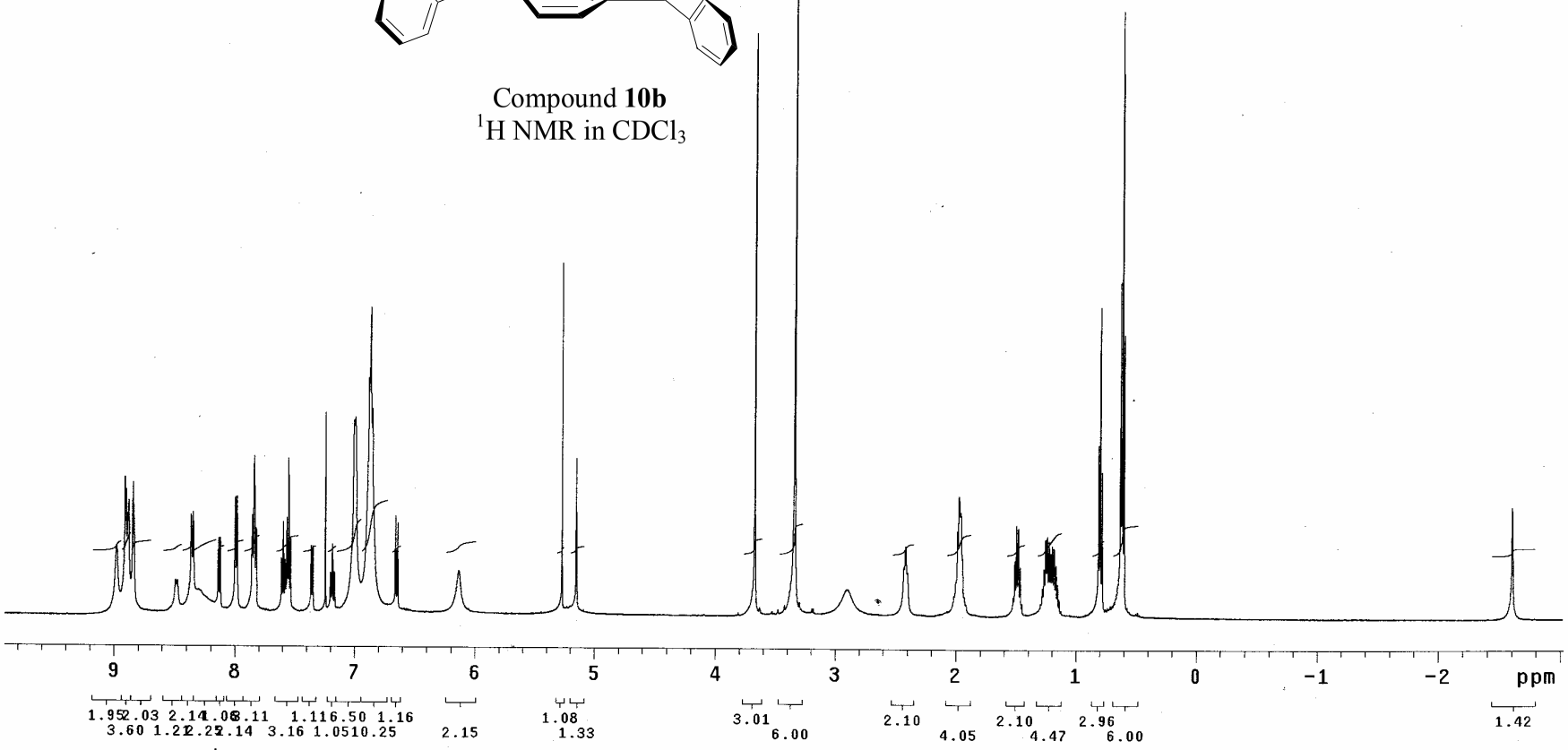
YANYILONG_051205_16381_YAN-4-26_HRMS 25 (1.064) Cn (Cen,4, 80.00, Ar); Sm (SG, 2x3.00); Sb (2,20.00); Cm (22:28)

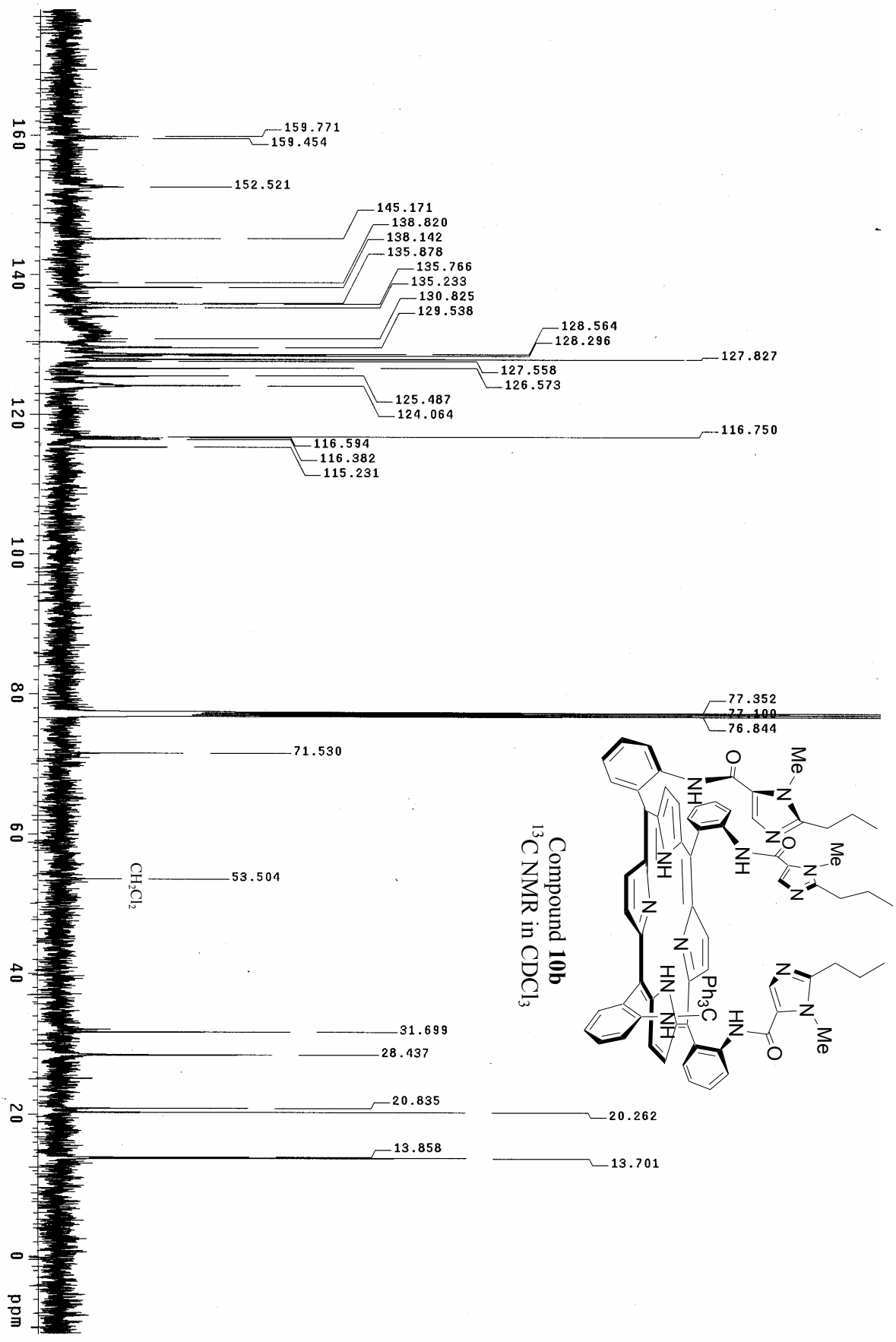


Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
Minimum:				-1.5		
Maximum:		200.0	5.0	120.0		
1241.5081	1241.5081	0.0	0.0	50.5	28	C84 H70 N2 O7 Na
	1241.5081	0.0	0.0	56.0	30	C83 H64 N9 O2 Na
	1241.5078	0.3	0.2	49.0	25	C83 H71 N O10
	1241.5078	0.3	0.2	54.5	27	C82 H65 N8 O5
	1241.5078	0.3	0.3	60.0	29	C81 H59 N15
	1241.5086	-0.5	-0.4	43.5	11	C69 H66 N14 O8 Na
	1241.5086	-0.5	-0.4	66.5	48	C98 H65
	1241.5091	-1.0	-0.8	59.5	32	C83 H61 N12 O
	1241.5091	-1.0	-0.8	54.0	31	C84 H67 N5 O6
	1241.5094	-1.3	-1.1	55.5	33	C85 H66 N6 O3 Na
	1241.5067	1.4	1.1	51.0	24	C82 H68 N5 O6 Na
	1241.5067	1.4	1.1	56.5	26	C81 H62 N12 O Na
	1241.5065	1.6	1.3	49.5	21	C81 H69 N4 O9
	1241.5065	1.6	1.3	55.0	23	C80 H63 N11 O4
	1241.5099	-1.8	-1.5	43.0	6	C71 H68 N11 O9 Na
	1241.5062	1.9	1.5	63.5	47	C96 H66 Na
	1241.5105	-2.4	-1.9	59.0	35	C85 H63 N9 O2
	1241.5105	-2.4	-1.9	53.5	34	C86 H69 N2 O7
	1241.5108	-2.7	-2.1	55.0	36	C87 H68 N3 O4 Na
	1241.5054	2.7	2.2	46.0	18	C81 H72 N O10 Na
	1241.5054	2.7	2.2	51.5	20	C80 H66 N8 O5 Na
	1241.5054	2.7	2.2	57.0	22	C79 H60 N15 Na
	1241.5110	-2.9	-2.3	46.5	3	C71 H65 N14 O8
	1241.5051	3.0	2.4	50.0	17	C79 H67 N7 O8
	1241.5051	3.0	2.4	55.5	19	C78 H61 N14 O3 ← $[M+H]^+$
	1241.5113	-3.2	-2.5	48.0	4	C72 H64 N15 O5 Na
	1241.5113	-3.2	-2.5	42.5	1	C73 H70 N8 O10 Na
	1241.5046	3.5	2.8	62.5	46	C93 H65 N2 O2
	1241.5118	-3.7	-3.0	58.5	37	C87 H65 N6 O3
	1241.5121	-4.0	-3.2	60.0	39	C88 H64 N7 Na
	1241.5121	-4.0	-3.2	54.5	38	C89 H70 O5 Na
	1241.5041	4.0	3.3	46.5	15	C79 H70 N4 O9 Na
	1241.5040	4.1	3.3	52.0	16	C78 H64 N11 O4 Na
	1241.5123	-4.2	-3.4	46.0	2	C73 H67 N11 O9
	1241.5038	4.3	3.5	50.5	14	C77 H65 N10 O7
	1241.5126	-4.5	-3.6	47.5	7	C74 H66 N12 O6 Na
	1241.5033	4.8	3.9	63.0	45	C91 H63 N5 O
	1241.5132	-5.1	-4.1	58.0	40	C89 H67 N3 O4
	1241.5134	-5.3	-4.3	59.5	42	C90 H66 N4 O Na
	1241.5027	5.4	4.3	47.0	12	C77 H68 N7 O8 Na
	1241.5027	5.4	4.3	52.5	13	C76 H62 N14 O3 Na
	1241.5137	-5.6	-4.5	51.0	8	C74 H63 N15 O5
	1241.5137	-5.6	-4.5	45.5	5	C75 H69 N8 O10
	1241.5024	5.7	4.6	51.0	10	C75 H63 N13 O6
	1241.5139	-5.8	-4.7	47.0	9	C76 H68 N9 O7 Na
	1241.5022	5.9	4.8	59.5	44	C91 H66 N2 O2 Na
	1241.5019	6.2	5.0	58.0	41	C90 H67 N O5
	1241.5019	6.2	5.0	63.5	43	C89 H61 N8

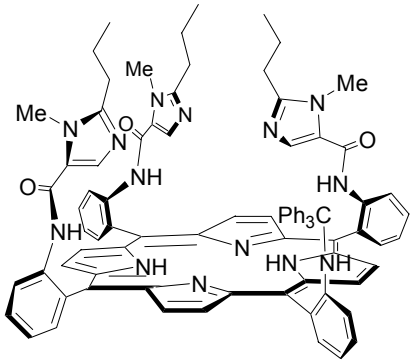


Compound **10b**
^{1}H NMR in $CDCl_3$

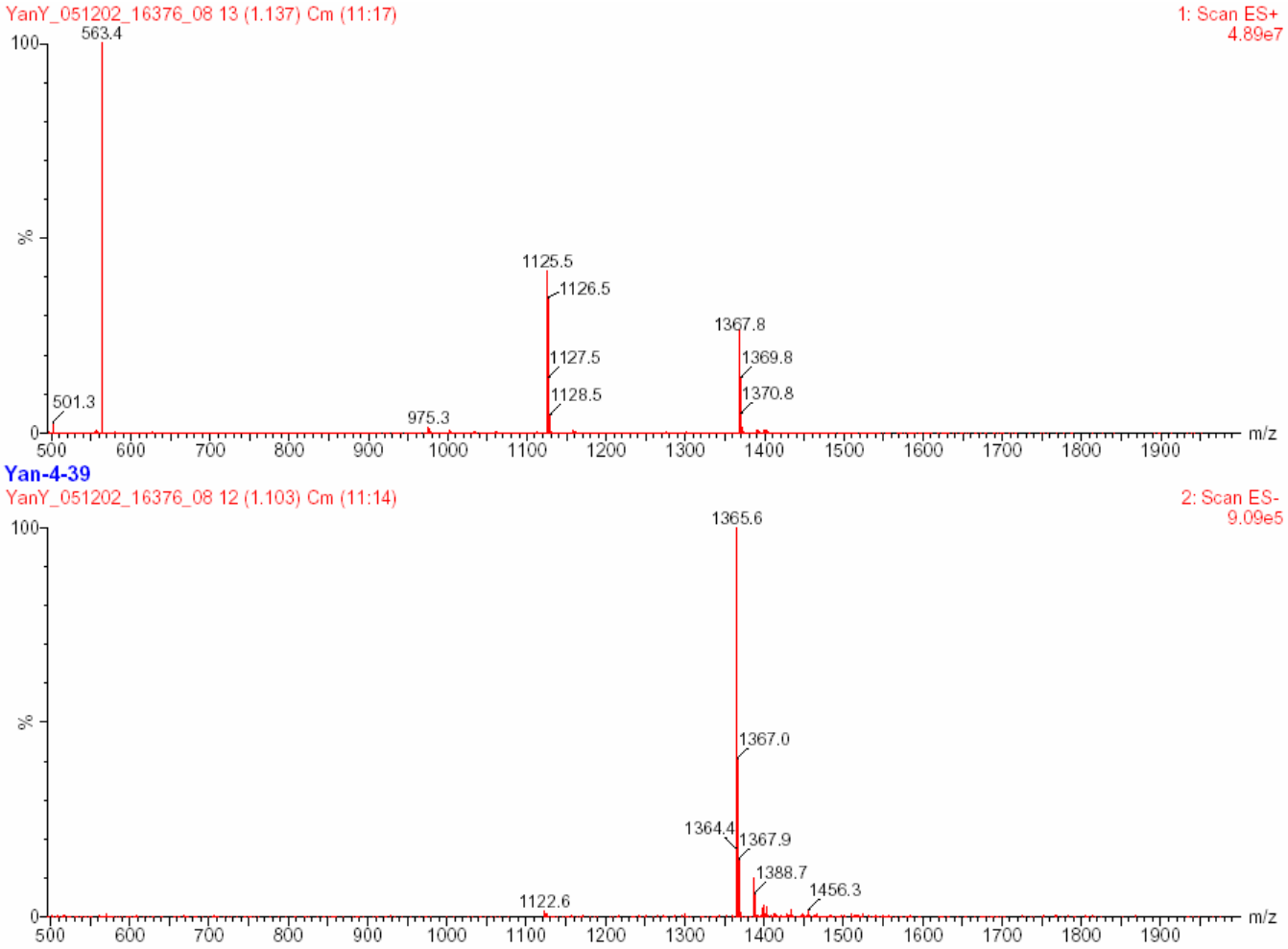


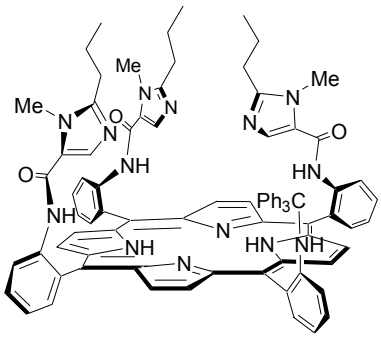


Compound **10b**
^{13}C NMR in $CDCl_3$



Compound **10b**, Low resolution mass analysis





Compound **10b**, High resolution mass analysis

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 120.0

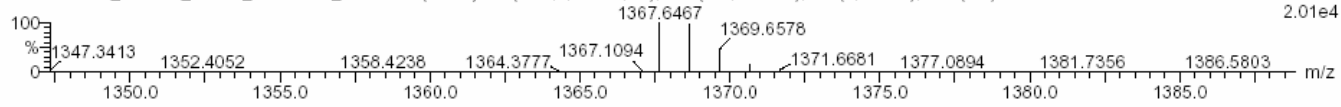
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

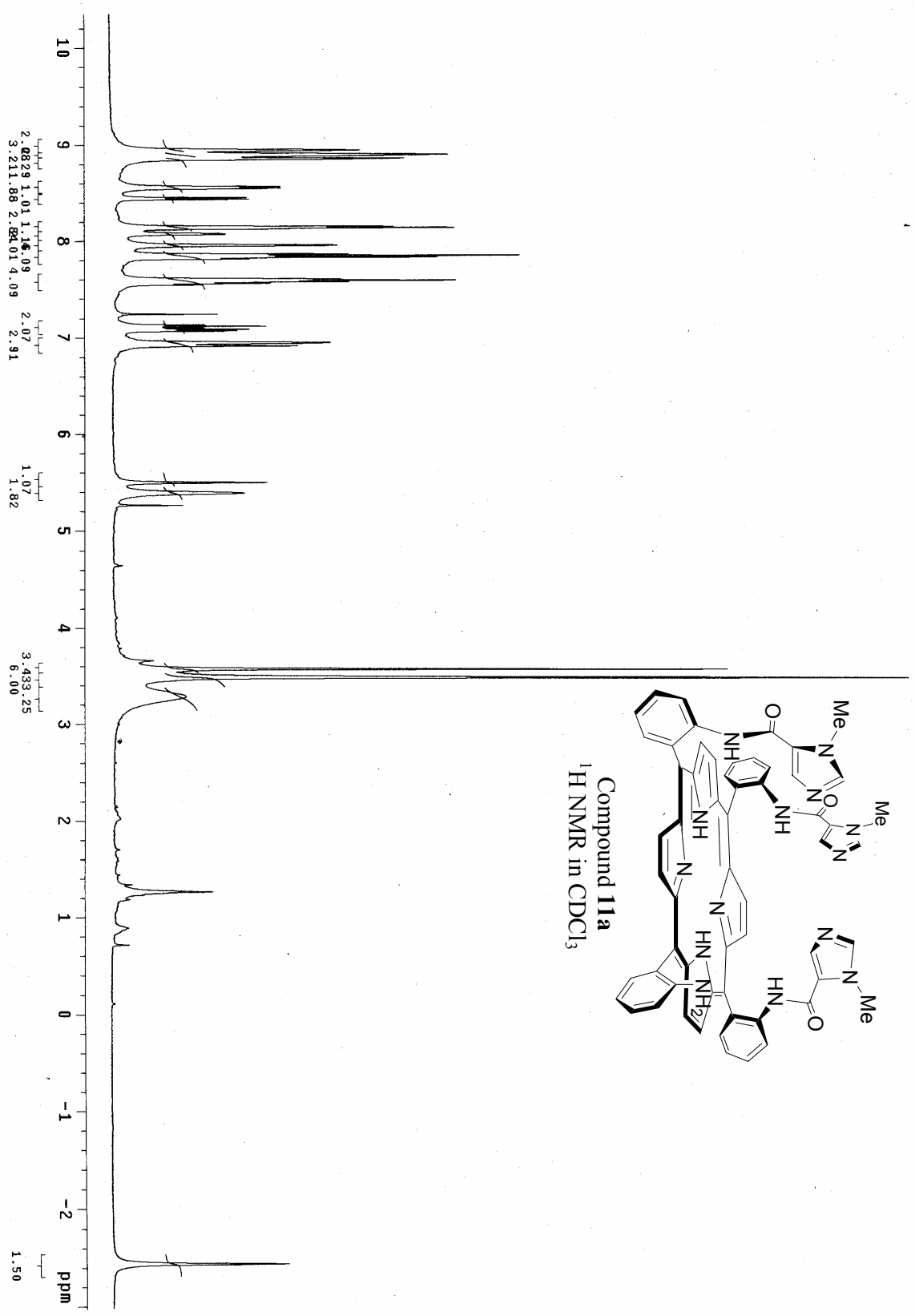
4885 formula(e) evaluated with 50 results within limits (up to 140 closest results for each mass)

Yilong Yan, Yan-4-39 | Cone V = 30, Coll V = 10.0 | 05-Dec-2005 17:18:44

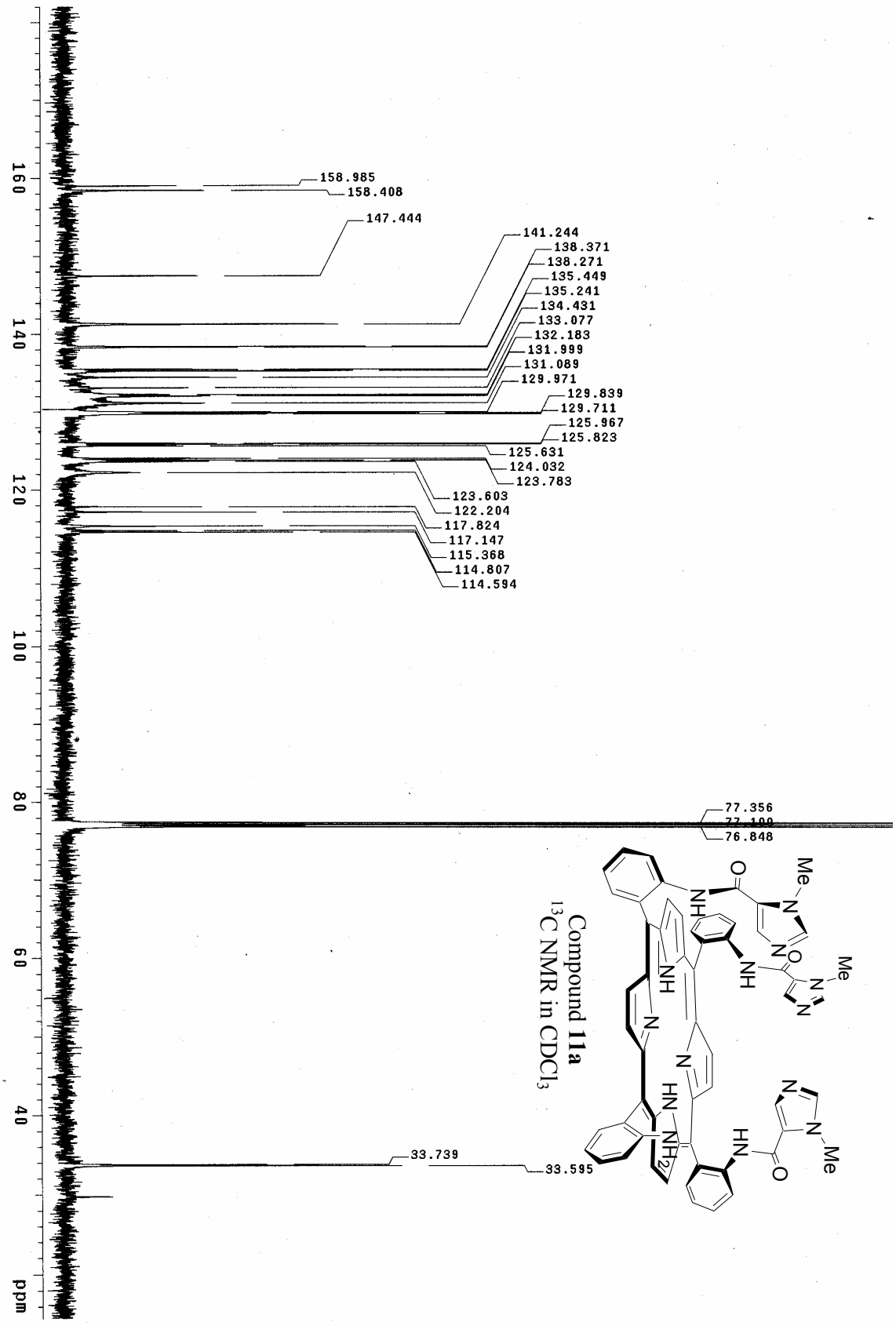
YANYILONG_051205_16381_YAN-4-39_HRMS 6 (0.261) Cn (Cen,4, 80.00, Ar); Sm (SG, 2x3.00); Sb (2,20.00); Cm (6:9) | TOF MS ES+ 2.01e4



Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
Minimum:				-1.5		
Maximum:		200.0	5.0	120.0		
1367.6467	1367.6462	0.5	0.3	46.0	20	C90 H90 N O10 Na
	1367.6462	0.5	0.3	51.5	21	C89 H84 N8 O5 Na
	1367.6462	0.5	0.3	57.0	22	C88 H78 N15 Na
	1367.6473	-0.6	-0.4	55.0	24	C89 H81 N11 O4
	1367.6473	-0.6	-0.4	49.5	23	C90 H87 N4 O9
	1367.6460	0.7	0.5	50.0	18	C88 H85 N7 O8
	1367.6460	0.7	0.5	55.5	19	C87 H79 N14 O3 ← $[M+H]^+$
	1367.6476	-0.9	-0.6	56.5	26	C90 H80 N12 O Na
	1367.6476	-0.9	-0.6	51.0	25	C91 H86 N5 O6 Na
	1367.6449	1.8	1.3	46.5	16	C88 H88 N4 O9 Na
	1367.6449	1.8	1.3	52.0	17	C87 H82 N11 O4 Na
	1367.6486	-1.9	-1.4	60.0	29	C90 H77 N15
	1367.6486	-1.9	-1.4	54.5	28	C91 H83 N8 O5
	1367.6486	-1.9	-1.4	49.0	27	C92 H89 N O10
	1367.6446	2.1	1.5	50.5	15	C86 H83 N10 O7
	1367.6489	-2.2	-1.6	56.0	31	C92 H82 N9 O2 Na
	1367.6489	-2.2	-1.6	50.5	30	C93 H88 N2 O7 Na
	1367.6441	2.6	1.9	63.0	50	C100 H81 N5 O
	1367.6494	-2.7	-2.0	43.5	7	C78 H84 N14 O8 Na
	1367.6436	3.1	2.3	47.0	13	C86 H86 N7 O8 Na
	1367.6436	3.1	2.3	52.5	14	C85 H80 N14 O3 Na
	1367.6500	-3.3	-2.4	59.5	33	C92 H79 N12 O
	1367.6500	-3.3	-2.4	54.0	32	C93 H85 N5 O6
	1367.6433	3.4	2.5	51.0	12	C84 H81 N13 O6
	1367.6503	-3.6	-2.6	55.5	34	C94 H84 N6 O3 Na
	1367.6431	3.6	2.7	59.5	49	C100 H84 N2 O2 Na
	1367.6428	3.9	2.9	58.0	47	C99 H85 N O5
	1367.6428	3.9	2.9	63.5	48	C98 H79 N8
	1367.6508	-4.1	-3.0	43.0	2	C80 H86 N11 O9 Na
	1367.6422	4.5	3.3	47.5	11	C84 H84 N10 O7 Na
	1367.6513	-4.6	-3.4	59.0	36	C94 H81 N9 O2
	1367.6513	-4.6	-3.4	53.5	35	C95 H87 N2 O7
	1367.6419	4.8	3.5	46.0	10	C83 H85 N9 O10
	1367.6516	-4.9	-3.6	55.0	38	C96 H86 N3 O4 Na
	1367.6417	5.0	3.7	60.0	46	C98 H82 N5 O Na
	1367.6518	-5.1	-3.8	46.5	1	C80 H83 N14 O8
	1367.6414	5.3	3.9	58.5	44	C97 H83 N4 O4
	1367.6521	-5.4	-4.0	48.0	3	C81 H82 N15 O5 Na
	1367.6521	-5.4	-4.0	42.5	5	C82 H88 N8 O10 Na
	1367.6409	5.8	4.3	48.0	6	C82 H82 N13 O6 Na
	1367.6527	-6.0	-4.4	58.5	41	C96 H83 N6 O3
	1367.6406	6.1	4.5	46.5	4	C81 H83 N12 O9
	1367.6529	-6.2	-4.6	60.0	45	C97 H82 N7 Na
	1367.6529	-6.2	-4.6	54.5	43	C98 H88 O5 Na
	1367.6404	6.3	4.6	55.0	40	C97 H86 N O5 Na
	1367.6404	6.3	4.6	60.5	42	C96 H80 N8 Na
	1367.6532	-6.5	-4.7	46.0	8	C82 H85 N11 O9
	1367.6401	6.6	4.8	53.5	37	C96 H87 O8
	1367.6401	6.6	4.8	59.0	39	C95 H81 N7 O3
	1367.6534	-6.7	-4.9	47.5	9	C83 H84 N12 O6 Na



Compound **11a**
^{1}H NMR in $CDCl_3$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129.839
129.711
125.967
125.823
125.631
124.032
123.783
123.603
122.204
117.824
117.147
115.368
114.807
114.594
77.356
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33.595
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140
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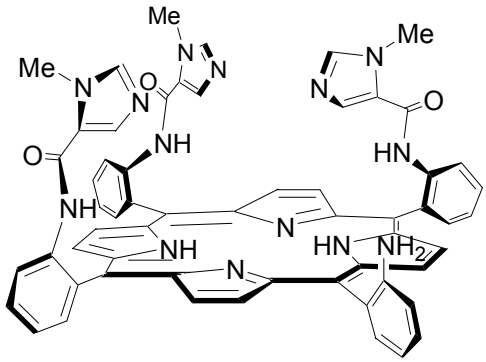
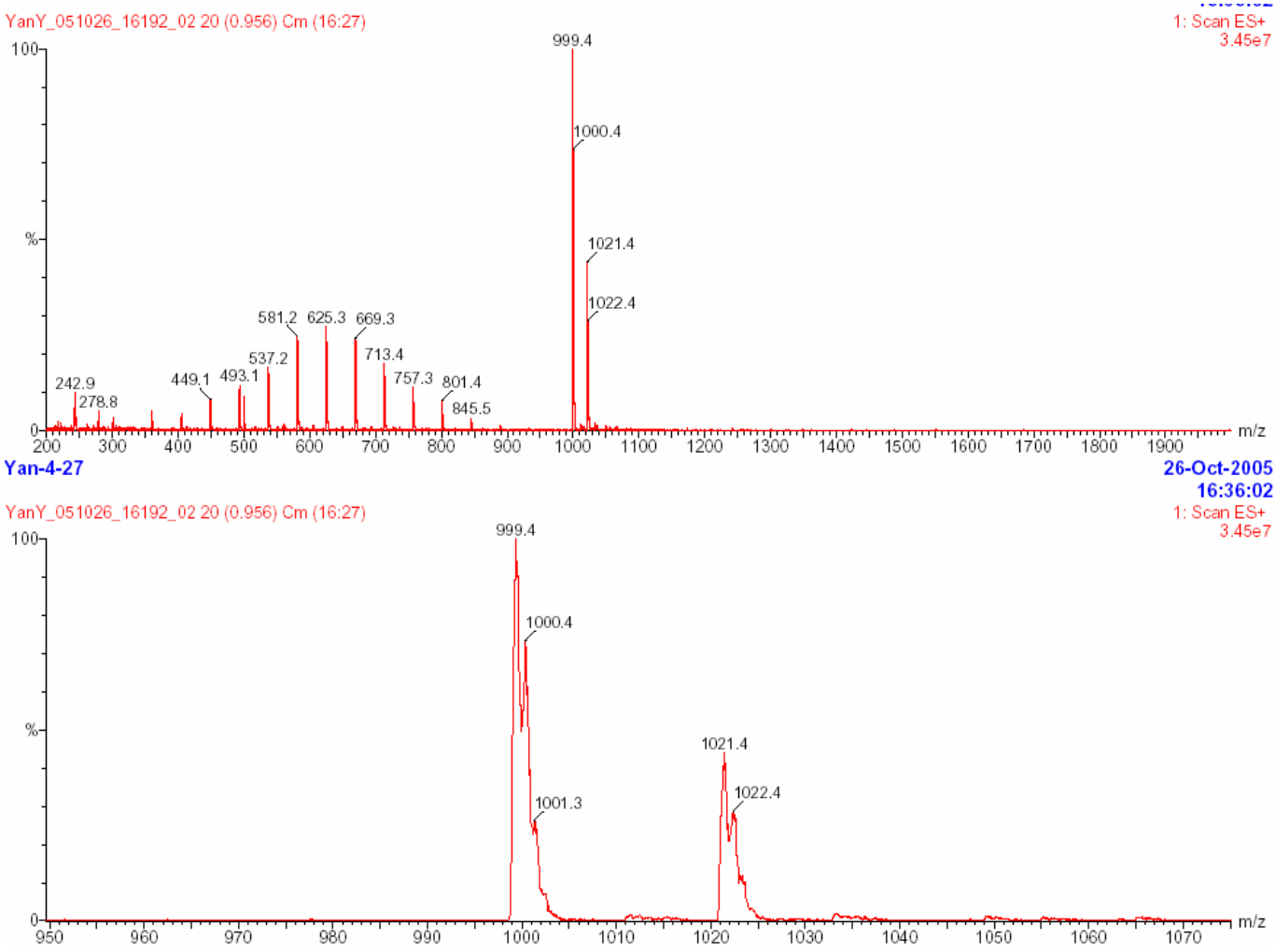
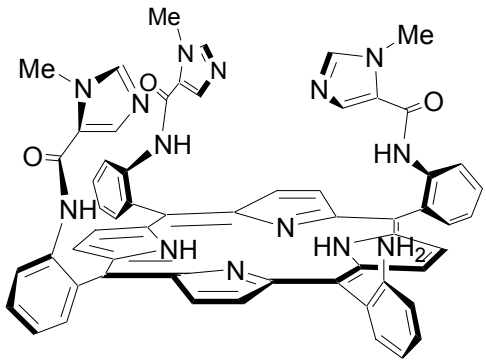
80
60
40
ppm
Compound 11a
13C NMR in CDCl3



Compound **11a**, Low resolution mass analysis





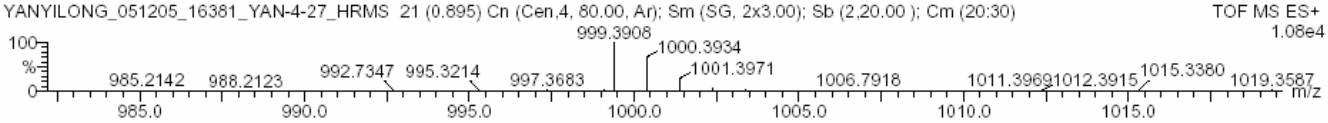
Compound **11a**, High resolution mass analysis

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 120.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
3897 formula(e) evaluated with 37 results within limits (up to 140 closest results for each mass)

Yilong Yan, Yan-4-27
Cone V = 30
Coll V = 10.0
05-Dec-2005
17:35:21
YANYILONG_051205_16381_YAN-4-27_HRMS 21 (0.895) Cn (Cen,4, 80.00, Ar); Sm (SG, 2x3.00); Sb (2,20.00); Cm (20:30)
TOF MS ES+
1.08e4



Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
Minimum:				-1.5		
Maximum:		200.0	5.0	120.0		
999.3908	999.3910	-0.2	-0.2	46.5	28	C69 H51 N4 O4
	999.3905	0.3	0.3	36.0	18	C54 H50 N13 O6 Na
	999.3913	-0.5	-0.5	48.0	30	C70 H50 N5 O Na
	999.3902	0.6	0.6	34.5	22	C53 H51 N12 O9
	999.3915	-0.7	-0.7	34.0	17	C55 H53 N9 O10
	999.3900	0.8	0.8	43.0	24	C69 H54 N O5 Na
	999.3900	0.8	0.8	48.5	27	C68 H48 N8 Na
	999.3918	-1.0	-1.0	35.5	12	C56 H52 N10 O7 Na
	999.3897	1.1	1.1	41.5	21	C68 H55 O8
	999.3897	1.1	1.1	47.0	23	C67 H49 N7 O3
	999.3924	-1.6	-1.6	51.5	32	C70 H47 N8
	999.3924	-1.6	-1.6	46.0	31	C71 H53 N O5
	999.3891	1.7	1.7	31.0	25	C53 H54 N9 O10 Na
	999.3926	-1.8	-1.9	47.5	34	C72 H52 N2 O2 Na
	999.3889	1.9	1.9	35.0	26	C51 H49 N15 O8
	999.3929	-2.1	-2.1	39.0	9	C56 H49 N13 O6
	999.3886	2.2	2.2	43.5	20	C67 H52 N4 O4 Na
	999.3932	-2.4	-2.4	40.5	6	C57 H48 N14 O3 Na
	999.3932	-2.4	-2.4	35.0	7	C58 H54 N7 O8 Na
	999.3884	2.4	2.4	42.0	16	C66 H53 N3 O7
	999.3883	2.5	2.5	47.5	19	C65 H47 N10 O2
	999.3937	-2.9	-2.9	51.0	35	C72 H49 N5 O
	999.3878	3.0	3.0	31.5	29	C51 H52 N12 O9 Na
	999.3942	-3.4	-3.4	38.5	5	C58 H51 N10 O7
	999.3873	3.5	3.5	38.5	13	C66 H56 O8 Na
	999.3873	3.5	3.5	44.0	15	C65 H50 N7 O3 Na
	999.3945	-3.7	-3.7	40.0	3	C59 H50 N11 O4 Na
	999.3945	-3.7	-3.7	34.5	2	C60 H56 N4 O9 Na
	999.3870	3.8	3.8	42.5	11	C64 H51 N6 O6
	999.3870	3.8	3.8	48.0	14	C63 H45 N13 O
	999.3951	-4.3	-4.3	50.5	36	C74 H51 N2 O2
	999.3865	4.3	4.3	55.0	37	C78 H49 N
	999.3864	4.4	4.4	32.0	33	C49 H50 N15 O8 Na
	999.3956	-4.8	-4.8	43.5	4	C59 H47 N14 O3 ← $[M+H]^+$
	999.3956	-4.8	-4.8	38.0	1	C60 H53 N7 O8
	999.3859	4.9	4.9	39.0	8	C64 H54 N3 O7 Na
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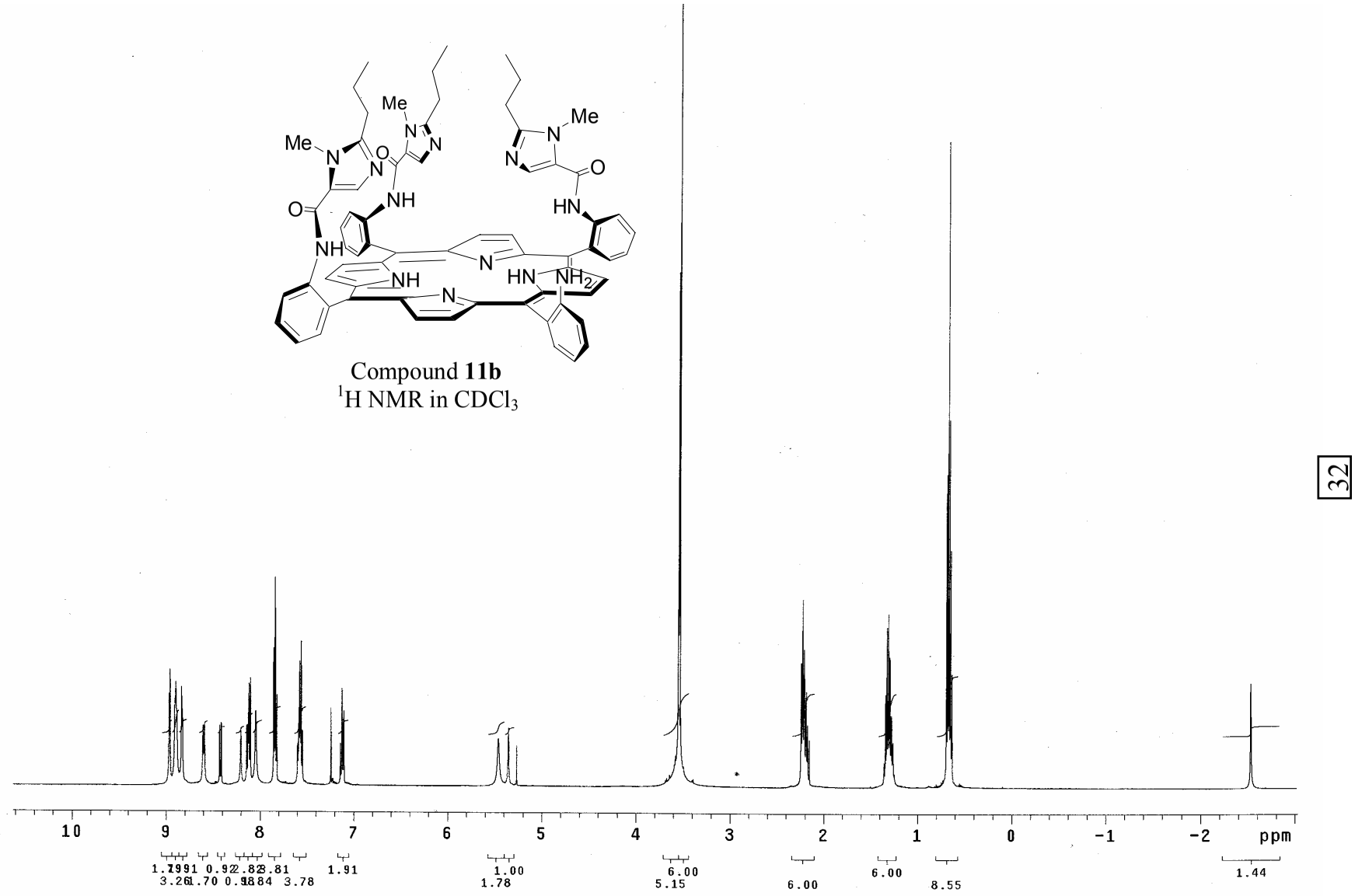
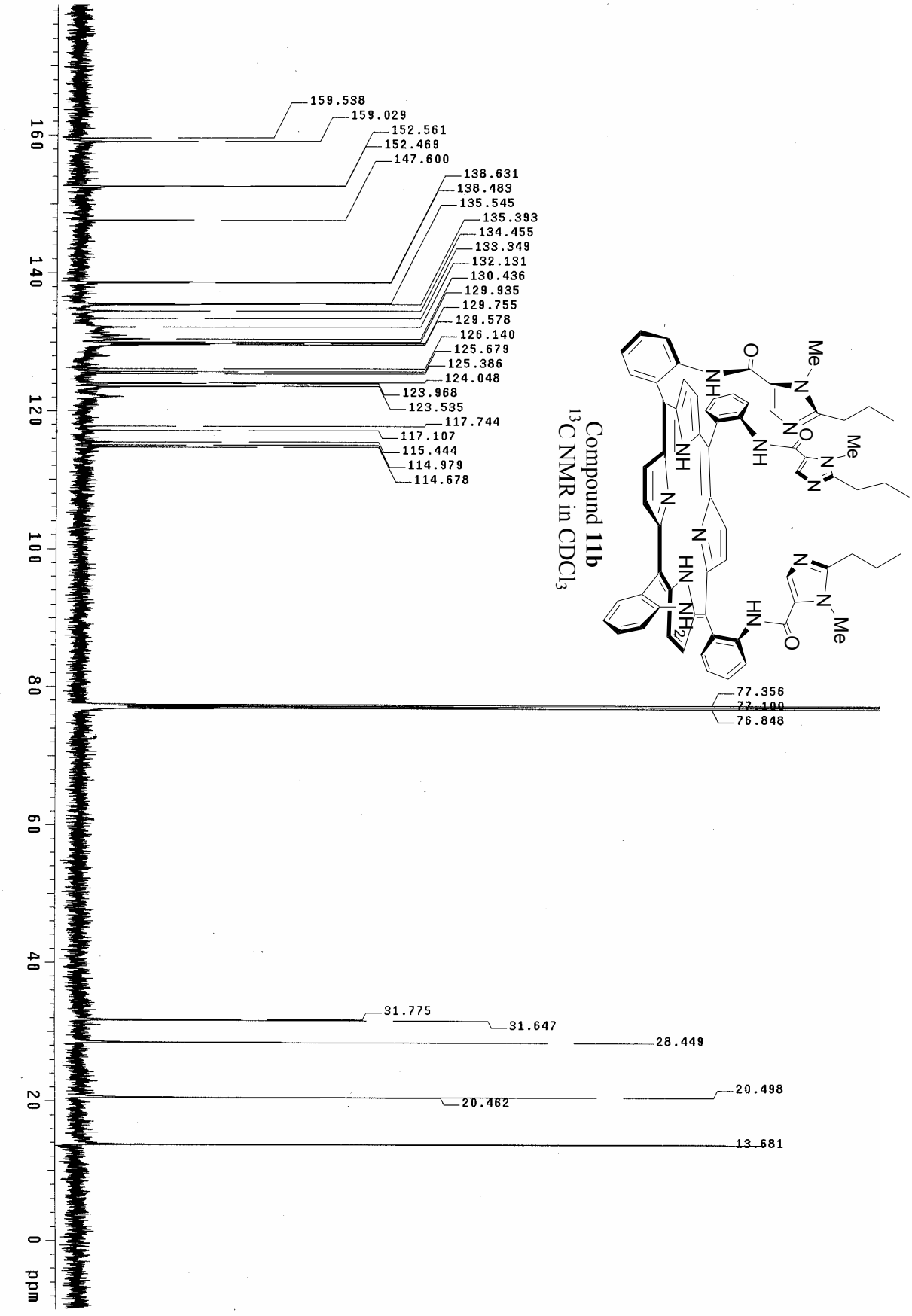


Compound **11b**
^{1}H NMR in $CDCl_3$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0
ppm
Compound 11b
13C NMR in CDCl3
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NH
N
N
HN
NH2
HN
O
O
Me
Me
N
N
N
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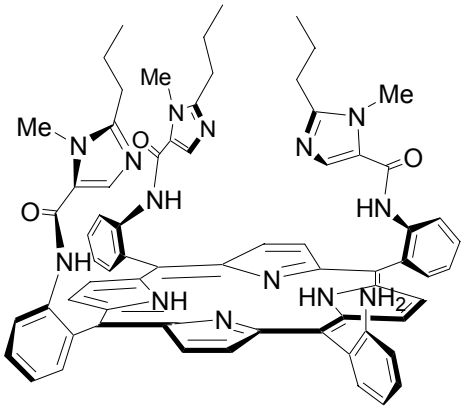
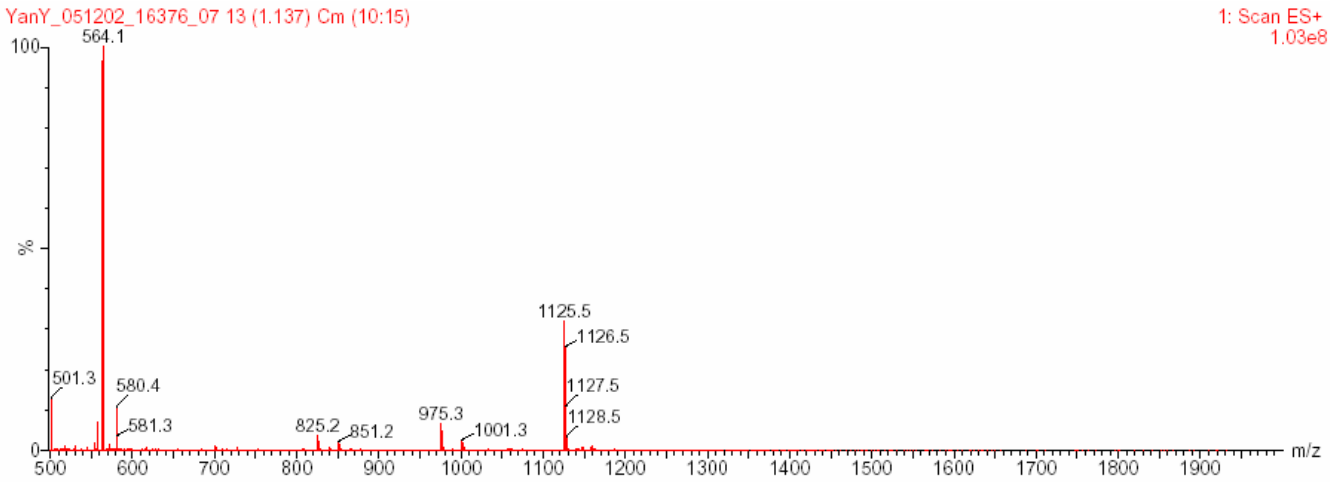
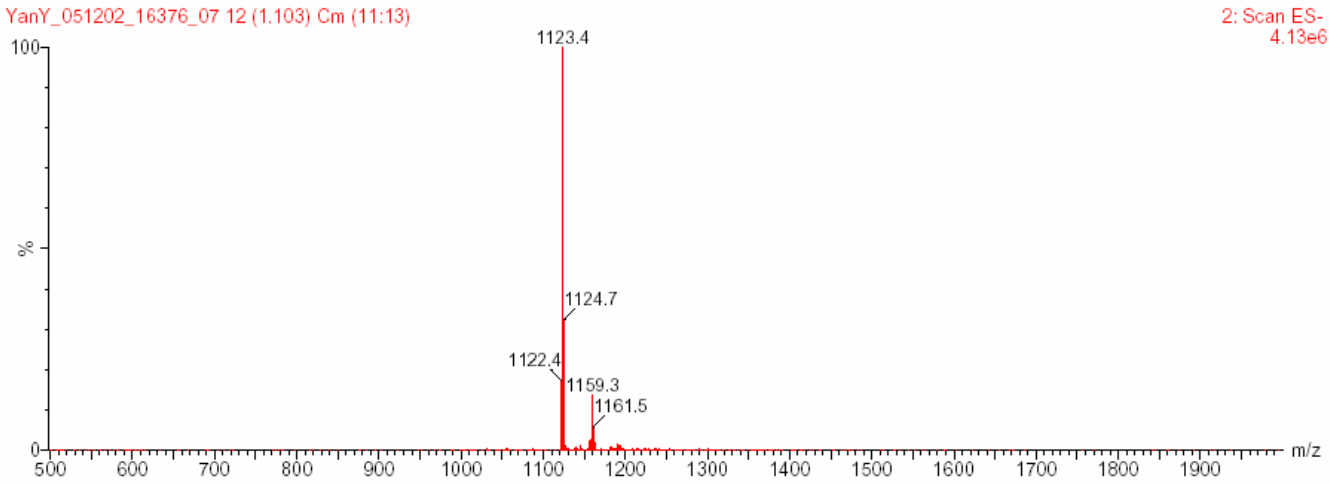
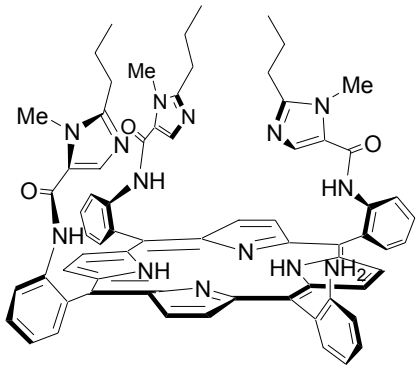


Compound **11b**, Low resolution mass analysis



Yan-4-40





Compound **11b**, High resolution mass analysis

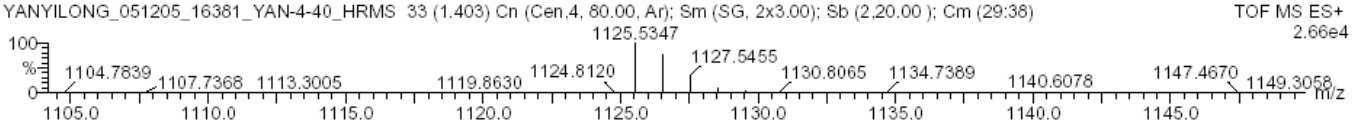
Elemental Composition Report

Single Mass Analysis

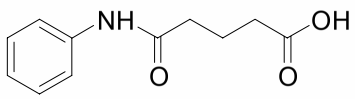
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 120.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
4454 formula(e) evaluated with 43 results within limits (up to 140 closest results for each mass)

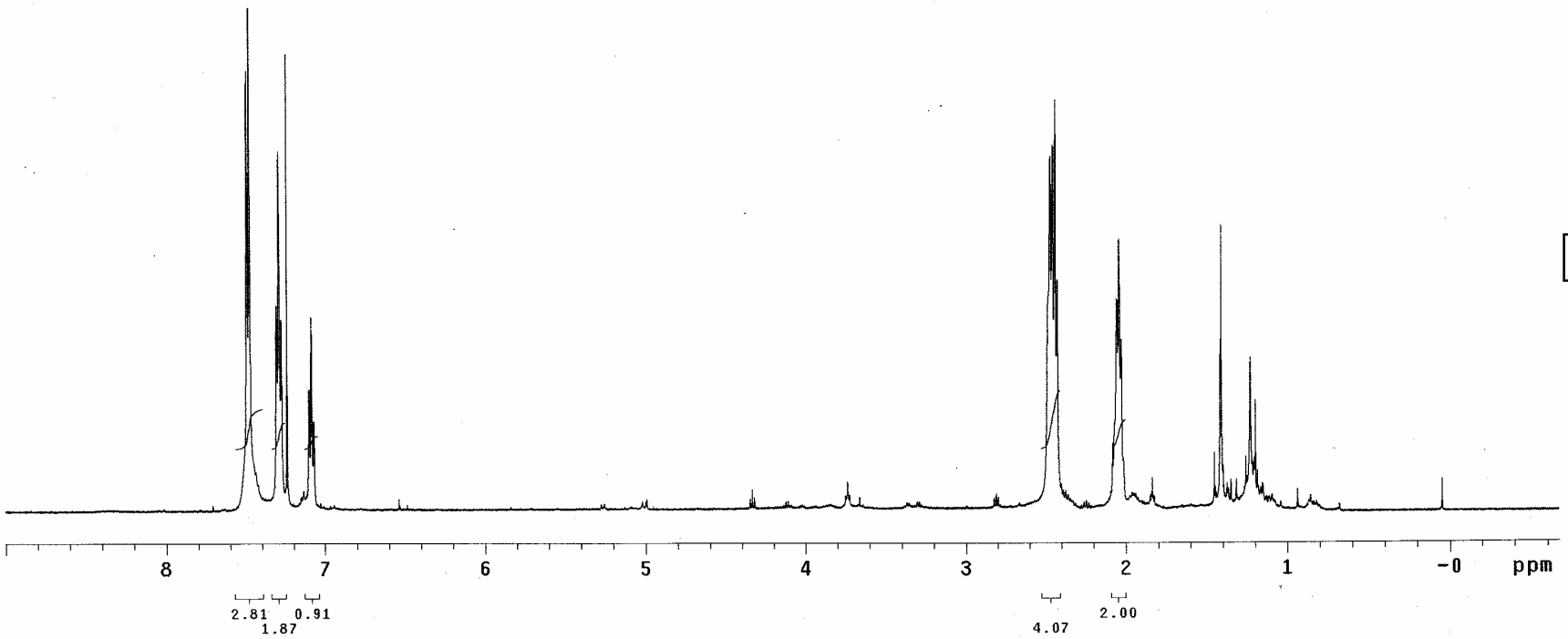
Yilong Yan, Yan-4-40 Cone V = 30 Coll V = 10.0 05-Dec-2005 16:53:50
YANYILONG_051205_16381_YAN-4-40_HRMS 33 (1.403) Cn (Cen,4, 80.00, Ar); Sm (SG, 2x3.00); Sb (2,20.00); Cm (29:38) TOF MS ES+ 2.66e4

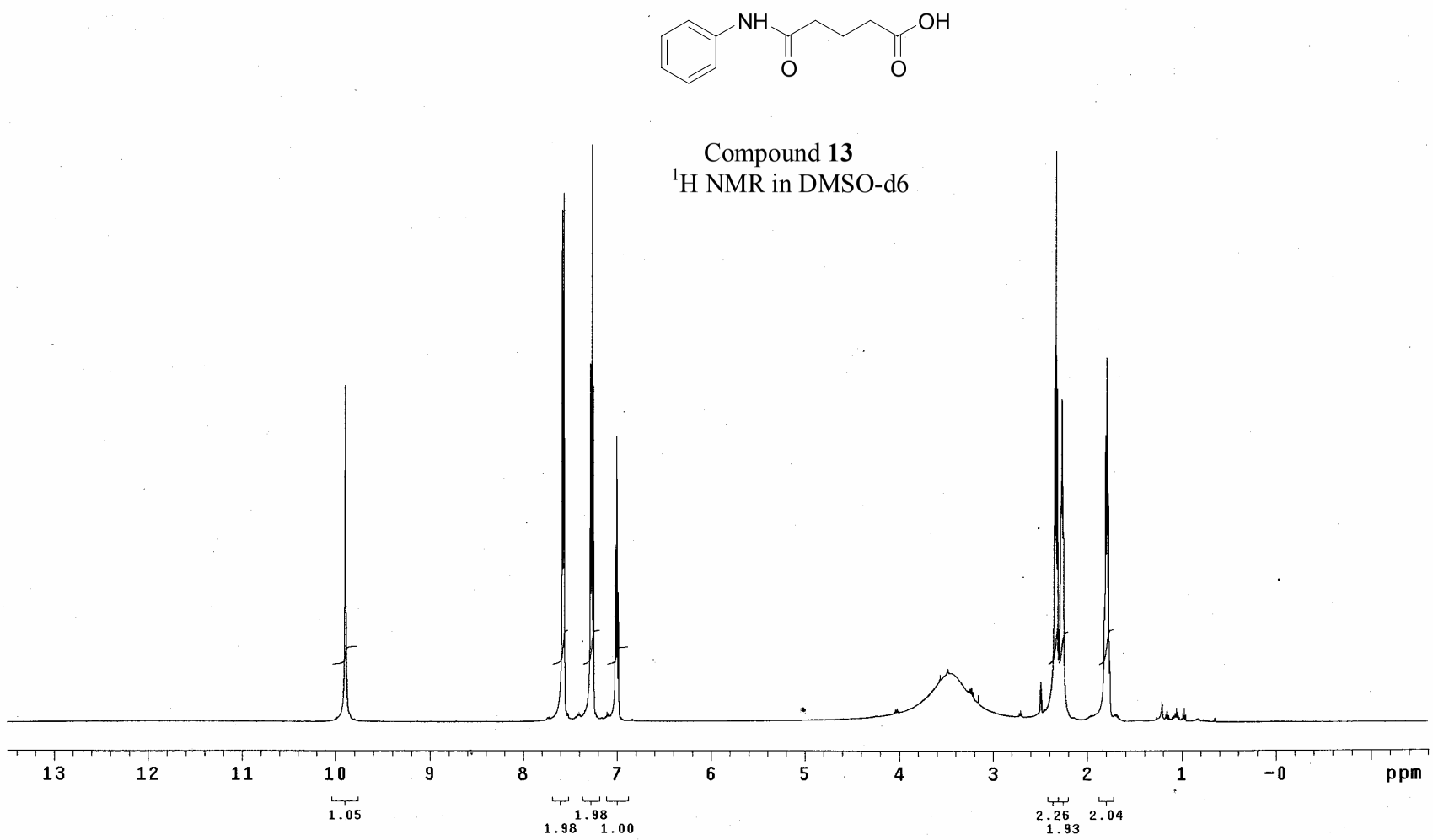


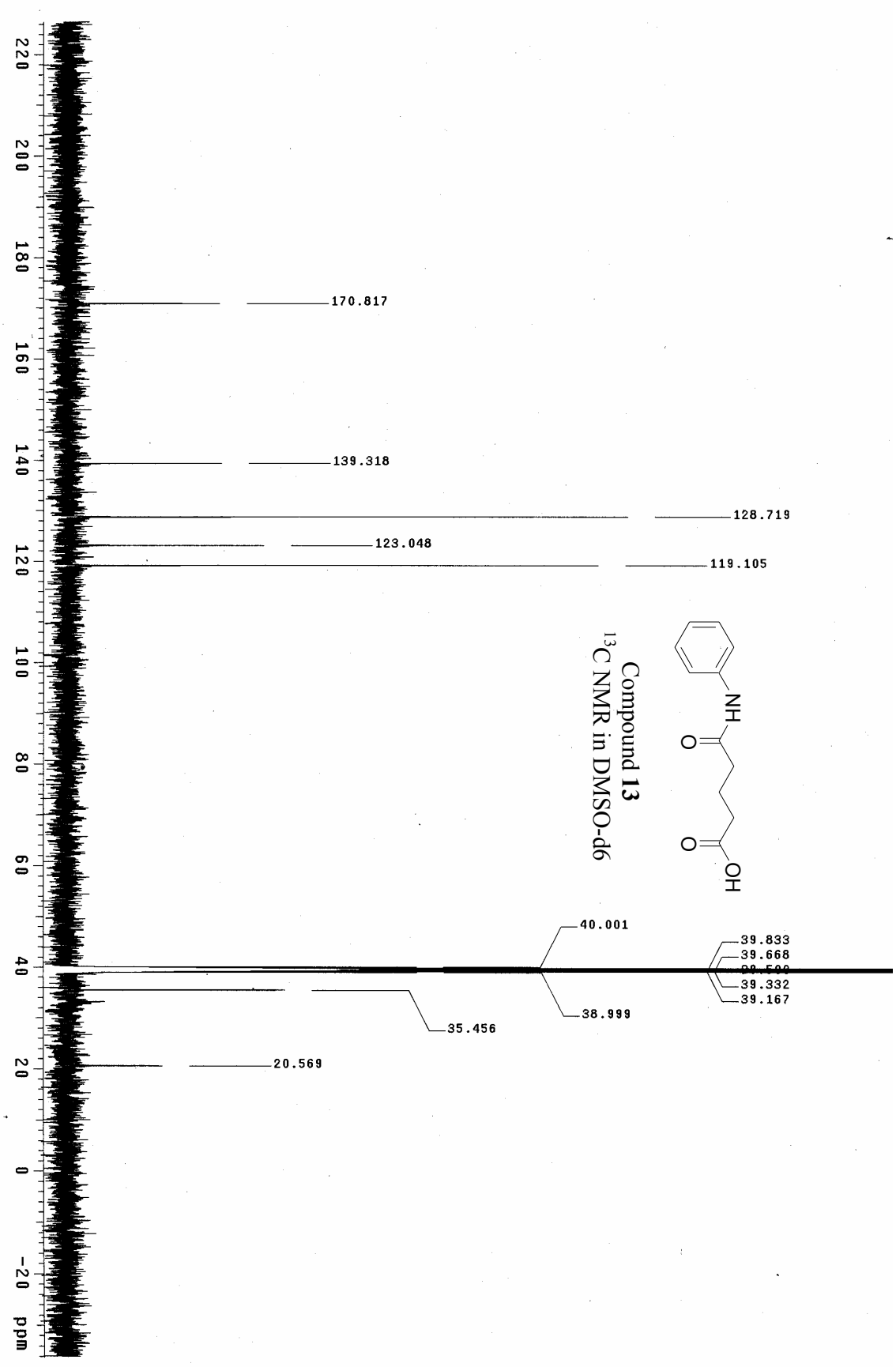
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula	
Minimum:				-1.5			
Maximum:		200.0	5.0	120.0			
1125.5347	1125.5346	0.1	0.1	51.0	40	C81 H67 N5 O	
	1125.5351	-0.4	-0.3	38.5	4	C67 H69 N10 O7	
	1125.5353	-0.6	-0.6	40.0	10	C68 H68 N11 O4 Na	
	1125.5354	-0.7	-0.6	34.5	2	C69 H74 N4 O9 Na	
	1125.5340	0.7	0.6	35.0	1	C67 H72 N7 O8 Na	
	1125.5340	0.7	0.6	40.5	8	C66 H66 N14 O3 Na	
	1125.5337	1.0	0.9	39.0	3	C65 H67 N13 O6	
	1125.5335	1.2	1.1	47.5	39	C81 H70 N2 O2 Na	
	1125.5359	-1.2	-1.1	50.5	41	C83 H69 N2 O2	
	1125.5332	1.5	1.3	46.0	37	C80 H71 N O5	
	1125.5332	1.5	1.3	51.5	38	C79 H65 N8	
	1125.5364	-1.7	-1.5	43.5	14	C68 H65 N14 O3	← [M+H]+
	1125.5364	-1.7	-1.5	38.0	9	C69 H71 N7 O8	
	1125.5367	-2.0	-1.8	45.0	19	C69 H64 N15 Na	
	1125.5367	-2.0	-1.8	39.5	15	C70 H70 N8 O5 Na	
	1125.5367	-2.0	-1.8	34.0	5	C71 H76 N O10 Na	
	1125.5327	2.0	1.8	35.5	6	C65 H70 N10 O7 Na	
	1125.5324	2.3	2.1	34.0	7	C64 H71 N9 O10	
	1125.5322	2.5	2.3	48.0	36	C79 H68 N5 O Na	
	1125.5319	2.8	2.5	46.5	35	C78 H69 N4 O4	
	1125.5375	-2.8	-2.5	51.5	42	C86 H70 Na	
	1125.5378	-3.1	-2.7	43.0	18	C70 H67 N11 O4	
	1125.5378	-3.1	-2.7	37.5	12	C71 H73 N4 O9	
	1125.5380	-3.3	-3.0	44.5	23	C71 H66 N12 O Na	
	1125.5380	-3.3	-3.0	39.0	20	C72 H72 N5 O6 Na	
	1125.5313	3.4	3.0	36.0	11	C63 H68 N13 O6 Na	
	1125.5310	3.7	3.2	34.5	13	C62 H69 N12 O9	
	1125.5308	3.9	3.4	43.0	33	C78 H72 N O5 Na	
	1125.5308	3.9	3.5	48.5	34	C77 H66 N8 Na	
	1125.5305	4.2	3.7	41.5	31	C77 H73 O8	
	1125.5305	4.2	3.7	47.0	32	C76 H67 N7 O3	
	1125.5391	-4.4	-3.9	48.0	26	C71 H63 N15	
	1125.5391	-4.4	-3.9	42.5	24	C72 H69 N8 O5	
	1125.5391	-4.4	-3.9	37.0	17	C73 H75 N O10	
	1125.5394	-4.7	-4.1	44.0	27	C73 H68 N9 O2 Na	
	1125.5394	-4.7	-4.2	38.5	22	C74 H74 N2 O7 Na	
	1125.5300	4.7	4.2	31.0	16	C62 H72 N9 O10 Na	
	1125.5297	5.0	4.4	35.0	21	C60 H67 N15 O8	
	1125.5399	-5.2	-4.6	31.5	25	C59 H70 N14 O8 Na	
	1125.5295	5.2	4.6	43.5	30	C76 H70 N4 O4 Na	
	1125.5399	-5.2	-4.6	54.5	43	C88 H69	
	1125.5292	5.5	4.9	42.0	28	C75 H71 N3 O7	
	1125.5292	5.5	4.9	47.5	29	C74 H65 N10 O2	

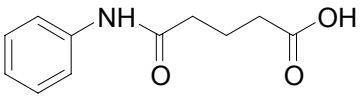


Compound **13**
^{1}H NMR in $CDCl_3$

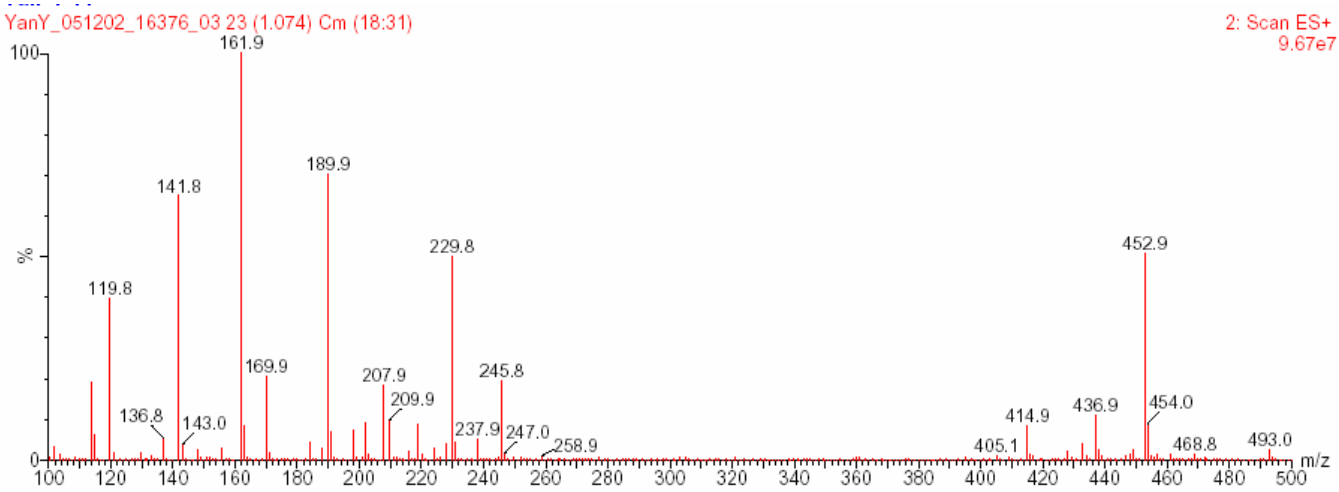



NH
OH
O
O
Compound 13
1H NMR in DMSO-d6
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.05
1.98
1.98
1.00
2.26
1.93
2.04


Compound 13
13C NMR in DMSO-d6
NH
O
O
OH
170.817
139.318
128.719
123.048
119.105
40.001
39.833
39.668
39.332
39.167
38.999
35.456
20.569
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm



Compound **13**: Low resolution mass analysis



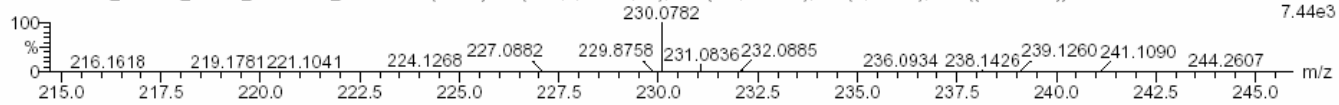
Compound **13**: High resolution mass analysis

Elemental Composition Report **Page 1**

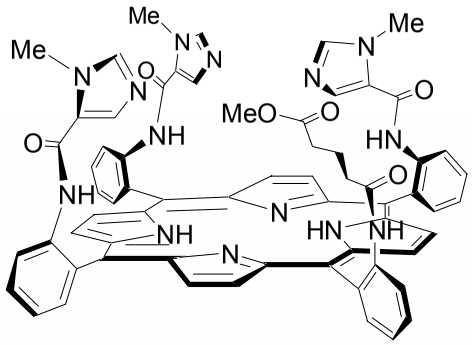
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 120.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
465 formula(e) evaluated with 6 results within limits (up to 140 closest results for each mass)

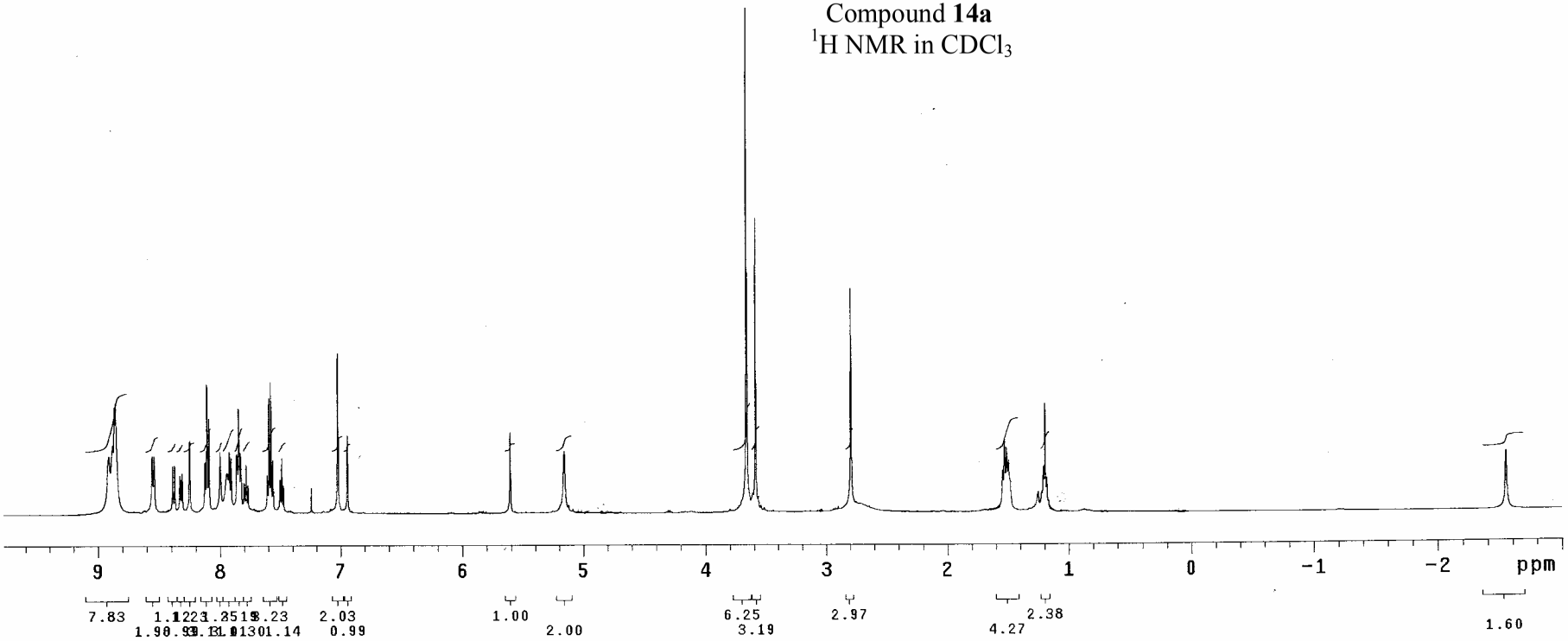
Yilong Yan, Yan-4-44 Cone V = 30 05-Dec-2005
Coll V = 10.0 16:24:02
YANYILONG_051205_16381_YAN-4-44_HRMS 39 (1.656) Cn (Cen,4, 80.00, Ar); Sm (SG, 2x3.00); Sb (2,20.00); Cm ((36:37+39)) TOF MS ES+
7.44e3

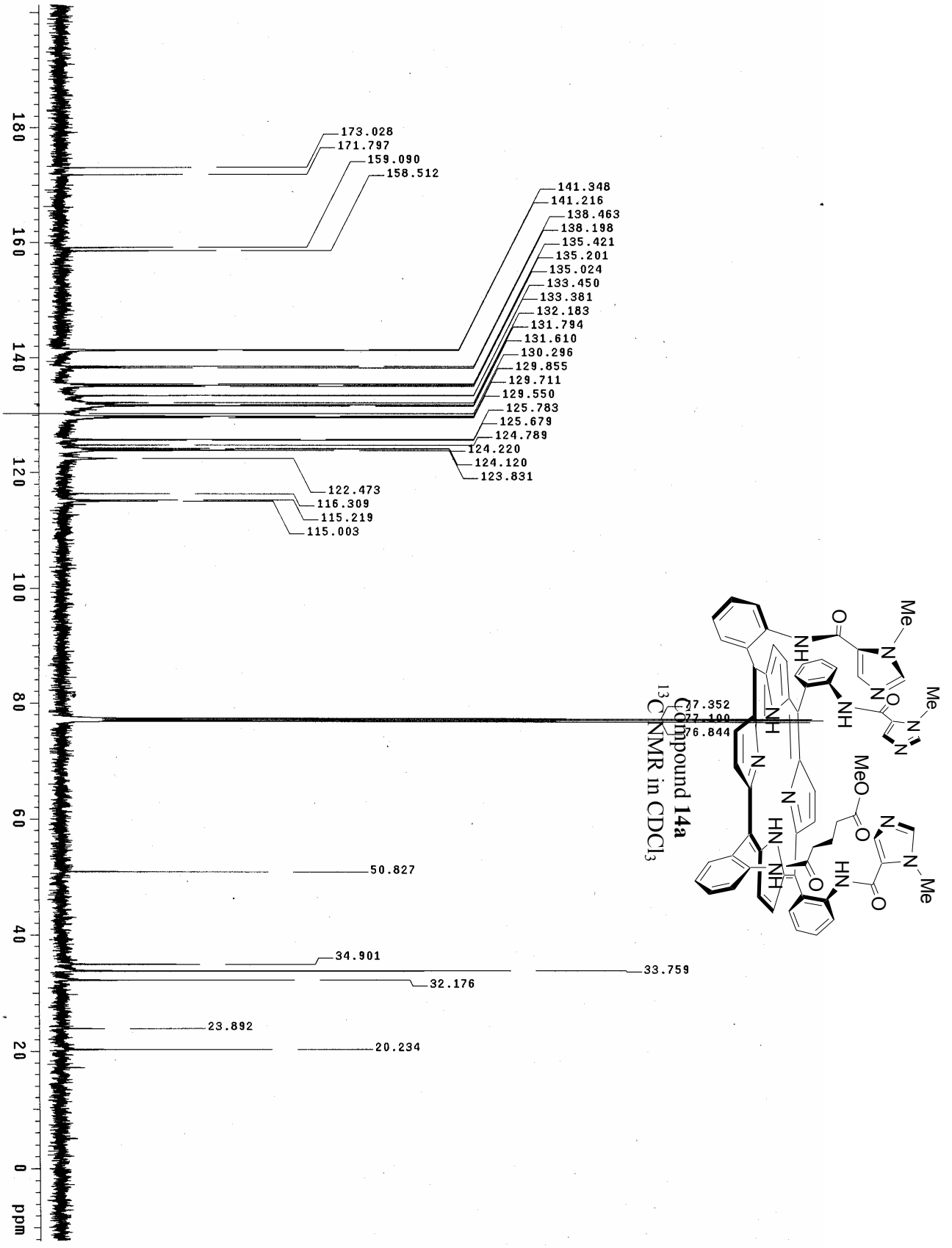


Minimum:				-1.5		
Maximum:		200.0	5.0	120.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
230.0782	230.0780	0.2	1.0	6.0	4	C9 H11 N4 O2 Na
	230.0777	0.5	2.2	4.5	6	C8 H12 N3 O5
	230.0777	0.5	2.2	10.0	5	C7 H6 N10
	230.0790	-0.8	-3.6	9.5	3	C9 H8 N7 O
	230.0790	-0.8	-3.6	4.0	2	C10 H14 O6
	230.0793	-1.1	-4.8	5.5	1	C11 H13 N O3 Na ← $[M+Na]^+$



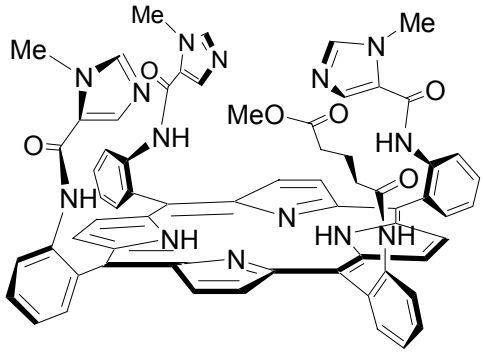
Compound **14a**
^{1}H NMR in $CDCl_3$



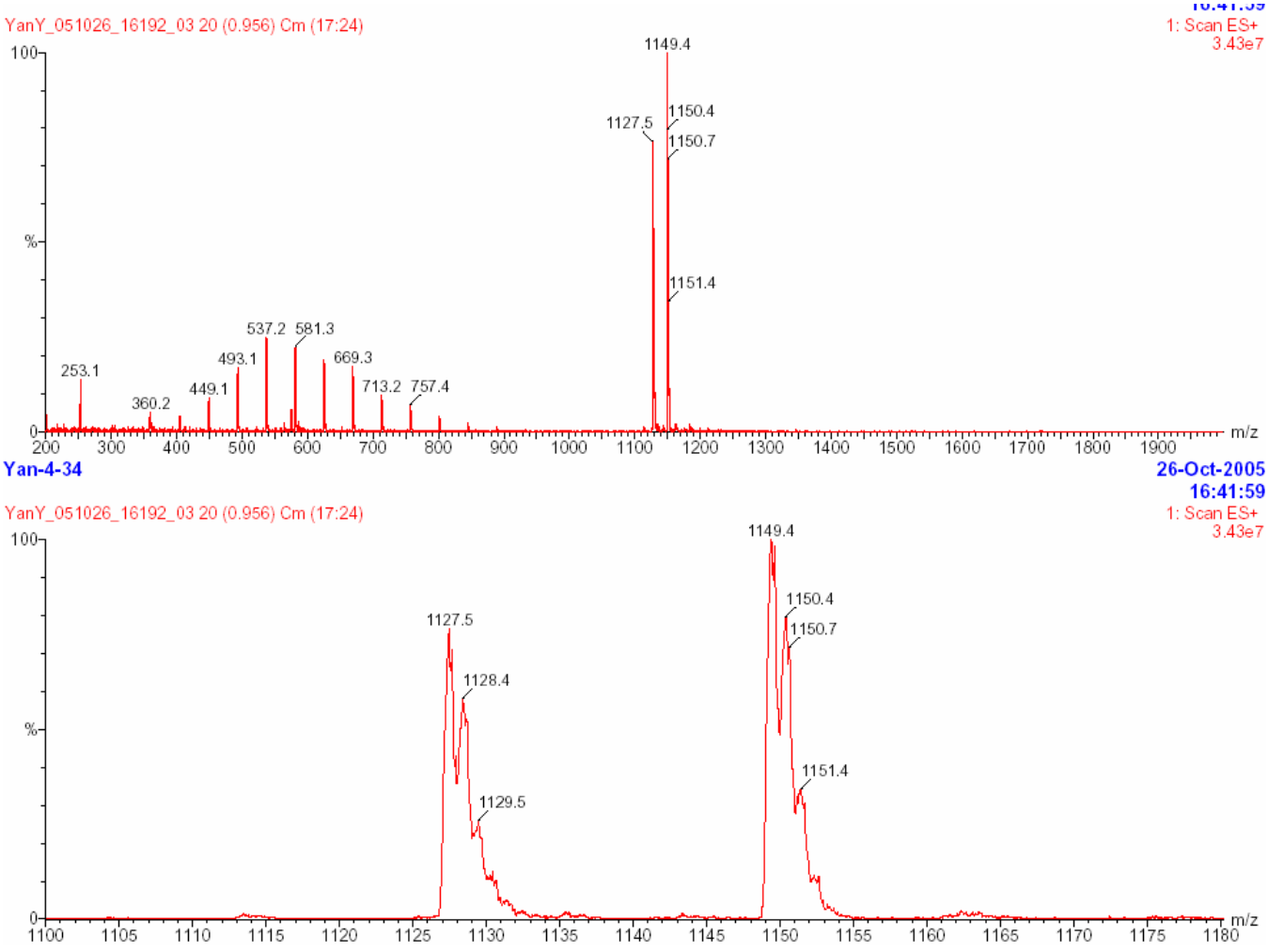


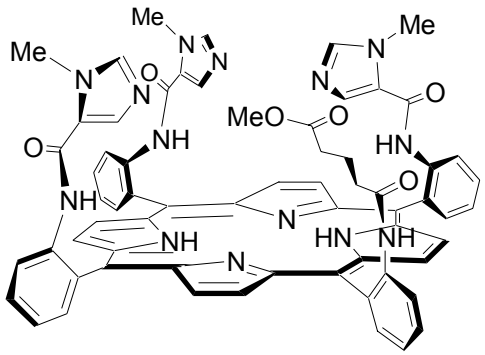
Compound **14a**

^{13}C NMR in $CDCl_3$



Compound **14a**: Low resolution mass analysis





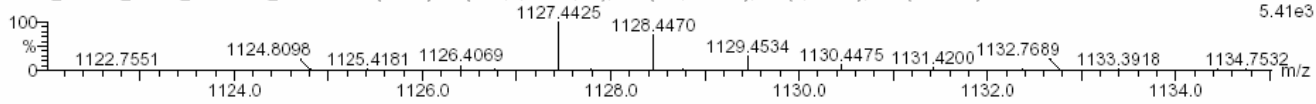
Compound **14a**: High resolution mass analysis

Elemental Composition Report **Page 1**

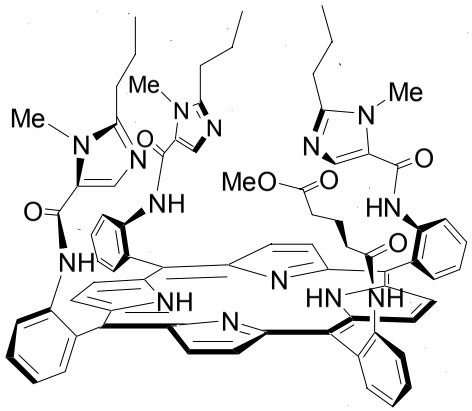
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 120.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
1805 formula(e) evaluated with 20 results within limits (up to 140 closest results for each mass)

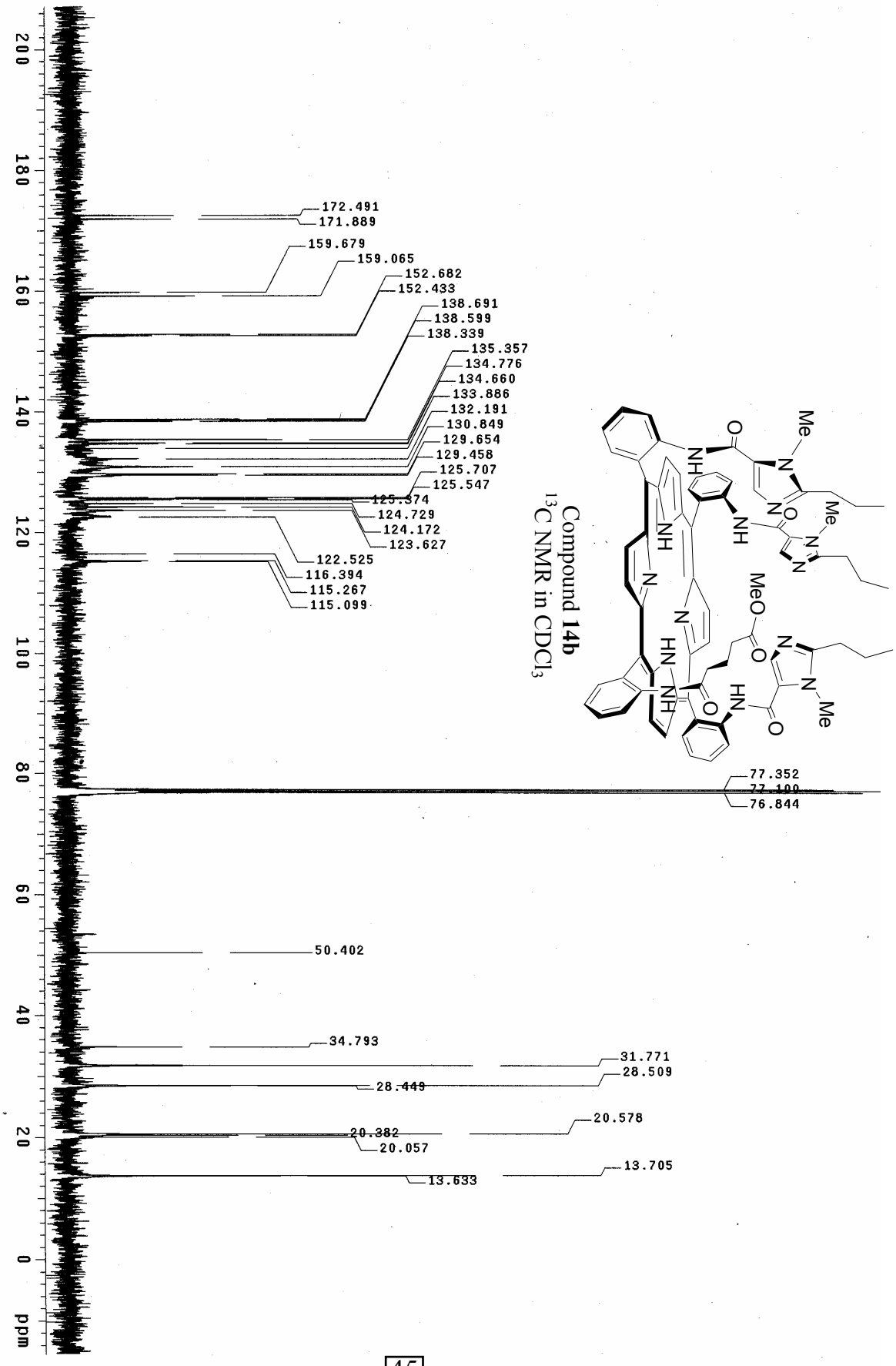
Yilong Yan, Yan-4-34 Cone V = 30 02-Nov-2005
16129 Coll V = 10.0 11:17:05
YANY_051102_16247_YAN-4-34_HRMS 118 (4.025) Cn (Cen,6, 80.00, Ar); Sm (SG, 2x3.00); Sb (2,20.00); Cm (108:118) TOF MS ES+
5.41e3

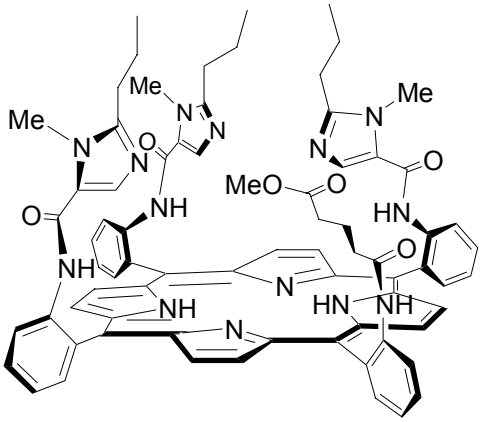


Minimum:				-1.5		
Maximum:		200.0	5.0	120.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
1127.4425	1127.4424	0.1	0.1	52.5	16	C80 H59 N2 O5
	1127.4424	0.1	0.1	58.0	17	C79 H53 N9
	1127.4429	-0.4	-0.4	45.5	3	C65 H55 N14 O6 ← $[M+H]^+$
	1127.4416	0.9	0.8	40.5	2	C64 H59 N10 O10
	1127.4437	-1.2	-1.1	57.5	18	C81 H55 N6 O
	1127.4411	1.4	1.3	53.0	15	C78 H57 N5 O4
	1127.4442	-1.7	-1.5	45.0	4	C67 H57 N11 O7
	1127.4402	2.3	2.0	41.0	1	C62 H57 N13 O9
	1127.4451	-2.6	-2.3	57.0	19	C83 H57 N3 O2
	1127.4397	2.8	2.5	48.0	12	C77 H61 N O8
	1127.4397	2.8	2.5	53.5	14	C76 H55 N8 O3
	1127.4456	-3.1	-2.7	50.0	7	C68 H53 N15 O3
	1127.4456	-3.1	-2.7	44.5	6	C69 H59 N8 O8
	1127.4464	-3.9	-3.5	56.5	20	C85 H59 O3
	1127.4384	4.1	3.7	48.5	10	C75 H59 N4 O7
	1127.4384	4.1	3.7	54.0	13	C74 H53 N11 O2
	1127.4469	-4.4	-3.9	49.5	8	C70 H55 N12 O4
	1127.4469	-4.4	-3.9	44.0	5	C71 H61 N5 O9
	1127.4370	5.5	4.8	49.0	9	C73 H57 N7 O6
	1127.4370	5.5	4.9	54.5	11	C72 H51 N14 O

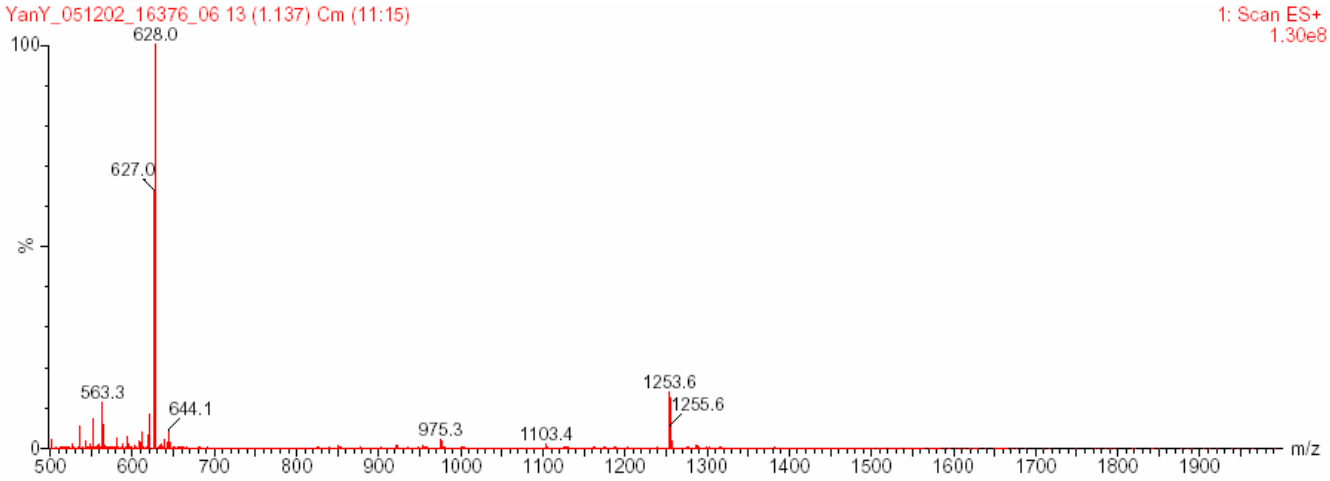


Compound **14b**
^{1}H NMR in $CDCl_3$

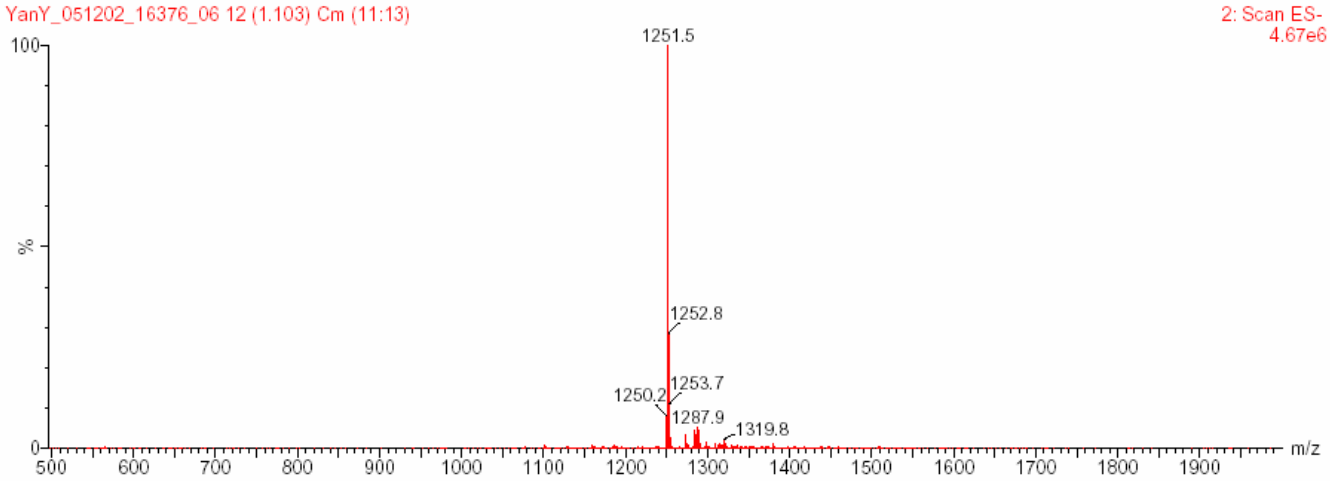

Compound 14b
13C NMR in CDCl3
172.491
171.889
159.679
159.065
152.682
152.433
138.691
138.599
138.339
135.357
134.776
134.660
133.886
132.191
130.849
129.654
129.458
125.707
125.547
125.374
124.729
124.172
123.627
122.525
116.394
115.267
115.099
77.352
77.100
76.844
50.402
34.793
31.771
28.509
28.449
20.578
20.382
20.057
13.705
13.633
ppm

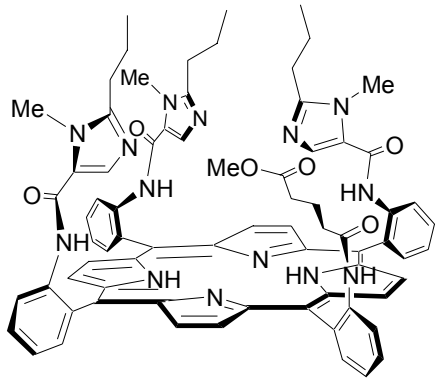


Compound **14b**, Low resolution mass analysis



Yan-4-41





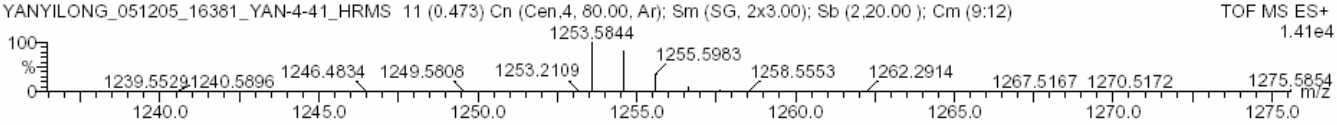
Compound **14b**, High resolution mass analysis

Elemental Composition Report **Page 1**

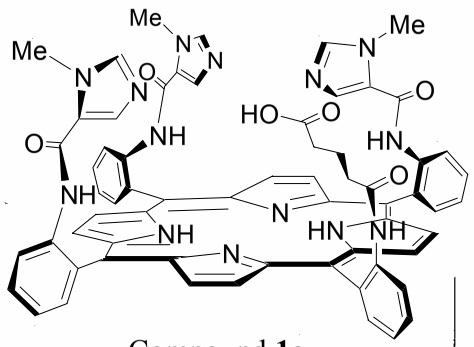
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 120.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
4972 formula(e) evaluated with 47 results within limits (up to 140 closest results for each mass)

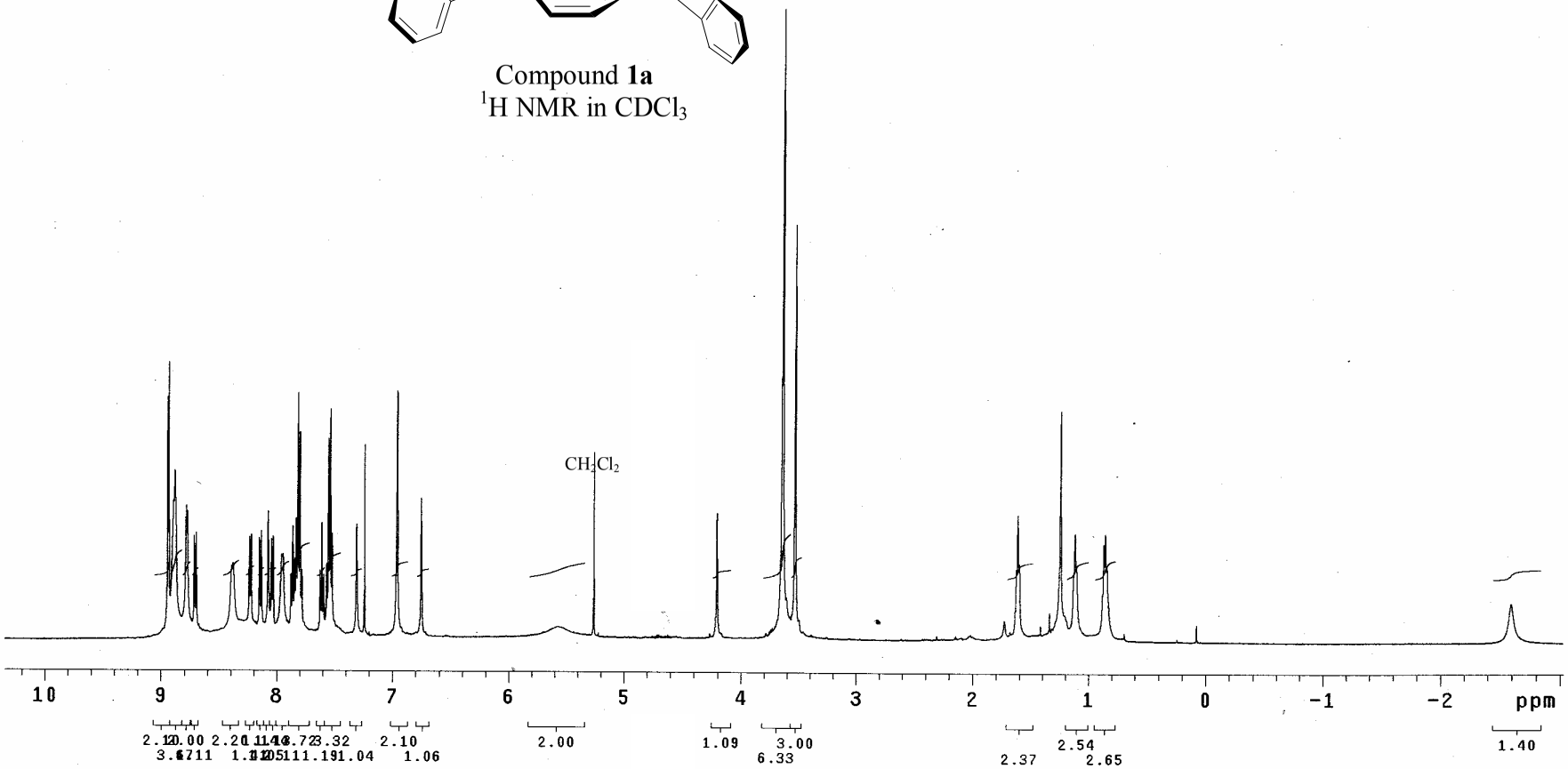
Yilong Yan, Yan-4-41 Cone V = 30 Coll V = 10.0 05-Dec-2005 17:12:13
YANYILONG_051205_16381_YAN-4-41_HRMS 11 (0.473) Cn (Cen,4, 80.00, Ar); Sm (SG, 2x3.00); Sb (2,20.00); Cm (9:12) TOF MS ES+ 1.41e4

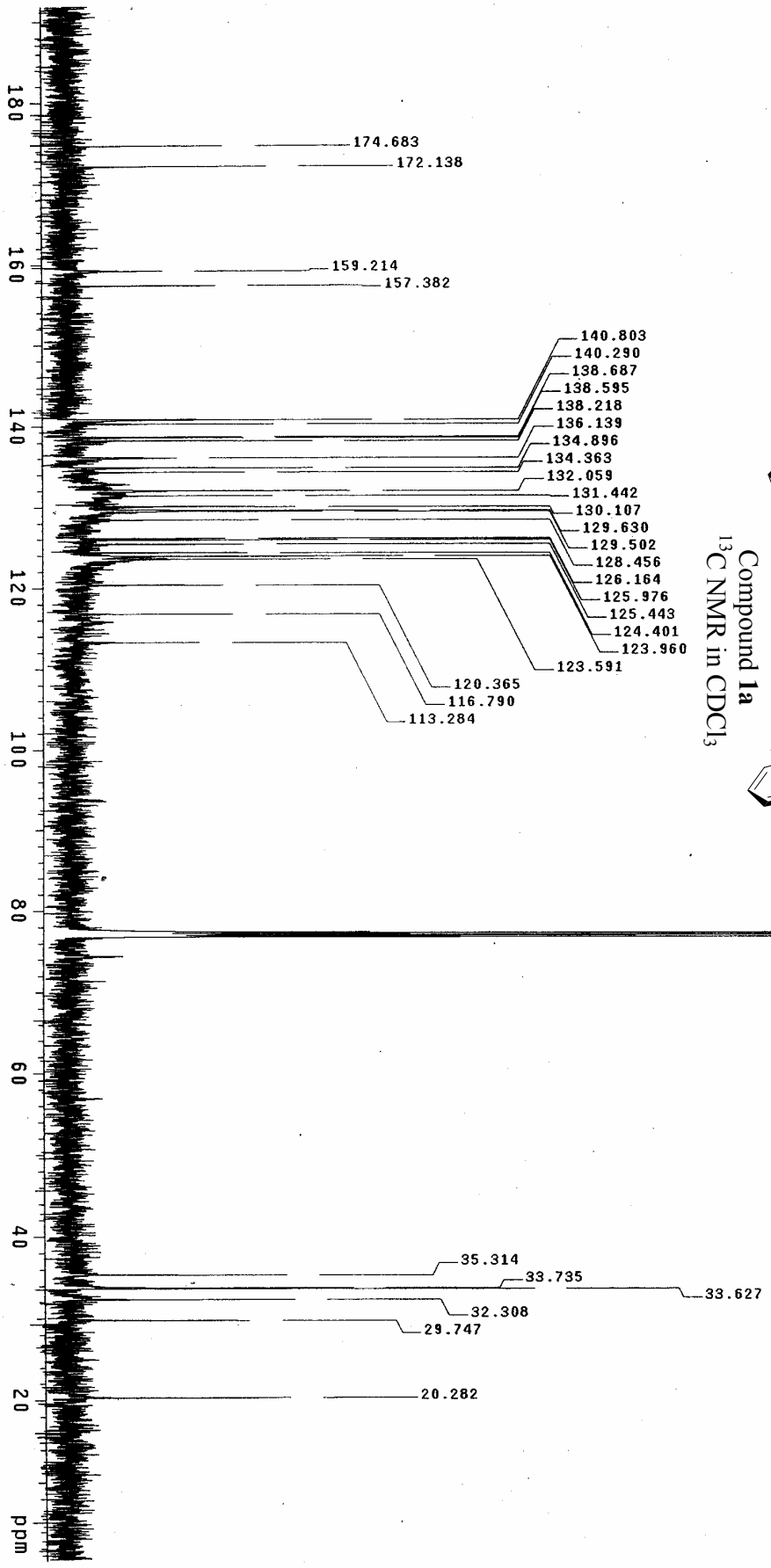


Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
Minimum:				-1.5		
Maximum:		200.0	5.0	120.0		
1253.5844	1253.5846	-0.2	-0.1	57.5	42	C90 H73 N6 O
	1253.5840	0.4	0.3	41.5	9	C76 H78 N8 O8 Na
	1253.5840	0.4	0.3	47.0	10	C75 H72 N15 O3 Na
	1253.5849	-0.5	-0.4	53.5	43	C92 H78 O3 Na
	1253.5838	0.6	0.5	45.5	8	C74 H73 N14 O6 ← [M+H]+
	1253.5851	-0.7	-0.6	45.0	11	C76 H75 N11 O7
	1253.5835	0.9	0.7	54.0	41	C90 H76 N3 O2 Na
	1253.5854	-1.0	-0.8	46.5	13	C77 H74 N12 O4 Na
	1253.5854	-1.0	-0.8	41.0	12	C78 H80 N5 O9 Na
	1253.5832	1.2	0.9	52.5	39	C89 H77 N2 O5
	1253.5832	1.2	0.9	58.0	40	C88 H71 N9
	1253.5859	-1.5	-1.2	57.0	44	C92 H75 N3 O2
	1253.5827	1.7	1.4	42.0	7	C74 H76 N11 O7 Na
	1253.5824	2.0	1.6	40.5	6	C73 H77 N10 O10
	1253.5864	-2.0	-1.6	50.0	15	C77 H71 N15 O3
	1253.5864	-2.0	-1.6	44.5	14	C78 H77 N8 O8
	1253.5822	2.2	1.8	54.5	38	C88 H74 N6 O Na
	1253.5867	-2.3	-1.8	46.0	17	C79 H76 N9 O5 Na
	1253.5867	-2.3	-1.8	40.5	16	C80 H82 N2 O10 Na
	1253.5819	2.5	2.0	53.0	37	C87 H75 N5 O4
	1253.5873	-2.9	-2.3	56.5	45	C94 H77 O3
	1253.5813	3.1	2.4	42.5	5	C72 H74 N14 O6 Na
	1253.5875	-3.1	-2.5	58.0	46	C95 H76 N Na
	1253.5811	3.3	2.7	41.0	4	C71 H75 N13 O9
	1253.5878	-3.4	-2.7	49.5	19	C79 H73 N12 O4
	1253.5878	-3.4	-2.7	44.0	18	C80 H79 N5 O9
	1253.5808	3.6	2.8	49.5	35	C87 H78 N2 O5 Na
	1253.5808	3.6	2.8	55.0	36	C86 H72 N9 Na
	1253.5880	-3.6	-2.9	51.0	21	C80 H72 N13 O Na
	1253.5881	-3.7	-2.9	45.5	20	C81 H78 N6 O6 Na
	1253.5806	3.8	3.1	48.0	33	C86 H79 N O8
	1253.5806	3.8	3.1	53.5	34	C85 H73 N8 O3
	1253.5800	4.4	3.5	37.5	3	C71 H78 N10 O10 Na
	1253.5891	-4.7	-3.8	49.0	23	C81 H75 N9 O5
	1253.5891	-4.7	-3.8	43.5	22	C82 H81 N2 O10
	1253.5795	4.9	3.9	50.0	32	C85 H76 N5 O4 Na
	1253.5894	-5.0	-4.0	50.5	25	C82 H74 N10 O2 Na
	1253.5894	-5.0	-4.0	45.0	24	C83 H80 N3 O7 Na
	1253.5792	5.2	4.1	48.5	30	C84 H77 N4 O7
	1253.5792	5.2	4.1	54.0	31	C83 H71 N11 O2
	1253.5899	-5.5	-4.4	38.0	1	C68 H76 N15 O8 Na
	1253.5900	-5.6	-4.4	61.0	47	C97 H75 N
	1253.5787	5.7	4.6	38.0	2	C69 H76 N13 O9 Na
	1253.5905	-6.1	-4.8	54.0	29	C82 H71 N13 O
	1253.5905	-6.1	-4.8	48.5	27	C83 H77 N6 O6
	1253.5782	6.2	5.0	45.0	26	C84 H80 N O8 Na
	1253.5782	6.2	5.0	50.5	28	C83 H74 N8 O3 Na

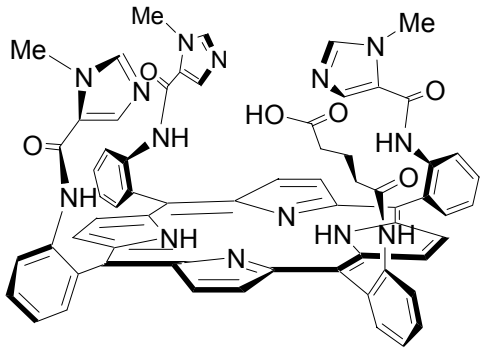


Compound **1a**
^{1}H NMR in $CDCl_3$

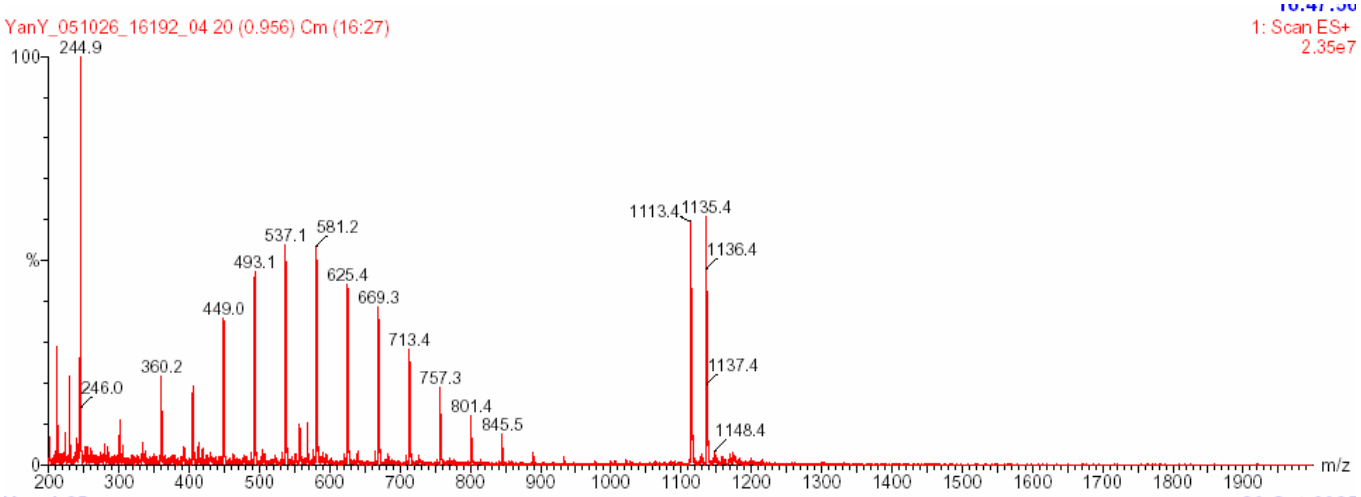


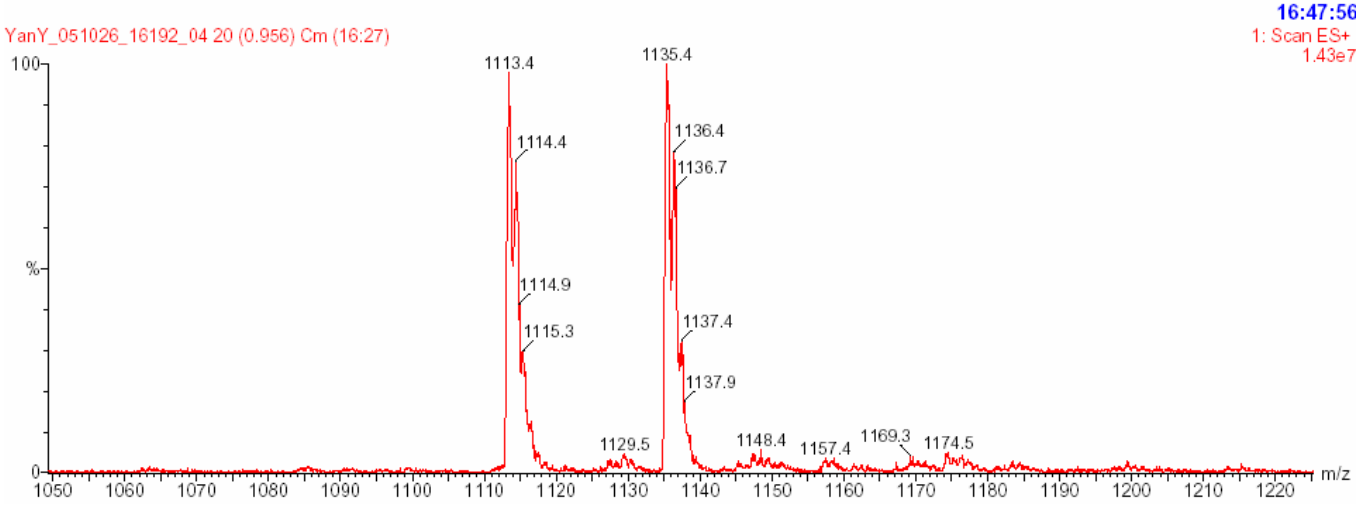


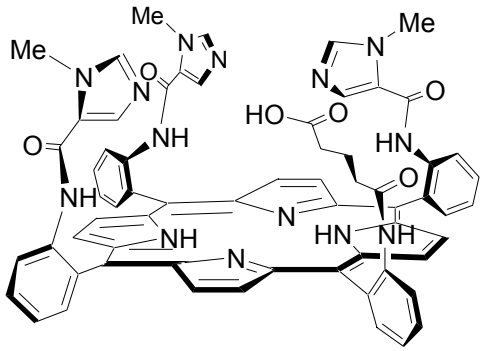
Compound **1a**
^{13}C NMR in $CDCl_3$



Compound **1a**, Low resolution mass analysis







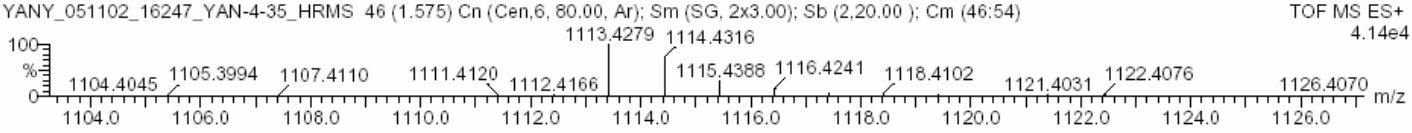
Compound **1a**, High resolution mass analysis

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 120.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

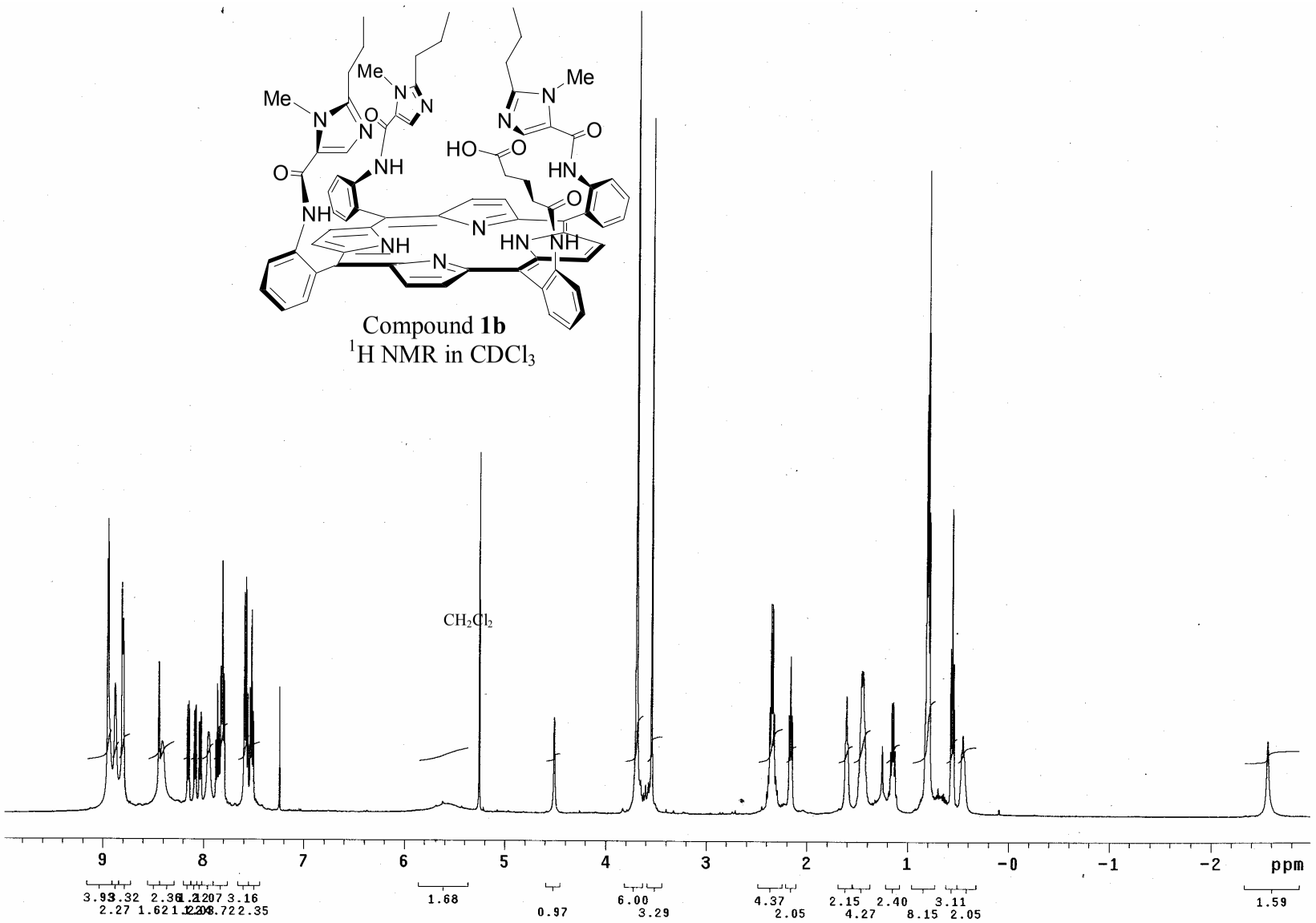
Monoisotopic Mass, Odd and Even Electron Ions
1811 formula(e) evaluated with 21 results within limits (up to 140 closest results for each mass)

Yilong Yan, Yan-4-35 Cone V = 30 02-Nov-2005
16129 Coll V = 10.0 11:25:01
YANY_051102_16247_YAN-4-35_HRMS 46 (1.575) Cn (Cen,6, 80.00, Ar); Sm (SG, 2x3.00); Sb (2,20.00); Cm (46:54) TOF MS ES+
4.14e4

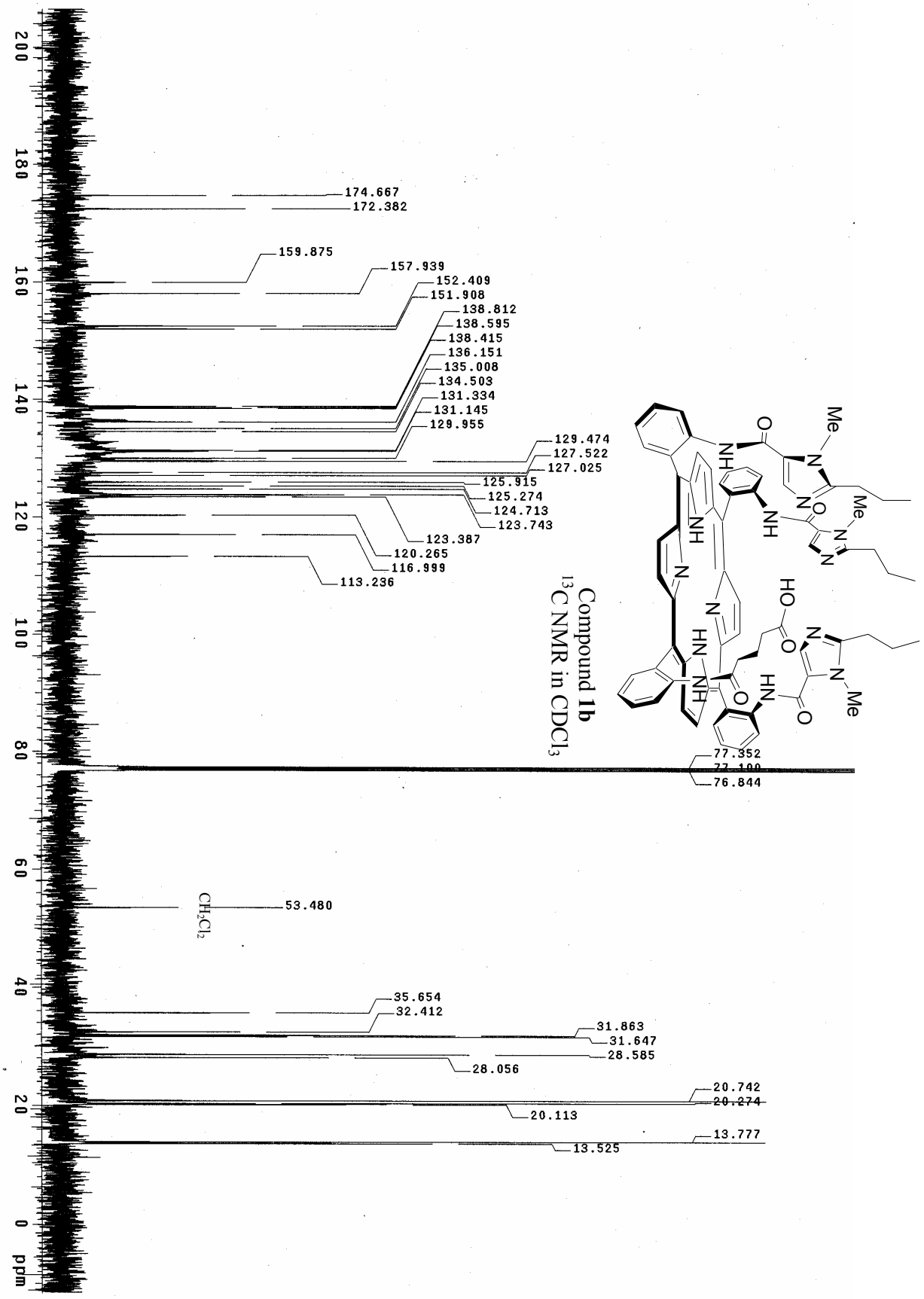


Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
Minimum:				-1.5		
Maximum:		200.0	5.0	120.0		
1113.4279	1113.4281	-0.2	-0.2	57.5	18	C80 H53 N6 O
	1113.4273	0.6	0.6	45.5	3	C64 H53 N14 O6
	1113.4286	-0.7	-0.6	45.0	4	C66 H55 N11 O7
	1113.4267	1.2	1.0	52.5	16	C79 H57 N2 O5
	1113.4267	1.2	1.0	58.0	17	C78 H51 N9
	1113.4294	-1.5	-1.4	57.0	19	C82 H55 N3 O2
	1113.4259	2.0	1.8	40.5	1	C63 H57 N10 O10
	1113.4299	-2.0	-1.8	50.0	8	C67 H51 N15 O3
	1113.4299	-2.0	-1.8	44.5	5	C68 H57 N8 O8
	1113.4254	2.5	2.2	53.0	15	C77 H55 N5 O4
	1113.4308	-2.9	-2.6	56.5	20	C84 H57 O3
	1113.4246	3.3	3.0	41.0	2	C61 H55 N13 O9
	1113.4313	-3.4	-3.0	49.5	9	C69 H53 N12 O4
	1113.4313	-3.4	-3.0	44.0	7	C70 H59 N5 O9
	1113.4241	3.8	3.4	48.0	12	C76 H59 N O8
	1113.4241	3.8	3.4	53.5	14	C75 H53 N8 O3
	1113.4326	-4.7	-4.2	49.0	10	C71 H55 N9 O5
	1113.4326	-4.7	-4.2	43.5	6	C72 H61 N2 O10
	1113.4227	5.2	4.6	48.5	11	C74 H57 N4 O7
	1113.4227	5.2	4.7	54.0	13	C73 H51 N11 O2
	1113.4335	-5.6	-5.0	61.0	21	C87 H55 N

$[M+H]^+$



Compound **1b**
^{1}H NMR in $CDCl_3$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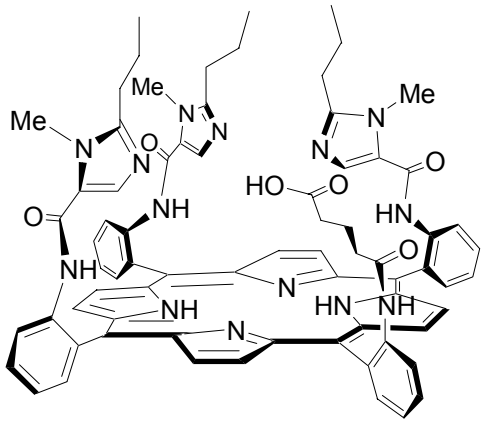
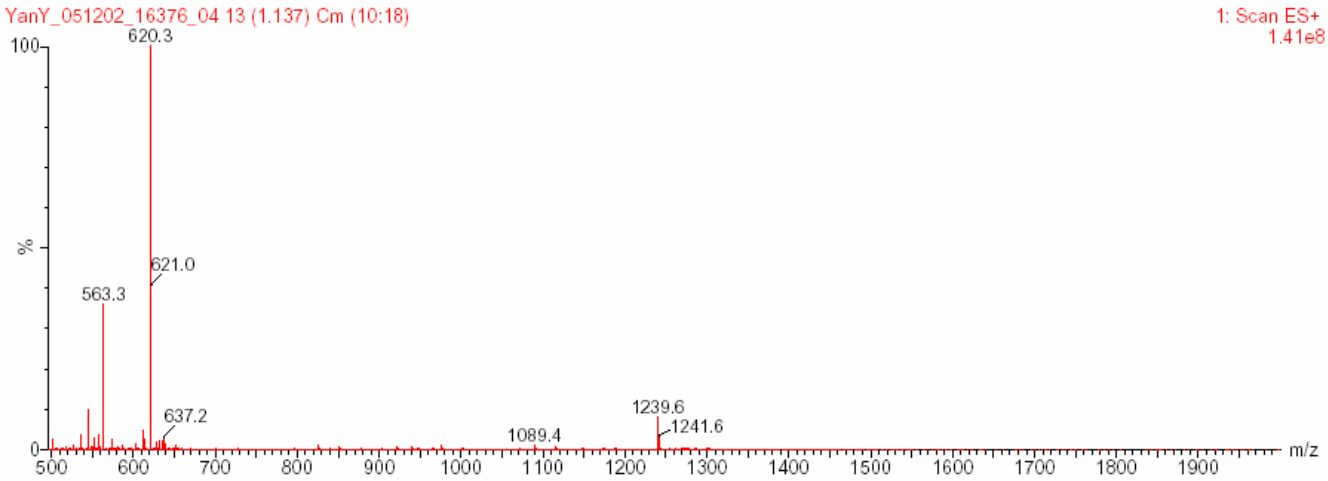
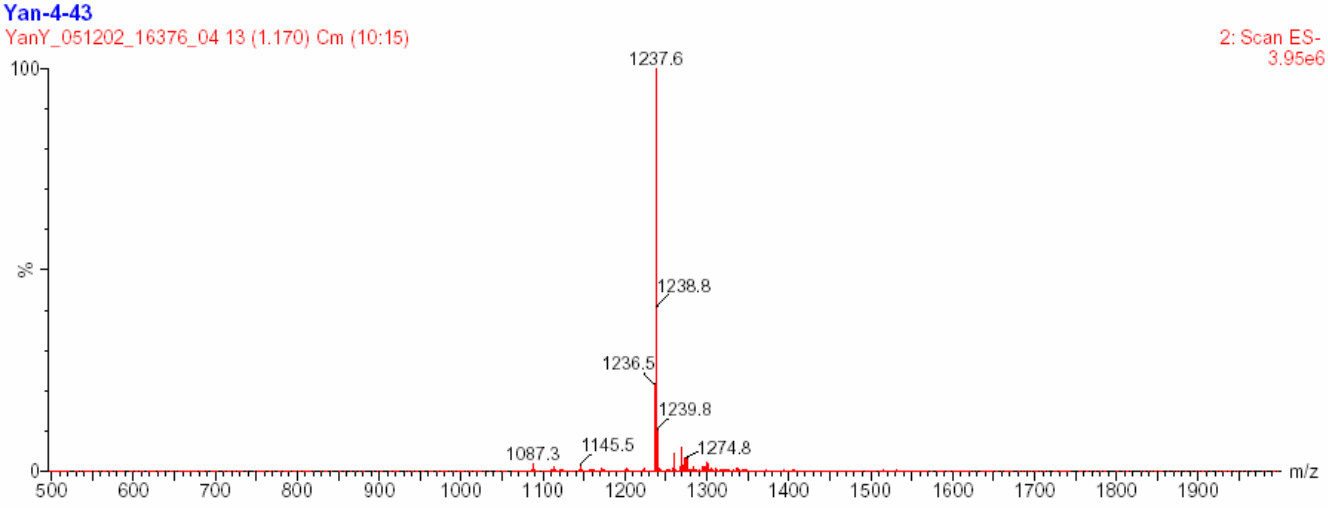
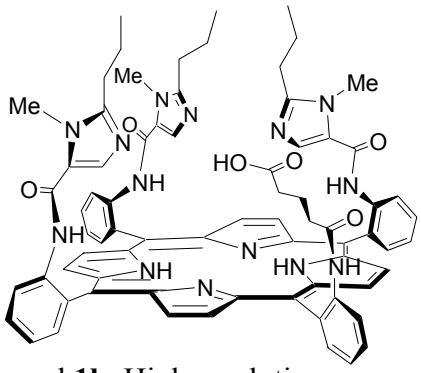
172.382
159.875
157.939
152.409
151.908
138.812
138.595
138.415
136.151
135.008
134.503
131.334
131.145
129.955
129.474
127.522
127.025
125.915
125.274
124.713
123.743
123.387
120.265
116.999
113.236
77.352
77.100
76.844
53.480
CH2Cl2
35.654
32.412
31.863
31.647
28.585
28.056
20.742
20.274
20.113
13.777
13.525
ppm
Compound 1b
13C NMR in CDCl3



Compound **1b**, Low resolution mass analysis







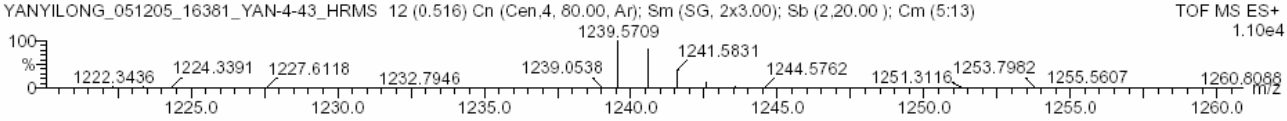
Compound **1b**, High resolution mass analysis

Elemental Composition Report **Page 1**

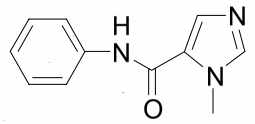
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 120.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
4924 formula(e) evaluated with 48 results within limits (up to 140 closest results for each mass)

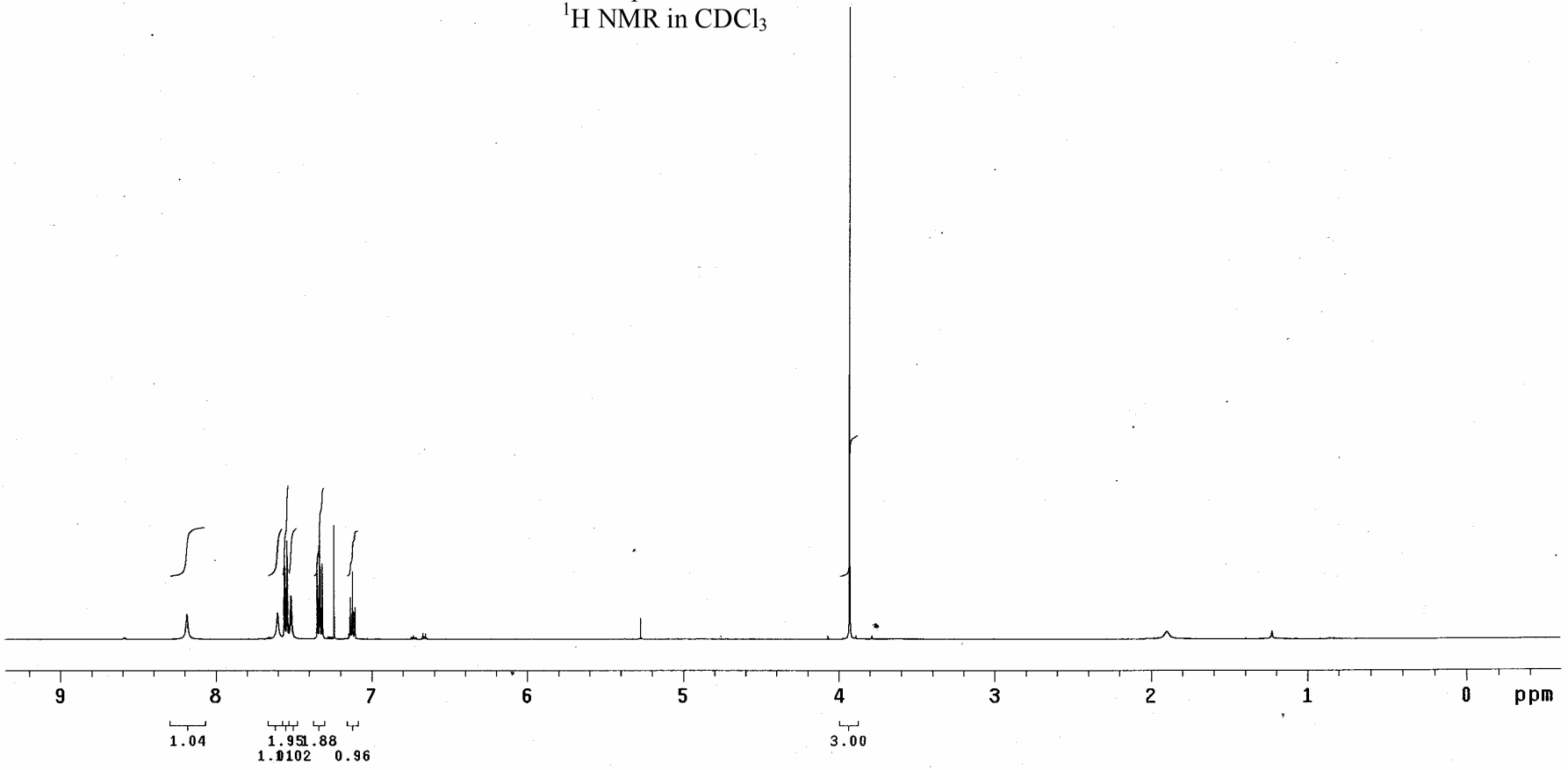
Yilong Yan, Yan-4-43 Cone V = 30 Coll V = 10.0 05-Dec-2005 17:01:48
YANYILONG_051205_16381_YAN-4-43_HRMS 12 (0.516) Cn (Cen,4, 80.00, Ar); Sm (SG, 2x3.00); Sb (2,20.00); Cm (5:13) TOF MS ES+ 1.10e4

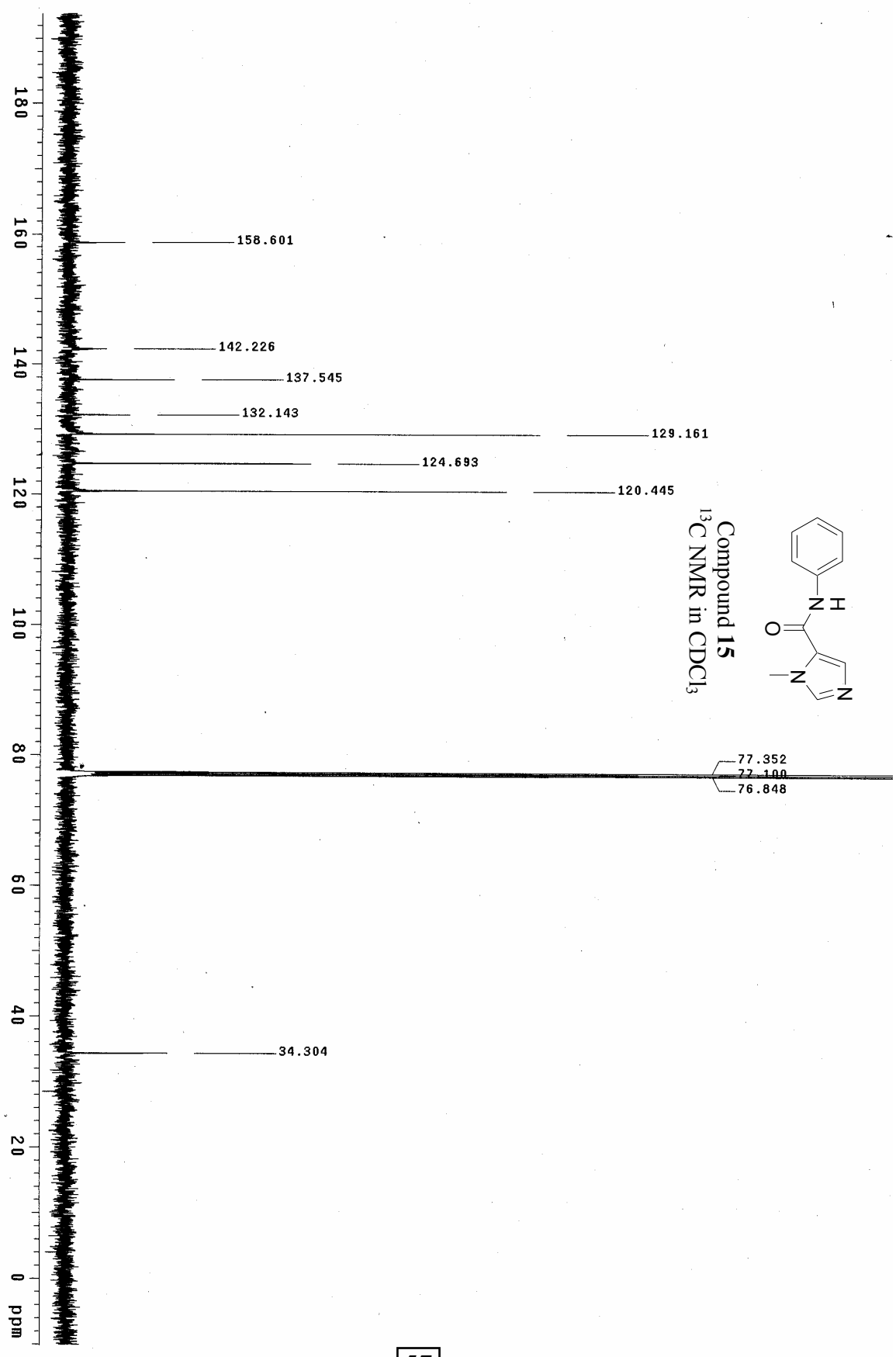


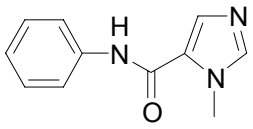
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
Minimum:				-1.5		
Maximum:		200.0	5.0	120.0		
1239.5709	1239.5708	0.1	0.1	44.5	15	C77 H75 N8 O8
	1239.5708	0.1	0.1	50.0	17	C76 H69 N15 O3
	1239.5711	-0.2	-0.1	46.0	18	C78 H74 N9 O5 Na
	1239.5711	-0.2	-0.1	40.5	16	C79 H80 N2 O10 Na
	1239.5703	0.6	0.5	57.0	45	C91 H73 N3 O2
	1239.5716	-0.7	-0.6	56.5	46	C93 H75 O3
	1239.5719	-1.0	-0.8	58.0	47	C94 H74 N Na
	1239.5697	1.2	0.9	41.0	13	C77 H78 N5 O9 Na
	1239.5697	1.2	1.0	46.5	14	C76 H72 N12 O4 Na
	1239.5721	-1.2	-1.0	49.5	20	C78 H71 N12 O4
	1239.5721	-1.2	-1.0	44.0	19	C79 H77 N5 O9
	1239.5694	1.5	1.2	45.0	12	C75 H73 N11 O7
	1239.5724	-1.5	-1.2	51.0	23	C79 H70 N13 O Na
	1239.5724	-1.5	-1.2	45.5	21	C80 H76 N6 O6 Na
	1239.5692	1.7	1.4	53.5	44	C91 H76 O3 Na
	1239.5689	2.0	1.6	57.5	43	C89 H71 N6 O
	1239.5684	2.5	2.0	41.5	9	C75 H76 N8 O8 Na
	1239.5684	2.5	2.0	47.0	10	C74 H70 N15 O3 Na
	1239.5735	-2.6	-2.1	49.0	24	C80 H73 N9 O5
	1239.5735	-2.6	-2.1	43.5	22	C81 H79 N2 O10
	1239.5681	2.8	2.3	45.5	8	C73 H71 N14 O6 ← $[M+H]^+$
	1239.5737	-2.8	-2.3	50.5	26	C81 H72 N10 O2 Na
	1239.5737	-2.8	-2.3	45.0	25	C82 H78 N3 O7 Na
	1239.5679	3.0	2.4	54.0	42	C89 H74 N3 O2 Na
	1239.5676	3.3	2.7	52.5	40	C88 H75 N2 O5
	1239.5676	3.3	2.7	58.0	41	C87 H69 N9
	1239.5742	-3.3	-2.7	38.0	11	C67 H74 N15 O8 Na
	1239.5743	-3.4	-2.7	61.0	48	C96 H73 N
	1239.5670	3.9	3.1	42.0	7	C73 H74 N11 O7 Na
	1239.5748	-3.9	-3.1	54.0	29	C81 H69 N13 O
	1239.5748	-3.9	-3.2	48.5	27	C82 H75 N6 O6
	1239.5668	4.1	3.3	40.5	3	C72 H75 N10 O10
	1239.5751	-4.2	-3.4	50.0	30	C83 H74 N7 O3 Na
	1239.5751	-4.2	-3.4	44.5	28	C84 H80 O8 Na
	1239.5665	4.4	3.5	54.5	39	C87 H72 N6 O Na
	1239.5663	4.6	3.7	53.0	38	C86 H73 N5 O4
	1239.5756	-4.7	-3.8	37.5	6	C69 H76 N12 O9 Na
	1239.5657	5.2	4.2	42.5	5	C71 H72 N14 O6 Na
	1239.5761	-5.2	-4.2	53.5	32	C83 H71 N10 O2
	1239.5762	-5.3	-4.2	48.0	31	C84 H77 N3 O7
	1239.5654	5.5	4.4	41.0	2	C70 H73 N13 O9
	1239.5764	-5.5	-4.5	49.5	34	C85 H76 N4 O4 Na
	1239.5652	5.7	4.6	49.5	36	C86 H76 N2 O5 Na
	1239.5652	5.7	4.6	55.0	37	C85 H70 N9 Na
	1239.5767	-5.8	-4.6	41.0	4	C69 H73 N15 O8
	1239.5649	6.0	4.8	48.0	33	C85 H77 N O8
	1239.5649	6.0	4.8	53.5	35	C84 H71 N8 O3
	1239.5769	-6.0	-4.9	37.0	1	C71 H78 N9 O10 Na



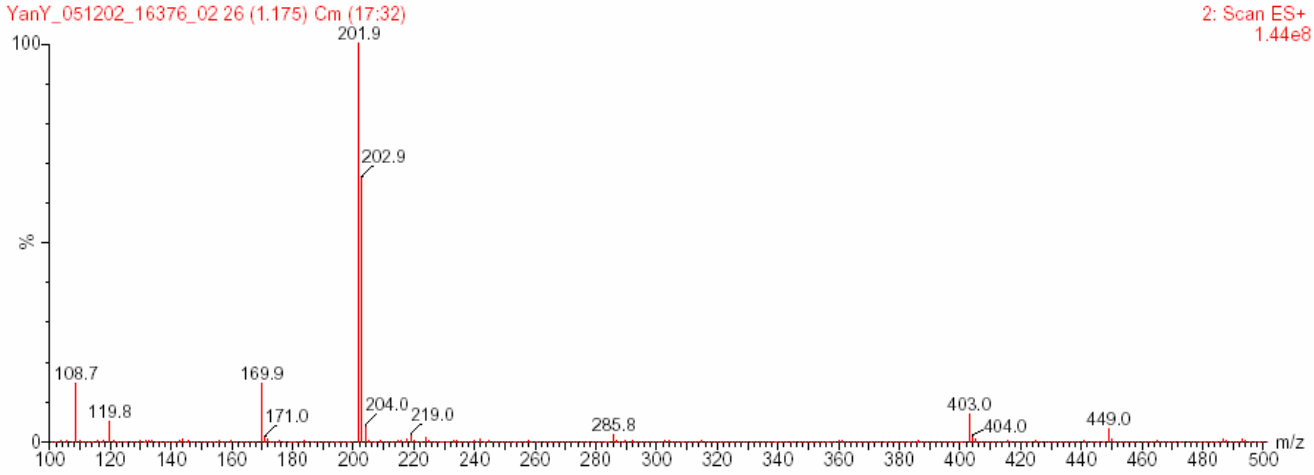
Compound **15**
^{1}H NMR in $CDCl_3$




158.601
142.226
137.545
132.143
129.161
124.693
120.445
77.352
77.100
76.848
34.304
180
160
140
120
100
80
60
40
20
0
ppm
Compound 15
13C NMR in CDCl3
H
N
O
N
N



Compound **15**, Low resolution mass analysis



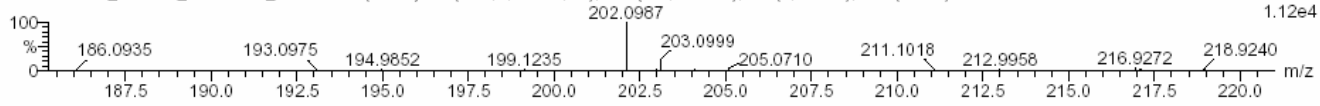
Compound **15**, High resolution mass analysis

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 120.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

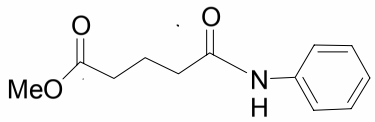
Monoisotopic Mass, Odd and Even Electron Ions
359 formula(e) evaluated with 2 results within limits (up to 140 closest results for each mass)

Yilong Yan, Yan-4-45
Cone V = 30
Coll V = 30.0
05-Dec-2005
16:16:46
YANYILONG_051205_YAN-4-45_HRMS 56 (2.375) Cn (Cen,4, 80.00, Ar); Sm (SG, 2x3.00); Sb (2,20.00); Cm (45:61)
TOF MS ES+
1.12e4

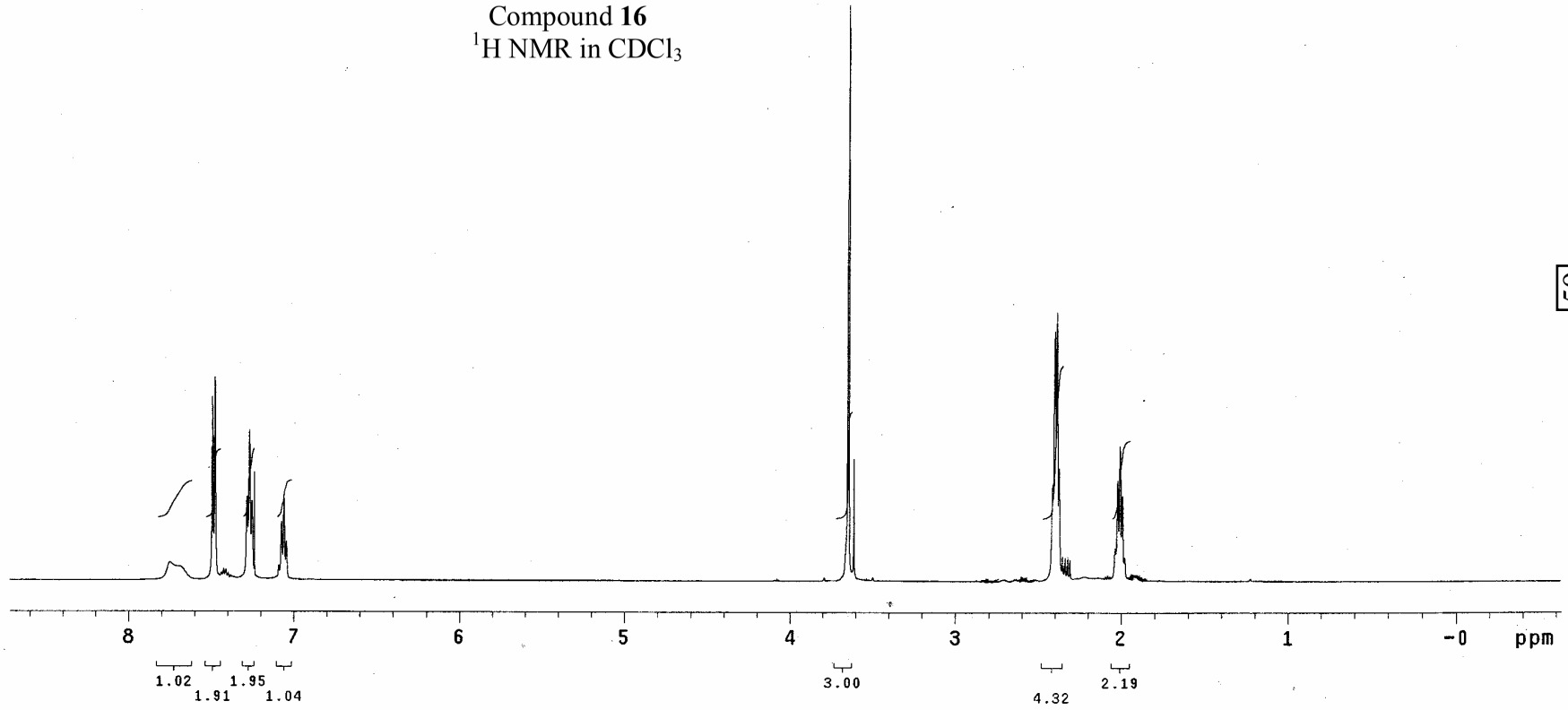


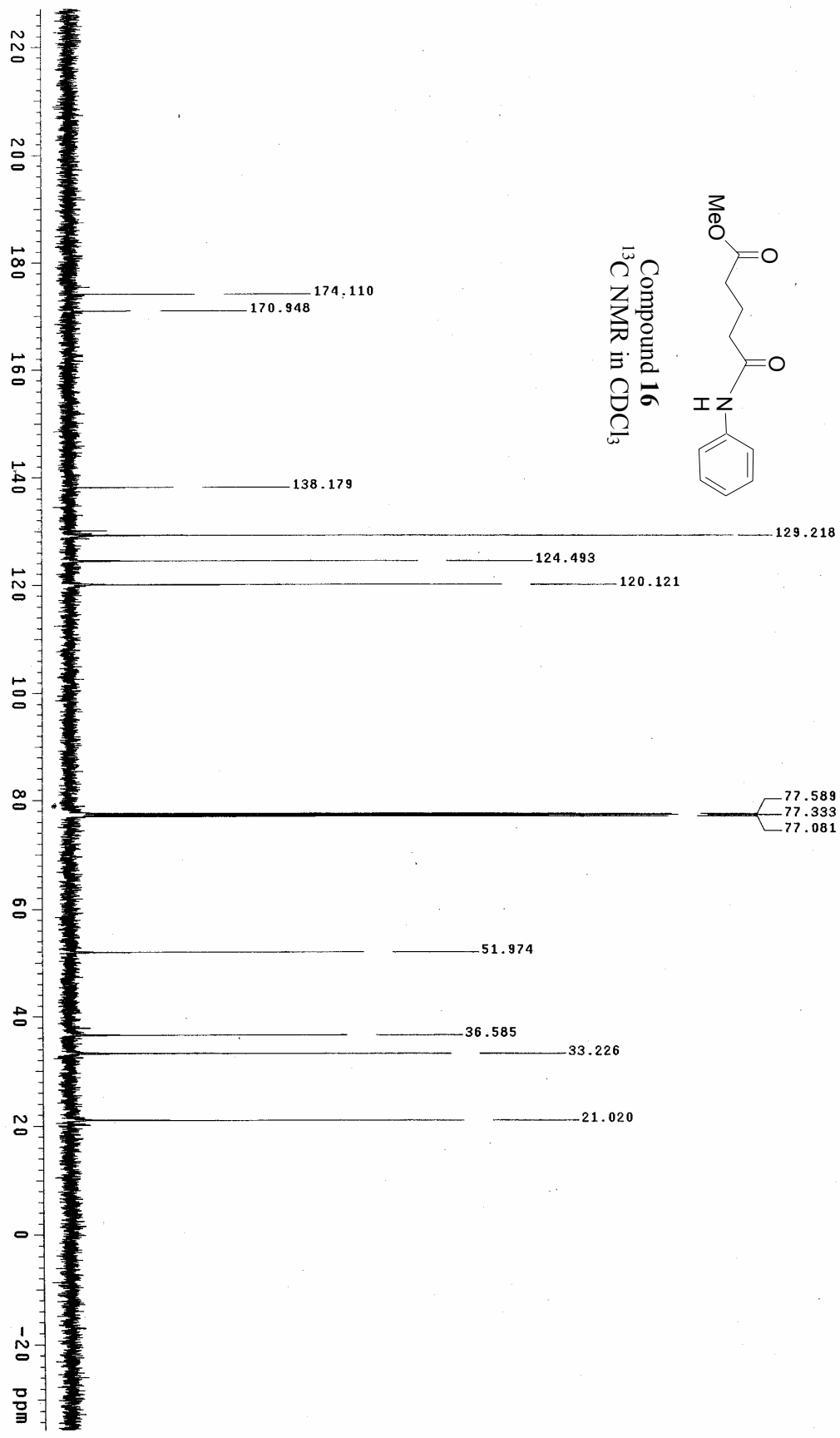
Minimum:				-1.5		
Maximum:		200.0	5.0	120.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
202.0987	202.0980	0.7	3.3	7.5	2	C11 H12 N3 O
	202.0994	-0.7	-3.4	7.0	1	C13 H14 O2

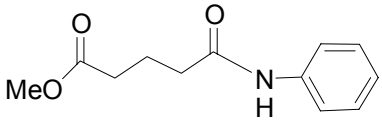
$[M+H]^+$



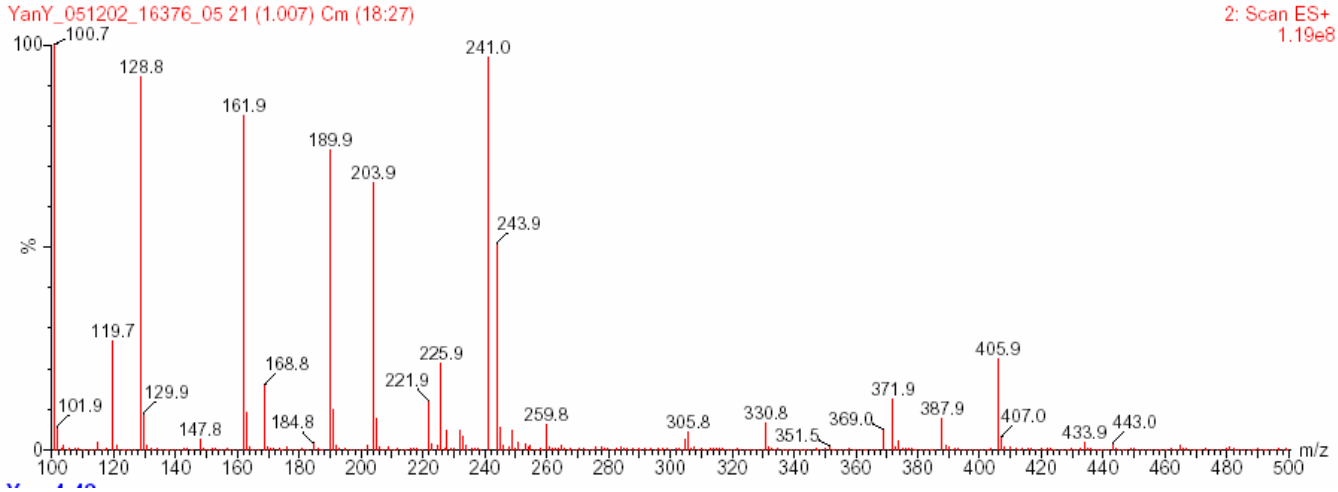
Compound **16**
^{1}H NMR in $CDCl_3$




Compound 16
^{13}C NMR in $CDCl_3$
MeO
O
O
N
H
174.110
170.948
138.179
129.218
124.493
120.121
77.589
77.333
77.081
51.974
36.585
33.226
21.020
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm



Compound **16**, Low resolution mass analysis



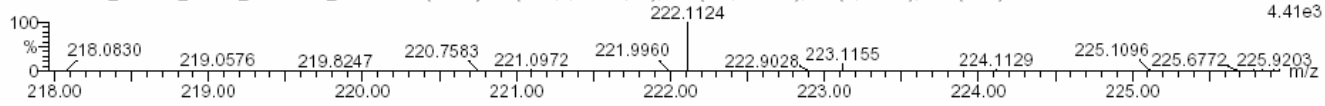
Compound **16**, High resolution mass analysis

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 120.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
439 formula(e) evaluated with 3 results within limits (up to 140 closest results for each mass)

Yilong Yan, Yan-4-42
Cone V = 30
Coll V = 10.0
05-Dec-2005
16:34:21
YANYILONG_051205_16381_YAN-4-42_HRMS 10 (0.430) Cn (Cen,4, 80.00, Ar); Sm (SG, 2x3.00); Sb (2,20.00); Cm (6:11)
TOF MS ES+
4.41e3



Minimum:				-1.5		
Maximum:		200.0	5.0	120.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
222.1124	222.1130	-0.6	-2.8	5.5	1	C12 H16 N O3
	222.1117	0.7	3.3	6.0	3	C10 H14 N4 O2
	222.1133	-0.9	-4.0	7.0	2	C13 H15 N2 Na

$[M+H]^+$