

# Dioxygen-binding of water-soluble iron(II) porphyrins in phosphate buffer at room temperature

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**$\alpha$ -5,10,15,20-{2-[3,3',3'',3''']-(*N,N,N',N''*-Tris(2-aminoethyl)amine)(*N',N''*-biscarboxy methyl)tetrapropionamido]tetraphenyl}porphyrin **3**.**  $\text{C}_{66}\text{H}_{64}\text{N}_{12}\text{O}_8$ . In a round bottom flask equipped with a stir bar porphyrin **2** (0.01 mmol, 12 mg) was charged with a methanol/ chloroform mixture (1/ 5) (10 mL) and potassium hydroxide (0.1 mmol, 5.5 mg). The reaction mixture was heated to 50 °C overnight then solvents were removed under vacuum. The resulting powder was dissolved in water then hydrochloric acid 2N was added to pH 7. The precipitate was filtrated, washed with diethyl ether and dried for several hours. The expected compound was obtained in 87% yield (10 mg).  $\delta_{\text{H}}$  (500.13 MHz, DMSO- $d_6$ , 323 K) -2.68 (2H, broad s), -2.49 (2H, s,  $-\text{NH}_{\text{pyr}}$ ), -1.24 (4H, broad s), 0.41 (8H, broad s), 1.60 (2H, broad s), 2.14-1.79 (10H, m), 2.21 (4H, broad s), 2.31 (2H, broad s), 7.42 (2H, t,  $^3J = 7.2$ ,  $\text{H}_{\text{aro}}$ ), 7.56 (4H, d,  $^3J = 6.8$ ,  $\text{H}_{\text{aro}}$ ), 7.62 (2H, t,  $^3J = 7.8$ ,  $\text{H}_{\text{aro}}$ ), 7.80 (2H, t,  $^3J = 8.2$ ,  $\text{H}_{\text{aro}}$ ), 7.86 (2H, t,  $^3J = 8.2$ ,  $\text{H}_{\text{aro}}$ ), 8.04 (2H, d,  $J = 7.3$ ,  $\text{H}_{\text{aro}}$ ), 8.24 (2H, broad s,  $\text{H}_{\text{aro}}$ ), 8.38 (2H, d,  $J = 8.3$ ,  $\text{H}_{\text{aro}}$ ), 8.57 (2H, broad

s, H<sub>βpyr</sub>), 8.67 (2H, s, H<sub>βpyr</sub>), 8.81 (2H, d, *J* = 4.6, H<sub>βpyr</sub>), 8.82 (2H, d, *J* = 4.6, H<sub>βpyr</sub>), 10.01 (2H, s, -NHCO) and 11.79 (2H, broad s, -NHCO); *m/z* (ESI HRMS) 1153.5052 ([M + H]<sup>+</sup> C<sub>66</sub>H<sub>65</sub>N<sub>12</sub>O<sub>8</sub> requires 1153.5048); λ<sub>max</sub>(CHCl<sub>3</sub>/ DMF (1/ 1))/nm 423.5 (10<sup>-3</sup>ε/dm<sup>3</sup> mol<sup>-1</sup> cm<sup>-1</sup> 264.0), 517.0 (16.5); 550.5 (4.4); 590.5 (4.9) and 645.5 (2.0).

**α-5,10,15,20-{2-[3,3',3'',3''']-(*N,N,N',N''*-Tris(2-aminoethyl)amine) (*N',N''*-(acetic acid 2-{2-[2-ureido-ethoxy]-ethoxy}-ethyl ester)tetrapropionamido]tetraphenyl} porphyrin 7.** C<sub>80</sub>H<sub>90</sub>N<sub>14</sub>O<sub>14</sub>. In a 100 mL round bottom flask equipped with a stir bar and a gas inlet compound **12** (1.20 mmol, 230 mg) was charged with CH<sub>2</sub>Cl<sub>2</sub> (50 mL) and Et<sub>3</sub>N (0.5 mL). The solution was cooled to 0 °C then diphosgene (0.60 mmol, 72 μL) was added dropwise. The solution was stirred at room temperature for 1 h then porphyrin **1** (0.14 mmol, 150 mg) was added. The solution was stirred overnight then solvent was removed under vacuum. The resulting powder was dissolved in CH<sub>2</sub>Cl<sub>2</sub> and directly loaded on a silica gel chromatography column. The expected compound eluted with 4 to 6% MeOH/ CH<sub>2</sub>Cl<sub>2</sub> was obtained in 78% yield (160 mg). <sup>1</sup>H NMR δ<sub>H</sub> (500 MHz, DMSO-*d*<sub>6</sub>, 323 K): δ = 10.01 (s, 2H, -NH); 8.94 (s, 2H, -NH); 8.71 (d, 2H, *J* = 4.6, H<sub>βpyr</sub>); 8.63 (d, 2H, *J* = 4.6, H<sub>βpyr</sub>); 8.53 (s, 2H, H<sub>βpyr</sub>); 8.48 (s, 2H, H<sub>βpyr</sub>); 8.27 (broad s, 2H, H<sub>aro</sub>); 8.10 (d, 2H, *J* = 7.1, H<sub>aro</sub>); 7.81 (m, 6H, H<sub>aro</sub>); 7.58 (m, 4H, H<sub>aro</sub>); 7.50 (t, 2H, *J* = 7.1 Hz, H<sub>aro</sub>); 6.07 (broad s, 2H, NCONH); 4.15 (t, 4H, *J* = 4.7); 3.65 (t, 4H, *J* = 4.7); 3.60 (m, 4H); 3.55 (m, 6H); 3.41 (t, 4H, *J* = 5.3); 3.12 (m, 4H); 2.99 (m, 4H); 2.30 (m, 2H); 2.13 (m, 2H); 2.01 (s, 6H, COCH<sub>3</sub>); 1.65 (m, 2H); 1.50 (broad s, 6H); 1.22 (broad s, 2H); 0.45 (t, 2H, *J* = 7.0); -1.26 (broad s, 2H); -1.70 (broad s, 2H); -2.82 (s, 2H, -NH<sub>pyr</sub>); -2.90 (broad s, 2H); *m/z* (ESI HRMS) 1493.6687 ([M + Na]<sup>+</sup> C<sub>80</sub>H<sub>90</sub>N<sub>14</sub>O<sub>14</sub>Na requires 1493.6658); λ<sub>max</sub>(CH<sub>2</sub>Cl<sub>2</sub>)/nm 419.0 (10<sup>-3</sup>ε/dm<sup>3</sup> mol<sup>-1</sup> cm<sup>-1</sup>) 419.0 (331.1); 512.0 (16.5); 544.5 (3.2); 585.0 (4.7); 640.5 (1.4).

**α-5,10,15,20-{2-[3,3',3'',3''']-(*N,N,N',N''*-Tris(2-aminoethyl)amine) (*N',N''*-bis(3-{2-[2-(2-hydroxy-ethoxy)-ethoxy]-ethyl}-urea)tetrapropionamido]tetraphenyl}porphyrin 8.** C<sub>76</sub>H<sub>86</sub>N<sub>14</sub>O<sub>12</sub>. In a 100 mL round bottom flask equipped with a stir bar compound **7** (0.033 mmol, 50 mg) and potassium carbonate (0.134 mmol, 16 mg) was charged with MeOH (20 mL). The solution was stirred at 60 °C overnight then solvent was removed under vacuum. The resulting powder was dissolved in CHCl<sub>3</sub> and directly loaded on a silica gel chromatography column. The expected compound eluted with 12% MeOH/ CHCl<sub>3</sub> was obtained in 83% yield (45 mg). δ<sub>H</sub> (500.13 MHz, DMSO-*d*<sub>6</sub>, 343 K) -2.82 (2H, broad s), -2.76 (2H, s, -NH<sub>pyr</sub>), -1.62 (2H, broad s), -1.24 (2H, broad s), 0.48 (2H, t, *J* = 8.5), 1.52 (6H, m), 1.66 (2H, m), 2.13 (2H, m), 2.30 (2H, m), 3.00 (4H, broad s), 3.10 (4H, m), 3.42 (4H, t, *J* = 5.7), 3.50 (4H, m), 3.55 (8H, m), 3.60 (4H, m), 4.31 (2H, broad s, -OH), 6.02 (2H, broad s, NCONH), 7.51 (2H, m, H<sub>aro</sub>), 7.59 (4H, m, H<sub>aro</sub>), 7.83 (6H, m, H<sub>aro</sub>), 8.10 (2H, d, *J* = 7.2, H<sub>aro</sub>), 8.28 (2H, broad d, *J* = 7.2, H<sub>aro</sub>), 8.49 (2H, s, H<sub>βpyr</sub>), 8.54 (2H, s, H<sub>βpyr</sub>), 8.63 (2H, d, *J* = 4.7, H<sub>βpyr</sub>), 8.72 (2H, d, *J* = 4.7, H<sub>βpyr</sub>), 8.89 (2H, s, -NH) and 9.91 (2H, s, -NH); *m/z* (ESI HRMS) 1409.6448 ([M + Na]<sup>+</sup> C<sub>76</sub>H<sub>86</sub>N<sub>14</sub>O<sub>12</sub>Na

requires 1409.6447);  $\lambda_{\max}(\text{CH}_2\text{Cl}_2)/\text{nm}$  419.0 ( $10^{-3}\epsilon/\text{dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$ ) 419.0 (329.6), 512.5 (14.6); 545.0 (3.2); 585.5 (4.6) and 641.5 (1.4).

**Acetic acid 2-[2-(2-hydroxy-ethoxy)-ethoxy]-ethyl ester 9.**  $\text{C}_8\text{H}_{16}\text{O}_5$ . In a 250 mL round bottom flask triethylene glycol (0.1 mol, 15 g) was charged with pyridine (100 mL). The reaction mixture was stirred at 0 °C then acetyl chloride (0.07 mol, 5 mL) was added dropwise over 1 h. The reaction mixture was stirred at room temperature overnight then solvent was removed under vacuum. The resulting oil dissolved in  $\text{CH}_2\text{Cl}_2$  was directly loaded on a silica gel column chromatography. The expected compound eluted with 2% MeOH/  $\text{CH}_2\text{Cl}_2$  was obtained as a colourless oil in 35% yield (6.7 g).  $\delta_{\text{H}}$  (200 MHz,  $\text{CDCl}_3$ , 298 K) 1.99 (3H, s,  $\text{COCH}_3$ ), 2.83 (1H, broad s, OH), 3.51 (2H, t,  $J = 4.7$ ), 3.58 (4H, s), 3.61 (4H, t,  $J = 4.9$ ) and 4.14 (2H, t,  $J = 4.9$ ).

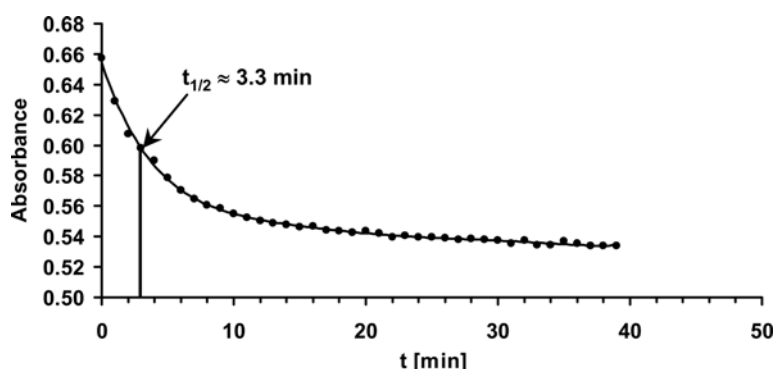
**Acetic acid 2-[2-(2-bromo-ethoxy)-ethoxy]-ethyl ester 10.**  $\text{C}_8\text{H}_{15}\text{O}_4\text{Br}$ . In a 100 mL round bottom flask compound **9** (7.8 mmol, 1.5 g) was charged with freshly distilled  $\text{CH}_2\text{Cl}_2$  (50 mL). The reaction mixture was stirred at -20 °C then triphenyl phosphine (9.4 mmol, 2.46 g) and *N*-bromosuccinimide (9.4 mmol, 1.53 g) were added. The reaction mixture was stirred at room temperature overnight then evaporated under vacuum. The resulting oil dissolved in  $\text{CH}_2\text{Cl}_2$  was directly loaded on a silica gel column chromatography. The expected compound eluted with  $\text{CH}_2\text{Cl}_2$  was obtained as a colourless oil in 78% yield (1.56 g).  $\delta_{\text{H}}$  (300.13 MHz,  $\text{CDCl}_3$ , 298 K) 2.08 (3H, s,  $\text{COCH}_3$ ), 3.47 (2H, t,  $J = 6.2$ ), 3.67 (4H, s), 3.70 (2H, t,  $J = 4.8$ ), 3.81 (2H, t,  $J = 6.2$ ) and 4.22 (2H, t,  $J = 4.5$ );  $\delta_{\text{C}}$  (50.3 MHz,  $\text{CDCl}_3$ , 298 K) 171.5; 71.8; 71.1; 71.0; 69.6; 64.0; 43.1; 21.4.  $m/z$  (ESI HRMS) 149.9680 ( $[\text{M}^+\text{C}_4\text{H}_8\text{O}_3]^+$  requires 149.9680).

**Acetic acid 2-{2-[2-(1,3-dioxo-1,3-dihydro-isoindol-2-yl)-ethoxy]-ethoxy}-ethyl ester 11.**  $\text{C}_{16}\text{H}_{19}\text{NO}_6$ . In a 250 mL round bottom flask compound **10** (5.2 mmol, 1 g) was charged with THF (50 mL). The reaction mixture was stirred at 0 °C then phthalimide (5.2 mmol, 765 mg) and triphenylphosphine (5.2 mmol, 1.36 g) were added. After dissolution, diisopropyl azodicarboxylate (5.2 mmol, 1.02 mL) was added dropwise. The reaction mixture was stirred 4 h at room temperature then solvent was removed under vacuum. The resulting oil dissolved in hexane was directly loaded on a silica gel column chromatography. The expected compound eluted with 20% ethyl acetate/ hexane was obtained as a colourless oil in 60% yield (1 g).  $\delta_{\text{H}}$  (300.13 MHz,  $\text{CDCl}_3$ , 298 K) 2.00 (3H, s,  $\text{COCH}_3$ ), 3.57 (2H, s), 3.58 (4H, s); 3.69 (2H, t,  $J = 5.4$ ), 3.85 (2H, t,  $J = 5.7$ ), 4.08 (2H, t,  $J = 4.5$ ), 7.66 (2H, m, Pht) and 7.79 (2H, m, Pht);  $\delta_{\text{C}}$  (75.47 MHz,  $\text{CDCl}_3$ , 298 K) 20.90, 37.20, 63.54, 67.88, 69.05, 70.04, 70.44, 123.20, 132.00, 133.90, 168.20 and 171.00.

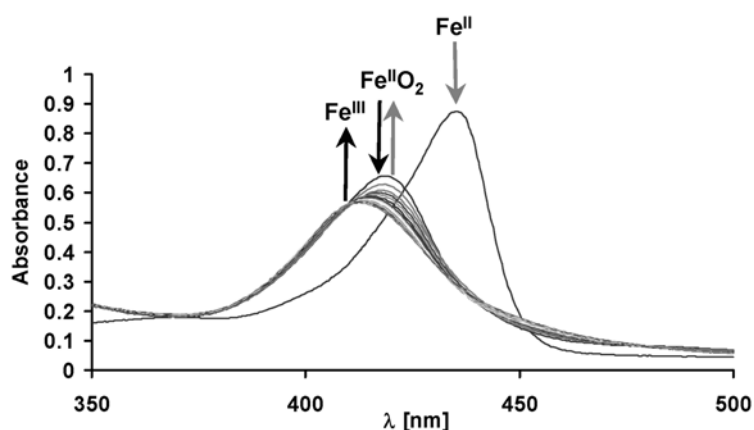
**Acetic acid 2-[2-(2-amino-ethoxy)-ethoxy]-ethyl ester 12.**  $\text{C}_8\text{H}_{17}\text{NO}_4$ . In a 100 mL round bottom flask compound **11** (3.1 mmol, 1 g) was charged with absolute ethanol (50 mL). The reaction mixture was

stirred at reflux then hydrazine monohydrate (3.7 mmol, 181  $\mu$ L) was added. The reaction mixture was stirred overnight then the reaction mixture was filtrated and evaporated under vacuum. The resulting oil dissolved in chloroform was directly loaded on a silica gel column chromatography. The reaction mixture was eluted firstly with 4% MeOH/ CHCl<sub>3</sub> and the expected compound eluted with CHCl<sub>3</sub>/ NH<sub>3</sub>g was obtained as a colourless oil in 91% yield (540 mg).  $\delta_{\text{H}}$  (500.13 MHz, CDCl<sub>3</sub>, 298 K) 1.43 (2H, broad s, -NH<sub>2</sub>), 2.01 (3H, s, COCH<sub>3</sub>), 2.80 (2H, t,  $J = 4.7$ , -CH<sub>2</sub>-N), 3.44 (2H, t,  $J = 4.7$ ), 3.57 (2H, m), 3.59 (2H, m), 3.64 (2H, t,  $J = 4.7$ , -CH<sub>2</sub>-CO) and 4.16 (2H, t,  $J = 4.4$ );  $\delta_{\text{C}}$  (125.76 MHz, CDCl<sub>3</sub>, 298 K) 20.8, 41.5, 63.5, 69.1, 70.2, 70.5, 72.9 and 170.8;  $m/z$  (ESI HRMS) 214.1051 ([M + Na]<sup>+</sup> C<sub>8</sub>H<sub>17</sub>NO<sub>4</sub>Na requires 214.1055).

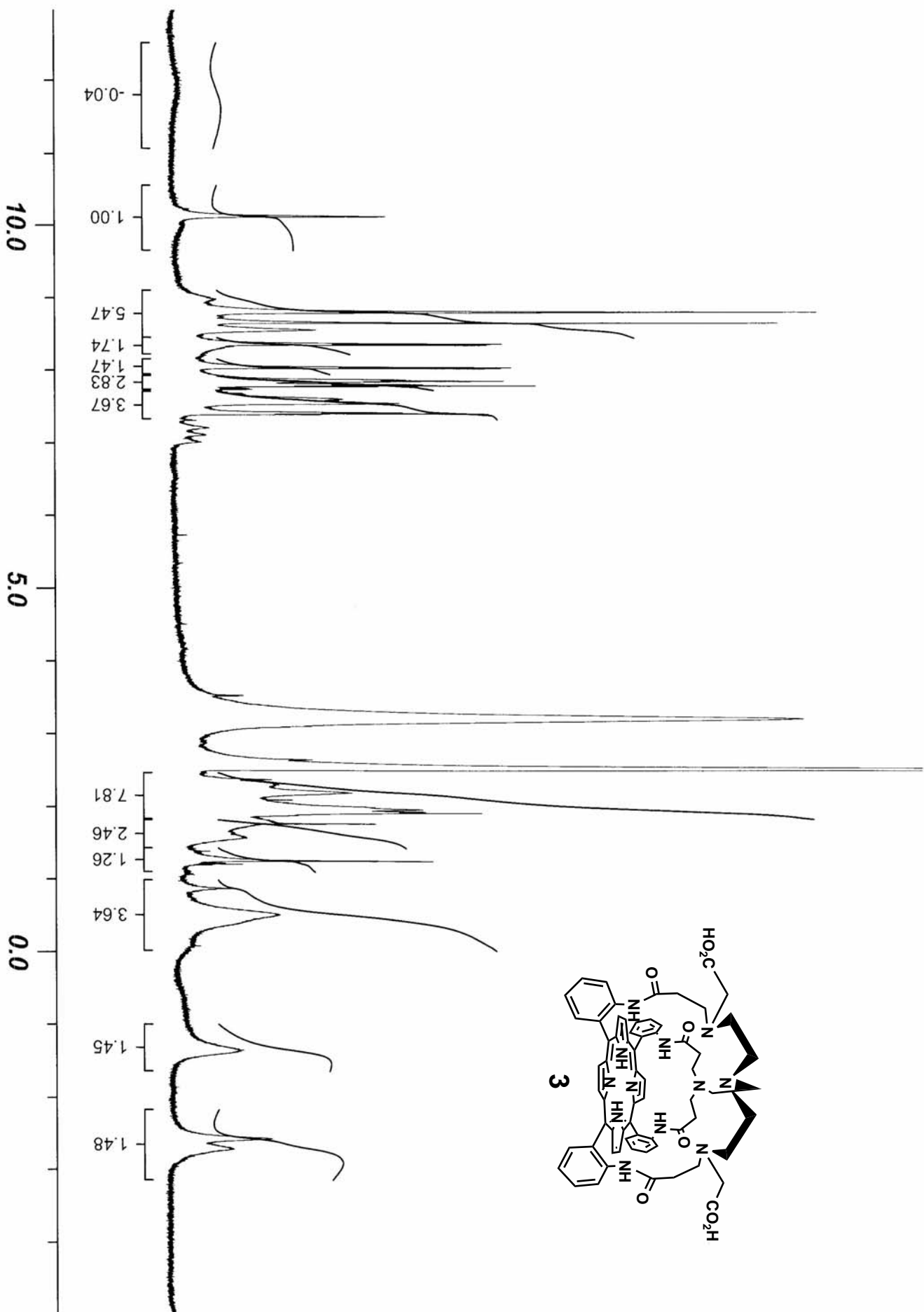
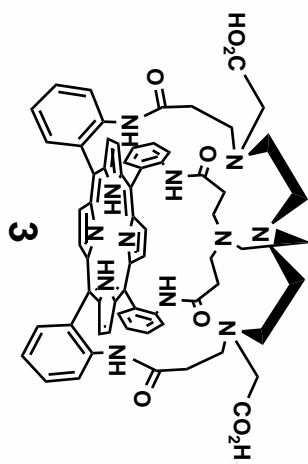
### Supplementary UV-visible data upon dioxygen binding for 4Fe.

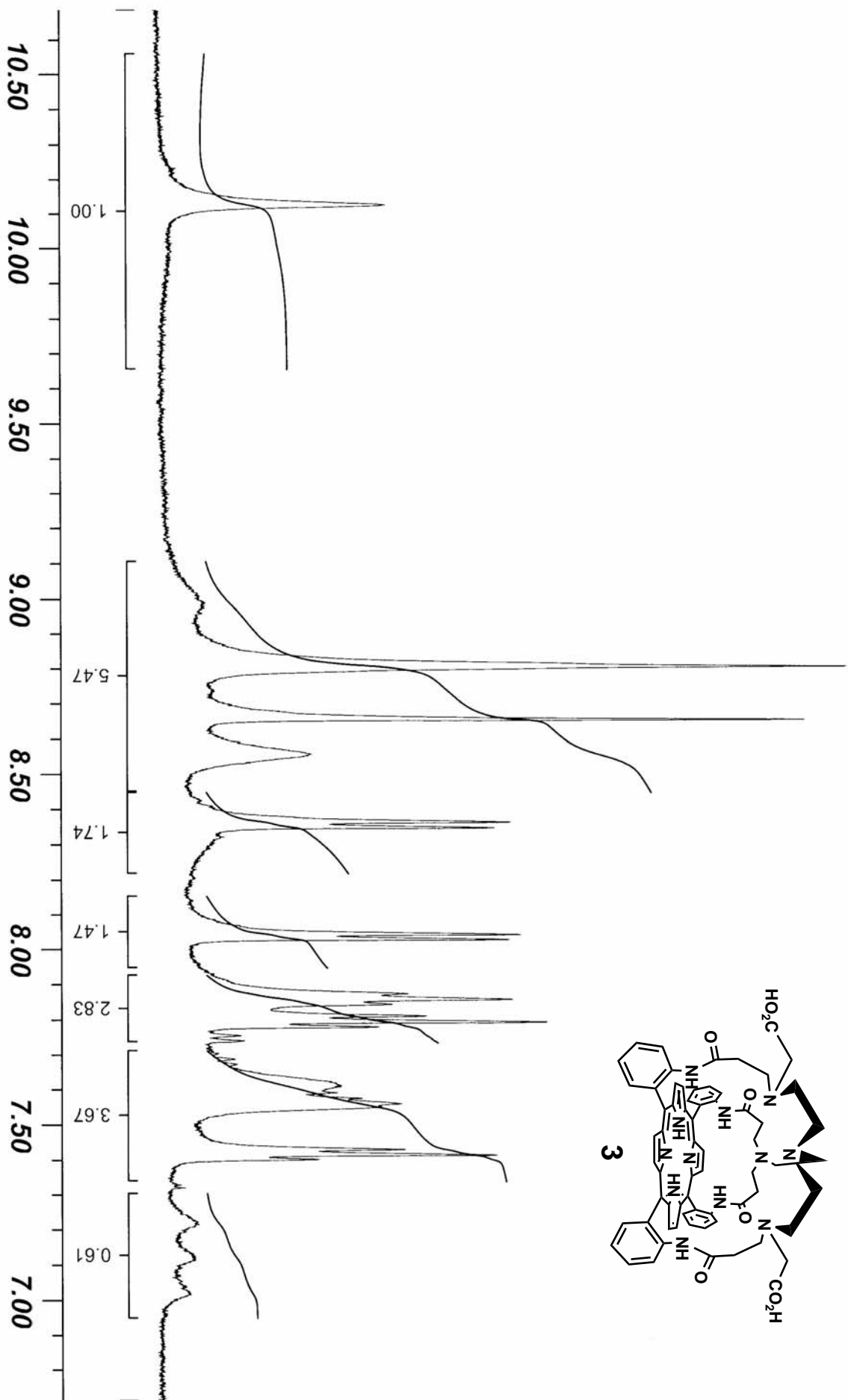
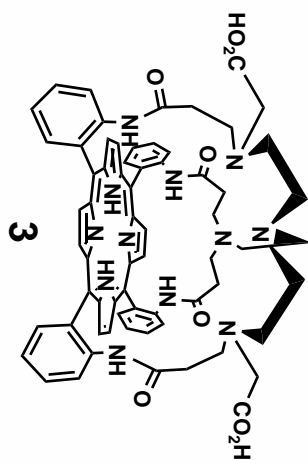


Decreasing of the absorbance at 436 nm upon dioxygen binding on **4Fe** + pyridine (phosphate buffer, pH = 7.4, 25 °C)

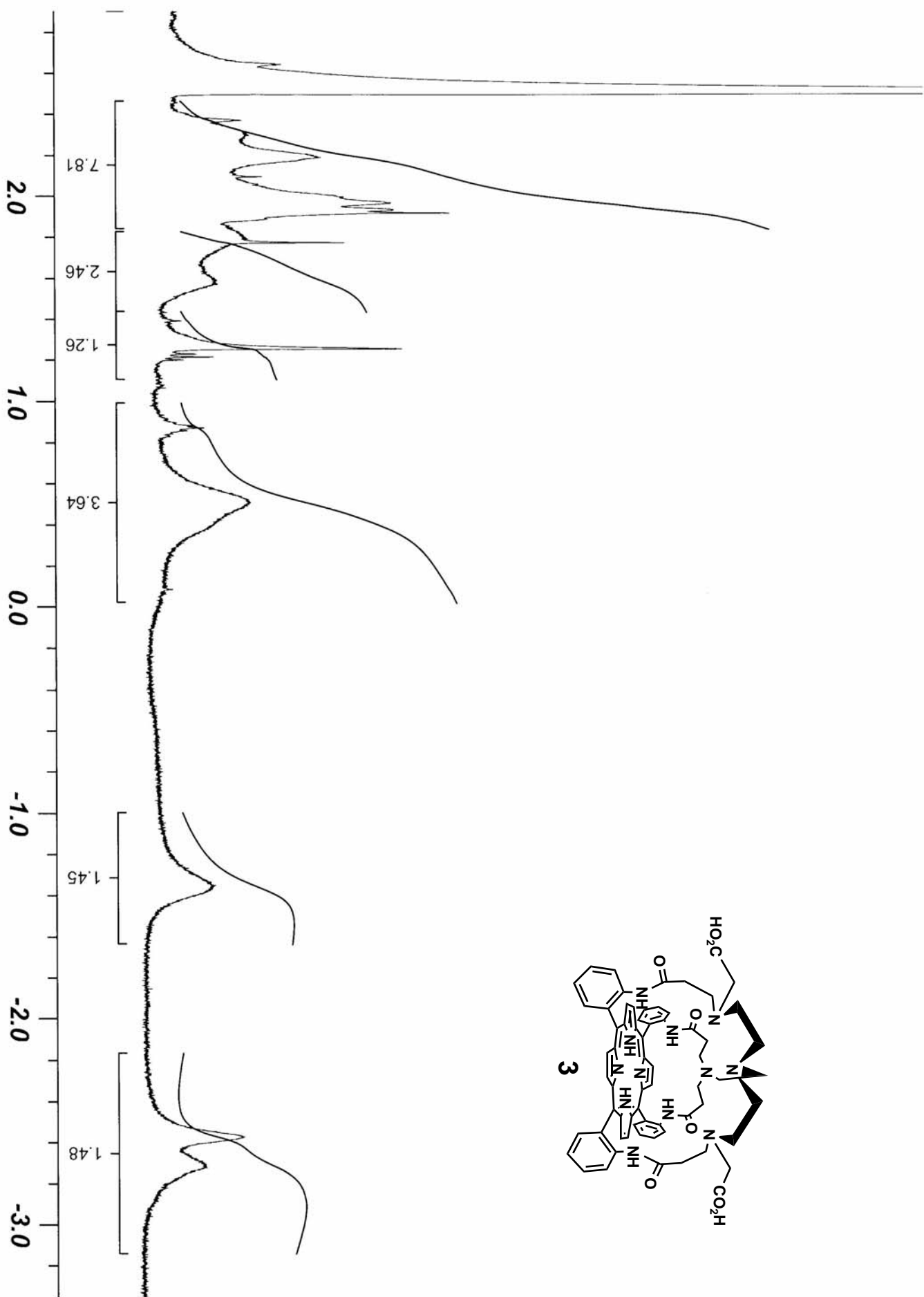
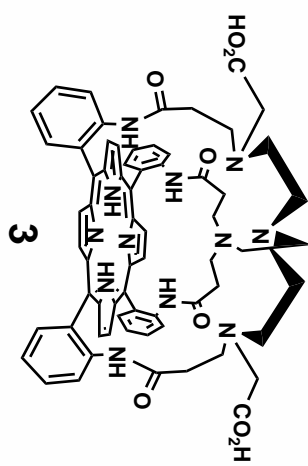


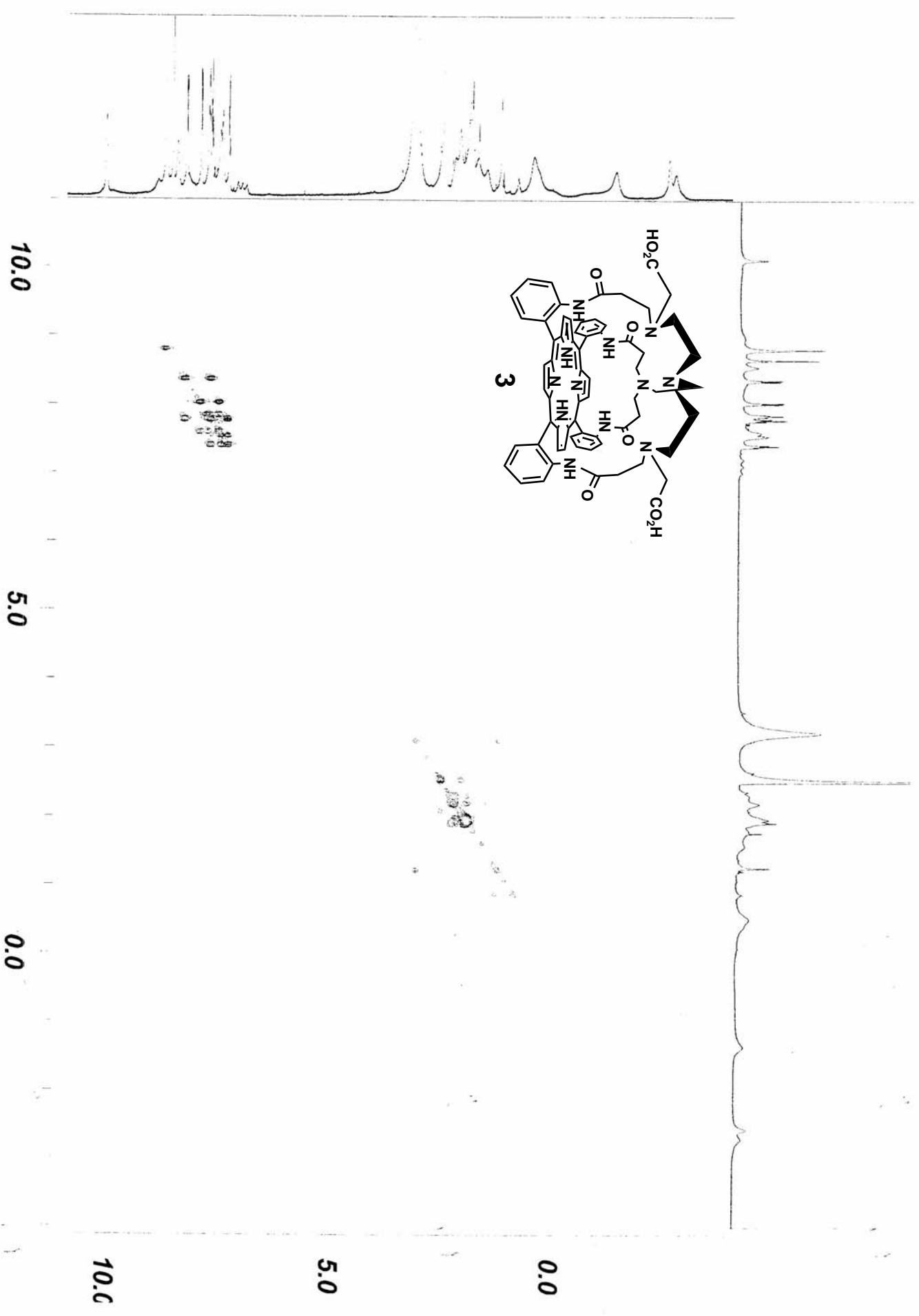
UV-vis. monitoring of dioxygen binding on **4Fe** + pyridine (phosphate buffer, pH = 7.4, 25 °C)











8.50

8.00

7.50

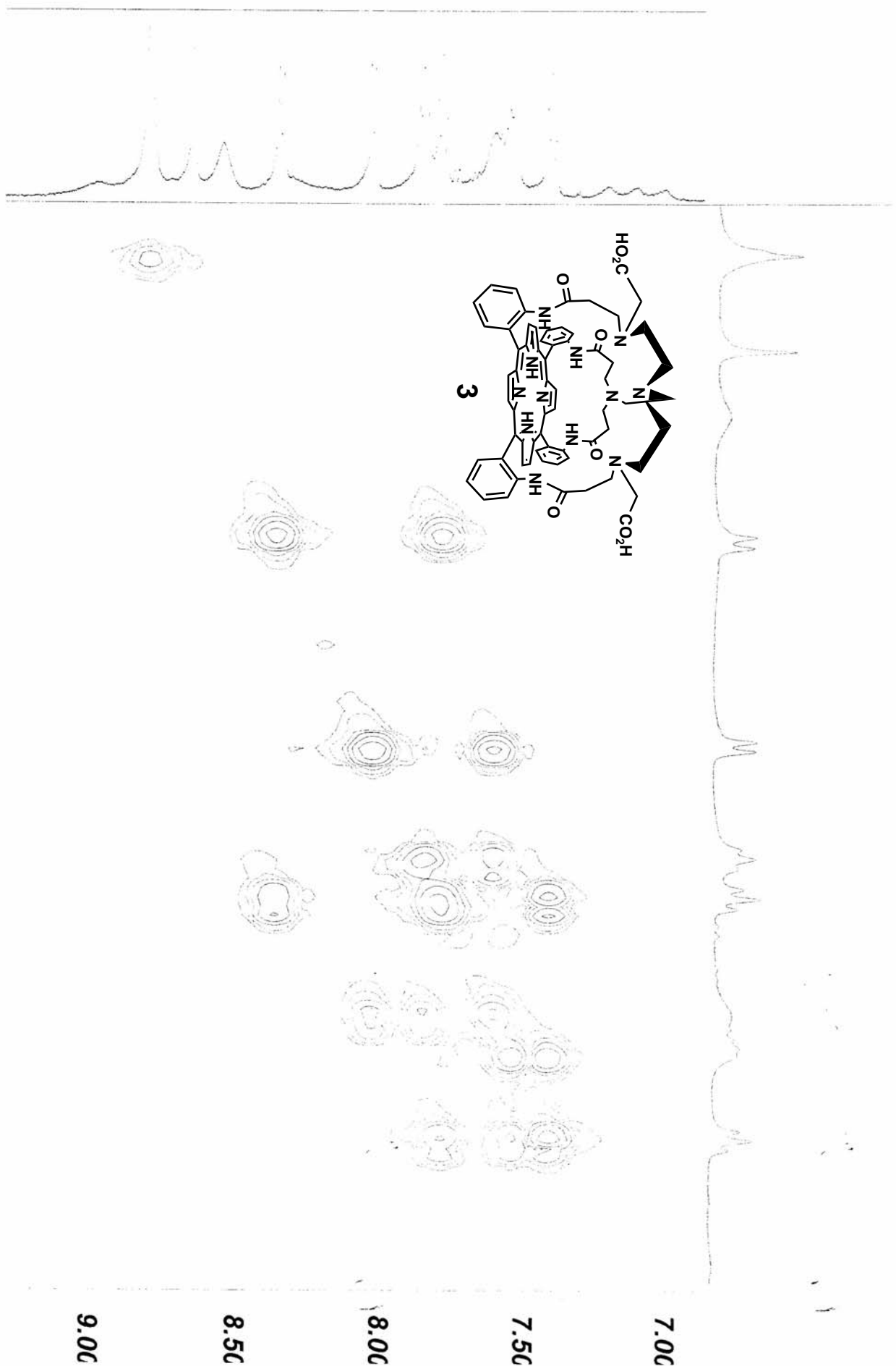
9.00

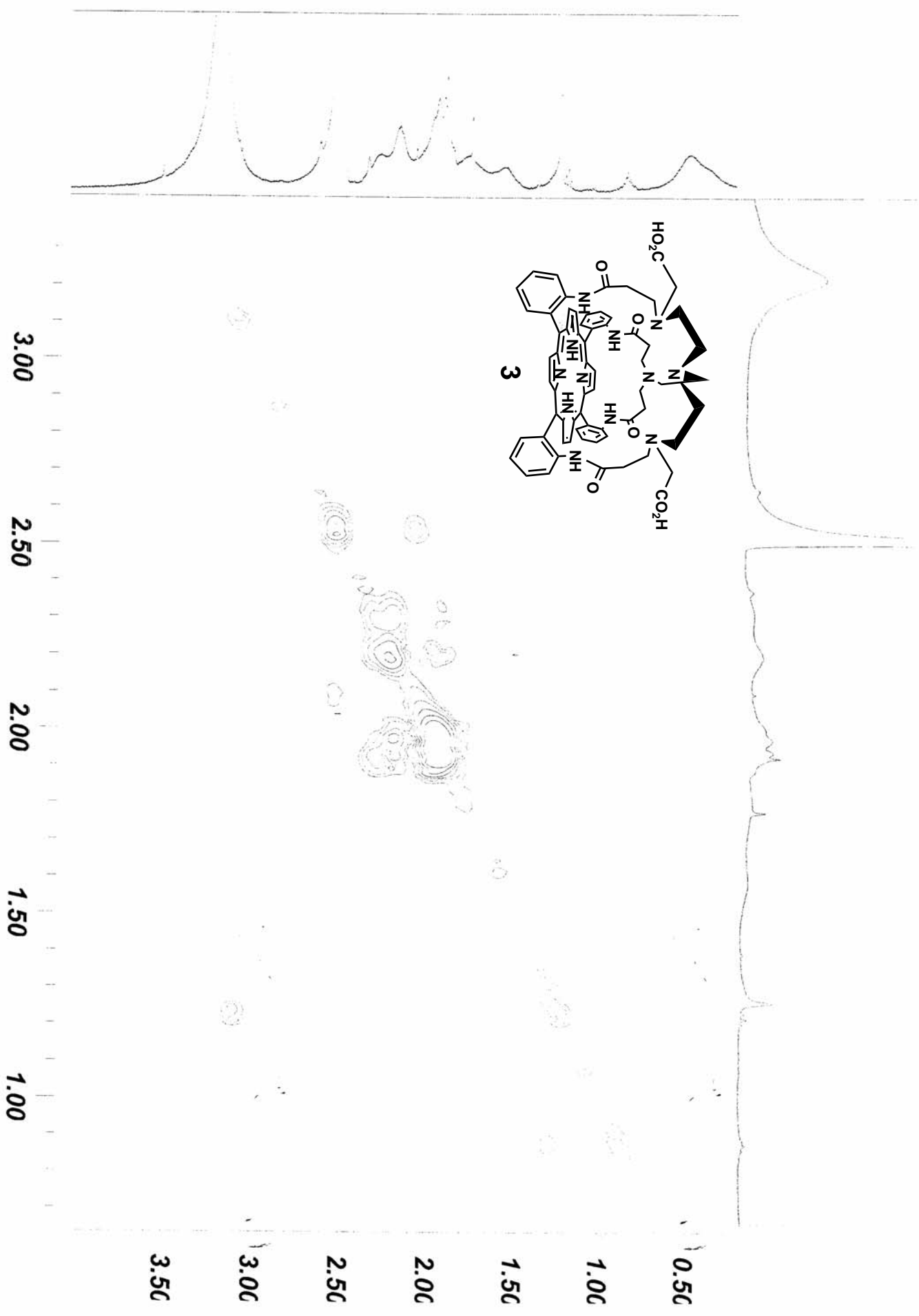
8.50

8.00

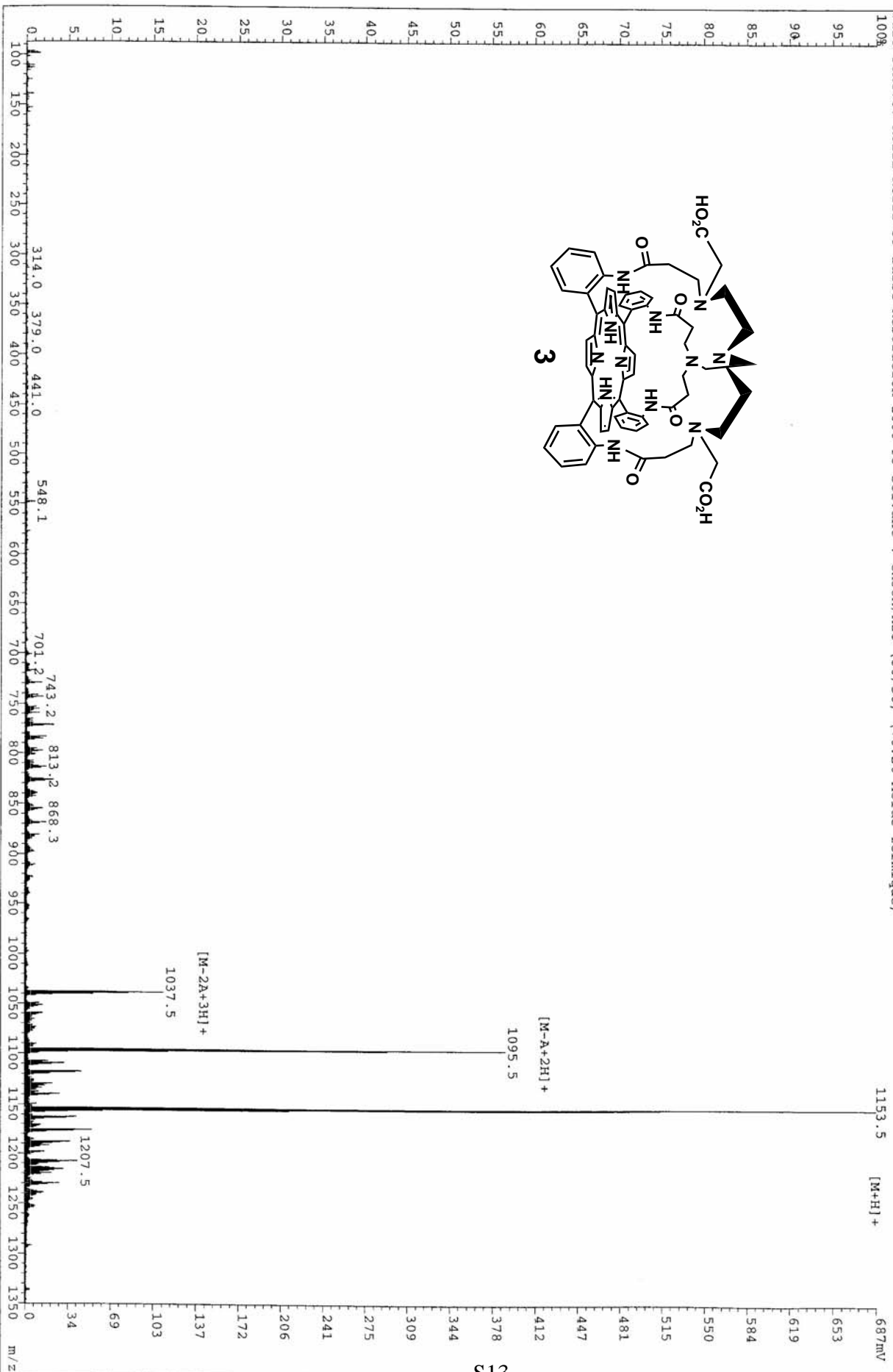
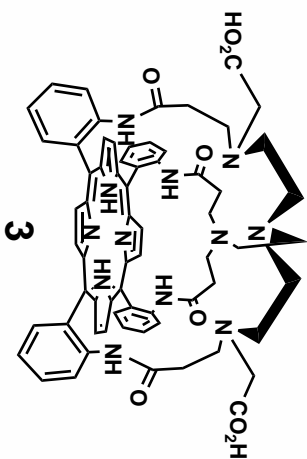
7.50

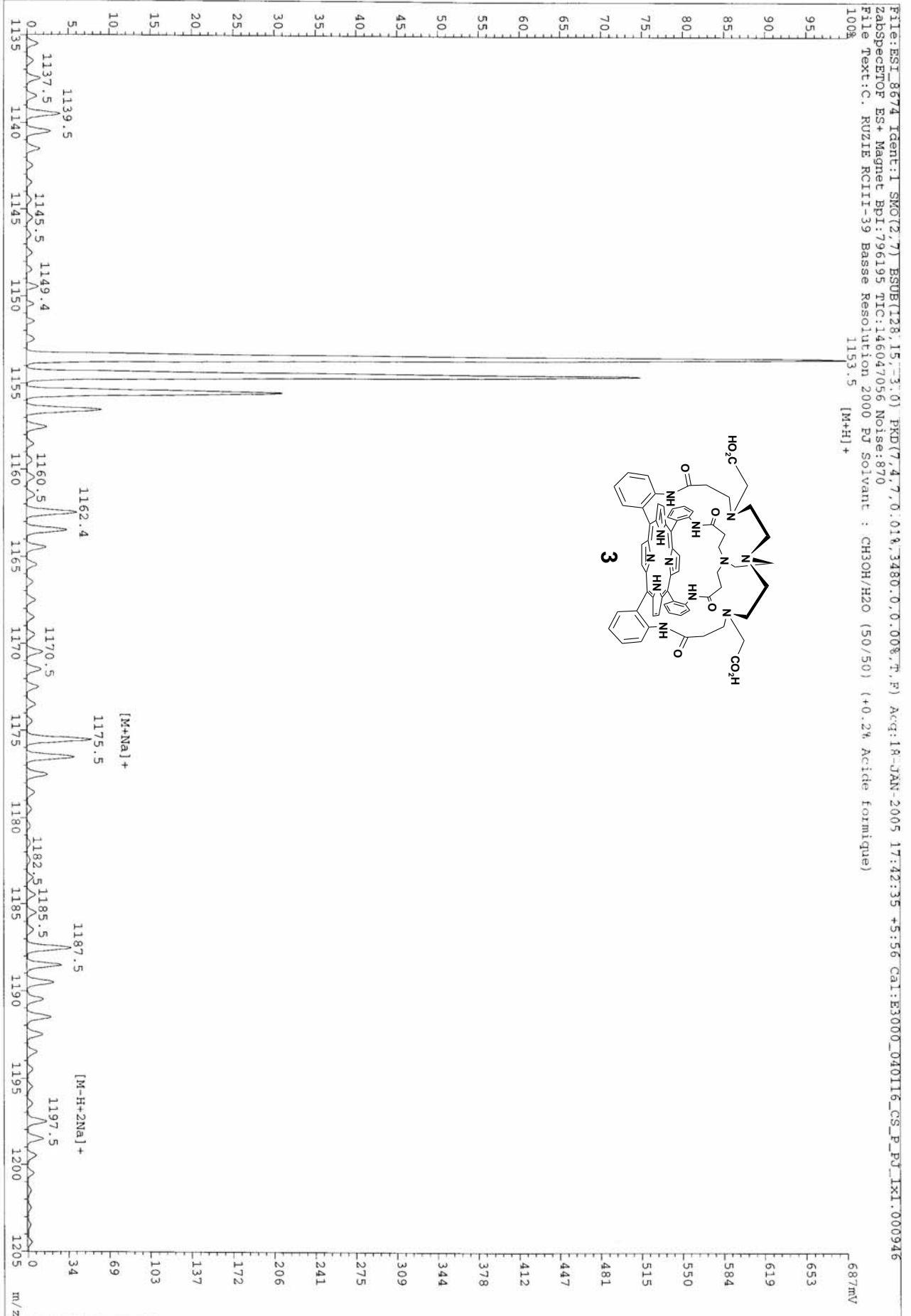
7.00



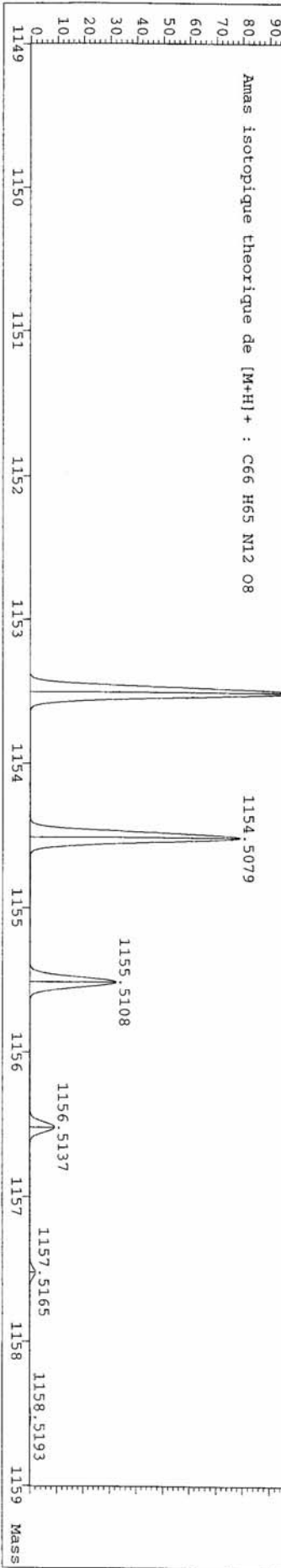
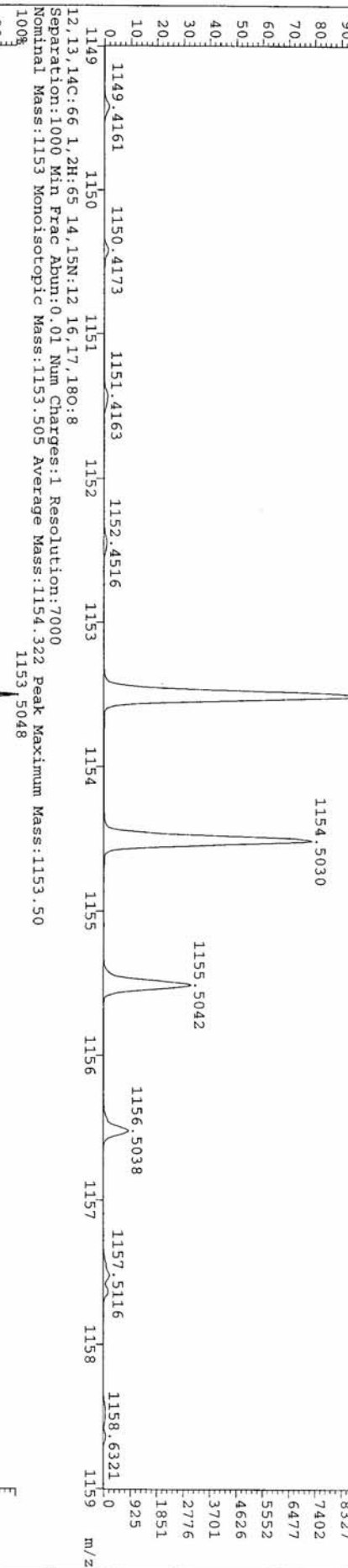
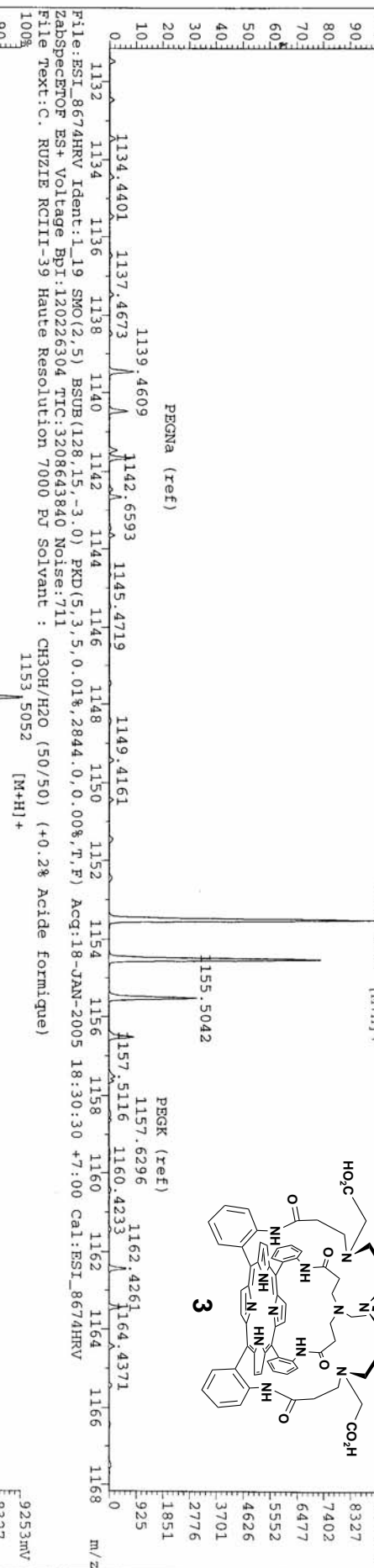
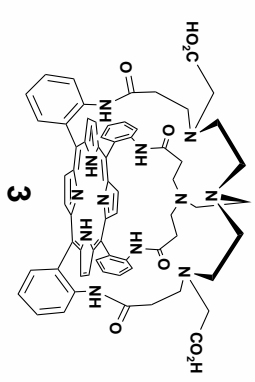


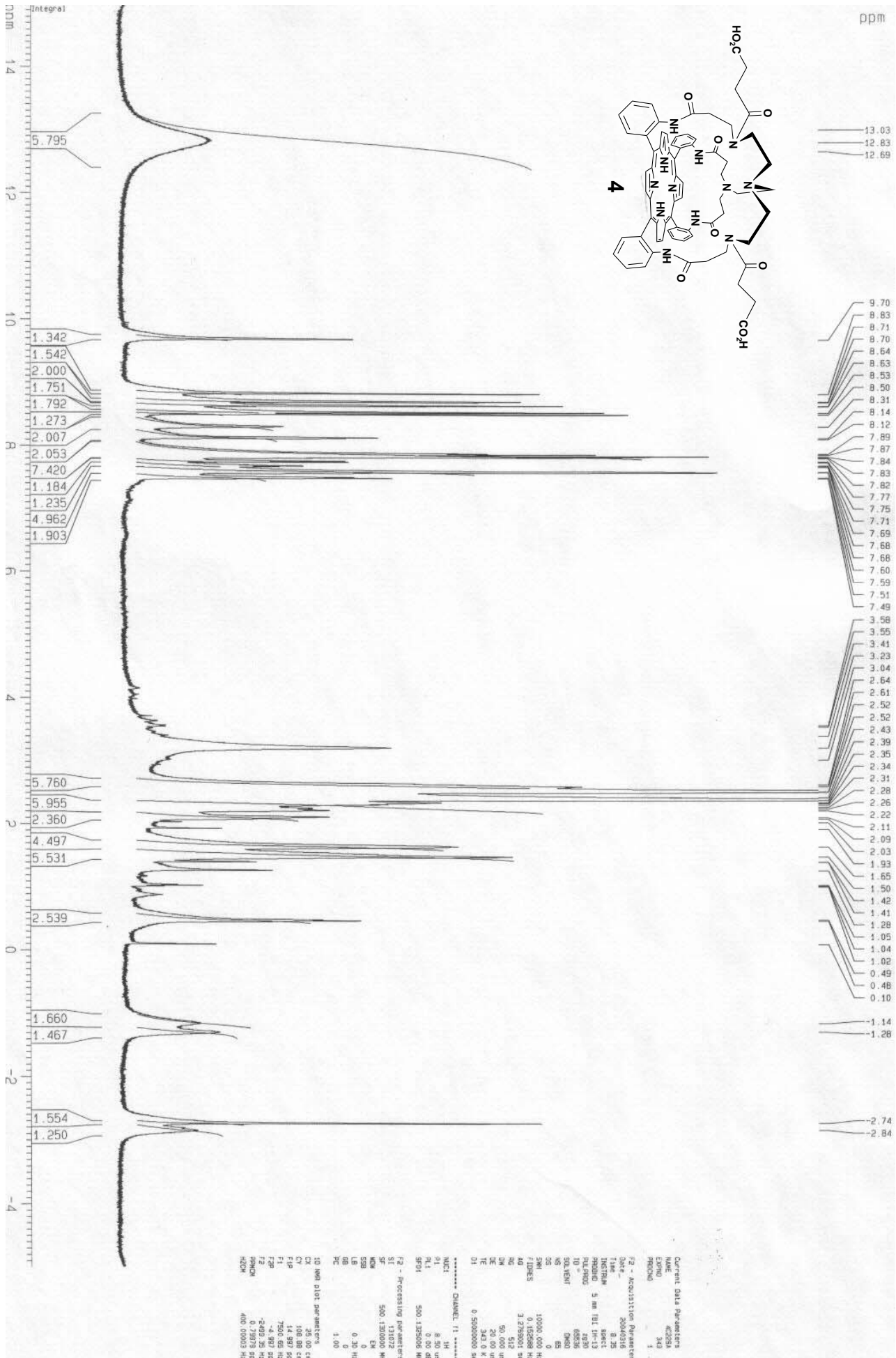
File:ESI\_8674 Ident:1 SMO(2,7) BS08(128\_15,-3\_0) EKD(7,4\_7,0\_01%,3480.0\_0.00%,T,F) Acq:18-JAN-2005 17:42:35 +5:56 Cal:E3000\_040116\_CS\_P\_PJ\_IxI\_000946  
 ZabspecTOP ES+ Magnet Bpt:796195 TIC:146047056 Noise:870  
 File text:C: RUIZIE RCUII-39 Basse Resolution 2000 Pj Solvant : CH3OH/H2O (50/50) (+0.2% Acide formique)



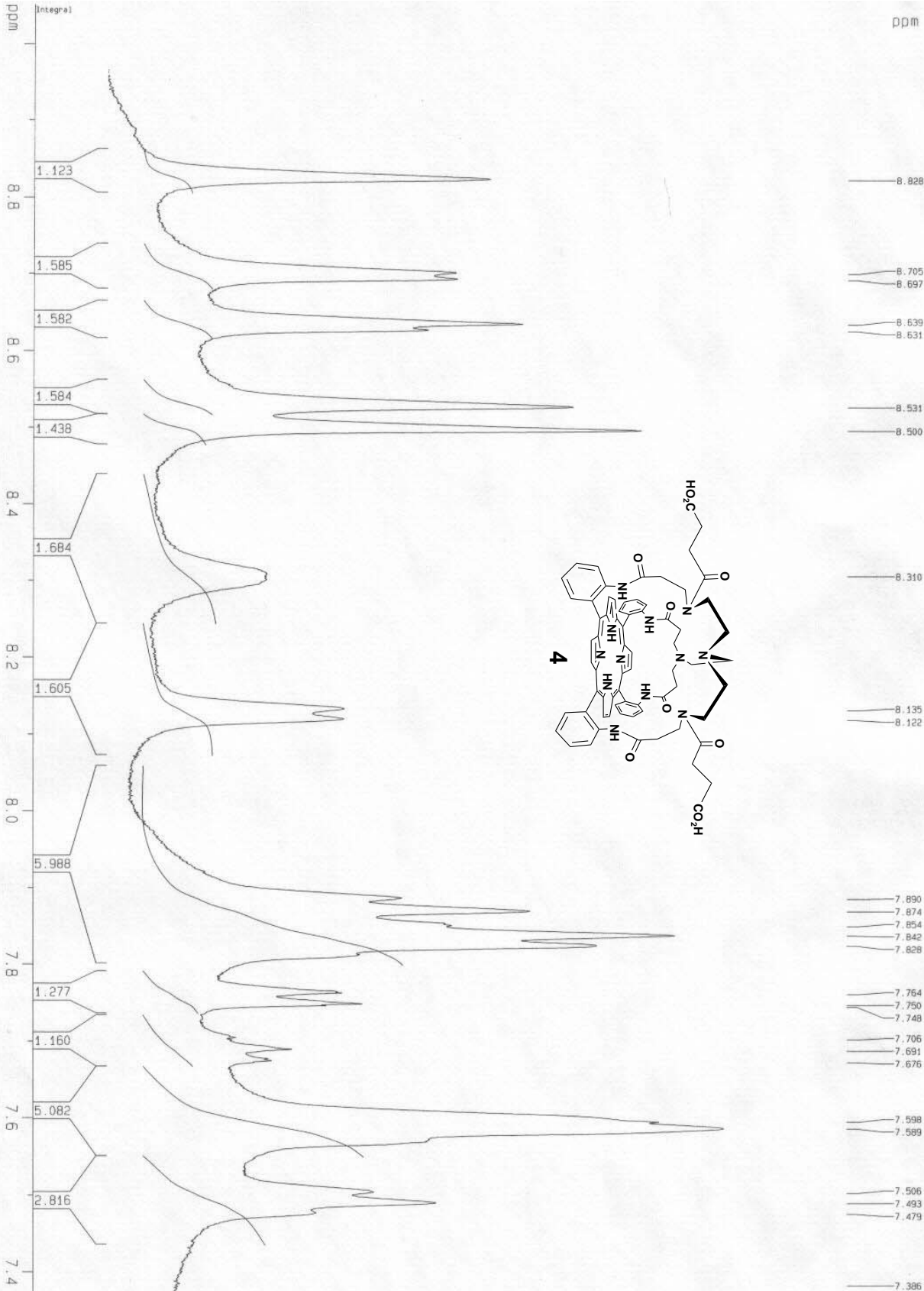


File:ESI\_8674HRV Ident:1\_19\_SMO(2,5) BSUB(128,15,-3,0) PKD(5,3,5,0,0.01%,2844,0,0.00%,T,F) Acq:18-JAN-2005 18:30:30 +7:00 Cal:ESI\_8674HRV  
 ZabsSpecTOP ES+ Voltage BpI:120226304 TIC:3208643840 Noise:711  
 File Text:C. RUZIE RCIIT-39 Haute Resolution 7000 PJ Solvant : CH3OH/H2O (50/50) (+0.2% Acide formique)









ppm

Integral

8.8

8.6

8.4

8.2

8.0

7.8

7.6

7.4

1.123

1.585

1.582

1.584

1.438

1.684

1.605

5.988

1.277

1.160

5.082

2.816

8.828

8.705

8.697

8.639

8.631

8.531

8.500

8.310

8.135

8.122

7.890

7.874

7.854

7.842

7.826

7.764

7.750

7.748

7.706

7.691

7.676

7.598

7.589

7.506

7.493

7.479

7.386

Current Data Parameters

NAME: 402324

EXPNO: 343

PROCNO: 1

F2 - Acquisition Parameters

Date\_ Time: 20060318

Time: 11.58

INSTRUM: spect

PROBHD: 5 mm 1H1-13

PULPROG: zgpg30

TD: 65536

TE: 300.2

NUC1: 13C

NUC2: 15N

SE: 0

SH: 10000.000 Hz

SI: 0

SDRES: 3.200000 Hz

LA: 0.000000 Hz

RG: 512

CH: 50.000 uM

DE: 24.00 uM

TE: 0.5000000 sec

SI: 0.5000000 sec

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*

NUC1: 13C

NUC2: 15N

P1: 9.00 uM

P2: 0.00 uM

SFO1: 500.125000 MHz

SFO2: 500.125000 MHz

F2 - Processing parameters

SI: 65536

WDW: EM

SSB: 0 Hz

GB: 0

PC: 1.00

1D NMR 010: parameters

SI: 65536

WDW: EM

SSB: 0 Hz

GB: 0

PC: 1.00

CT: 100.00 Hz

CV: 100.00 Hz

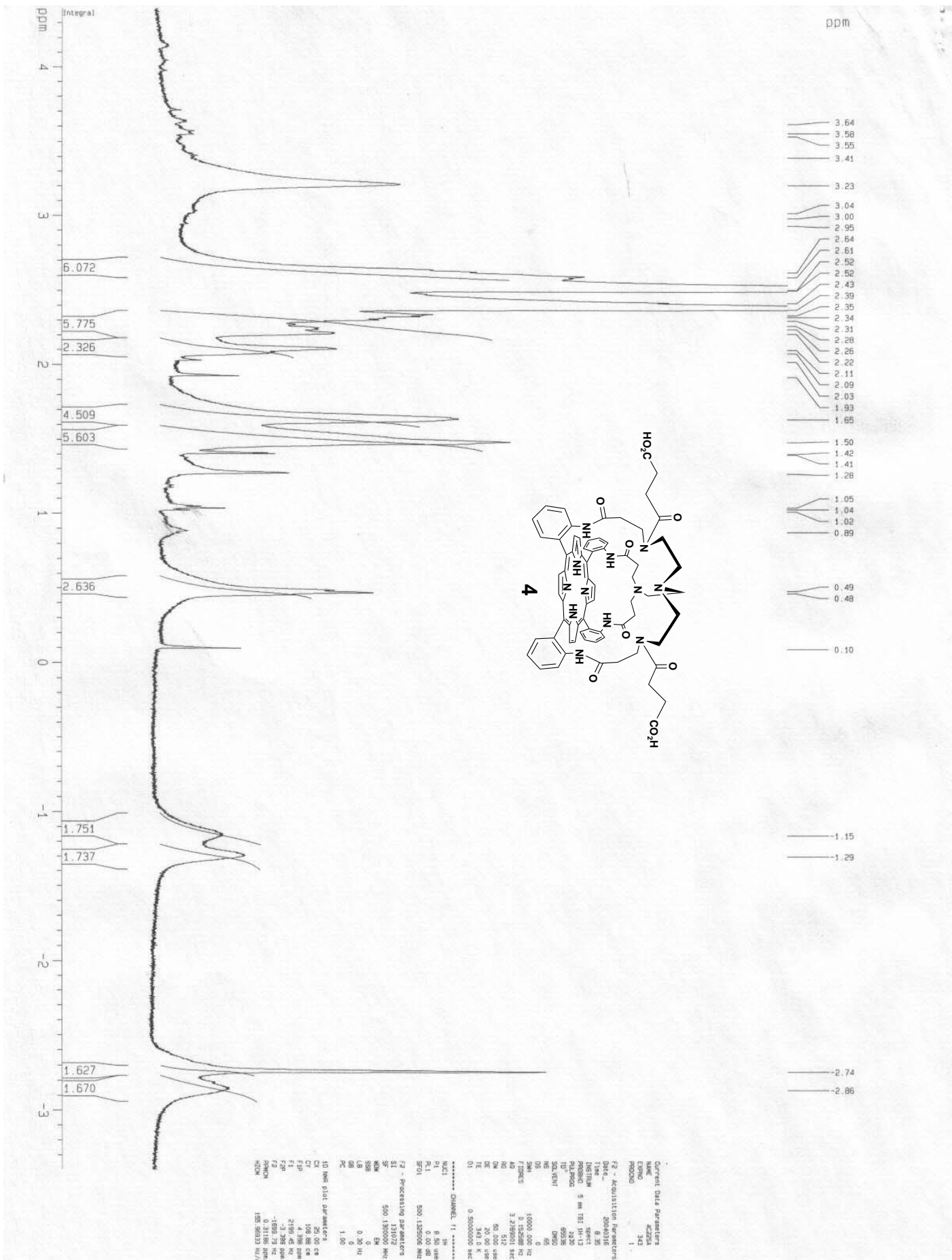
F1P: 9.161000 MHz

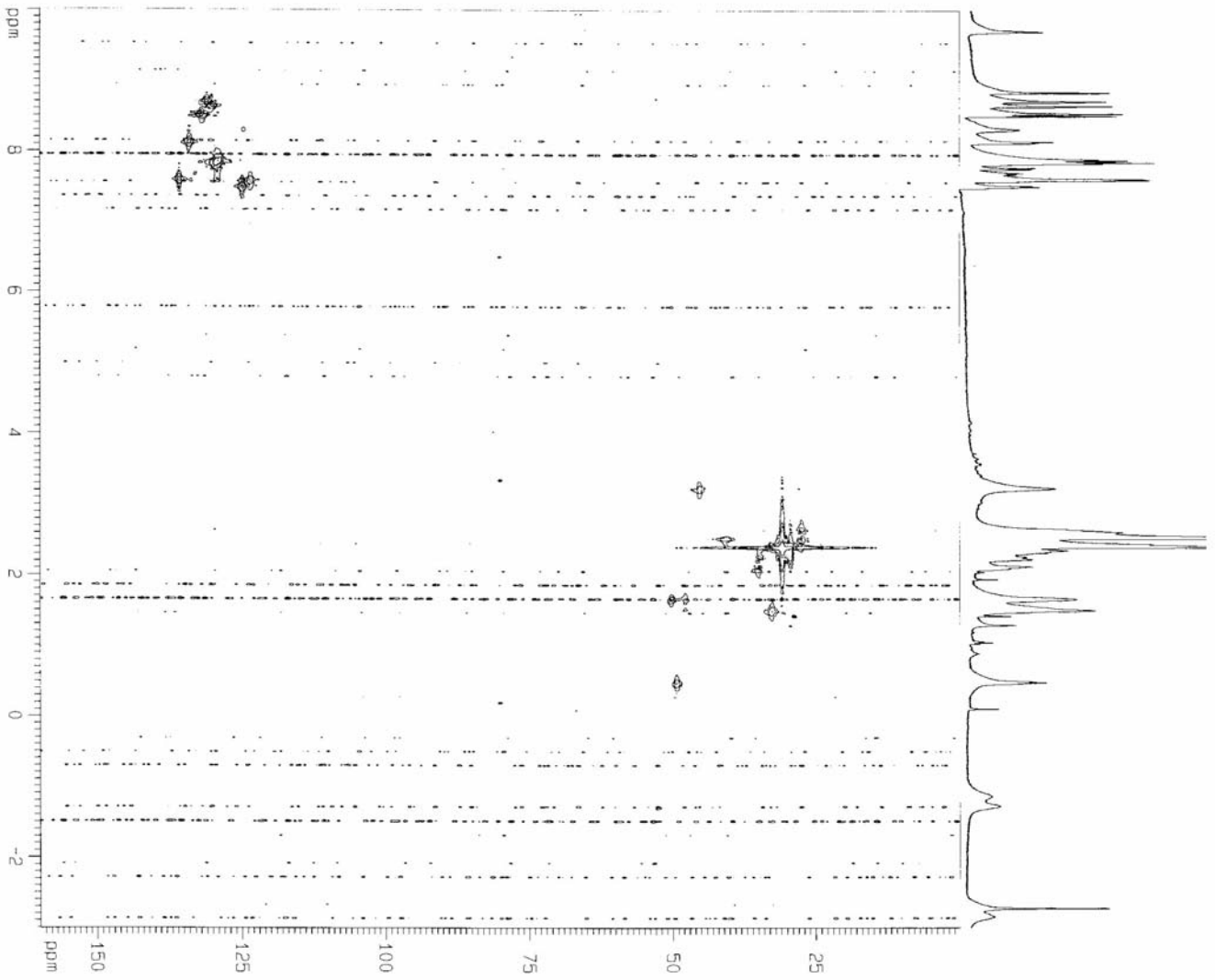
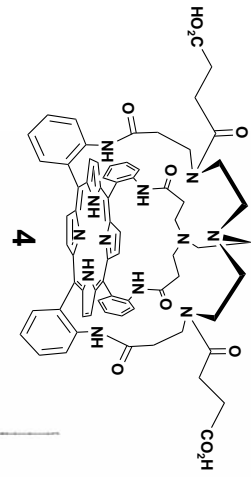
F2P: 46264.92 Hz

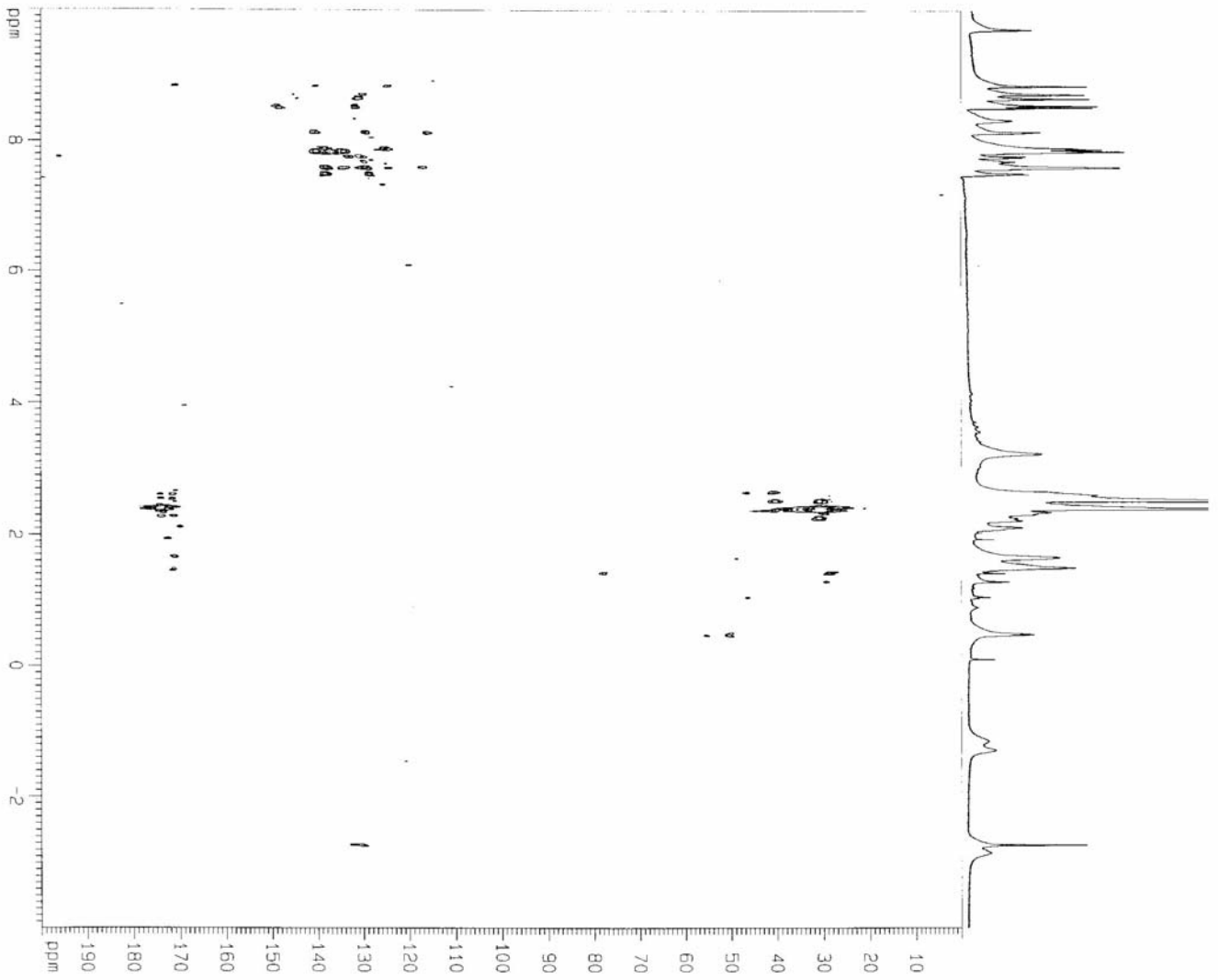
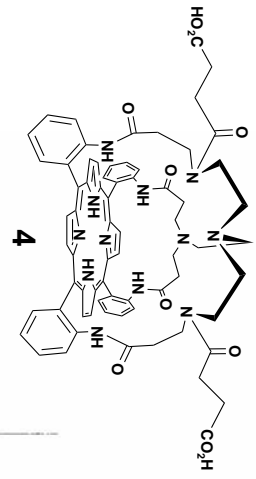
F3P: 30.701000 MHz

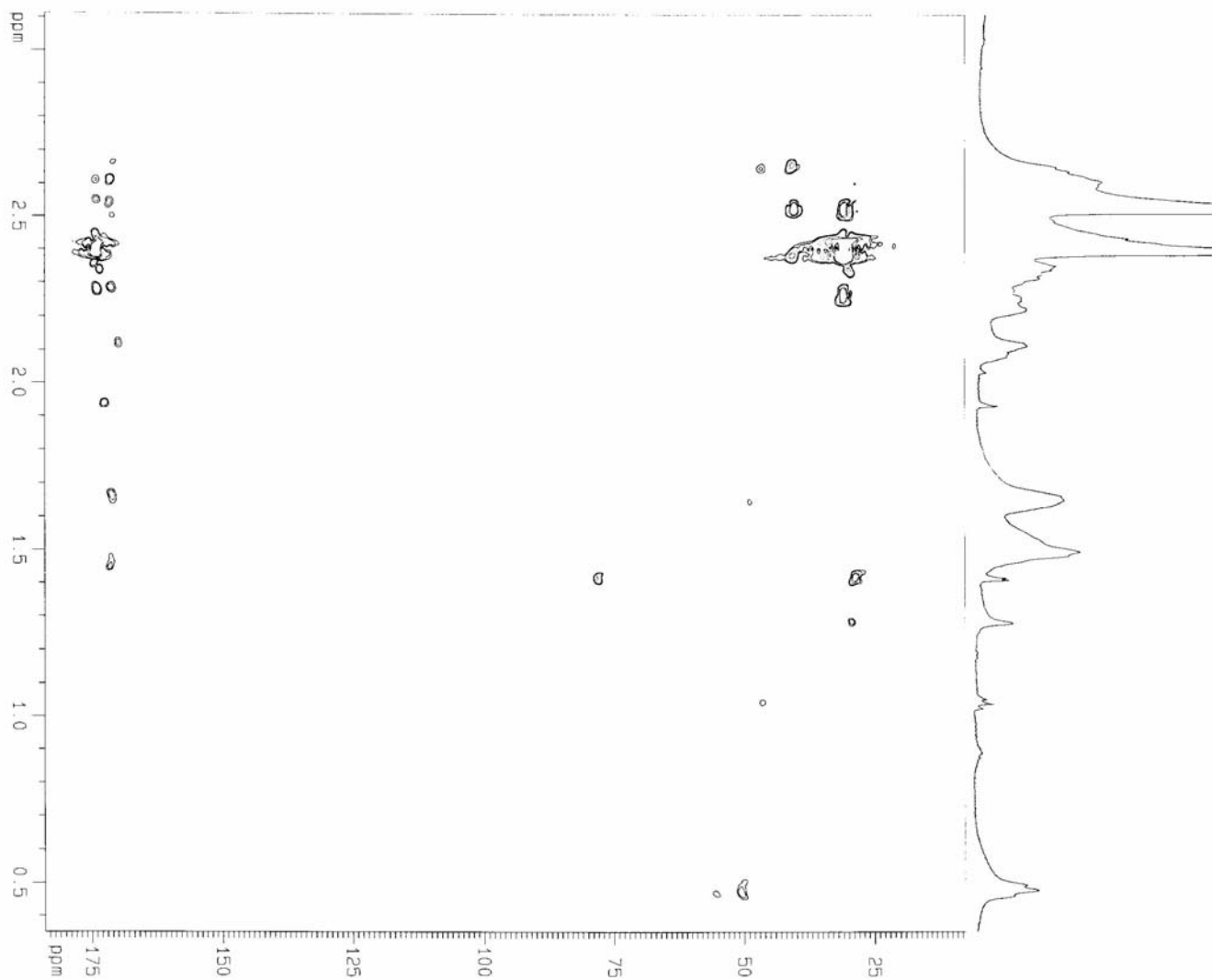
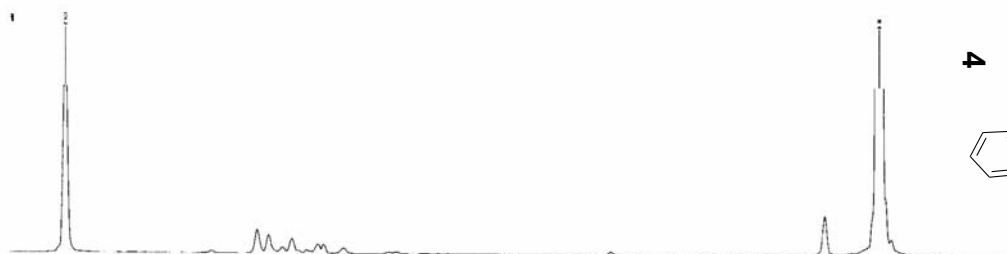
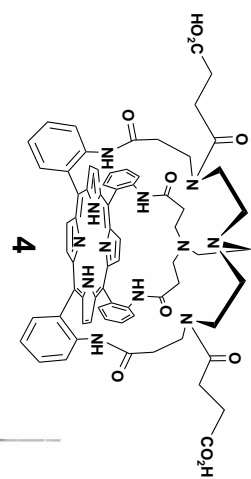
PRNDM: 0.00000000

PCNOM: 34.02942 Hz/2

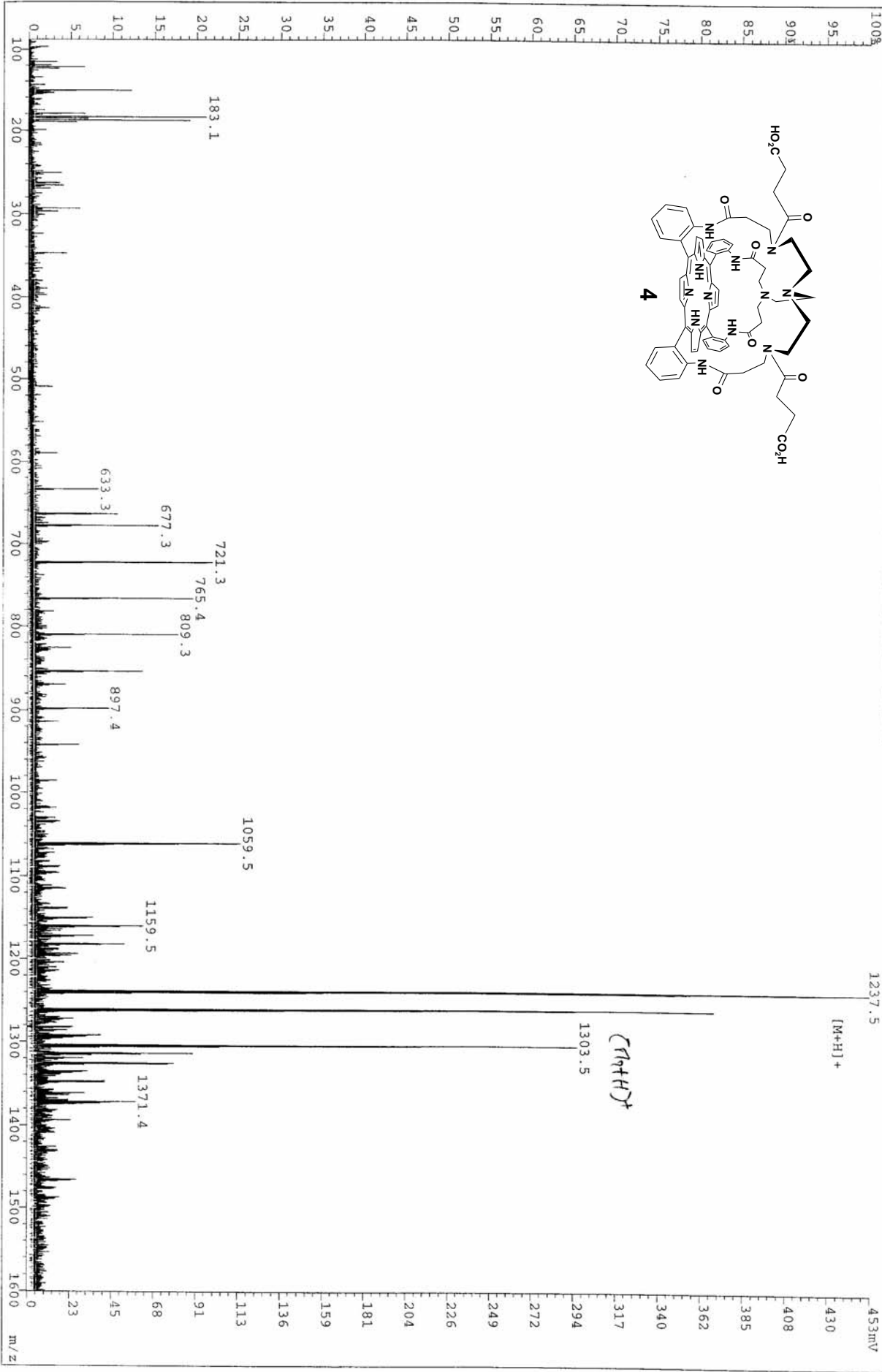
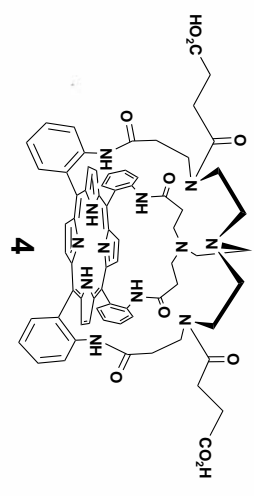




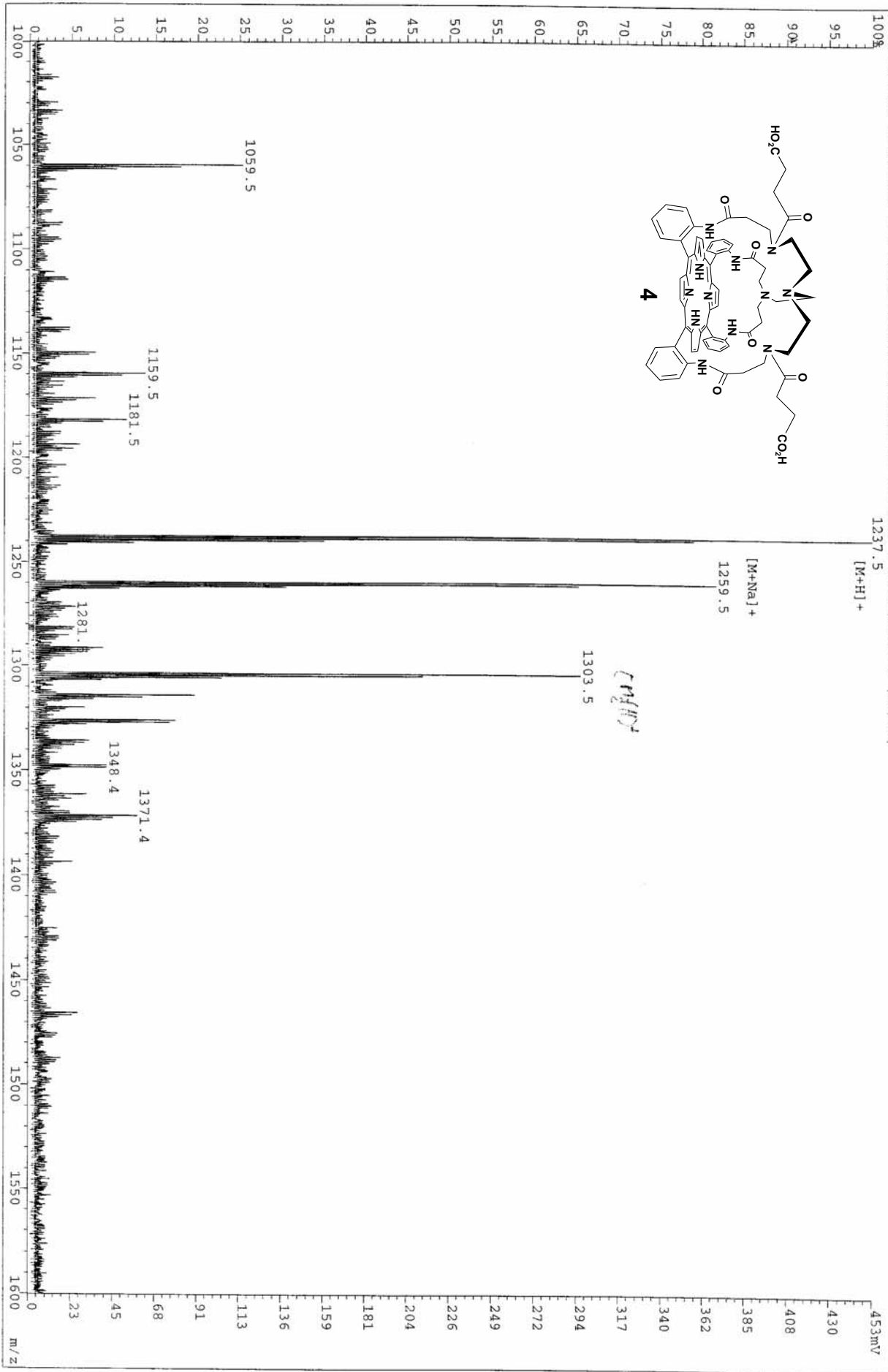
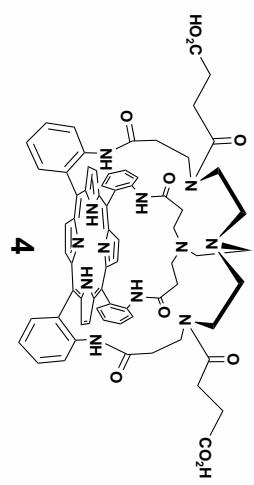




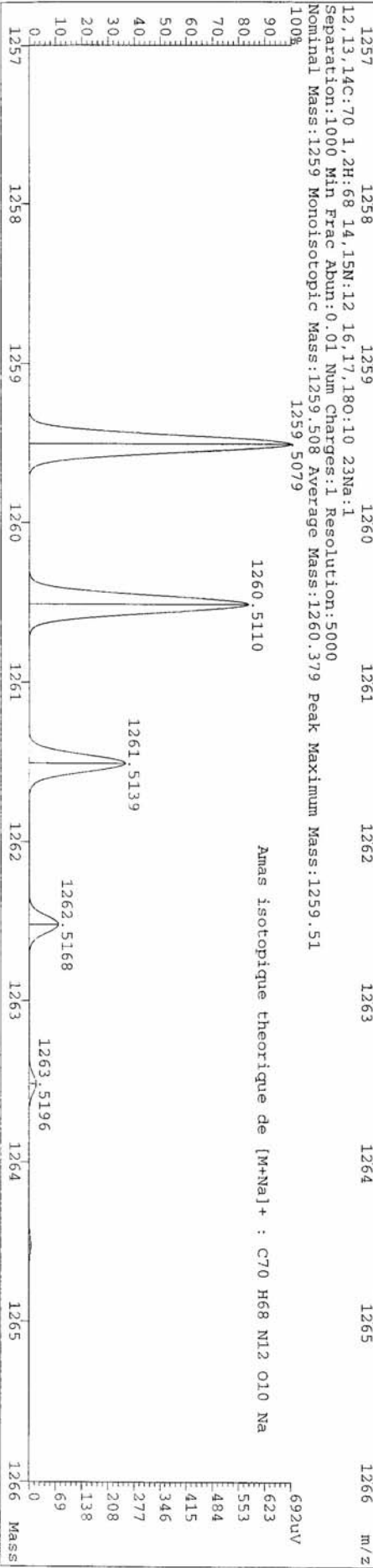
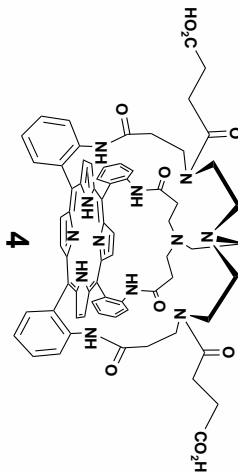
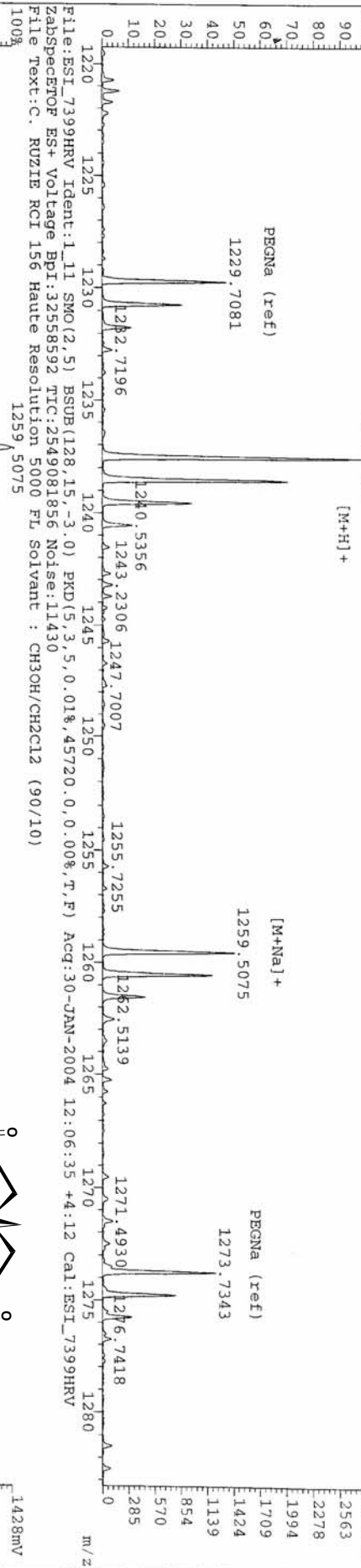
File: EST\_7399 Ident: 1 SMO(2.75) BSUB(128.15,-3.0) PKD(5.3,5,0.018,190880.0,0.008,T,F) Acq: 30-JAN-2004 11:48:02 +10:57 Cal: E3000\_040116\_CS\_P\_FJ Txt: 00035\*  
 ZabspecETOP ES+ Magnet Bp1: 538638 TTC: 455710080 Noise: 47720  
 File Text: C. RUZIE RCI 156 Basse Resolution 2000 FL Solvant : CH3OH/CH2Cl2 (90/10)



File: EST\_7399 Ident: 1 SMO(2,5) BSUB(128,15,-3,0) PKD(5,3,5,0,0.018,190880,0,0,0.008,T,F) Acq: 30-JAN-2004 11:48:02 +10:57 Cal: E3000\_040116\_CS\_F\_P1\_1X1\_000355  
 ZABSPECTOP ES+ Magnet EPI: 538638 TIC: 455710080 Noise: 47720  
 File Text: C. RUIZIE RCI 156 Basse Resolution 2000 FL Solvant : CH3OH/CH2Cl2 (90/10) 1237.5 [M+H]<sup>+</sup>  
 1008 [M+H]<sup>+</sup>

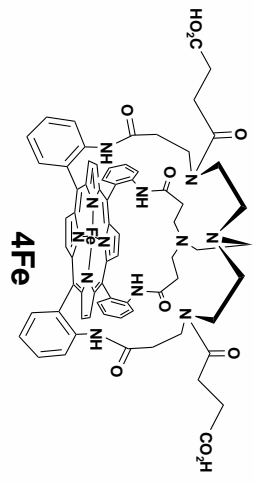
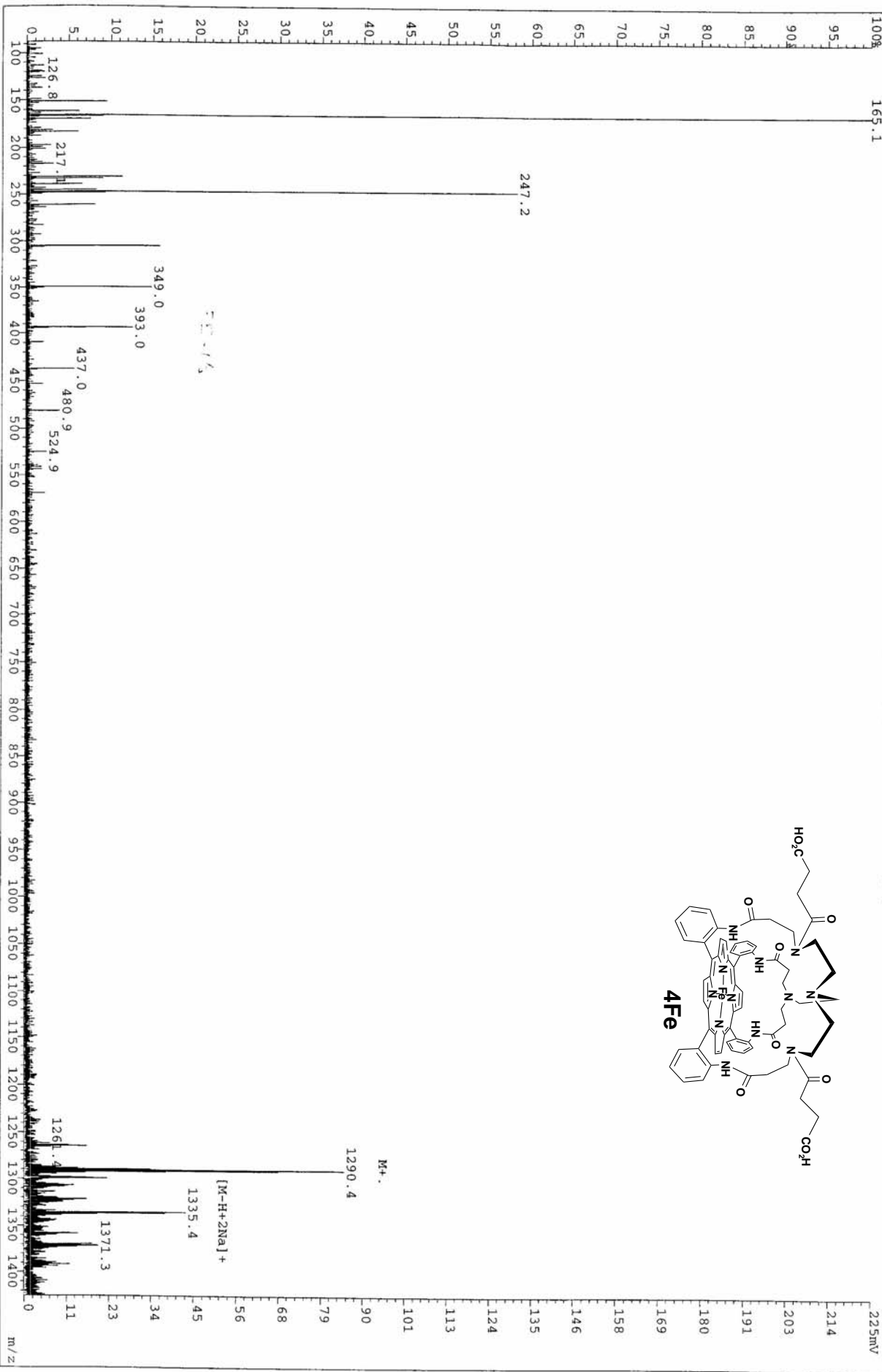


File: EST\_7399HRV Ident: 1\_11 SMO(2,5) ESUB(128,15,-3,0) PKD(5,3,5,0,0.018,45720,0,0.008,T,F) Acq: 30-JAN-2004 12:06:35 +4:12 Cal: EST\_7399HRV  
 Zabspector ES+ Voltage BpI:32558592 TIC:2549081856 Noise:11430  
 File Text: C. RUZIE RCI 156 Haute Resolution 5000 FL Solvant : CH3OH/CH2Cl2 (90/10)

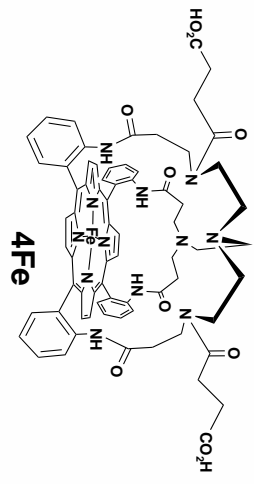
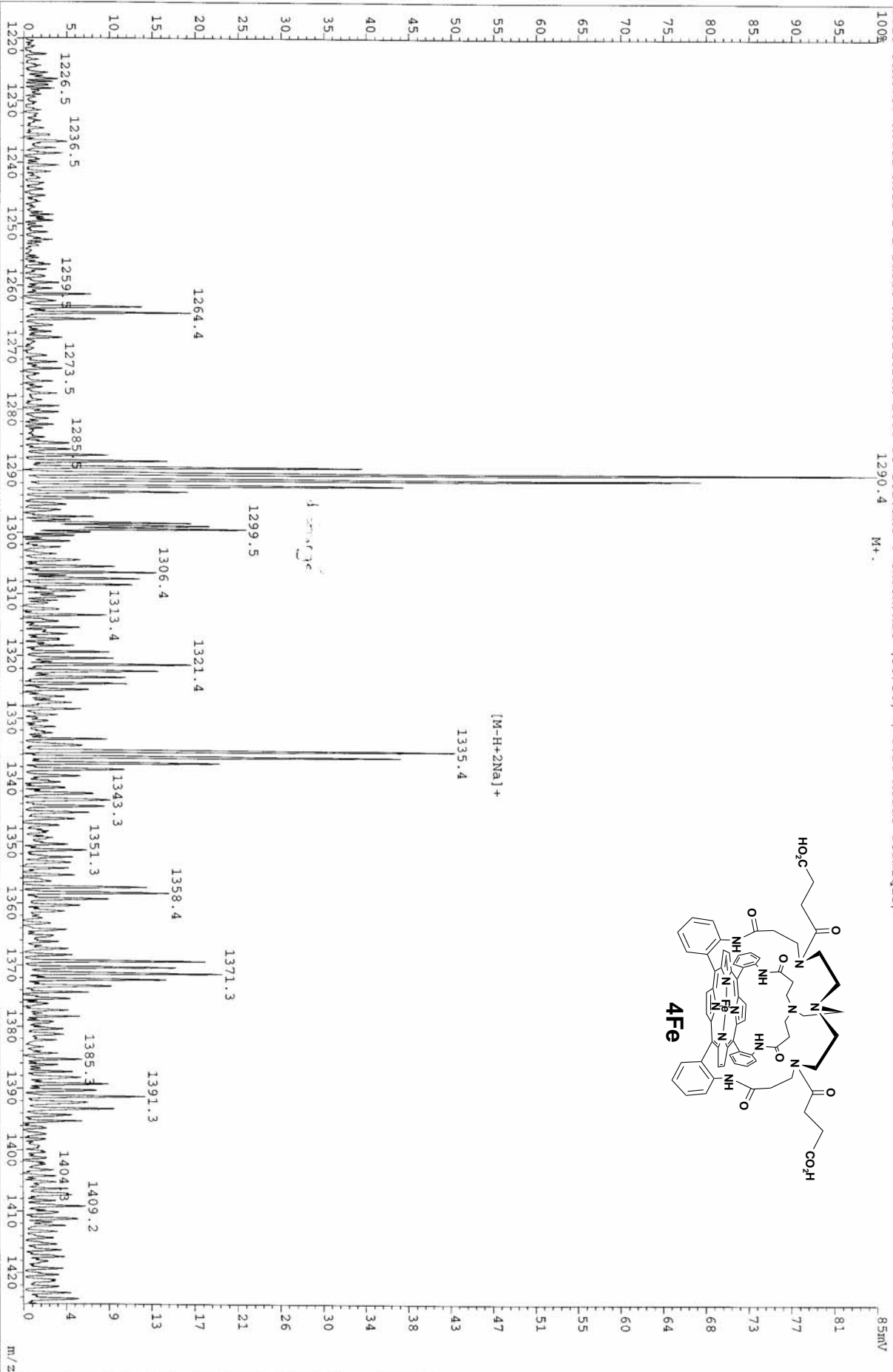




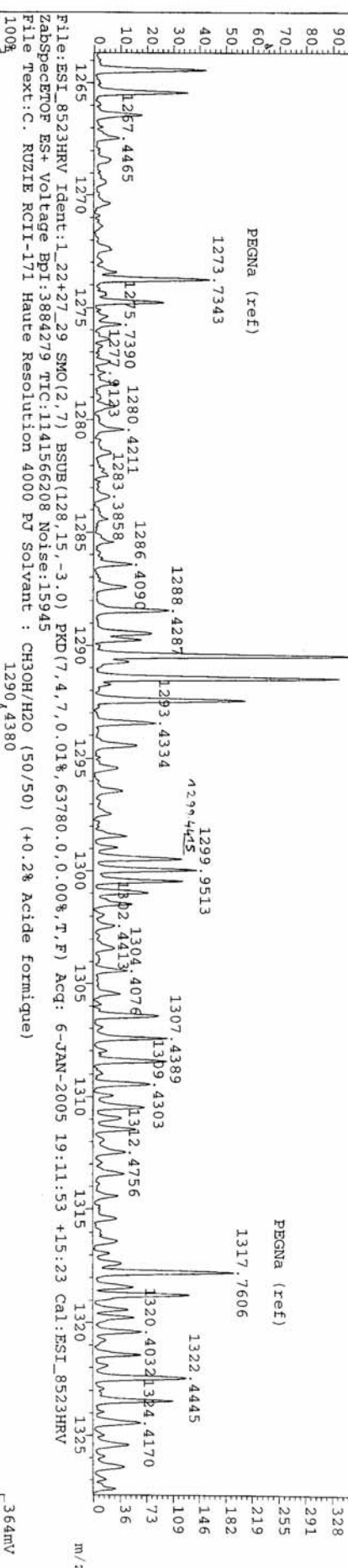
File:ESI\_85238 Ident:1 SMO(2.7) ESDB(128.15.-3.0) PRD(7.4.7.0.01% 23476.0.0.00% T.F) Acq: 6-JAN-2005 18:39:09 +12:43 Cal:ES000\_061018\_CS\_P\_FJ\_1X0.999419  
 ZabSpecTOP ES+ Magnet BpI:215325 TIC:62792620 Noise:5869  
 File Text:C. RUIZIE KClI-171 Basse Resolution 2000 FJ Solvant : CH3OH/H2O (50/50) (+0.2% Acide formique)



File: EST\_85238 Ident: 1 SMO72.7) ESUB(128.15--3.0) PKD7.4.7.0.018.23476.0.0.008.T.F) Acq: 6-JAN-2005 18:39:09 +12:43 Cal: E3000\_061018\_CS\_P\_PJ\_1x0.999419  
 ZABSPECTOR ES+ Magnet BpI:215325 TIC:62792620 Noise:5869  
 File Text: C. RUZIE RCII-171 Basse Resolution 2000 PU Solvant : CH3OH/H2O (50/50) (+0.28 Acide formique) 1290.4 M+.



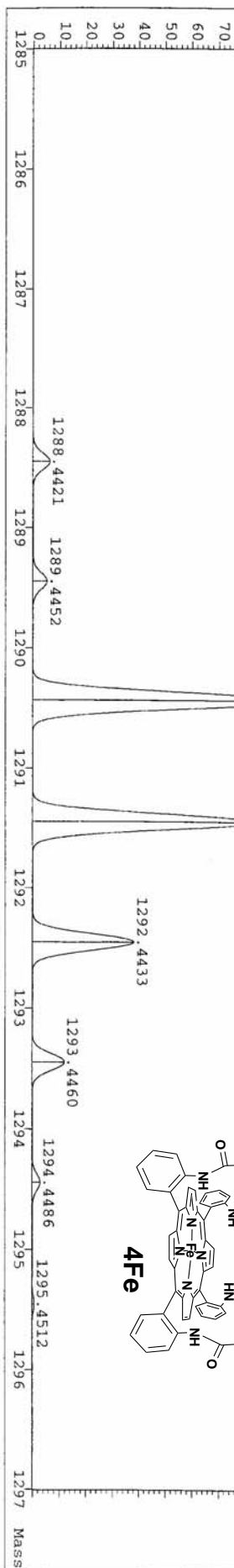
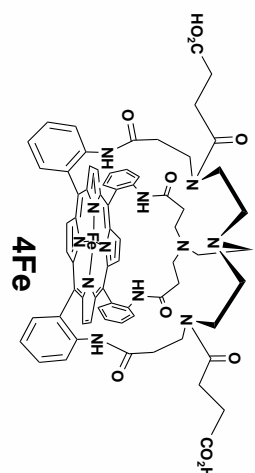
File:ESI\_8523HRV Ident:1\_22+27\_29 SMO(2,7) ESUB(128,15,-3.0) PKD(7,4,7,0.01%,63780,0,0.00%,T,F) Acq:6-JAN-2005 19:11:53 +15:23 Cal:ESI\_8523HRV  
 ZABSPECTOF ES+ Voltage BpI:3884279 TIC:1141566208 Noise:15945  
 File Text:C. RUZIE RCII-171 Haute Resolution 4000 PJ Solvant : CH3OH/H2O (50/50) (+0.2% Acide formique)  
 1290.4380 M+.

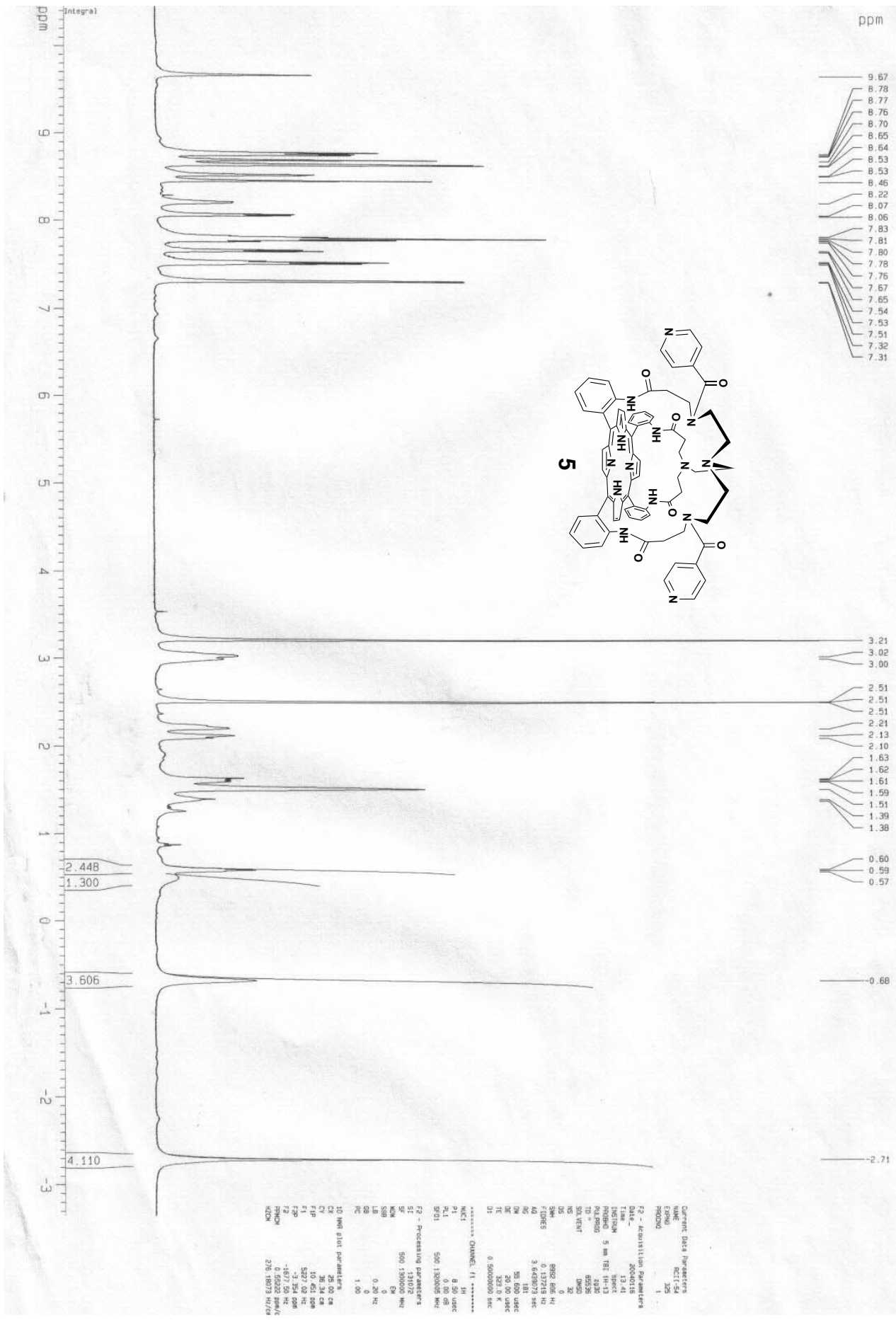


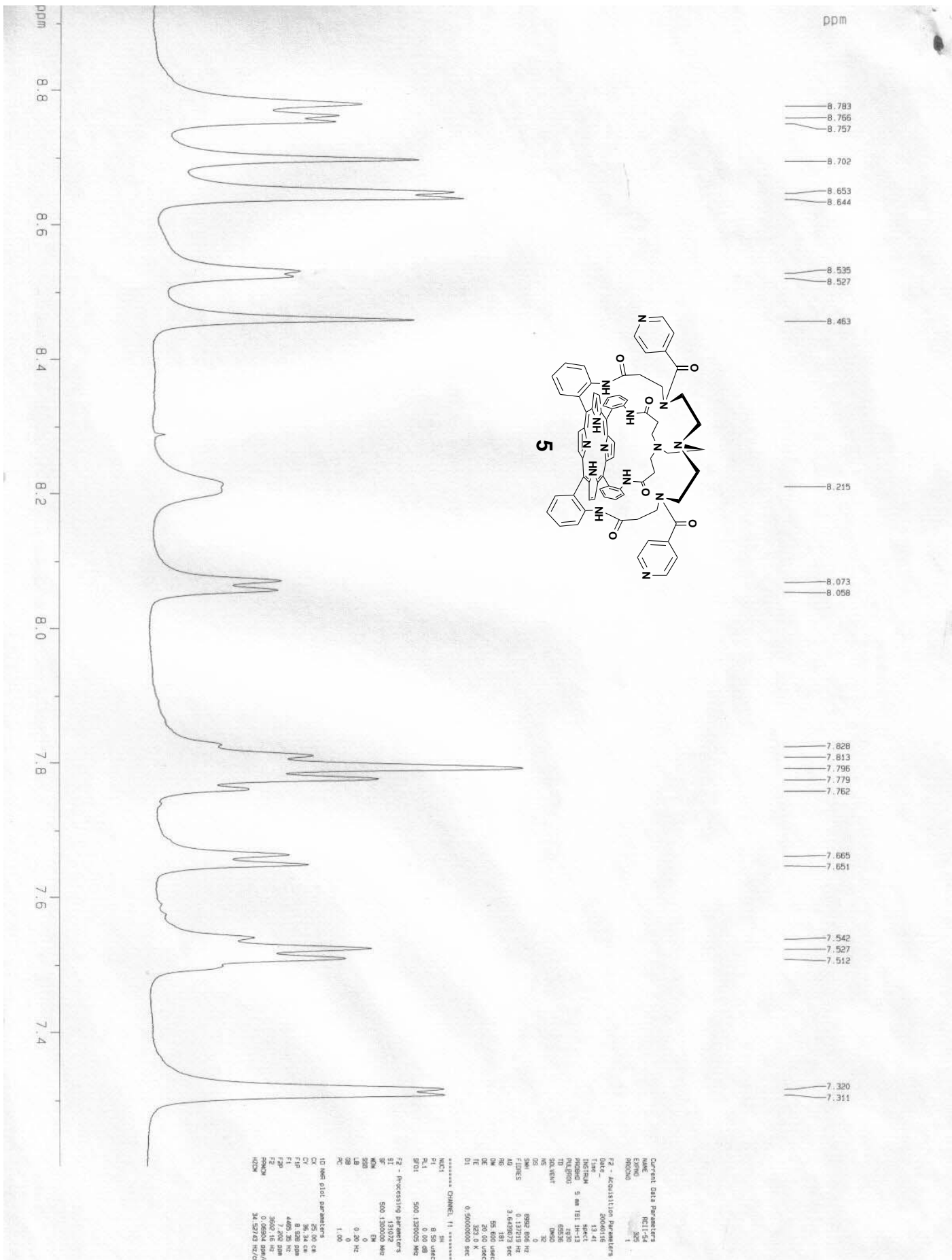
File:ESI\_8523HRV Ident:1\_22+27\_29 SMO(2,7) ESUB(128,15,-3.0) PKD(7,4,7,0.01%,63780,0,0.00%,T,F) Acq:6-JAN-2005 19:11:53 +15:23 Cal:ESI\_8523HRV  
 ZABSPECTOF ES+ Voltage BpI:3884279 TIC:1141566208 Noise:15945  
 File Text:C. RUZIE RCII-171 Haute Resolution 4000 PJ Solvant : CH3OH/H2O (50/50) (+0.2% Acide formique)  
 1290.4380 M+.

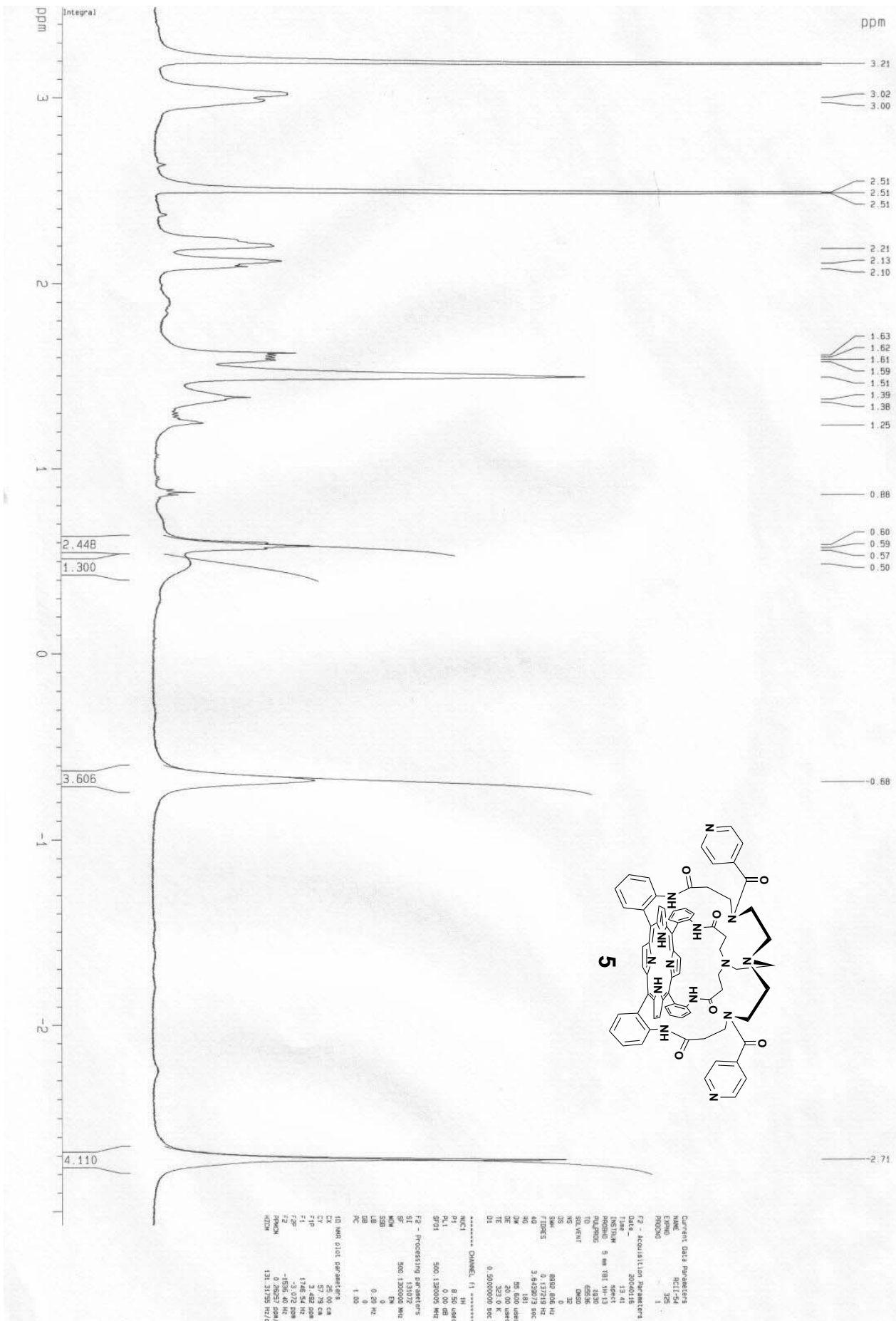
Amas isotopique theorique de M+ : C70 H66 N12 O10 Fe

12.13, 14C:70 1,2H:66 14,15N:12 16,17,18O:10 54,56,57,58Fe:1  
 Separation:1000 Min Frac Abun:0.01 Num Charges:1 Resolution:4000  
 Nominal Mass:1290 Monoisotopic Mass:1290.437 Average Mass:1291.220 Peak Maximum Mass:1290.44



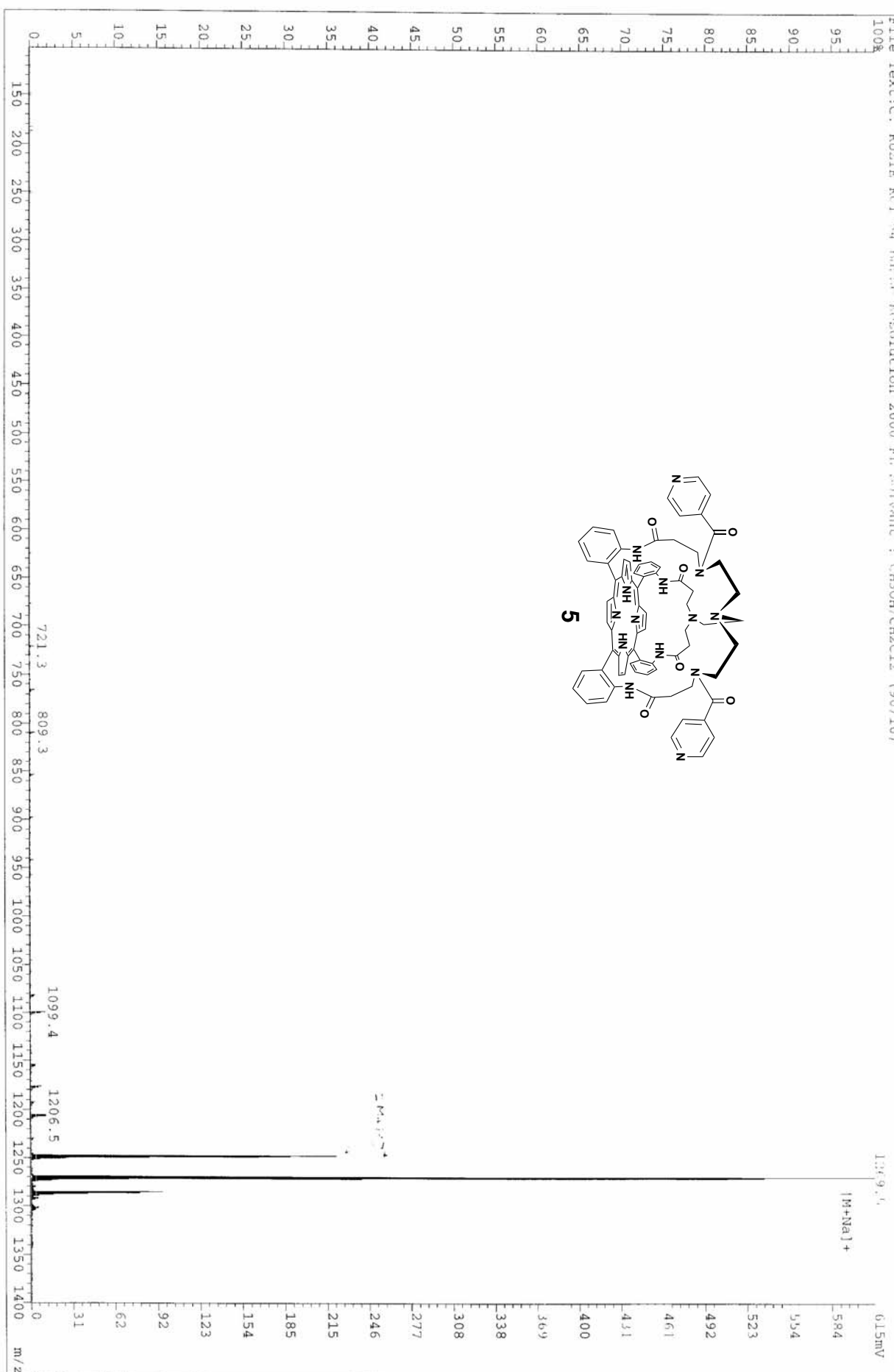
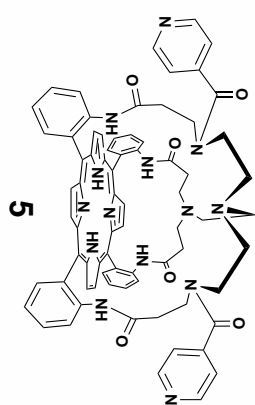




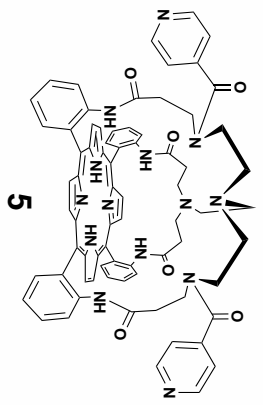
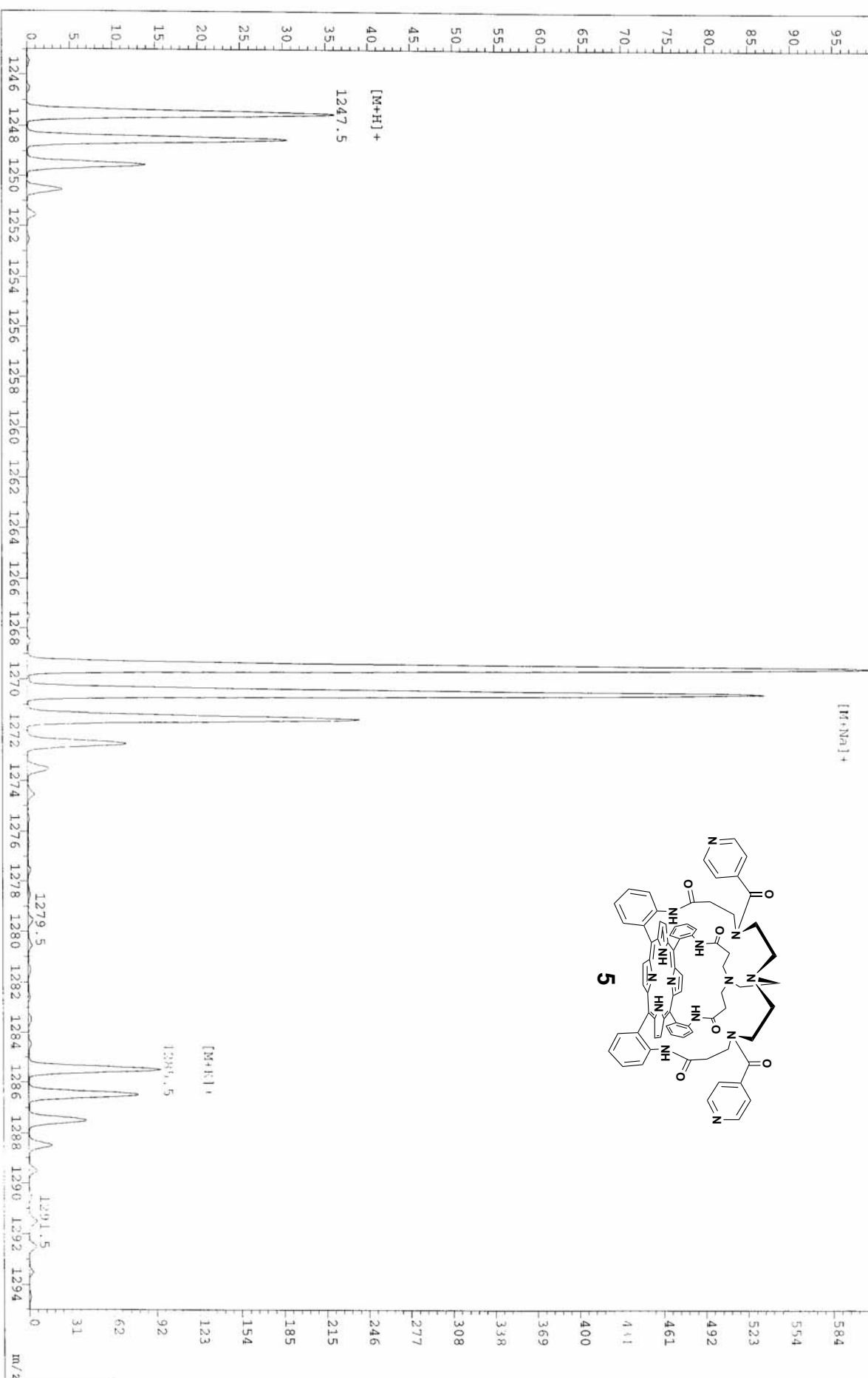


Current Data Parameters  
 NAME: RELI-54  
 EXPNO: 323  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_: 20040418  
 Time: 13:41  
 File: 54131313  
 PROBRG: 5 ms 101: 13  
 PULPROG: zgpg30  
 TO: 62536  
 SFO: 500.132405  
 DS: 4  
 SWH: 0  
 FIDRES: 0.1317219 Hz  
 AQ: 3.8423181 sec  
 RG: 65  
 SW: 65.600 usec  
 DE: 20.00 usec  
 TE: 323.0 K  
 DT: 0.50000000 sec  
 \*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1: 1H  
 P1: 0.20 usec  
 PL1: 0.00 dB  
 SFO1: 500.132405 MHz  
 F2 - Processing parameters  
 SI: 500.132405 MHz  
 KW: 64  
 SFO: 0.20 Hz  
 LB: 0.20 Hz  
 GB: 0  
 PC: 1.00  
 ID: NMR 9101: parameters  
 CX: 25.00 cm  
 CR: 2.00 cm  
 FI: 1.00 cm  
 FIP: 3.46200e  
 F1: 1746.54 Hz  
 F2P: 131.07200e  
 FREQ: 131.07200 MHz  
 ZPCW: 0.28257 Hz  
 HZCW: 131.31750 Hz

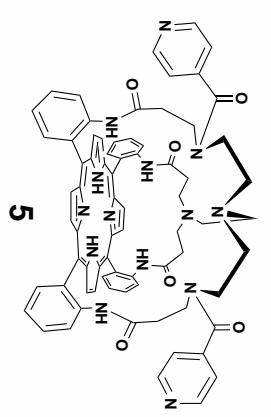
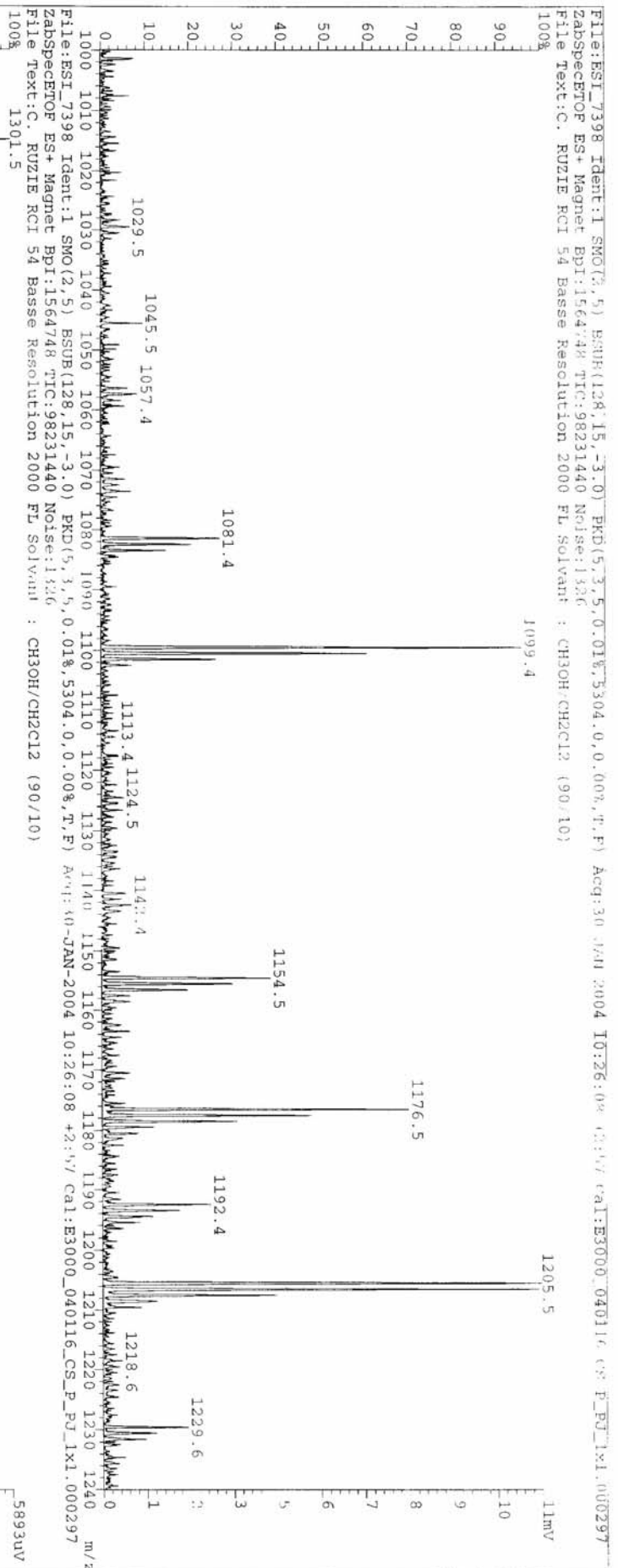
File: EST\_7398 Ident: 1 SMO(T, 4) H2O(128,15,-3.0) PKD(5,3,5,70,018,5304,0,0,00%,T,F) Acq: 10 JAN 2004 10:56:00 (01:53000\_04011) 00 P RT 101.000297  
 Zabspectof ES+ Magnet EPl: 100kV48 Pie: 98231440 Noise: 1326  
 File Text: C. RUIZIE K01 64 Intra-Resolution 2000 Pl. Solvant : CH3OH/CH2Cl2 (90/10)  
 100%



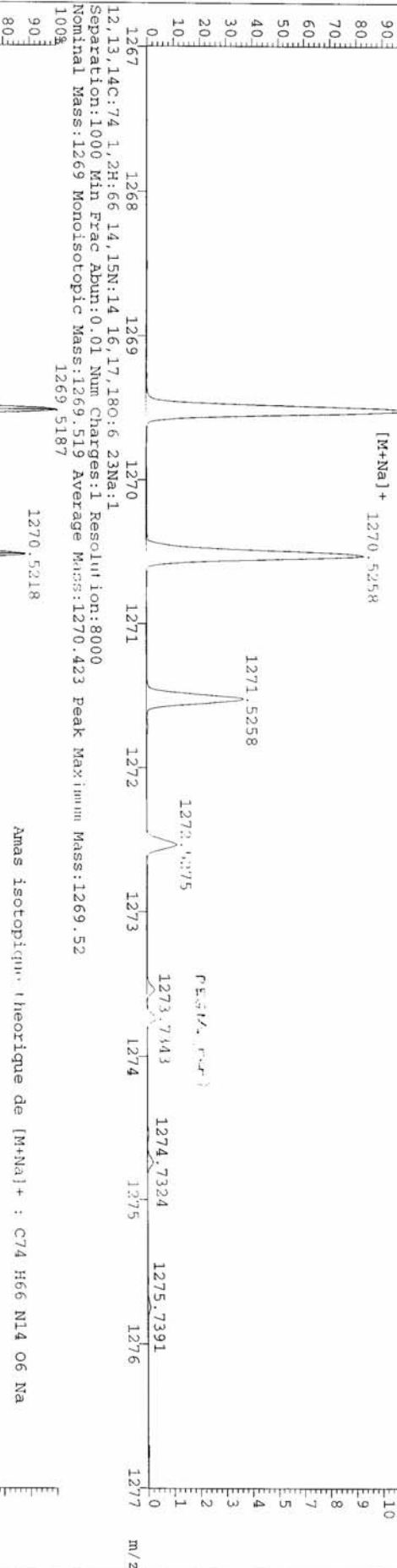
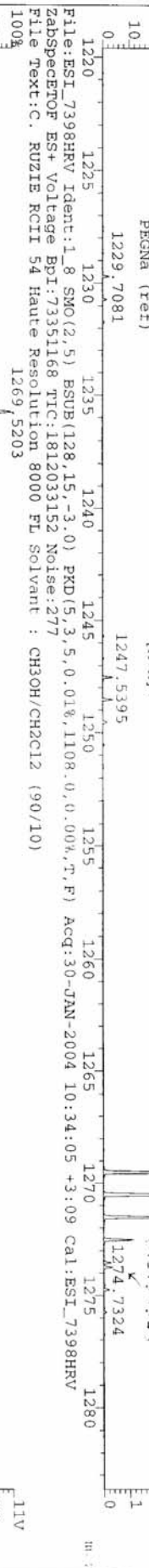
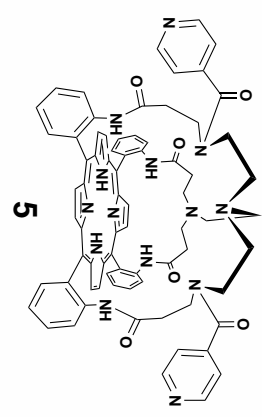
File: EST\_7398\_IDENT1.SMO(2,5) INSTR(128,15,-3,0) EPD(5,3,5,0,0,0,0) T.F.F. Acq:30 JAN 2004 10:26:08 12:17 CAL: E3000\_040110 (9, F, FT) x1.000297  
 Zabspectof ES+ Magnet PFI:156448 TIC:98231440 Noise:1326  
 File Text: C. RUZIE RCI V4 Base Resolution 2000 PL Solvant : CH3OH/CH2Cl2 (90/10)  
 1269.5







File:ESI\_7398HRV Ident:1\_8 SMO(2,5) BSUB(128,15,-3,0) PKD(5,3,5,0,01%,1108,0,0,00%,T,F) Acq: 00 JAN 2004 10:34:05 11:09 Cal:ESI\_7398HRV  
 ZabspecTOP ES+ Voltage BpI:73351168 TIC:1812033152 Noise:277  
 File Text: C. RUZIE RCI1 54 Haute Resolution 8000 FL Solvant : CH3OH (H2O)12 (90/10)

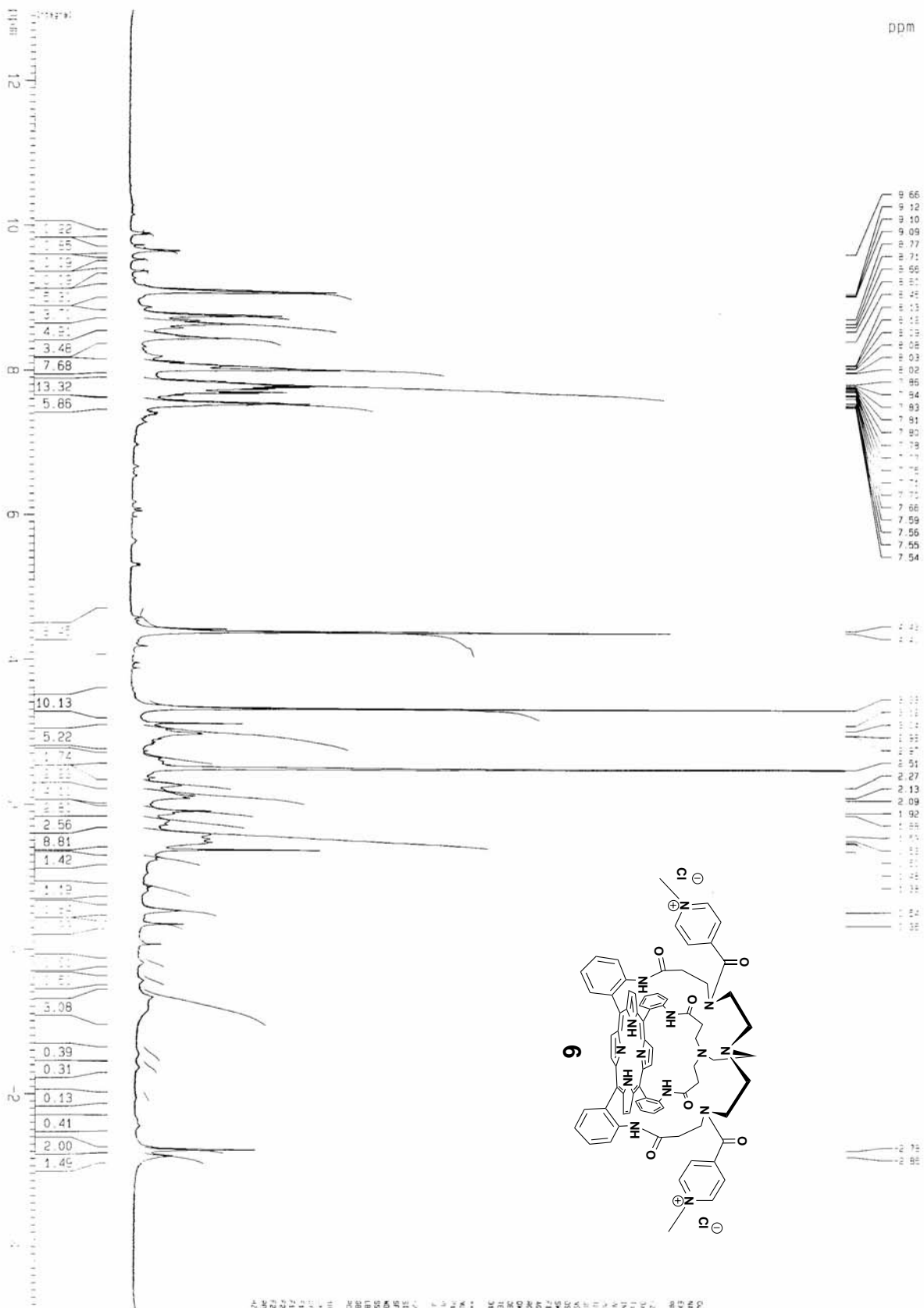


File:ESI\_7398HRV Ident:1\_8 SMO(2,5) BSUB(128,15,-3,0) PKD(5,3,5,0,01%,1108,0,0,00%,T,F) Acq: 00 JAN 2004 10:34:05 11:09 Cal:ESI\_7398HRV  
 ZabspecTOP ES+ Voltage BpI:73351168 TIC:1812033152 Noise:277  
 File Text: C. RUZIE RCI1 54 Haute Resolution 8000 FL Solvant : CH3OH/CH2Cl2 (90/10)

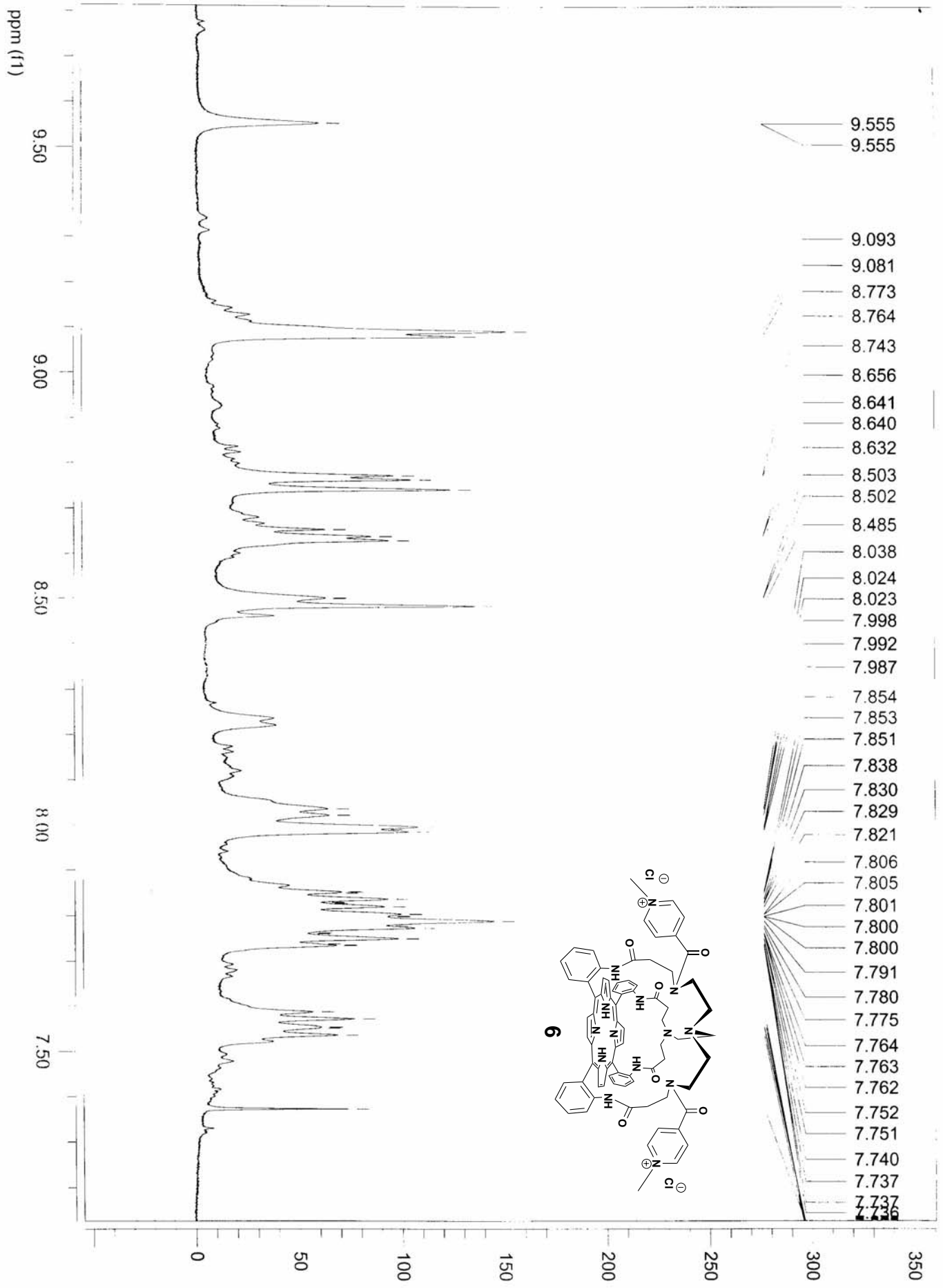
1267 1268 1269 1270 1271 1272 1273 1274 1275 1276 1277

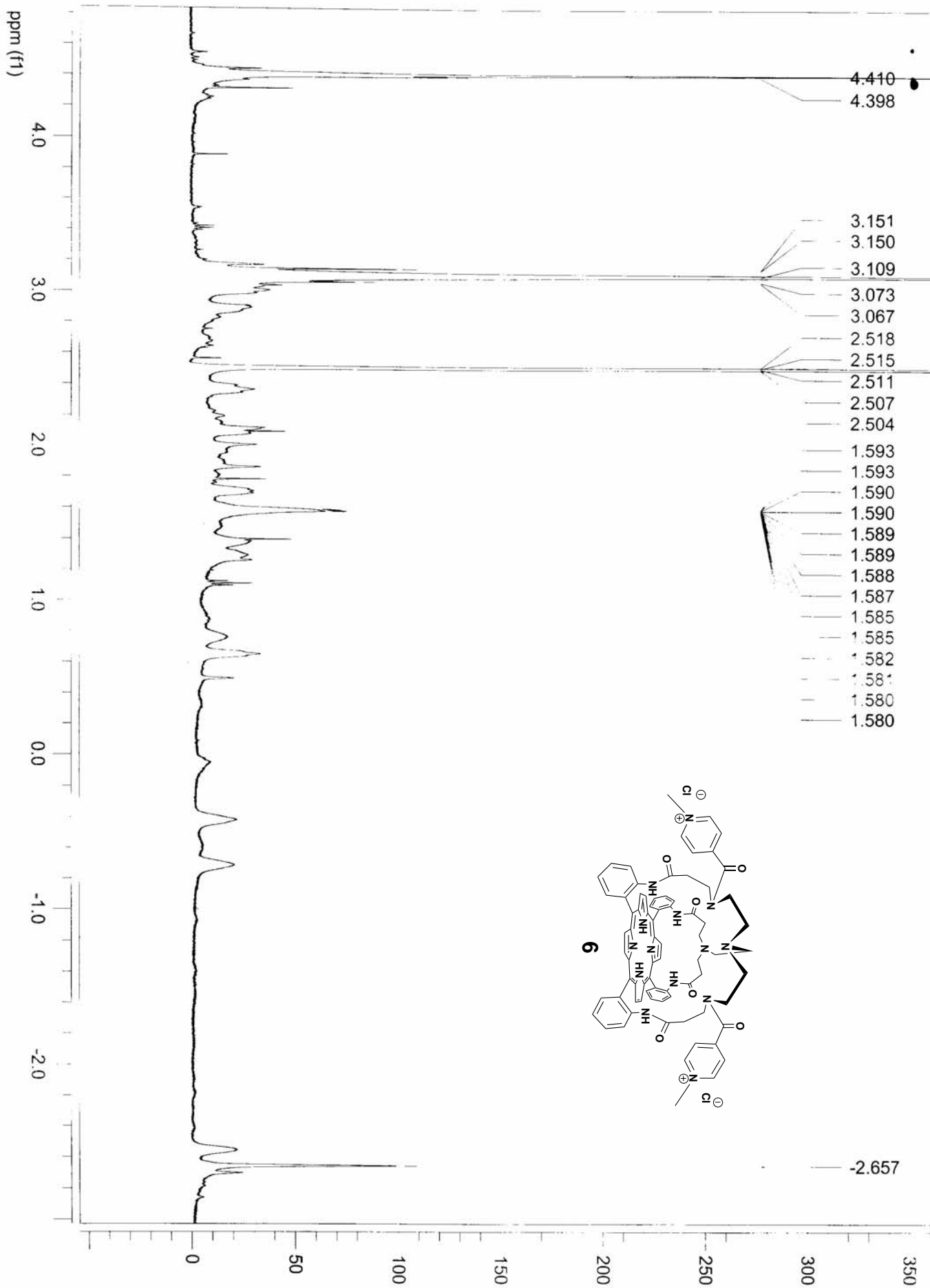
1267 1268 1269 1270 1271 1272 1273 1274 1275 1276 1277

12.13, 14C: 7.4 1.2H: 6.6 1.4, 15N: 1.4 1.6 1.7, 18O: 6.23Na: 1  
 Separation: 1000 Min Frag Abun: 0.01 Num Charges: 1 Resolution: 8000  
 Nominal Mass: 1269 Monoisotopic Mass: 1269.519 Average Mass: 1270.423 Peak Maximum Mass: 1269.52

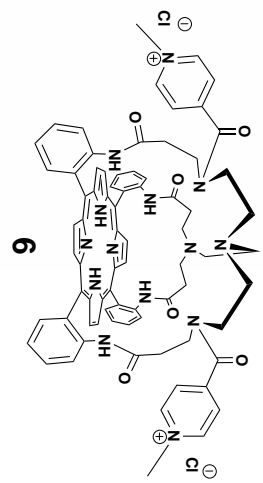
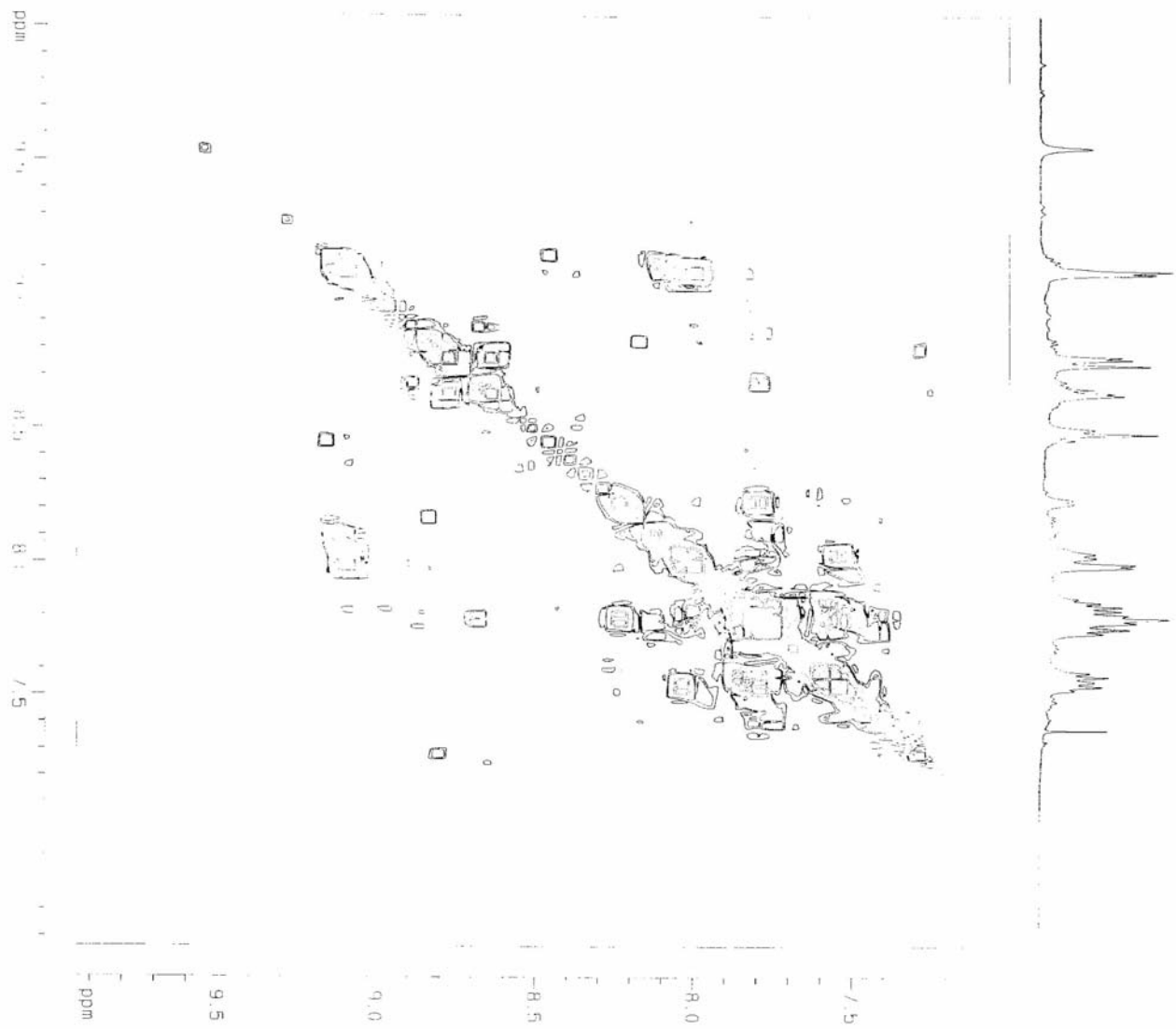


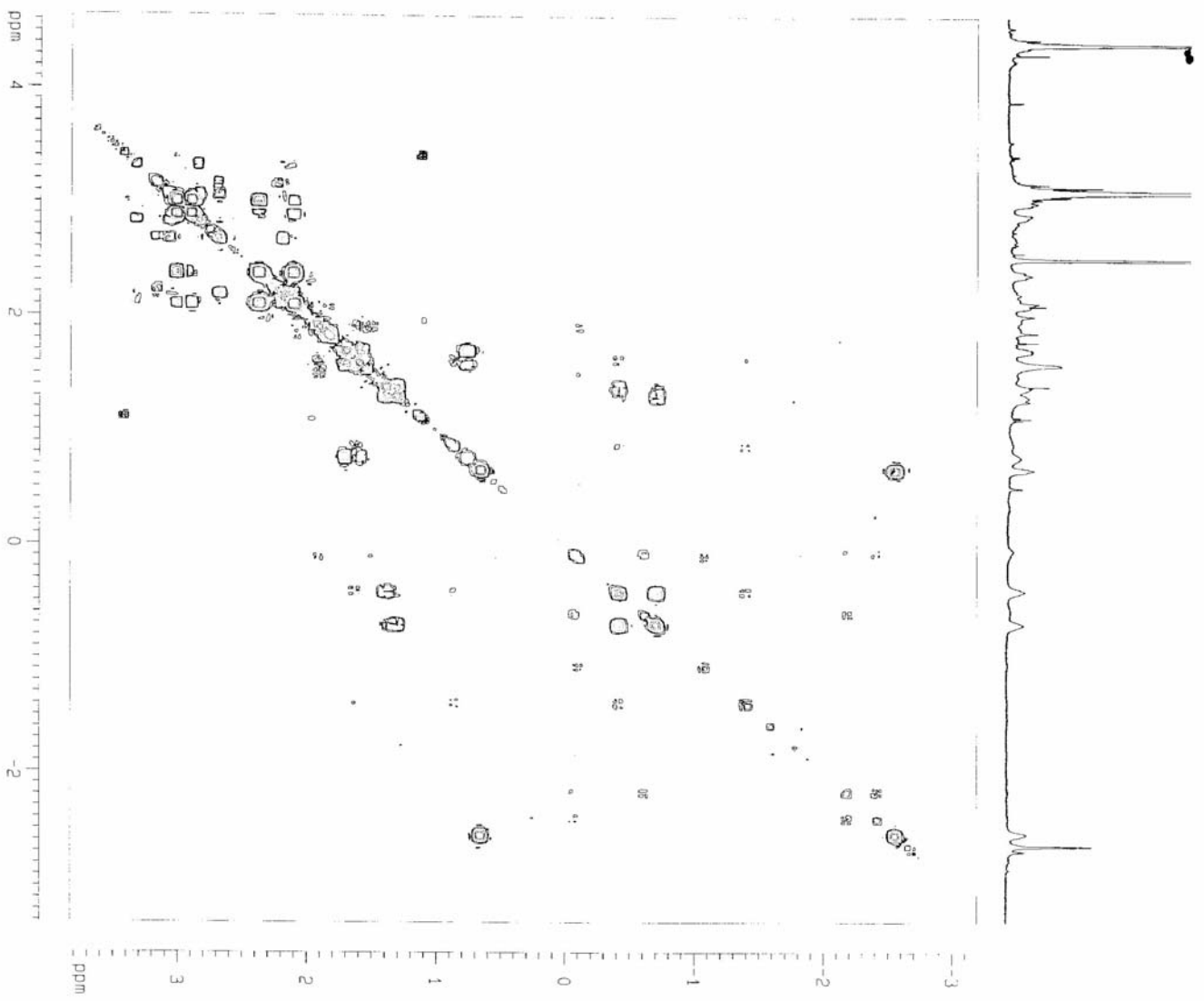
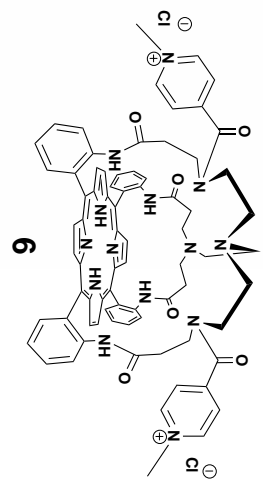
Report Data Parameters  
 Name: 6  
 EXPID: 6111  
 PROCNO: 1  
 F2: 500.130000 MHz  
 F1: 125.760000 MHz  
 A10: 5.000000 MHz  
 Acquisition Parameters  
 Date\_ Time: 20080313 10:11:11  
 Instrument: spect  
 Processor: zgpg30  
 F2: 500.130000 MHz  
 F1: 125.760000 MHz  
 A10: 5.000000 MHz  
 SFO: 500.130000 MHz  
 P1: 0.000000 sec  
 T1: 0.000000 sec  
 D1: 0.000000 sec  
 A1: 0.000000 sec  
 SFO: 500.130000 MHz  
 P1: 0.000000 sec  
 T1: 0.000000 sec  
 D1: 0.000000 sec  
 A1: 0.000000 sec  
 \*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1: 13  
 P1: 0.000000 sec  
 F1: 125.760000 MHz  
 A10: 5.000000 MHz  
 Acquisition Parameters  
 Name: 6  
 EXPID: 6111  
 PROCNO: 1  
 F2: 500.130000 MHz  
 F1: 125.760000 MHz  
 A10: 5.000000 MHz  
 SFO: 500.130000 MHz  
 P1: 0.000000 sec  
 T1: 0.000000 sec  
 D1: 0.000000 sec  
 A1: 0.000000 sec  
 SFO: 500.130000 MHz  
 P1: 0.000000 sec  
 T1: 0.000000 sec  
 D1: 0.000000 sec  
 A1: 0.000000 sec



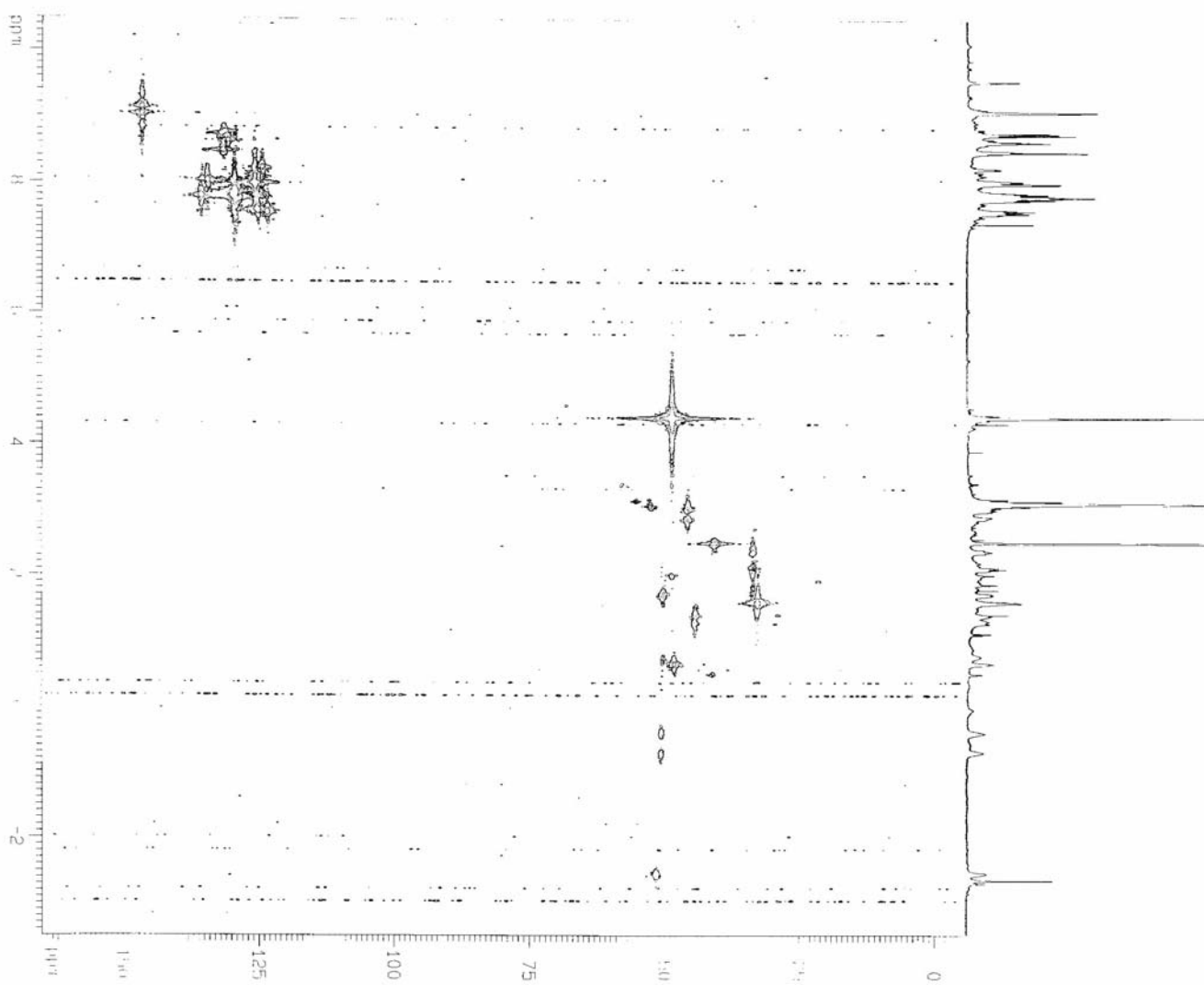
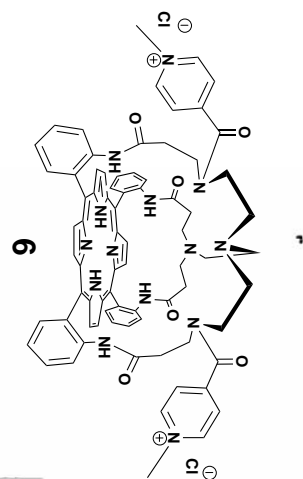


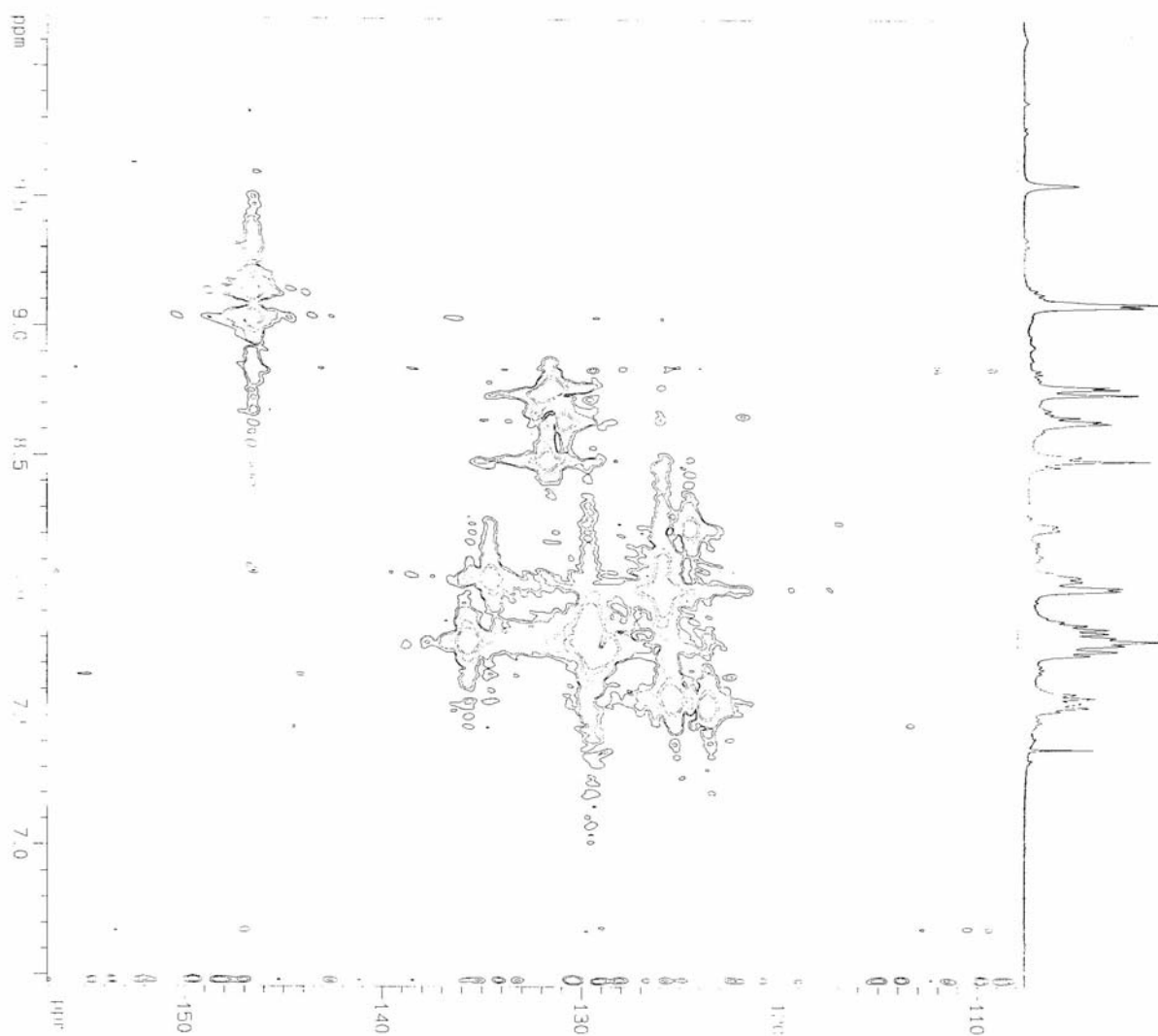
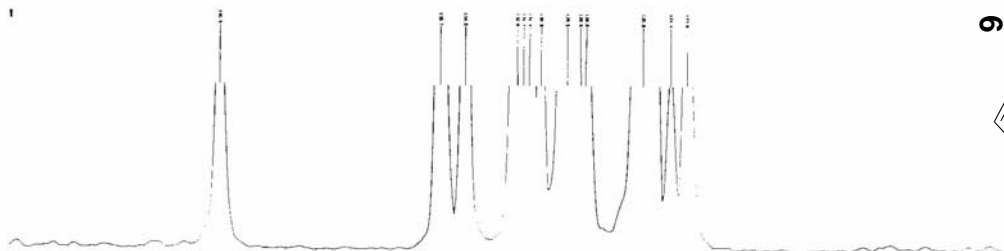
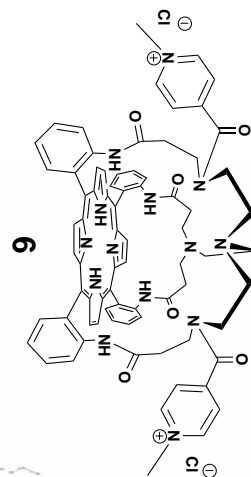


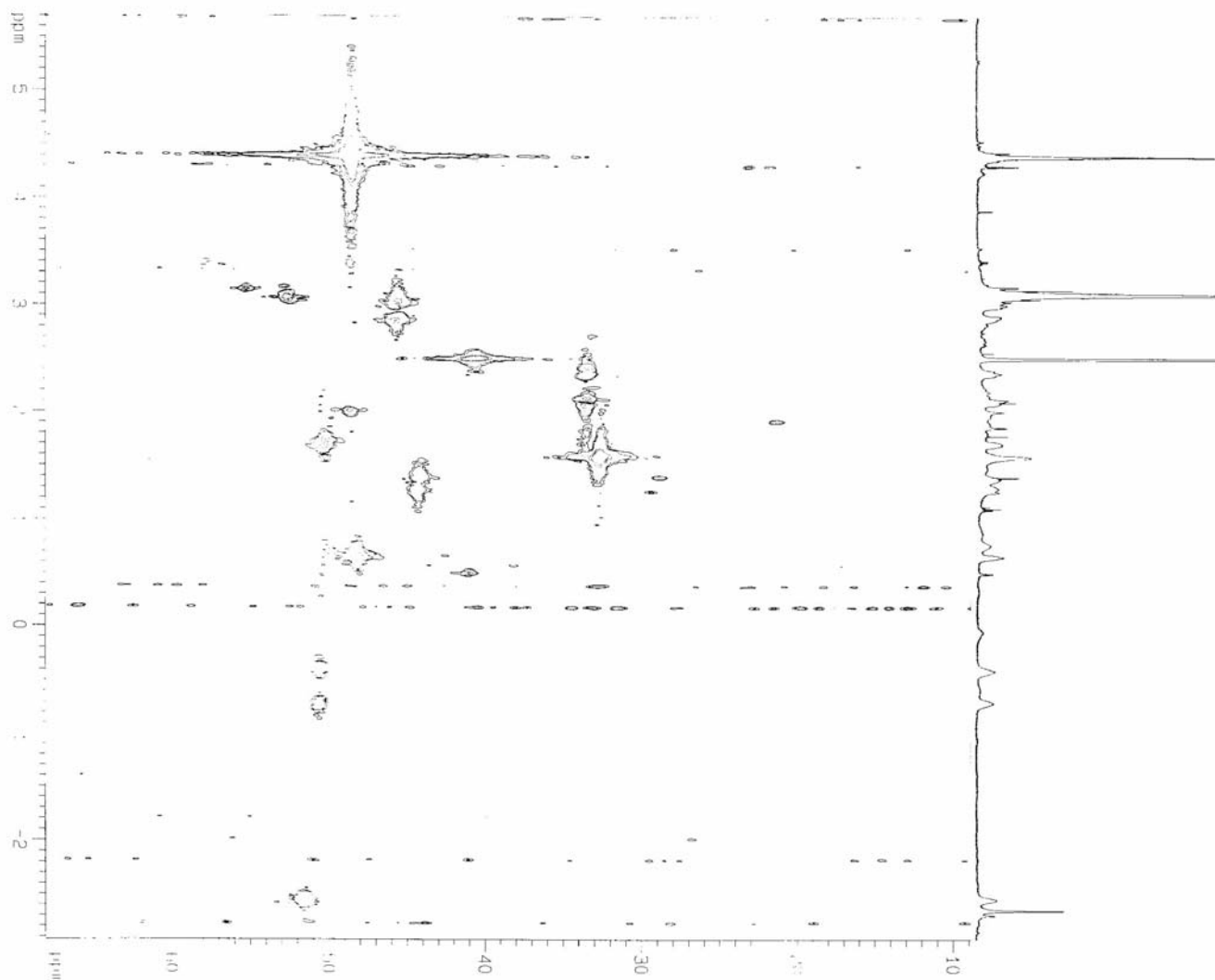
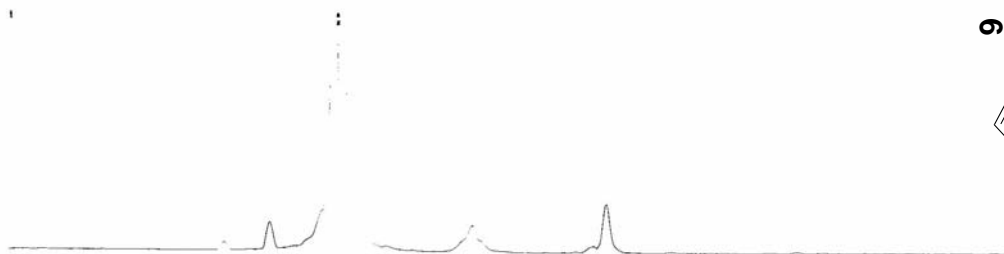
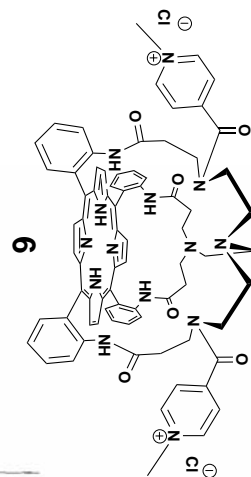


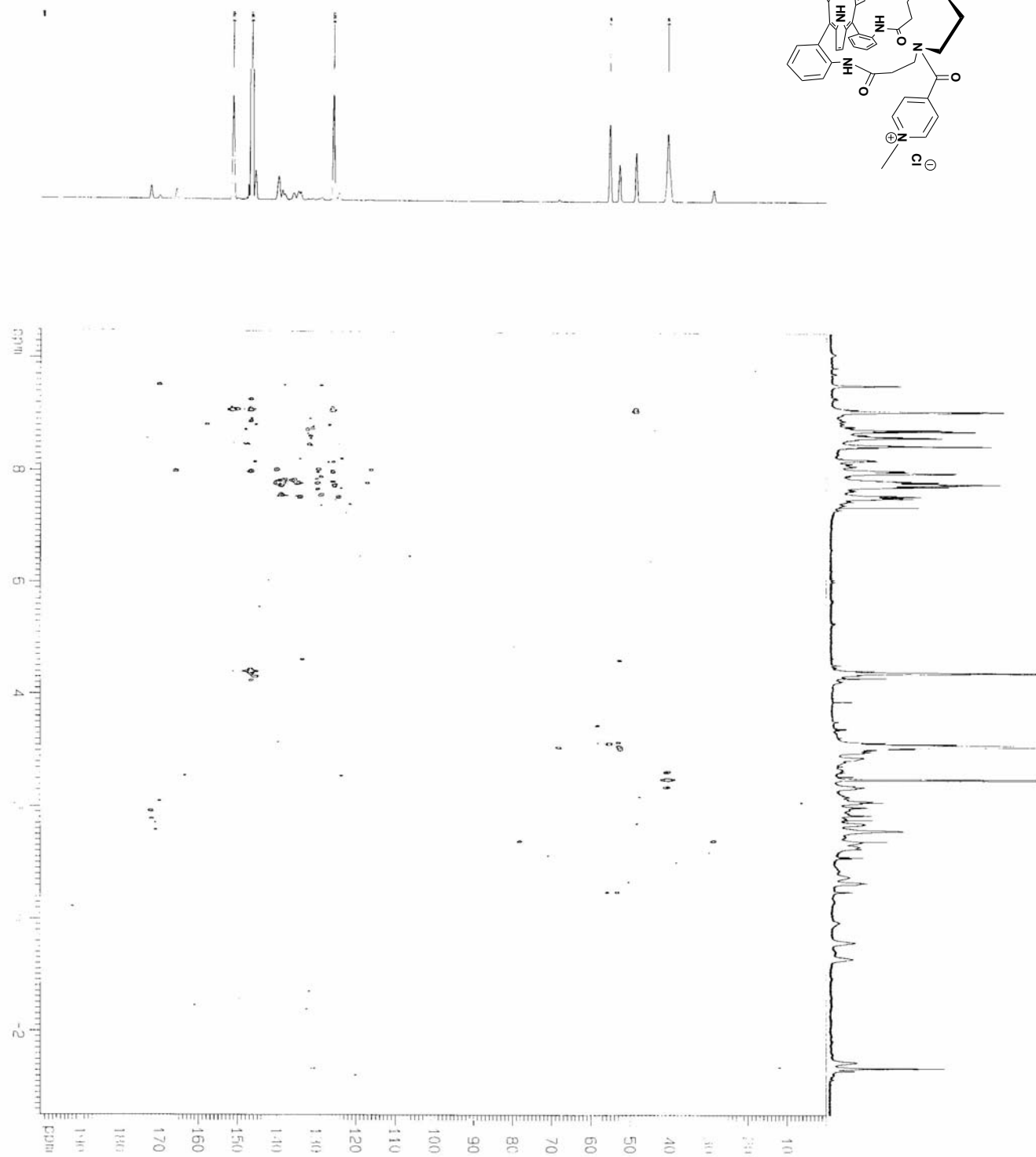
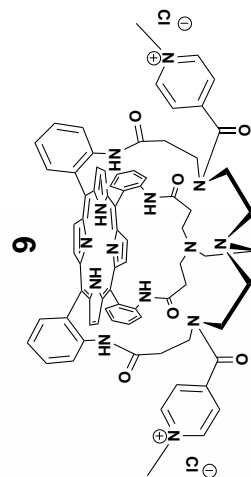




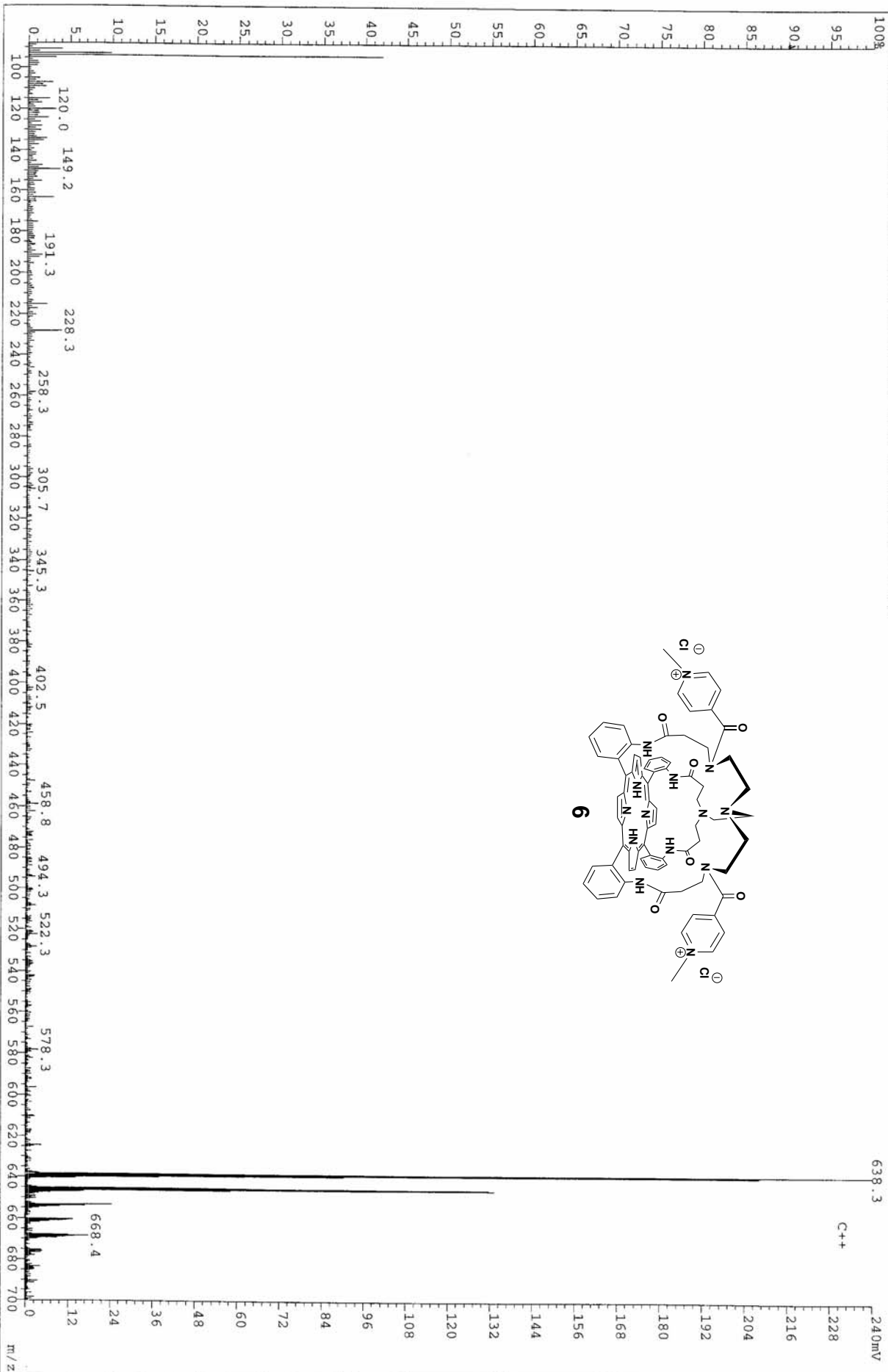
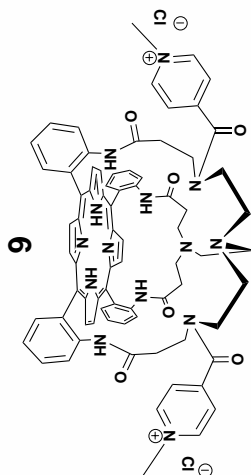




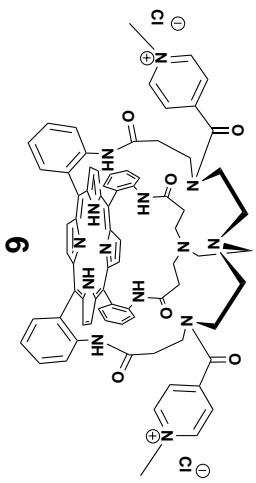
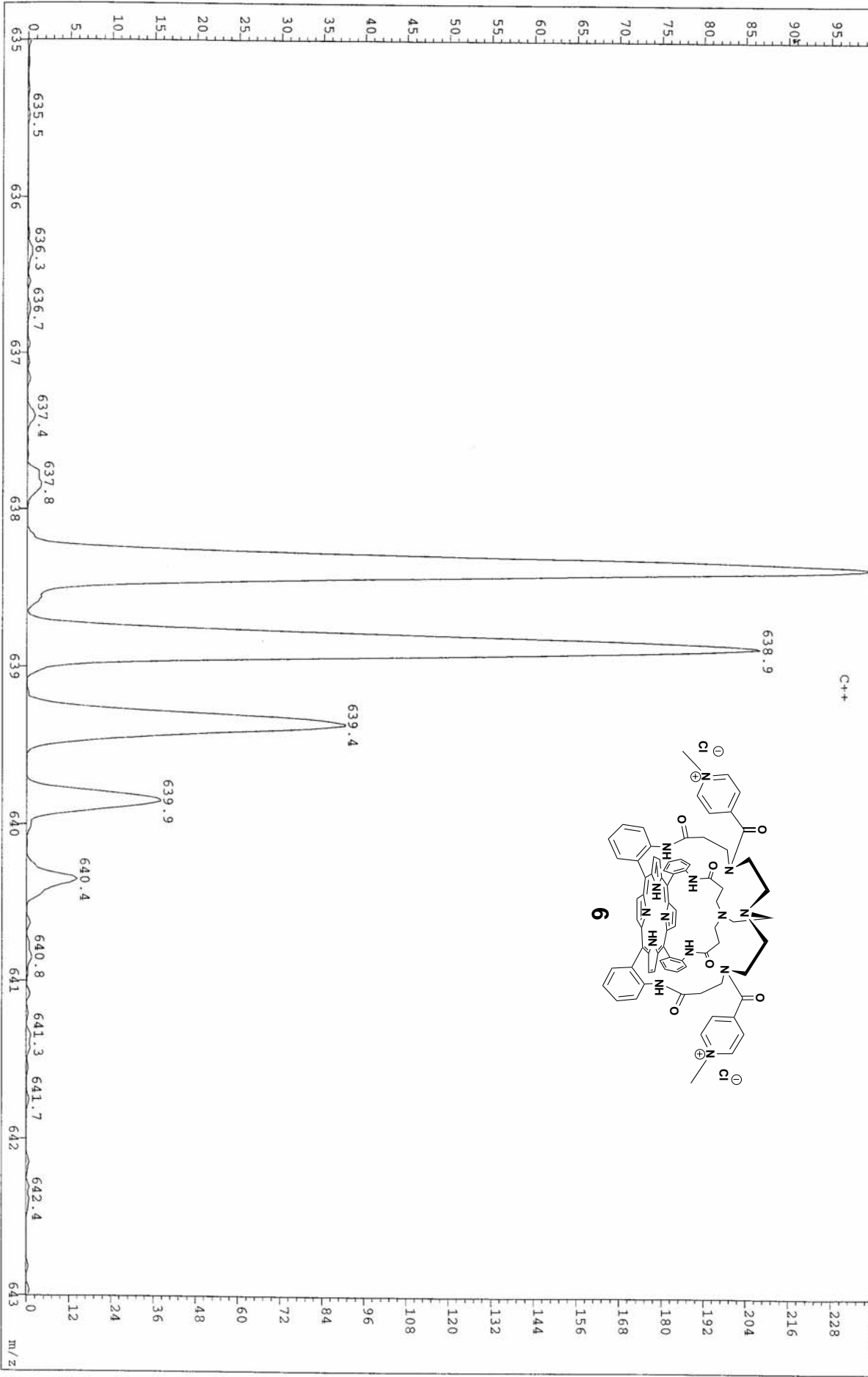




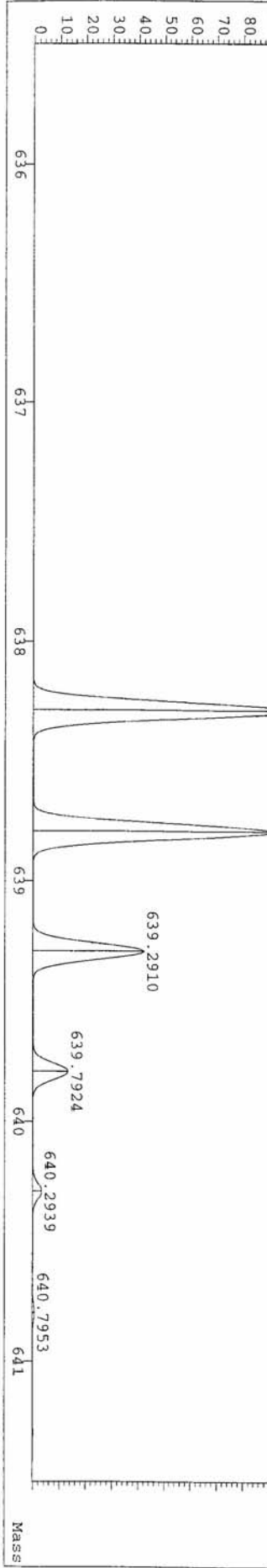
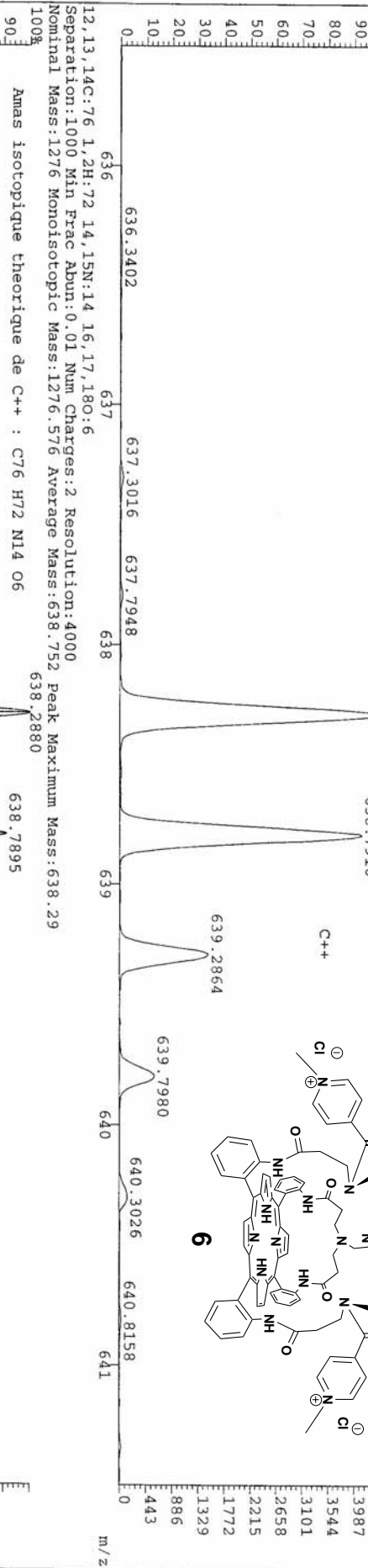
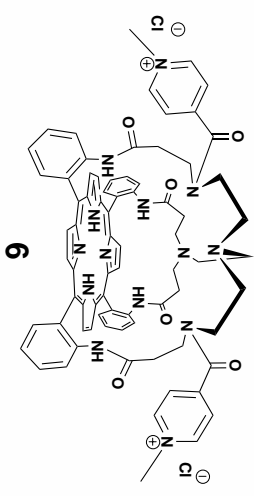
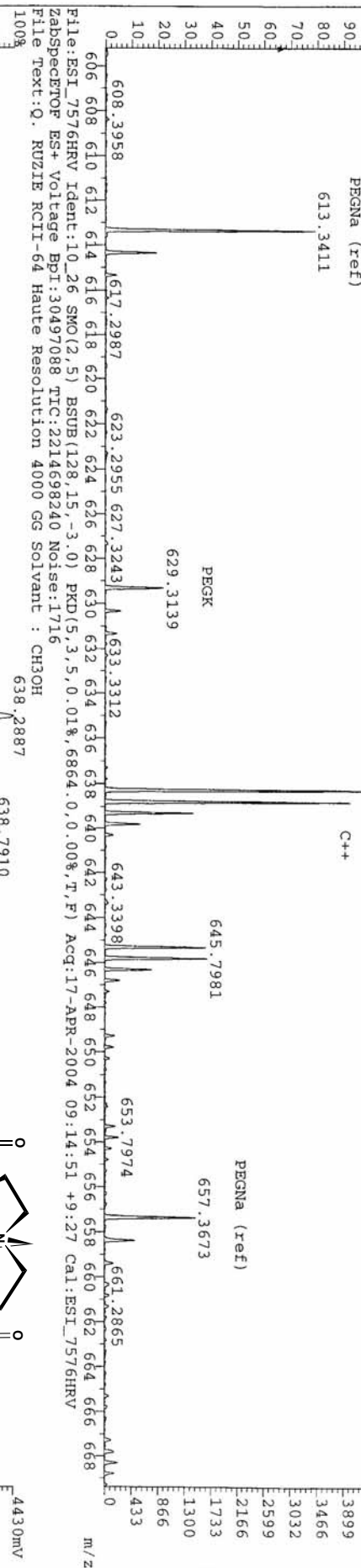
File:ESI\_7576 Ident:1 SM012,5) BSUB(128,15,-3.0) PKD(5,3,5,0.01%,108,0,0.00%,T,F) Acq:17-APR-2004 09:01:04 +6:31 Cal:E3000\_040116\_CS\_P\_EJ\_LX0.999937  
 ZabsPECTOR ES+ Magnet Bpl:490198 TIC:70164168 Noise:27  
 File Text:Q. RUIE RCI1-64 Basse Resolution 2000 GG Solvant : CH3OH



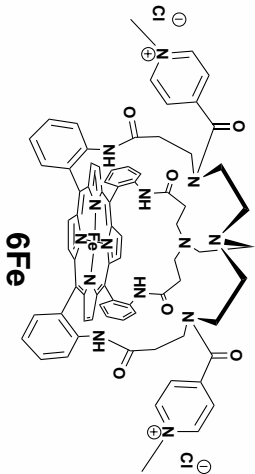
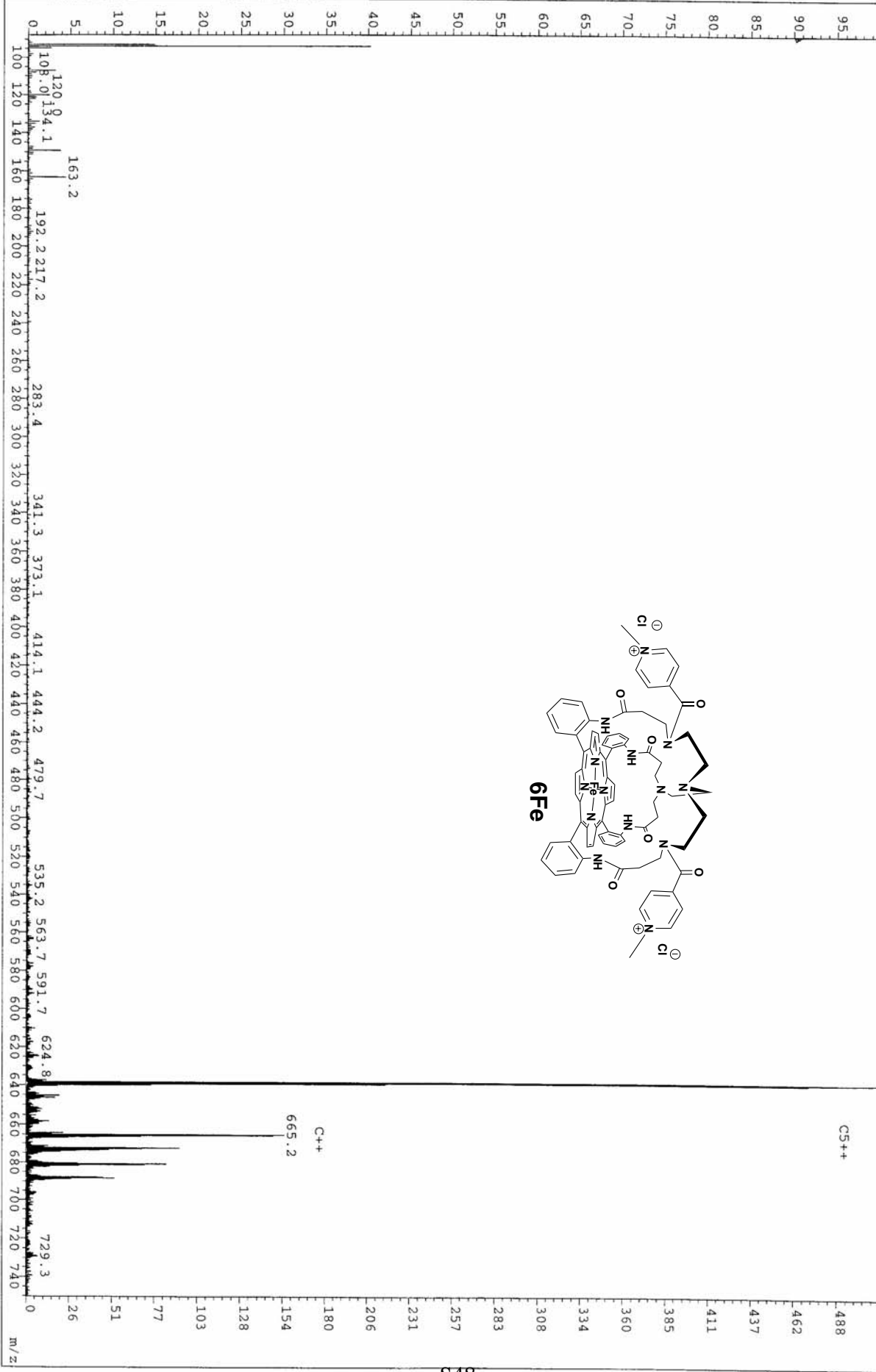
File:ESI\_7576 Ident:1 SMO(2.5) ESDB(128.15,-3.0) PKD15.3.5,0.01%,108.0,0.00%,T,F) Acq:17-APR-2004 09:01:04 +6:31 Cal:E3000\_040116\_CS\_P\_Pf\_Ix0.999937  
 ZabspecTOF ES+ Magnet BpI:490198 TIC:70164168 Noise:27  
 File Text:Q. RUIZIE KCI1-64 Basse Resolution 2000 GG Solvant : CH3OH  
 100%



File: EST\_7576HRV Ident: 11\_26\_SMO(2,5) BSUB(128,15,-3.0) PKD(5,3,5,0.018,6852.0,0.008,T,F) Acq: 17-APR-2004 09:14:51 +9:27 Cal: EST\_7576HRV  
 ZabsSPECTOP ES+ Voltage BpI: 29934400 TIC: 2033758976 Noise: 1713  
 File Text: Q, RUIZIE RCI1-64 Haute Resolution 4000 GG Solvant : CH3OH

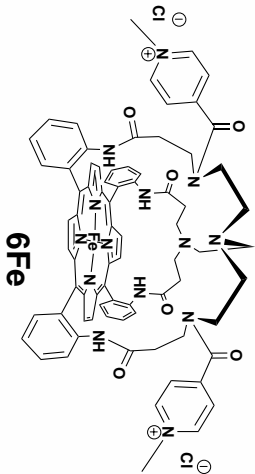
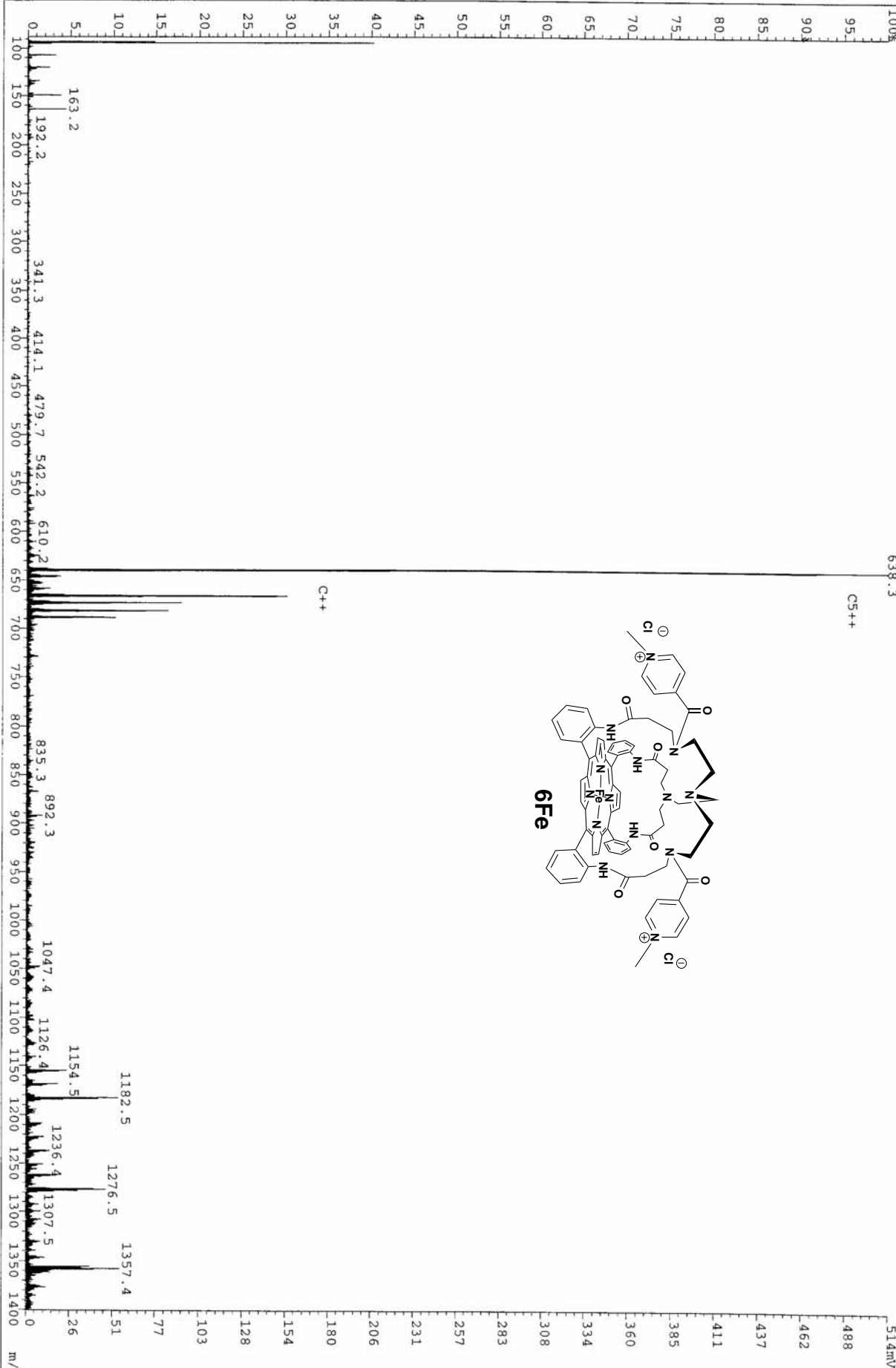


File: EST\_8584A Ident: 1 SMO(2.7) BSUB(128.15 -3.0) PKD(7.3.7, 0.01%, 12844.0, 0.00%, T.F) Acq: 24-JAN-2005 18:14:53 +3:33 Cal: E3000\_050119\_CS\_P\_F0\_1x0.999959  
 ZabsSpecTOP ES+ Magnet BPI: 766021 TTC: 130571608 Noise: 3211  
 File Text: C. RUZIE RCLII-40 Basse Resolution 3000 GG Solvant : CH3OH/H2O (95:5)

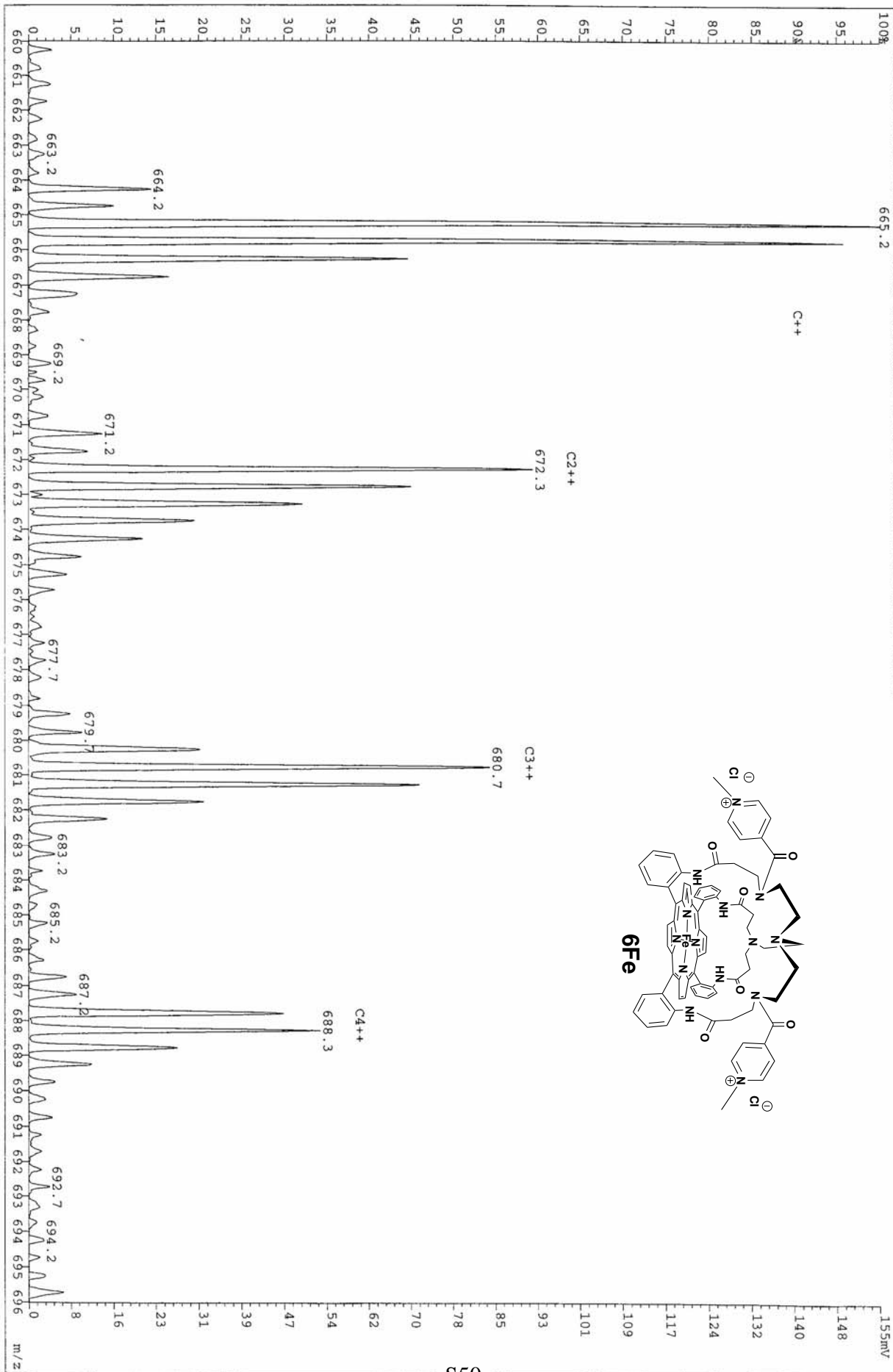
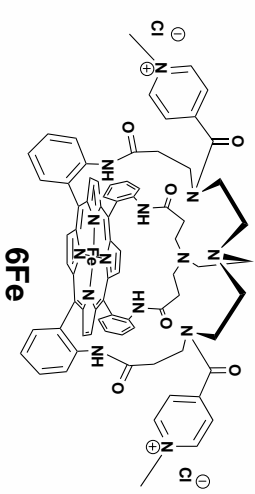


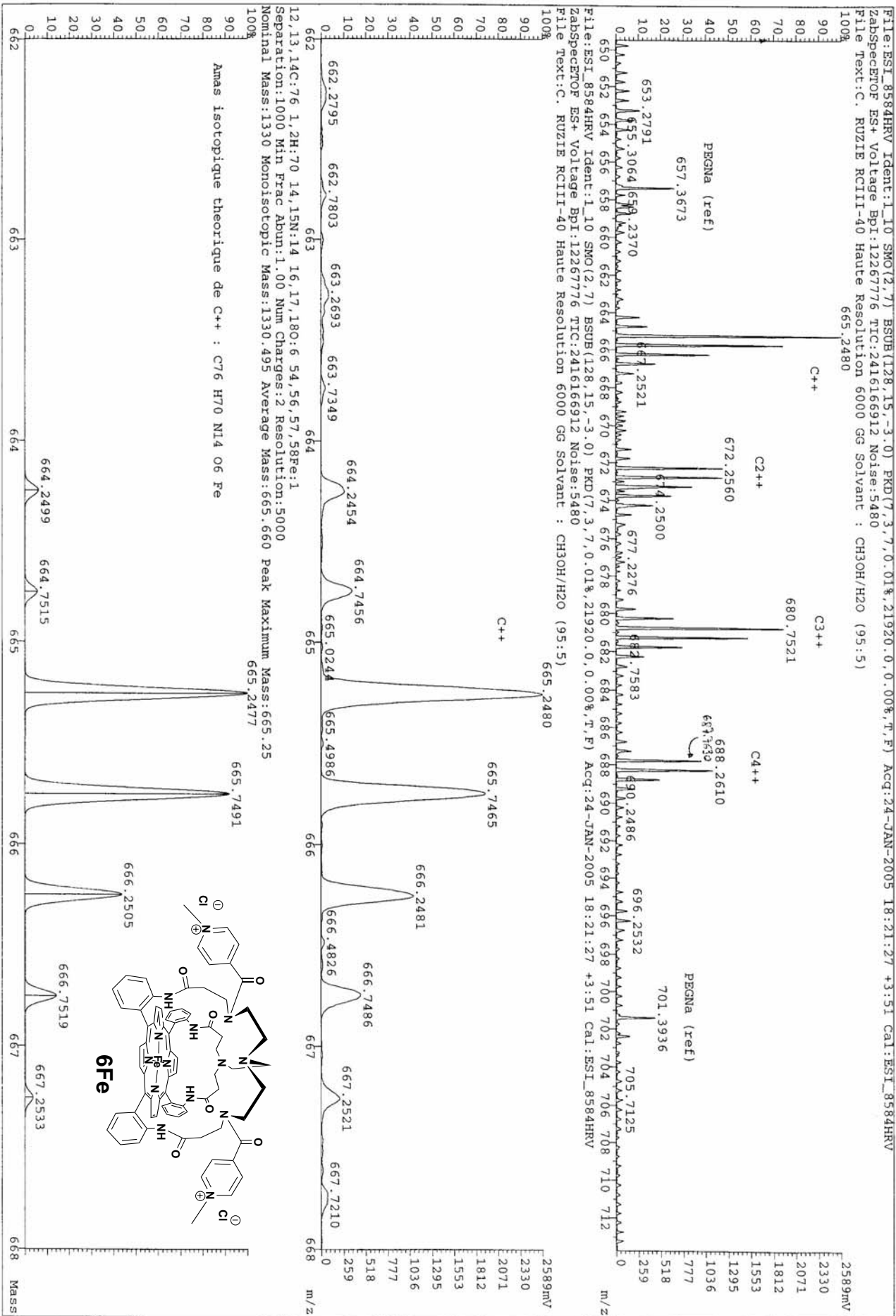


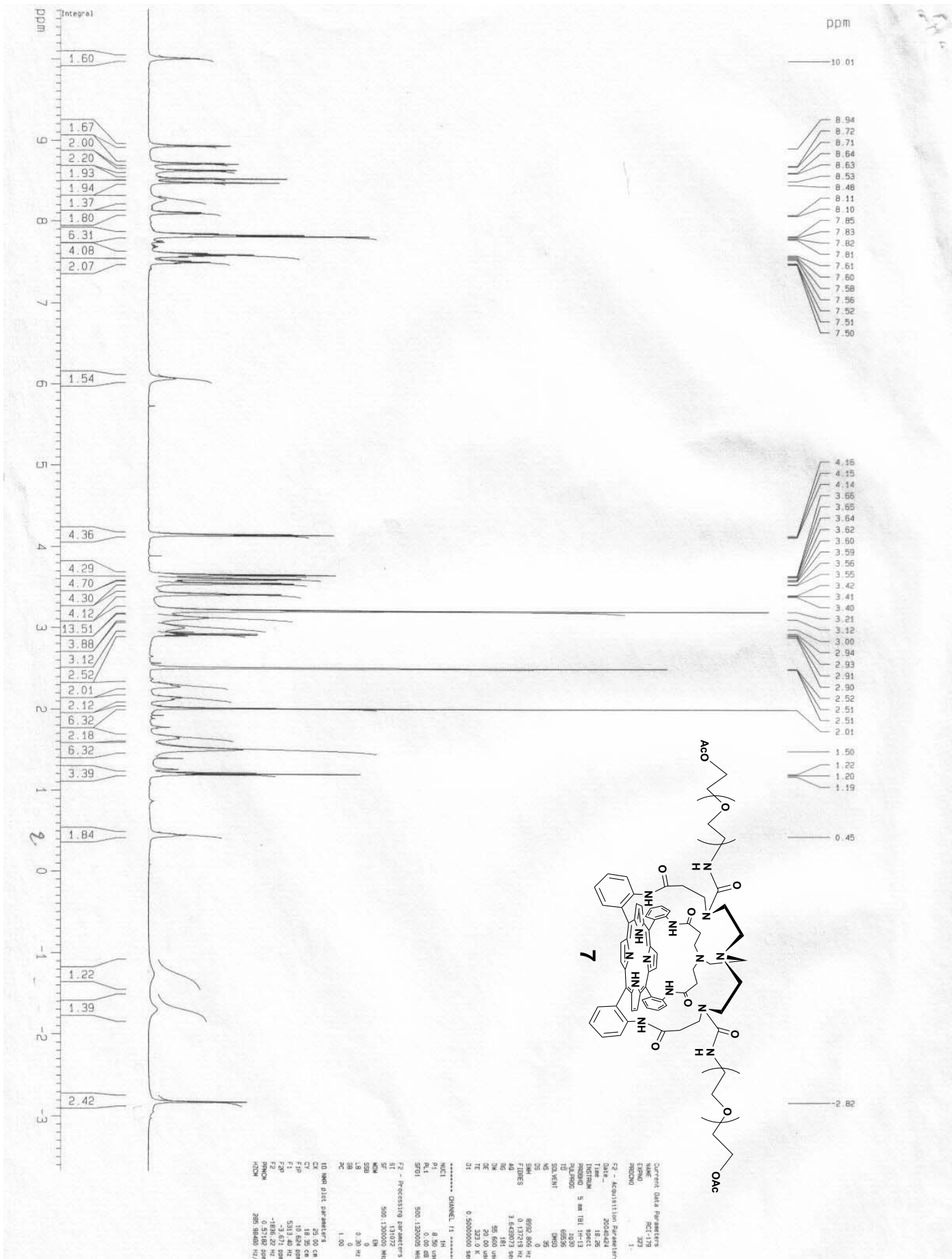
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 ZabsSPECTOP ES+ Magnet BpI:766021 TIC:130571608 Noise:3211  
 File Text:C, RUIZIE RCI11-40 Basse Resolution 3000 Gg Solvant : CH3OH/H2O (95:5)  
 100% 638.3

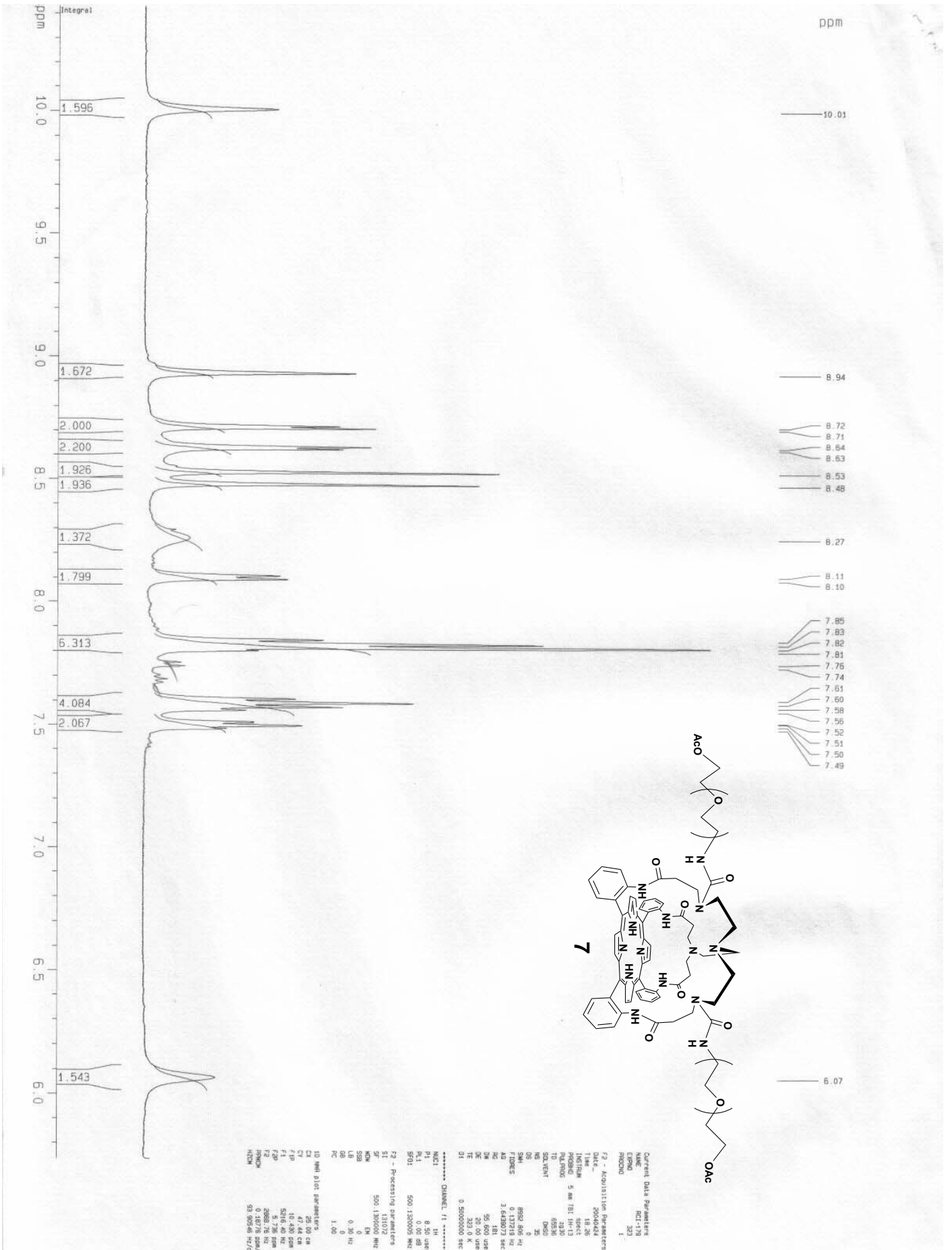


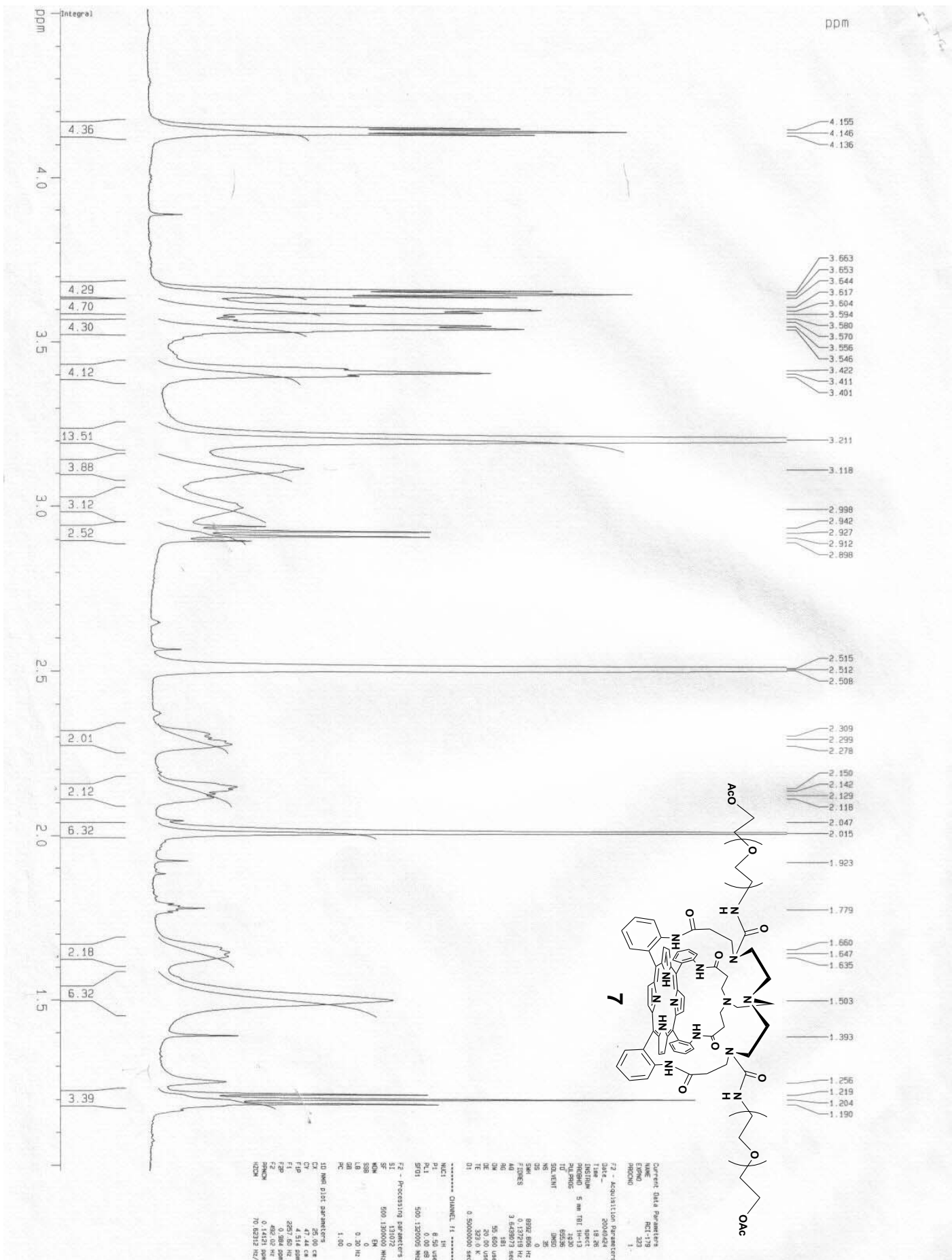
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 ZAPSpecTOP ES+ Magnet Bp1:766021 TIC:130571608 Noise:3211  
 File Text:C. RUZIE RCUIT-40 Basse Resolution 3000 GG Solvant : CH3OH/H2O (95:5)

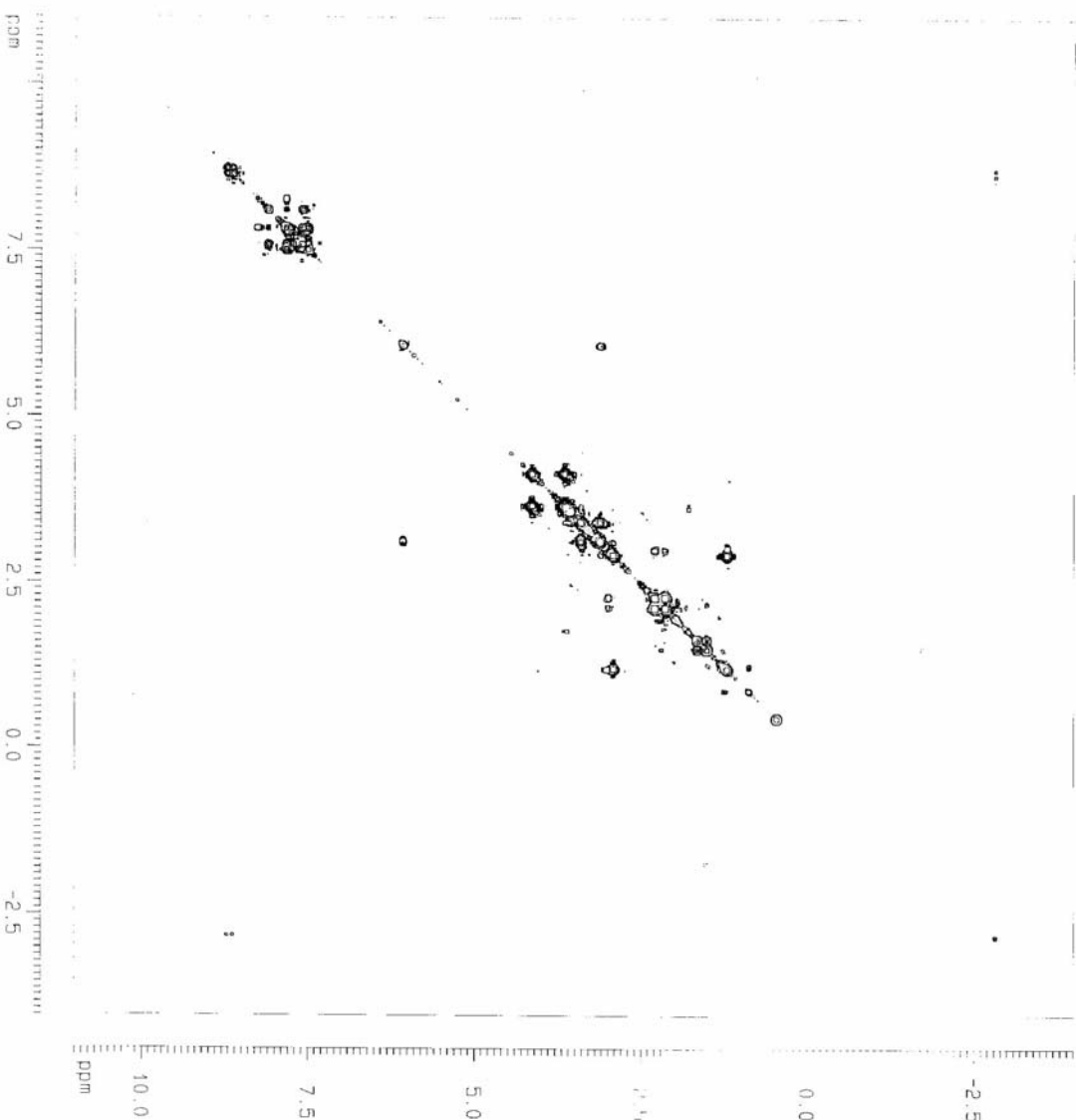
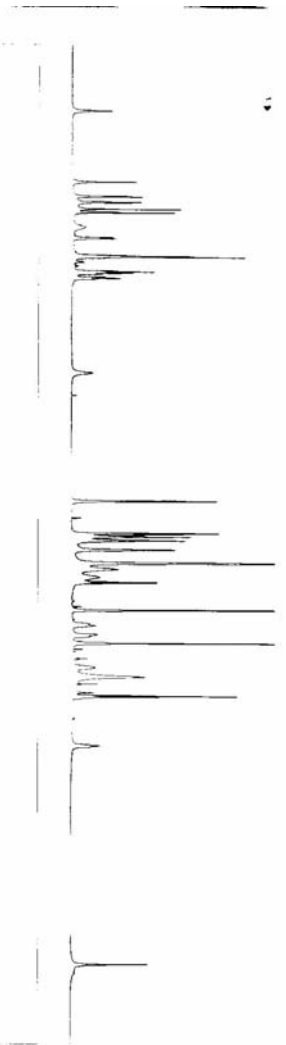
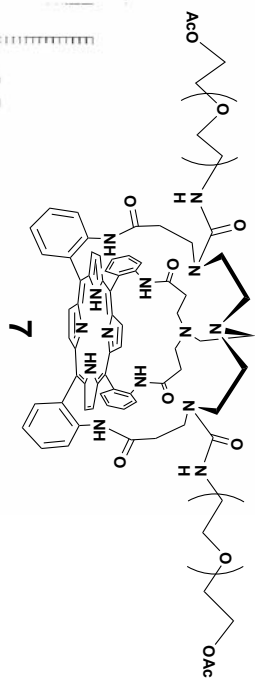


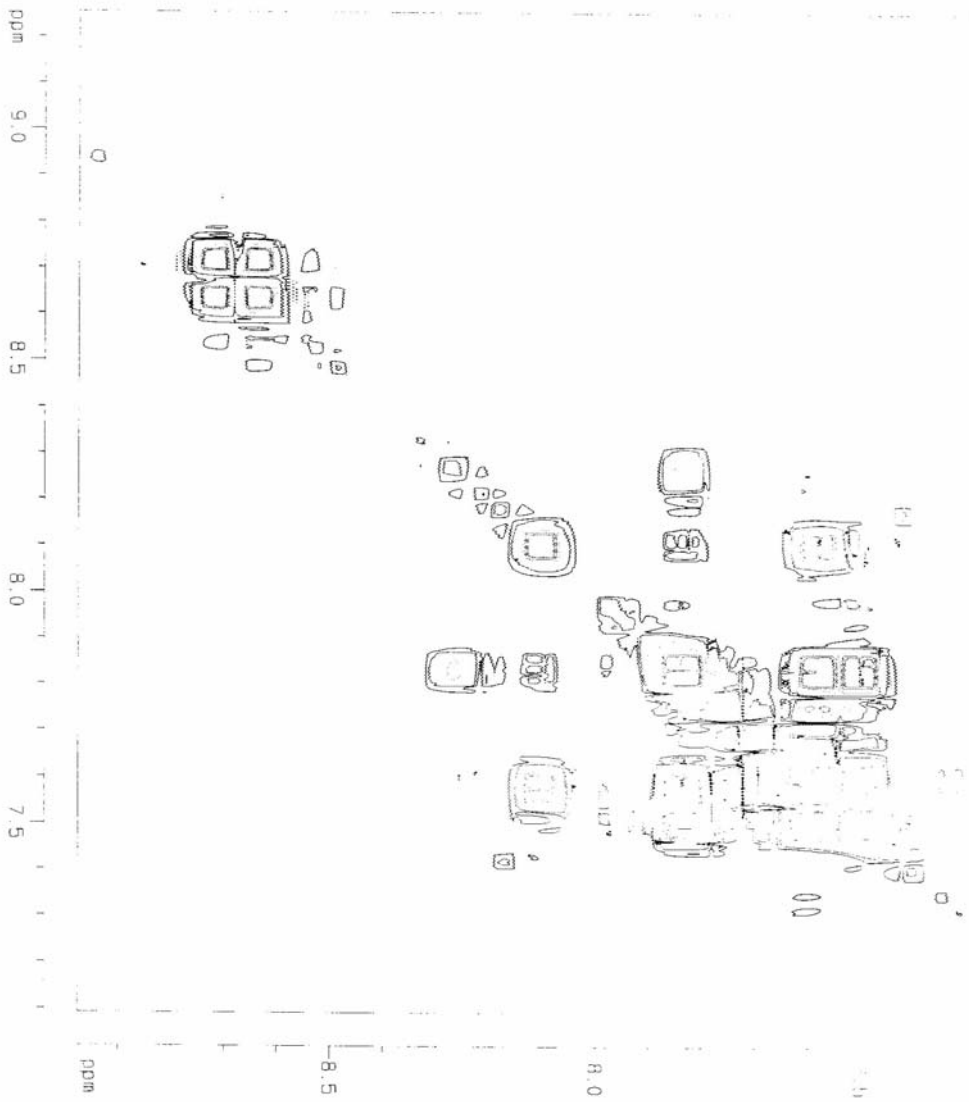
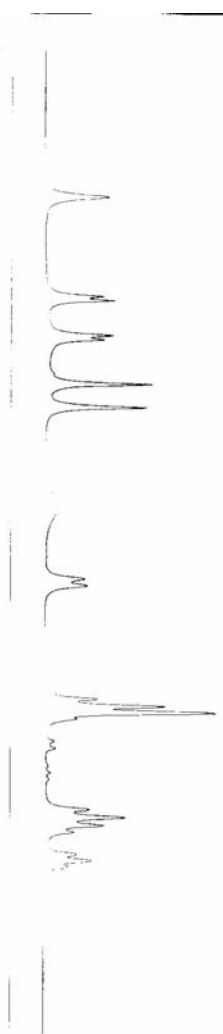
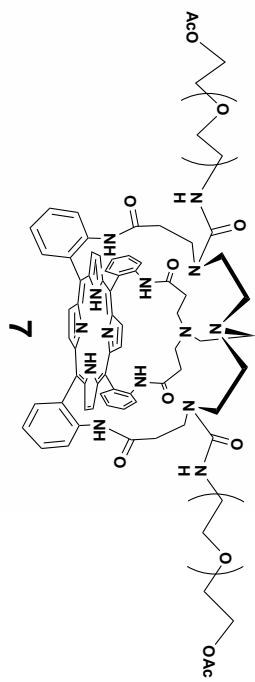




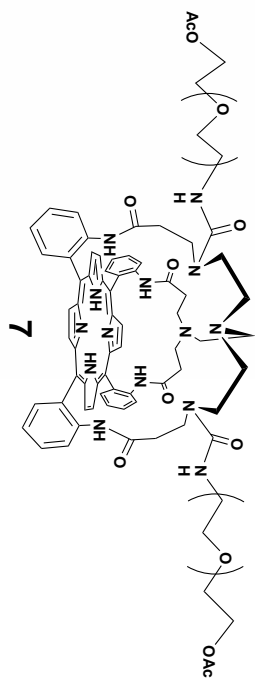
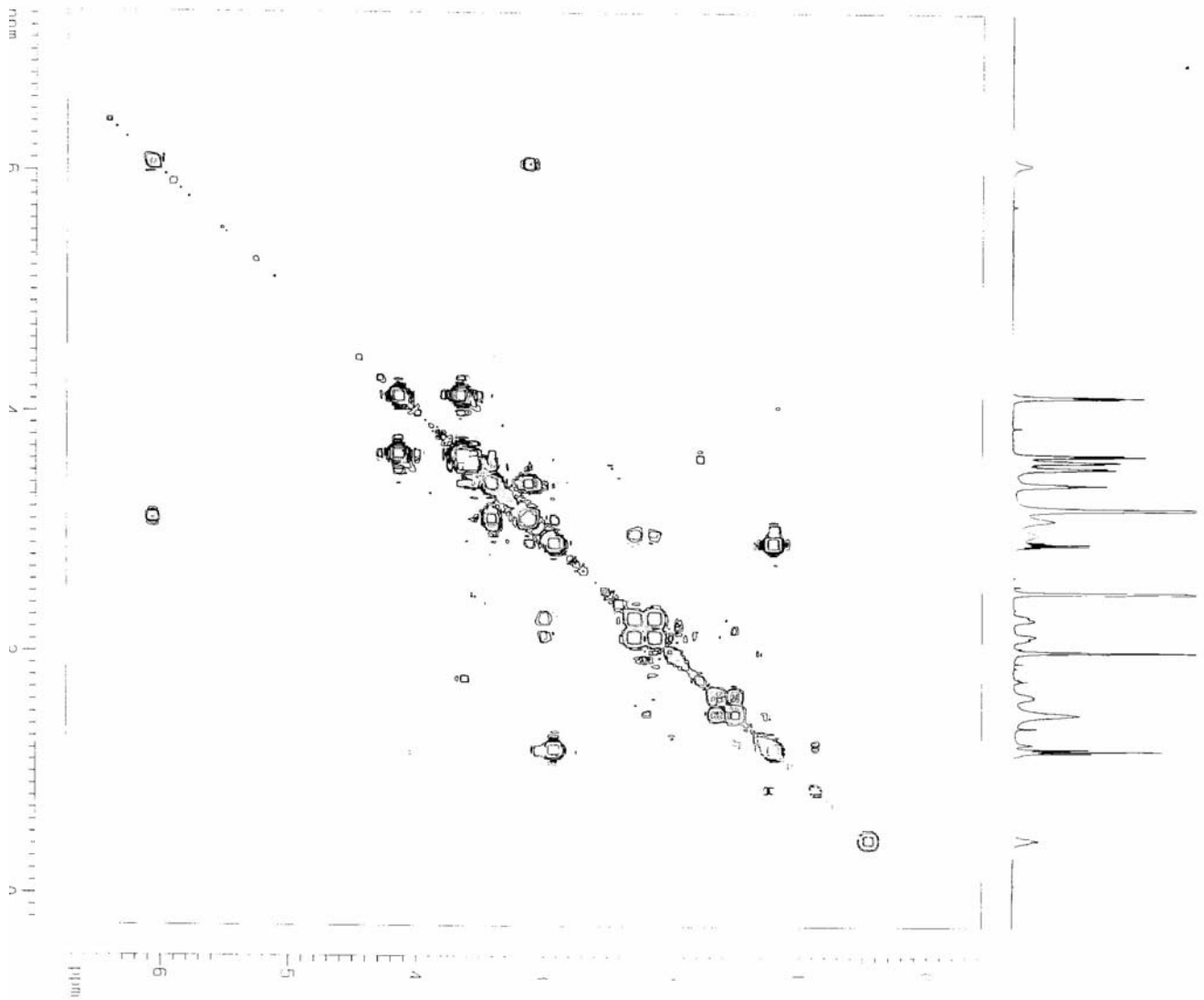


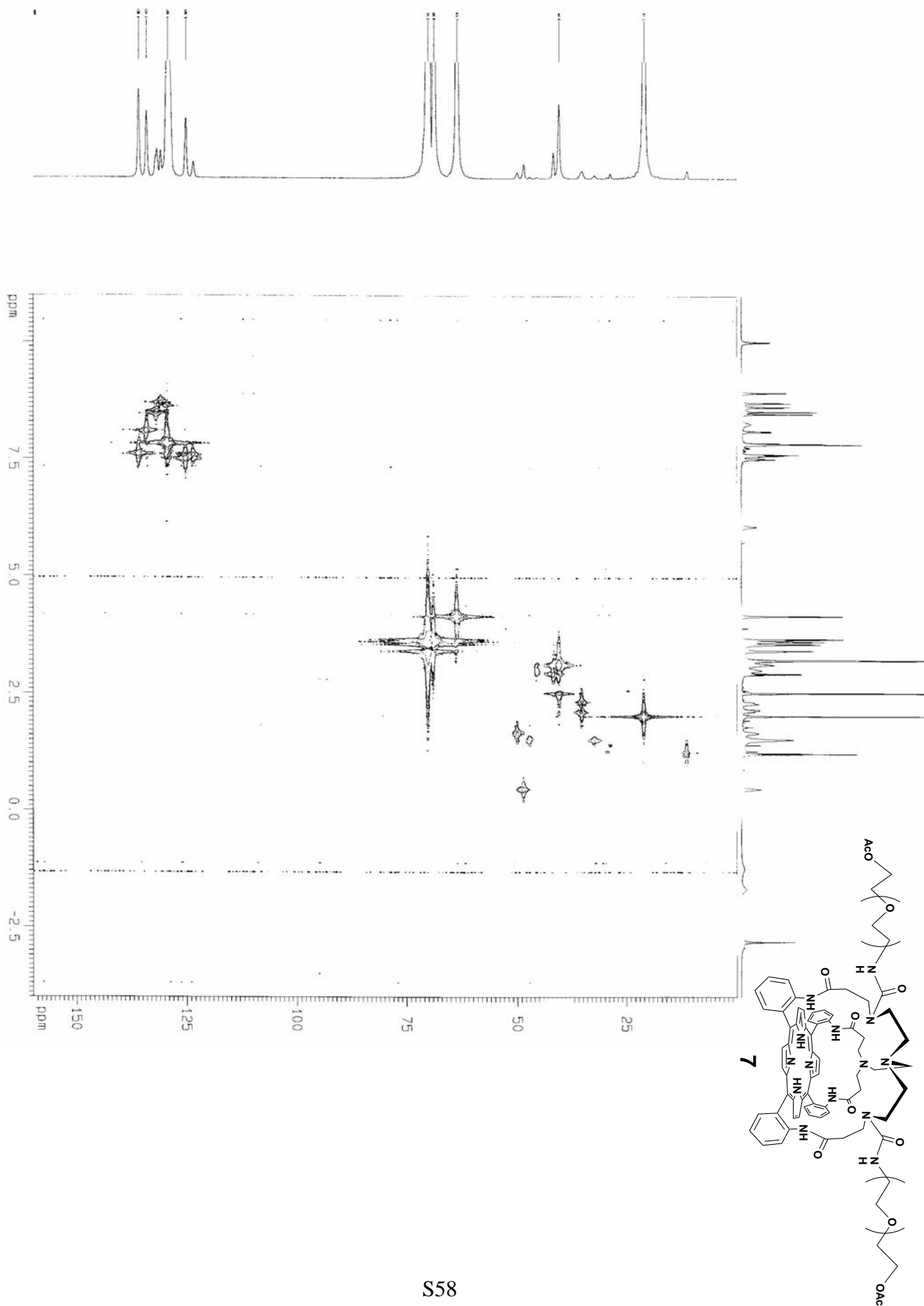


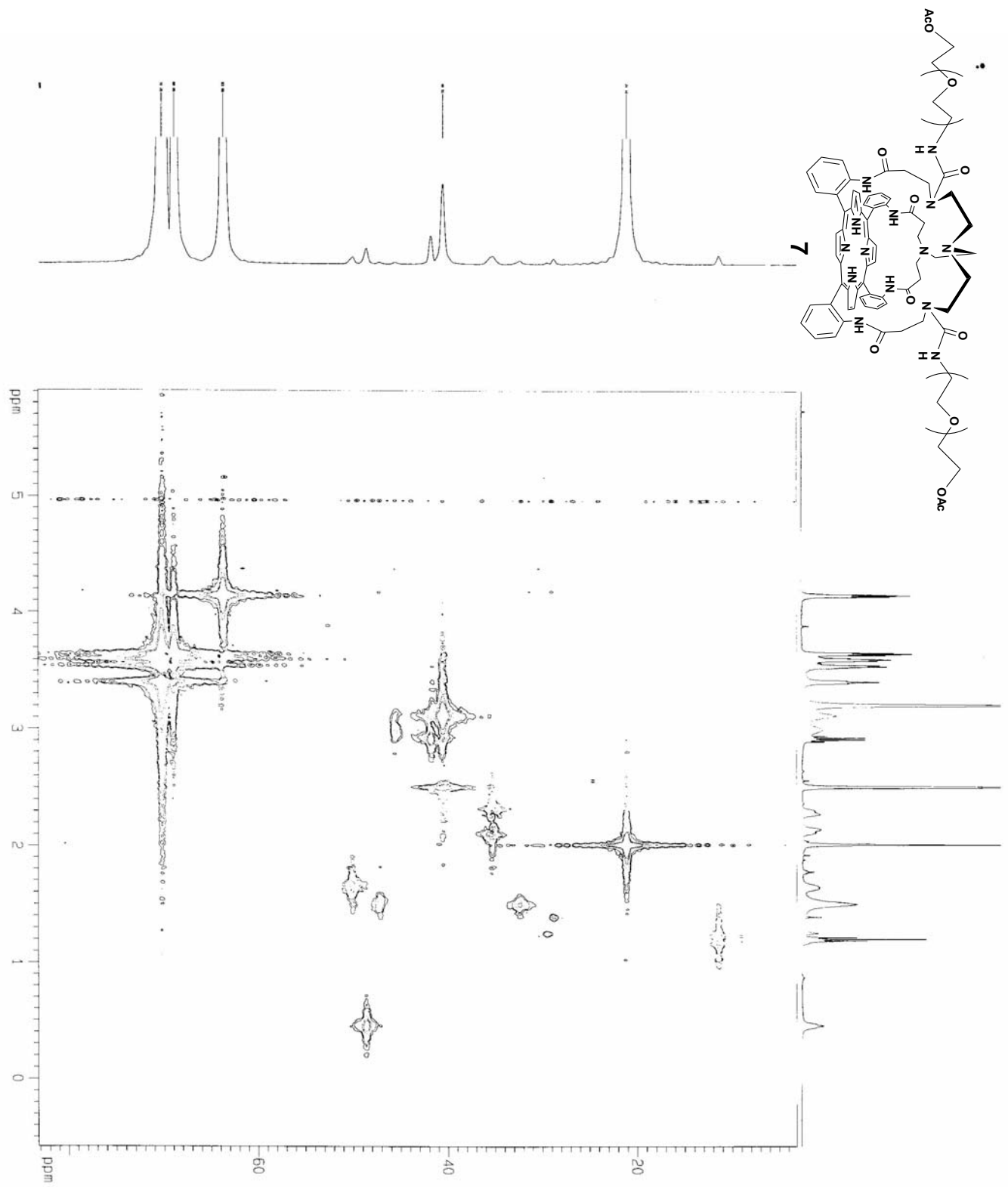




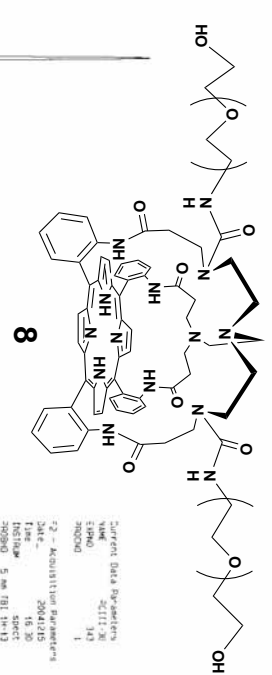
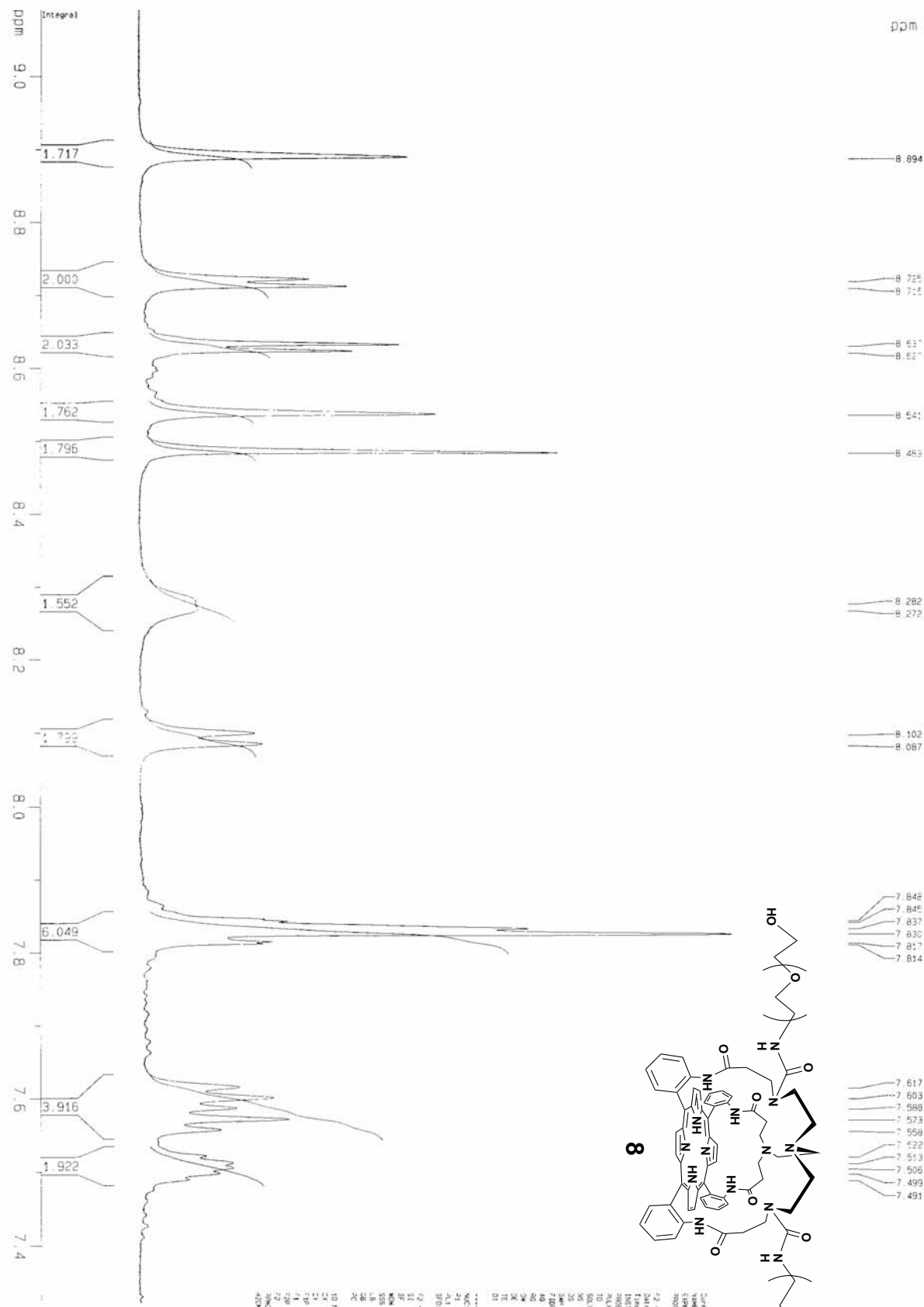








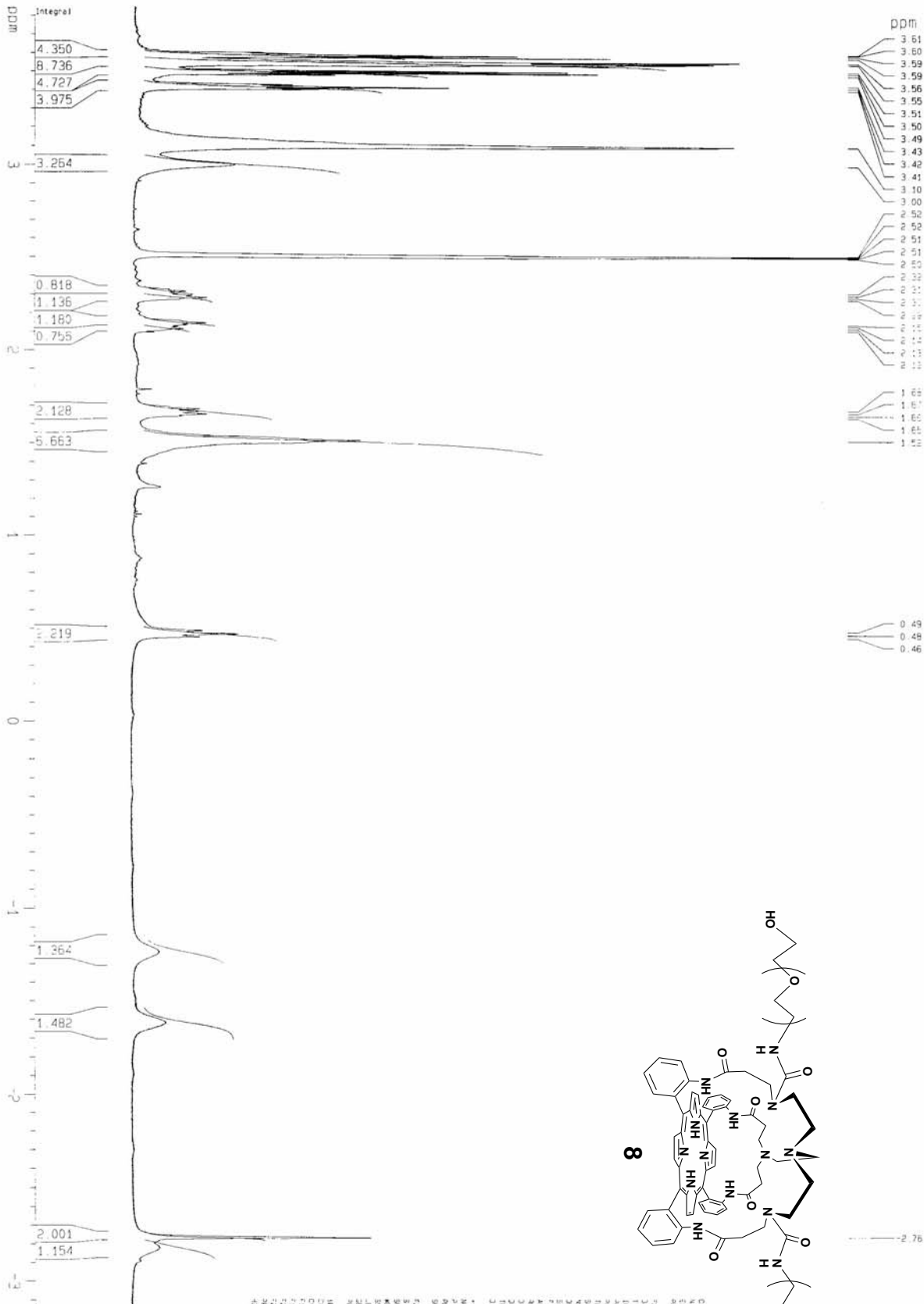




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Name      2-117-01
EXPNO    143
PROCNO   1
Date_     20040415
Time      16.30
INSTRUM  spect
PROBHD   5 mm TBI 1H/1
PULPROG  zgpg30
TD        65536
SOLVENT  DMSO
NS        0
DS        0
SWH       8899.000 Hz
F2RES    0.130218 Hz
AQ        3.86173 sec
RG        161.7
AQ        55.655 usec
DE        231.00 usec
DI        0.50000000 usec
***** CHANNEL f1 *****
NUC1      13C
PC1       8.00 usec
PL1       0.00 dB
PL12      500.1350005 MHz
SFO1      500.1350005 MHz
F2 - Processing parameters
SI        32768
SF        500.1350005 MHz
WDW       EM
SSB       0
LB        0.00 Hz
GB        0
CB        0
PC        1.00
*****
1D 1H NMR Parameters
CY        25.00 usec
CY        20.70 usec
RG        327.68
RG        327.68
FIDRES   7.300 usec
AQ        3.86173 sec
WDW       EM
SSB       0
LB        0.00 Hz
GB        0
CB        0
PC        1.00
*****

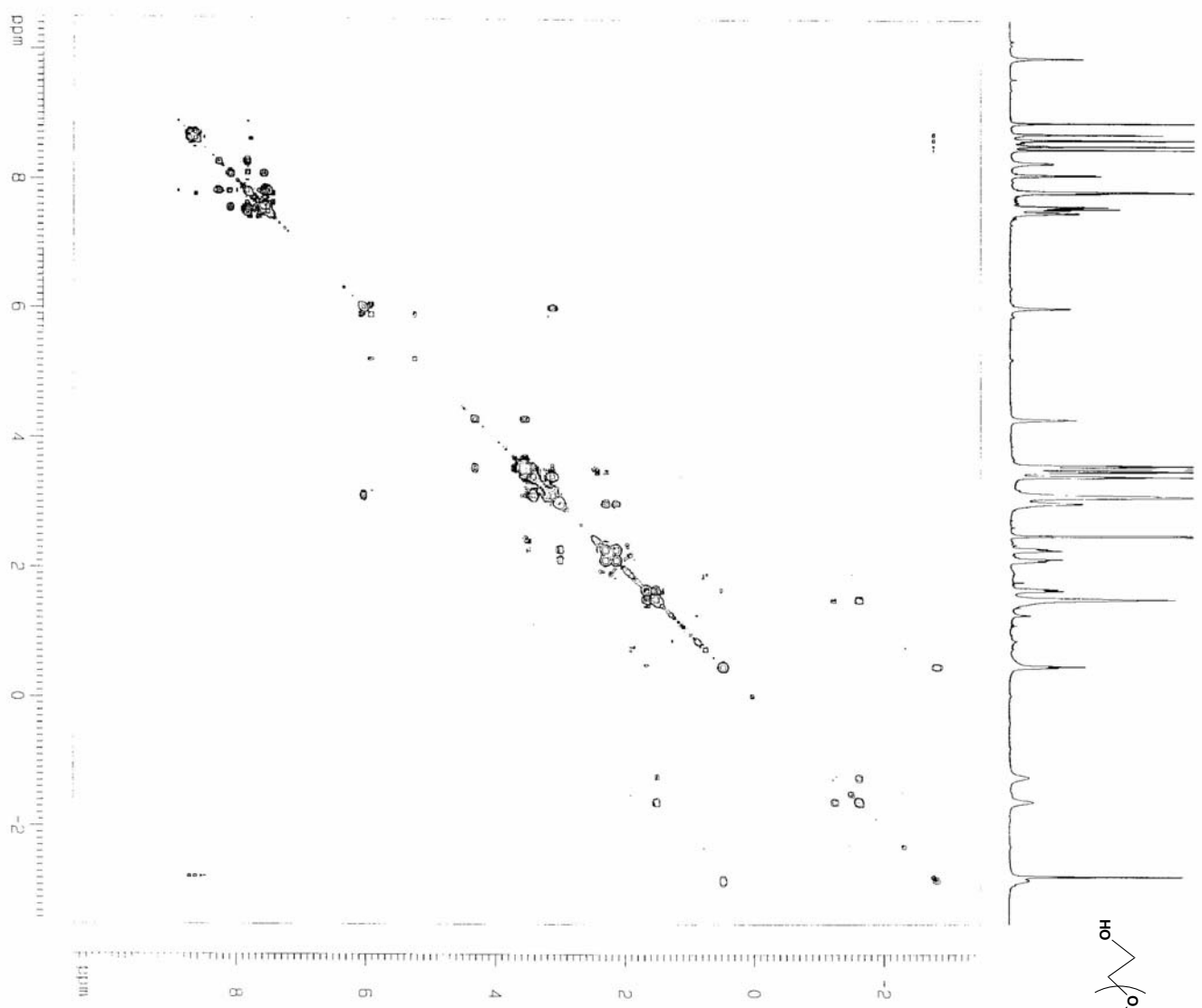
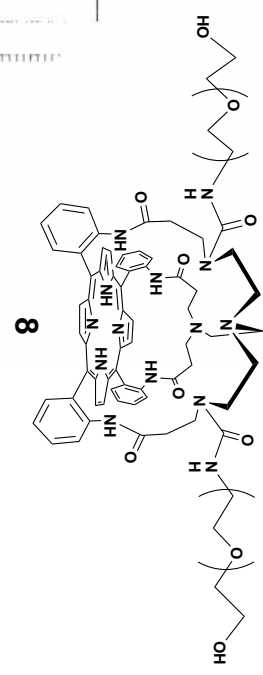
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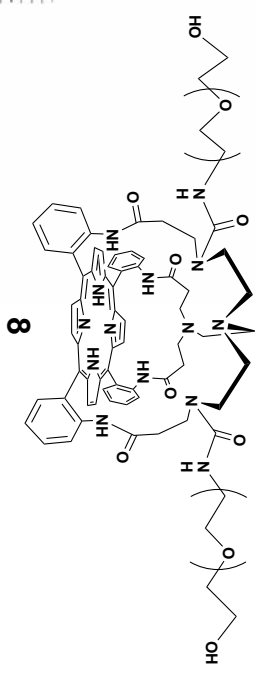
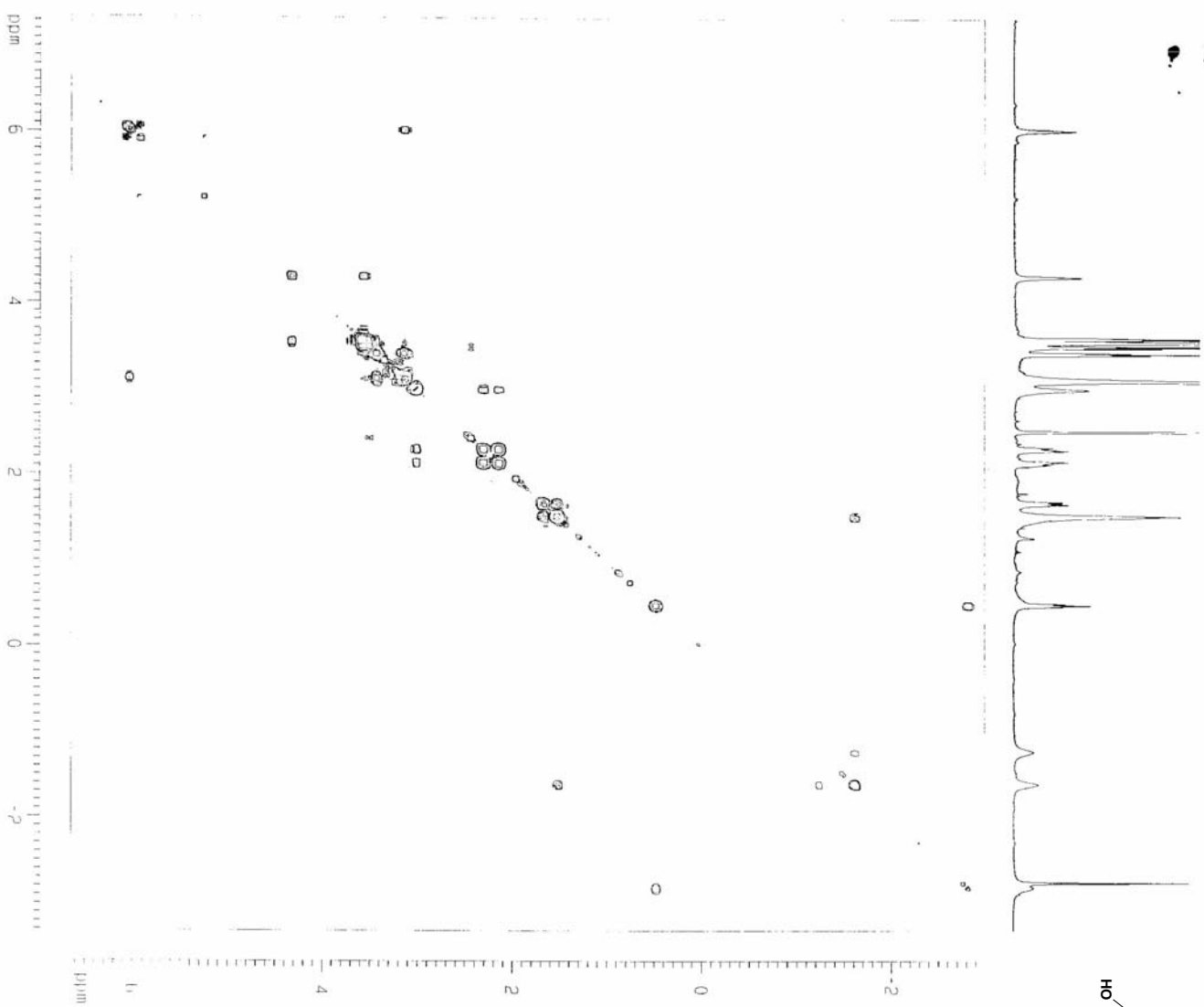


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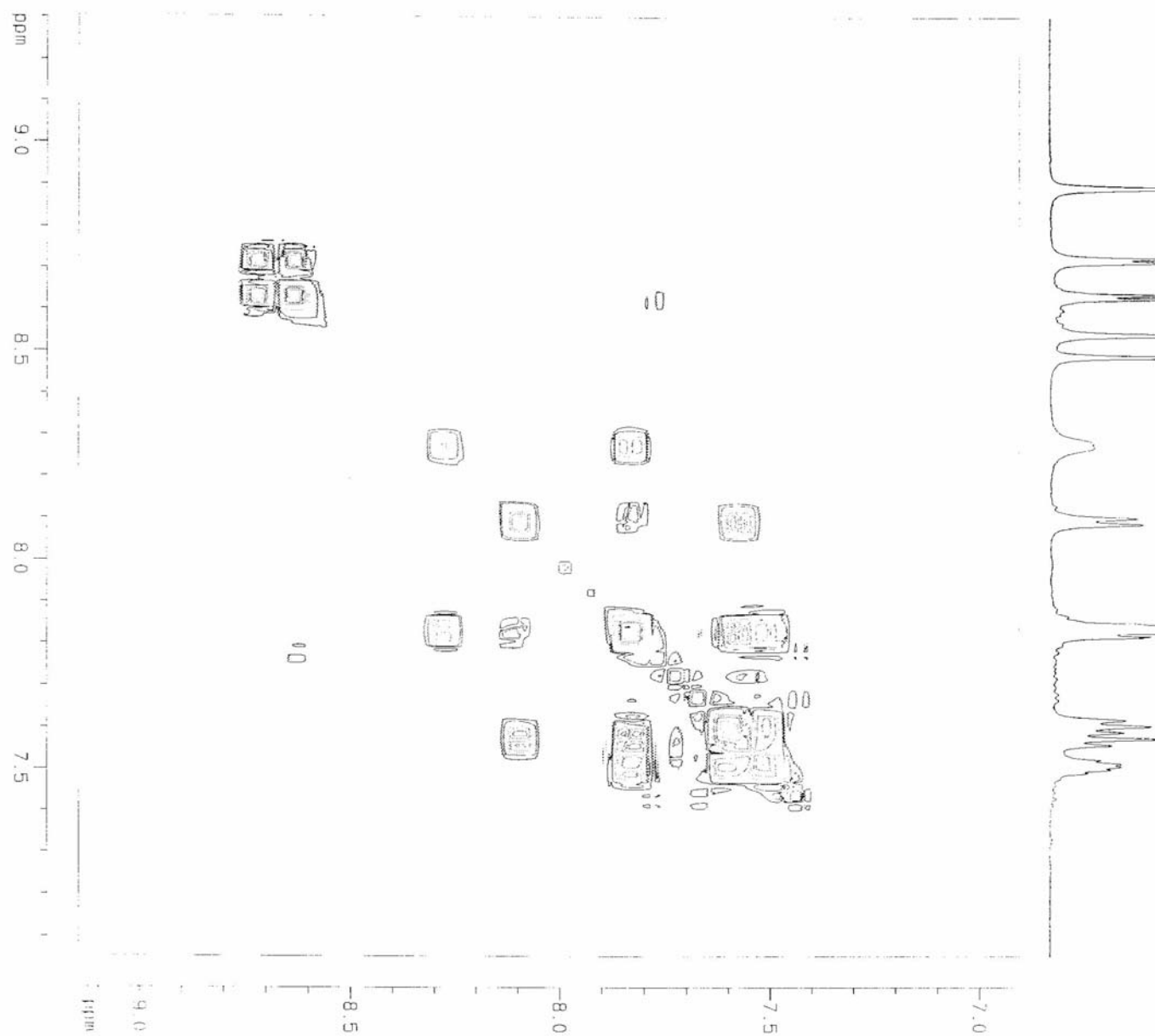
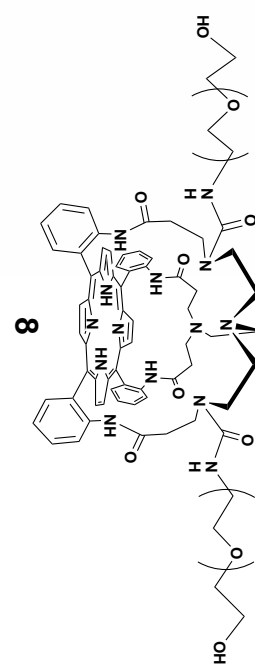
Current Data Parameters
Name: 8
EXPNO: 1
PROCNO: 1
PROCNAME:
Date_ : 20040115
Time: 16.30
INSTRUM: spect
PROBHD: 5 mm QNP 1H/1
PULPROG: zgpg30
TD: 65536
AQ: 0.0263
RG: 327.5
GB: 0
PC: 0
SFO: 800.136 MHz
F2 - Acquisition Parameters
Date_ : 20040115
Time: 16.30
INSTRUM: spect
PROBHD: 5 mm QNP 1H/1
PULPROG: zgpg30
TD: 65536
AQ: 0.0263
RG: 327.5
GB: 0
PC: 0
SFO: 800.136 MHz
F2 - Processing parameters
SI: 32768
SF: 500.136000 MHz
AQ: 0.0263
RG: 327.5
GB: 0
PC: 0
SFO: 800.136 MHz
F2 - Acquisition Parameters
Name: 8
EXPNO: 1
PROCNO: 1
PROCNAME:
Date_ : 20040115
Time: 16.30
INSTRUM: spect
PROBHD: 5 mm QNP 1H/1
PULPROG: zgpg30
TD: 65536
AQ: 0.0263
RG: 327.5
GB: 0
PC: 0
SFO: 800.136 MHz
F2 - Processing parameters
SI: 32768
SF: 500.136000 MHz
AQ: 0.0263
RG: 327.5
GB: 0
PC: 0
SFO: 800.136 MHz

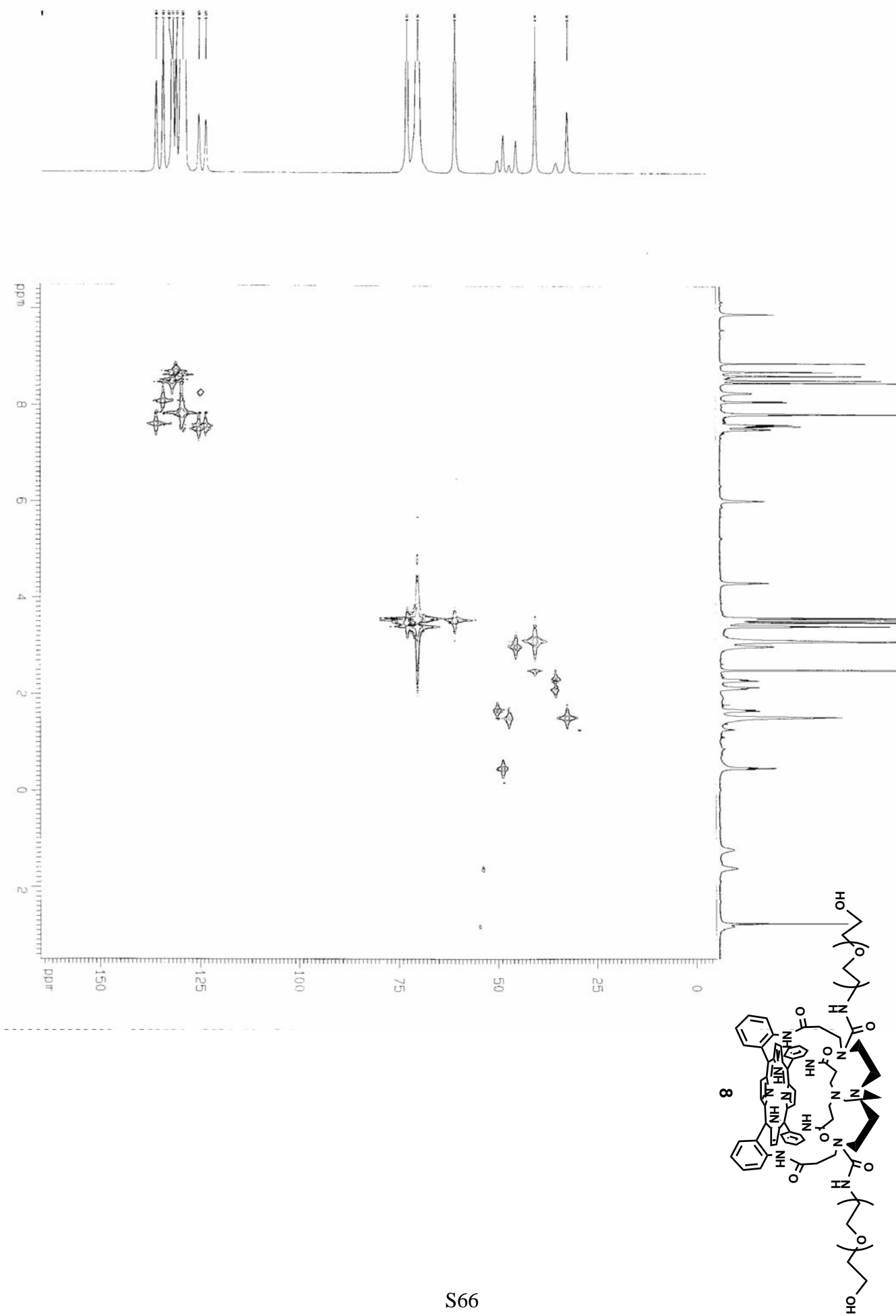
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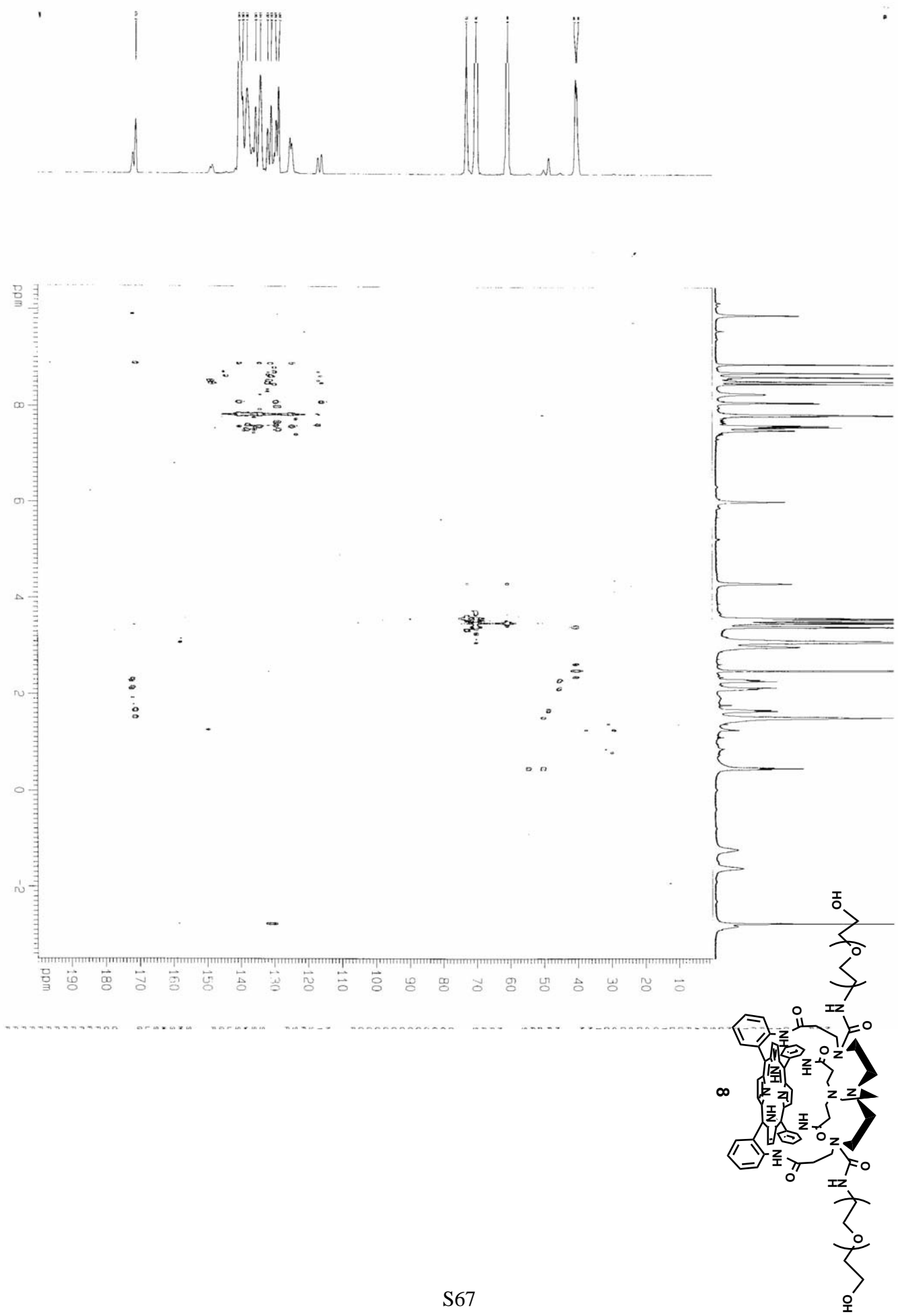


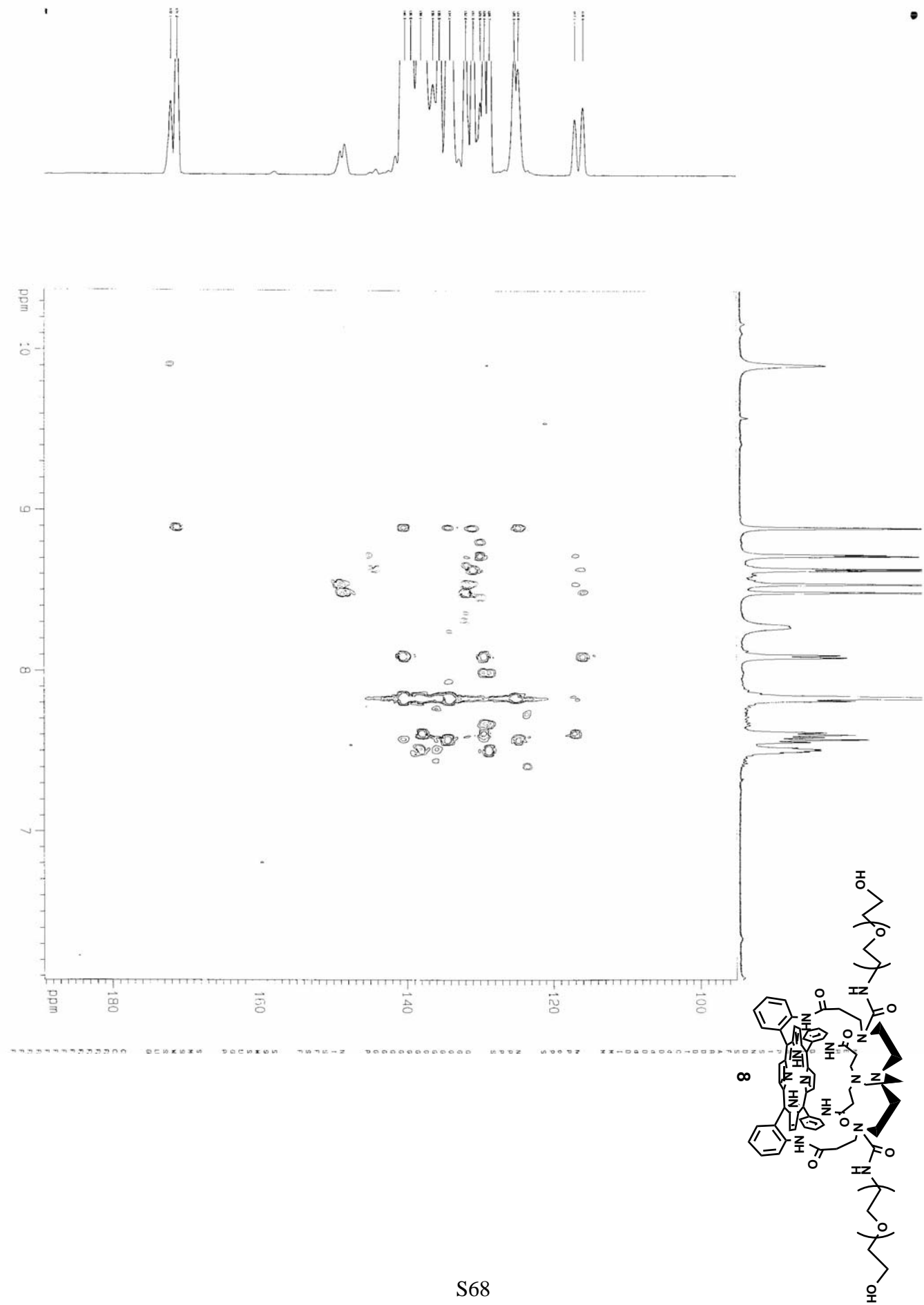


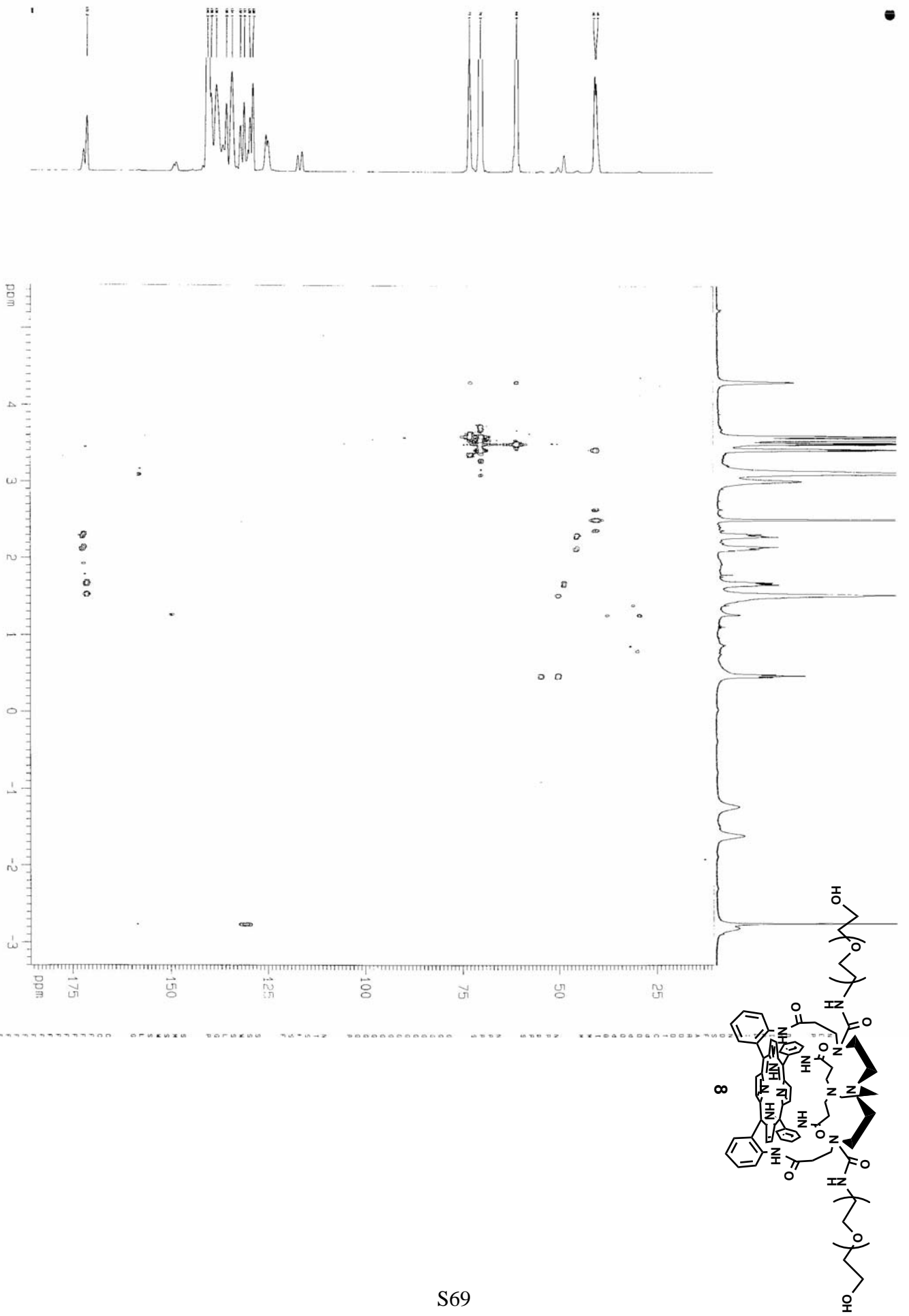




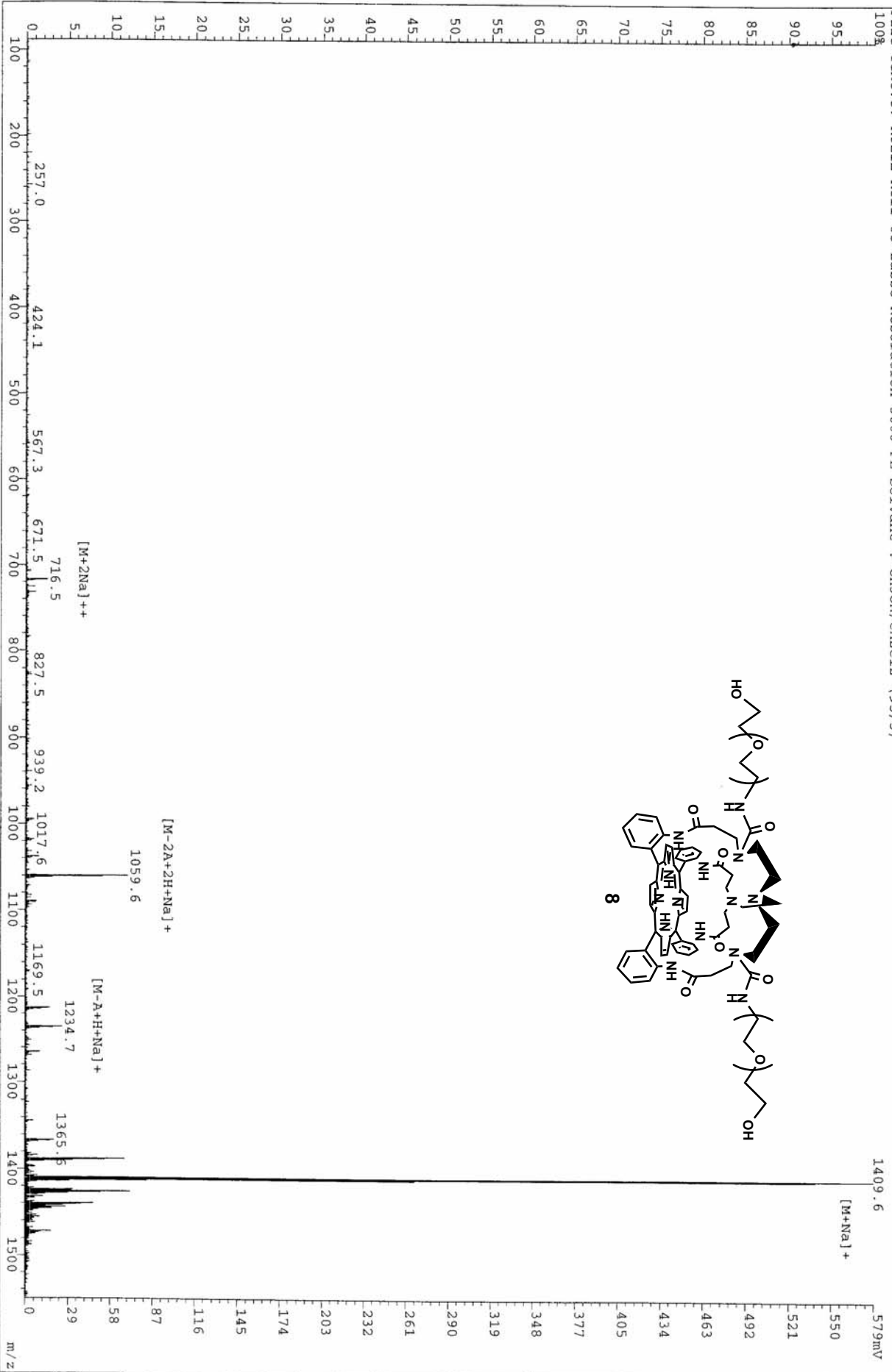
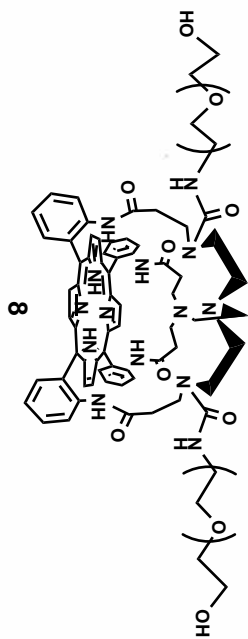




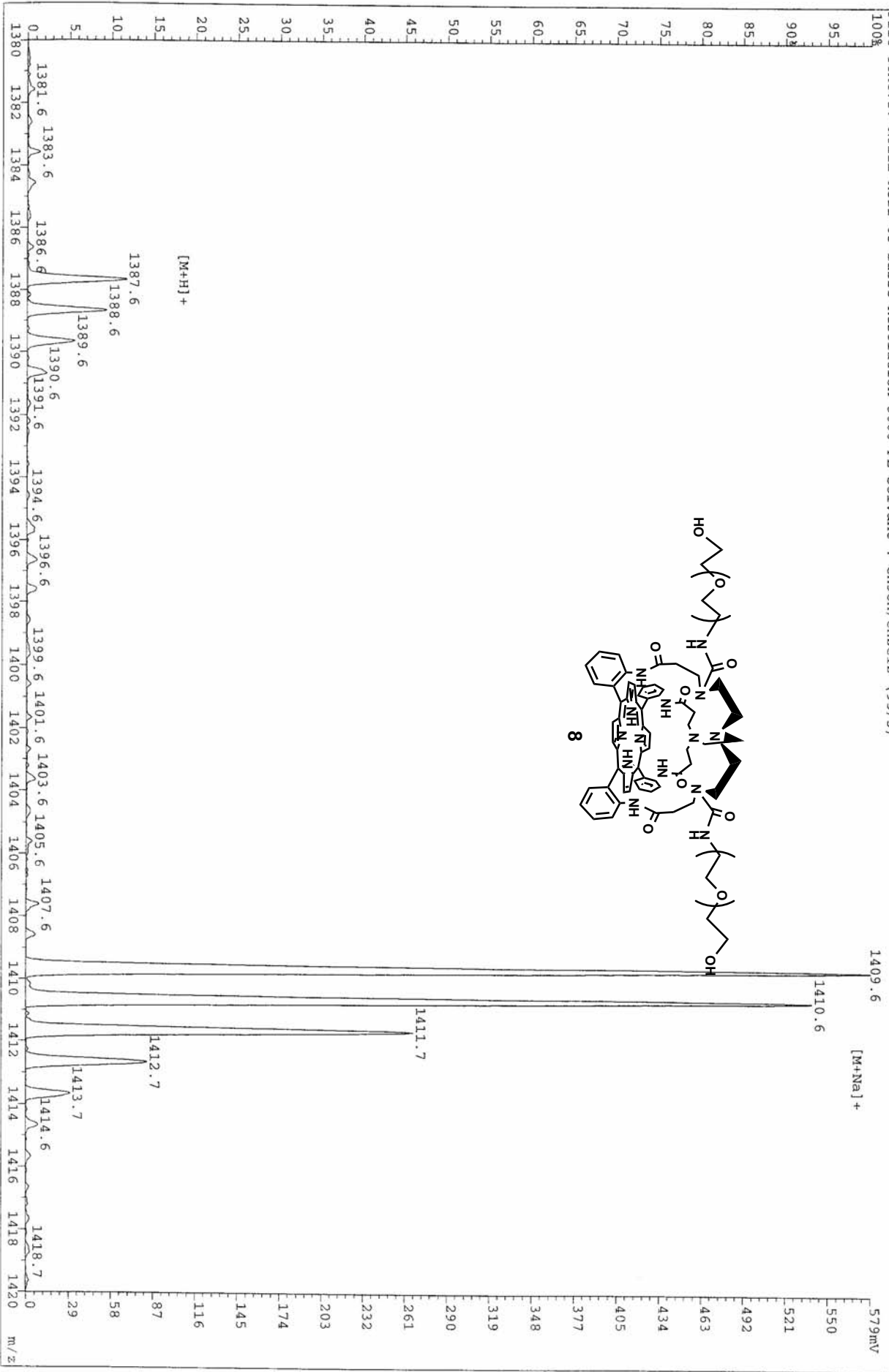
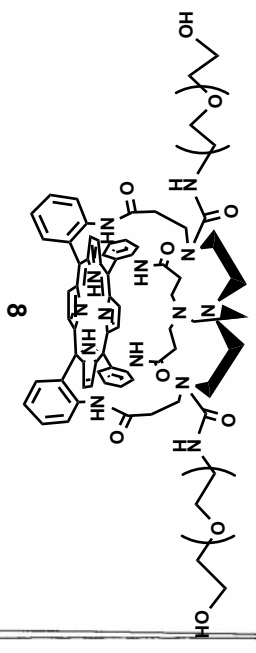




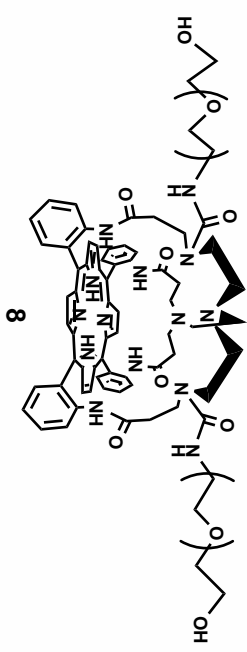
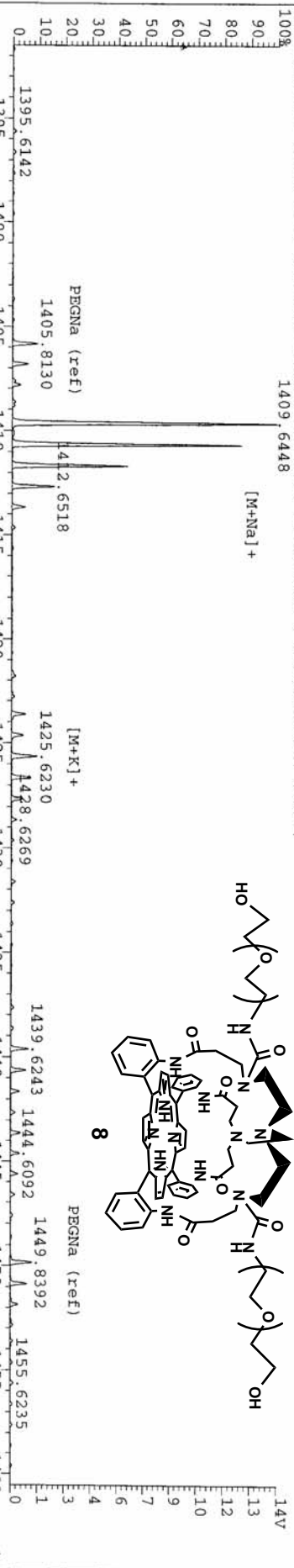
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 Zabpector ES+ Magnet BPI:1042004 TIC:70232792 Noise:11  
 File Text:C. RUZIE KCI-05 Basse Resolution 3000 FL Solvant : CH3OH/CH2Cl2 (95/5)



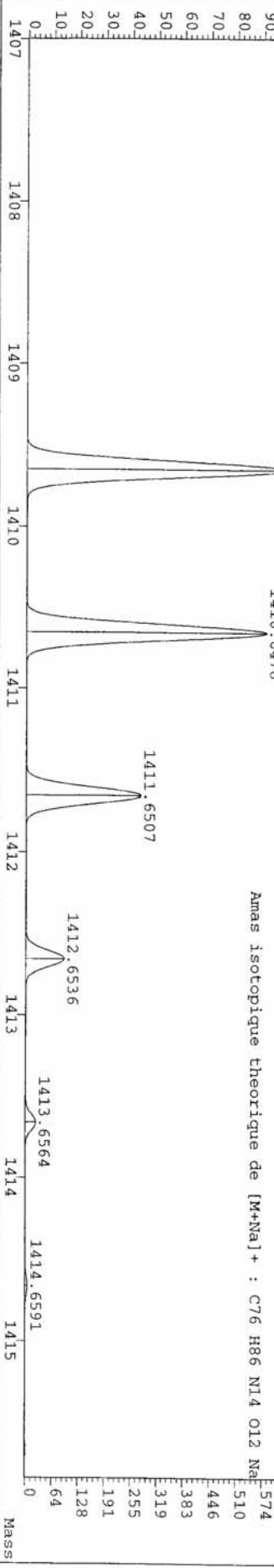
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 ZabspectOP ES+ Magnet BpI:1042004 TIC:70232792 Noise:11  
 File Text:C. RUIZIE RCU1-05 Basse Resolution 3000 FL Solvant : CH3OH/CH2Cl2 (95/5)



File:ESI\_6881HRV Ident:5\_19 SMO(2,5) BSUB(128,15,-3.0) PKD(5,3,5,0.01%,22520.0,0.00%,T,F) Acq:16-SEP-2003 12:21:04 +7:00 Cal:ESI\_6881HRV  
 ZabsPECTOP ES+ Voltage BpI:120088576 TIC:5708548096 Noise:5630  
 File Text:C. RUIZIE RCI1-05 Haute Resolution 6000 FL Solvant : CH3OH/CH2Cl2 (95/5)

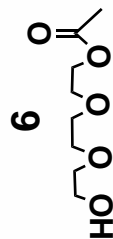


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 ZabsPECTOP ES+ Voltage BpI:120088576 TIC:5708548096 Noise:5630  
 File Text:C. RUIZIE RCI1-05 Haute Resolution 6000 FL Solvant : CH3OH/CH2Cl2 (95/5)



Amas isotopique theorique de [M+Na]<sup>+</sup> : C76 H86 N14 O12 Na





4.164  
4.163  
4.161  
4.160  
4.158

3.524  
3.523  
3.522  
3.521  
3.520  
3.519  
3.518  
3.517  
3.516  
3.499

2.915

2.004  
1.993

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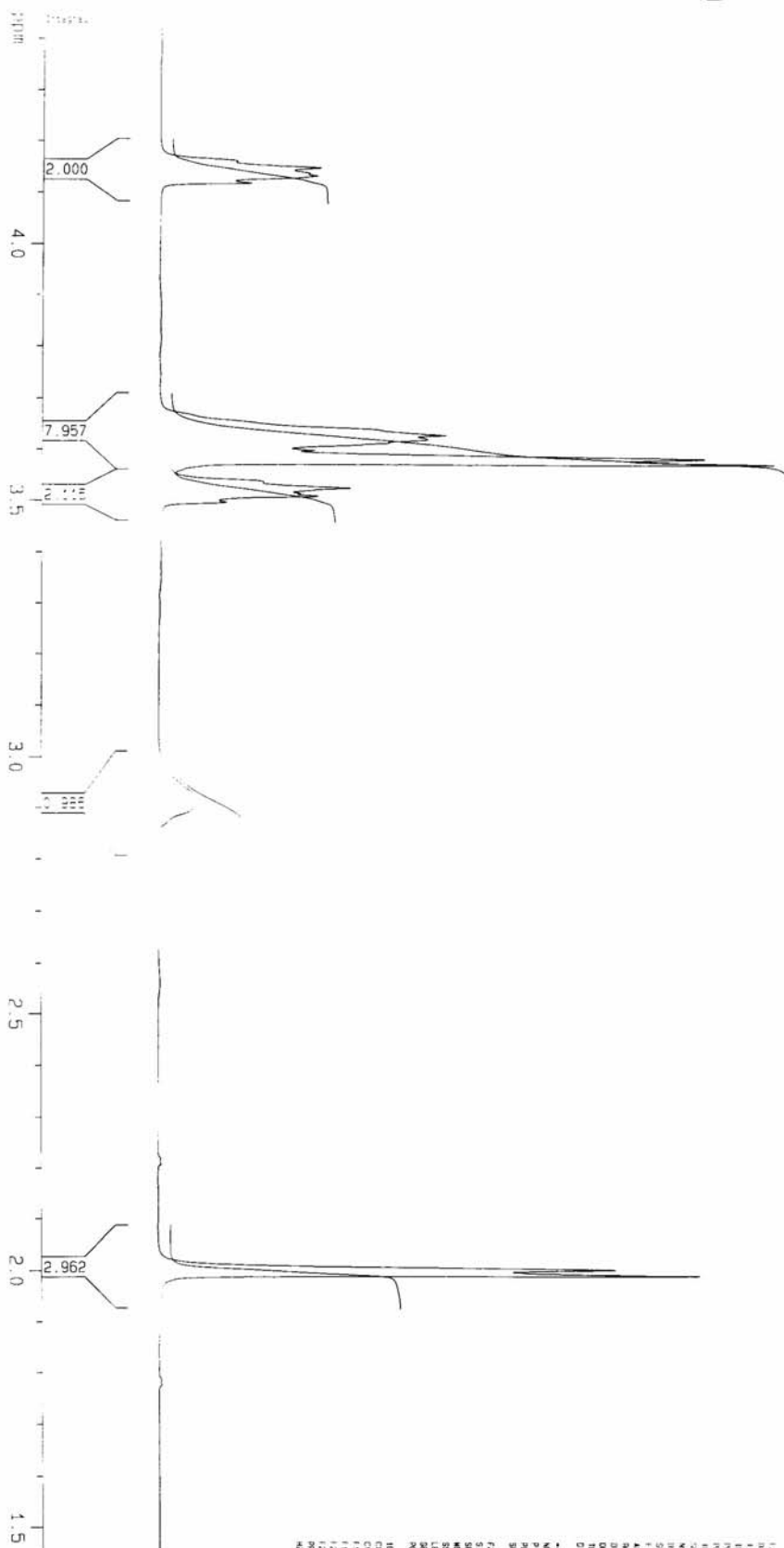
Current Data Parameters
NAME      DCI-98
EXPNO     2
PROCNO    1

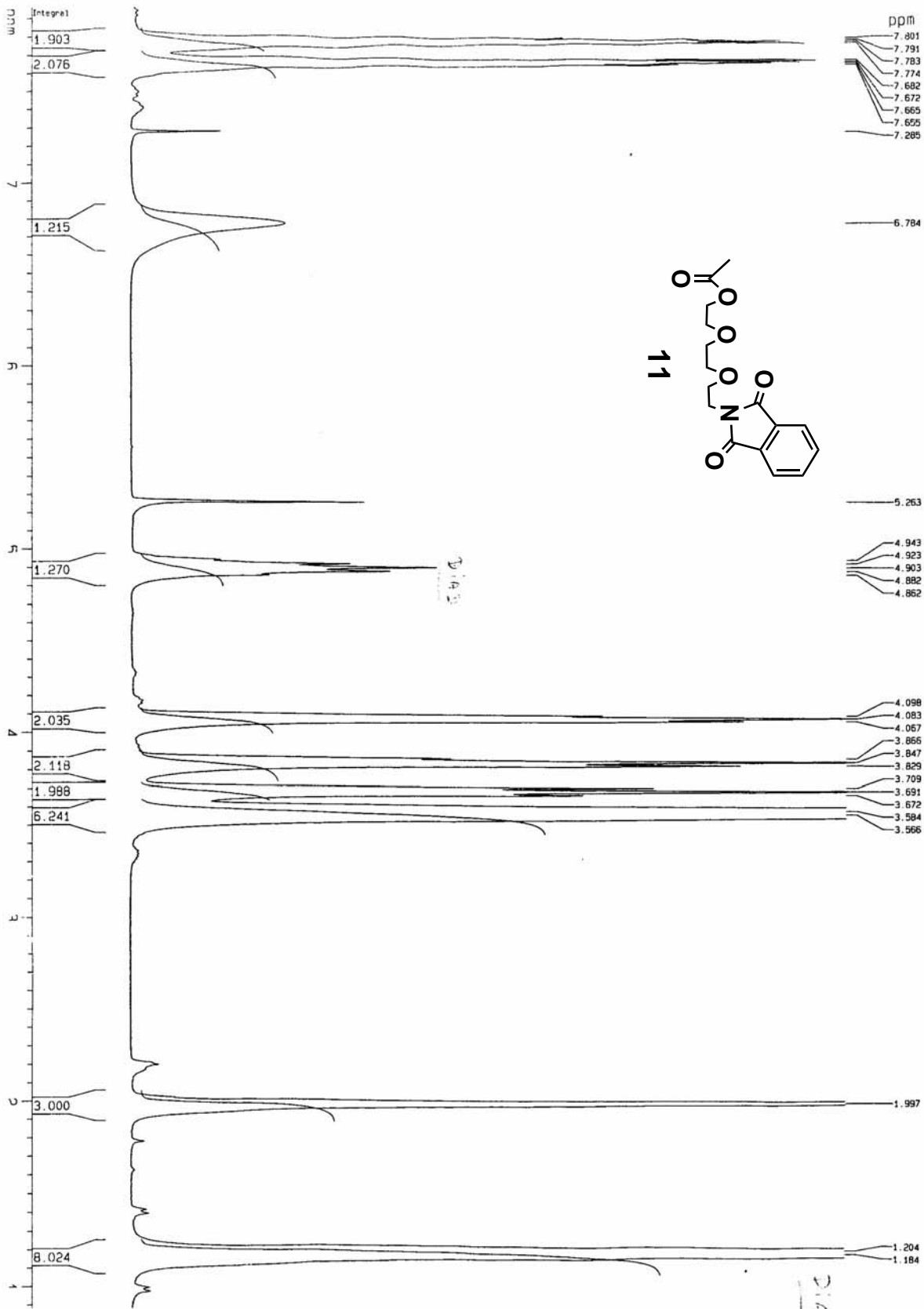
F2 - Acquisition Parameters
Date_     20030927
Time      13.24
INSTRUM   spect
PROBHD    5 mm HHH HT H1
PULPROG   zgpg30
RG         327.68
AQ         0.00146
RG2        327.68
AQ2        0.00146
SFO       300
WDW         EM
SSB         0
LB          0
GB          0
PC         1.00

===== CHANNEL f1 =====
NUC1       13
P1         8.50 usec
PL1        0.00 dB
SFO1       300.1310005 MHz

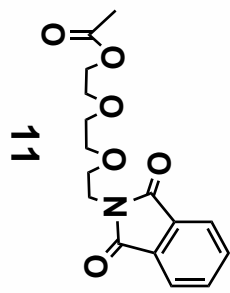
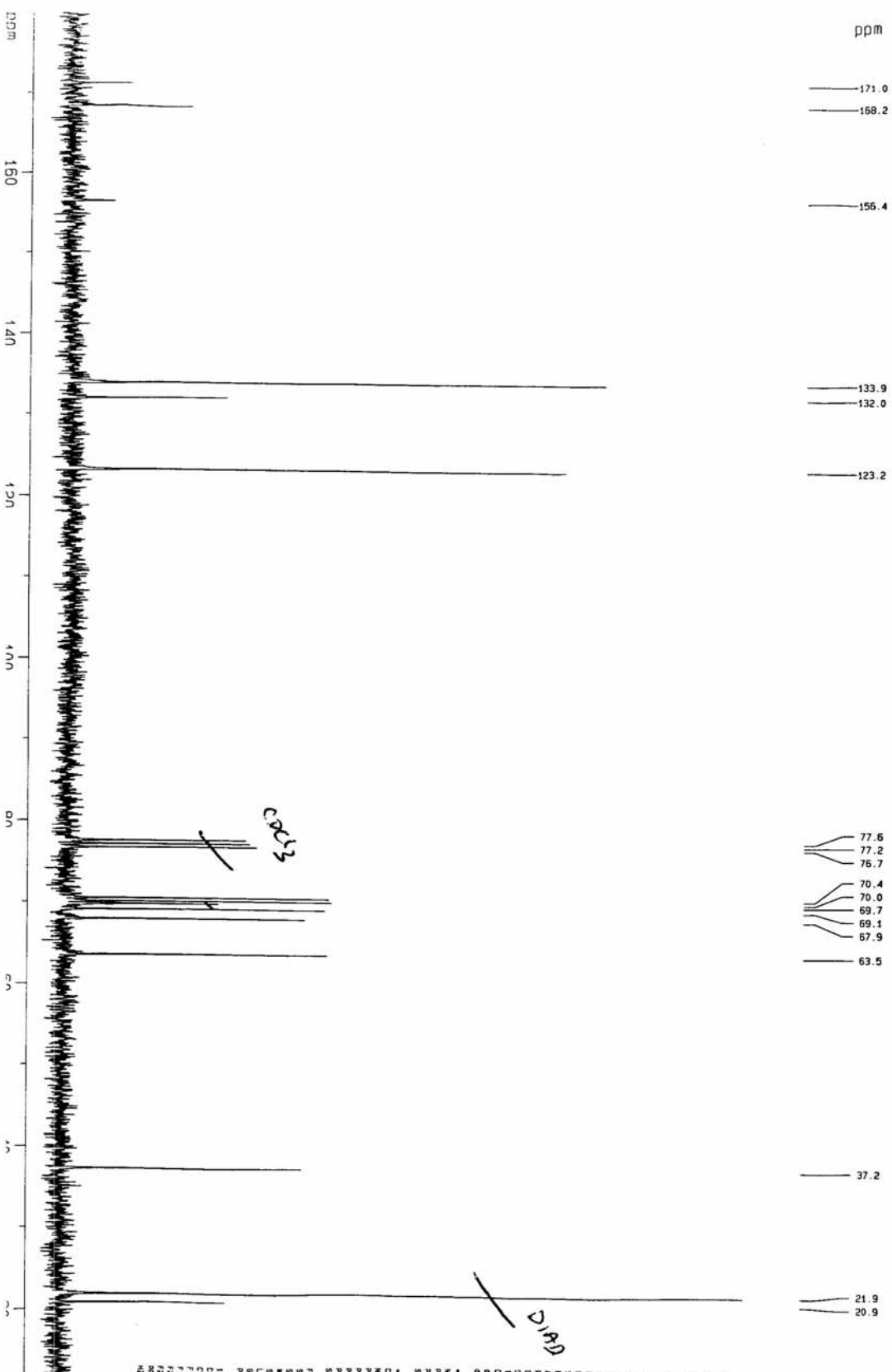
F2 - Processing parameters
SI         300.1300000 MHz
SF         300.1300000 MHz
WDW         EM
SSB         0
LB          0.00 Hz
GB          0
PC         1.00

ID: Name of parameters
CP         29.00 cm
CY         10.00 cm
CX         10.00 cm
FL1        135.28 Hz
FL2        1.448 ppm
F2 - 2D NMR
PC12000 30.00000 Hz/cm
  
```





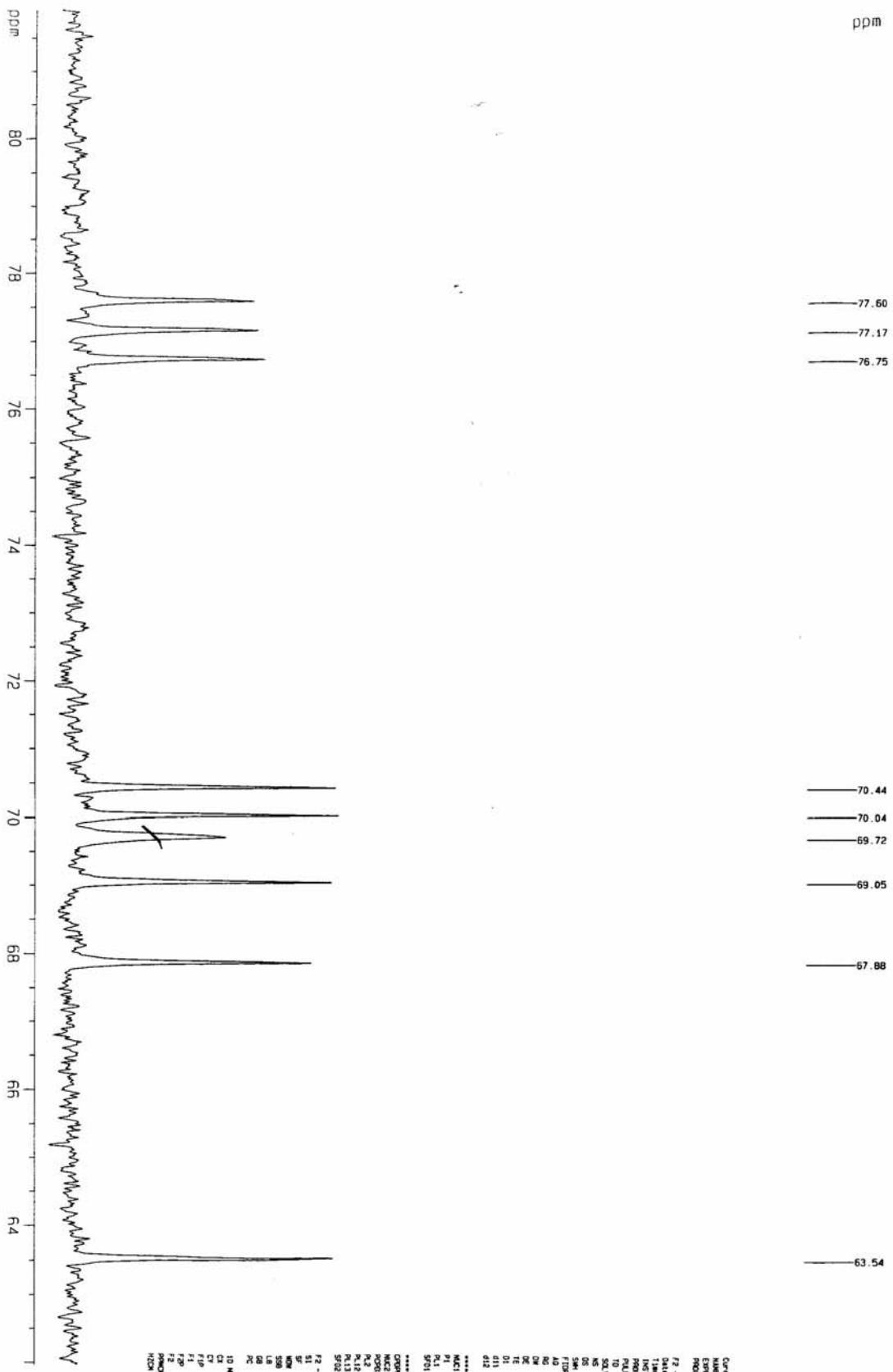
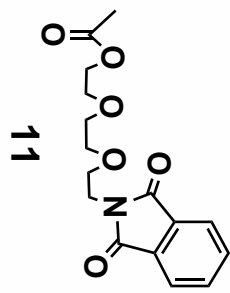
Current Data 1  
NAME  
SMNO  
PRODS  
F2 - Acetamin  
DATE  
INSTRUM  
PROBND 5 mm  
PULPROG  
TD  
SOLVENT  
NS  
DS  
F1RES  
AQ  
RG  
OR  
DI  
TE  
OI  
\*\*\*\*\*  
NAME  
P1  
P1.1  
SFO1  
F2 - Acetamin  
SI  
SM  
SBR  
UB  
OR  
RG  
CX  
CX  
TO WMT D181 SU  
CX  
CX  
F1P  
F1  
F2P  
F2  
PNAME  
NCDM



```

Current Data Parameters
NAME      DCI-44F2
PROCNO   1
PULPROG  zgpg30
Date_    20030906
TIME     11.42
INSTRUM  spect
PROBHD   5 mm TCI 13C Z
PULPROG  zgpg30
AQ       4.0000000
SI       32768
SF       125.7611635
WDW       EM
SS       2.00
GB       0
PC       1.40
----- CHANNEL f1 -----
NUC1      13C
P1        17.00 usec
PL1       0 dB
SFO1      75.46302 MHz
----- CHANNEL f2 -----
NAME      waltz16
NUC2      1H
PCPD2     60.00 usec
PC2       3.00 dB
PC12      3.00 dB
PC13      3.00 dB
SFO2      500.0000000 MHz
F3 - Pre-cycling parameters:
SI        32768
SF        75.4631760 MHz
MCH       EM
LB        2.00 Hz
GB        0
PC        1.40
10 MHz 210: parameters
CX        25.00 cm
CY        12.17 cm
CZ        179.658 cm
F1        1.50
F2        11.522 ppm
F3        6.7346 ppm
MCH       500.0001760 MHz

```



```

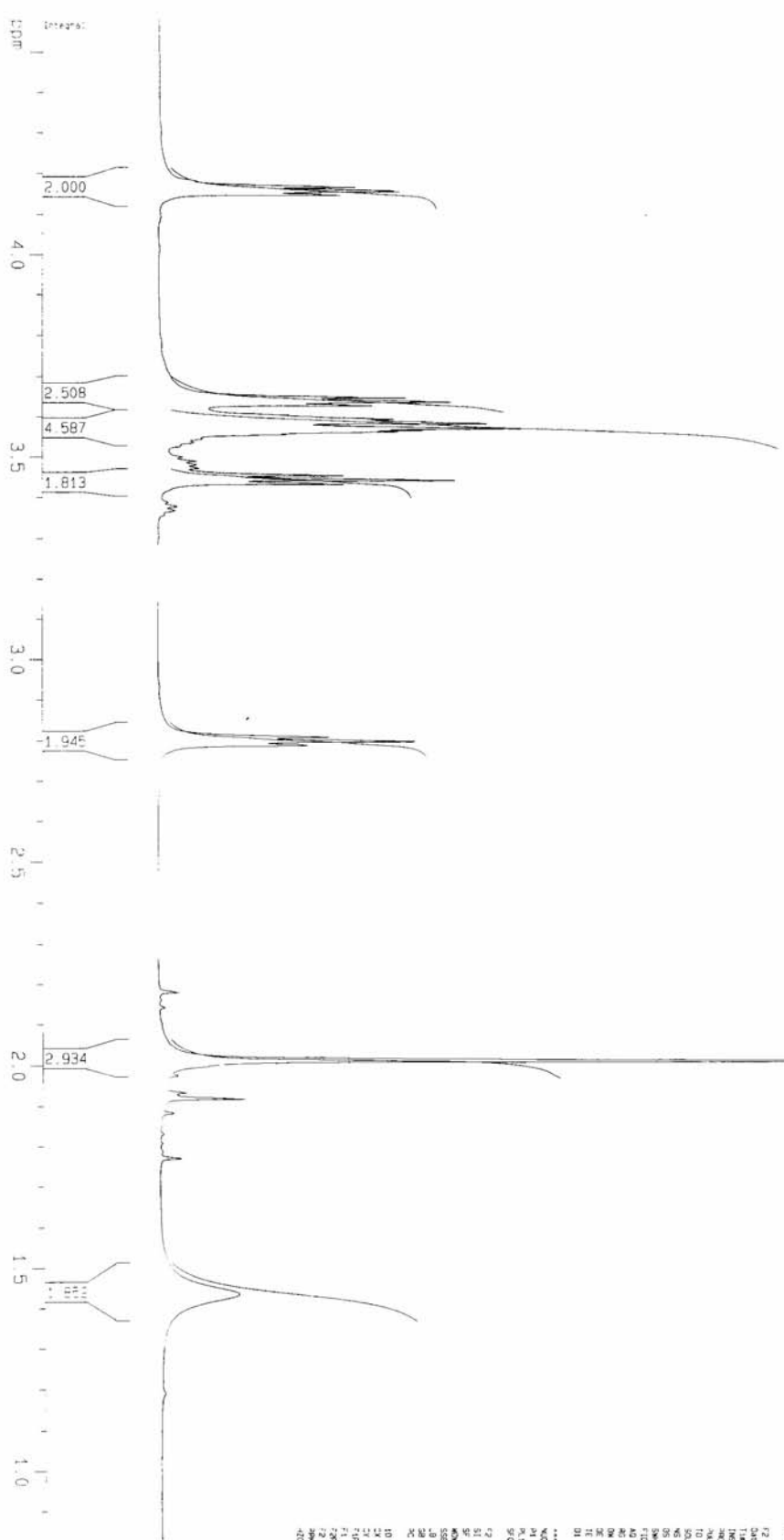
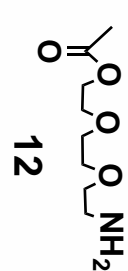
Current Data Parameters
NAME      RCI-14172
EXPNO    8
PROCNO   1
F2 - Acquisition Parameters
Date_    20030506
Time     13:42
INSTRUM  spect
PROBHD   5 mm BB1-13C-7
PULPROG  zgpg30
TD        32768
SOLVENT  CDCl3
NS       512
DS       2
SWH       15960.000 Hz
FIDRES    0.000113 Hz
AQ        0.00000000 sec
RG         3648.1
AQ        0.00000000 sec
OR        25.000 usec
DE        1.00000000 sec
DI        0.111
D1        2.00000000 sec
d12       0.00000000 sec
***** CHANNEL f1 *****
NUC1      13C
P1        17.00 usec
PL1       0.00 dB
SFO1     75.468307 MHz
***** CHANNEL f2 *****
NAME      waltz16
NUC2      1H
P2        80.00 usec
PL2       0.00 dB
PCPRG2   3.00 usec
PCPRG2   0.00 dB
PCPRG2   28.50 dB
PCPRG2   28.50 dB
SFO2     300.0813004 MHz
F2 - Processing parameters
SI        32768
SF        75.4681500 MHz
WDW       EM
SSB       0
LB        2.00 Hz
GB        0
PC        1.40
F2 - 1D NMR data parameters
CX        25.00 cm
CY        12.73 cm
CZ        61.508 cm
F1        61.511 Hz
F2        61.862 cm
F3        46.7536 Hz
PACMON    0.79885 ppm/c
NCDM      01.22925 Hz/cm

```

4.250  
4.230  
4.184  
4.171  
4.164  
4.161  
4.154  
4.152  
4.096  
3.791  
3.781  
3.772  
3.652  
3.644  
3.642  
3.535  
3.632  
3.608  
3.599  
3.596  
3.593  
3.589  
3.577  
3.571  
3.568  
3.566  
3.560  
3.549  
3.545  
3.542  
3.539  
3.528  
3.511  
3.451  
3.453  
3.461  
3.473  
3.457  
3.447  
3.438  
3.436  
3.389  
3.378  
3.368  
3.304  
3.293  
2.933  
2.922  
2.811  
2.801  
2.790

2.180  
2.153  
2.141  
2.133  
2.115  
2.114  
2.111  
2.108  
2.106  
2.104  
1.943  
1.933  
1.927  
1.919  
1.882  
1.881  
1.830  
1.806  
1.786  
1.769

1.436  
1.219  
1.190



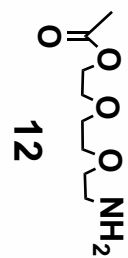
Current Data Parameters  
NAME: 021150  
EXPNO: 288  
PROCNO: 1

F2 - Acquisition Parameters  
Date\_ : 20070528  
Time: 9:14  
Time2: 9:14  
INSTRUM: spect  
PROBHD: 5 mm BBI  
NUC1: 13C  
PULPROG: zgpg30  
TD: 32768  
SOLVENT: CDCl3  
AQ: 3.00  
RG: 320  
SI: 0  
SM: 10000.000 Hz  
FIDRES: 0.00078 Hz  
AQRES: 1.000000 Hz  
AQ: 49.3 Hz  
NUC2: 13C  
NUC2A: 13C  
NUC2F1: 101.325 kHz  
NUC2F2: 101.325 kHz  
NUC2F3: 101.325 kHz  
NUC2F4: 101.325 kHz  
NUC2F5: 101.325 kHz  
NUC2F6: 101.325 kHz  
NUC2F7: 101.325 kHz  
NUC2F8: 101.325 kHz  
NUC2F9: 101.325 kHz  
NUC2F10: 101.325 kHz  
NUC2F11: 101.325 kHz  
NUC2F12: 101.325 kHz  
NUC2F13: 101.325 kHz  
NUC2F14: 101.325 kHz  
NUC2F15: 101.325 kHz  
NUC2F16: 101.325 kHz  
NUC2F17: 101.325 kHz  
NUC2F18: 101.325 kHz  
NUC2F19: 101.325 kHz  
NUC2F20: 101.325 kHz  
NUC2F21: 101.325 kHz  
NUC2F22: 101.325 kHz  
NUC2F23: 101.325 kHz  
NUC2F24: 101.325 kHz  
NUC2F25: 101.325 kHz  
NUC2F26: 101.325 kHz  
NUC2F27: 101.325 kHz  
NUC2F28: 101.325 kHz  
NUC2F29: 101.325 kHz  
NUC2F30: 101.325 kHz  
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NUC2F33: 101.325 kHz  
NUC2F34: 101.325 kHz  
NUC2F35: 101.325 kHz  
NUC2F36: 101.325 kHz  
NUC2F37: 101.325 kHz  
NUC2F38: 101.325 kHz  
NUC2F39: 101.325 kHz  
NUC2F40: 101.325 kHz  
NUC2F41: 101.325 kHz  
NUC2F42: 101.325 kHz  
NUC2F43: 101.325 kHz  
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NUC2F45: 101.325 kHz  
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NUC2F49: 101.325 kHz  
NUC2F50: 101.325 kHz  
NUC2F51: 101.325 kHz  
NUC2F52: 101.325 kHz  
NUC2F53: 101.325 kHz  
NUC2F54: 101.325 kHz  
NUC2F55: 101.325 kHz  
NUC2F56: 101.325 kHz  
NUC2F57: 101.325 kHz  
NUC2F58: 101.325 kHz  
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NUC2F62: 101.325 kHz  
NUC2F63: 101.325 kHz  
NUC2F64: 101.325 kHz  
NUC2F65: 101.325 kHz  
NUC2F66: 101.325 kHz  
NUC2F67: 101.325 kHz  
NUC2F68: 101.325 kHz  
NUC2F69: 101.325 kHz  
NUC2F70: 101.325 kHz  
NUC2F71: 101.325 kHz  
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NUC2F74: 101.325 kHz  
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NUC2F76: 101.325 kHz  
NUC2F77: 101.325 kHz  
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NUC2F85: 101.325 kHz  
NUC2F86: 101.325 kHz  
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NUC2F88: 101.325 kHz  
NUC2F89: 101.325 kHz  
NUC2F90: 101.325 kHz  
NUC2F91: 101.325 kHz  
NUC2F92: 101.325 kHz  
NUC2F93: 101.325 kHz  
NUC2F94: 101.325 kHz  
NUC2F95: 101.325 kHz  
NUC2F96: 101.325 kHz  
NUC2F97: 101.325 kHz  
NUC2F98: 101.325 kHz  
NUC2F99: 101.325 kHz  
NUC2F100: 101.325 kHz

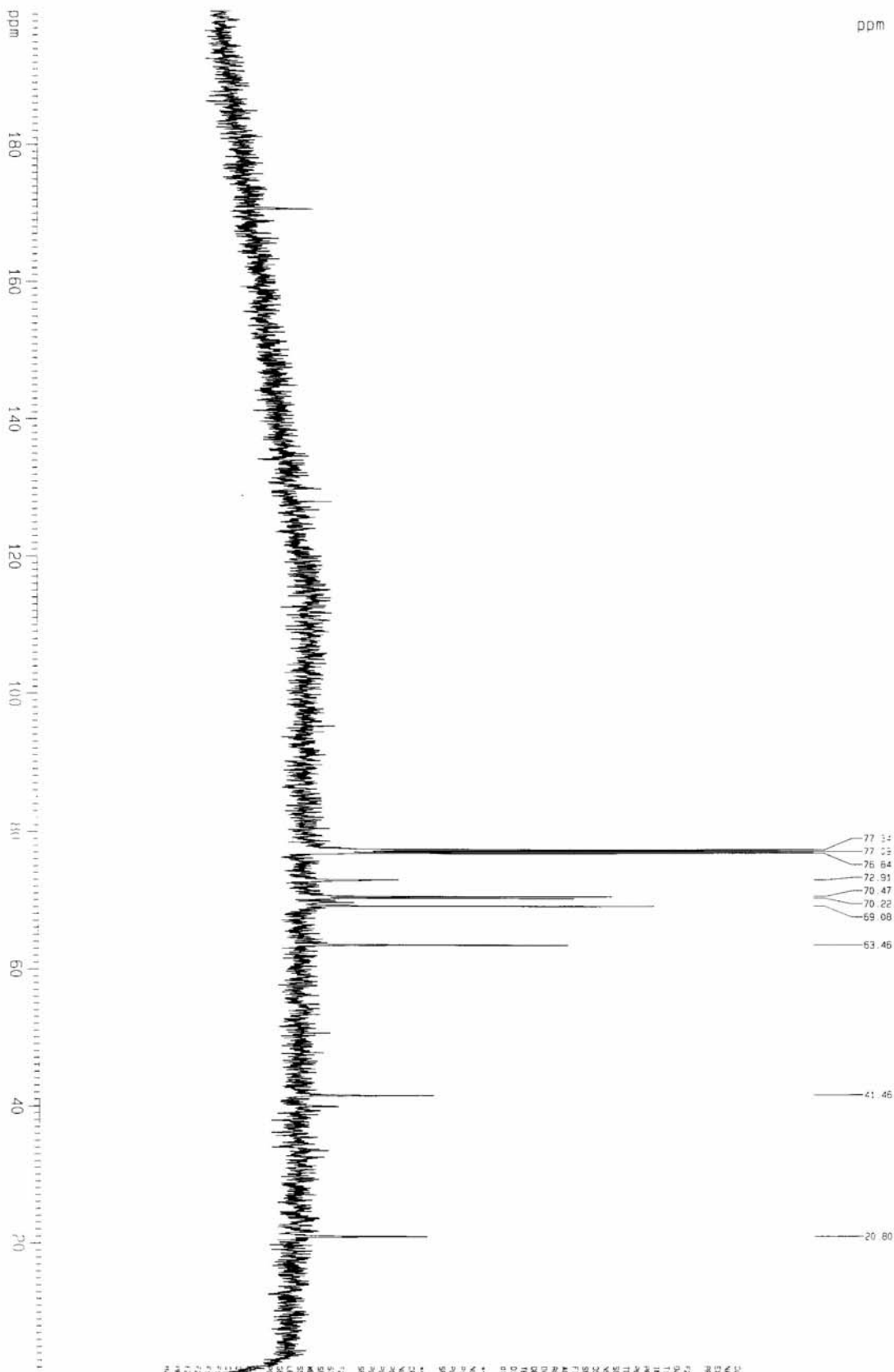
F2 - Processing parameters  
SI: 65536  
SF: 500.1300000 MHz  
WDW: EM  
SSB: 0  
GB: 0  
PC: 1.00

1D NMR list parameters  
SI: 65536  
SF: 500.1300000 MHz  
WDW: EM  
SSB: 0  
GB: 0  
PC: 1.00

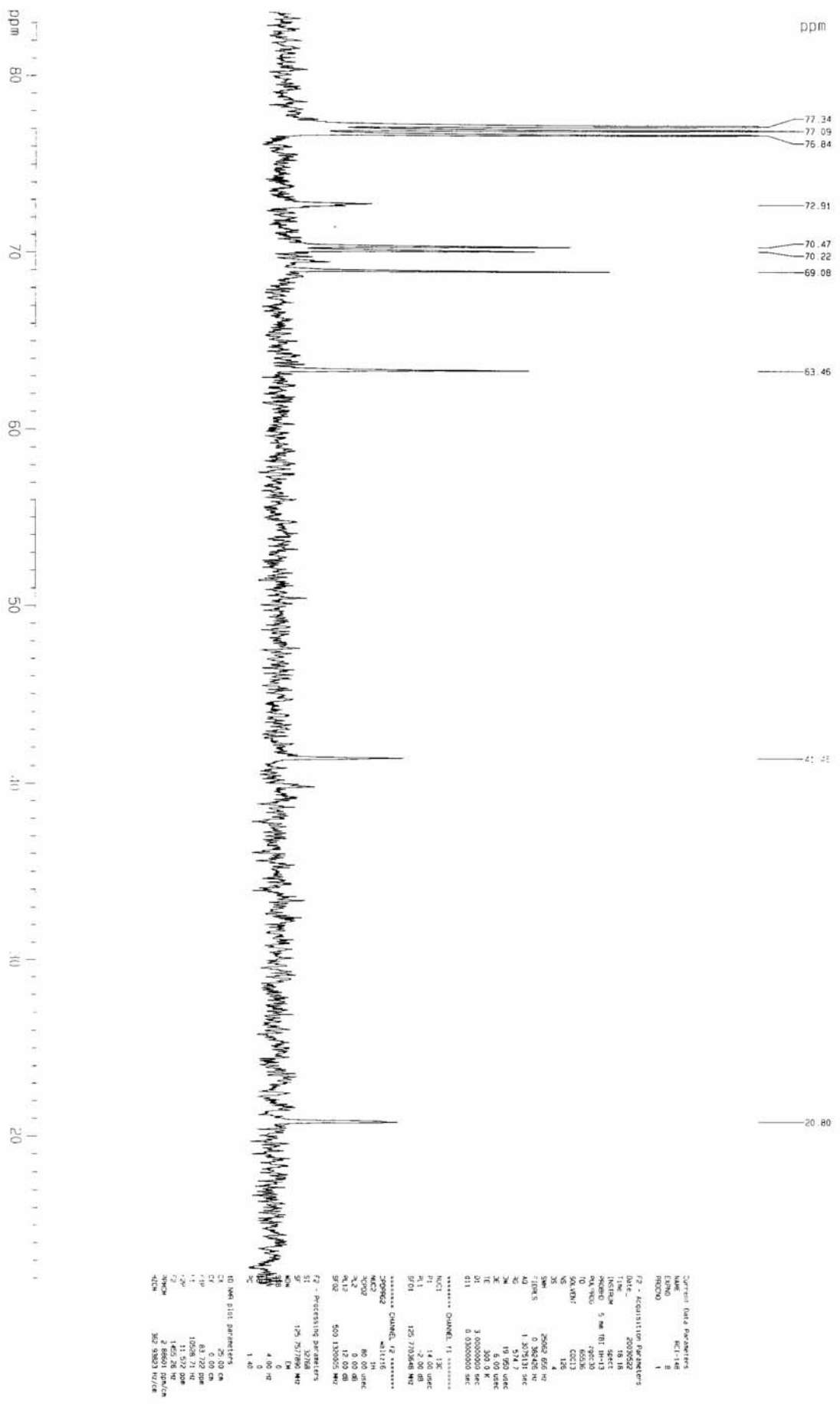
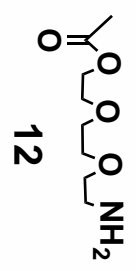
1D NMR list parameters  
SI: 65536  
SF: 500.1300000 MHz  
WDW: EM  
SSB: 0  
GB: 0  
PC: 1.00



ppm



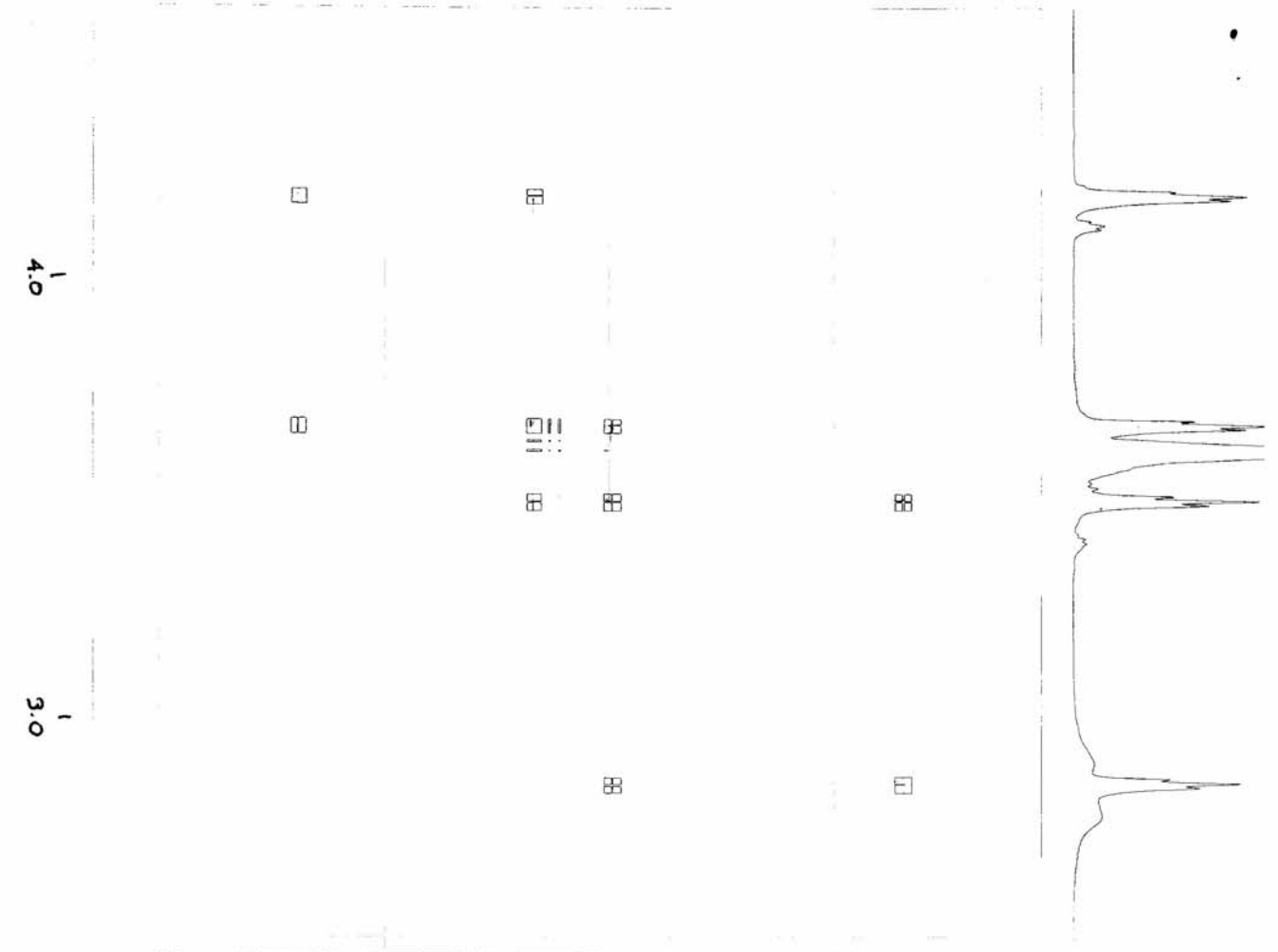
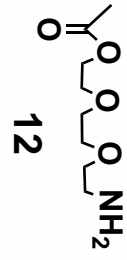
Current Data Parameters  
 NAME: ECL-148  
 EXPNO: 8  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_: 20030522  
 Time: 18.18  
 INSTRUM: spect  
 CHANNEL: hnu1hpc  
 PULPROG: zgpg30  
 TD: 65536  
 SOLVENT: CDCl3  
 NS: 163  
 DS: 4  
 SWH: 25042.636 Hz  
 FIDRES: 0.382426 Hz  
 AQ: 1.3075131 sec  
 RG: 327  
 DE: 19.950 uVsec  
 FE: 6.00 uVsec  
 TE: 300.0 K  
 D1: 1.0000000 sec  
 D11: 0.0000000 sec  
 \*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1: 13C  
 P1: 1.00 uVsec  
 PL1: -2.00 dB  
 SFO1: 125.760349 MHz  
 \*\*\*\*\* CHANNEL f2 \*\*\*\*\*  
 NUC2: 1H  
 P2: 1.00 uVsec  
 PL2: -2.00 dB  
 SFO2: 500.136099 MHz  
 F2 - Processing parameters  
 SI: 32768  
 SF: 125.760349 MHz  
 KMC: 0  
 SSB: 0  
 LB: 4.00 Hz  
 GB: 0  
 PC: 1.40  
 \*\*\*\*\* D1H1 Parameters \*\*\*\*\*  
 SI: 32768  
 SF: 500.136099 MHz  
 KMC: 0  
 SSB: 0  
 LB: 4.00 Hz  
 GB: 0  
 PC: 1.40



```

Current Data Properties
Name: 12
EXPNO: 5
PROCNO: 1
F2 - Acquisition Parameters
Date_ 20080522
Time 18:18
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SFO 125
AQ 5.00
RG 512
DELTA 0.0000000 sec
DE 0.0000000 sec
TE 300.2 K
D1 3.00000000 sec
d11 0.00000000 sec
***** CHANNEL f1 *****
NUC1 13C
P1 14.00 usec
PL 0.00 dB
SFO1 125.770349 MHz
***** CHANNEL f2 *****
NAME 12
PROCNO 1
AQ 5.00 usec
RG 512
DELTA 0.0000000 sec
DE 0.00000000 sec
TE 300.2 K
D1 3.00000000 sec
d11 0.00000000 sec
***** CHANNEL f3 *****
NAME 12
PROCNO 1
AQ 5.00 usec
RG 512
DELTA 0.0000000 sec
DE 0.00000000 sec
TE 300.2 K
D1 3.00000000 sec
d11 0.00000000 sec
F2 - Processing parameters
SI 32768
SF 125.770349 MHz
WDW EM
SSB 0
GB 0
PC 4.00 usec
MC 1.40
DC 0
***** NMR 010: parameters *****
C1 25.00 usec
C2 1.00 usec
C3 1.00 usec
C4 1.00 usec
C5 1.00 usec
C6 1.00 usec
C7 1.00 usec
C8 1.00 usec
C9 1.00 usec
C10 1.00 usec
C11 1.00 usec
C12 1.00 usec
C13 1.00 usec
C14 1.00 usec
C15 1.00 usec
C16 1.00 usec
C17 1.00 usec
C18 1.00 usec
C19 1.00 usec
C20 1.00 usec
C21 1.00 usec
C22 1.00 usec
C23 1.00 usec
C24 1.00 usec
C25 1.00 usec
C26 1.00 usec
C27 1.00 usec
C28 1.00 usec
C29 1.00 usec
C30 1.00 usec
C31 1.00 usec
C32 1.00 usec
C33 1.00 usec
C34 1.00 usec
C35 1.00 usec
C36 1.00 usec
C37 1.00 usec
C38 1.00 usec
C39 1.00 usec
C40 1.00 usec
C41 1.00 usec
C42 1.00 usec
C43 1.00 usec
C44 1.00 usec
C45 1.00 usec
C46 1.00 usec
C47 1.00 usec
C48 1.00 usec
C49 1.00 usec
C50 1.00 usec
C51 1.00 usec
C52 1.00 usec
C53 1.00 usec
C54 1.00 usec
C55 1.00 usec
C56 1.00 usec
C57 1.00 usec
C58 1.00 usec
C59 1.00 usec
C60 1.00 usec
C61 1.00 usec
C62 1.00 usec
C63 1.00 usec
C64 1.00 usec
C65 1.00 usec
C66 1.00 usec
C67 1.00 usec
C68 1.00 usec
C69 1.00 usec
C70 1.00 usec
C71 1.00 usec
C72 1.00 usec
C73 1.00 usec
C74 1.00 usec
C75 1.00 usec
C76 1.00 usec
C77 1.00 usec
C78 1.00 usec
C79 1.00 usec
C80 1.00 usec
C81 1.00 usec
C82 1.00 usec
C83 1.00 usec
C84 1.00 usec
C85 1.00 usec
C86 1.00 usec
C87 1.00 usec
C88 1.00 usec
C89 1.00 usec
C90 1.00 usec
C91 1.00 usec
C92 1.00 usec
C93 1.00 usec
C94 1.00 usec
C95 1.00 usec
C96 1.00 usec
C97 1.00 usec
C98 1.00 usec
C99 1.00 usec
C100 1.00 usec

```



Current Data Parameters

NAME: DCI-148  
 INSTR: 1  
 PROCNO: 1

F2 - Acquisition Parameters

Date\_: 20030627  
 Time: 18.12  
 INSTRUM: 5 m HPL SOLCT  
 METHOD: 039700141  
 P1/P2/Q1: 2048  
 TO: DCI13  
 SOLVENT: DCI13  
 NS: 4  
 DS: 16  
 SWH: 2900.000 Hz  
 FIDRES: 1.620000 Hz  
 AQRES: 0.400000 sec  
 RG: 10321.3  
 DM: 200.000 usec  
 DE: 6.00 usec  
 TE: 300.0 K  
 D0: 0.0000000 sec  
 D1: 1.0000000 sec  
 D2: 0.0000000 sec  
 d13: 0.0000000 sec  
 d15: 0.0005000 sec  
 INO: 0.00040000 sec  
 MCHRES: 0.00000000 sec  
 MCHRES1: 1.00000000 sec

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*

NUC1: 1H  
 P1: 8.50 usec  
 PL1: 0.00 dB  
 SFO1: 500.1312603 MHz

\*\*\*\*\* GRADIENT CHANNEL \*\*\*\*\*

GRAD1: 100.0000000  
 GRAD2: 100.0000000  
 GRAD3: 100.0000000  
 GRAD4: 100.0000000  
 GRAD5: 100.0000000  
 GRAD6: 100.0000000  
 GRAD7: 100.0000000  
 GRAD8: 100.0000000  
 GRAD9: 100.0000000  
 GRAD10: 100.0000000  
 GRAD11: 100.0000000  
 GRAD12: 100.0000000  
 GRAD13: 100.0000000  
 GRAD14: 100.0000000  
 GRAD15: 100.0000000  
 GRAD16: 100.0000000  
 GRAD17: 100.0000000  
 GRAD18: 100.0000000  
 GRAD19: 100.0000000  
 GRAD20: 100.0000000  
 GRAD21: 100.0000000  
 GRAD22: 100.0000000  
 GRAD23: 100.0000000  
 GRAD24: 100.0000000  
 GRAD25: 100.0000000  
 GRAD26: 100.0000000  
 GRAD27: 100.0000000  
 GRAD28: 100.0000000  
 GRAD29: 100.0000000  
 GRAD30: 100.0000000  
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 GRAD33: 100.0000000  
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 GRAD35: 100.0000000  
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 GRAD92: 100.0000000  
 GRAD93: 100.0000000  
 GRAD94: 100.0000000  
 GRAD95: 100.0000000  
 GRAD96: 100.0000000  
 GRAD97: 100.0000000  
 GRAD98: 100.0000000  
 GRAD99: 100.0000000  
 GRAD100: 100.0000000

F1 - Acquisition Parameters

NO: 1  
 TO: 30  
 SFO1: 500.1313 MHz  
 FIDRES: 83.33336 Hz  
 SW: 41999 DPM  
 FWHZ: 0  
 PC: 1.40

F2 - Processing Parameters

SF: 4996  
 F2: 500.1300000 MHz  
 KW: SINE  
 SSB: 0  
 GB: 0  
 PC: 1.40

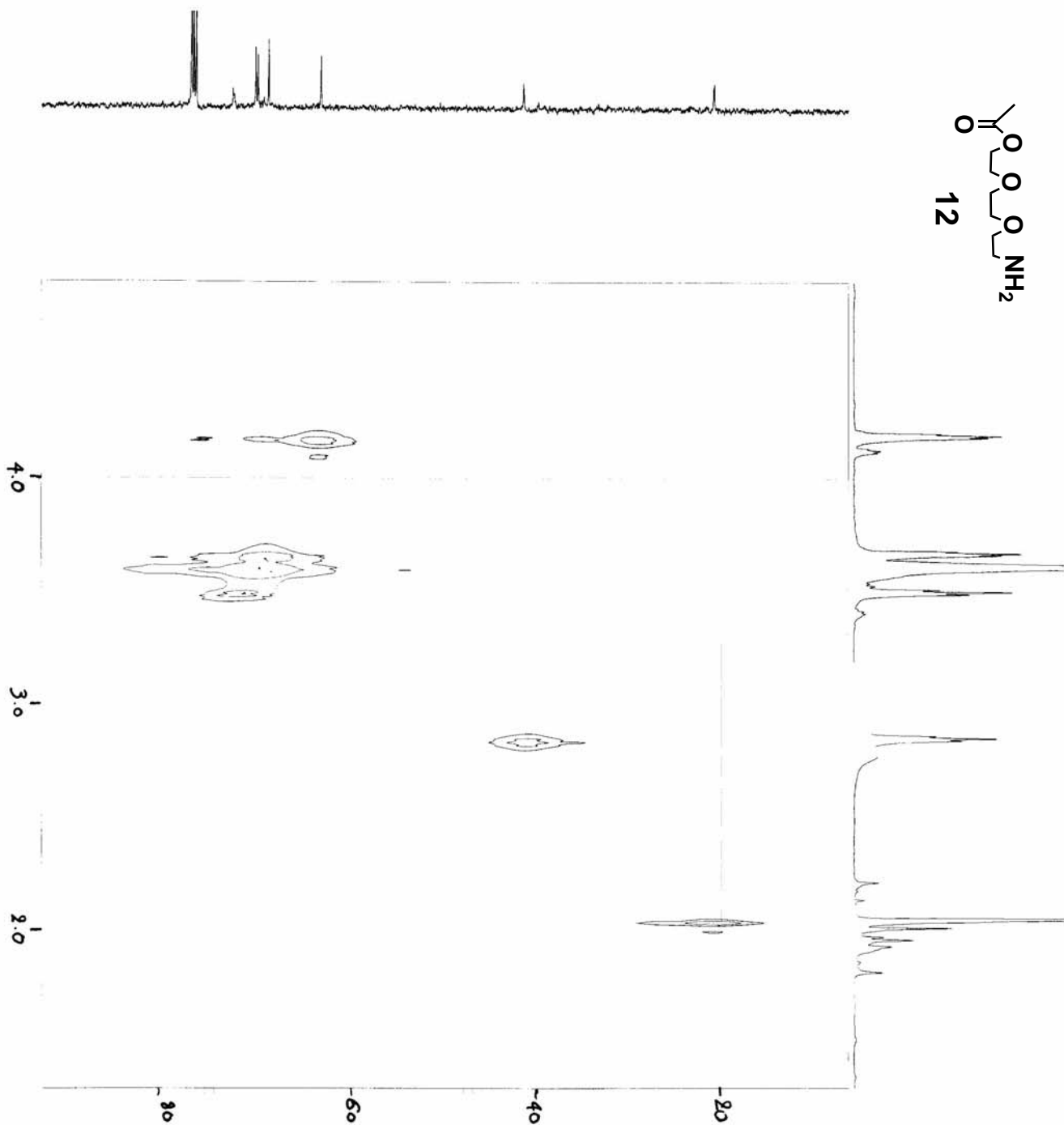
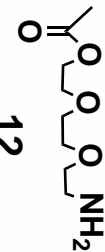
F1 - Processing Parameters

SF: 2048  
 F2: 500.1300000 MHz  
 KW: SINE  
 SSB: 0  
 GB: 0  
 PC: 0

20 MHz list parameters

CX1: 15.00 cm  
 F2H0: 4.620 GPM  
 F2H1: 2310.51 MHz  
 F2H2: 2.448 GPM  
 F2H3: 1028.51 Hz  
 F1H0: 2324.54 Hz  
 F1H1: 2.538 GPM  
 F1H2: 1504.86 Hz  
 F2PCK1: 0.14405 GPM/cm  
 F2PCK2: 0.06417 Hz/cm  
 F1PCK1: 0.14008 GPM/cm  
 F1PCK2: 0.31200 Hz/cm

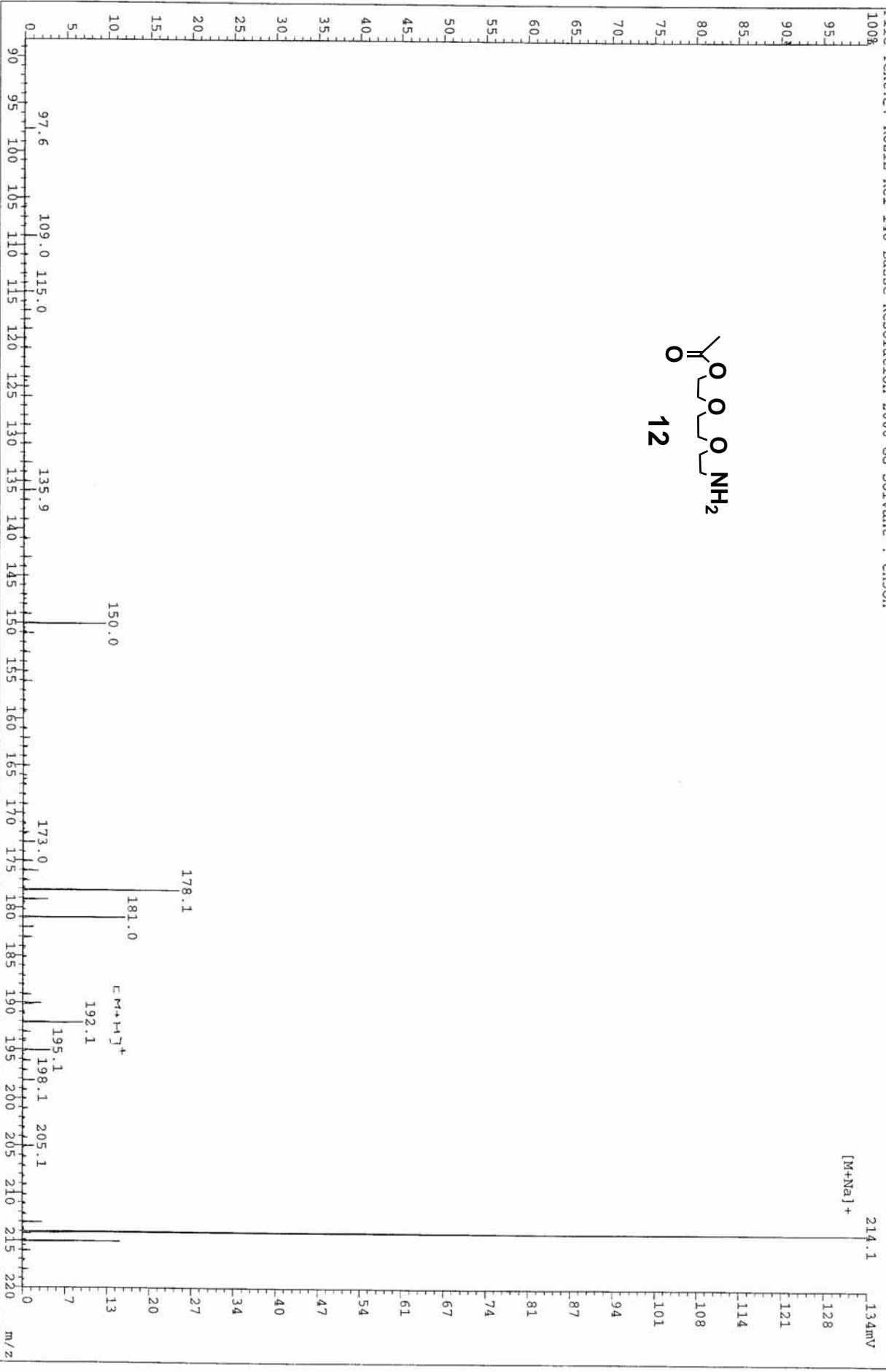
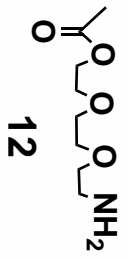




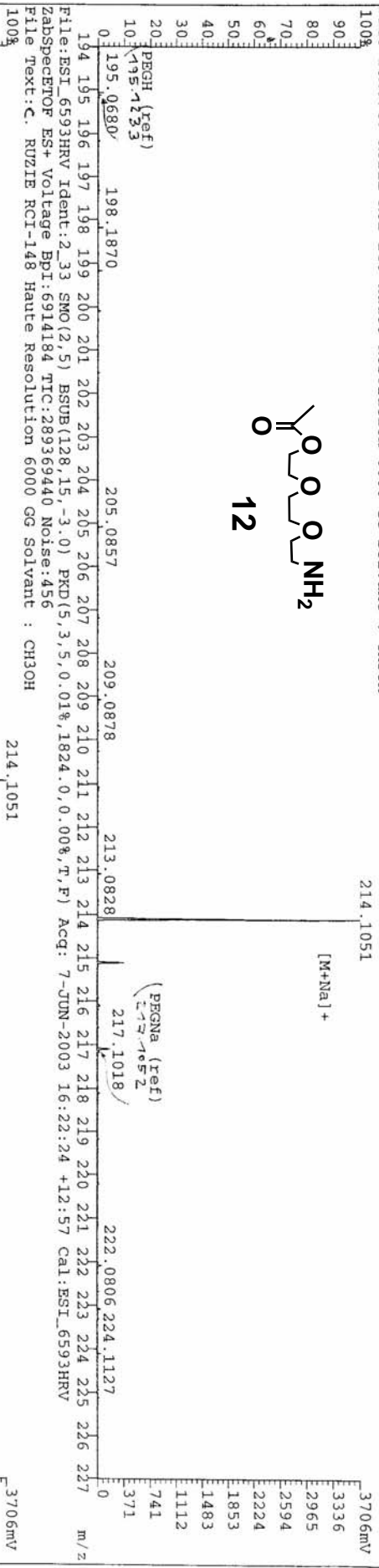
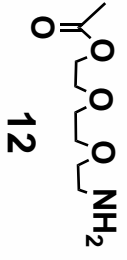
Current Data Parameters

NAME: 12-11-11  
 INSTRUM: 1  
 DATE\_: 08-SEP-11  
 TIME: 14.41  
 F1NAME: 12-11-11-1  
 PULPROG: zgpg30  
 TD: 65536  
 SFO: 500.136261 MHz  
 DS: 4  
 SWH: 2500.0000 MHz  
 FIDRES: 0.4666666666666667  
 AQ: 0.1000000000000000  
 INJ: 51795.2  
 DM: 200.00000000000000  
 TE: 300.2 K  
 CQ: 14.000000000000000  
 RG: 0.0000000000000000  
 DI: 0.0000000000000000  
 DE: 0.0000000000000000  
 FI: 0.0000000000000000  
 F2: 0.0000000000000000  
 F3: 0.0000000000000000  
 F4: 0.0000000000000000  
 F5: 0.0000000000000000  
 F6: 0.0000000000000000  
 F7: 0.0000000000000000  
 F8: 0.0000000000000000  
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 F10: 0.0000000000000000  
 F11: 0.0000000000000000  
 F12: 0.0000000000000000  
 F13: 0.0000000000000000  
 F14: 0.0000000000000000  
 F15: 0.0000000000000000  
 F16: 0.0000000000000000  
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 F90: 0.0000000000000000  
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 F92: 0.0000000000000000  
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 F96: 0.0000000000000000  
 F97: 0.0000000000000000  
 F98: 0.0000000000000000  
 F99: 0.0000000000000000  
 F100: 0.0000000000000000

File:ESI\_6593 Ident:1 SMO(2,5) SUBS(128,15,-3,0) PKD(5,3,5,0,0.01%,68,0,0,0.00%,T,F) Acq: 7-JUN-2003 16:14:28 +2:58 Cal: E3000\_030410\_GR\_P\_LN\_Ix0.999963  
 ZabsPECTOP ES+ Magnet BpI:797704 TIC:32176804 Noise:17  
 File Text:C, RUIE RCI-148 Basse Resolution 2000 GG Solvant : CH3OH



File: EST\_6593HRV Ident: 2\_33 SMO(2,5) ESUB(128,15,-3,0) PKD(5,3,5,0,0.018,1824,0,0.008,T,F) Acq: 7-JUN-2003 16:22:24 +12:57 Cal: EST\_6593HRV  
 ZabsSpecTOP ES+ Voltage BPL:6914184 TIC:289369440 Noise:456  
 File Text: C. RUIZIE RCI-148 Haute Resolution 6000 GG Solvant : CH3OH



File: EST\_6593HRV Ident: 2\_33 SMO(2,5) ESUB(128,15,-3,0) PKD(5,3,5,0,0.018,1824,0,0.008,T,F) Acq: 7-JUN-2003 16:22:24 +12:57 Cal: EST\_6593HRV  
 ZabsSpecTOP ES+ Voltage BPL:6914184 TIC:289369440 Noise:456  
 File Text: C. RUIZIE RCI-148 Haute Resolution 6000 GG Solvant : CH3OH

Amas isotopique theorique de [M+Na]<sup>+</sup> : C8 H17 N O4 Na

12,13,14C: 8, 1,2H: 17, 14,15N: 1, 16,17, 18O: 4, 23Na: 1  
 Separation: 1000 Min Frac Abun: 0.01 Num Charges: 1 Resolution: 6000  
 Nominal Mass: 214 Monoisotopic Mass: 214.106 Average Mass: 214.217 Peak Maximum Mass: 214.11  
 214.1055

