Preparation of Hypermonomer 1. In a typical experiment allyl amine (3.2 gm, 56 mmol) was allowed to react with ethyl bromohexanoate (25.0 gm, 112 mmol) in the presence of K_2CO_3 (30.9 gm, 224 mmol) in DMF (100 mL) at 50 °C for 12 hrs. After cooling, the reaction mixture was passed through a bed of celite to remove the inorganic base. The filtrate was concentrated under reduced pressure. Pure product was obtained either by column chromatography (35-40% EtOAc in hexane) or by vacuum distillation. Yield 17.8 gm, 93%. ^{1}H NMR, $CDCl_3$, δ 5.8-5.7 (m, 1H), 5.1-5.0 (m, 2H), 4.14-4.01 (m, 4H), 3.0 (br t, 2H), 2.40-2.18 (m, 8H), 1.61-1.15 (m, 18H). ^{13}C NMR $CDCl_3$, δ 173.58, 135.94, 116.83, 60.01, 57.13, 53.43, 34.19, 26.94, 24.81, 14.13.

Compound 2. $Pd(PPh_3)_4$ (20 mg) was added under argon to a round bottom flask. A solution of N,N dimethyl barbituric acid (4.68 gm, 30 mmol)in 30 mL of dichloromethane (DCM) and hypermonomer **1** (6.84 gm, 20 mmol) in 20 mL of DCM was added to the round bottom flask under argon. Once the reaction was over (TLC analysis), the mixture was diluted with ether and concentrated. Purification was performed by ion-exchange chromatography (Dowex 50 X2). The crude product was loaded on to the ion-exchange resin. The resin was washed with DCM (25 mL), diethyl ether (25 mL). The pure product was obtained by washing the resin column using 10% triethyl amine in DCM. The pure fractions of the product were combined and concentrated. Yield 5.35 gm, 89%. ^{1}H NMR, $CDCl_3$, δ 4.03 (q, 4H), 2.51 (t, 3H), 2.21 (t, 4H), 1.75 (br, NH), 1.61-1.56 (m, 4H) 1.54-1.38 (m, 4H), 1.32-1.23 (m, 4H), 1.16 (t, 6H). ^{13}C NMR $CDCl_3$, δ 173.57, 60.07, 49.61, 34.14, 29.50, 26.79, 24.74, 14.14.

Compound 3. The hypermonomer **1** (6.84 gm, 20 mmol) was dissolved in 25 mL of MeOH. To this at 0 °C 5N NaOH (15 mL) was added slowly. The reaction was monitored using TLC with ninhydrin staining. Once the hydrolysis was complete, the reaction mixture was neutralized with HCl and concentrated under high vacuum. Using silica gel chromatography (25:75 MeOH/$CHCl_3$), the pure product was isolated from the residue.[24] Yield 5.2 gm, 92%. ^{1}H NMR, CD_3OD, δ 6.1-5.9 (m, 1H), 5.6-5.53 (d, 2H), 3.76 (d, 2H), 3.07 (t, 4H), 2.25 (t, 4H), 1.75-1.60 (m, 8H), 1.42-1.37 (m, 4H). ^{13}C NMR CD_3OD, δ 174.72, 127.08, 125.17, 54.84, 51.93, 49.12, 33.70, 26.01, 24.20, 23.01. ESI-Mass ($C_{15}H_{28}NO_4$) 286.05.

Compound 4. In a typical experiment compound **3** (906 mg, 3 mmol) was dissolved in minimum amount of DMF. To this, a solution of HBTU (1.08 gm, 2.85 mmol) was added followed by the addition of DIEA (1.04 mL, 6 mmol). The mixture was stirred for few min. Finally the compound **2** (427 mg, 1.5 mmol) in 1.0 mL of DMF was added to this stirring mixture, and the reaction was continued over night (12-14 hrs). Once all the starting materials were consumed (TLC analysis), the reaction was quenched with addition of 1 mL of water, and the reaction mixture was diluted with ether. The major portion of DMF was removed by aqueous workup. The organic layer was dried over Na_2SO_4 and concentrated under reduced pressure. Chromatography of the residue was performed using silica gel and 5:95 MeOH/$CHCl_3$. Thus the tetrameric dendrimer (**4**) was obtained in high yield 1.10 gm, 86%. ^{1}H NMR, $CDCl_3$, δ 5.72-5.67 (m, 1H), 5.04-4.94 (d, 2H), 4.01 (q, 8H), 3.20-3.0 (m, 12H), 2.95 (d, 2H), 2.25 (t, 4H), 2.20-2.10 (m, 12H), 1.70 (t, 4H), 1.60-1.25 (m, 16H). 1.22-1.15 (m, 24H). ^{13}C NMR $CDCl_3$, δ 173.41, 173.18, 172.28, 135.32, 117.26, 60.09, 59.97, 56.95, 53.41, 47.62, 45.49, 40.82, 34.03, 33.91, 32.89, 28.73, 28.15, 27.30, 27.25, 26.52, 26.36, 26.22, 25.21, 24.54, 24.47, 14.09. Analysis C, H, N.

Compound 5. In a typical experiment compound **4** (530 mg, 0.62 mmol) in DCM was added to $Pd(PPh_3)_4$ under argon. A solution of N,N dimethyl barbituric acid (146 mg, 0.93 mmol) was added under argon to the reaction mixture. Once the reaction was over (as analyzed by TLC), the mixture was diluted with ether and concentrated. Purification was performed by ion-exchange chromatography. The crude product was loaded on to the ion-exchange resin. The resin was washed with DCM (15 mL), diethyl ether (10 mL). The pure product was obtained by washing the resin column using 10% triethyl amine in DCM. The pure fractions of the product were combined and concentrated. Yield 422 mg, 84%. ^{1}H NMR, $CDCl_3$, δ 4.13-4.04 (2q, 8H), 3.28-3.10 (m, 12H), 2.95 (t, 4H), 2.25 (qt, 12H), 1.80 (t, 4H), 1.65-1.31 (m, 20H) 1.29-1.19 (m, 20). ^{13}C NMR $CDCl_3$, δ 173.61, 173.42, 172.26, 60.31, 60.19, 50.59, 47.72, 47.44, 45.82, 34.12, 34.00, 32.23, 28.68, 27.36, 26.32, 25.70, 24.60, 24.53, 14.189. Analysis C, H, N.

Compound 6. A solution of compound 4 (530 mg, 0.62 mmol) in 5 mL of MeOH was cooled to 0 °C. To this 2 mL of 5N NaOH was added slowly. The reaction was monitored using TLC with ninhydrin staining. Once the hydrolysis was complete, the reaction mixture was neutralized with HCl and concentrated under high vacuum. Using silica gel chromatography, the pure product was isolated from the residue[24]. Yield 325 mg, 71%. ^{1}H NMR, CD_3OD, δ 6.1-5.9 (m, 1H), 5.7-5.53 (d, 2H), 3.76 (d, 2H), 3.35 (t, 8H), 3.16 (br, 4H), 2.5-2.25 (m, 12H), 1.9-1.78 (m, 4H), 1.78-1.50 (m, 24H), 1.5-1.32 (m, 12H). ^{13}C NMR CD_3OD, δ 175.92, 174.42, 173.76, 126.29, 125.27, 54.93, 52.26, 48.50, 45.84, 33.37, 33.24, 33.22, 32.98, 32.00, 28.24, 26.86, 26.00, 25.83, 24.58, 24.28, 23.96. ESI-Mass ($C_{39}H_{69}N_3O_{10}$) 740.5.

Compound 7. In a typical experiment compound **6** (74 mg, 0.1 mmol) was dissolved in minimum amount of DMF. To this solution HBTU (151 mg, 0.4 mmol) was added followed by DIEA (140 μL, 0.8 mmol). The mixture was stirred for few min. Finally compound **5** (324 mg, 0.4 mmol) in 1.0 mL of DMF was added to this stirring mixture, and the reaction was continued over night (12-14 hrs). Once all the starting materials were consumed (TLC analysis), the reaction was quenched with addition of 1 mL of water, and the reaction mixture was diluted with ether. The major portion of DMF was removed by aqueous workup. The organic layer was dried over Na_2SO_4 and concentrated under reduced pressure. Chromatography of the residue was performed using silica gel and 10:90 MeOH/$CHCl_3$. Yield 230 mg, 60%. MALDI-TOF ($C_{215}H_{385}N_{15}O_{46}$) Calculated 3916.54 observed 3917.14.

Compound 8. In a typical experiment N-fmoc-amino hexanoic acid (660 mg, 1.86 mmol) in 3 mL of DCM and diisopropyl ethyl amine (646 μL, 3.72 mmol) in 2 mL of DCM were added to 600 mg of 2-chlorotrityl chloride resin. After 30 min of mixing another fresh batch of amino acid and the base were added and allowed to react with the resin. The resin was then washed with DCM followed by DMF. A solution of 20% piperidine in DMF (5 mL) was added to the resin and mixed for 15 min. After the fmoc removal, the resin was washed with DMF followed by DCM and then DMF. N-fmoc Sarcosine (578 mg, 1.86 mmol) was dissolved in 2 mL of DMF. To this, a solution of HBTU (669 mg, 1.76 mmol) in 2 mL of DMF was added followed by DIPEA (646 μL, 3.72 mmol) in 1 mL of DMF. After 30 min of coupling a second coupling was performed. N-fmoc removal was effected by piperidine as per standard protocol. Biotin (453 mg, 1.86 mmol) in 8 mL of NMP was added to the resin followed by addition of a solution of HBTU (669 mg, 1.76 mmol) in 1 mL of DMF was added followed by DIPEA (646 μL, 3.72 mmol) in 1 mL of DMF. After 30 min of coupling a second coupling was performed. Using standard protocol the product was cleaved from the resin and purified using RP-HPLC (10% to 45% of ACN in 65 min). The major peak was isolated and lyophilized. Yield 207 mg, 77%. ^{1}H NMR, $CDCl_3$, δ 4.53-4.49 (m, 1H), 4.35-4.32 (m, 1H), 4.05 (2s, 2H), 3.56 (t, 1H), 3.32-3.30 (m, 1H), 3.26-3.18 (m, 4H), 3.10 (s, 3H), 2.95 (s, 2H), 2.7 (d, 1H), 2.50-2.42 (m, 2H), 2.40-2.25 (m, 4 H), 1.9-1.45 (m, 16H), 1.45-1.3 (m, 3H). ^{13}C NMR CD_3OD, δ 174.80, 169.54, 168.99, 164.62, 163.67, 163.28, 162.88, 61.83, 60.64, 60.19, 55.57, 52.38, 50.60, 39.74, 38.96, 38.83, 36.13, 35.23, 34.94, 34.65, 34.10, 33.46, 32.37, 32.20, 29.96, 29.67, 29.39. ESI-Mass ($C_{19}H_{32}N_4O_5S$) 429.2.

Compound 9. In a typical experiment compound **8** (51 mg, 0.12 mmol) was dissolved in minimum amount of NMP, and HBTU (46 mg, 0.12 mmol) was added, followed by DIEA (31 mg, 0.24 mmol). After stirring for 1 min, compound **5** (97 mg, 0.12 mmol) in 500 μL of NMP was added and the reaction was allowed to proceed overnight (12-14 hrs). Once all the starting materials were consumed (TLC), the reaction was quenched with addition of a few drops of water. NMP was removed using high vacuum, and the residue was purified using RP-HPLC (20% to 75% of ACN in 80 min). The major peak was isolated and lyophilized. Yield 52 mg, 35%. ESI-Mass ($C_{63}H_{111}N_7O_{14}S$) 1222.72.

Compound 10. In a typical experiment compound **9** was hydrolyzed using a NaOH/H_2O mixture as per procedure reported for compound **3**. Following the workup method mentioned above, compound **10** was obtained as a white solid. ESI-Mass ($C_{55}H_{95}N_7O_{14}S$) 1110.53.

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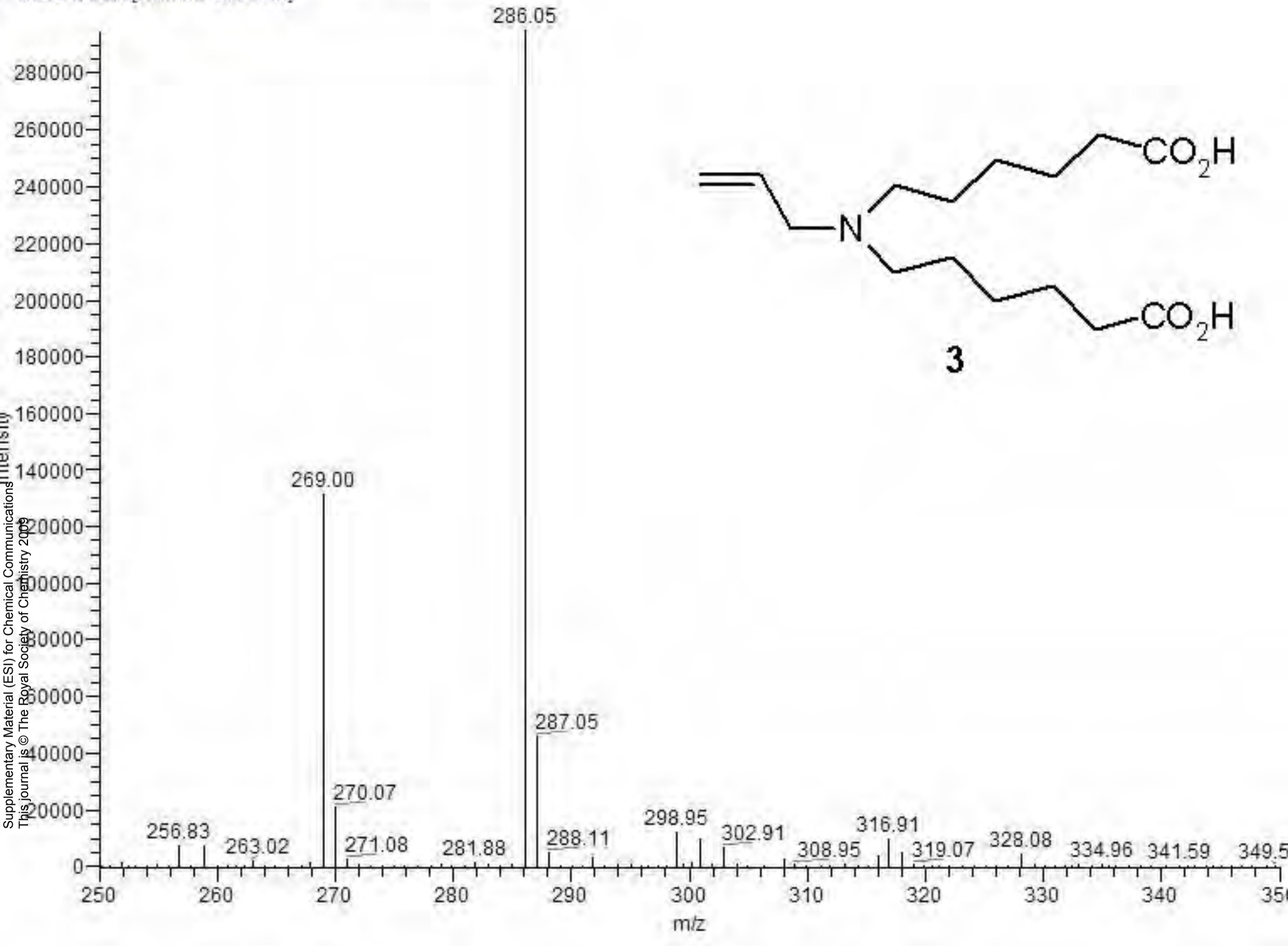

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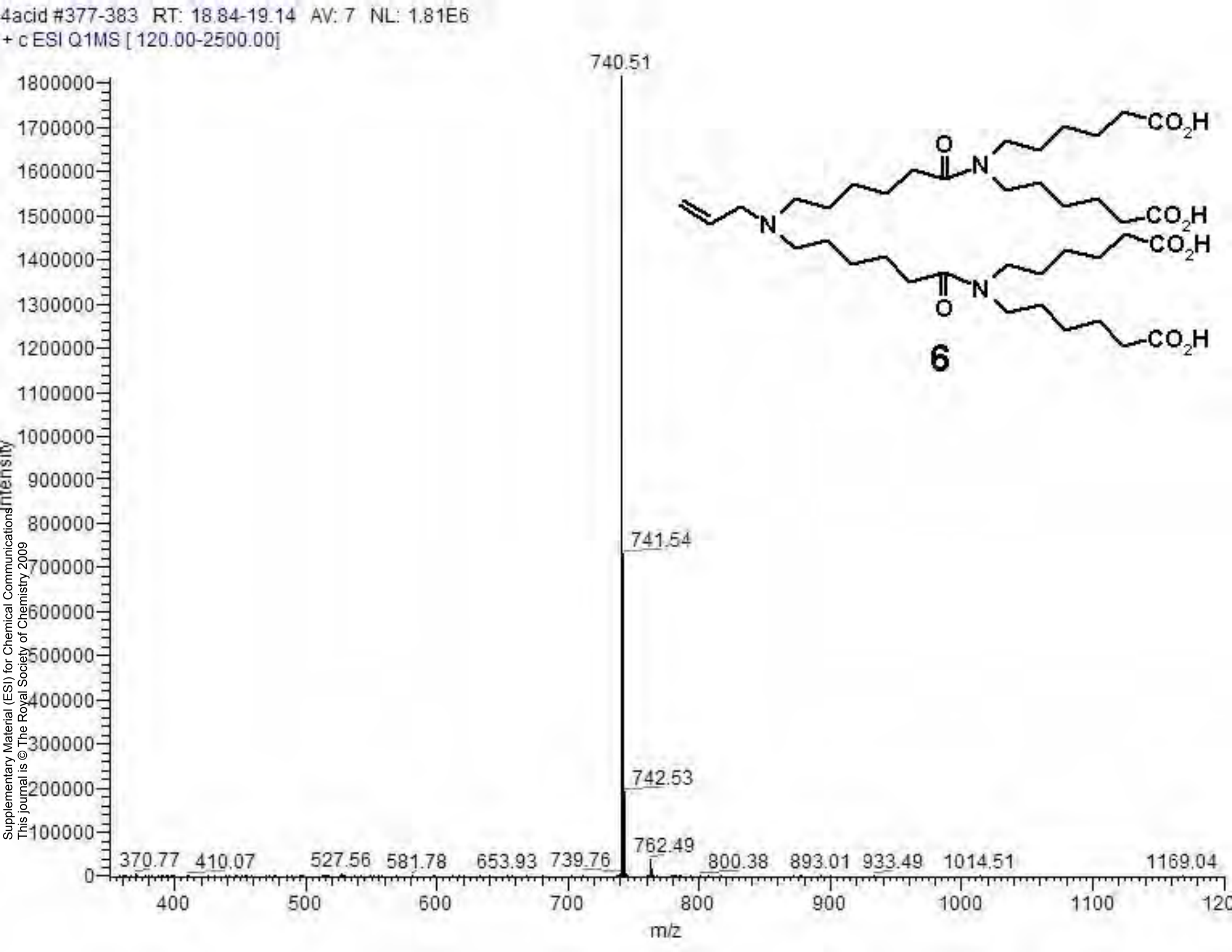

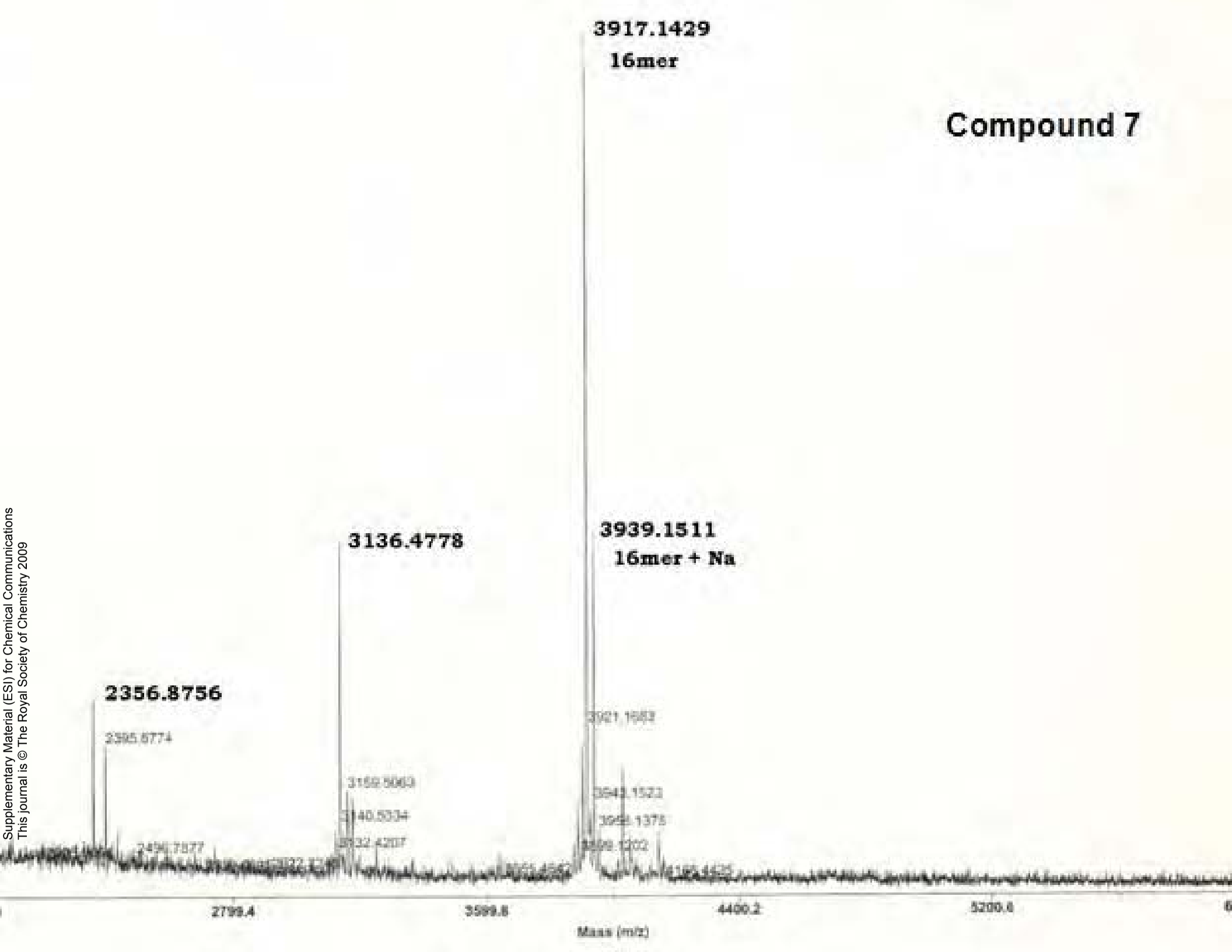

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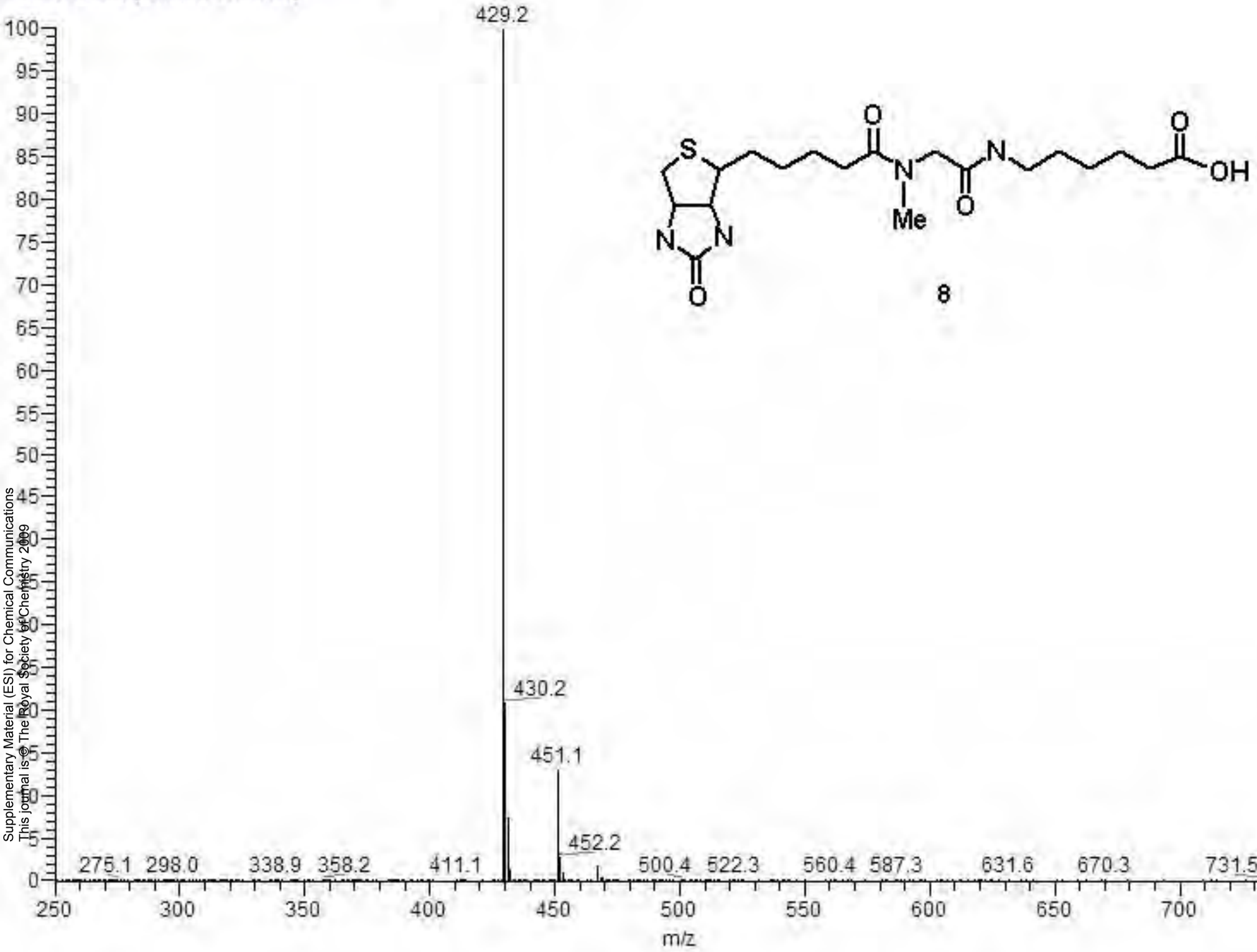

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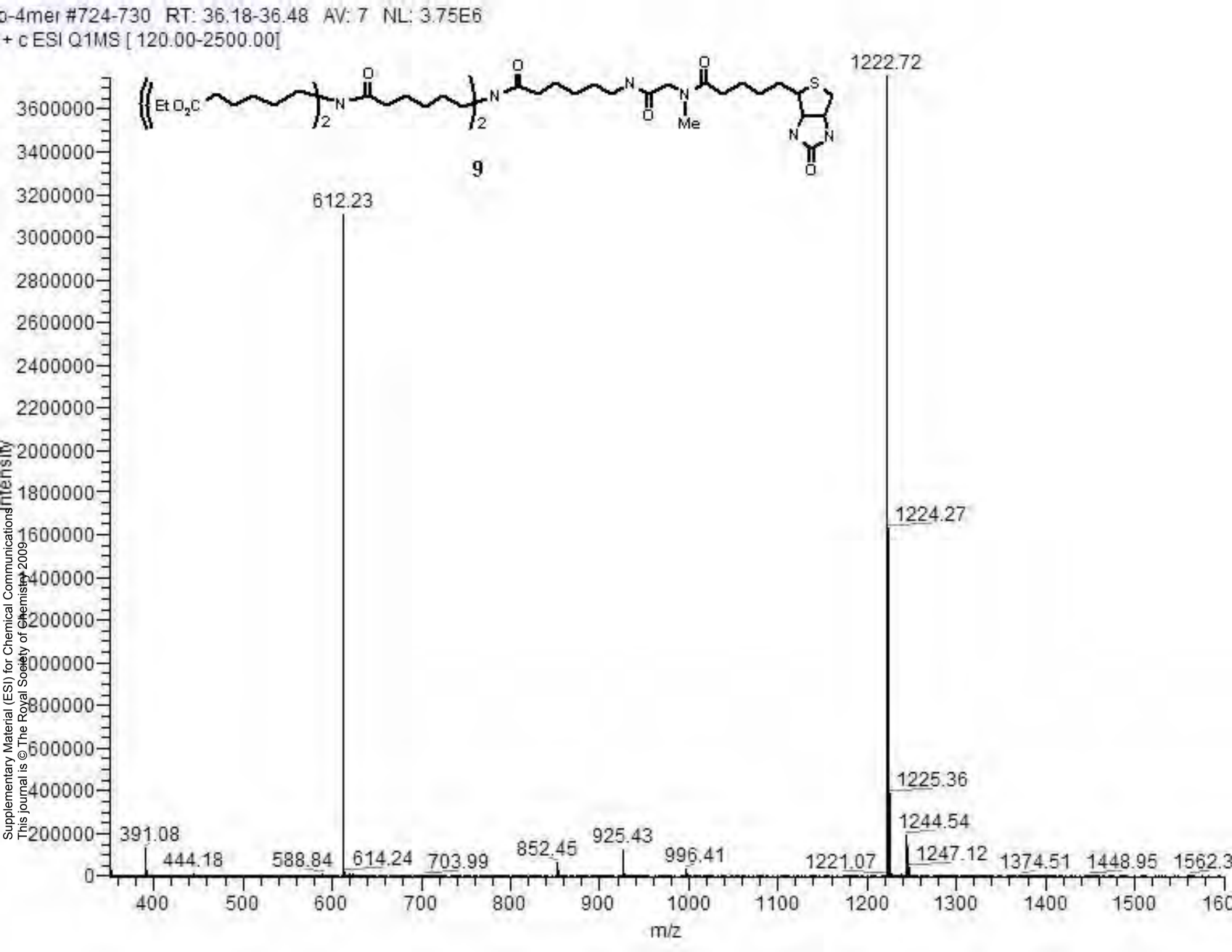

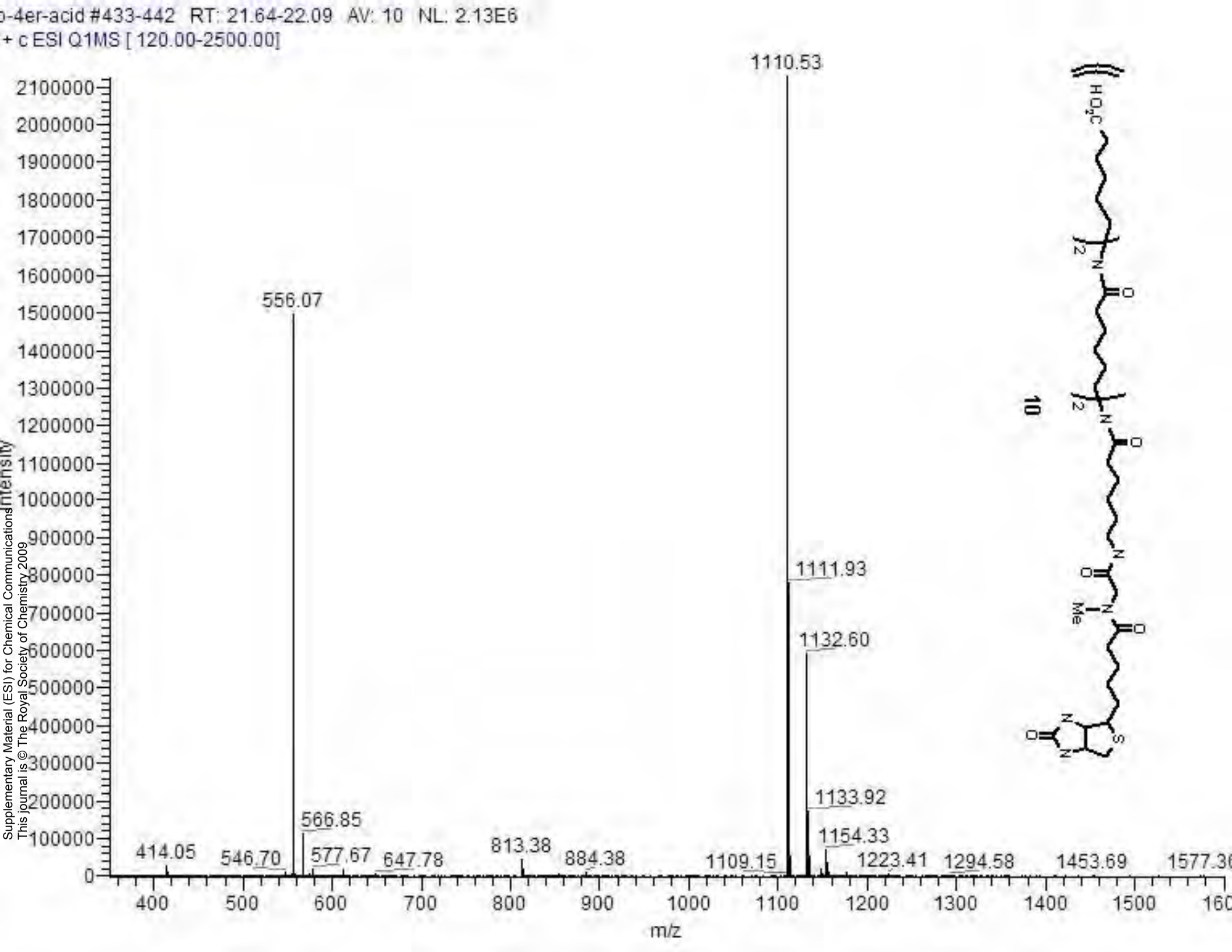

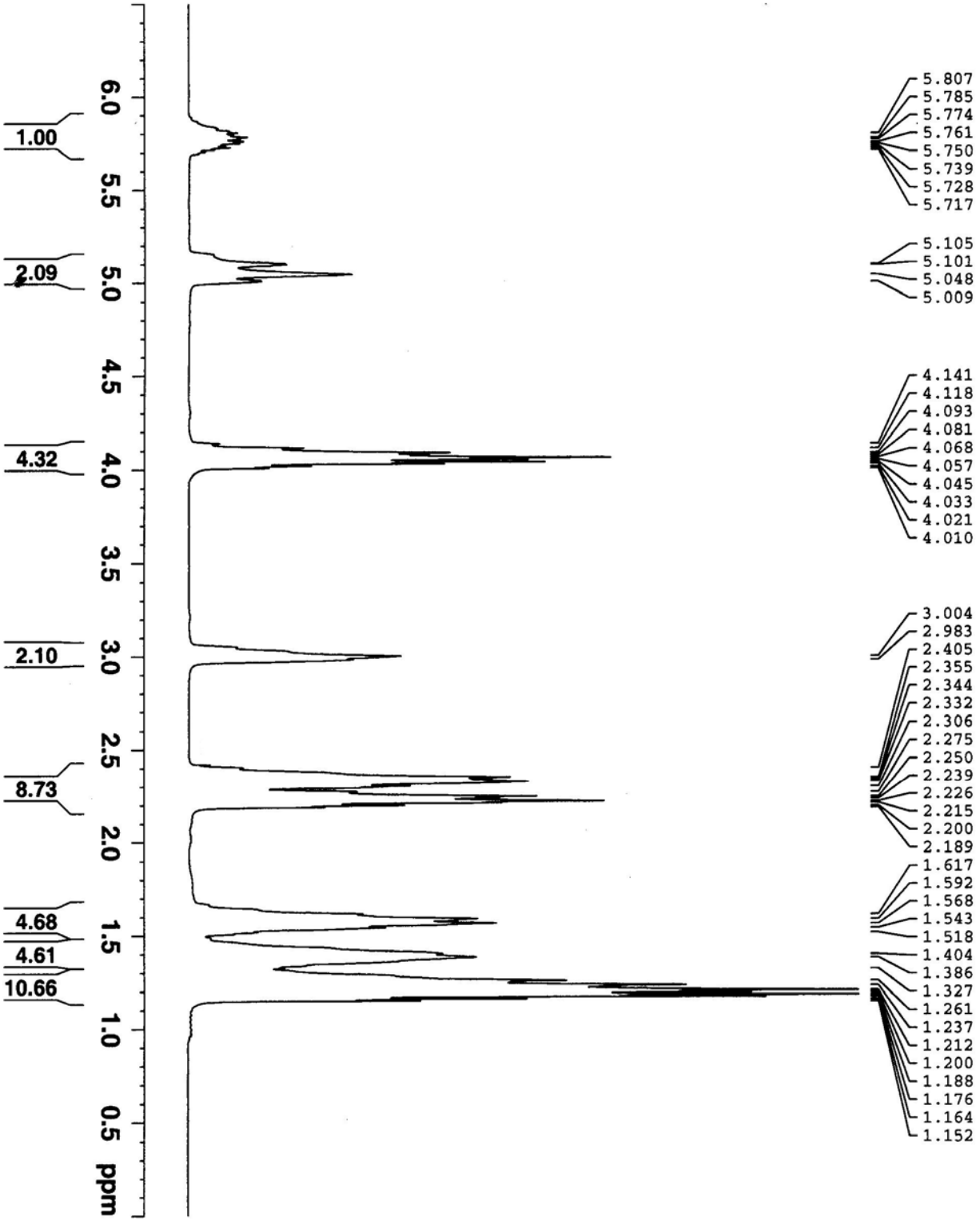

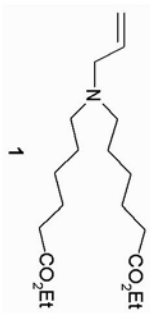


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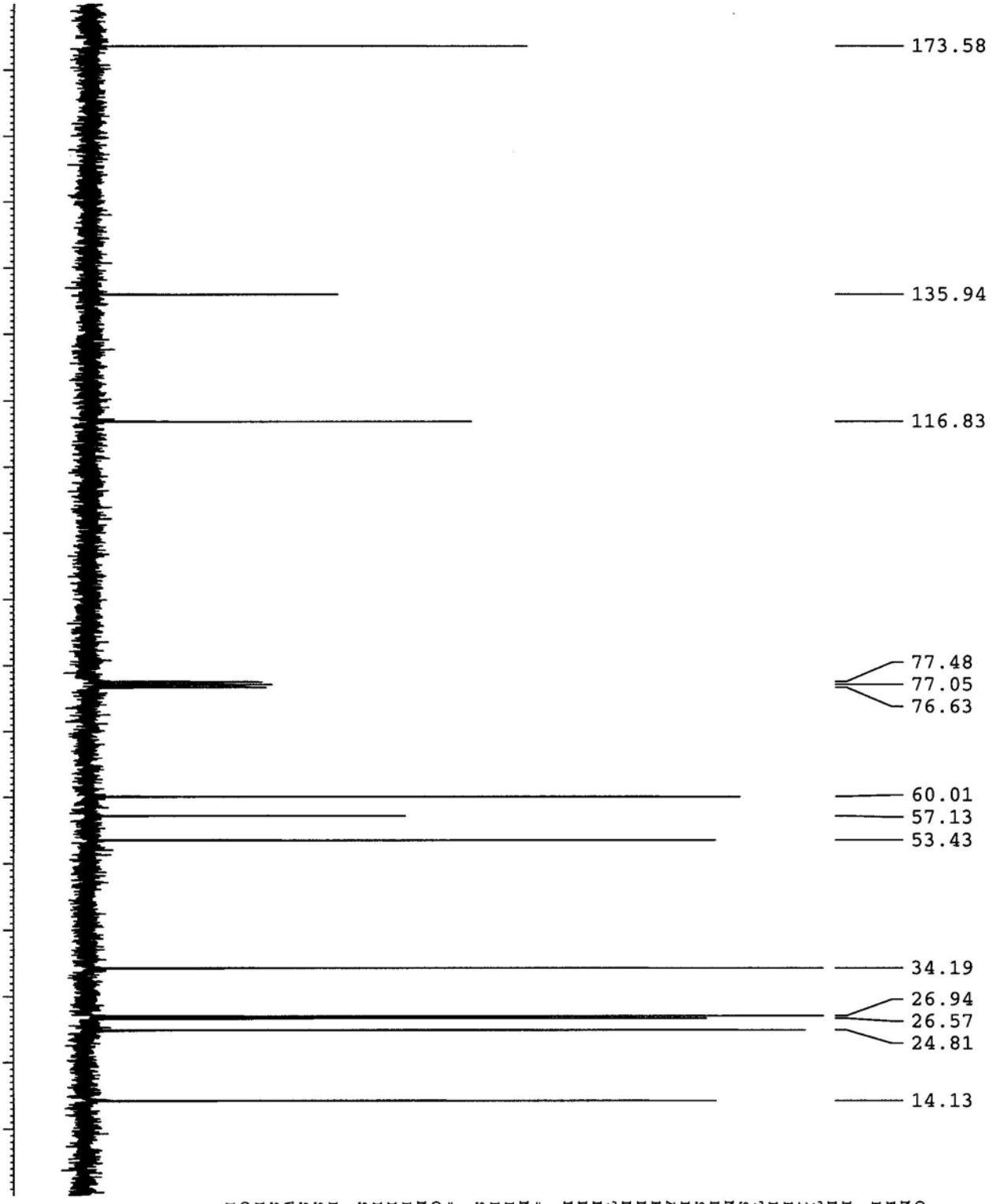

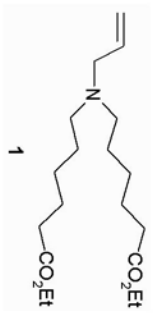

13C NMR

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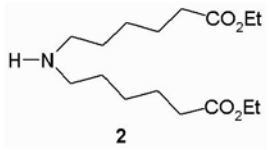
13C



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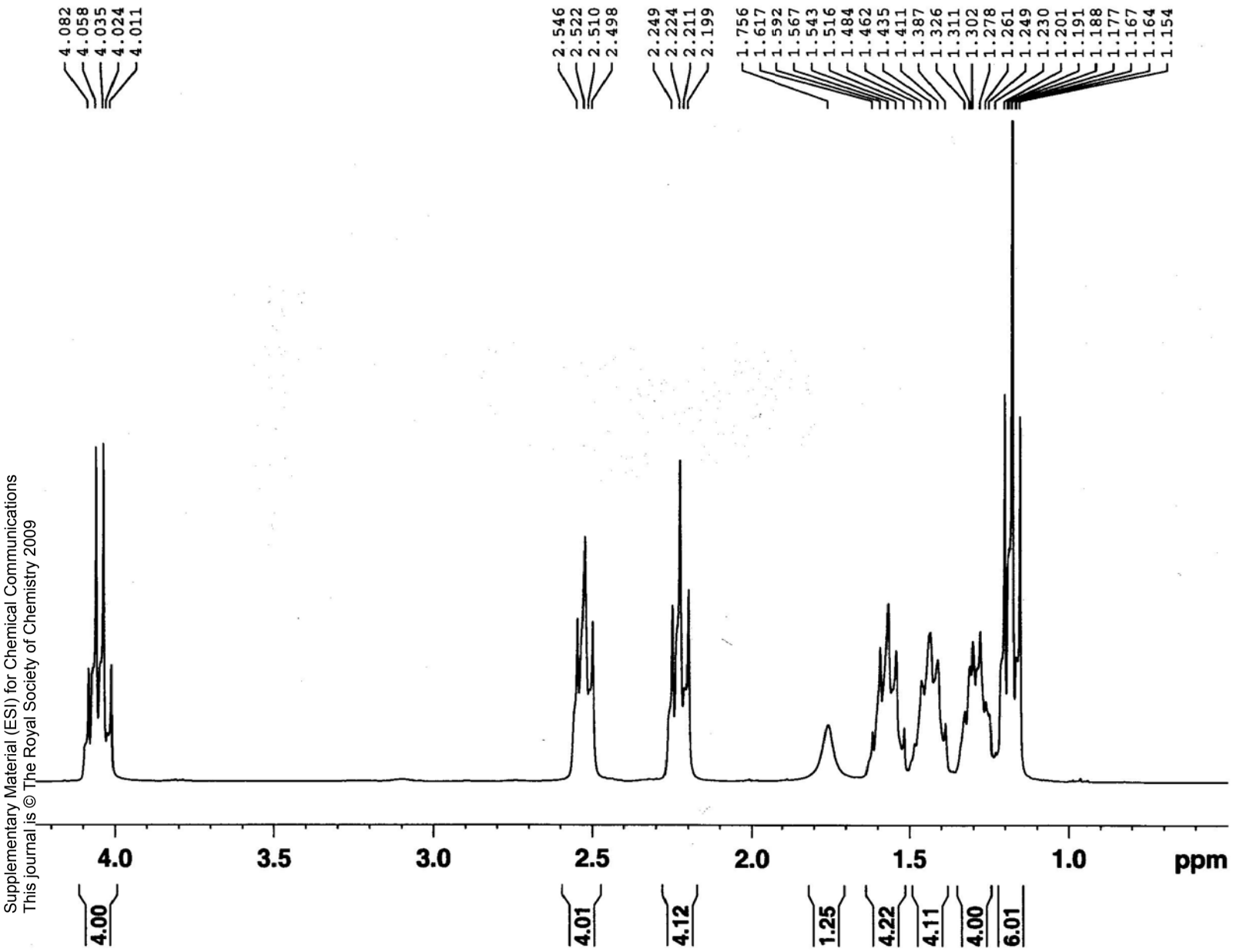


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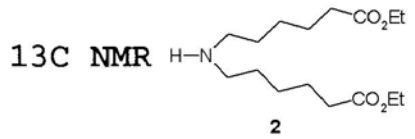
13C NMR



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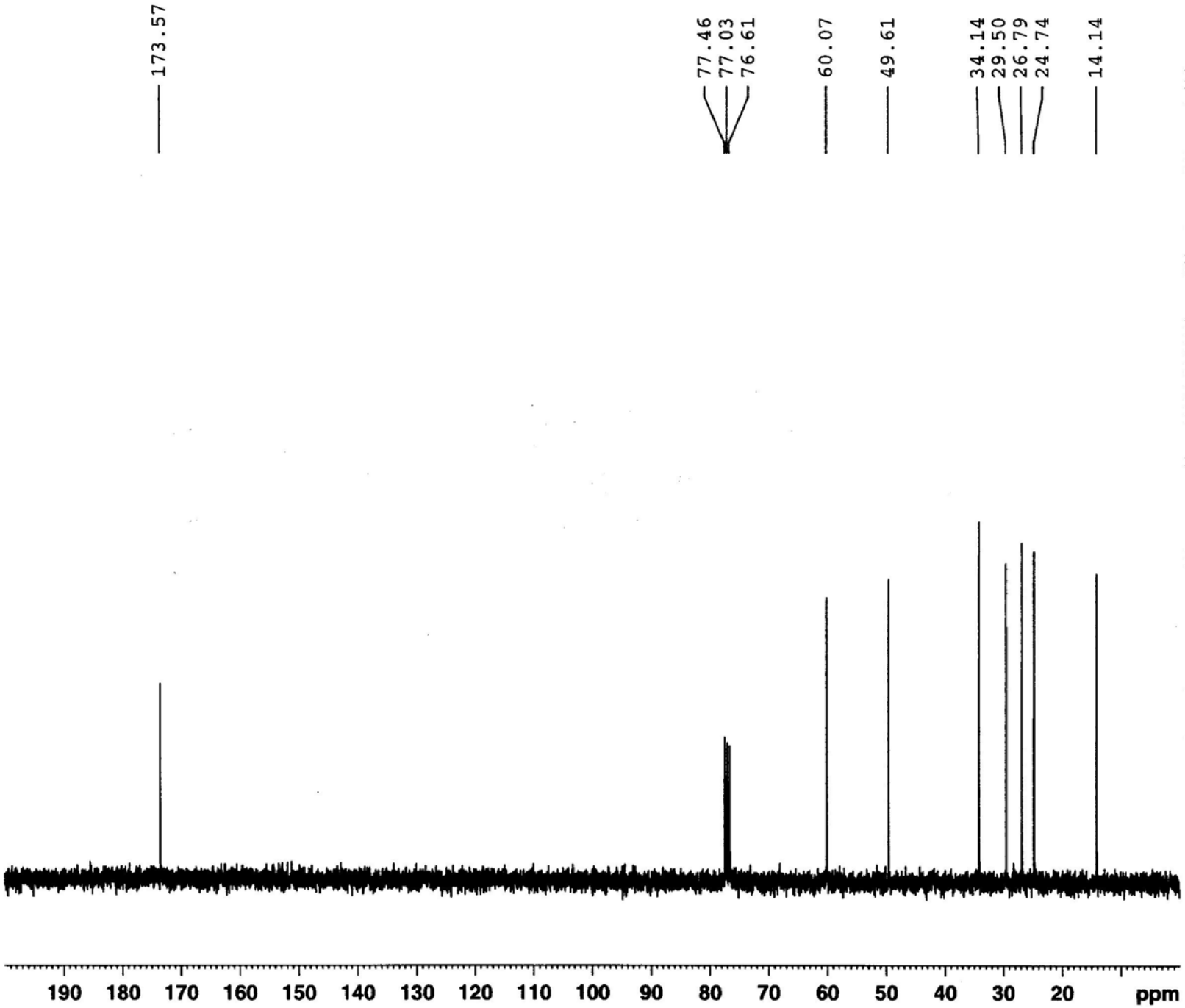


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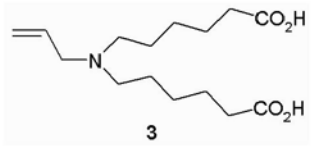
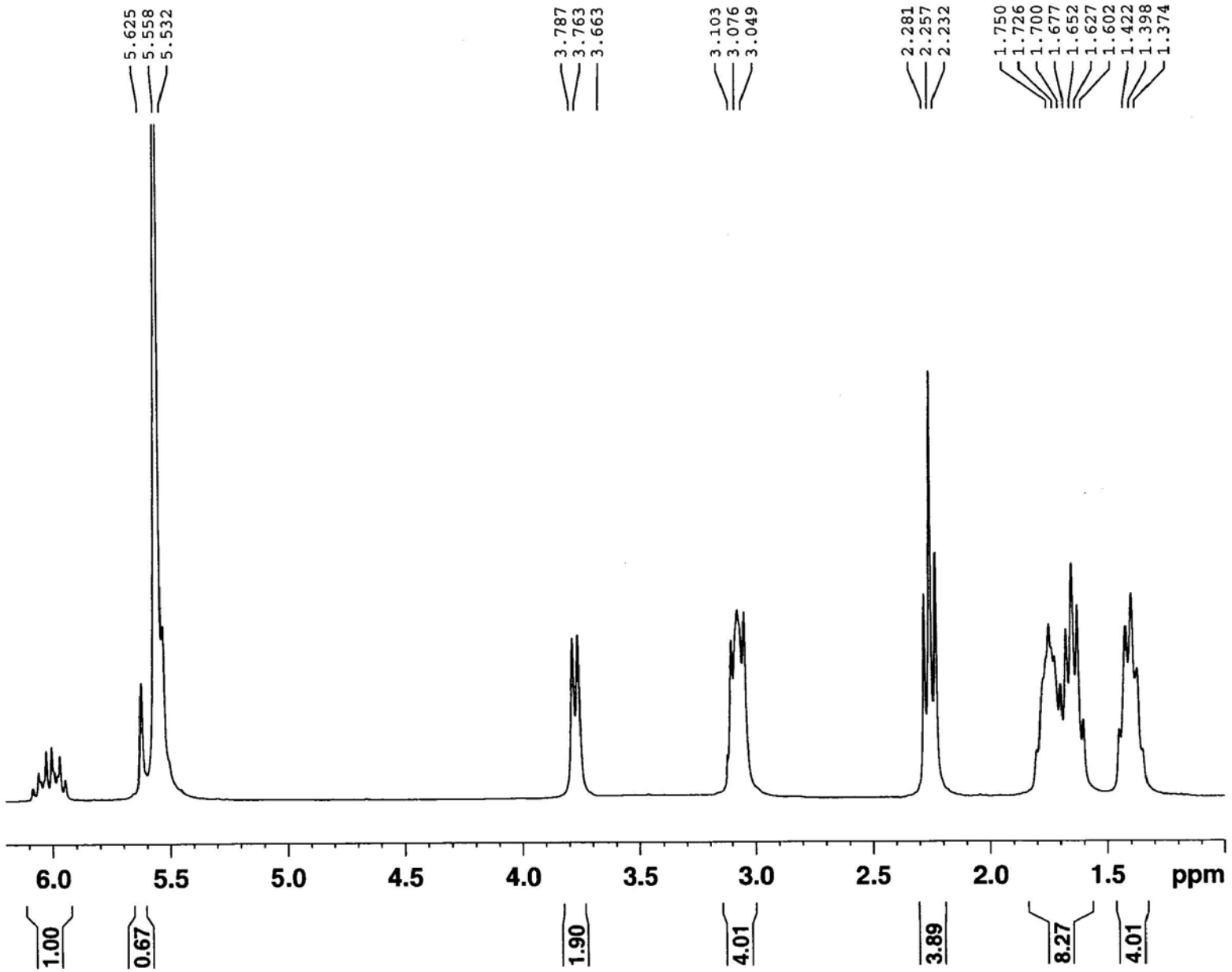


1H NMR

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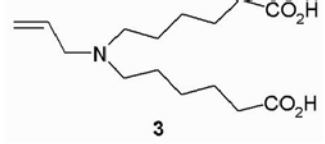

3

13C NMR

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125.17
79.30
78.86
78.43
54.84
51.93
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39.92
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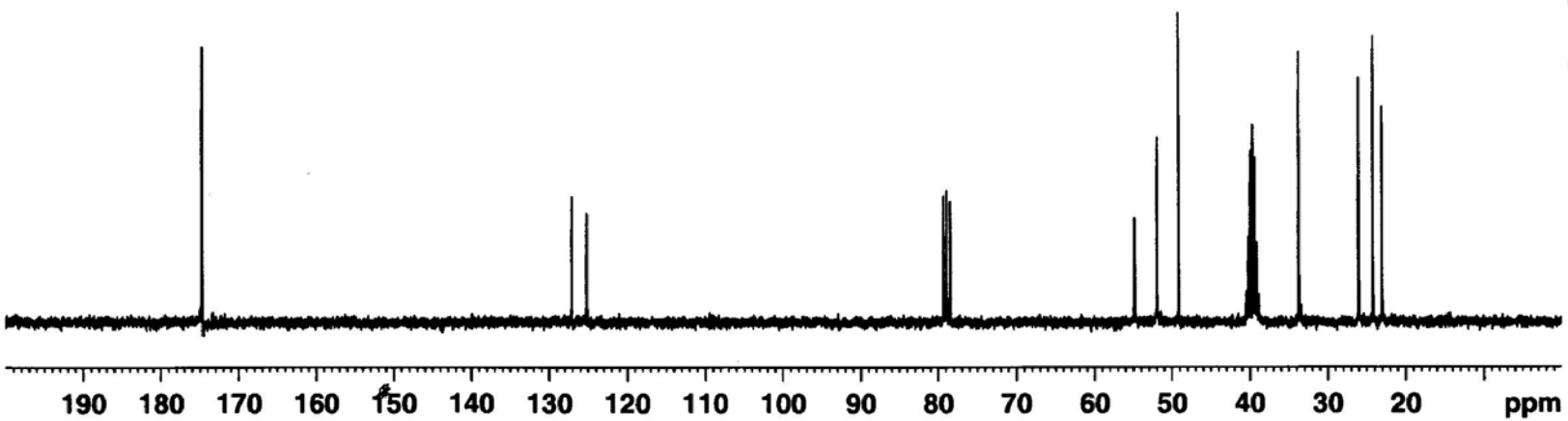
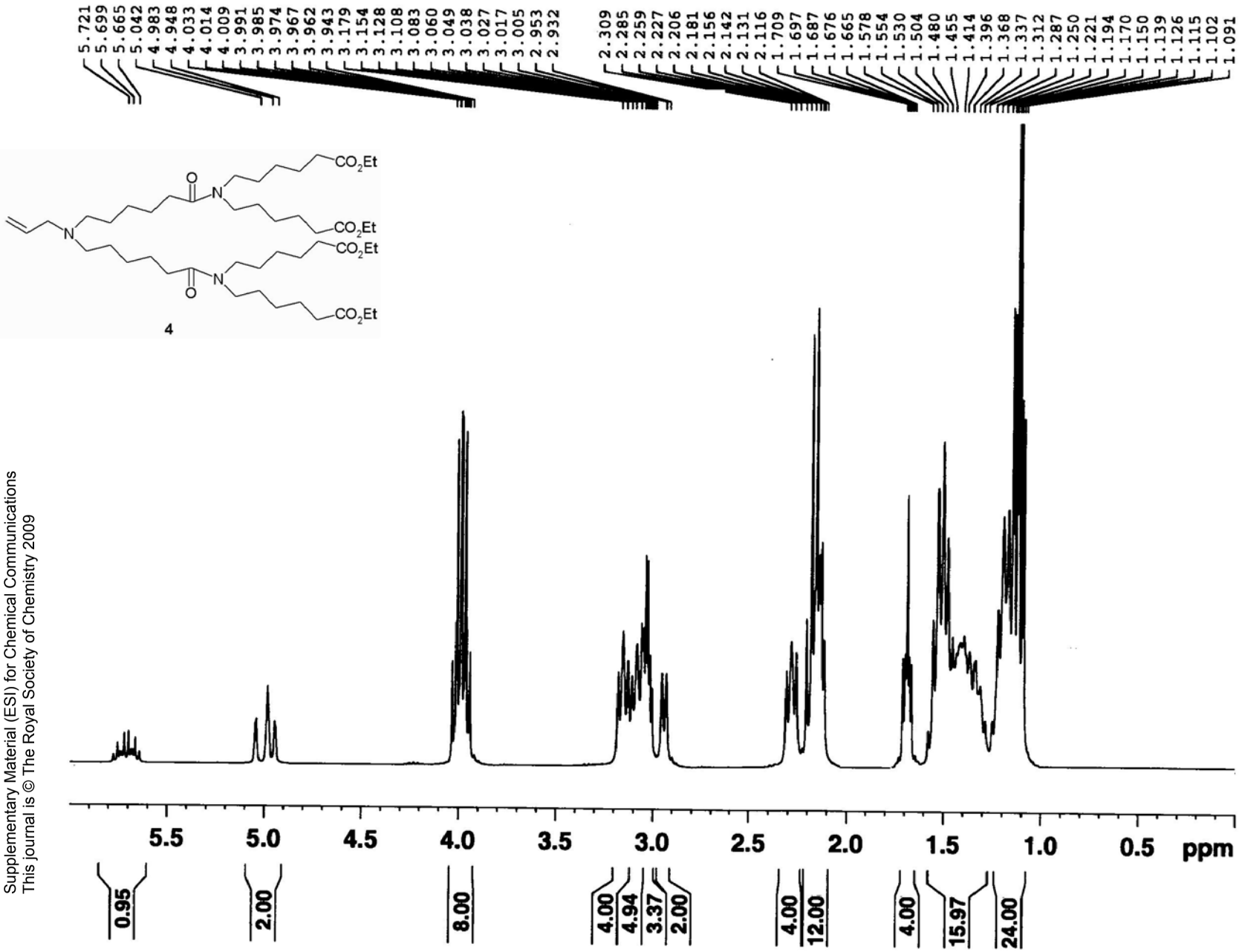


1H NMR

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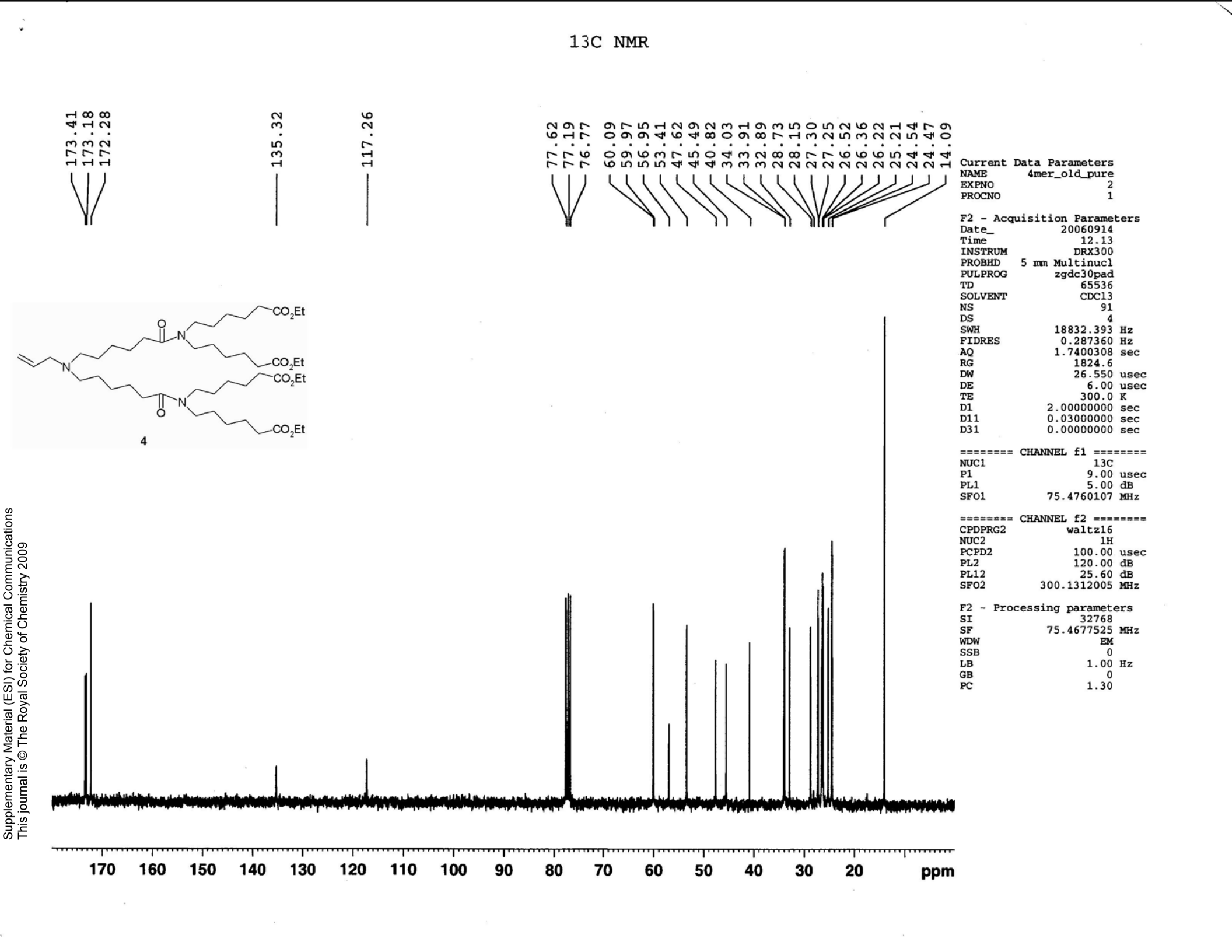
PULPROG                zg30pad
TD                       32768
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                 6172.839 Hz
FIDRES              0.188380 Hz
AQ                  2.6542580 sec
RG                        28.5
DW                   81.000 usec
DE                    6.00 usec
TE                    300.0 K
D1                  1.00000000 sec
D31                 0.00000000 sec

======== CHANNEL f1 ========
NUC1                        1H
P1                    7.05 usec
PL1                    0.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                       32768
SF            300.1300022 MHz
WDW                         EM
SSB                          0
LB                    0.30 Hz
GB                           0
PC                        1.30
```

13C NMR



Current Data Parameters

NAME	4mer_old_pure
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20060914
Time	12.13
INSTRUM	DRX300
PROBHD	5 mm Multinucl
PULPROG	zgdc30pad
TD	65536
SOLVENT	CDC13
NS	91
DS	4
SWH	18832.393 Hz
FIDRES	0.287360 Hz
AQ	1.7400308 sec
RG	1824.6
DW	26.550 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
D31	0.00000000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	9.00 usec
PL1	5.00 dB
SFO1	75.4760107 MHz

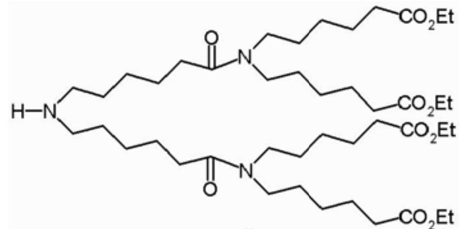
======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	25.60 dB
SFO2	300.1312005 MHz

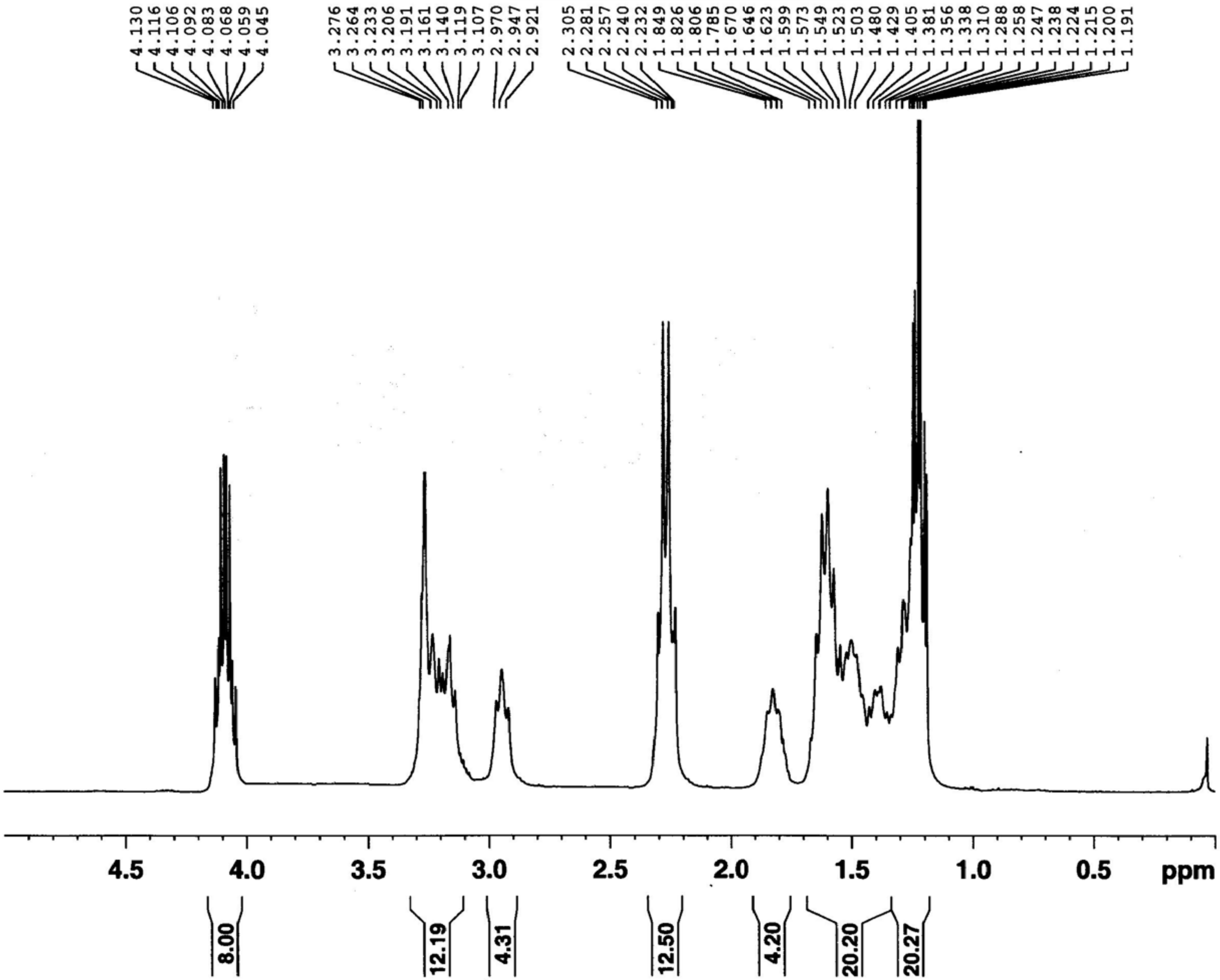
F2 - Processing parameters

SI	32768
SF	75.4677525 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.30

Supplementary Material (ESI) for Chemical Communications



5

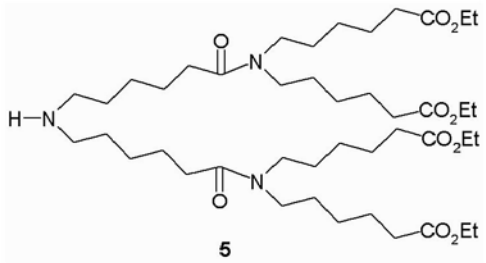



```
Current Data Parameters
NAME          NH4mer_spot1
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20070623
Time                 17.35
INSTRUM             DRX300
PROBHD   5 mm Multinucl
PULPROG            zg30pad
TD                   32768
SOLVENT              CDC13
NS                      16
DS                       2
SWH            6172.839 Hz
FIDRES         0.188380 Hz
AQ           2.6542580 sec
RG                    90.5
DW              81.000 usec
DE                6.00 usec
TE                 300.0 K
D1          1.00000000 sec
D31         0.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                7.05 usec
PL1                0.00 dB
SFO1        300.1318534 MHz

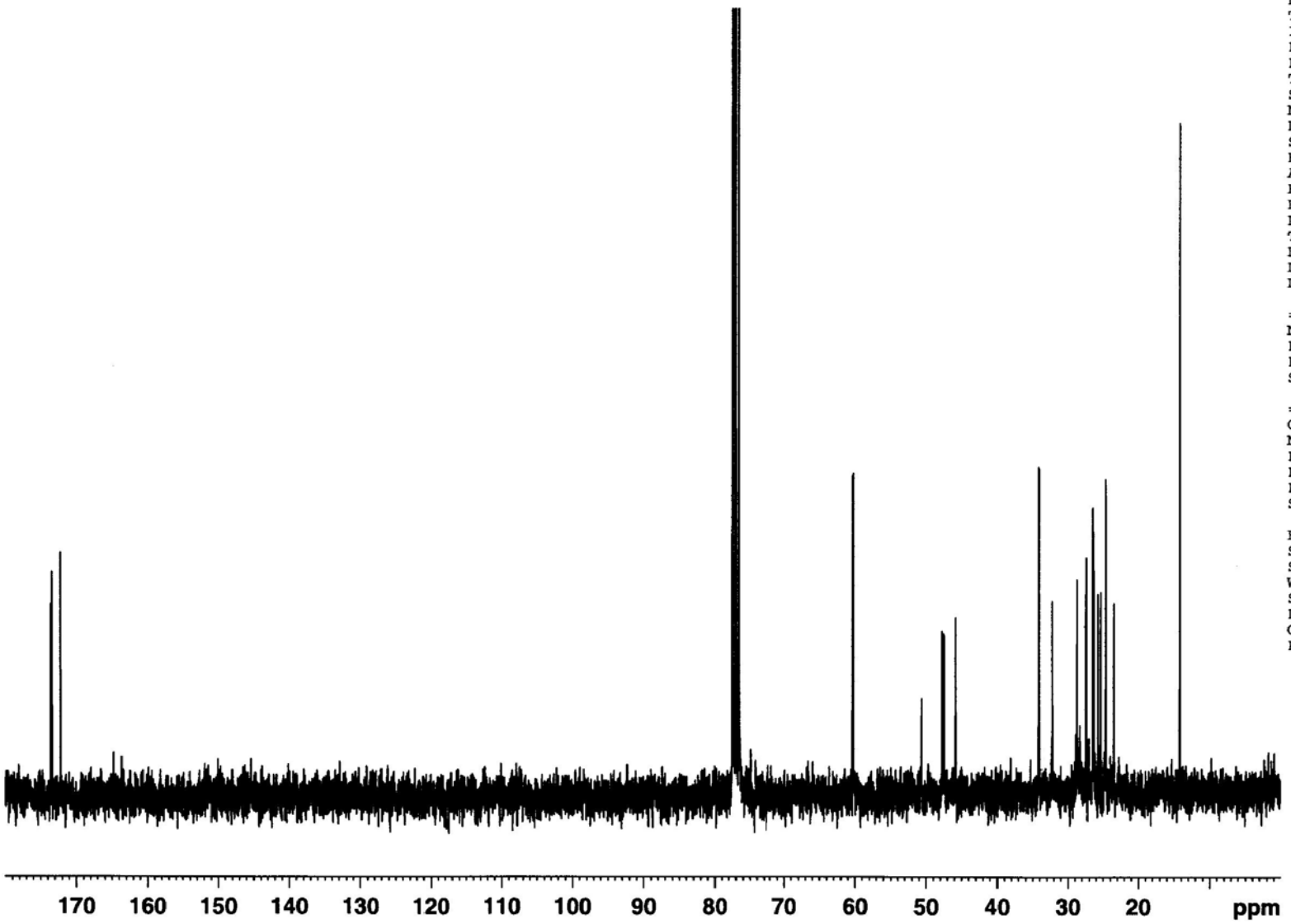
F2 - Processing parameters
SI                   32768
SF          300.1300022 MHz
WDW                     EM
SSB                      0
LB                 0.30 Hz
GB                       0
PC                    1.40
```



5

13C NMR

173.61
173.42
172.26
77.44
77.02
76.59
60.31
60.19
50.59
47.72
47.44
45.82
34.12
34.00
32.23
28.68
27.36
26.47
26.32
25.70
25.27
24.60
24.53
23.48
14.18



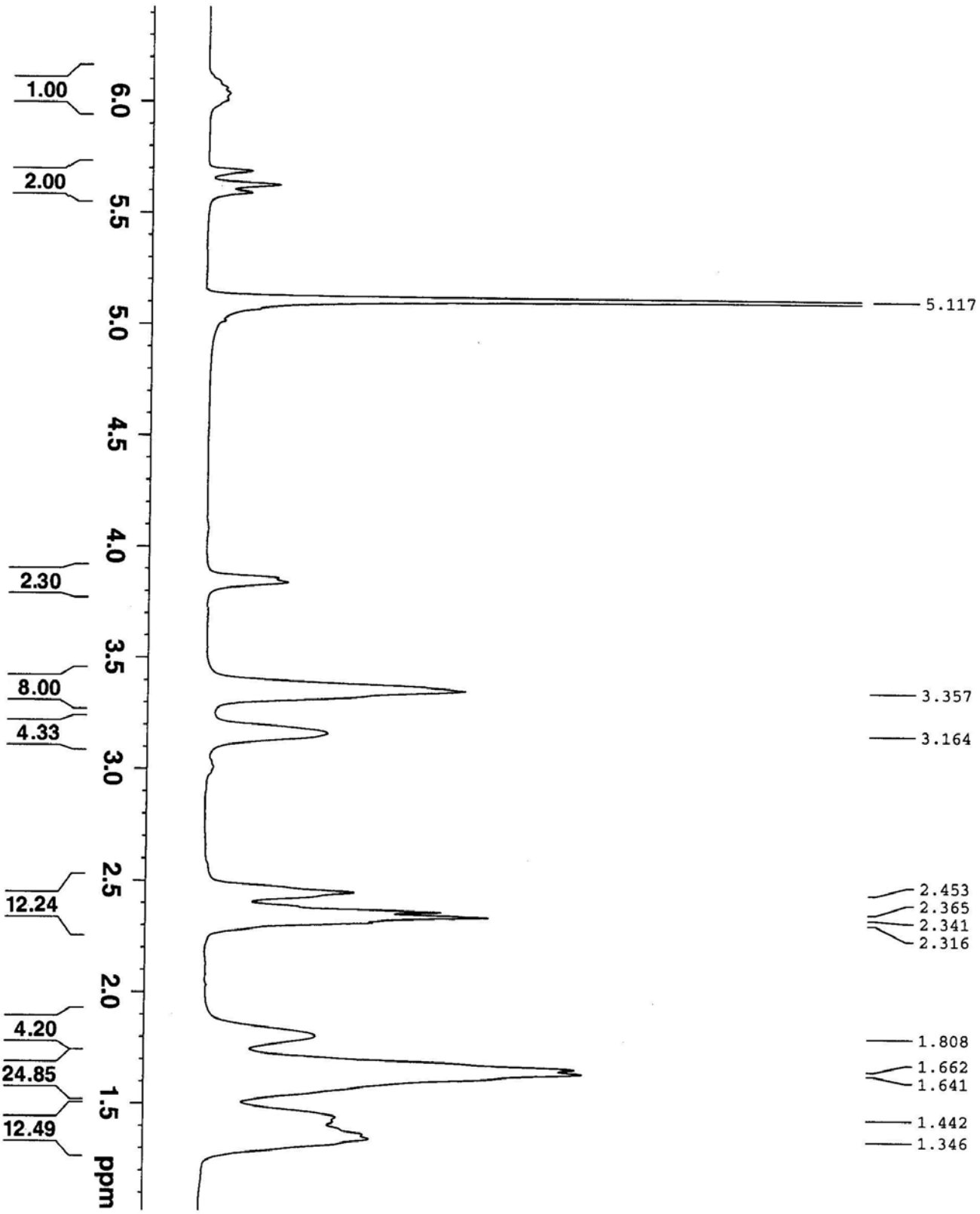
Current Data Parameters
NAME NH_4mer_col_3
EXPNO 2
PROCNO 1

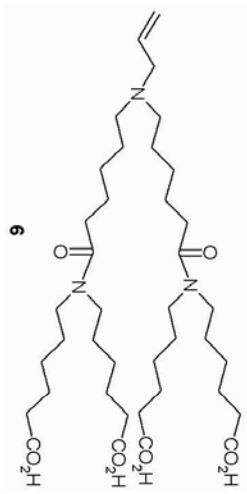
F2 - Acquisition Parameters
Date_ 20070622
Time 16.11
INSTRUM DRX300
PROBHD 5 mm Multinucl
PULPROG zgdc30pad
TD 65536
SOLVENT CDCl3
NS 318
DS 4
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 4096
DW 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D31 0.00000000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 5.00 dB
SFO1 75.4760107 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 21.41 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677525 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.30



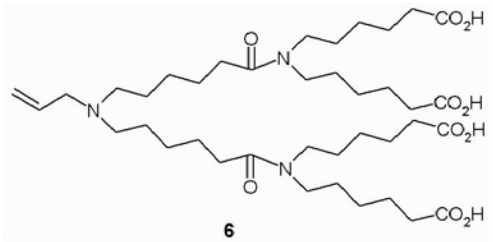



```
Current Data Parameters
NAME                 4mer_acid
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_               20060914
Time                   11.40
INSTRUM                DRX300
PROBHD     5 mm Multinucl
PULPROG             zg30pad
TD                    32768
SOLVENT                MeOD
NS                       16
DS                        2
SWH                6172.839 Hz
FIDRES                0.188380 Hz
AQ                 2.6542580 sec
RG                      50.8
DW                   81.000 usec
DE                    6.00 usec
TE                   300.0 K
D1                   1.00000000 sec
D31                  0.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                    7.05 usec
PL1                   0.00 dB
SFO1           300.1318534 MHz

F2 - Processing parameters
SI                    32768
SF             300.1300022 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.40
```



13C NMR

175.92
174.42
173.76
126.29
125.27
54.93
52.26
48.50
48.21
47.93
47.64
47.36
47.08
46.79
45.84
33.37
33.24
33.22
32.98
32.00
28.24
26.86
26.00
25.83
24.58
24.28
23.96

```
Current Data Parameters
NAME                 4mer_acid
EXPNO                        2
PROCNO                       1

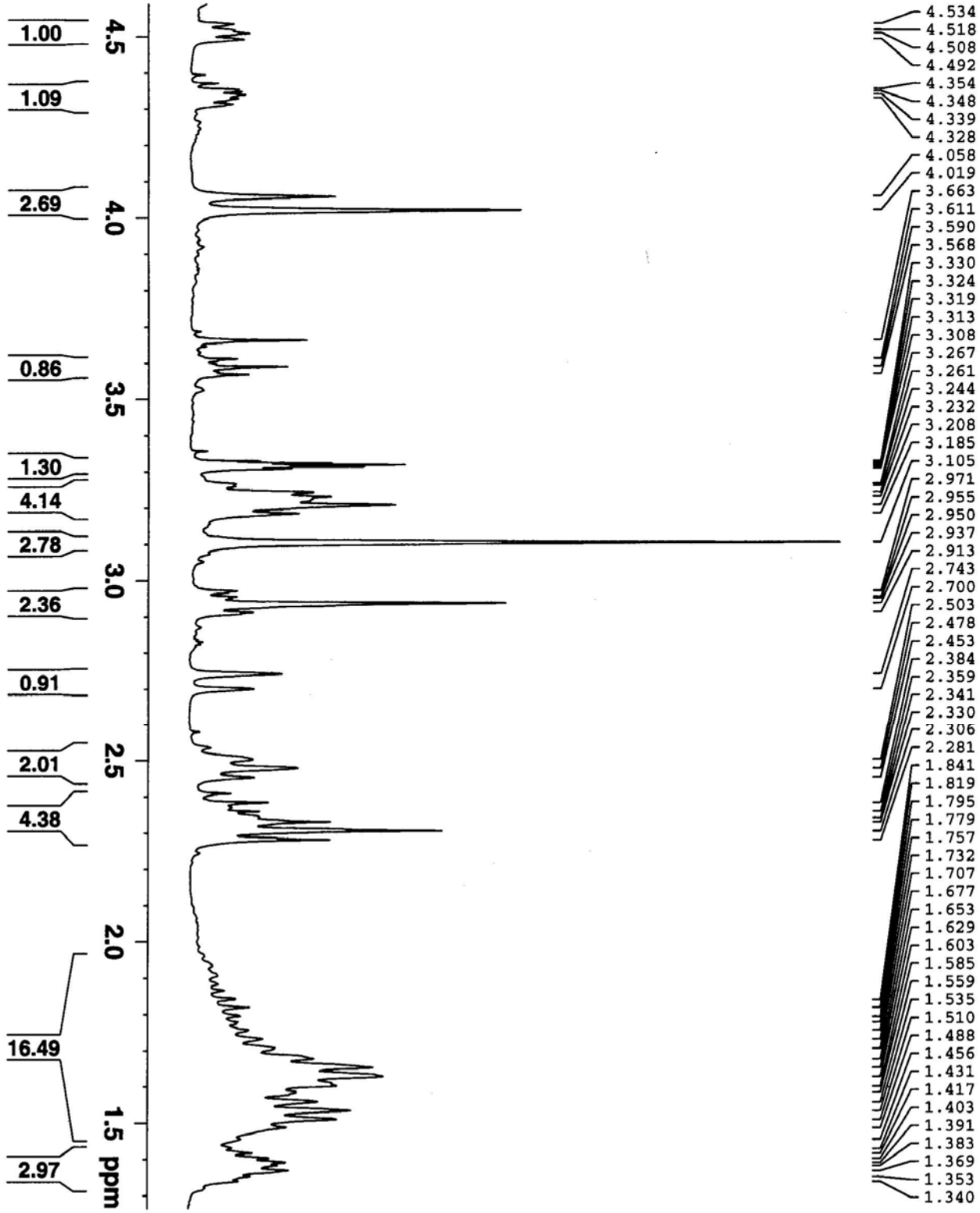
F2 - Acquisition Parameters
Date_                 20060914
Time                     11.43
INSTRUM                DRX300
PROBHD    5 mm Multinucl
PULPROG              zgdc30pad
TD                       65536
SOLVENT                  CDCl3
NS                         242
DS                           4
SWH                  18832.393 Hz
FIDRES                0.287360 Hz
AQ                    1.7400308 sec
RG                       1149.4
DW                       26.550 usec
DE                        6.00 usec
TE                       300.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
D31                   0.00000000 sec

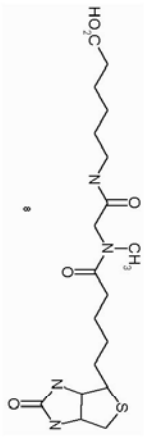
======== CHANNEL f1 ========
NUC1                        13C
P1                         9.00 usec
PL1                        5.00 dB
SFO1                 75.4760107 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    100.00 usec
PL2                      120.00 dB
PL12                      25.60 dB
SFO2                300.1312005 MHz

F2 - Processing parameters
SI                         32768
SF                   75.4677525 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.30
```


170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



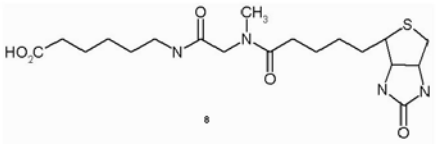



```
Current Data Parameters
NAME                  BioSarAHex_500mg
EXPNO                              1
PROCNO                             1

F2 - Acquisition Parameters
Date_                       20070712
Time                           17.31
INSTRUM                       DRX300
PROBHD     5 mm Multinucl
PULPROG                    zg30pad
TD                           32768
SOLVENT                       MeOD
NS                               16
DS                                2
SWH                        6172.839 Hz
FIDRES                     0.188380 Hz
AQ                         2.6542580 sec
RG                             161.3
DW                         81.000 usec
DE                          6.00 usec
TE                             300.0 K
D1                         1.00000000 sec
D31                        0.00000000 sec

======== CHANNEL f1 ========
NUC1                             1H
P1                          7.05 usec
PL1                          0.00 dB
SFO1                  300.1318534 MHz

F2 - Processing parameters
SI                           32768
SF                    300.1300022 MHz
WDW                             EM
SSB                              0
LB                          0.30 Hz
GB                               0
PC                            1.30
```



13C NMR

174.80
169.54
168.99
164.62
163.67
163.28
162.88

78.31
77.87
77.44
61.83
60.64
60.19
55.57
52.38
50.60
48.58
48.30
48.01
47.73
47.45
47.16
46.88
39.74
38.96
38.83
36.13
35.23
34.94
34.65
34.10
33.46
32.37
32.20
29.96
29.67
29.39

Current Data Parameters
NAME BioSarAHex_500mg
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070712
Time 17.34
INSTRUM DRX300
PROBHD 5 mm Multinucl
PULPROG zgdc30pad
TD 65536
SOLVENT CDCl3
NS 250
DS 4
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 1824.6
DW 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D31 0.00000000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 5.00 dB
SFO1 75.4760107 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 21.41 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677525 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm