

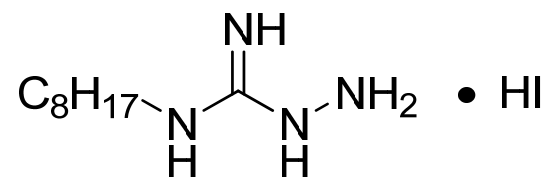
Supporting Data

Structure-Activity Relationships of Lipopolysaccharide Sequestration in Guanylhydrazone-bearing Lipopolyamines

*Wenyan Wu, Diptesh Sil, Michal L. Szostak, Lakshmi S. Malladi,
Hemamali J. Warshakoon,
Matthew R. Kimbrell, Jens R. Cromer, Sunil A. David**

¹H, ¹³C NMR, and Mass-Spectral Data
of all key intermediates and final compounds
and
LC-MS Data of final compounds

¹H NMR Spectrum

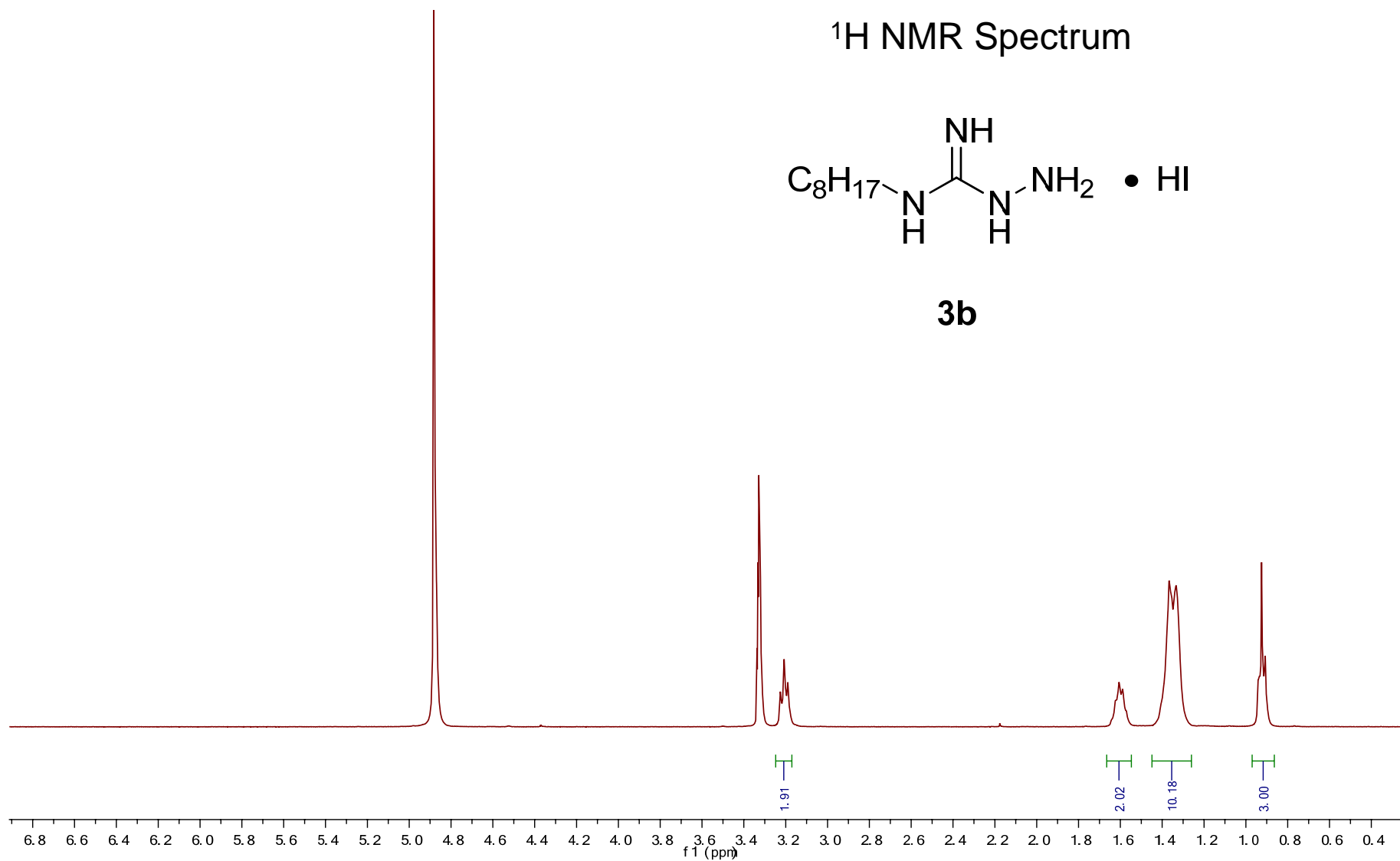


3.23
3.21
3.19

1.61
1.59

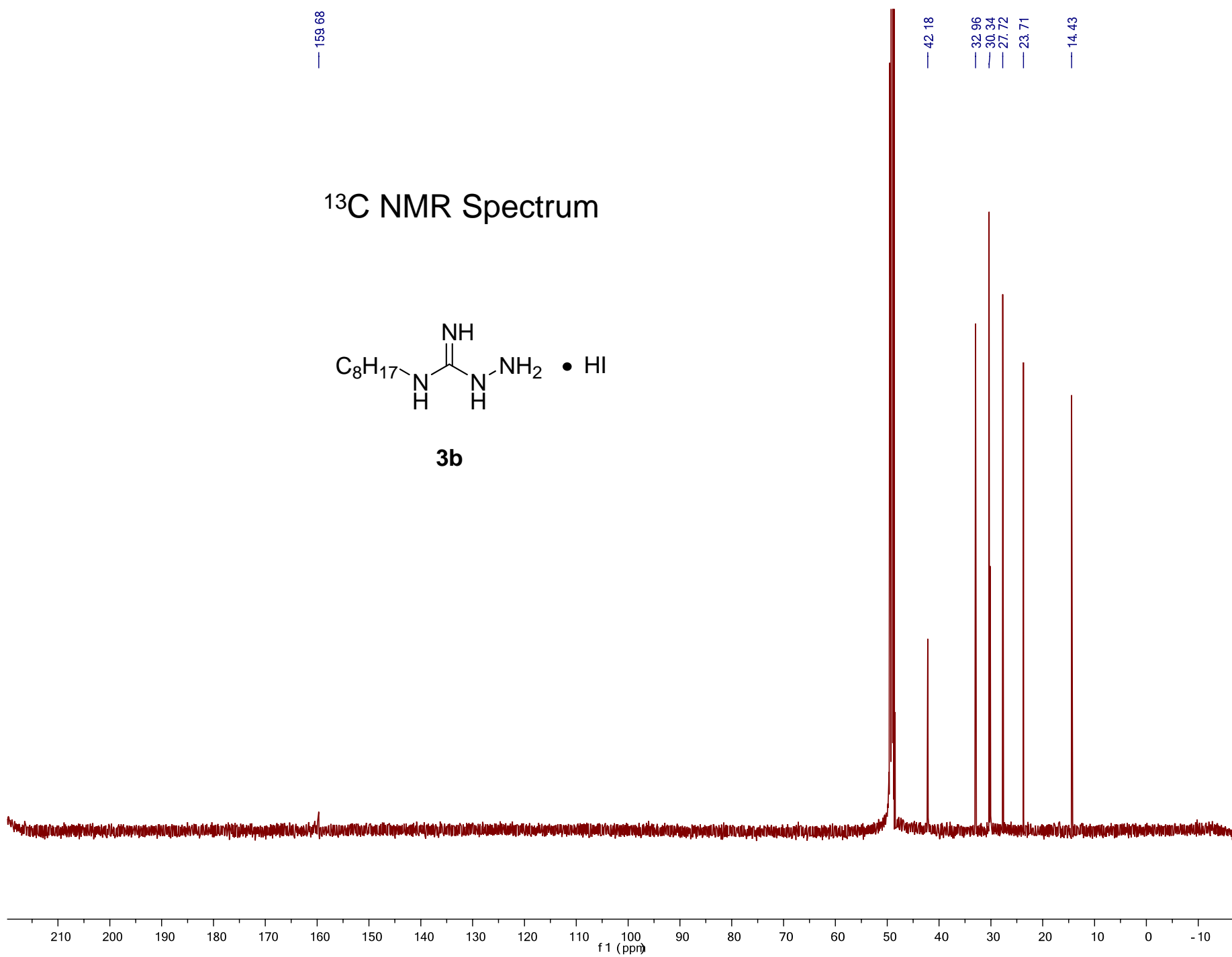
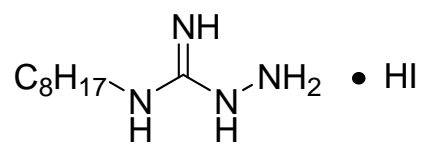
1.37
1.33

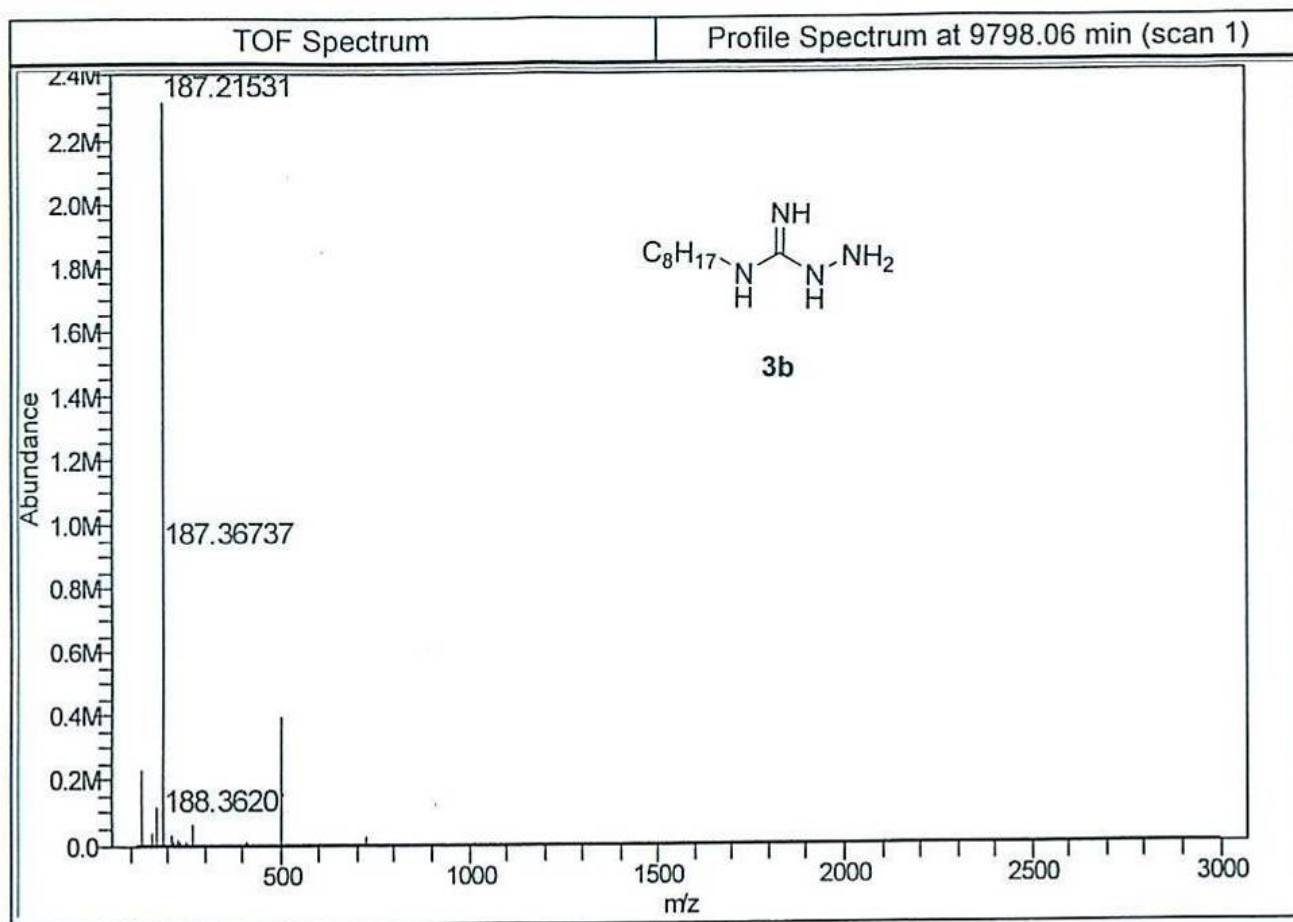
0.93
0.91



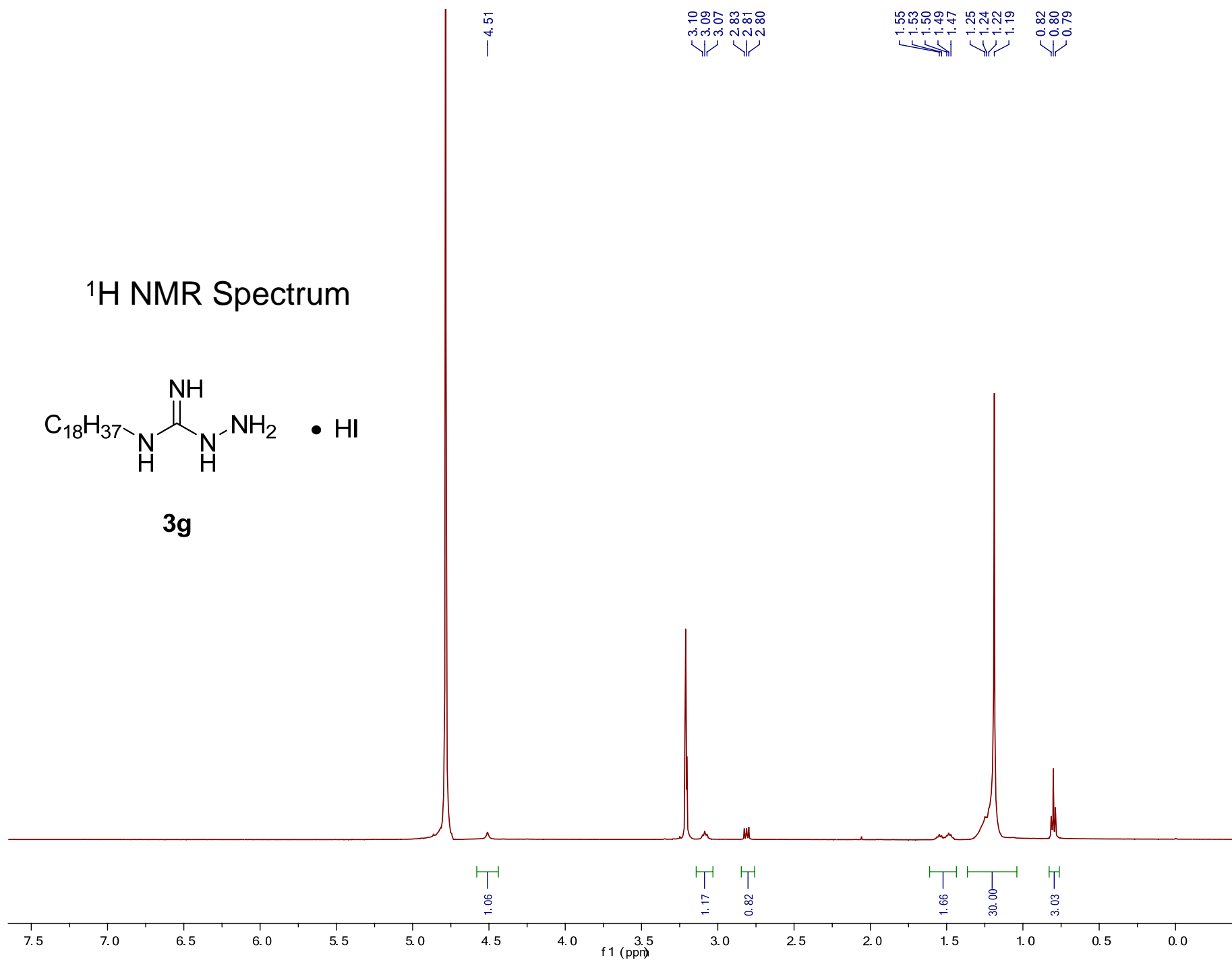
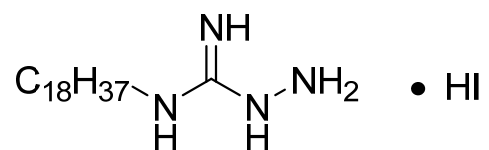
— 159.68

¹³C NMR Spectrum



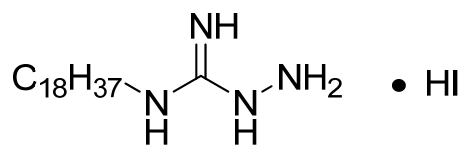


¹H NMR Spectrum

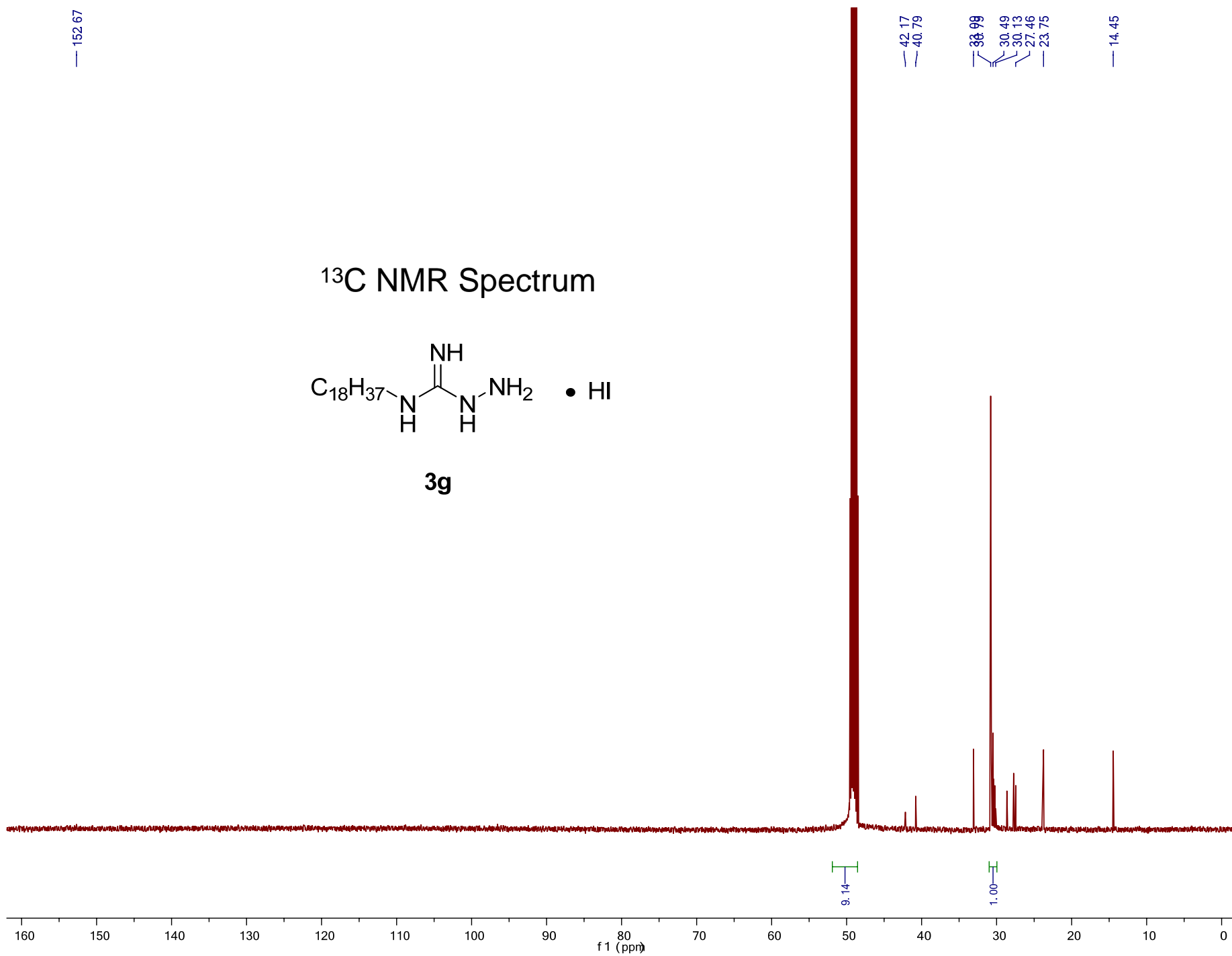


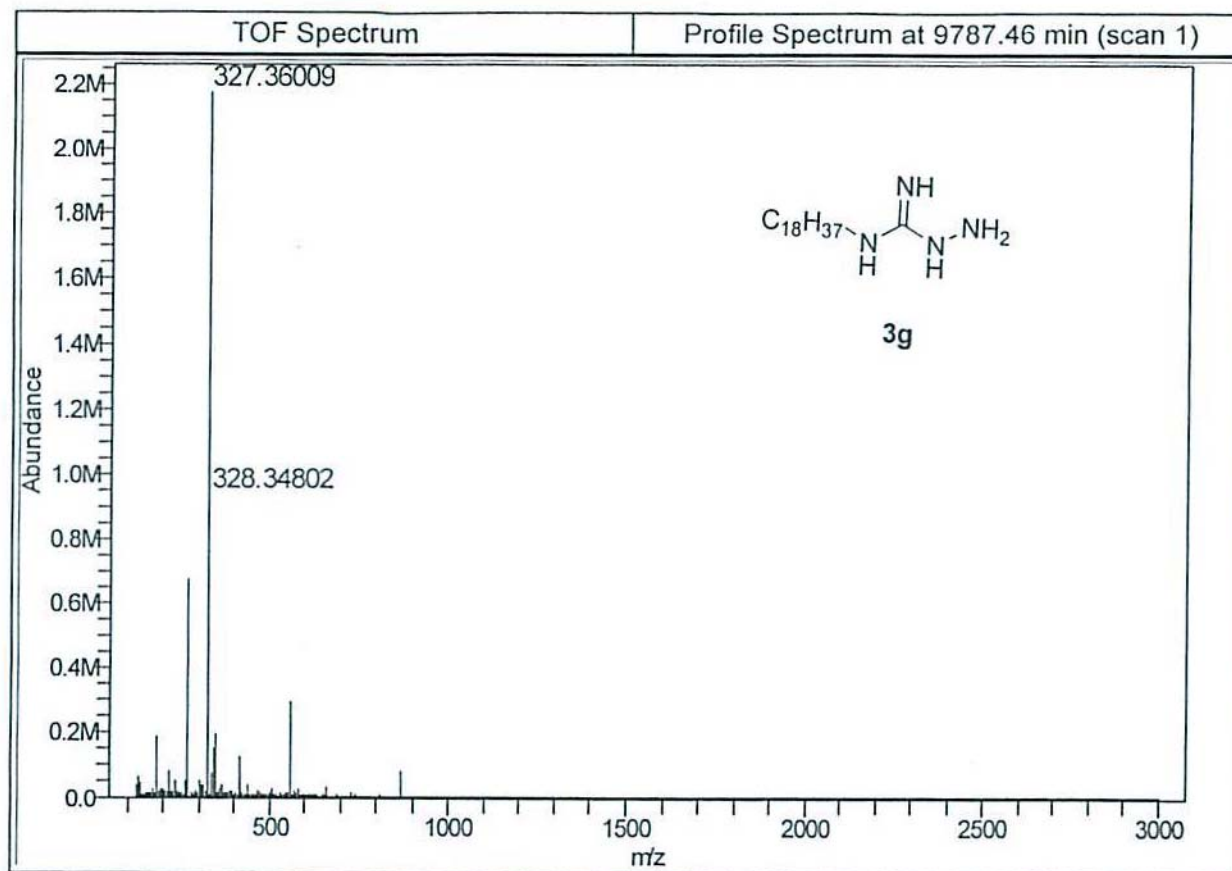
— 152.67

^{13}C NMR Spectrum

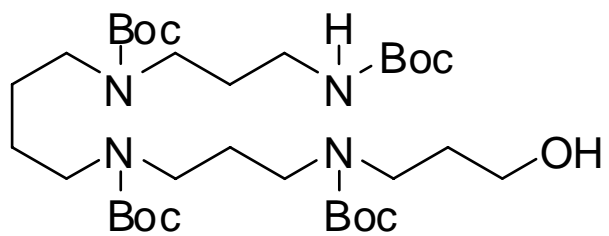


3g

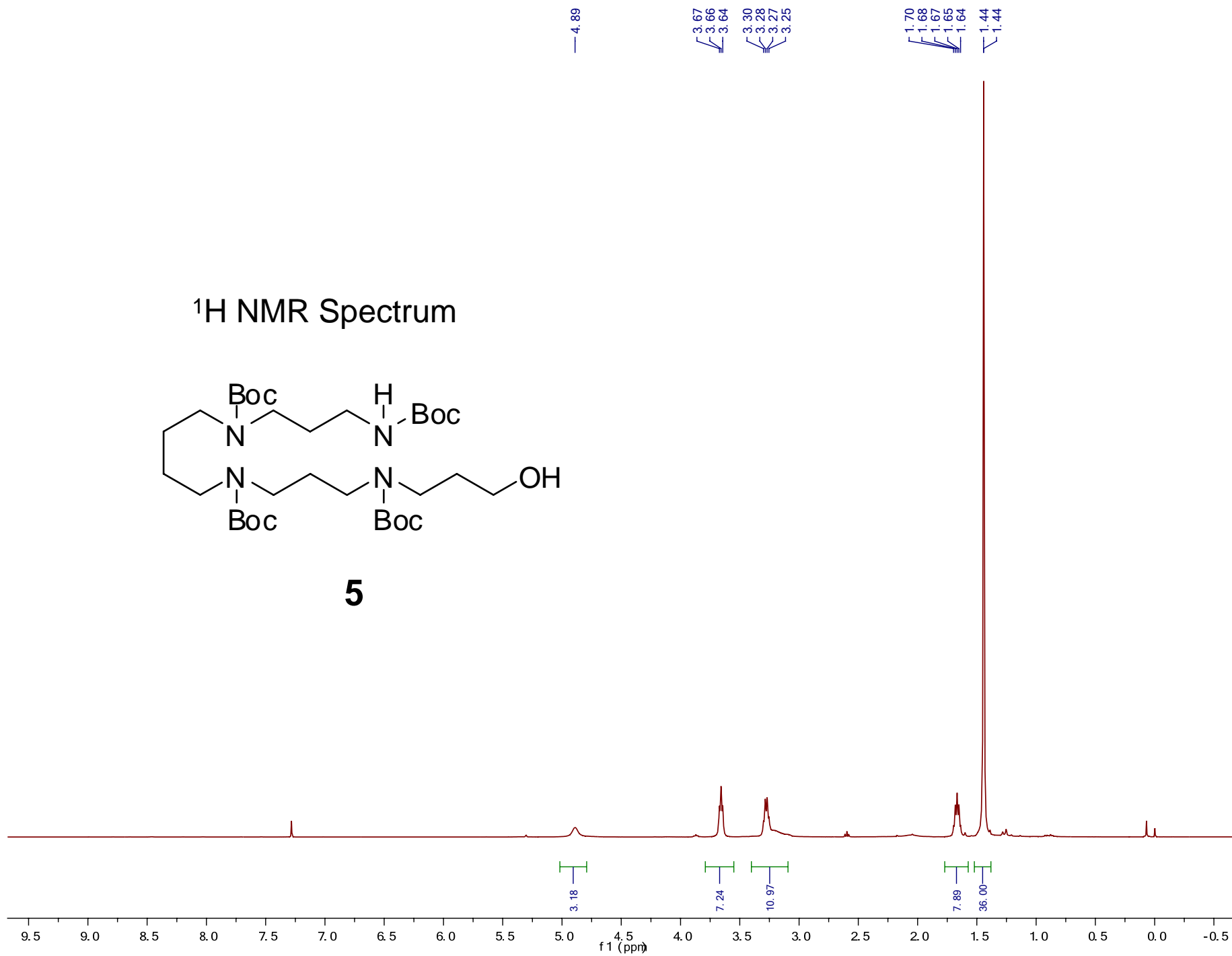




¹H NMR Spectrum



5



— 156.20

— 78.60

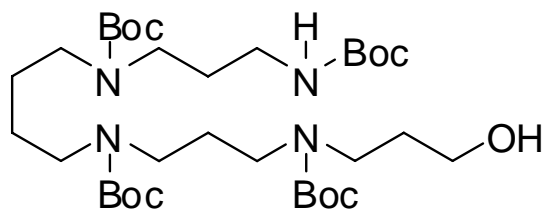
— 58.16

— 35.86

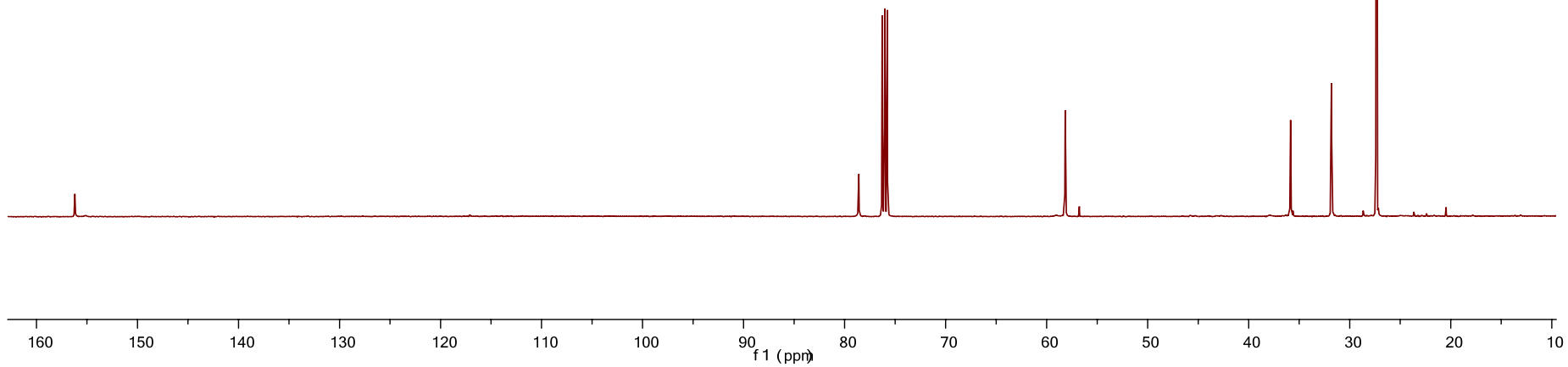
— 31.81

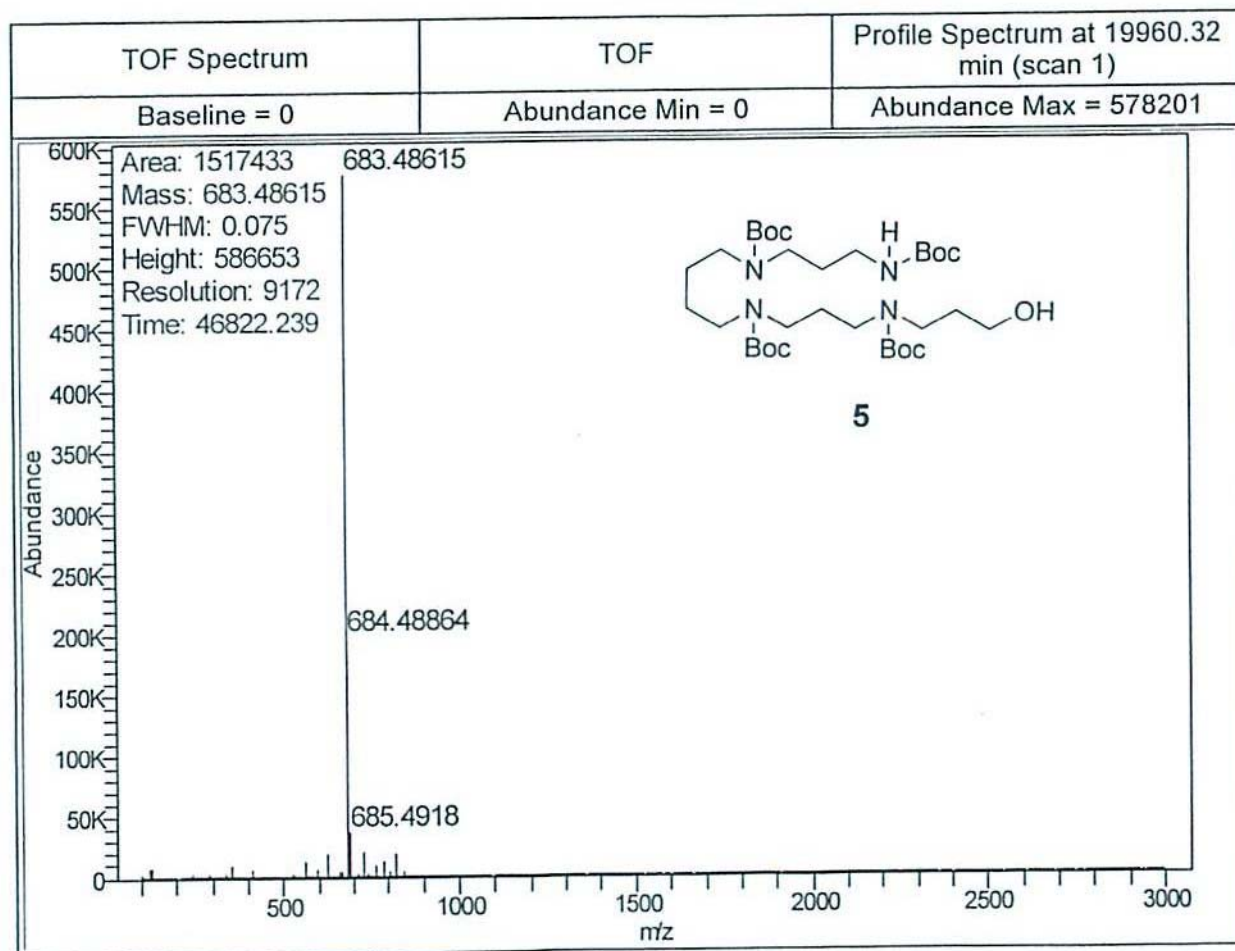
27.42
27.34

¹³C NMR Spectrum

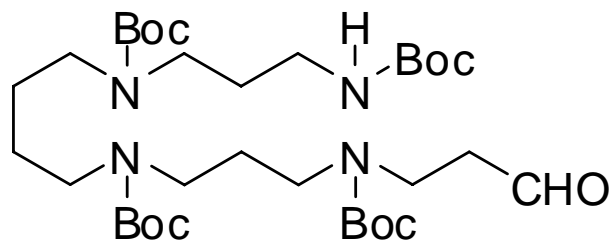


5

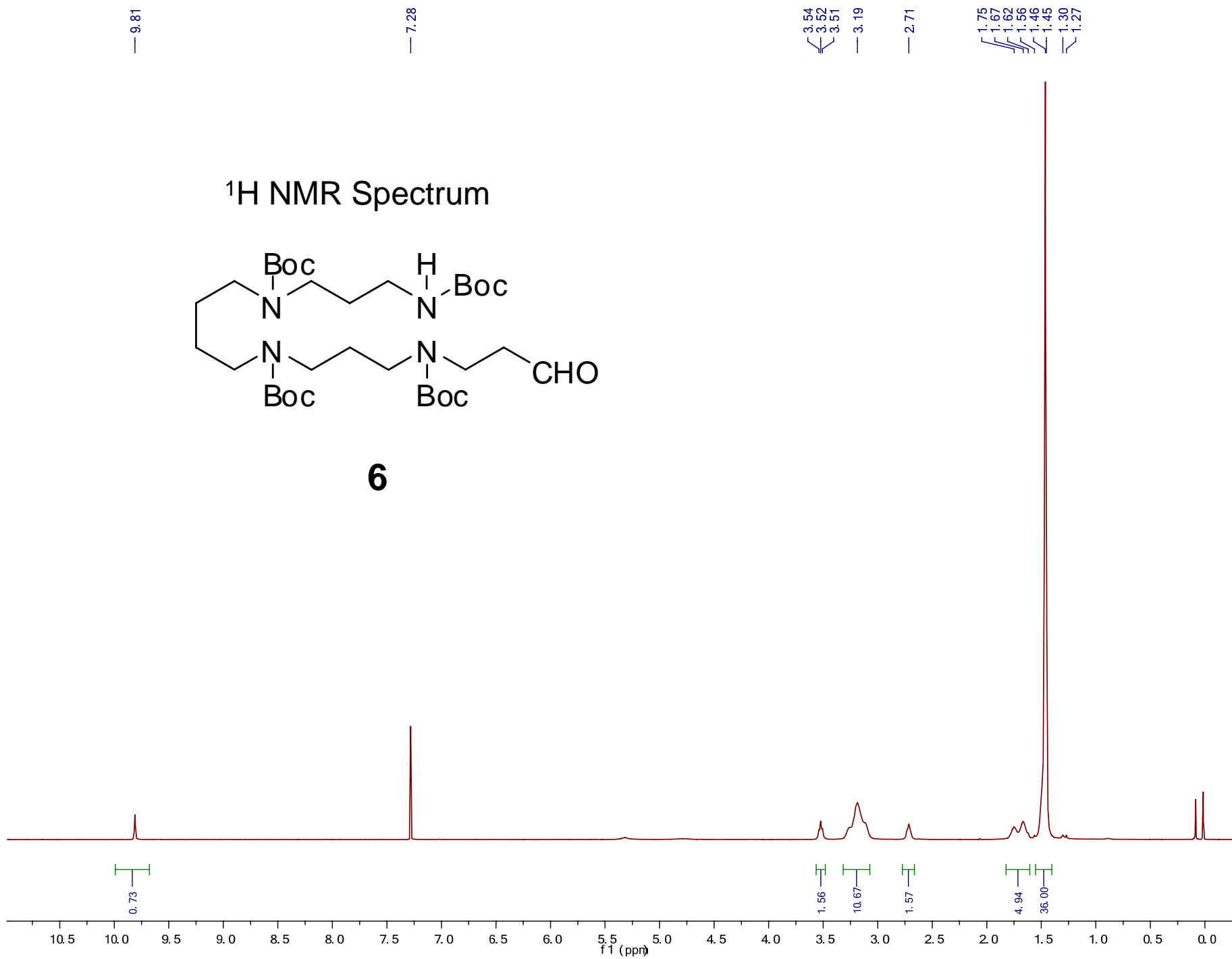




¹H NMR Spectrum



6



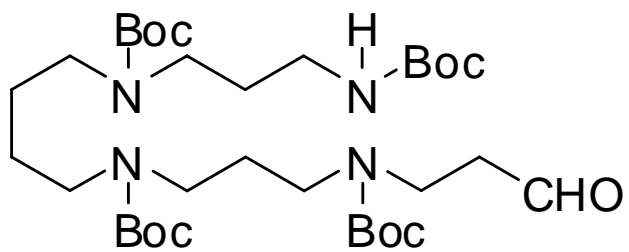
— 199.95

155.09
154.42

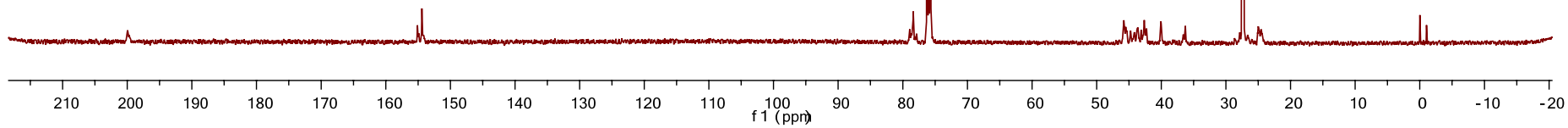
78.93
78.37

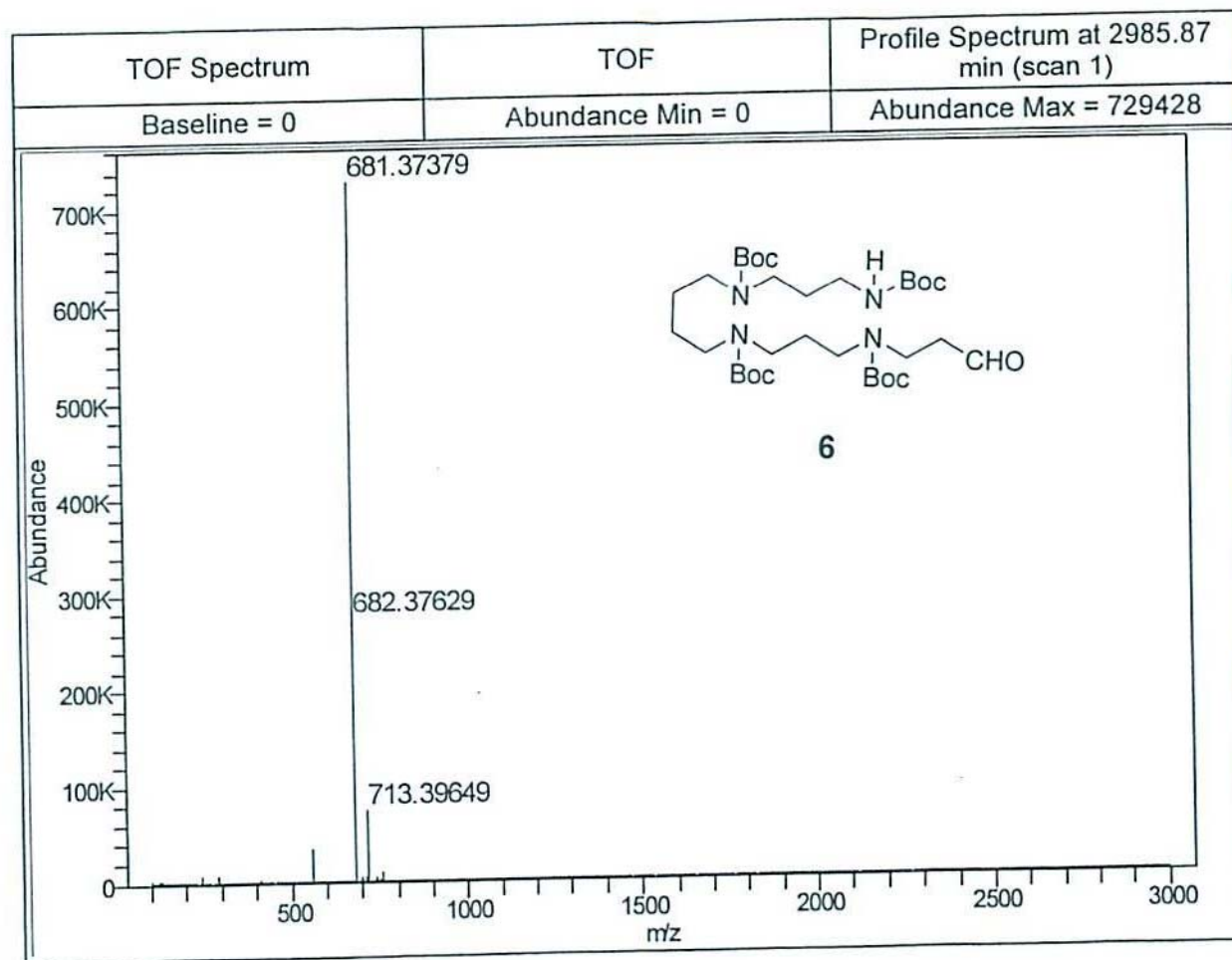
45.80
45.51
44.76
44.15
43.61
43.12
42.64
42.29
40.05
36.28
27.87
27.46
27.42
27.39
27.26
25.00
24.55

^{13}C NMR Spectrum



6





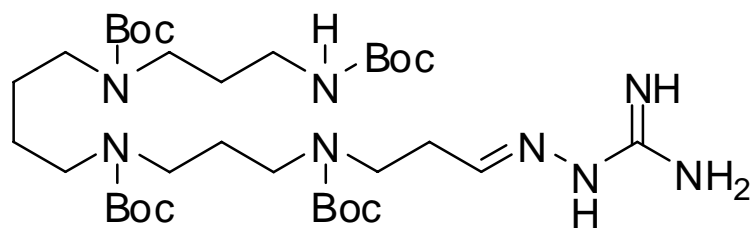
—7.49

3.50
3.48
3.47
3.45
3.16
3.09

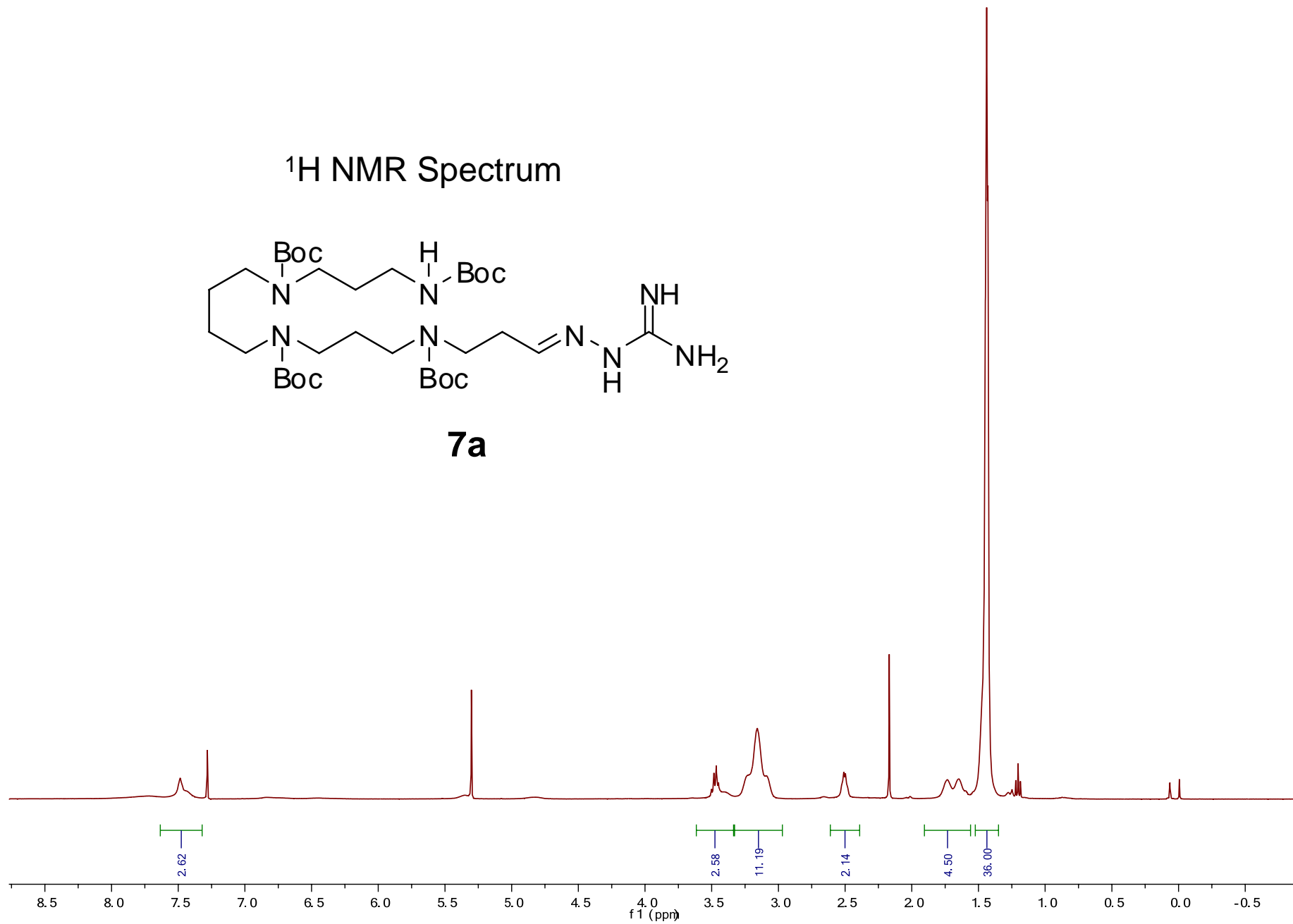
2.51
2.50

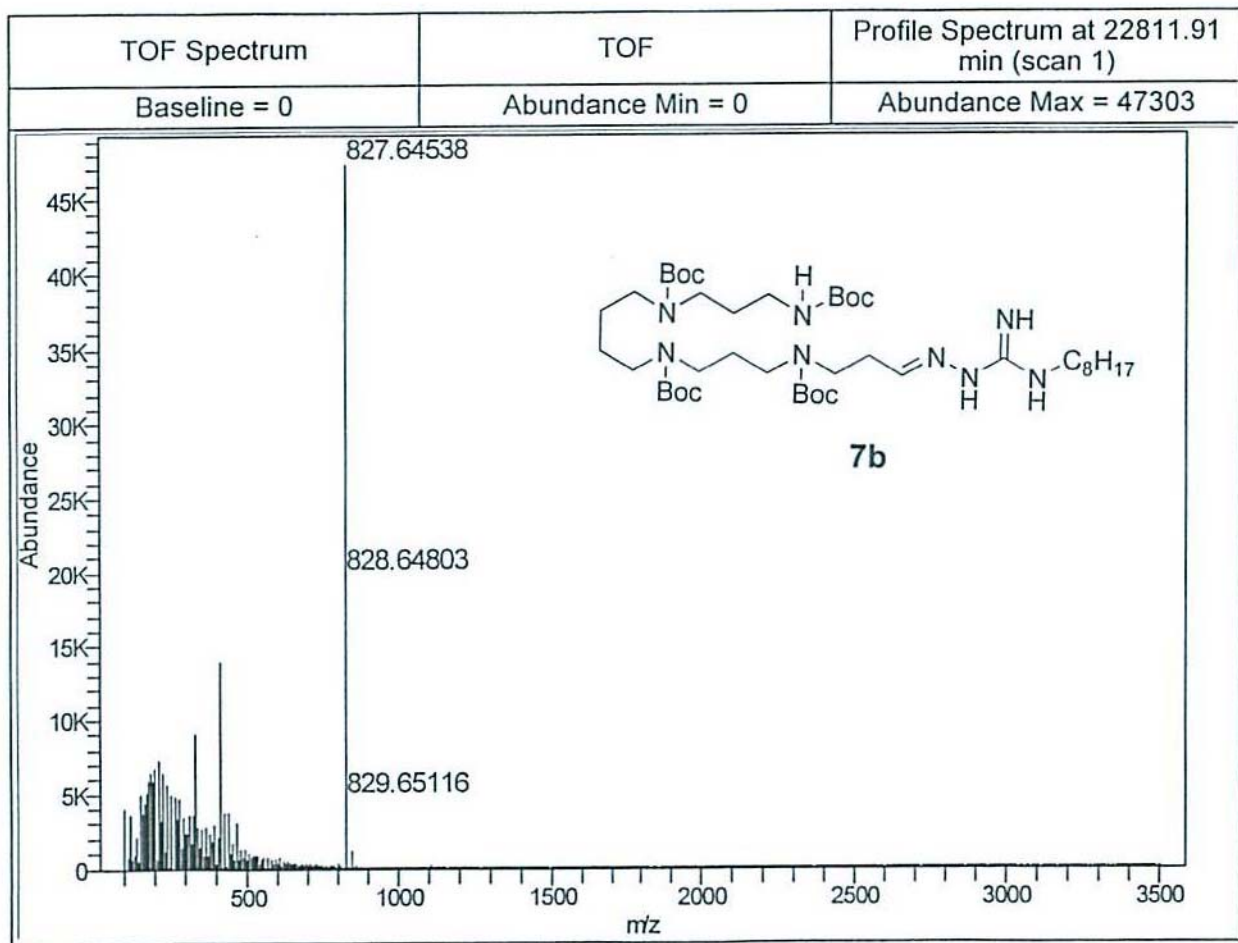
1.73
1.65
1.44
1.43

^1H NMR Spectrum

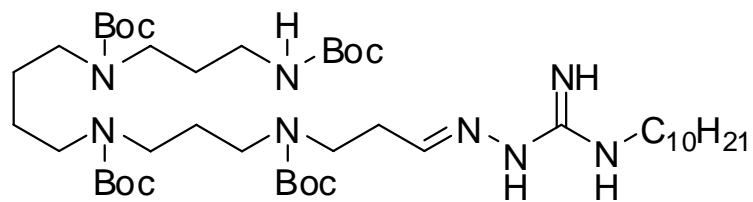


7a

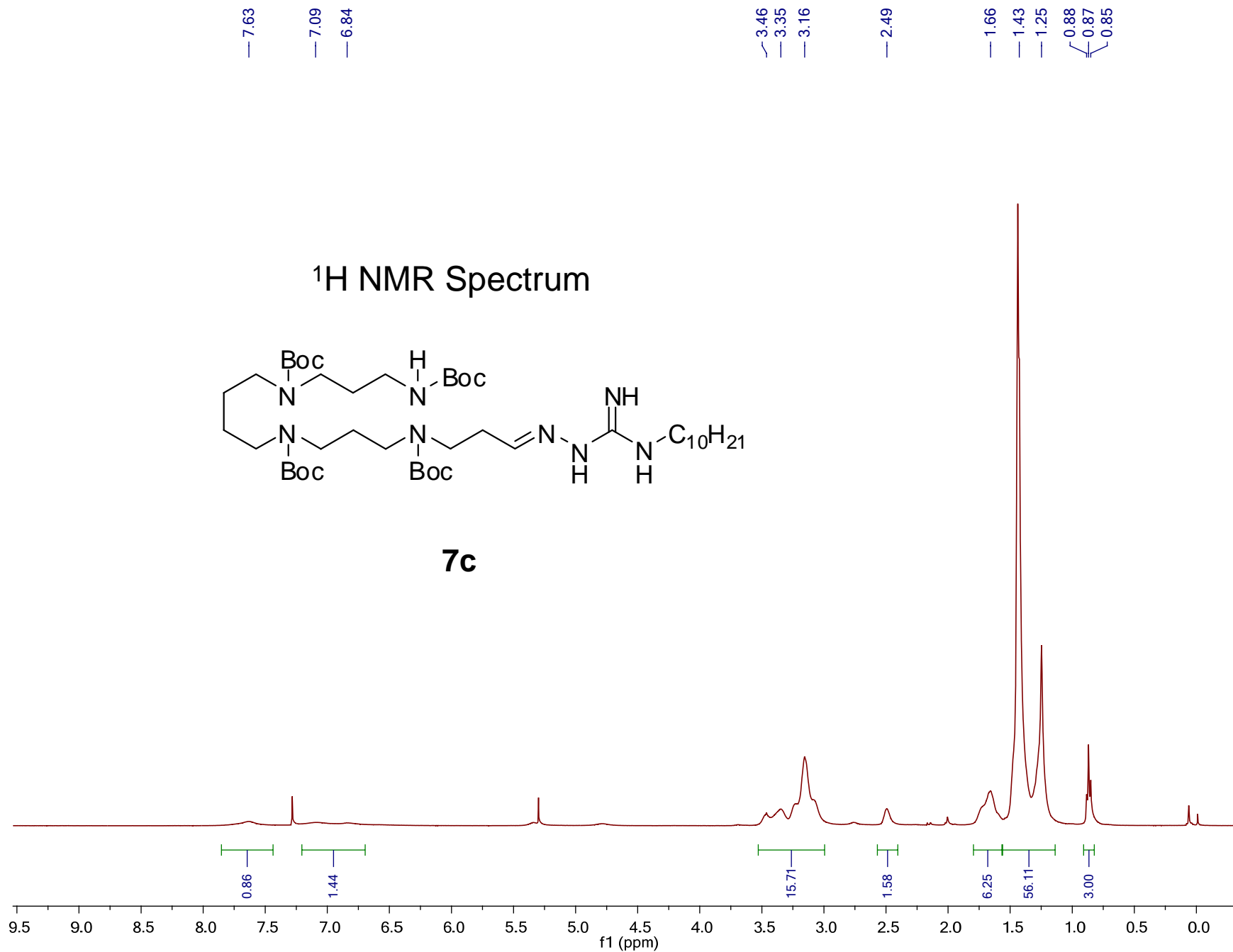




¹H NMR Spectrum



7c



155.25
155.21
155.13
154.57
154.51

78.96
78.92
78.85
78.83
78.52

64.84

52.45

45.83

45.46

43.89

43.26

41.17

36.69

36.42

36.40

30.84

27.43

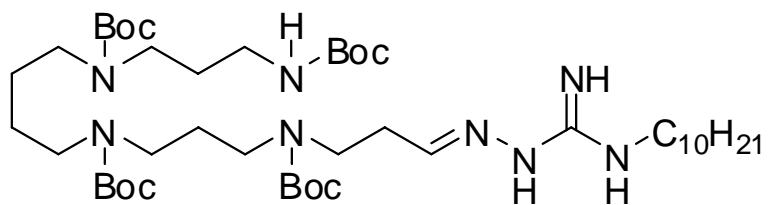
24.57

21.64

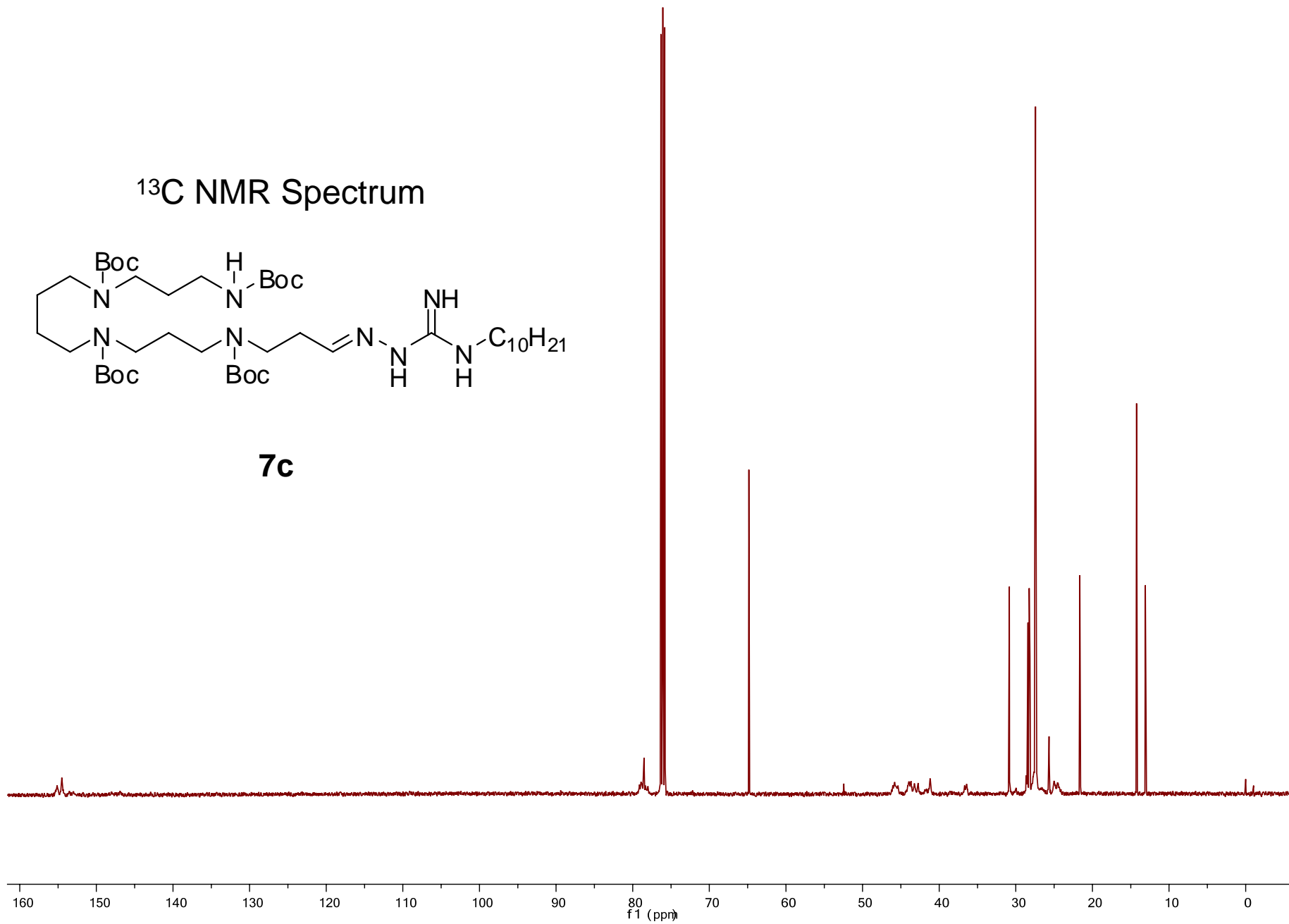
14.25

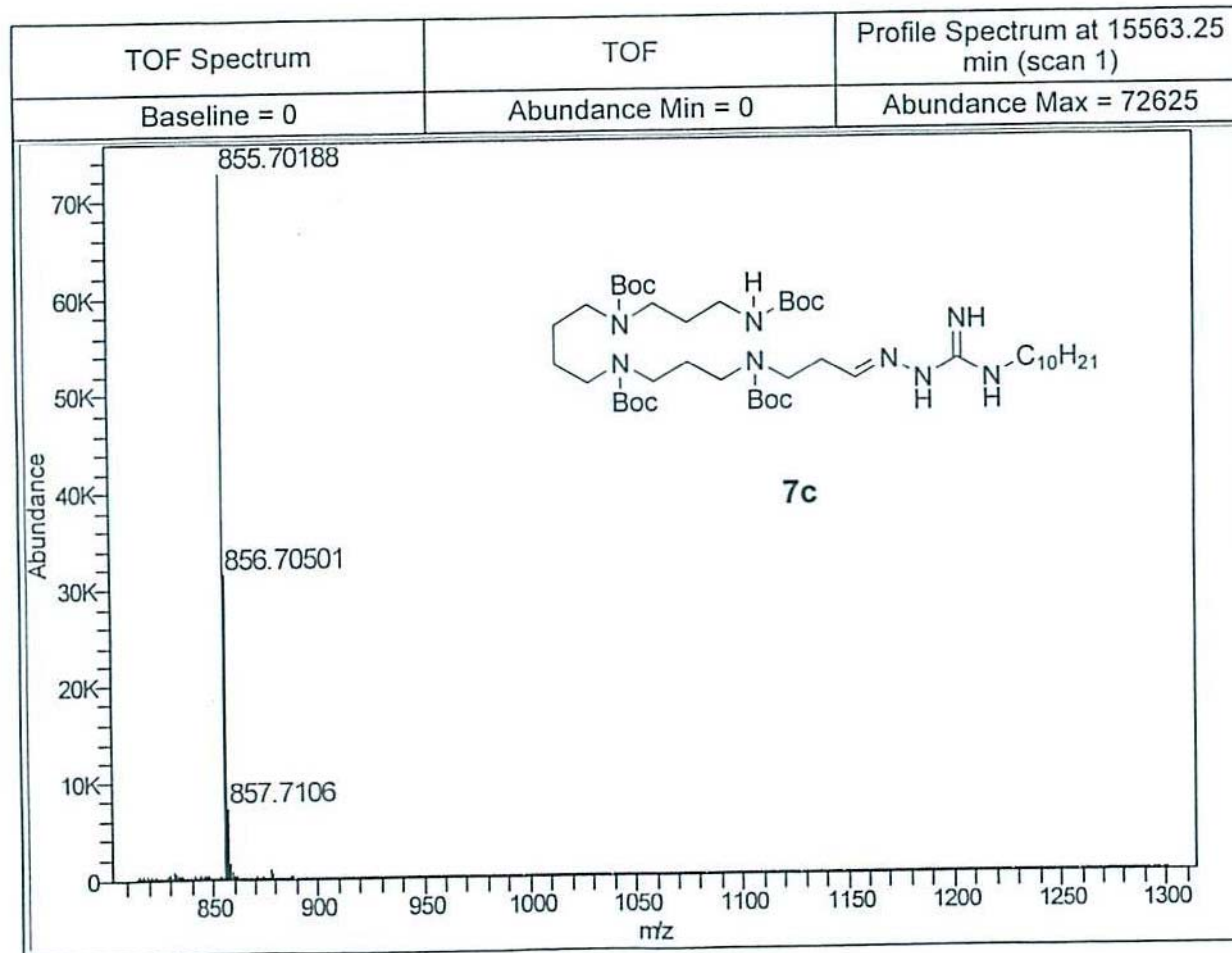
13.10

¹³C NMR Spectrum

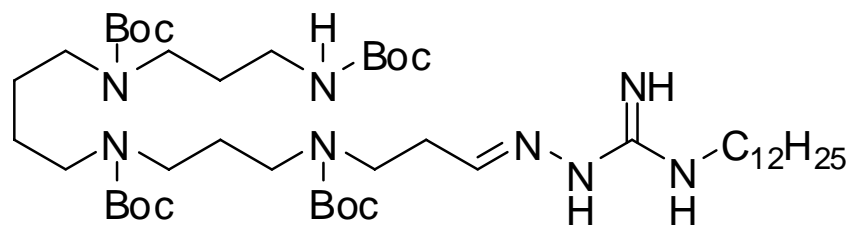


7c

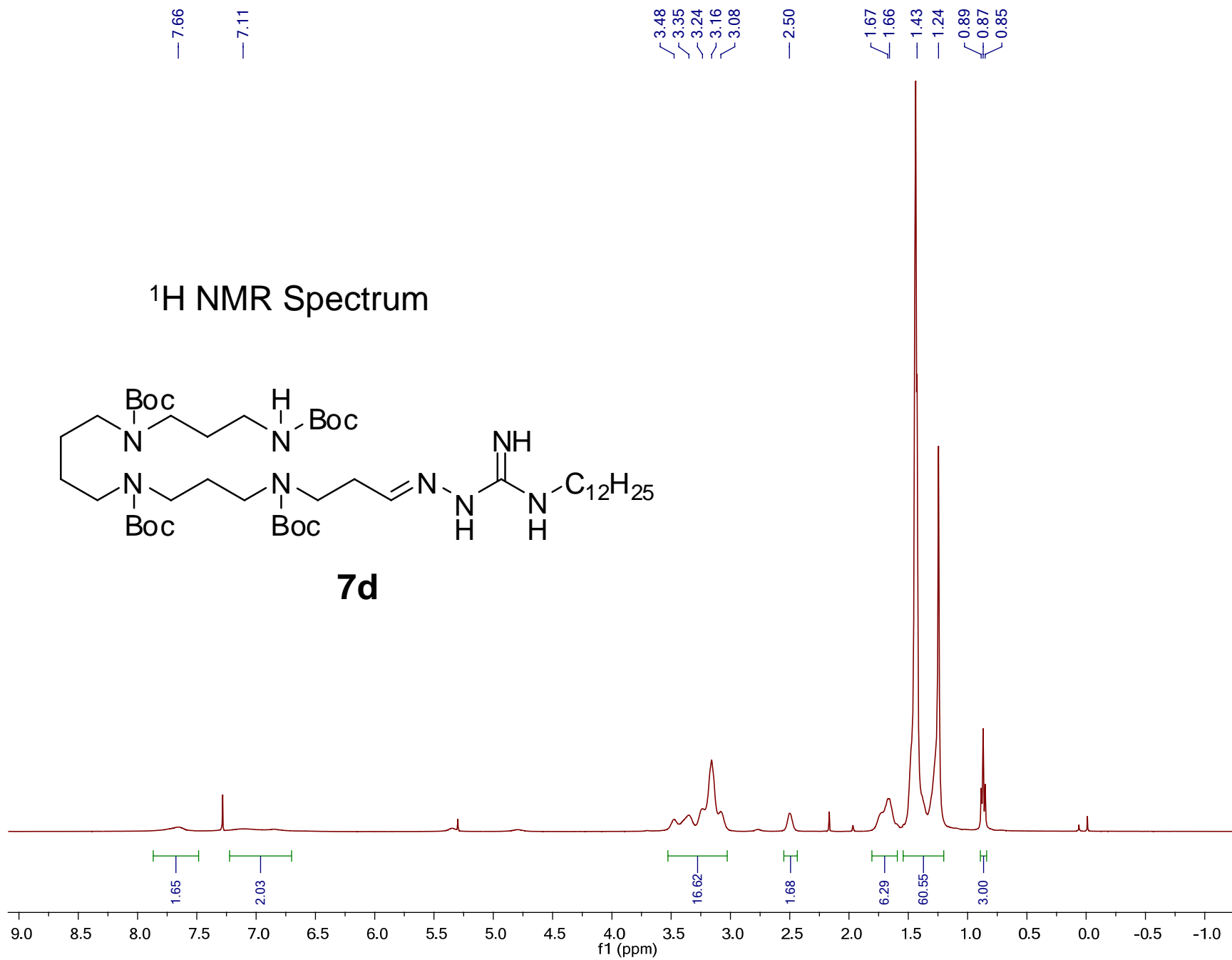




¹H NMR Spectrum



7d



156.24
156.11
156.06
155.50
155.31
155.27

80.15
80.11
80.00
79.89
79.54
79.37
79.06
79.02

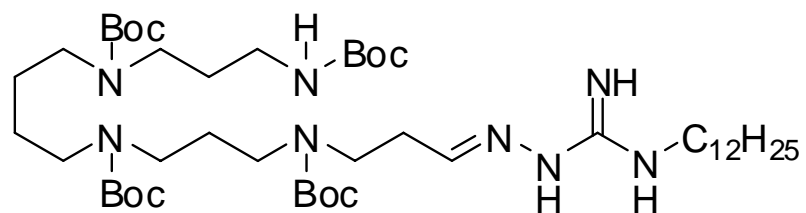
65.86

45.00
44.96
44.91
44.83
44.69
44.26
43.79
42.23

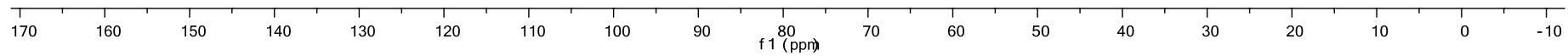
37.69
37.53
37.42
29.59
29.34
28.69
28.51
26.69
22.68

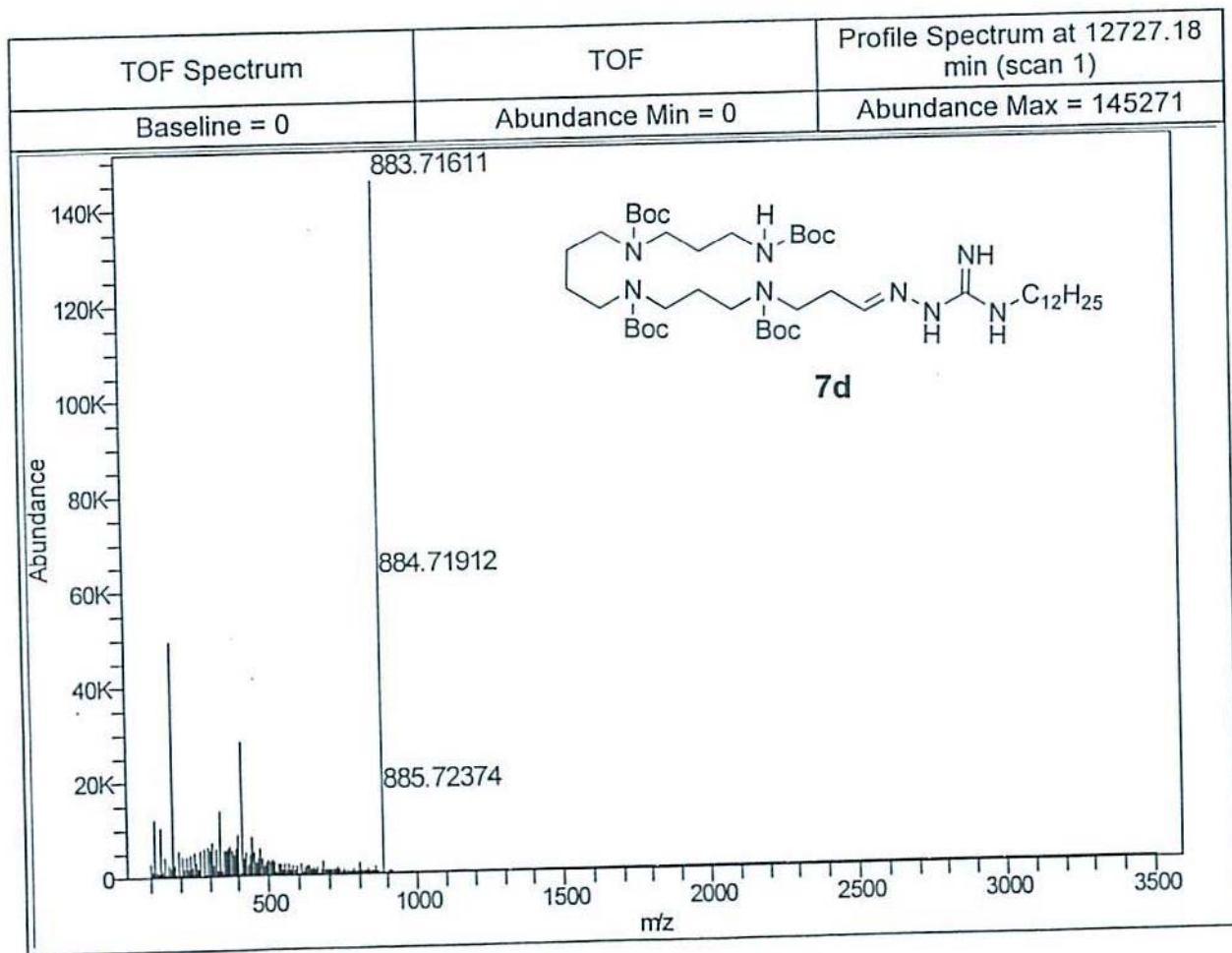
15.27
14.13

¹³C NMR Spectrum

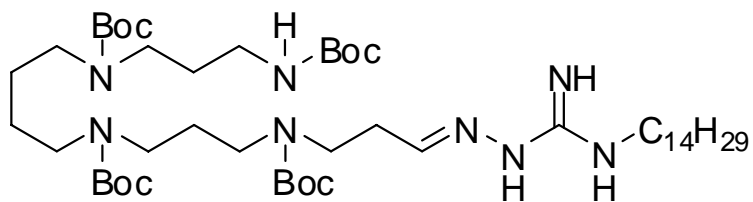


7d

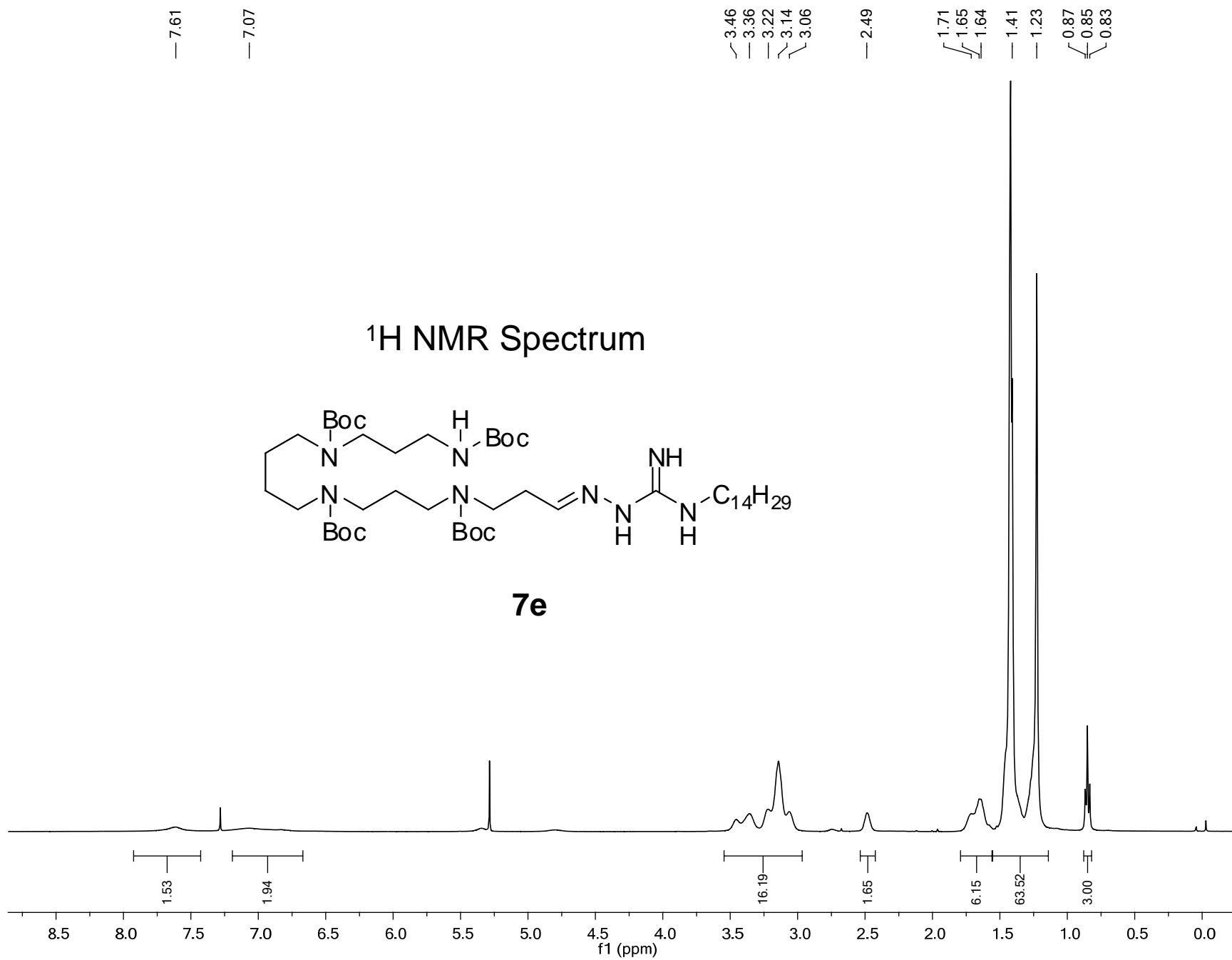


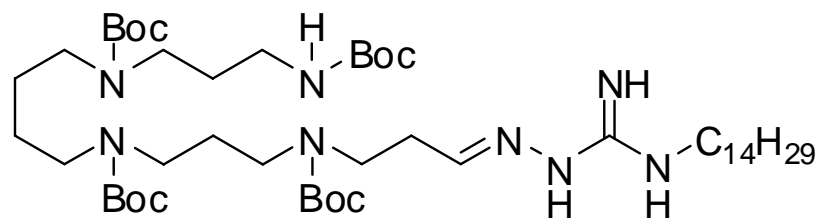


¹H NMR Spectrum



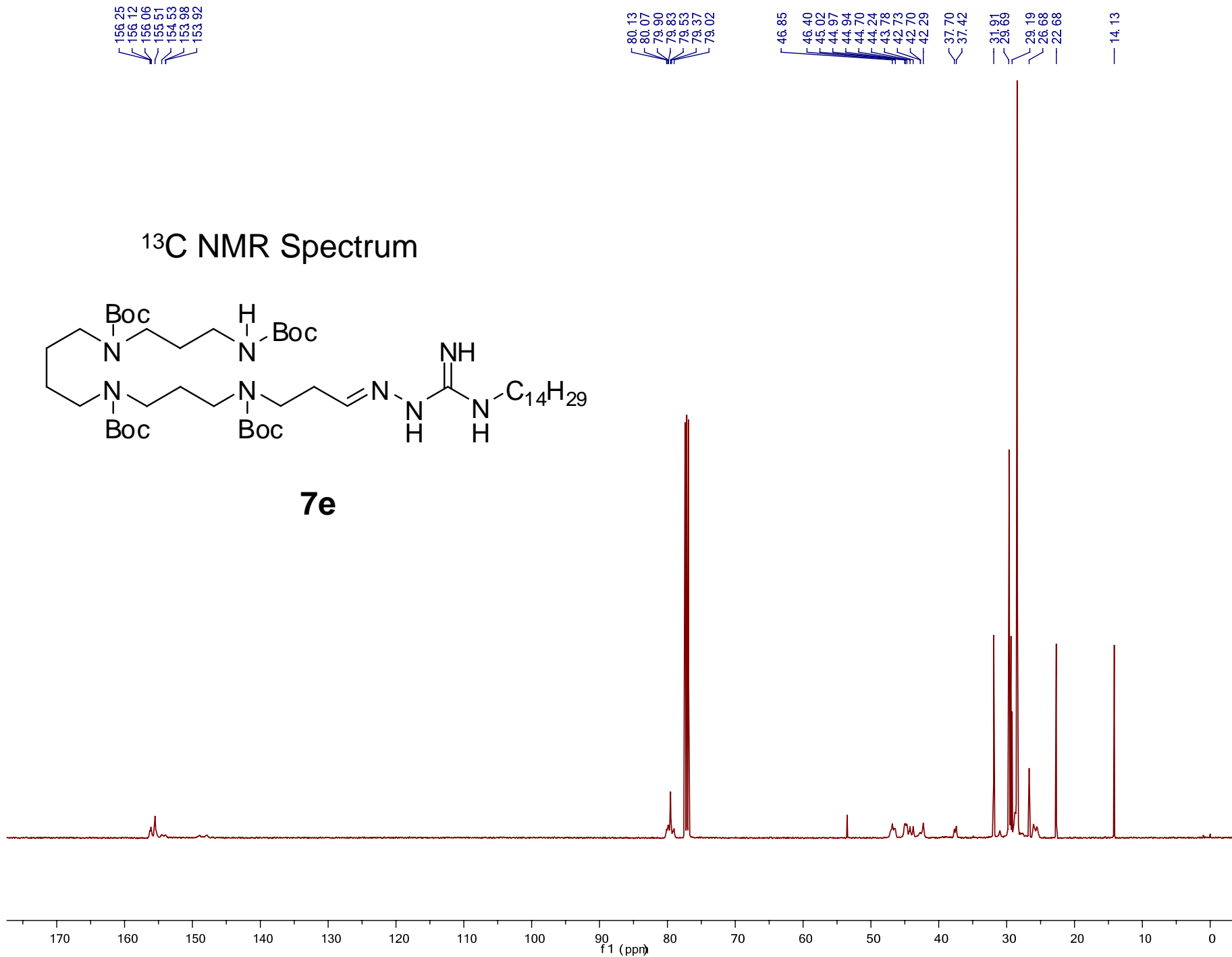
7e

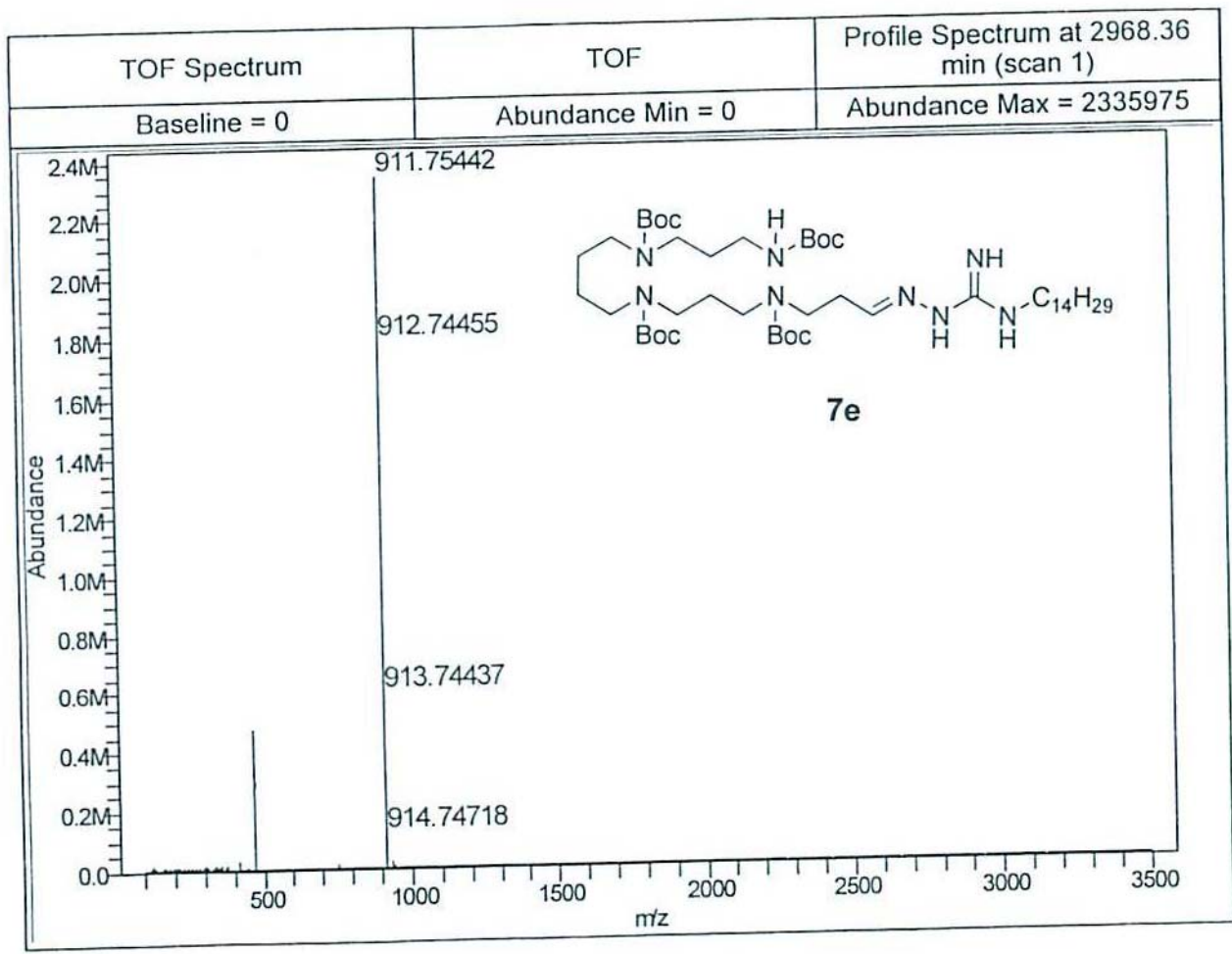




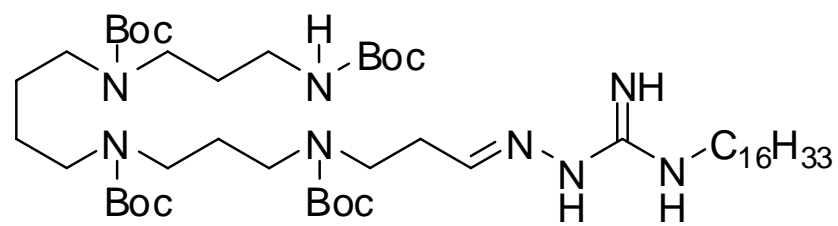
7e

¹³C NMR Spectrum

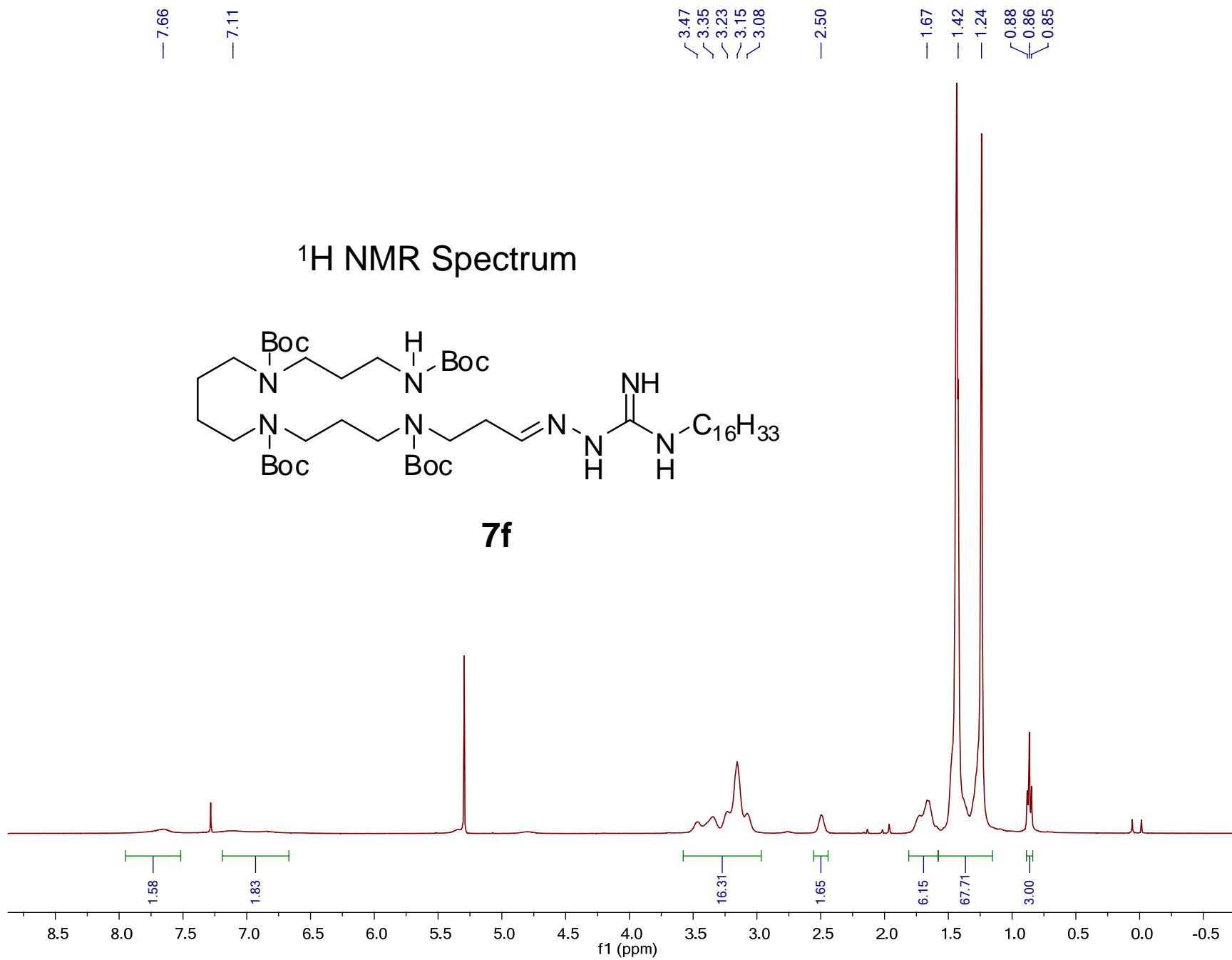




¹H NMR Spectrum



7f



155.25
155.10
154.52

148.09
147.00

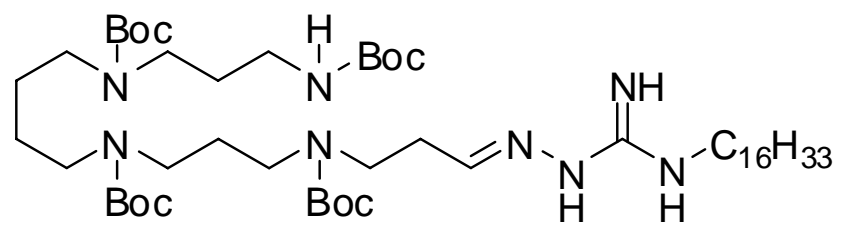
79.12
78.83
78.52
78.36
78.03

52.46
45.83
43.90
43.69
43.22
42.75
41.20
36.69
36.42

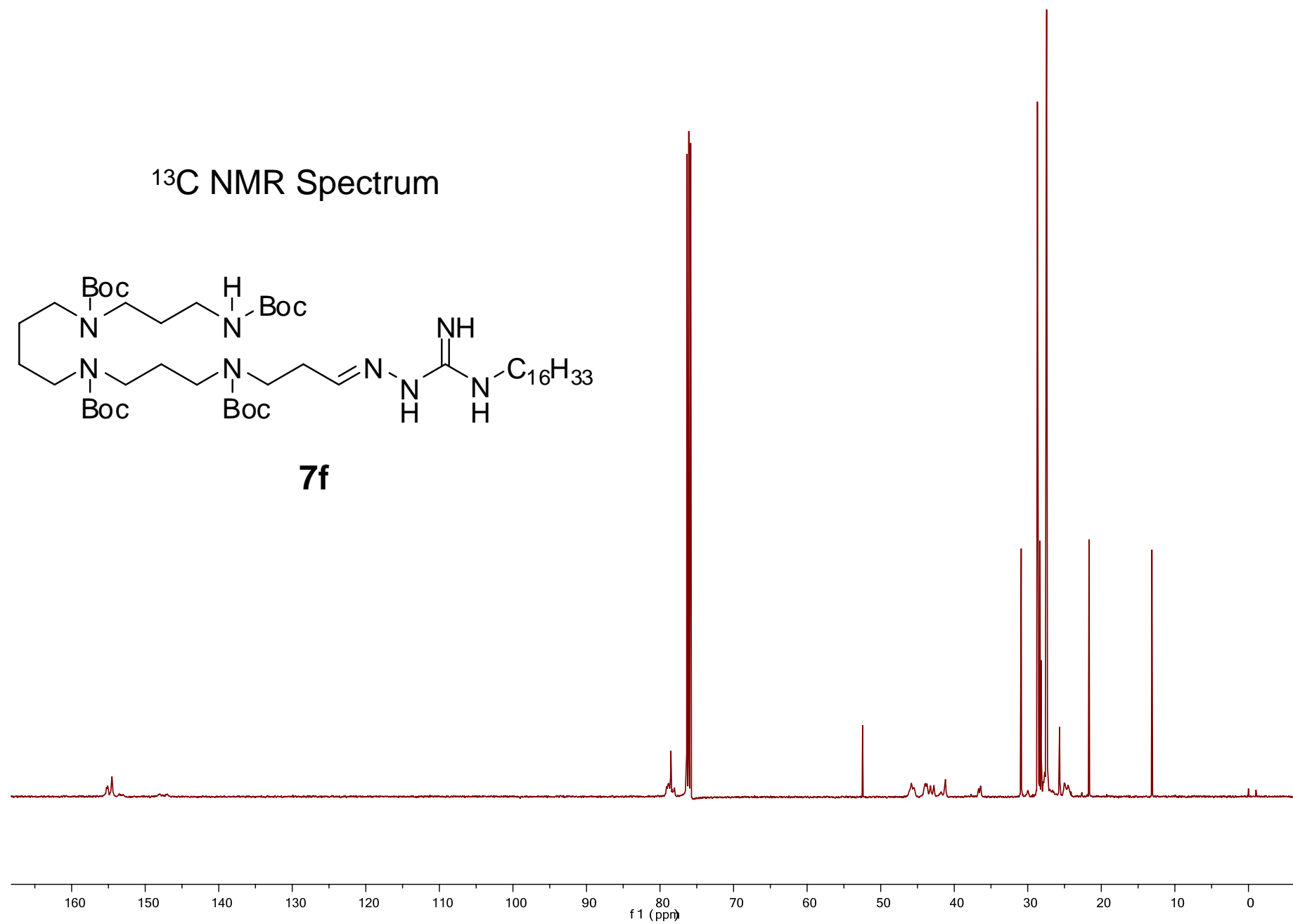
30.98
25.68
24.54
21.66

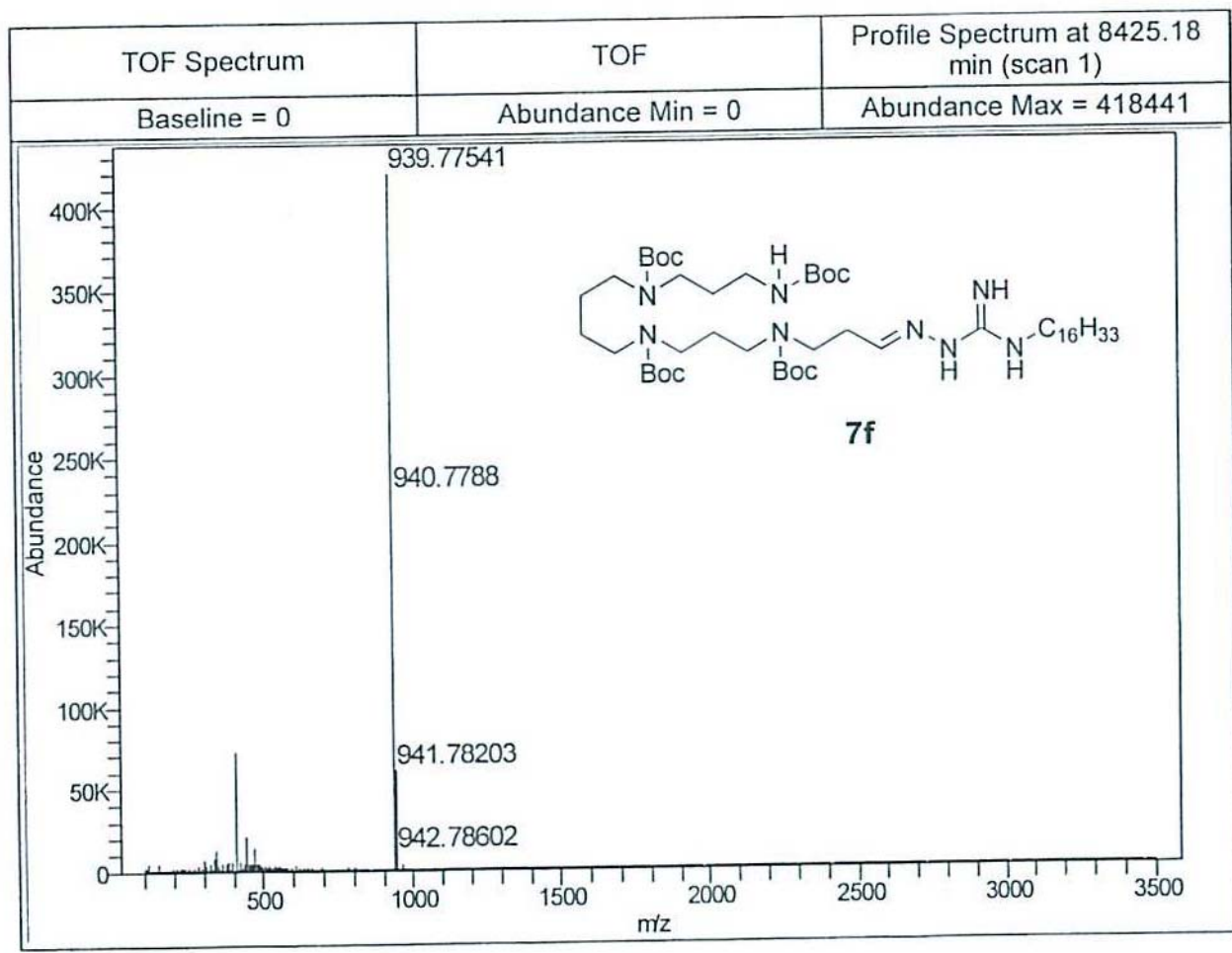
13.11

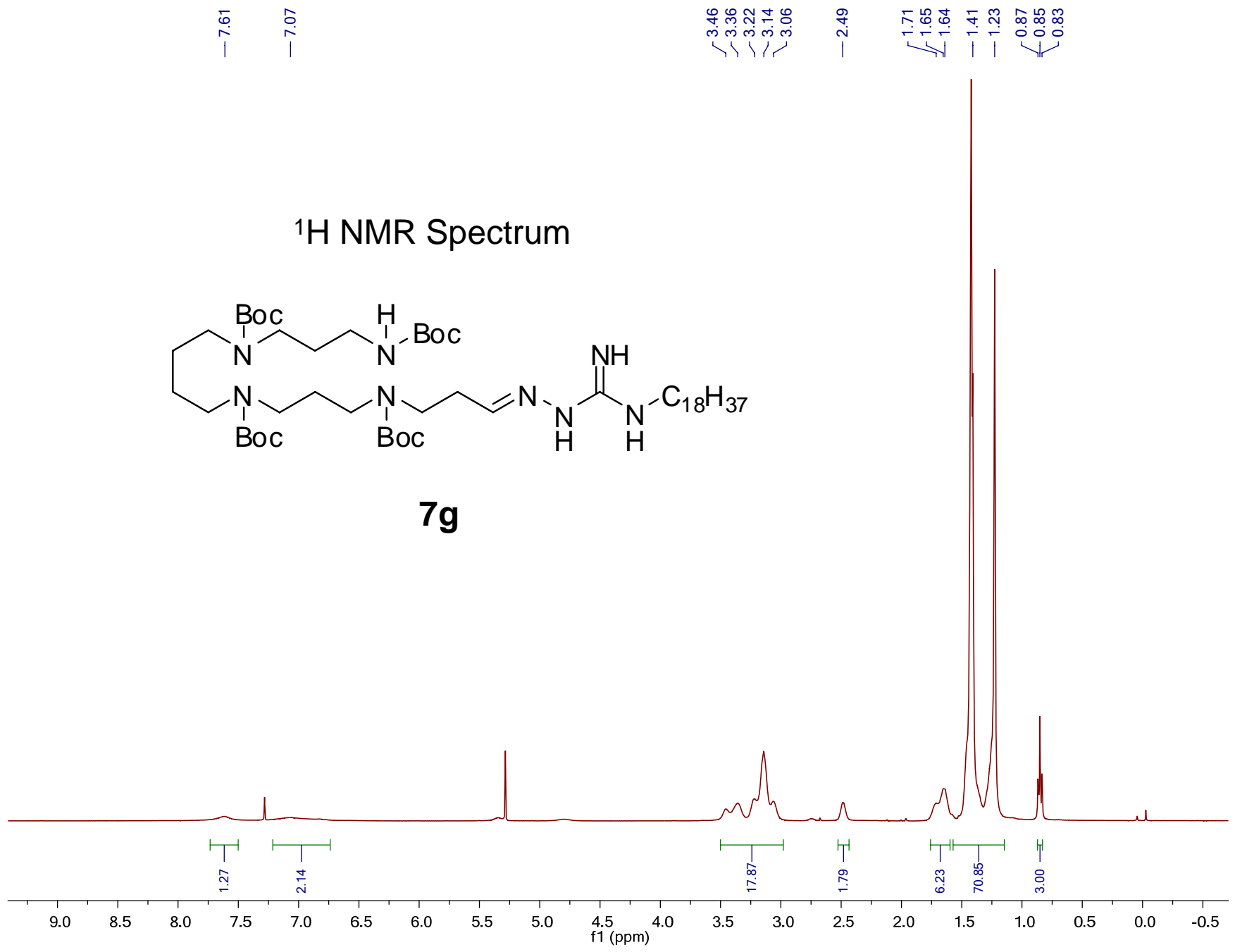
¹³C NMR Spectrum



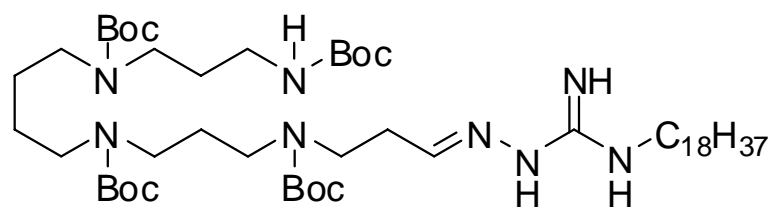
7f



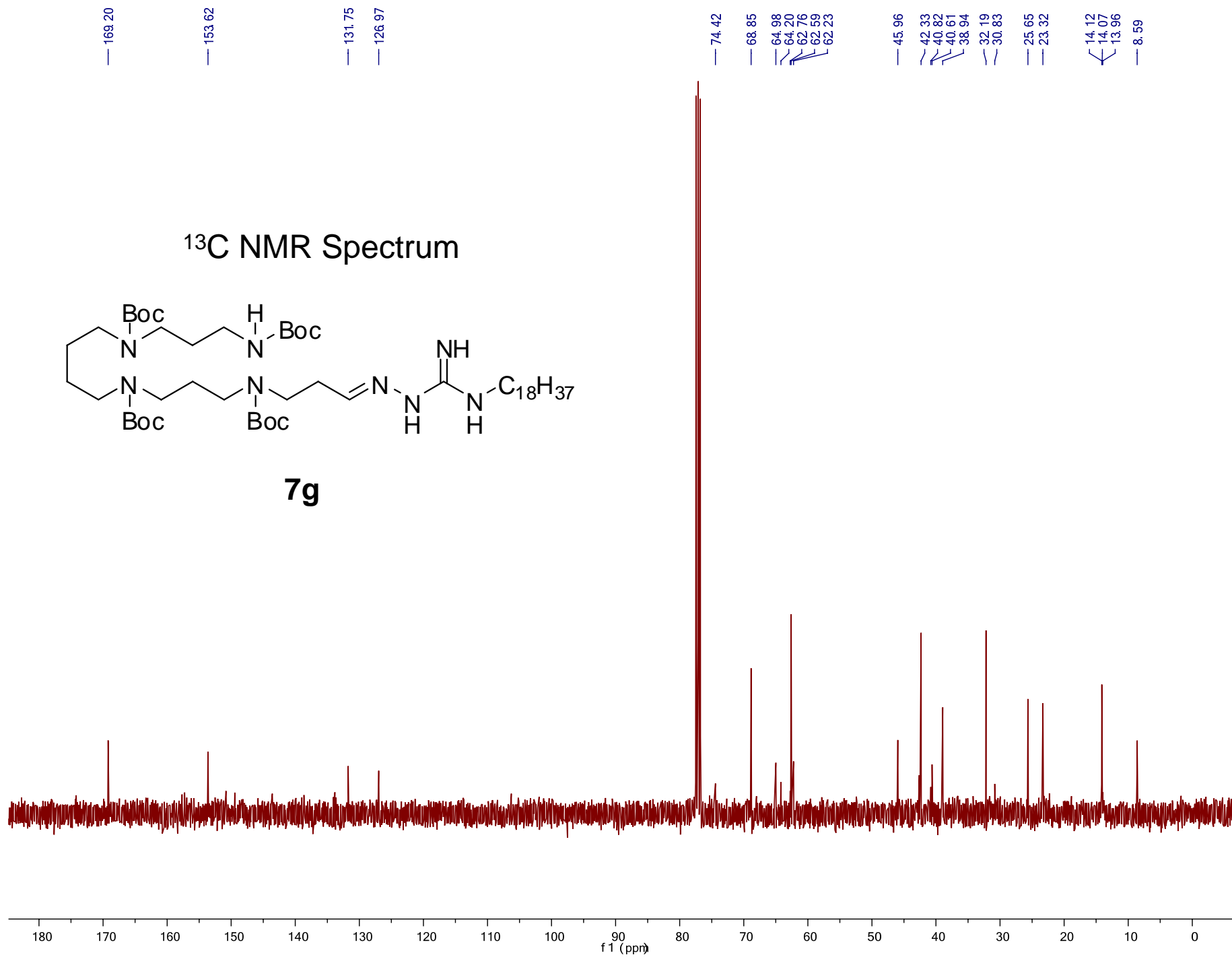


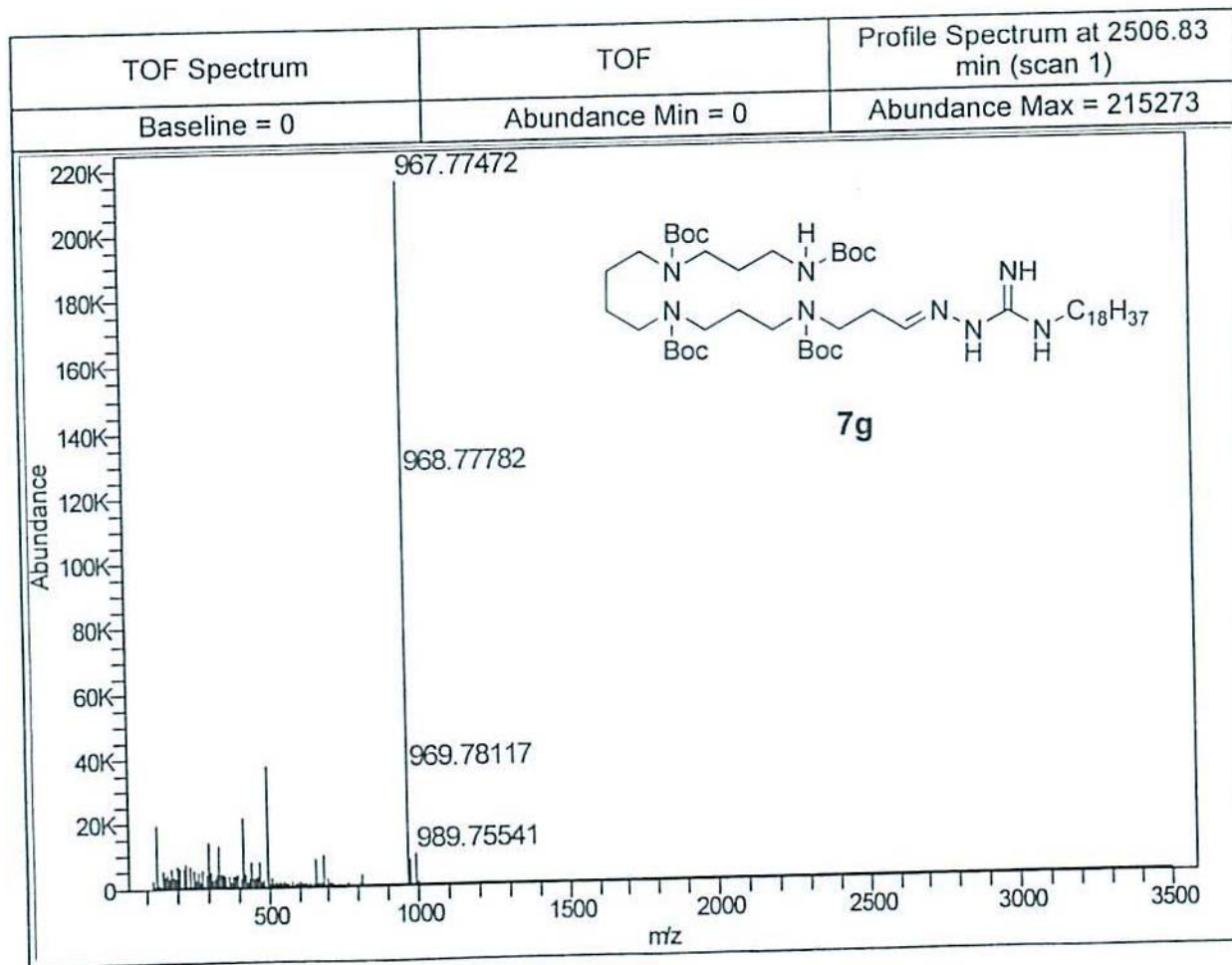


¹³C NMR Spectrum



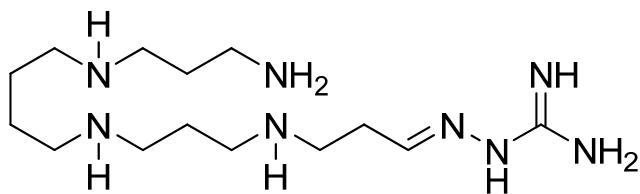
7g



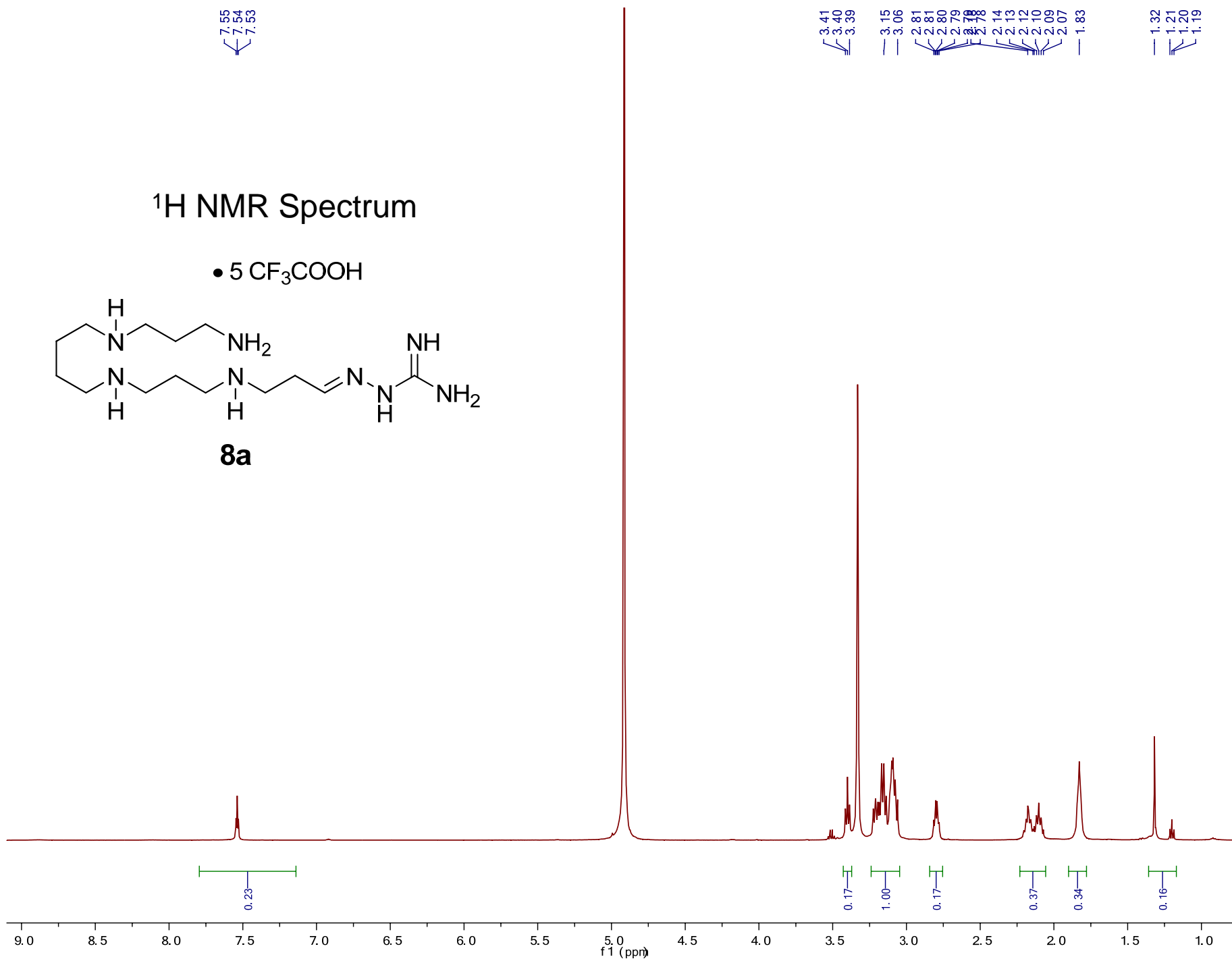


¹H NMR Spectrum

• 5 CF₃COOH



8a



— 155.70

— 147.12

— 117.75

— 115.43

— 46.80

44.59

44.42

44.39

43.55

— 36.38

— 28.47

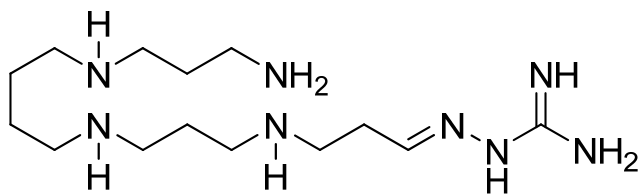
23.97

22.80

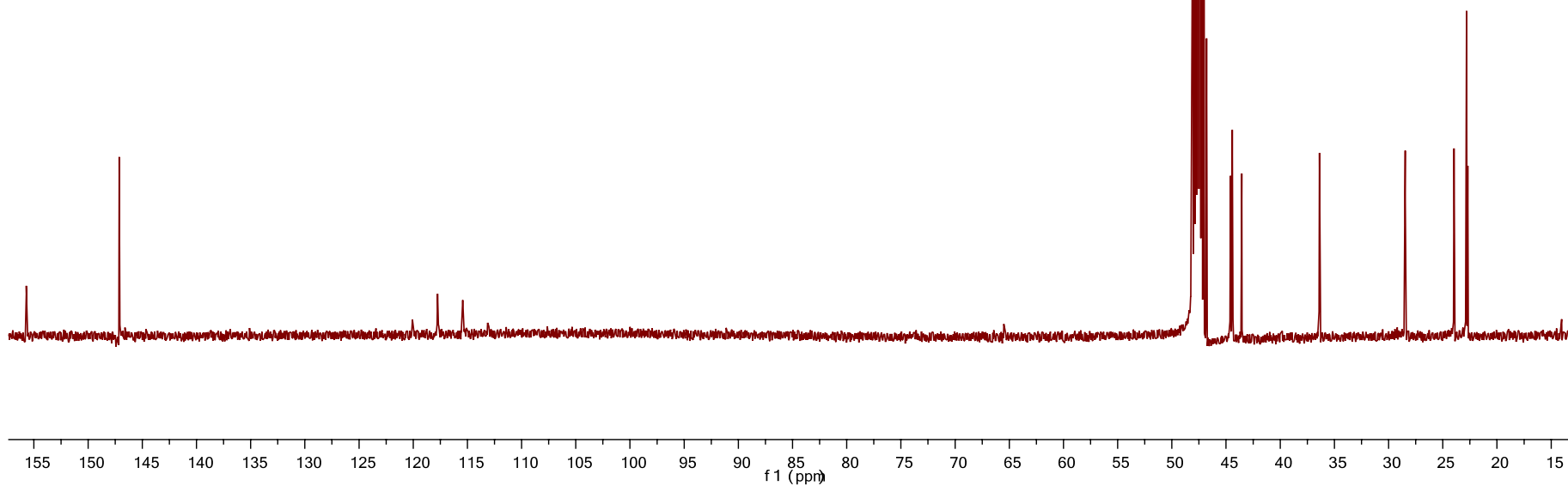
22.69

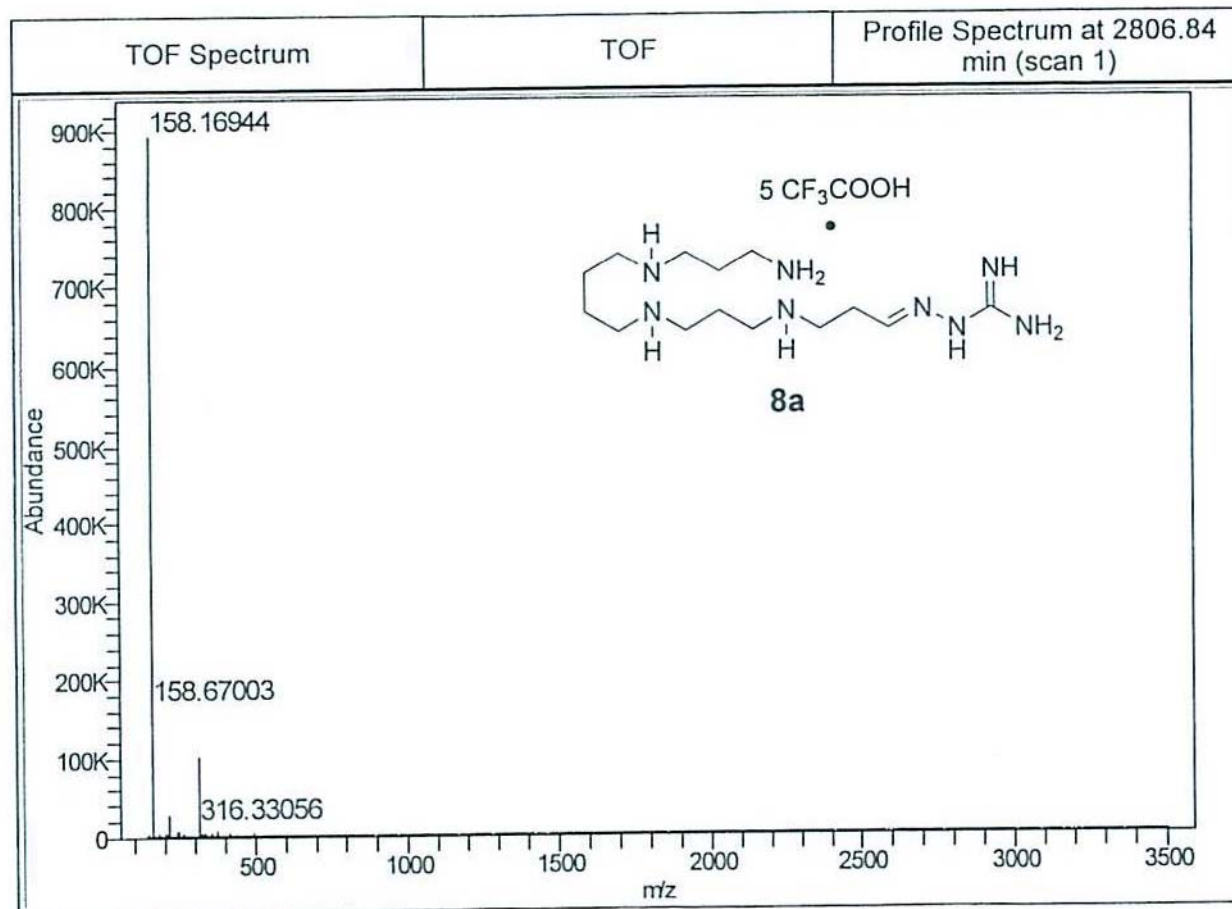
¹³C NMR Spectrum

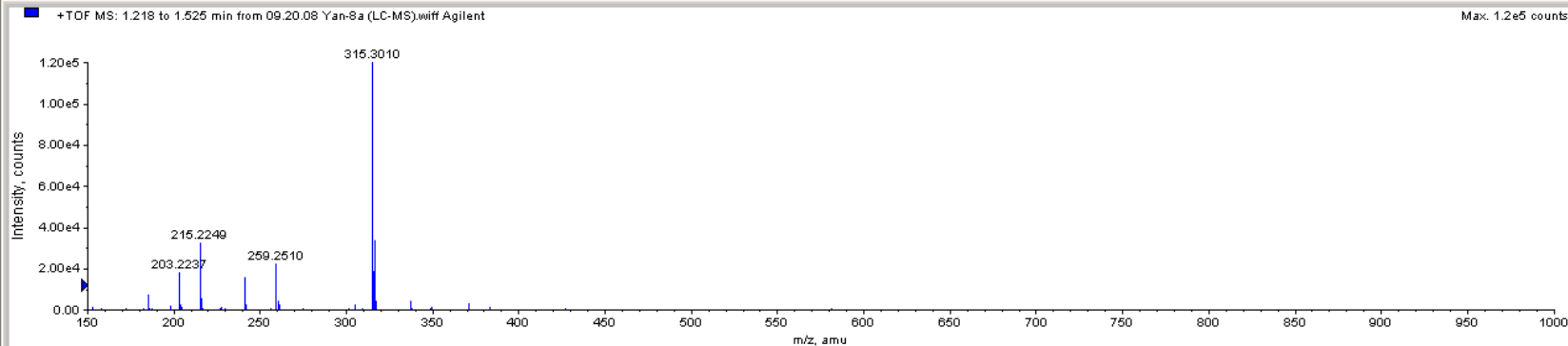
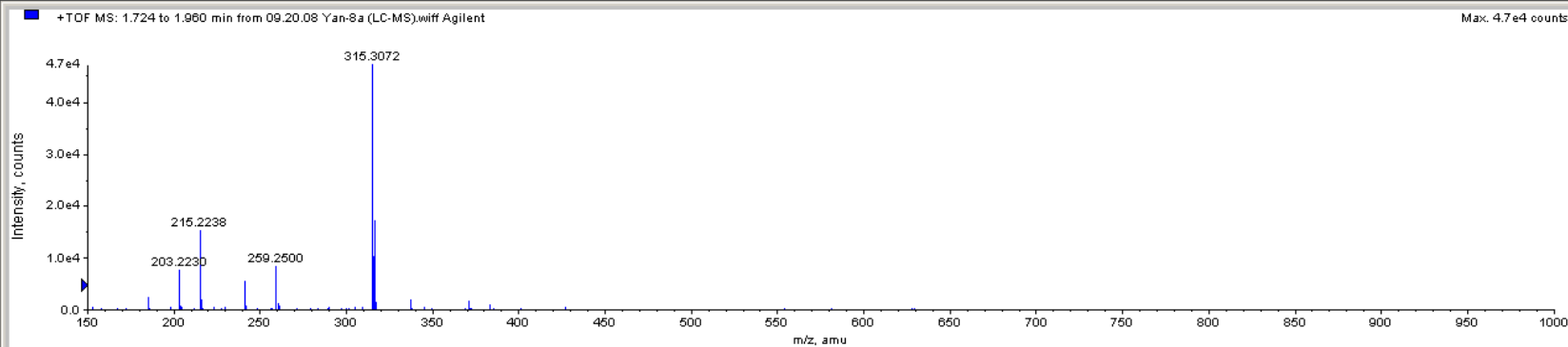
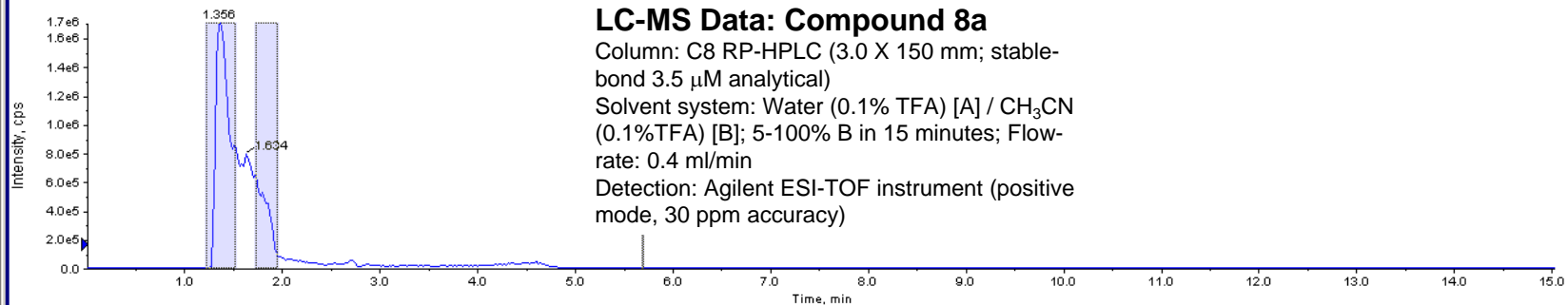
• 5 CF₃COOH



8a



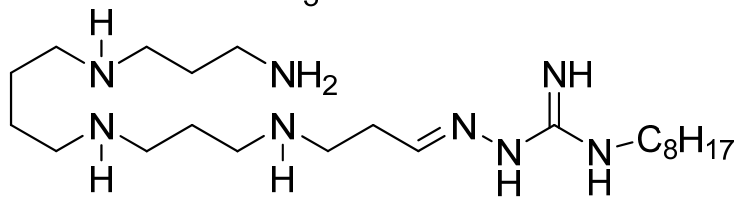




7.54
7.53

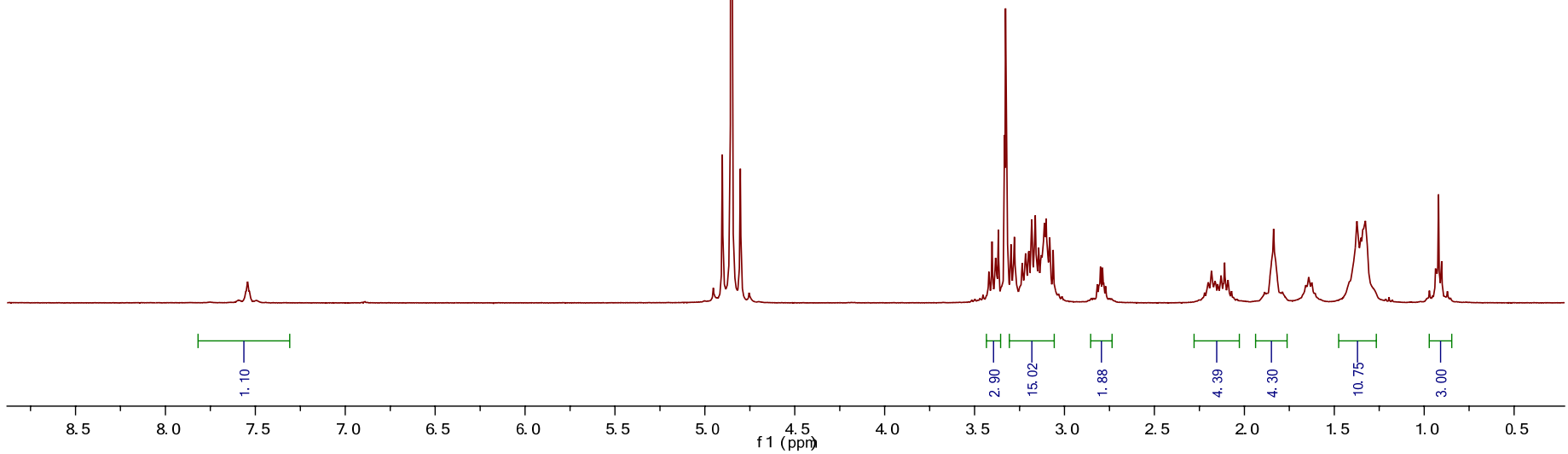
¹H NMR Spectrum

• 5 CF₃COOH



8b

3.45
3.42
3.40
3.38
3.38
3.37
3.37
3.35
3.30
3.28
3.25
3.24
3.22
3.20
3.18
3.16
3.14
3.13
3.11
3.10
3.08
3.06
3.03
2.81
2.79
2.78
2.77
2.13
2.12
2.11
2.09
2.04
1.82
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1.34
1.33
0.97
0.94
0.92
0.90
0.87



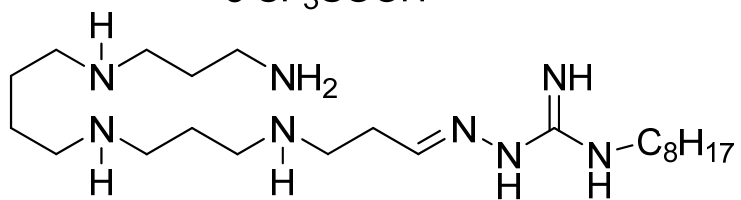
161.86
161.59

117.86
115.55

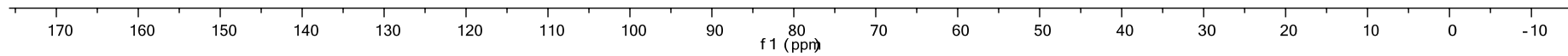
46.81
43.71
41.14
36.41
31.54
28.53
26.28
23.97
22.81
22.70
22.29
13.01

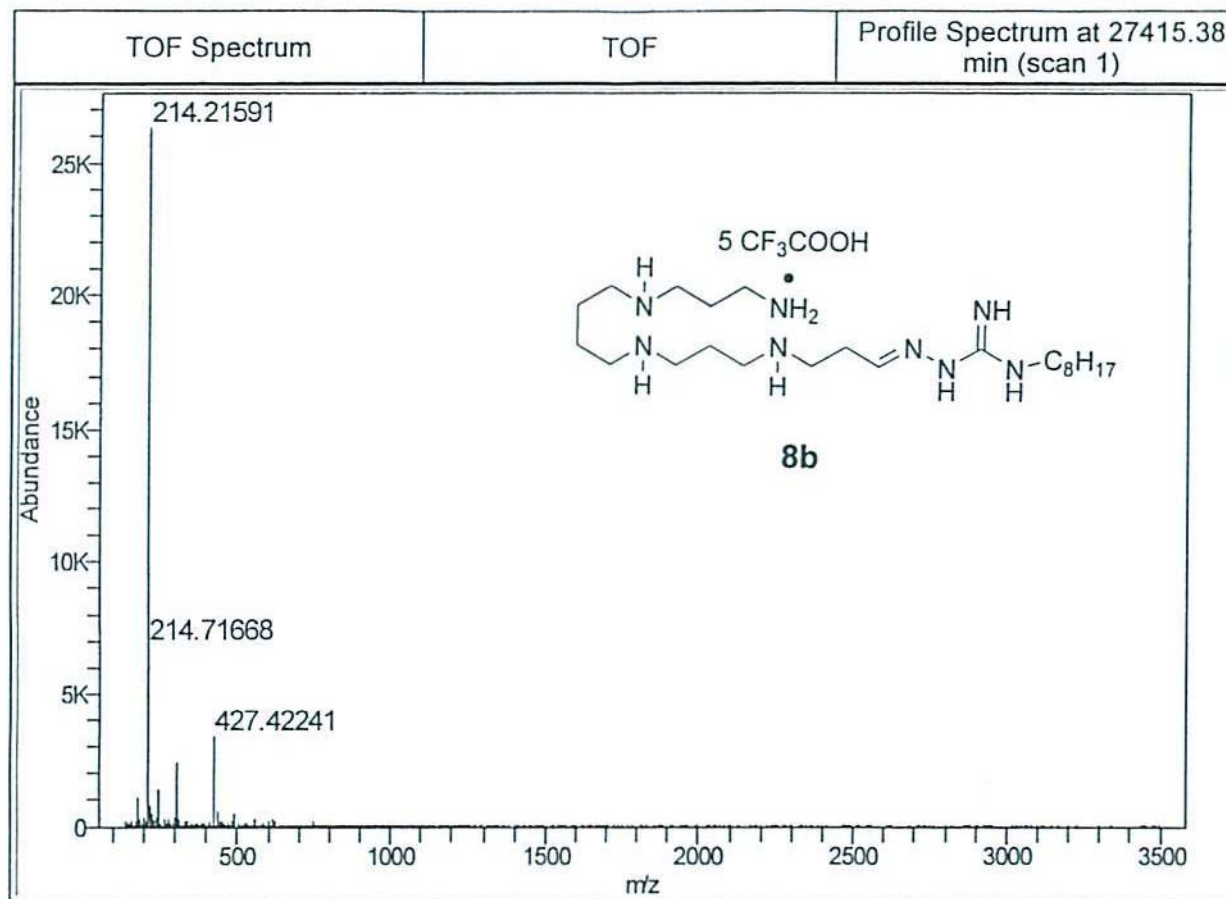
^{13}C NMR Spectrum

• 5 CF_3COOH



8b



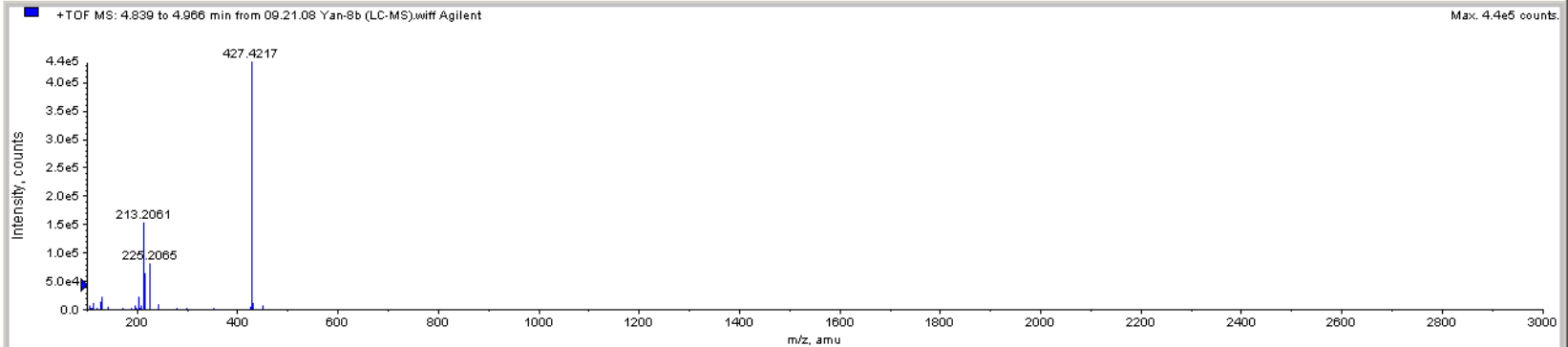
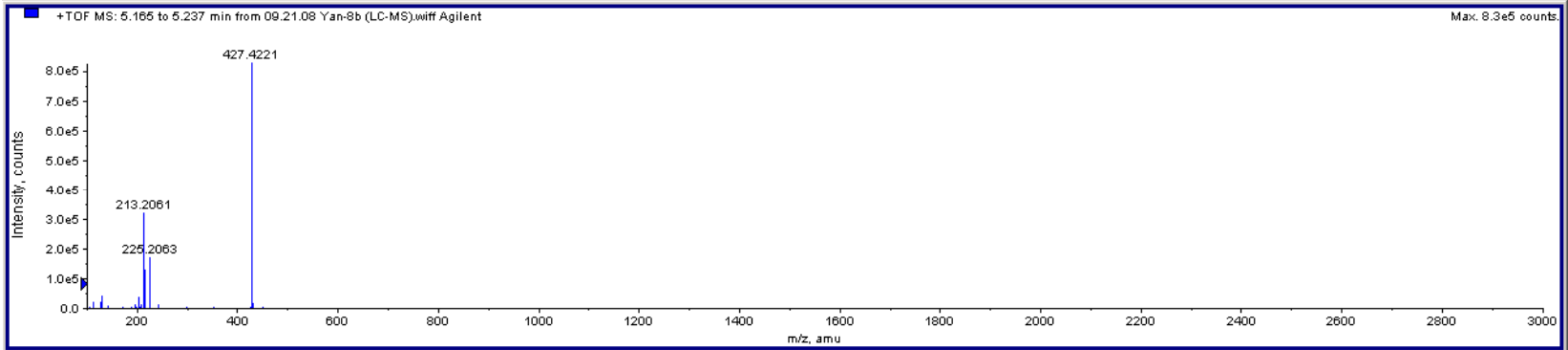
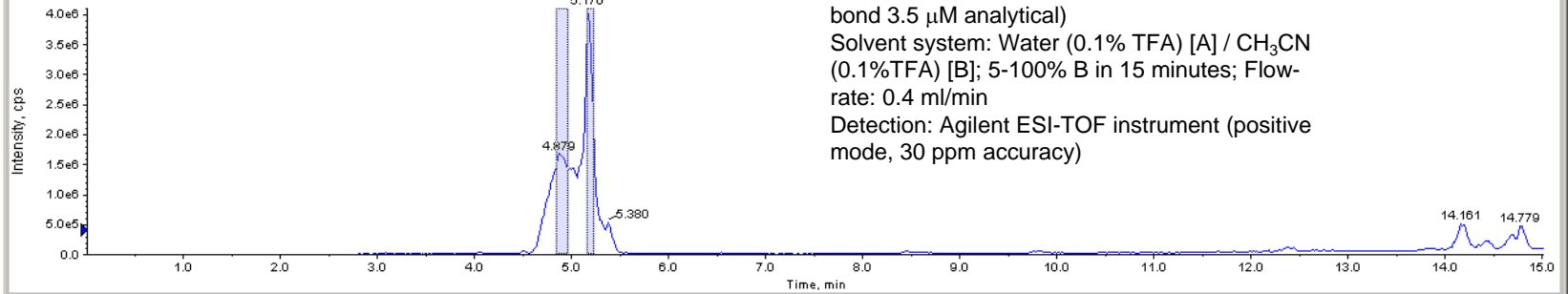


LC-MS Data: Compound 8b

Column: C8 RP-HPLC (3.0 X 150 mm; stable-bond 3.5 μ M analytical)

Solvent system: Water (0.1% TFA) [A] / CH₃CN (0.1%TFA) [B]; 5-100% B in 15 minutes; Flow-rate: 0.4 ml/min

Detection: Agilent ESI-TOF instrument (positive mode, 30 ppm accuracy)

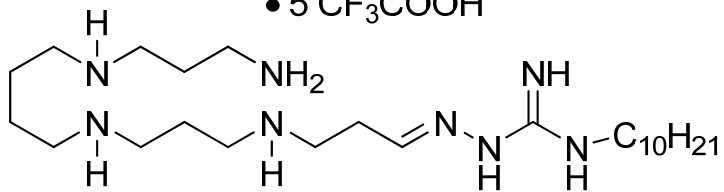


— 7.54

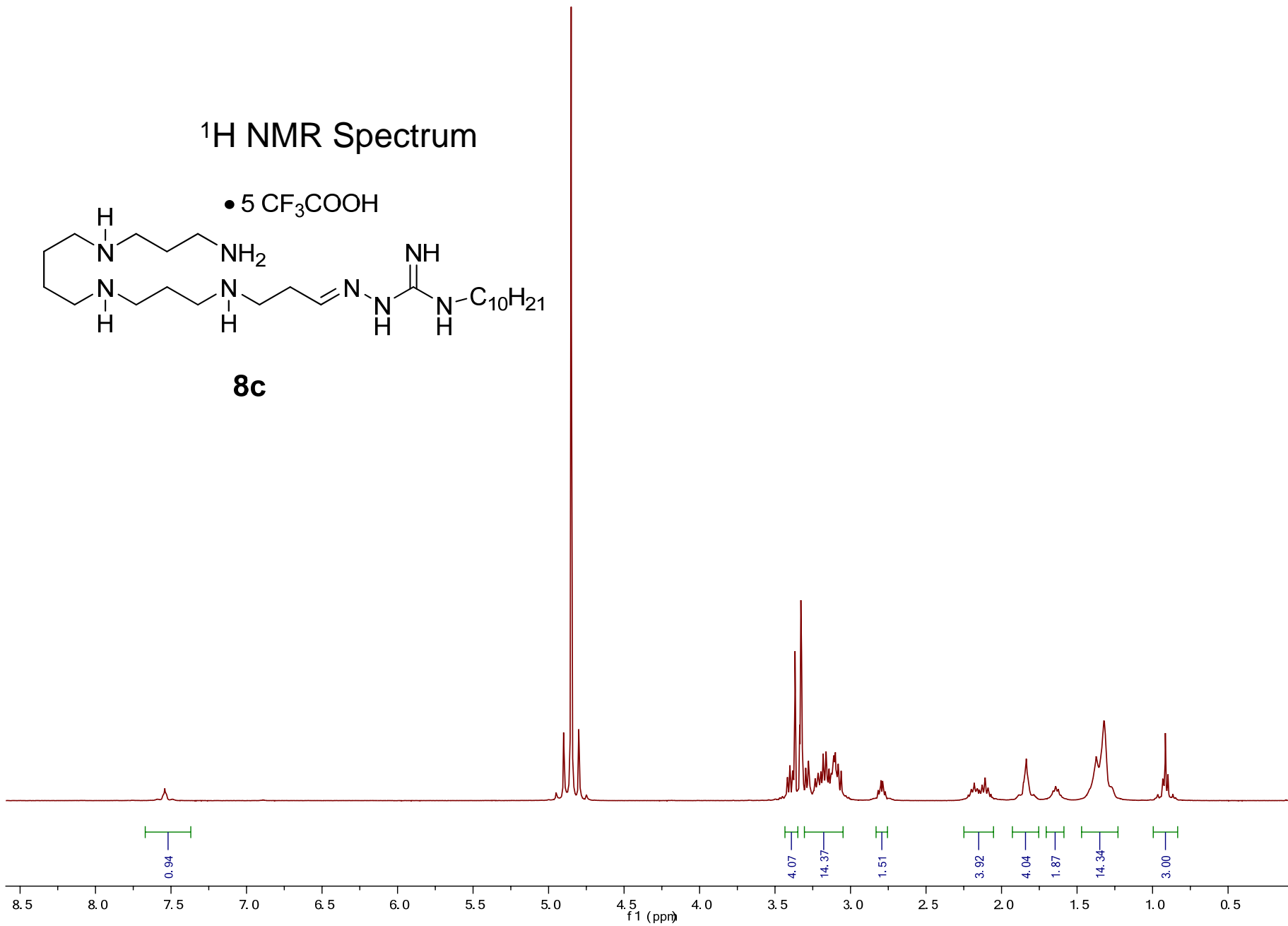
3.42
3.40
3.38
3.38
3.37
3.30
3.28
3.23
3.22
3.20
3.18
3.16
3.14
3.13
3.11
3.10
3.08
3.06
2.82
2.80
2.79
2.78
2.77
2.18
2.16
2.15
2.13
2.11
2.09
— 1.84
1.64
1.62
1.37
1.32
0.93
0.90

¹H NMR Spectrum

• 5 CF₃COOH



8c



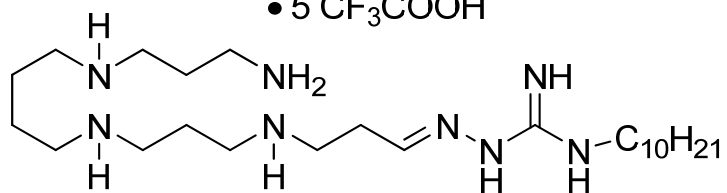
— 154.36

146.54
146.49
146.44

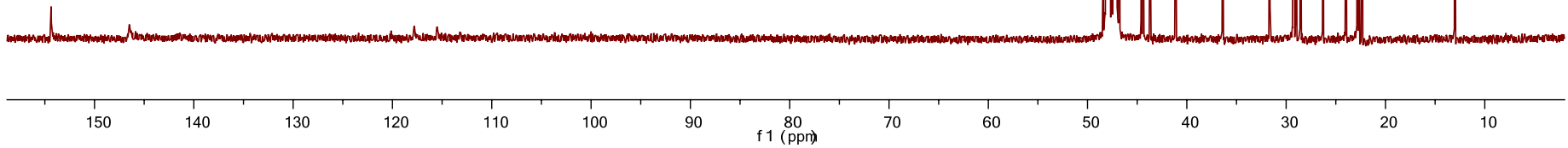
44.59
44.40
43.71
41.14
36.40
31.64
28.53
26.28
23.98
22.81
22.70
22.32
13.03

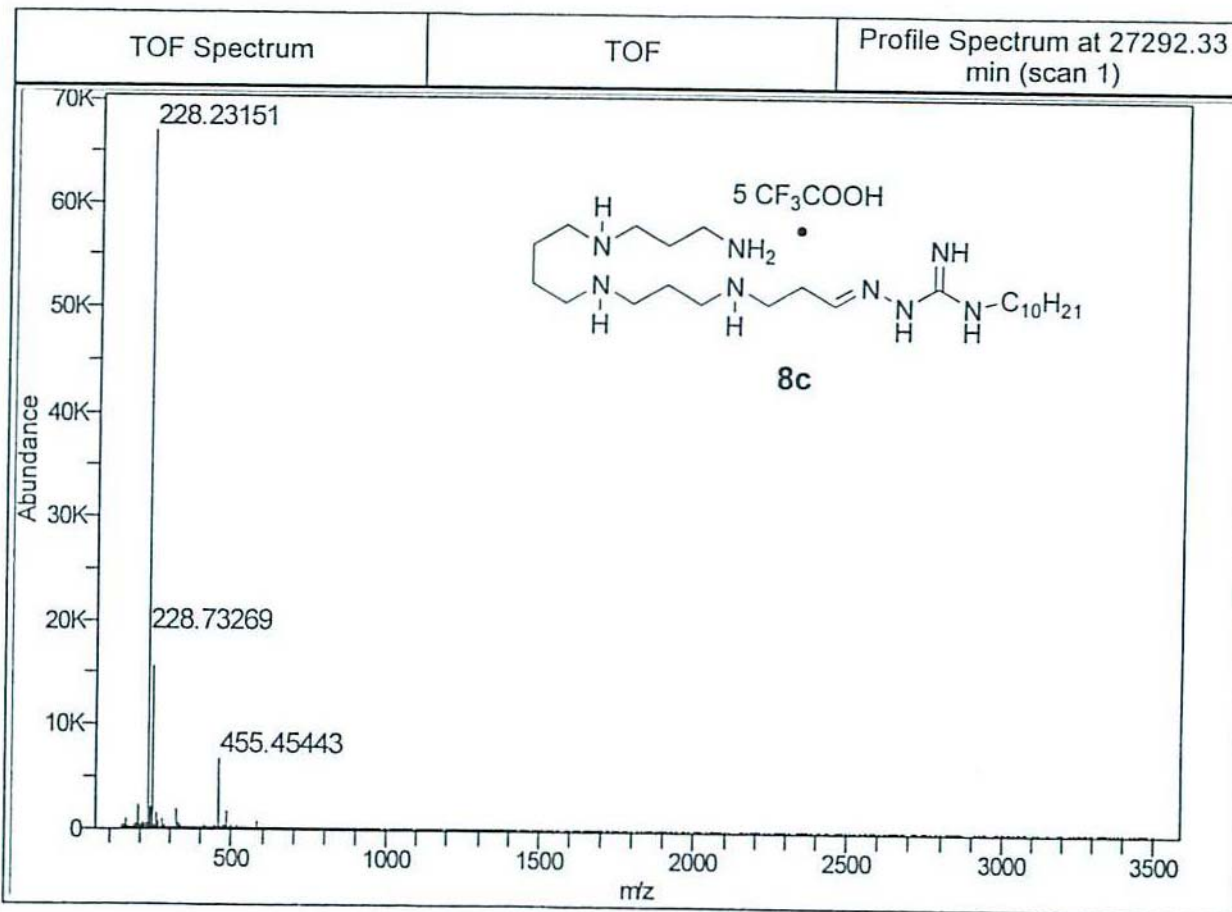
¹³C NMR Spectrum

• 5 CF₃COOH



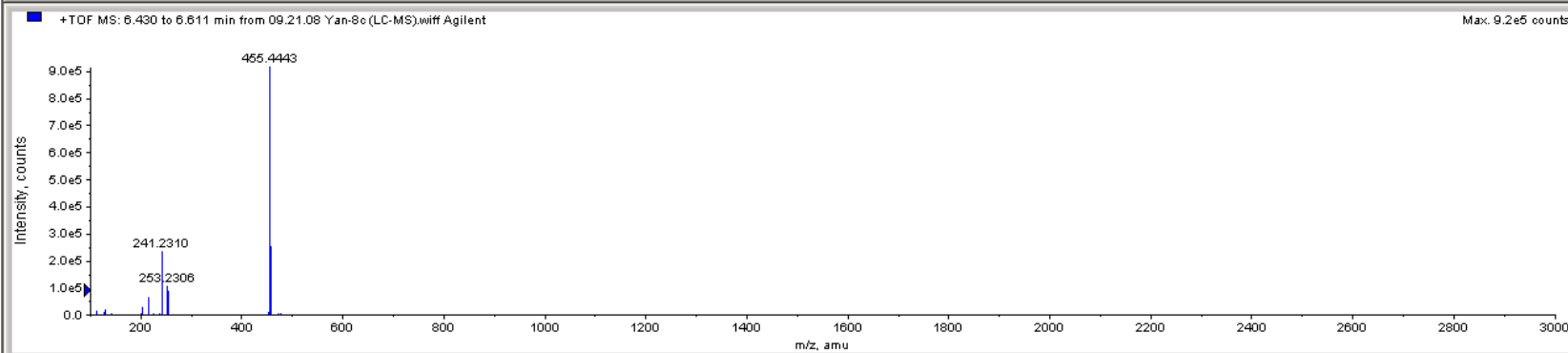
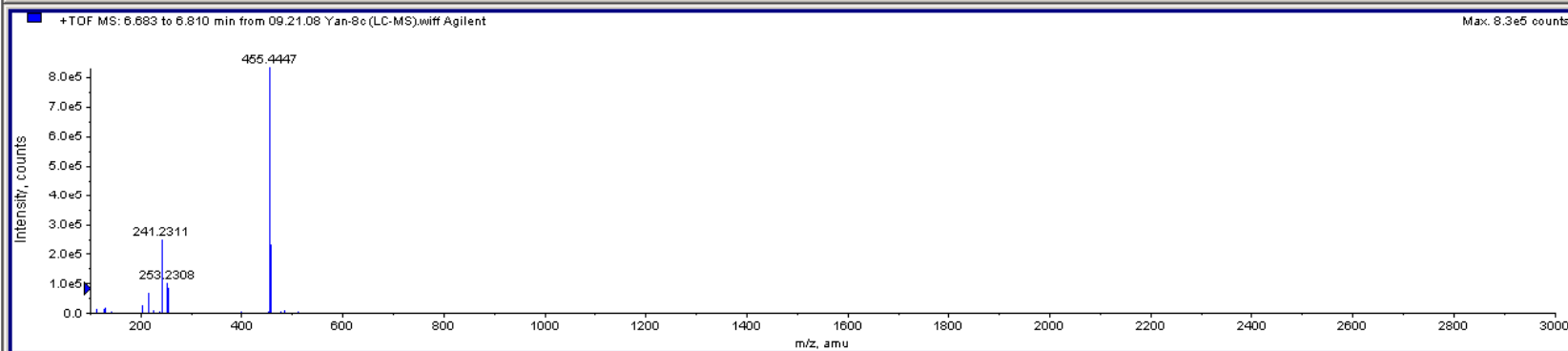
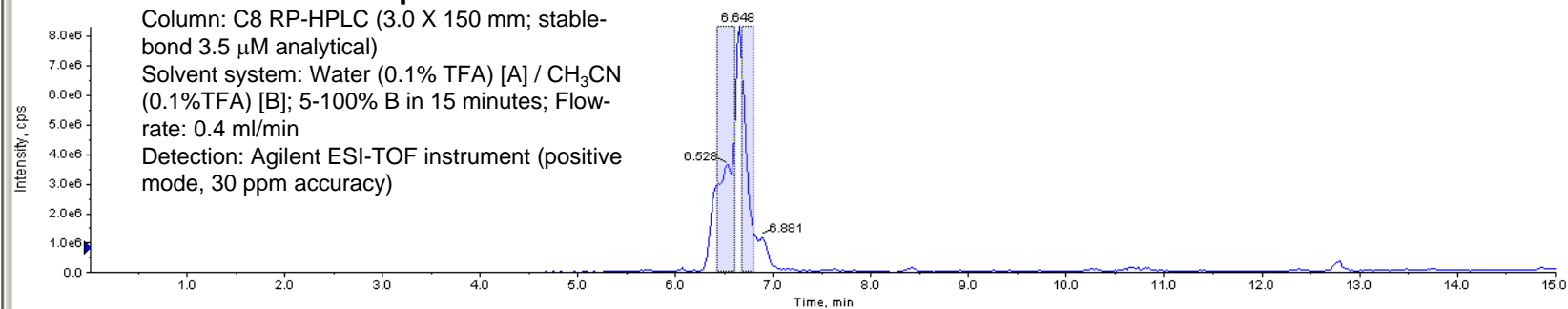
8c

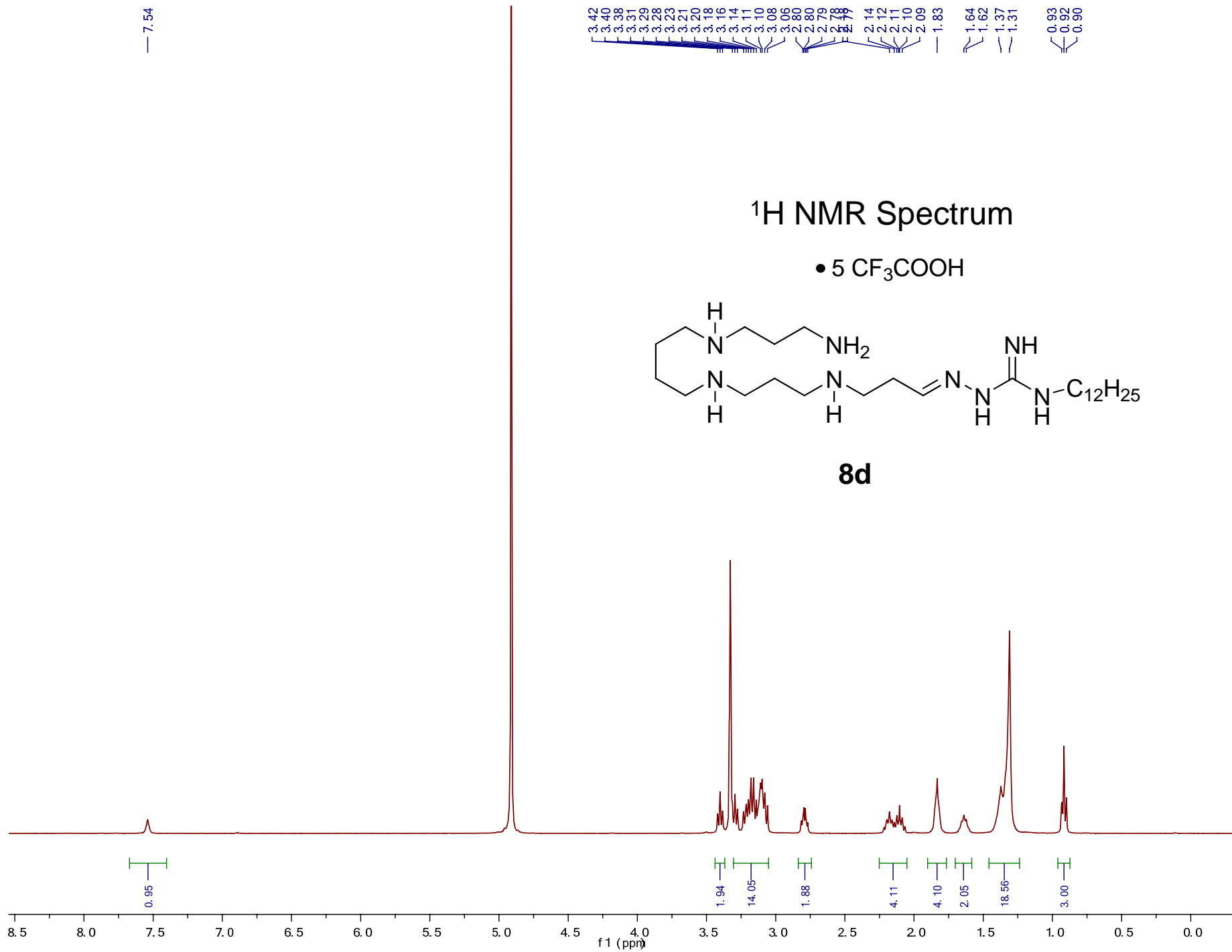




LC-MS Data: Compound 8c

Column: C8 RP-HPLC (3.0 X 150 mm; stable-bond 3.5 μ M analytical)
Solvent system: Water (0.1% TFA) [A] / CH₃CN (0.1%TFA) [B]; 5-100% B in 15 minutes; Flow-rate: 0.4 ml/min
Detection: Agilent ESI-TOF instrument (positive mode, 30 ppm accuracy)





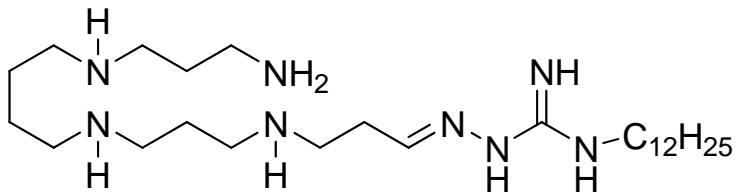
— 154.37
146.62
146.57
146.54

— 117.77
115.46
115.43

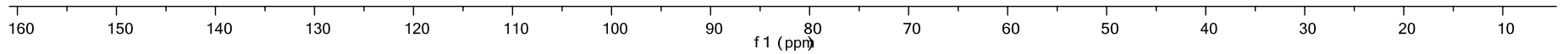
44.59
44.43
44.40
43.71
41.14
36.40
31.67
28.96
26.29
23.97
22.81
22.70
22.33
13.03

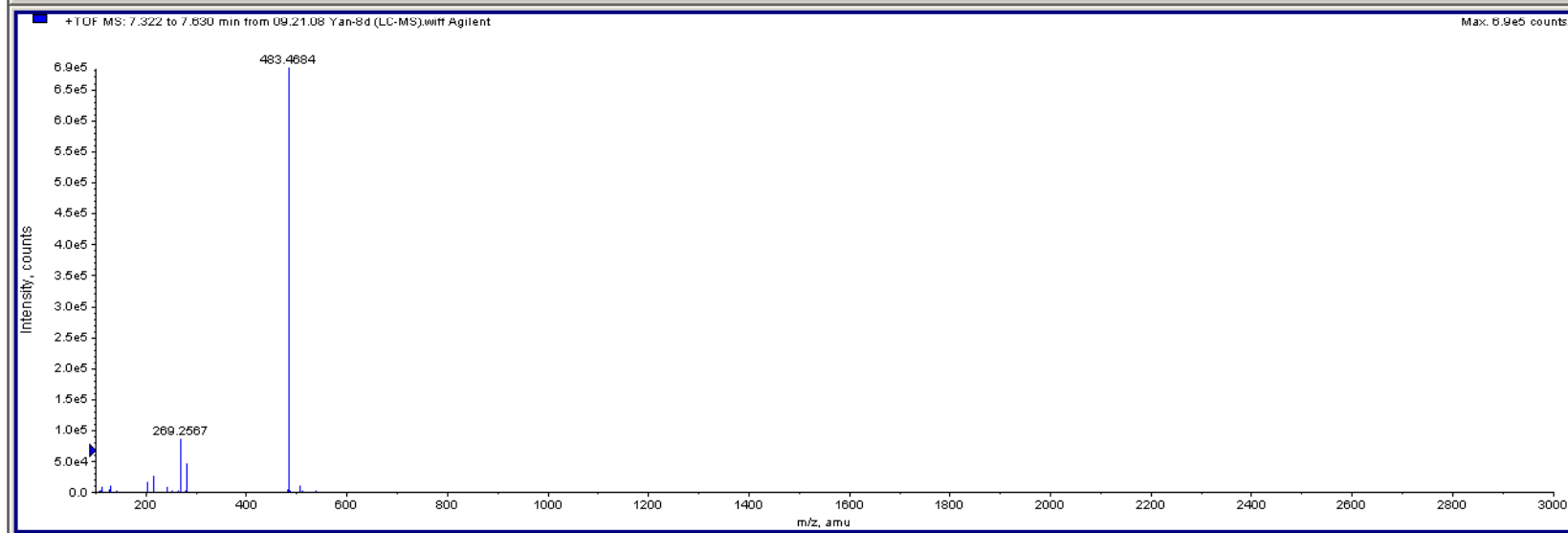
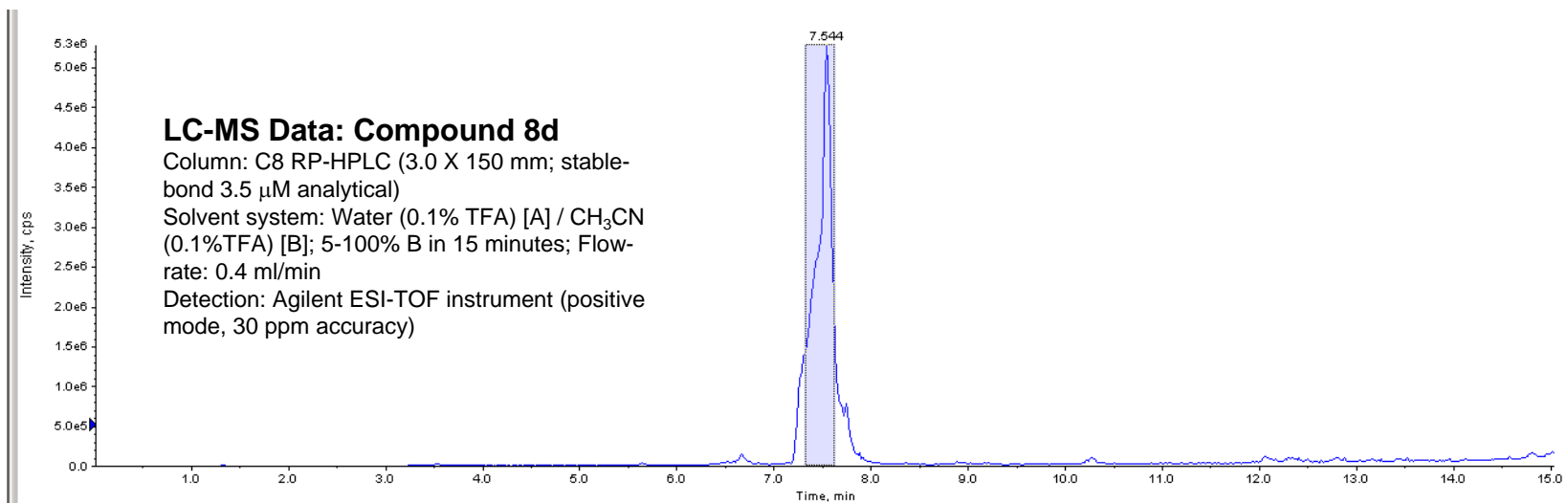
¹³C NMR Spectrum

• 5 CF₃COOH



8d

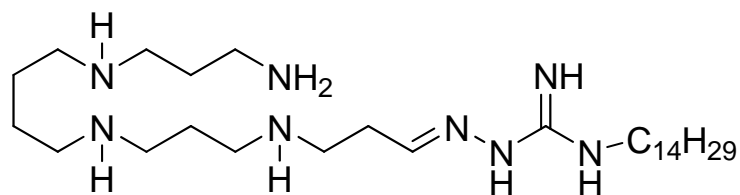




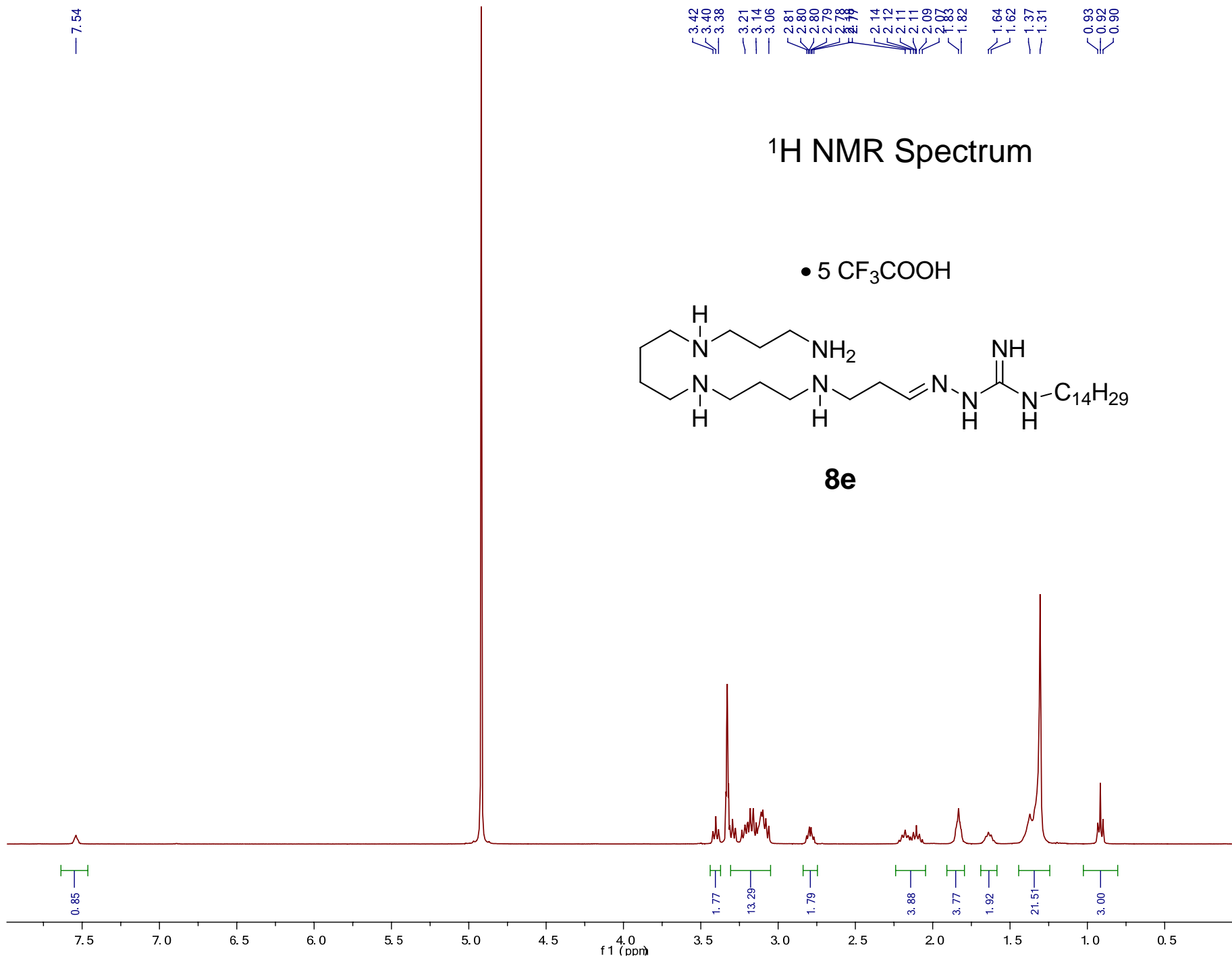
— 7.54

^1H NMR Spectrum

• 5 CF_3COOH

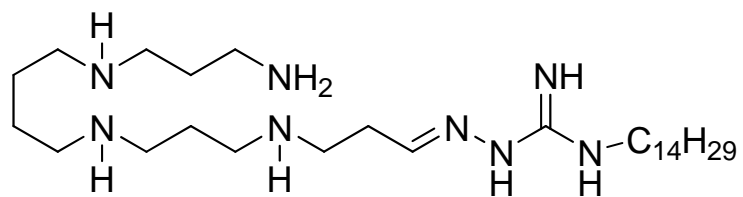


8e

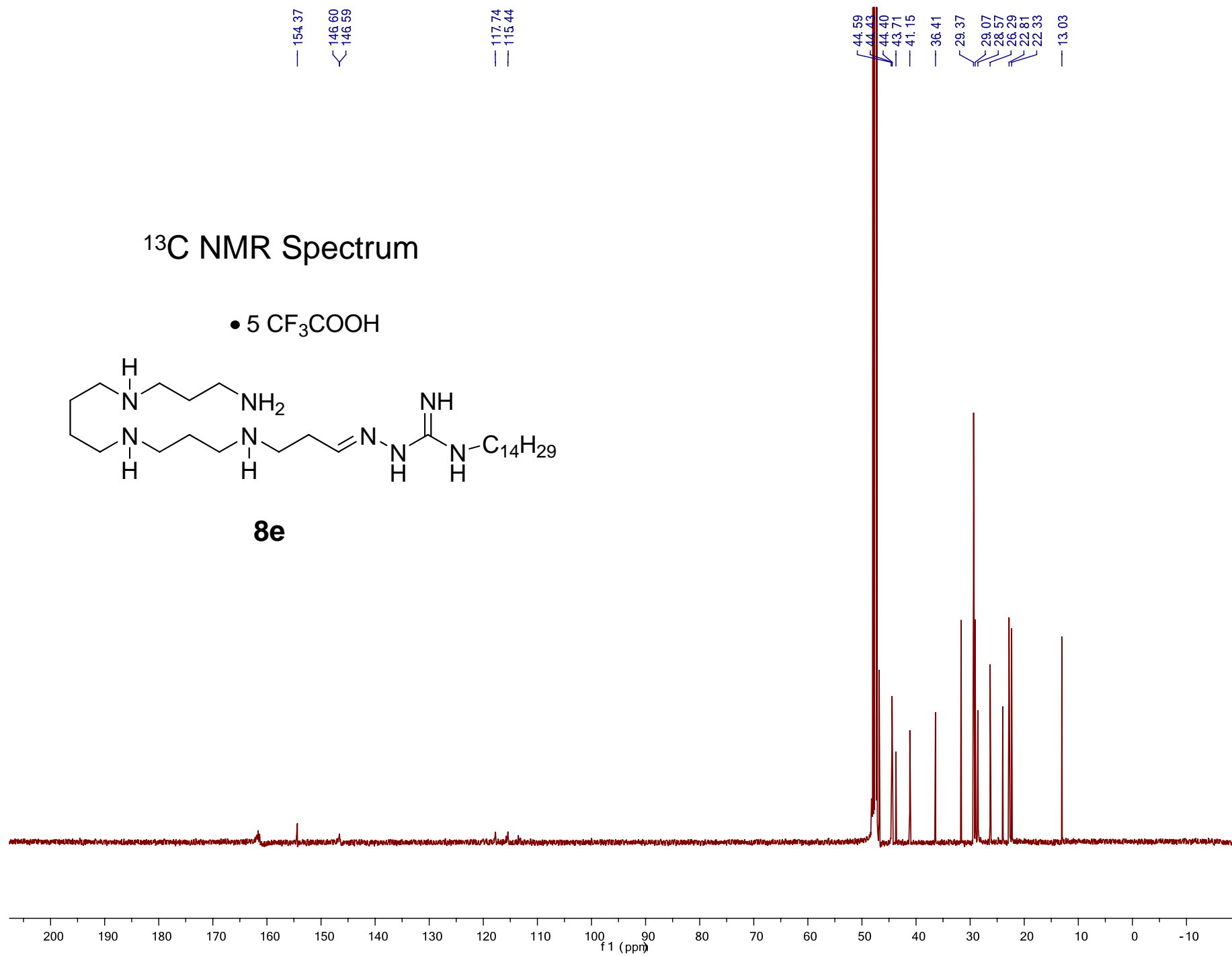


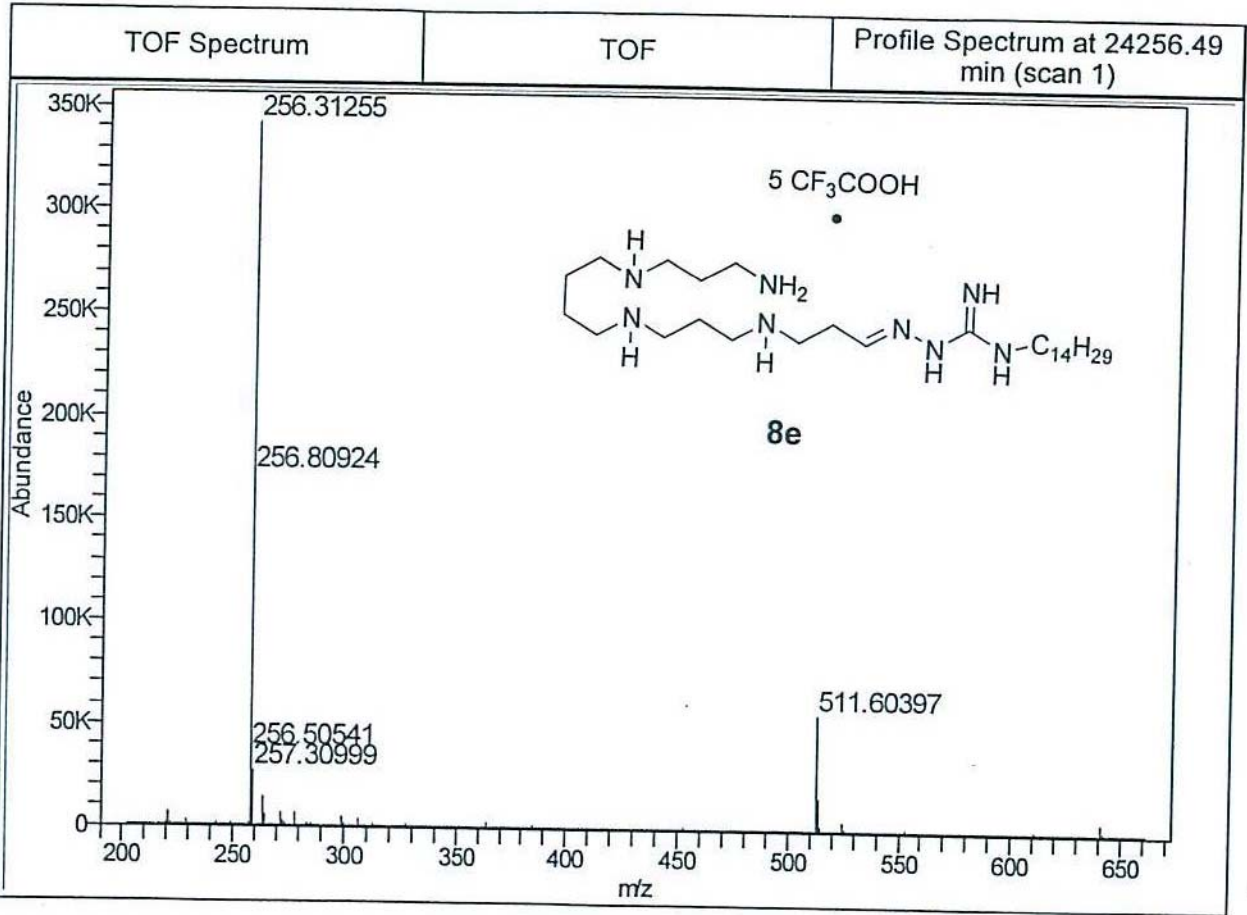
¹³C NMR Spectrum

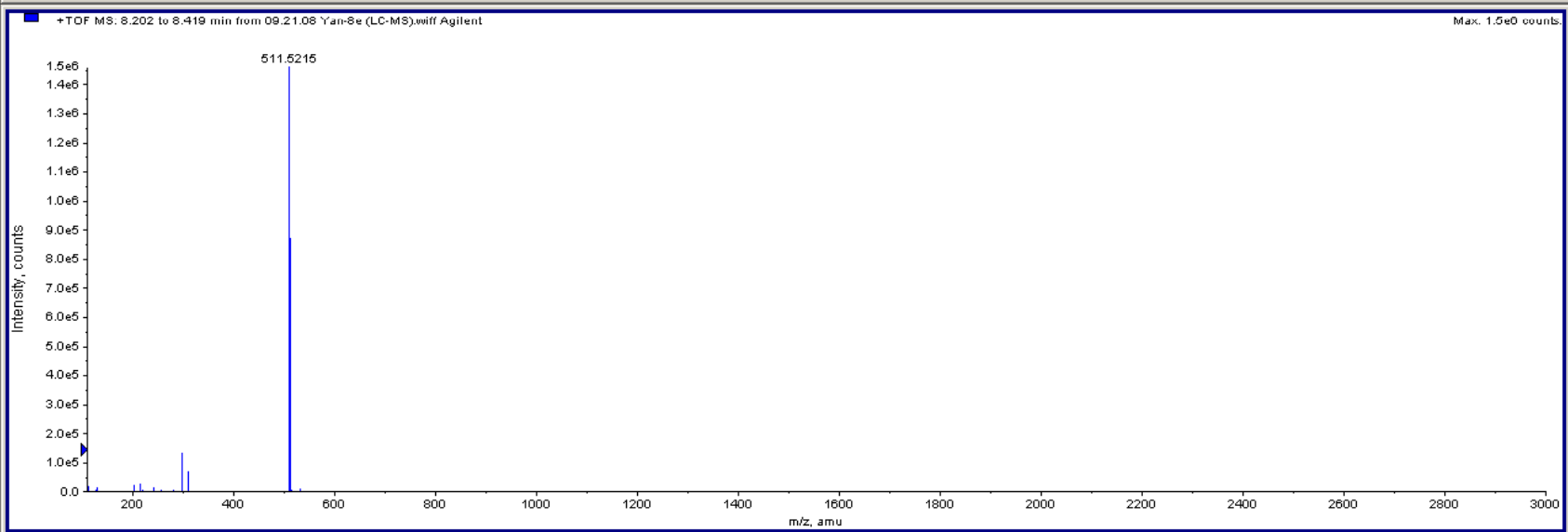
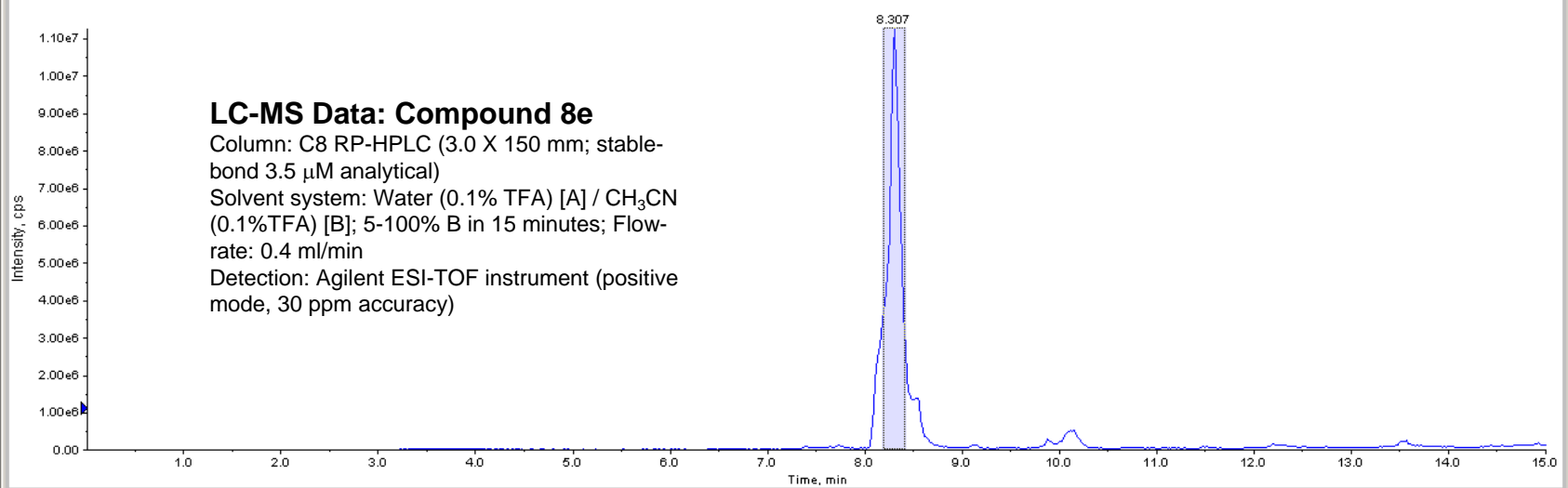
• 5 CF₃COOH



8e

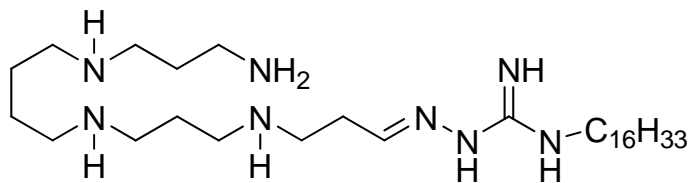




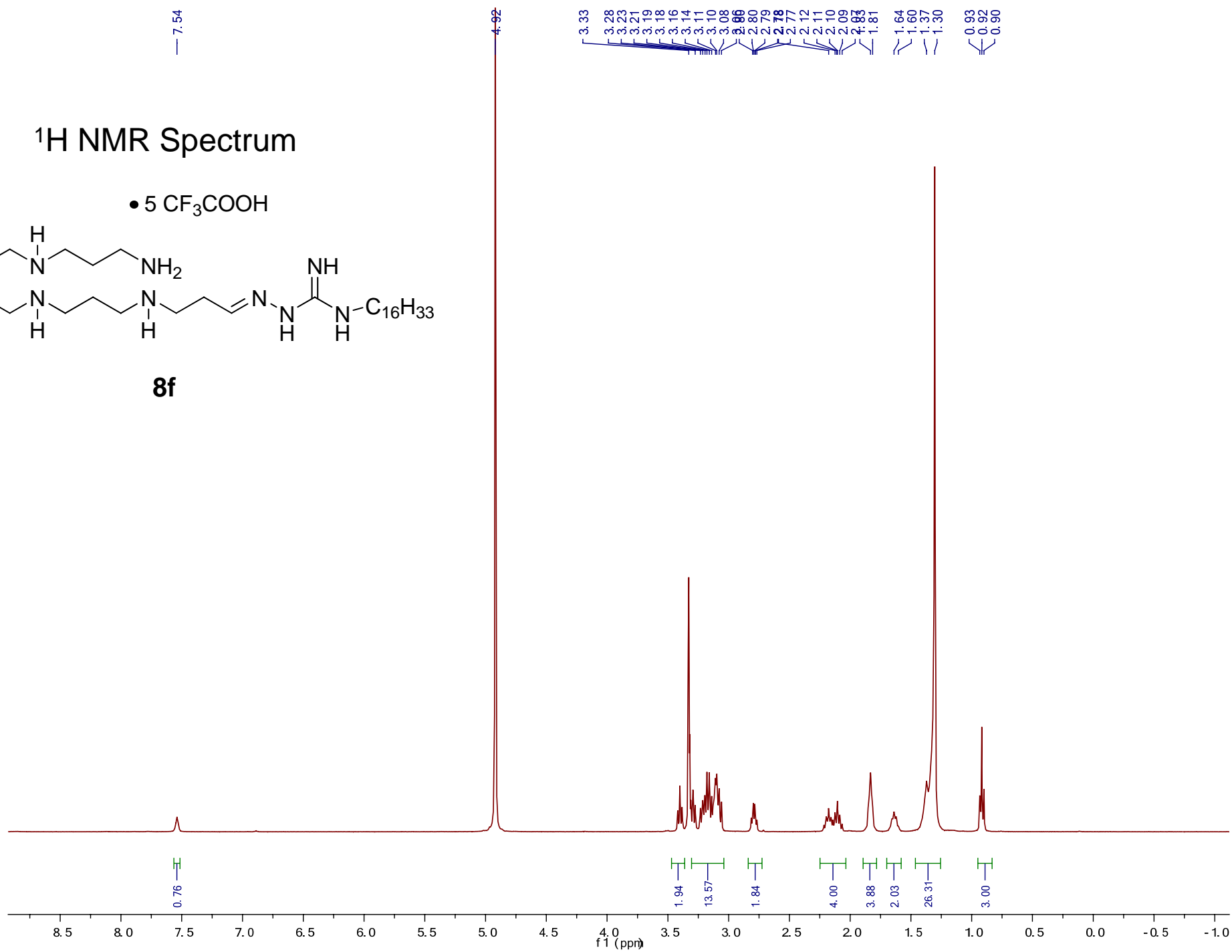


¹H NMR Spectrum

• 5 CF₃COOH



8f



— 154.36

146.63
146.59
146.55

— 117.81
— 115.47

44.60

44.41

43.72

41.15

— 36.41

— 31.67

— 28.57

— 26.29

— 23.96

22.81

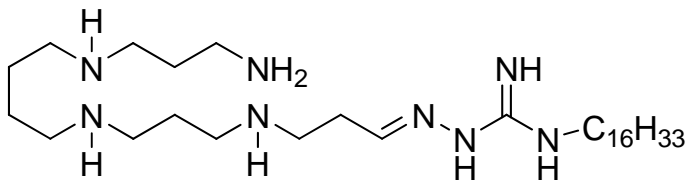
22.70

22.33

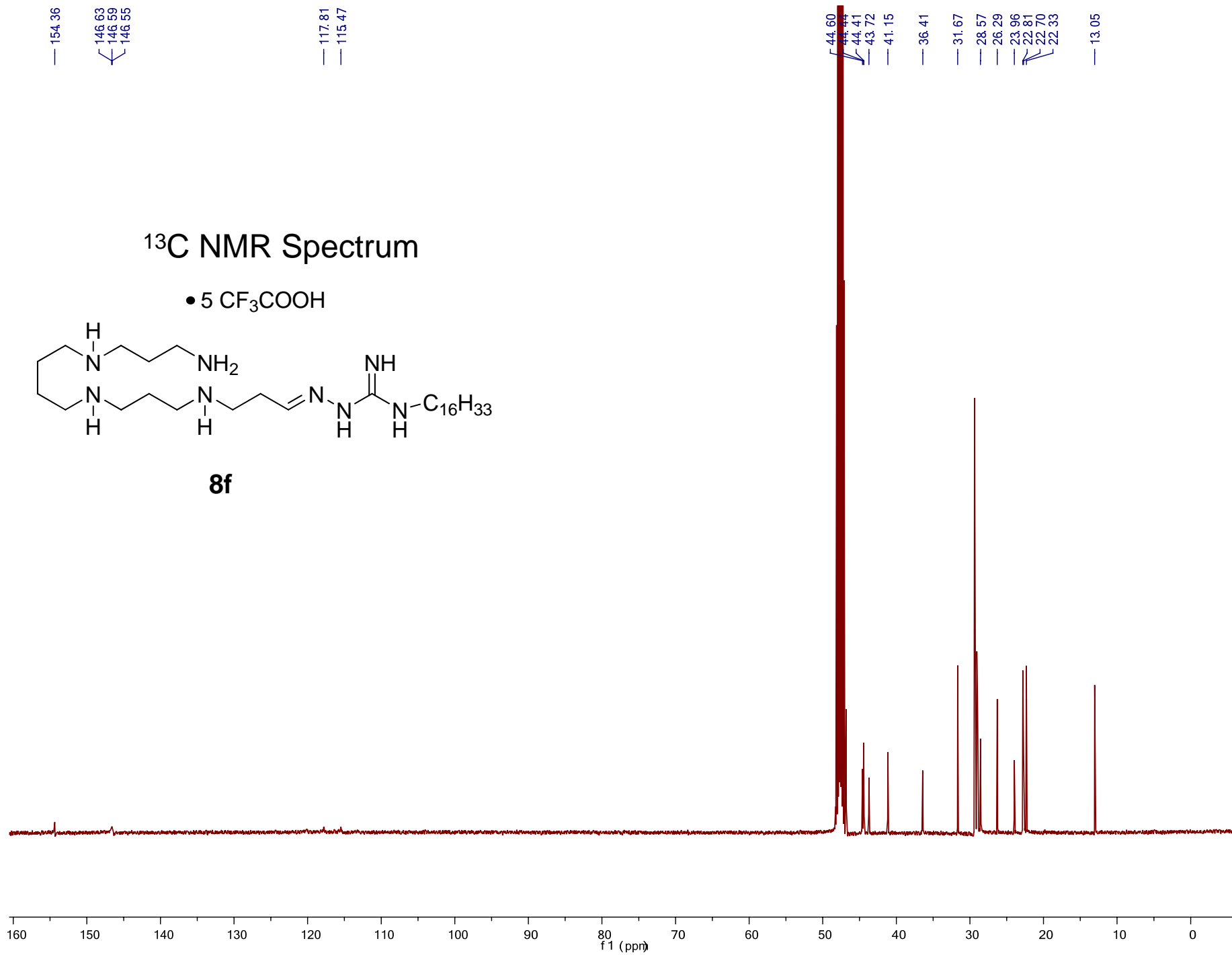
— 13.05

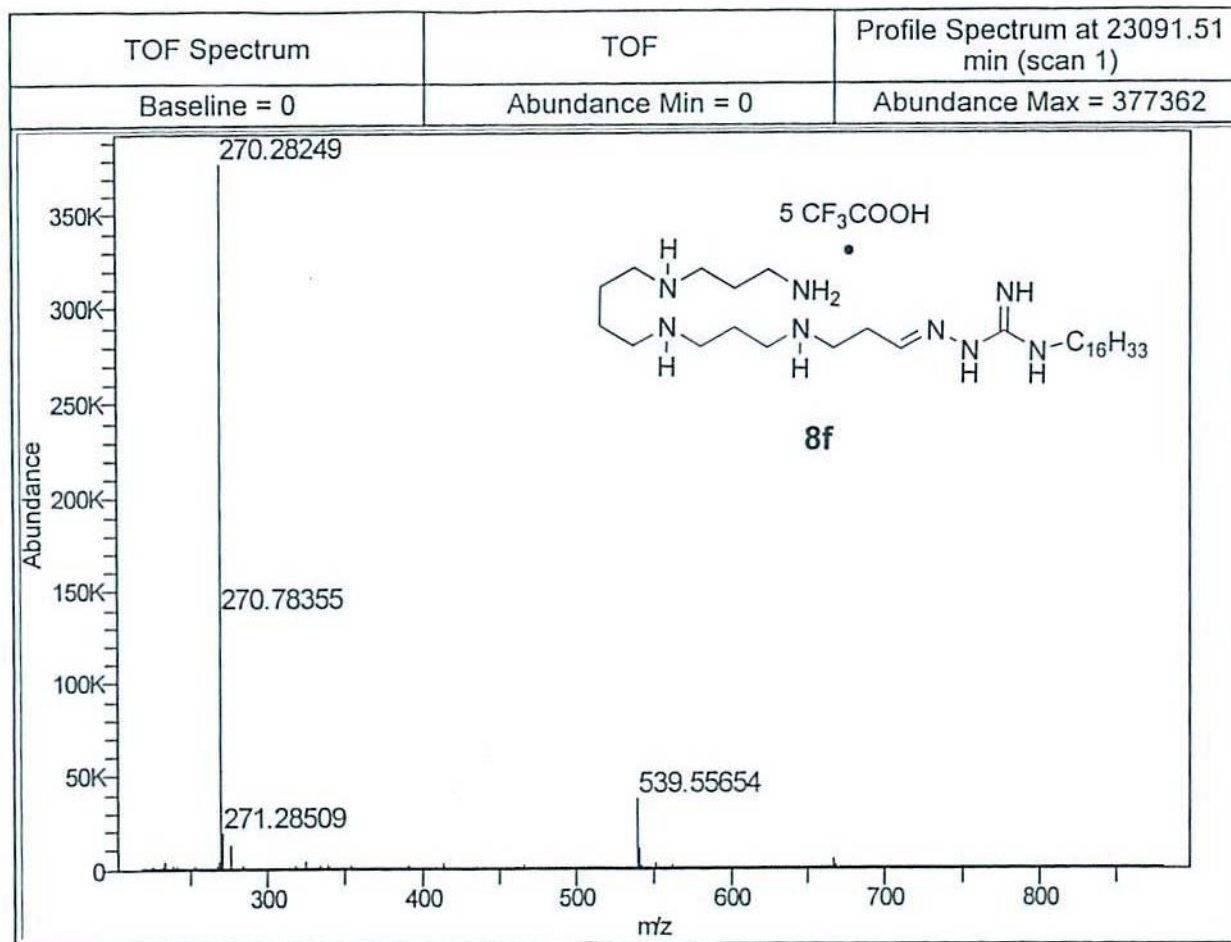
¹³C NMR Spectrum

• 5 CF₃COOH



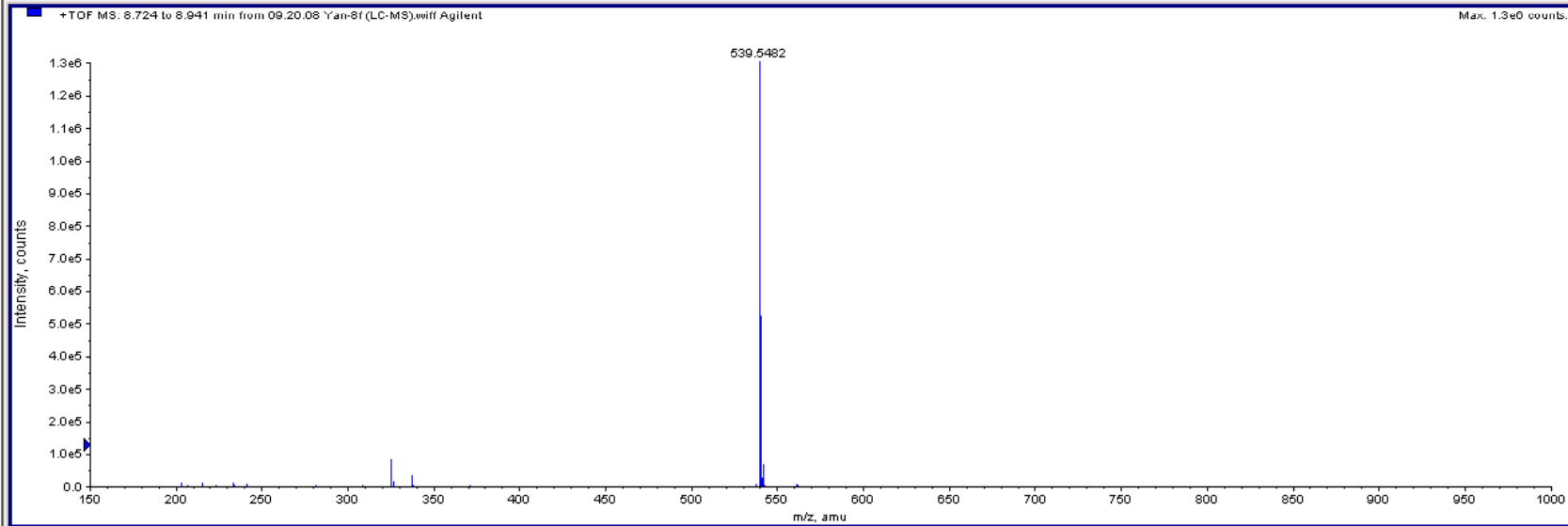
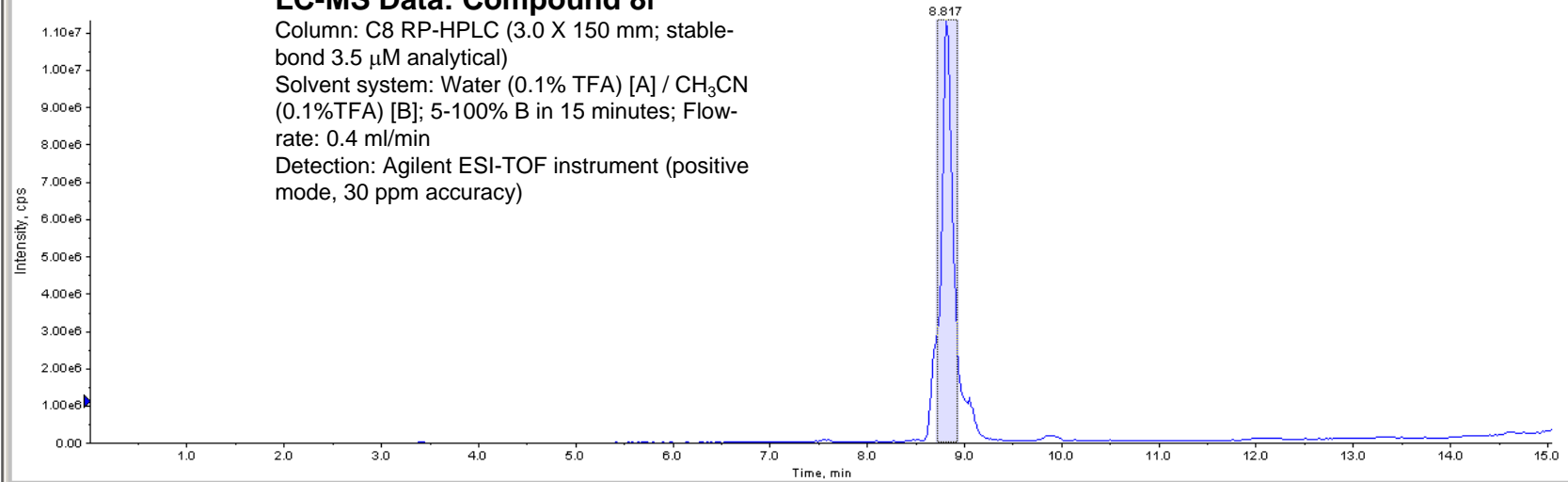
8f



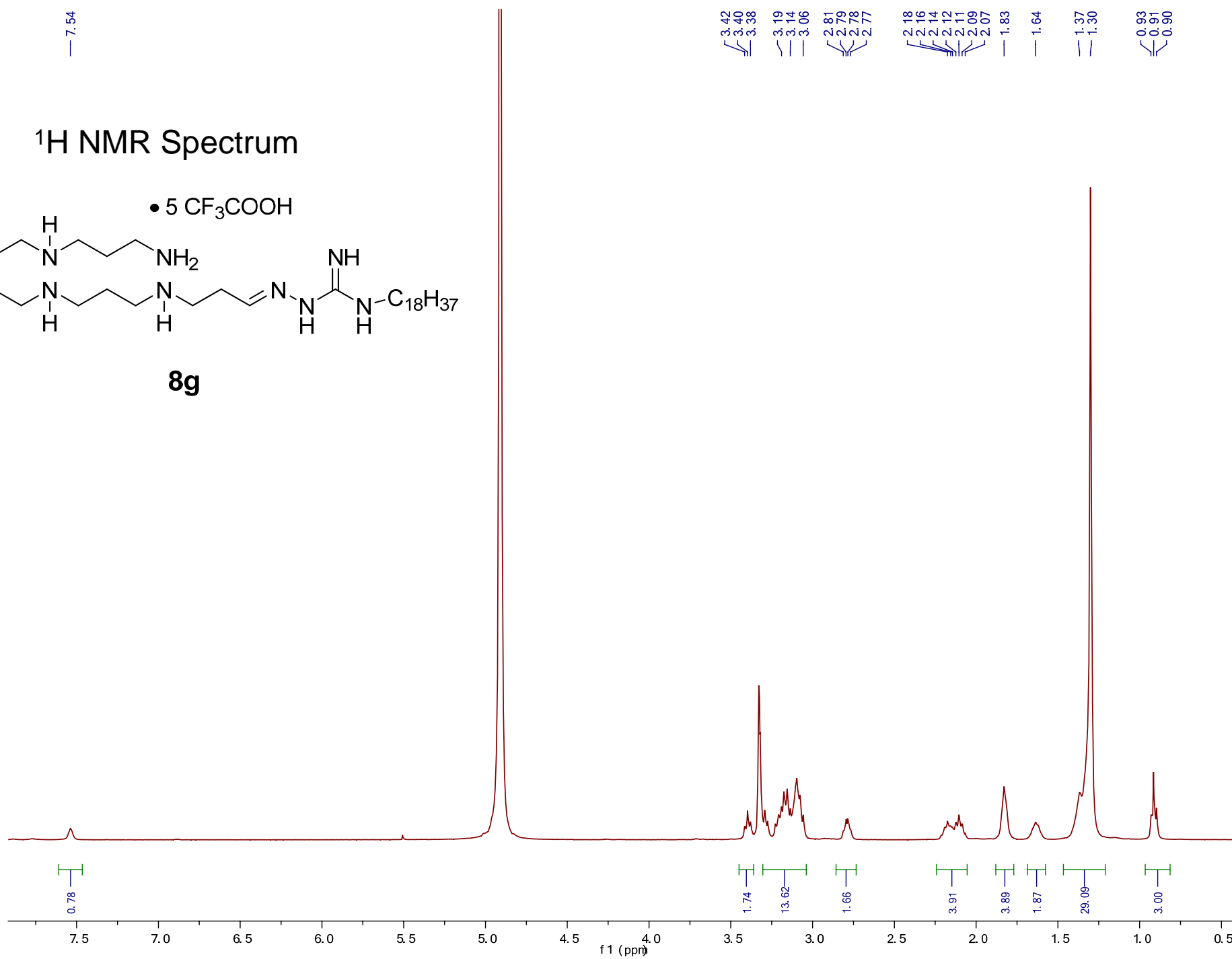
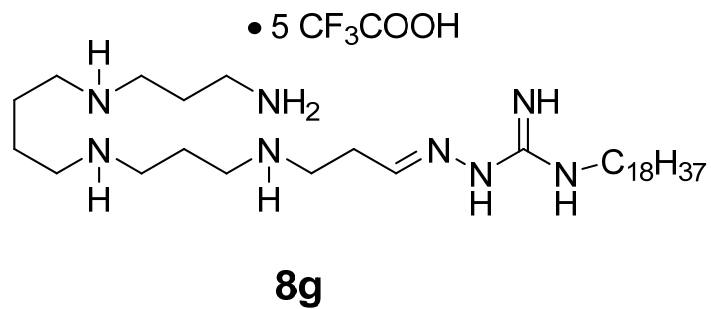


LC-MS Data: Compound 8f

Column: C8 RP-HPLC (3.0 X 150 mm; stable-bond 3.5 μ M analytical)
Solvent system: Water (0.1% TFA) [A] / CH₃CN (0.1%TFA) [B]; 5-100% B in 15 minutes; Flow-rate: 0.4 ml/min
Detection: Agilent ESI-TOF instrument (positive mode, 30 ppm accuracy)



¹H NMR Spectrum

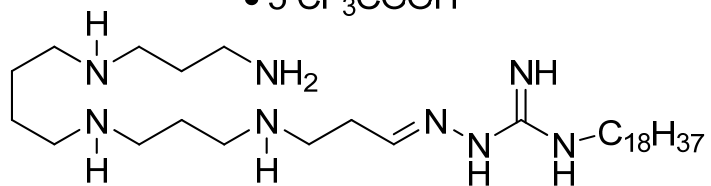


— 154.78

147.09
147.03

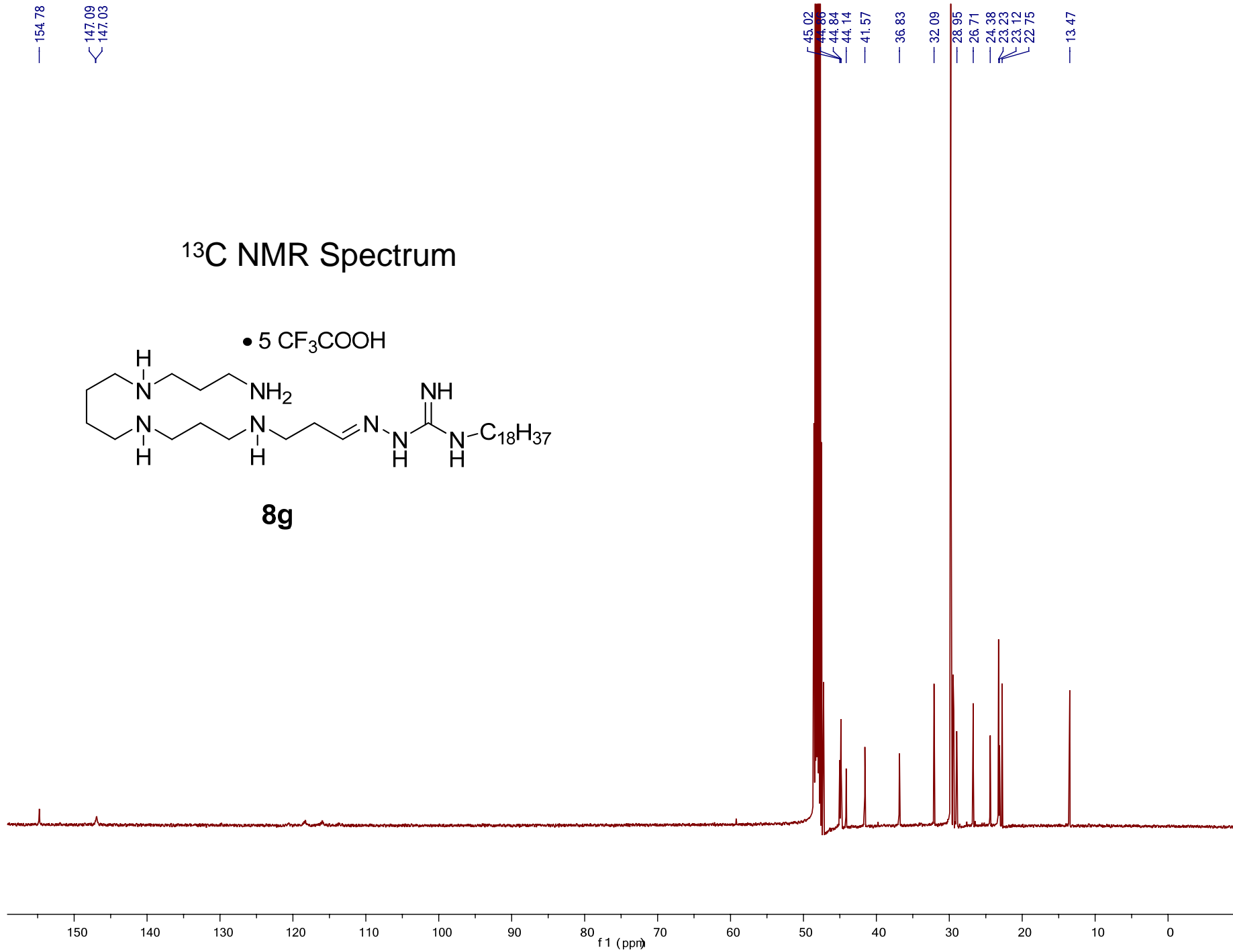
¹³C NMR Spectrum

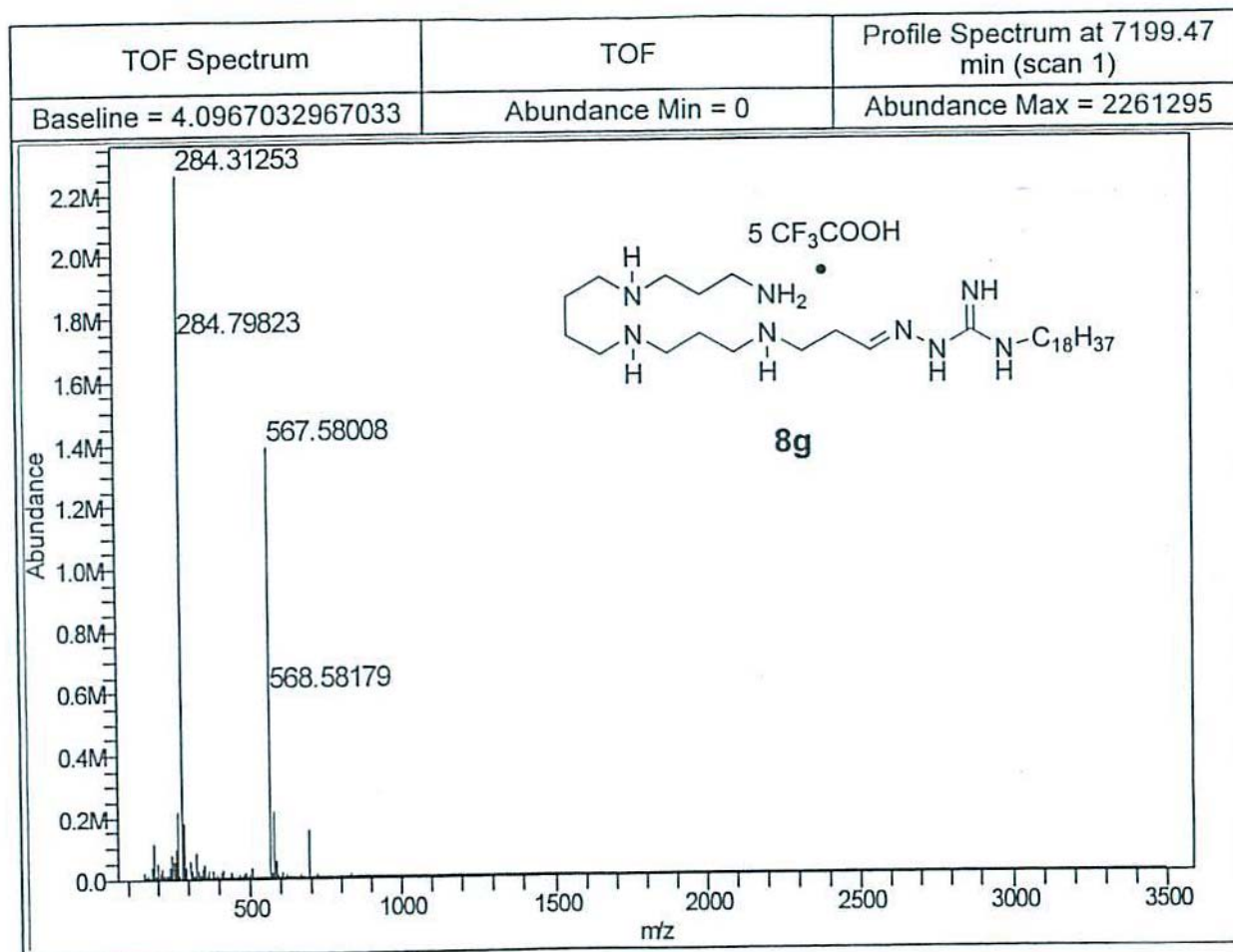
• 5 CF₃COOH



8g

45.02
44.84
44.14
41.57
36.83
32.09
28.95
26.71
24.38
23.23
23.12
22.75
13.47



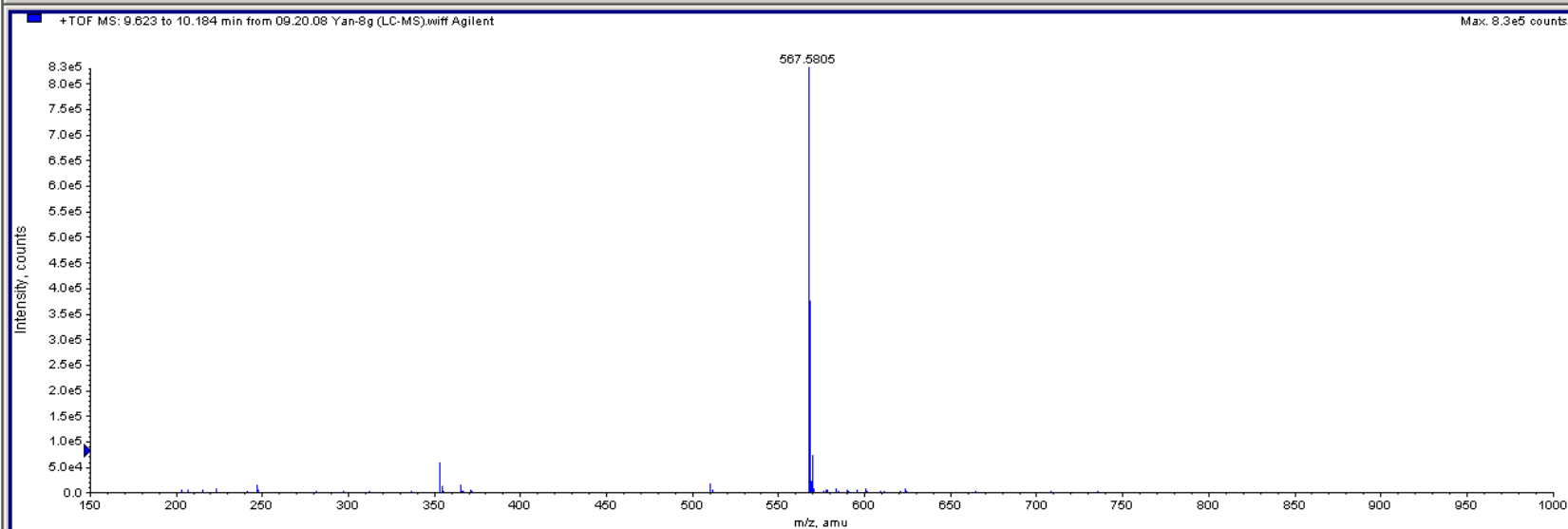
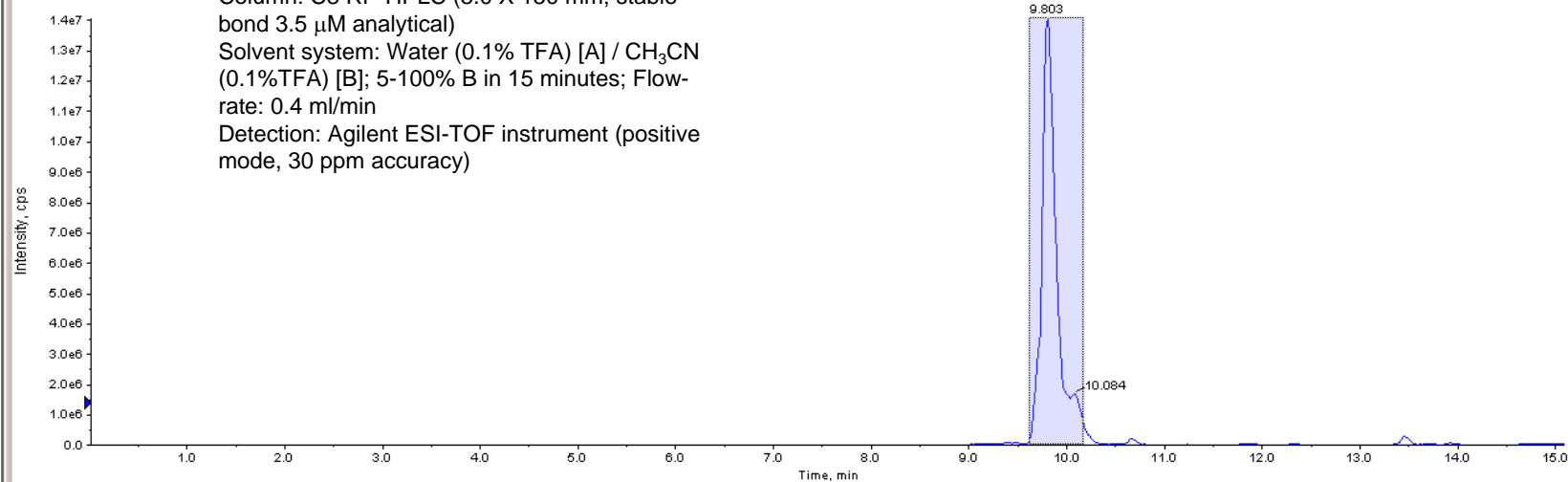


LC-MS Data: Compound 8g

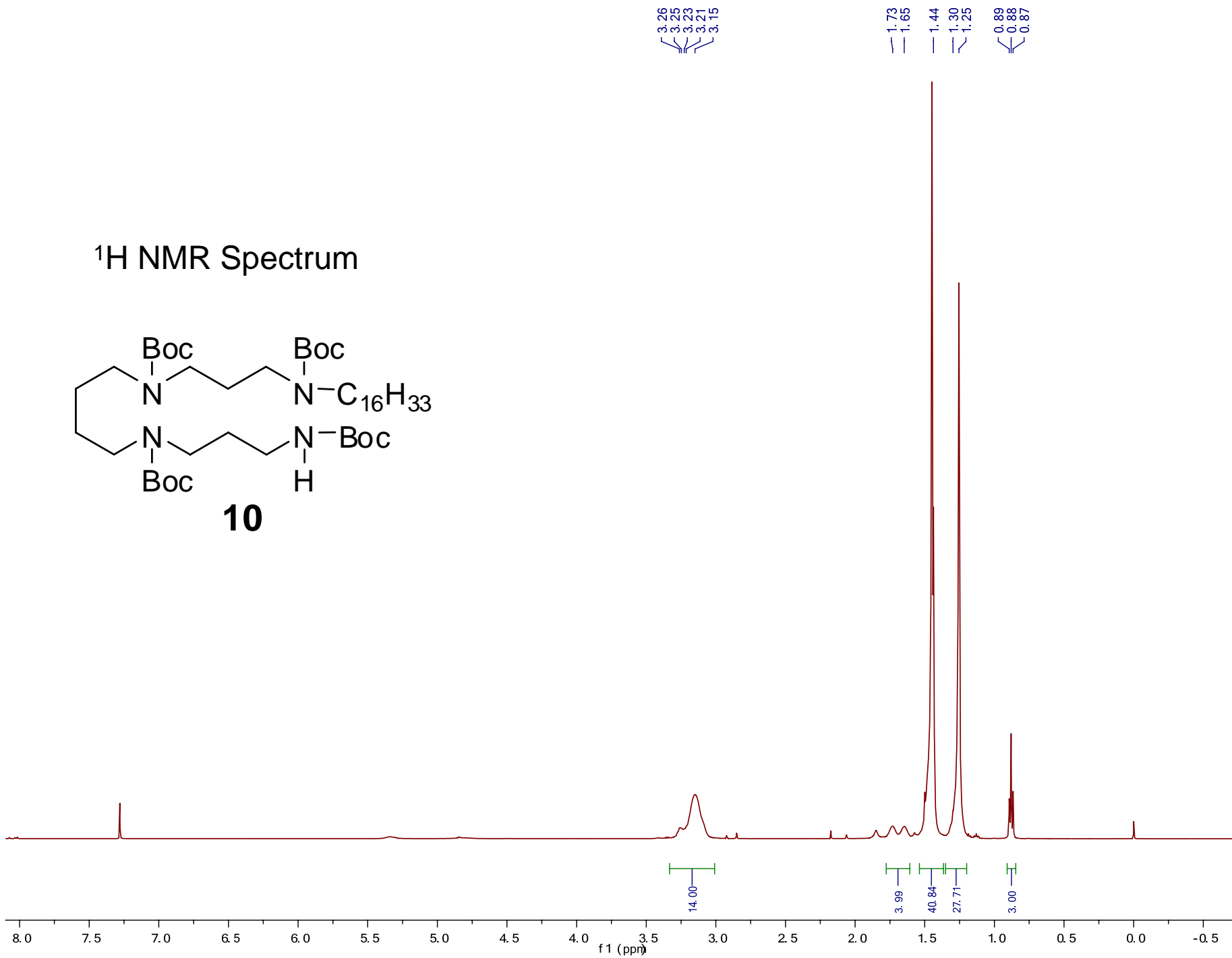
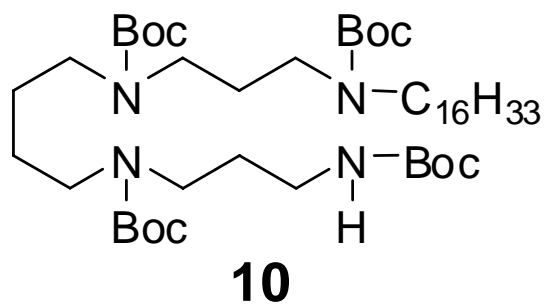
Column: C8 RP-HPLC (3.0 X 150 mm; stable-bond 3.5 μ M analytical)

Solvent system: Water (0.1% TFA) [A] / CH₃CN (0.1%TFA) [B]; 5-100% B in 15 minutes; Flow-rate: 0.4 ml/min

Detection: Agilent ESI-TOF instrument (positive mode, 30 ppm accuracy)



^1H NMR Spectrum



156.11
155.95
155.48

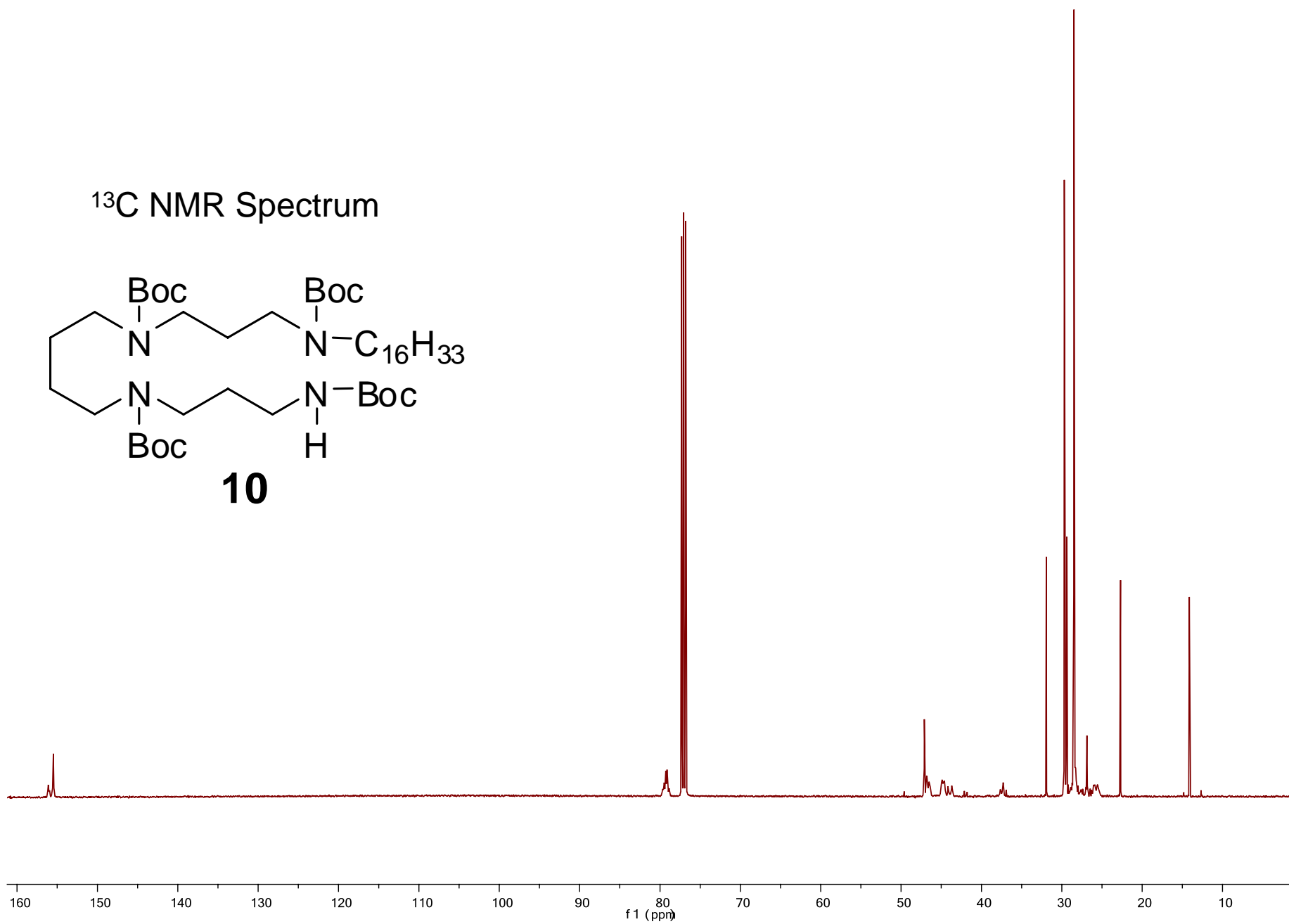
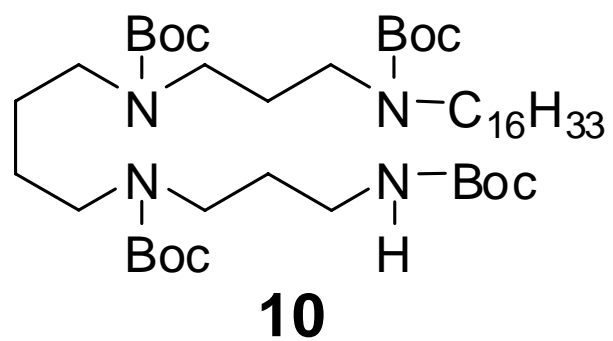
79.27
79.12

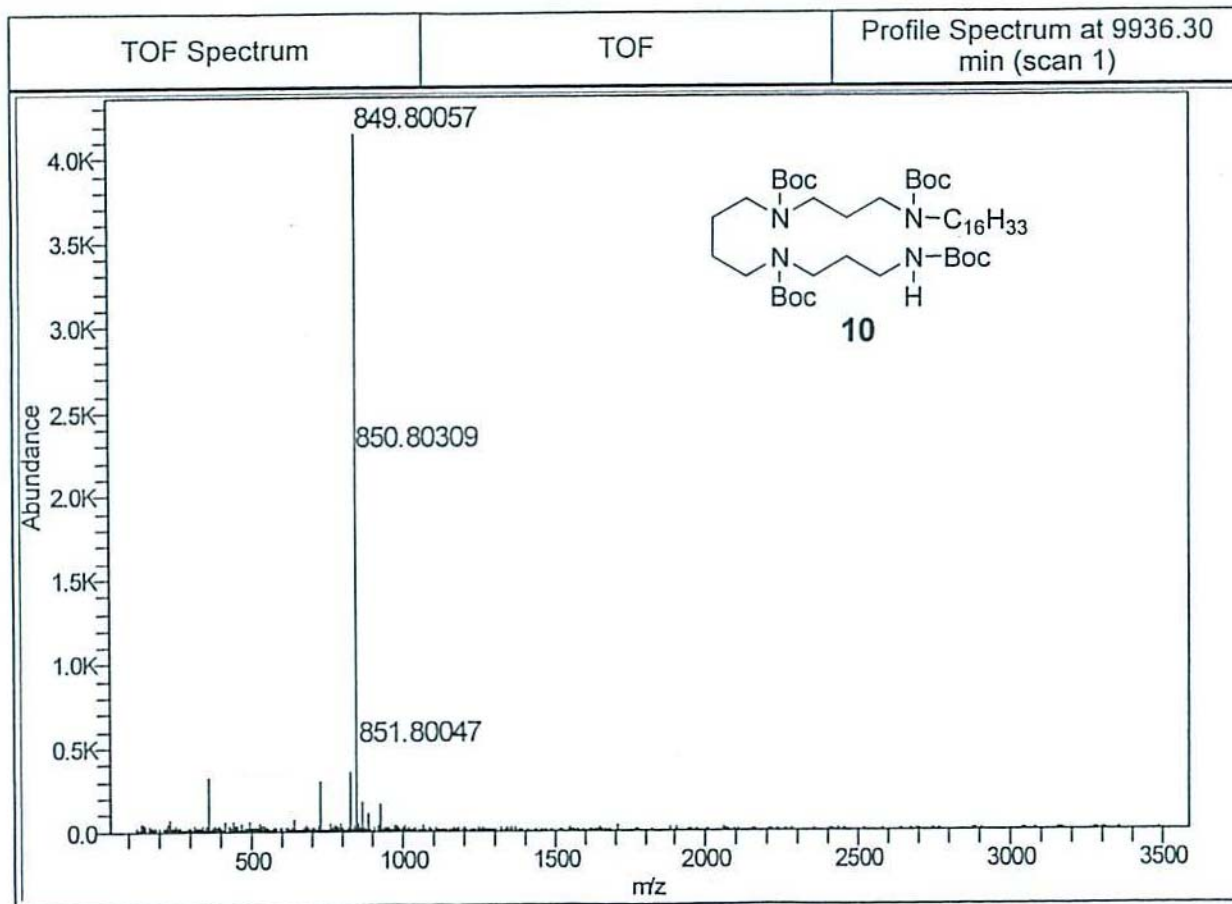
47.10
46.80
46.63
46.52
44.90
44.66
44.60
44.18
43.68

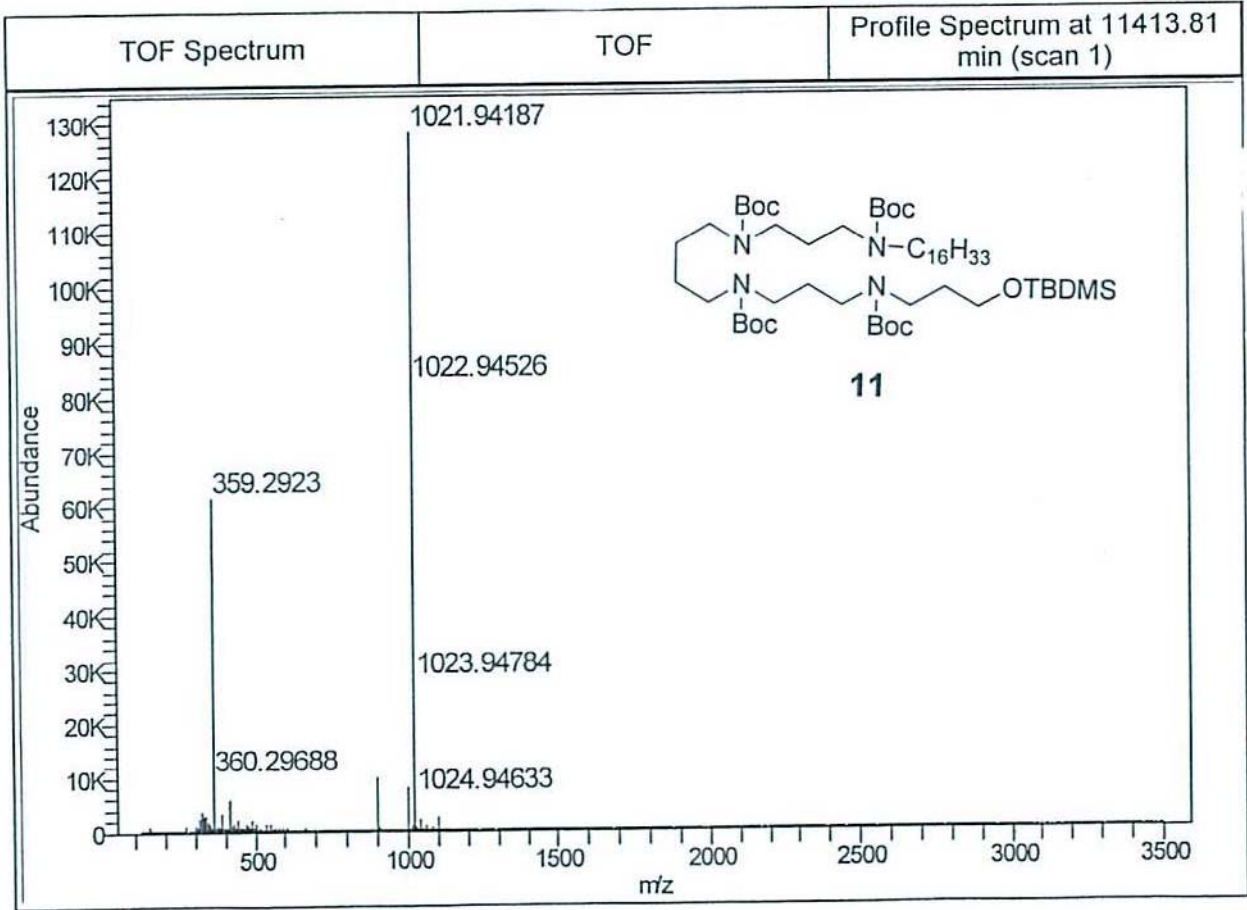
37.66
37.29
31.93
29.43
26.87
25.55
22.69

14.13

¹³C NMR Spectrum







— 9.79

— 3.51

— 3.16

— 2.70

— 1.73

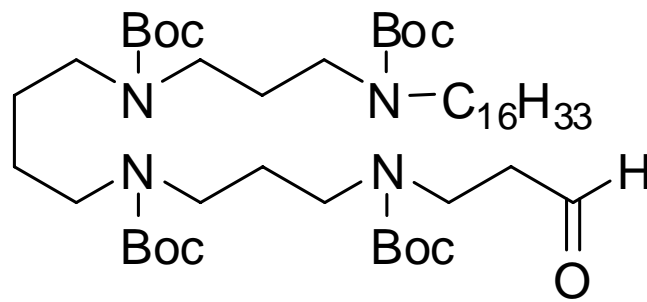
— 1.25

— 0.89

— 0.88

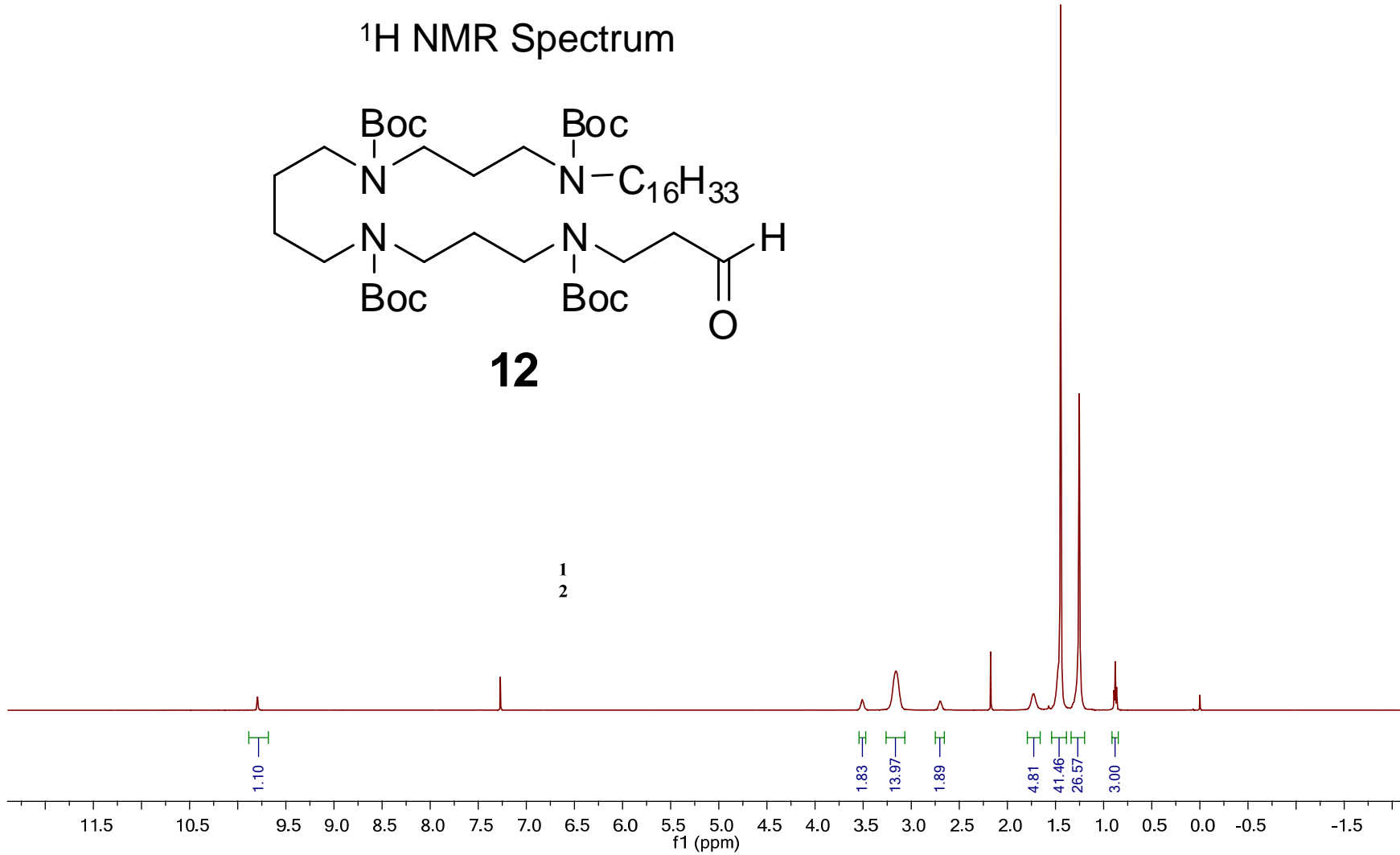
— 0.87

¹H NMR Spectrum



12

1
2



— 200.99

— 155.47

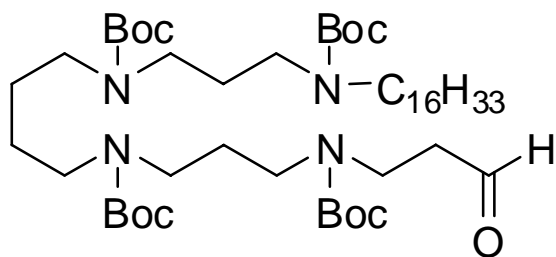
79.93
79.34
79.25
79.11

47.08
45.82
45.80
45.16
44.69
44.63
44.61
43.63
43.32
41.12
41.09

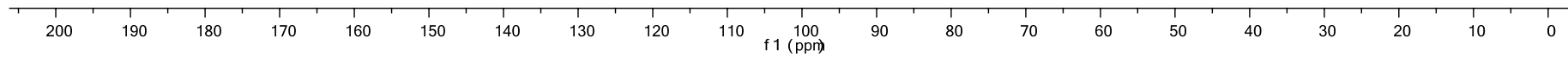
29.67
29.43
28.42
27.52
25.60
22.69

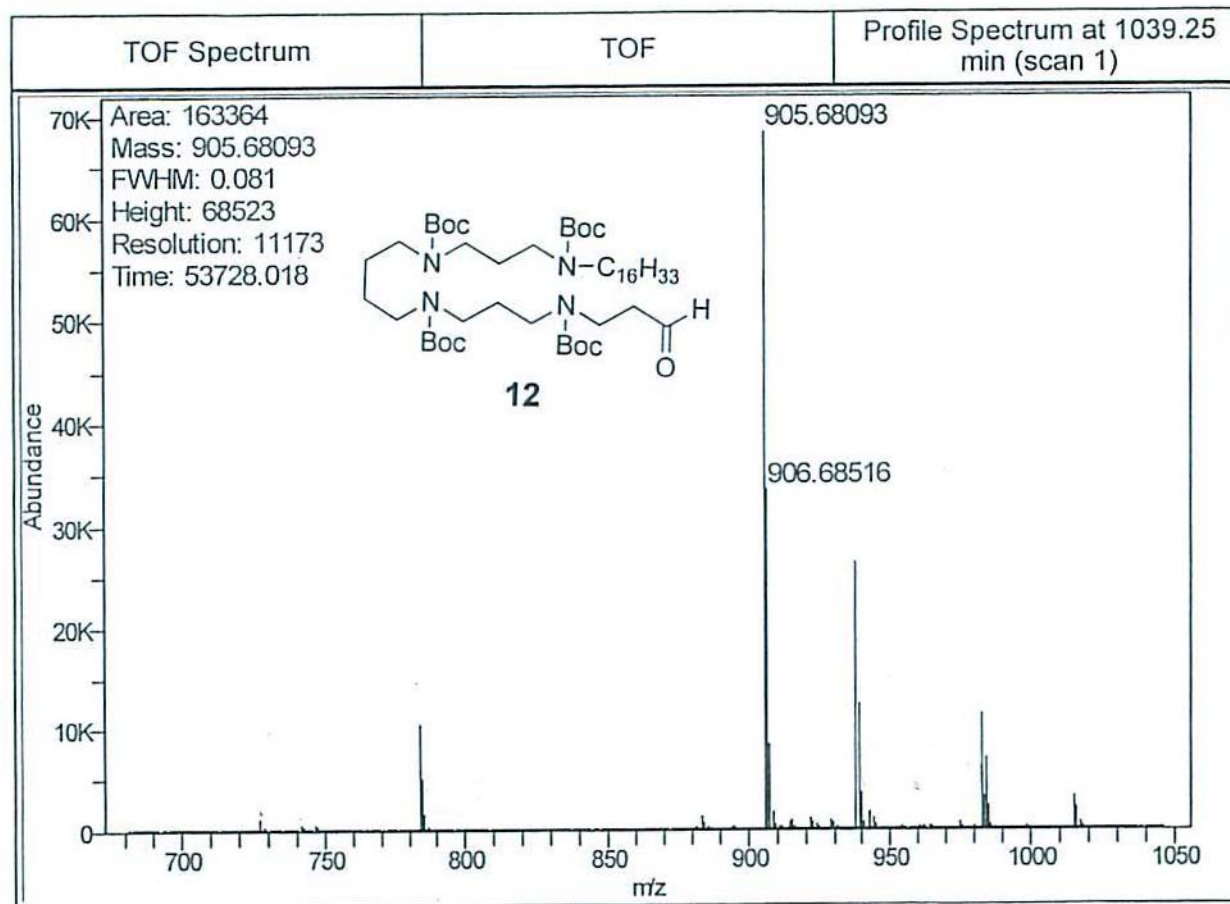
— 14.13

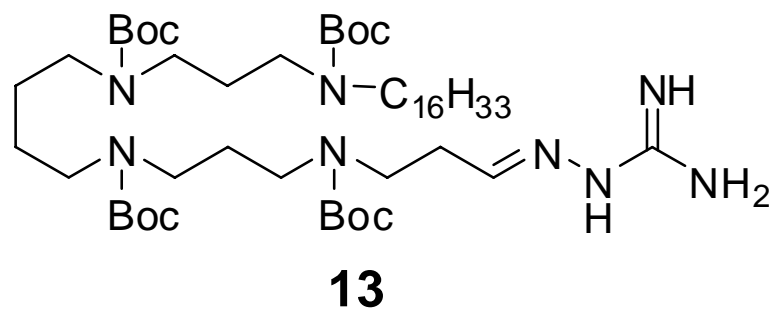
¹³C NMR Spectrum



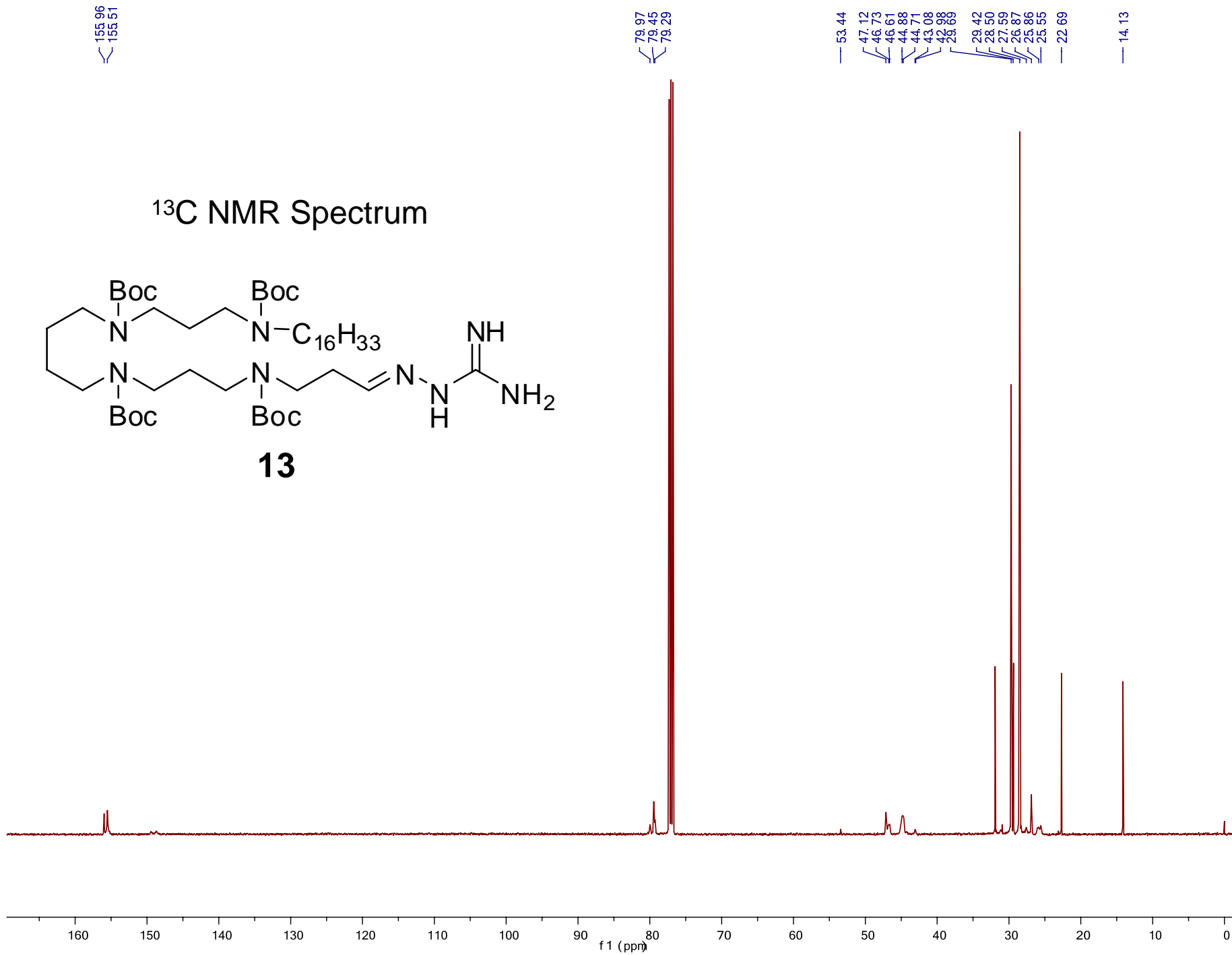
12



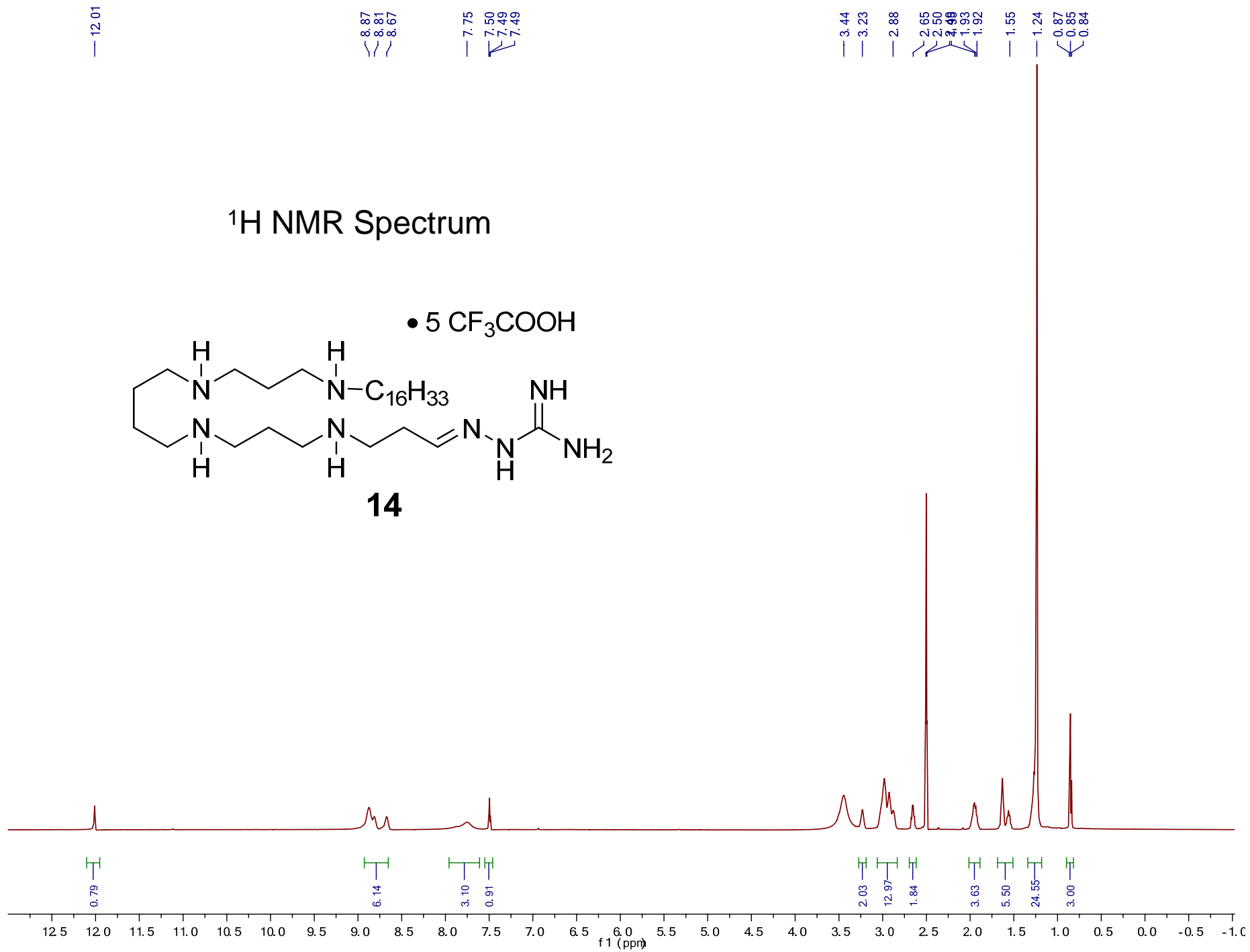
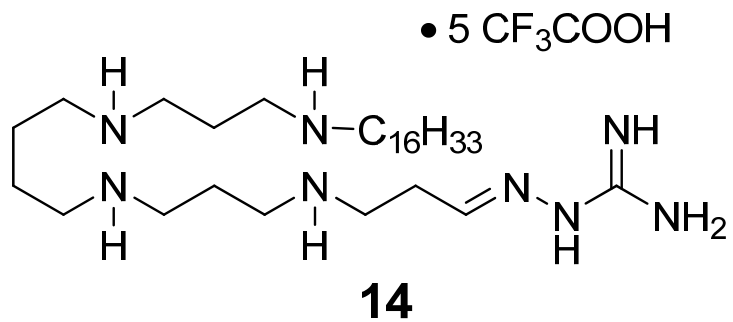




¹³C NMR Spectrum

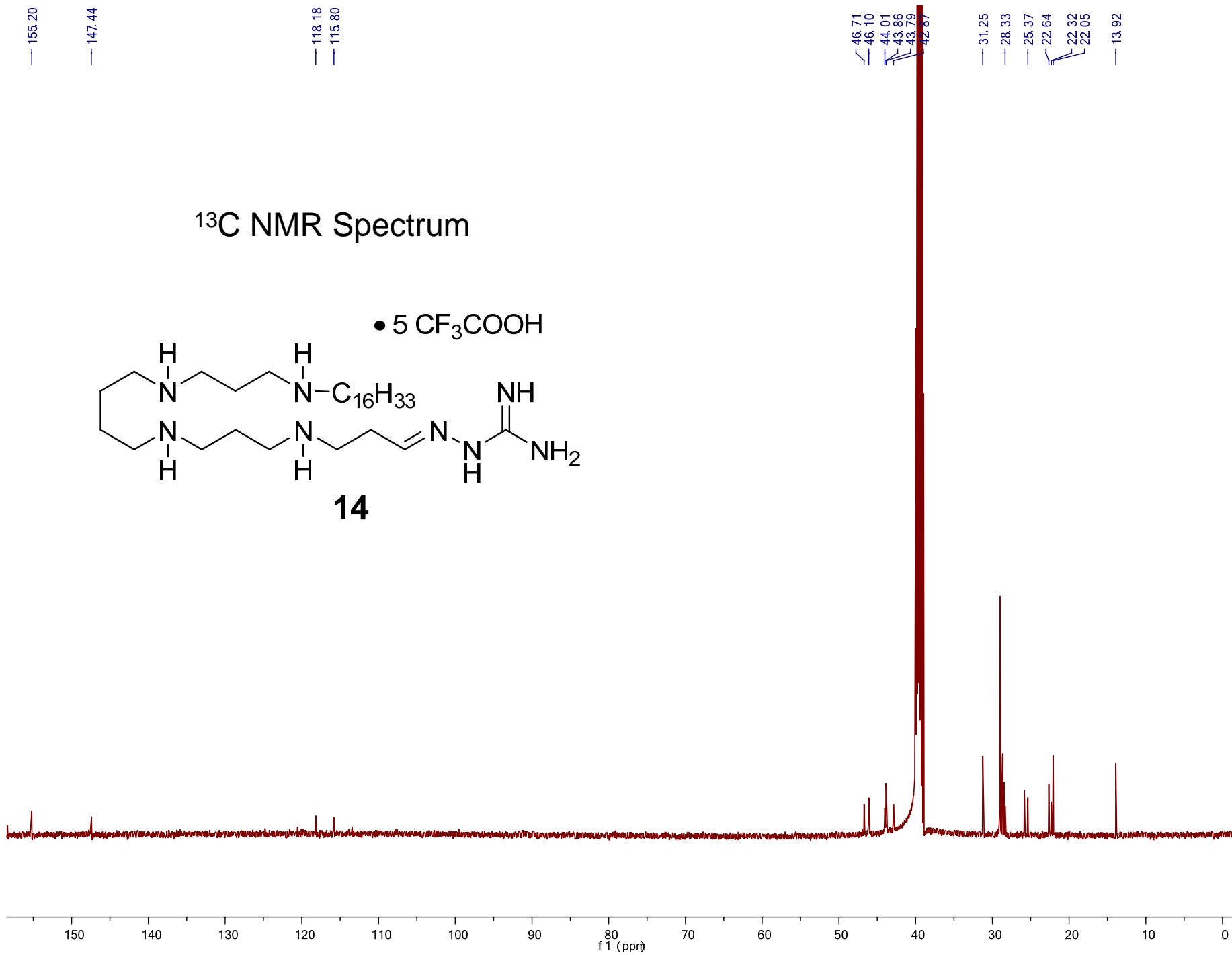
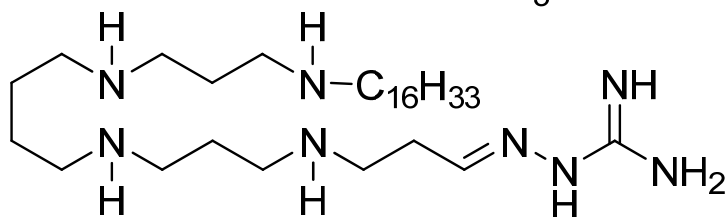


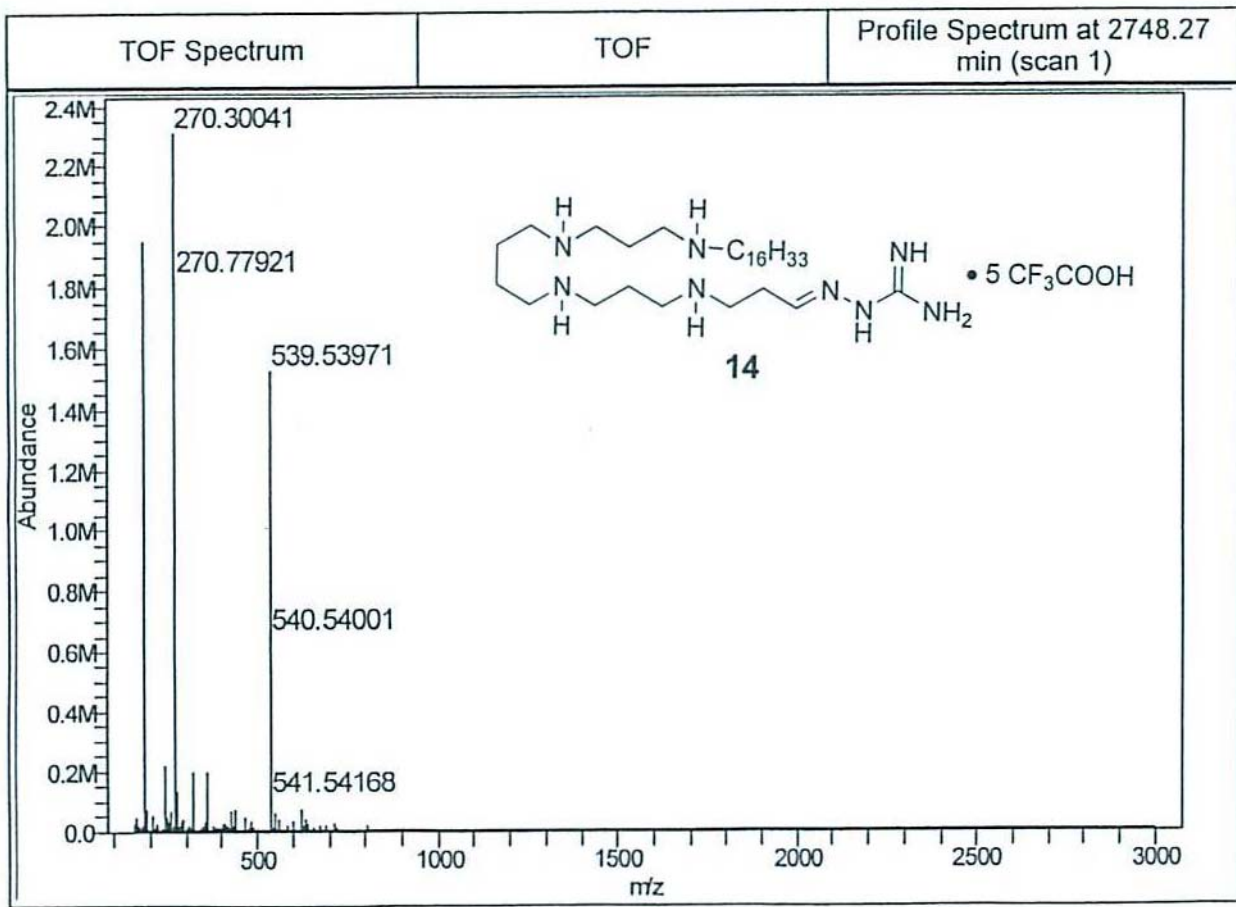
^1H NMR Spectrum



¹³C NMR Spectrum

• 5 CF₃COOH





LC-MS Data: Compound 14

Column: C8 RP-HPLC (3.0 X 150 mm; stable-bond 3.5 μ M analytical)

Solvent system: Water (0.1% TFA) [A] / CH₃CN (0.1%TFA) [B]; 5-100% B in 15 minutes; Flow-rate: 0.4 ml/min

Detection: Agilent ESI-TOF instrument (positive mode, 30 ppm accuracy)

