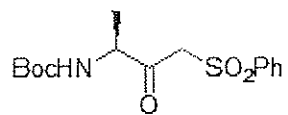# Supplementary Materials: Synthesis of Chiral, Enantiopure Allylic Amines by the Julia Olefination of α-Amino Esters

Fabio Benedetti, Federico Berti, Lidia Fanfoni, Michele Garbo, Giorgia Regini and Fulvia Felluga
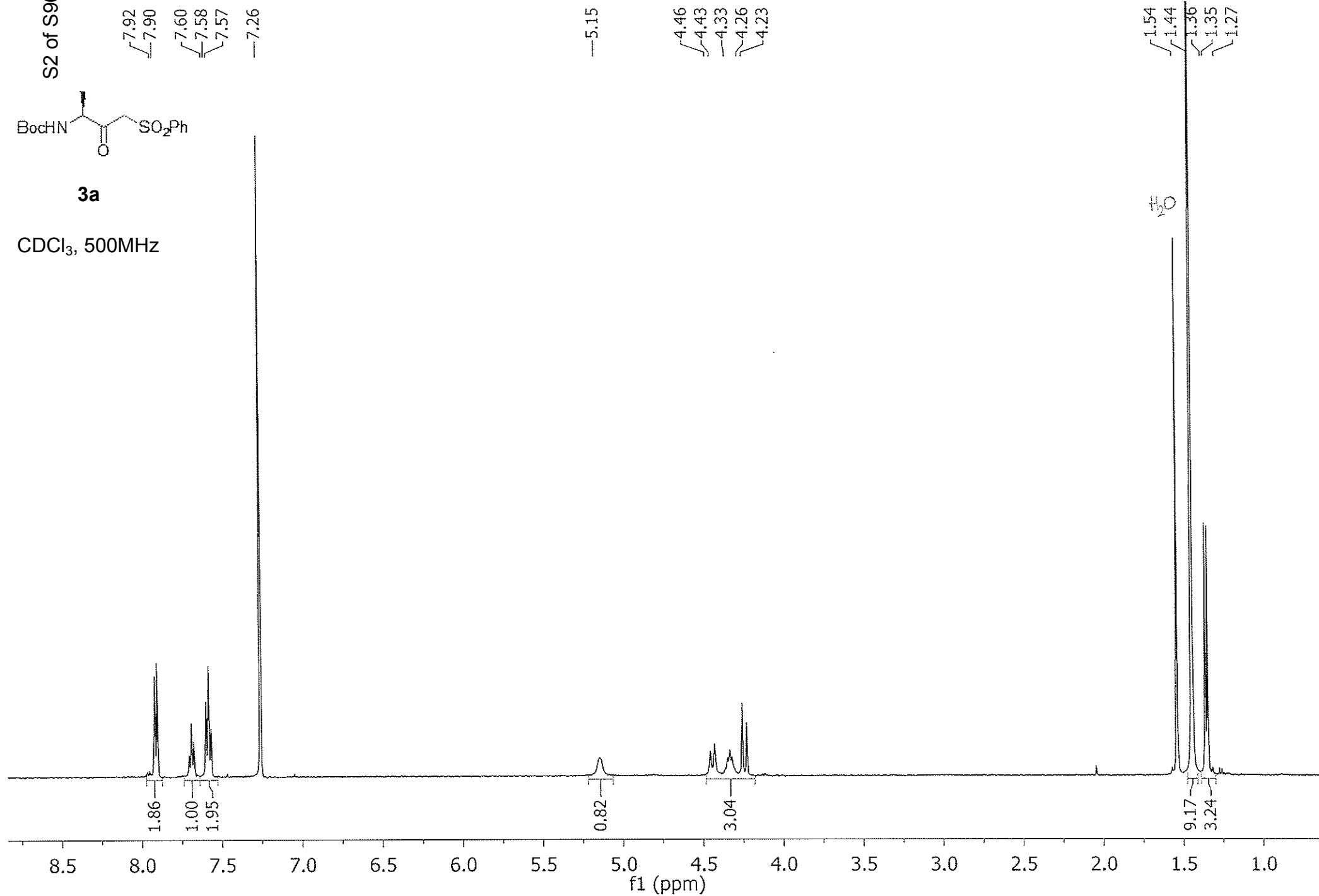
Table of Contents

7.92
7.90
7.60
7.58
7.57
7.26
5.15
4.46
4.43
4.33
4.26
4.23
1.54
1.44
1.36
1.35
1.27

BocHN—⟨⟩—SO$_2$Ph

O

3a

CDCl$_3$, 500MHz

H$_2$O

1.86
1.00
1.95
0.82
3.04
9.17
3.24

8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
f1 (ppm)

198.38

155.32

138.99
134.31
129.30
128.43

80.21

63.25
55.89

28.08

16.10

BocHN—CH(CH₃)—C(=O)—CH₂—SO₂Ph

3a

CDCl₃, 125.68 MHz

190   170   150   130   110   90   80   70   60   50   40   30   20   10   0
f1 (ppm)

7.93
7.92
7.68
7.59
7.56
7.26

5.11
5.10

4.44
4.41
4.28
4.27
4.26
4.25
4.22
4.19

2.31
2.29
2.28
2.27

1.44

0.99
0.98
0.81
0.80

BocHN
SO$_2$Ph
O

3b

CDCl$_3$, 500MHz

2.23
1.13
2.25
1.00
1.08
1.14
1.17
1.17
9.93
3.42
3.40

8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

197.81

155.96

139.13
134.39
129.40
128.65

80.50
77.41
77.16
76.91

65.21
64.00

28.94
28.41

19.93
16.97

BocHN

SO$_2$Ph

O

3b

CDCl$_3$, 125.68 MHz

f1 (ppm)

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

CDCl$_3$, 500MHz

7.93, 7.93, 7.91, 7.91, 7.70, 7.68, 7.67, 7.59, 7.58, 7.56

5.07, 5.05

4.50, 4.47, 4.28, 4.23, 4.20

1.67

1.44

0.95, 0.94, 0.93, 0.93

2.28, 1.17, 2.41, 0.92, 1.00, 2.32, 2.67, 12.54, 7.45

3c

198.65
155.53
138.90
134.22
129.23
128.43
80.48
63.30
58.90
39.08
28.25
24.77
23.15
21.37

3c

CDCl$_3$, 125.68 MHz

7.96, 7.95, 7.94, 7.93, 7.69, 7.68, 7.67, 7.66, 7.66, 7.65, 7.60, 7.59, 7.59, 7.58, 7.57, 7.56

5.09, 5.07

4.45, 4.42, 4.29, 4.28, 4.27, 4.26, 4.23, 4.20

1.67, 1.44, 1.27, 1.05, 0.97, 0.95, 0.89, 0.88, 0.86

−0.39

3d

CDCl$_3$, 500MHz

4.45, 4.42, 4.29, 4.28, 4.27, 4.26, 4.23, 4.20

f1 (ppm)

f1 (ppm)

197.59

155.60

139.11
134.07
129.34
129.17
128.49

80.25

64.84
63.76

35.36

28.11
24.37

16.03
11.24

3d

CDCl$_3$, 125.68 MHz

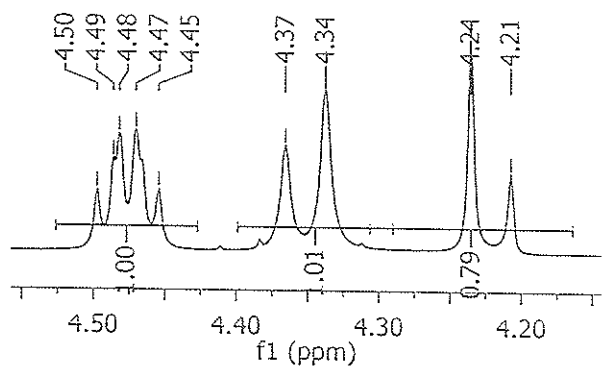200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

7.87/
7.86
7.85
7.69
7.68
7.66
7.58
7.56
7.55
7.31
7.30
7.29
7.26
7.25
7.23
7.16
7.14
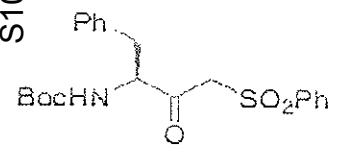
5.13
5.11

4.50
4.49
4.48
4.47
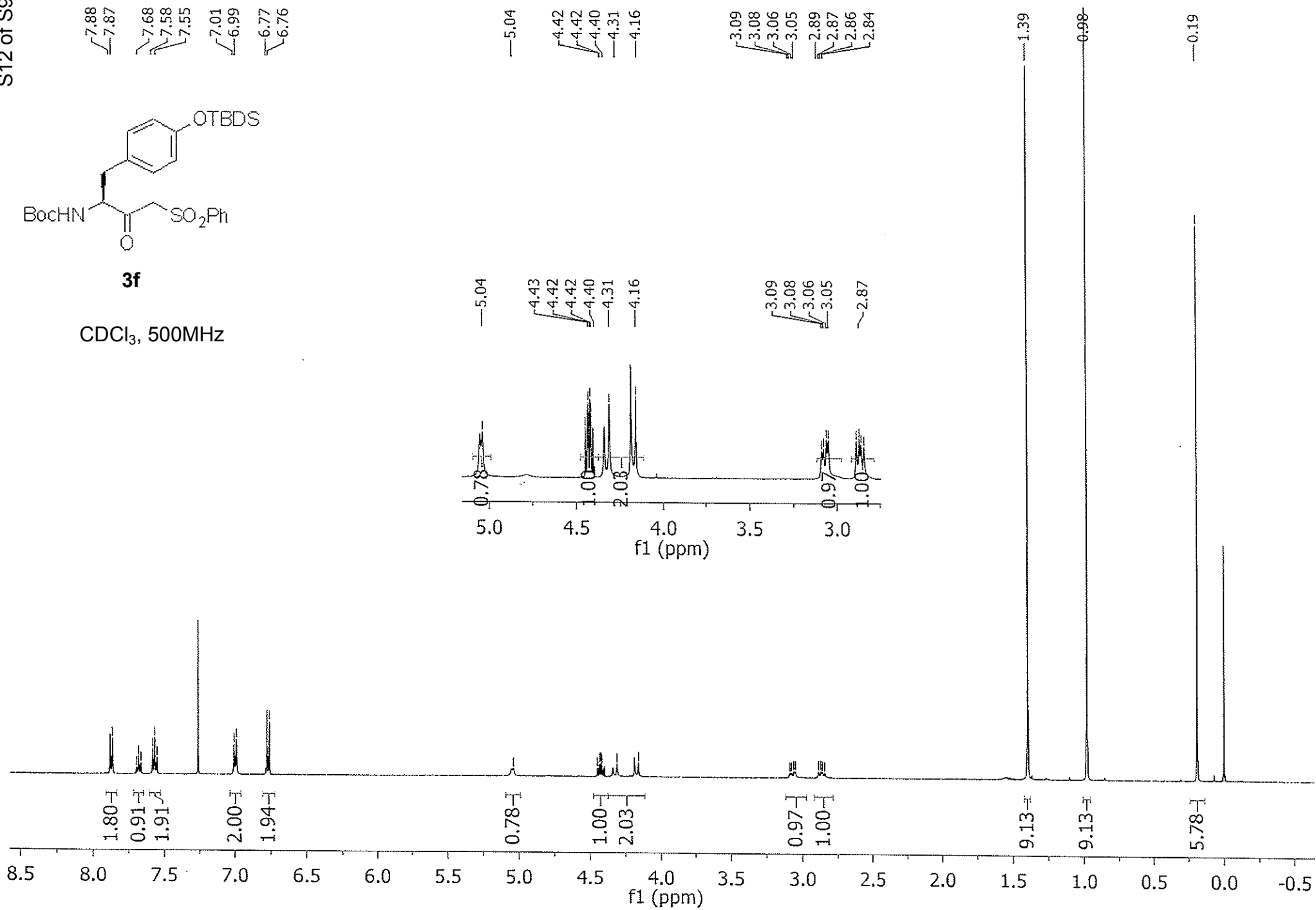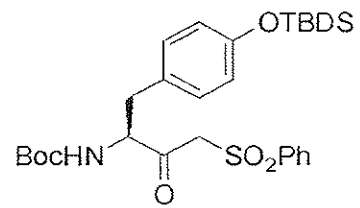4.45
4.37
4.34
4.24
4.21

3.18
3.17
3.16
3.15
2.94
2.93
2.91
2.90

1.41
1.38

3e

CDCl$_3$, 500MHz

4.50
4.49
4.48
4.47
4.45
4.37
4.34
4.24
4.21

197.38

155.37

138.82
136.14
134.26

129.26
128.78
128.43
127.08

80.64

63.74
61.37

36.34

28.21

Ph

BocHN

SO$_2$Ph

O

3e

CDCl$_3$, 125.68 MHz

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

7.88, 7.87
7.68, 7.58, 7.55
7.01, 6.99
6.77, 6.76
5.04
4.42, 4.42, 4.40, 4.31, 4.16
3.09, 3.08, 3.06, 3.05
2.89, 2.87, 2.86, 2.84
1.39
0.98
0.19

OTBDS

BocHN — SO$_2$Ph

O

3f

CDCl$_3$, 500MHz

5.04
4.43, 4.42, 4.42, 4.40, 4.31, 4.16
3.09, 3.08, 3.06, 3.05
2.87

0.78
1.00
2.03
0.97
1.00

5.0   4.5   4.0   3.5   3.0
f1 (ppm)

1.80
0.91
1.91
2.00
1.94
0.78
1.00
2.03
0.97
1.00
9.13
9.13
5.78

8.5   8.0   7.5   7.0   6.5   6.0   5.5   5.0   4.5   4.0   3.5   3.0   2.5   2.0   1.5   1.0   0.5   0.0   -0.5
f1 (ppm)

197.60

155.37
154.79

138.85
134.23
130.20
129.24
128.54
128.45
120.39

80.59
77.25
77.00
76.75

63.85
61.46

35.71

28.23
25.65

18.18

-0.01
-4.43

OTBDS

BocHN

SO₂Ph

O

3f

CDCl₃, 125.68 MHz

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

7.96, 7.94, 7.92, 7.69, 7.68, 7.66, 7.65, 7.60, 7.59, 7.58, 7.56

4.42, 4.41, 4.41, 4.39, 4.37, 4.34, 4.32, 4.31, 4.28, 4.21, 4.18, 3.57, 3.55, 3.48, 3.46, 3.46, 3.44, 3.43, 2.14, 2.10, 2.08, 2.04, 2.01, 1.94, 1.91, 1.88, 1.86, 1.59, 1.43, 1.36

3g

CDCl$_3$, 500MHz

H2O

1.58 1.05 2.02 3.00 2.45 4.30 8.59

8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

198.60
198.07
154.99
153.67
139.32
134.20
129.40
129.28
128.60
81.12
80.61
66.27
65.73
64.08
62.79
47.17
46.97
29.43
28.49
28.40
24.78
23.86

3g

CDCl$_3$, 125.68 MHz

f1 (ppm)

7.68, 7.67, 7.57, 7.55, 7.54

7.34, 7.33, 7.31, 7.31, 7.29, 7.21, 7.20, 7.19

5.41, 5.40

4.98, 4.97

4.69

4.51, 4.50, 4.46

3.32, 3.31, 3.24, 3.23, 3.21, 3.20

3.01, 2.88, 2.86, 2.86, 2.84

1.40, 1.29, 1.28

3h

CDCl$_3$, 500MHz

BocHN — C(=O) — SO$_2$Ph

6.64

6.09

1.00

1.14

1.04

1.14

1.24

4.98

8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5

f1 (ppm)

202.62
200.79
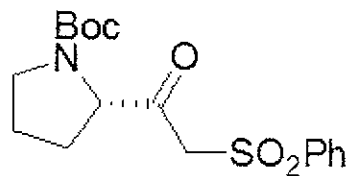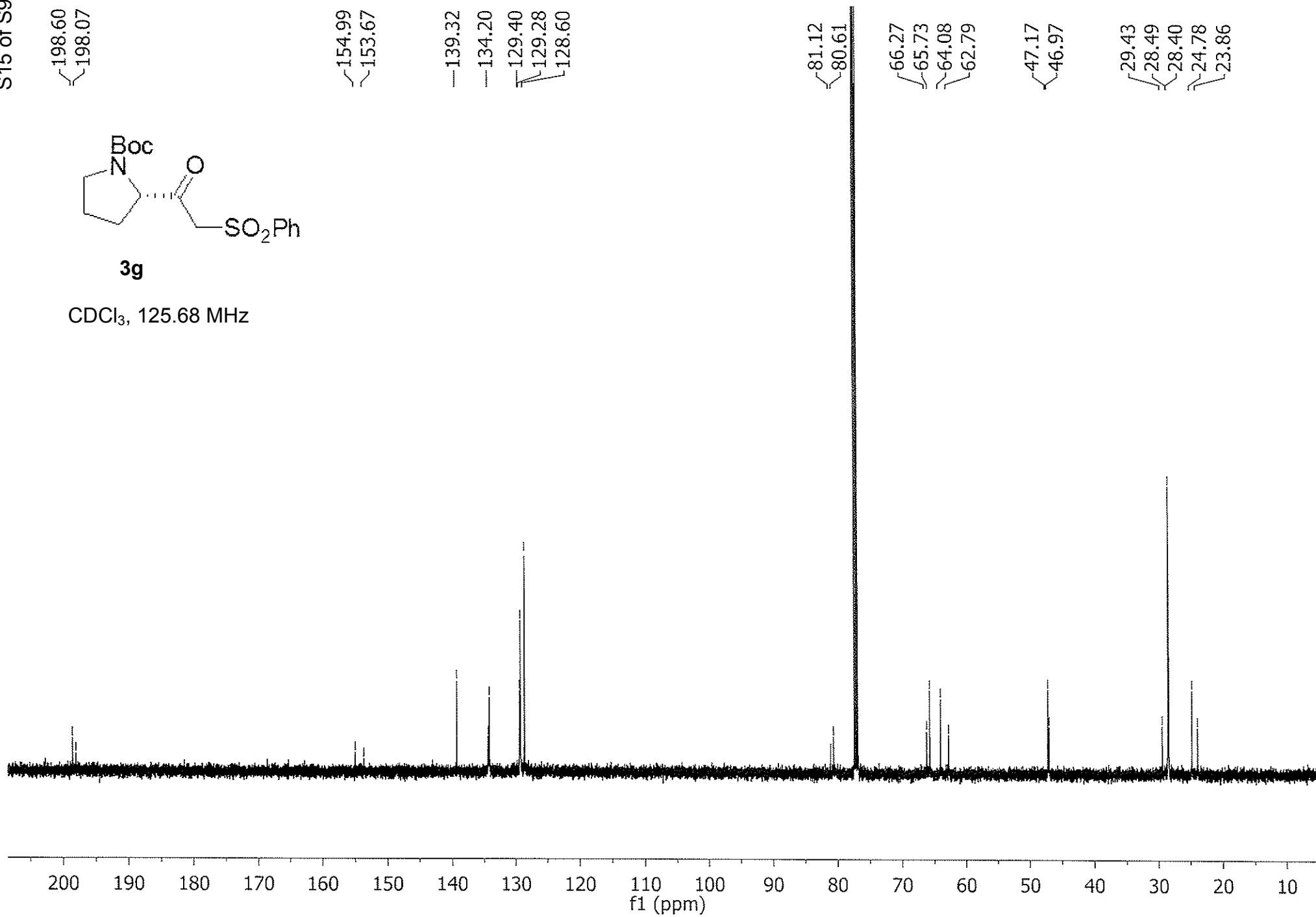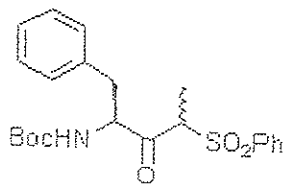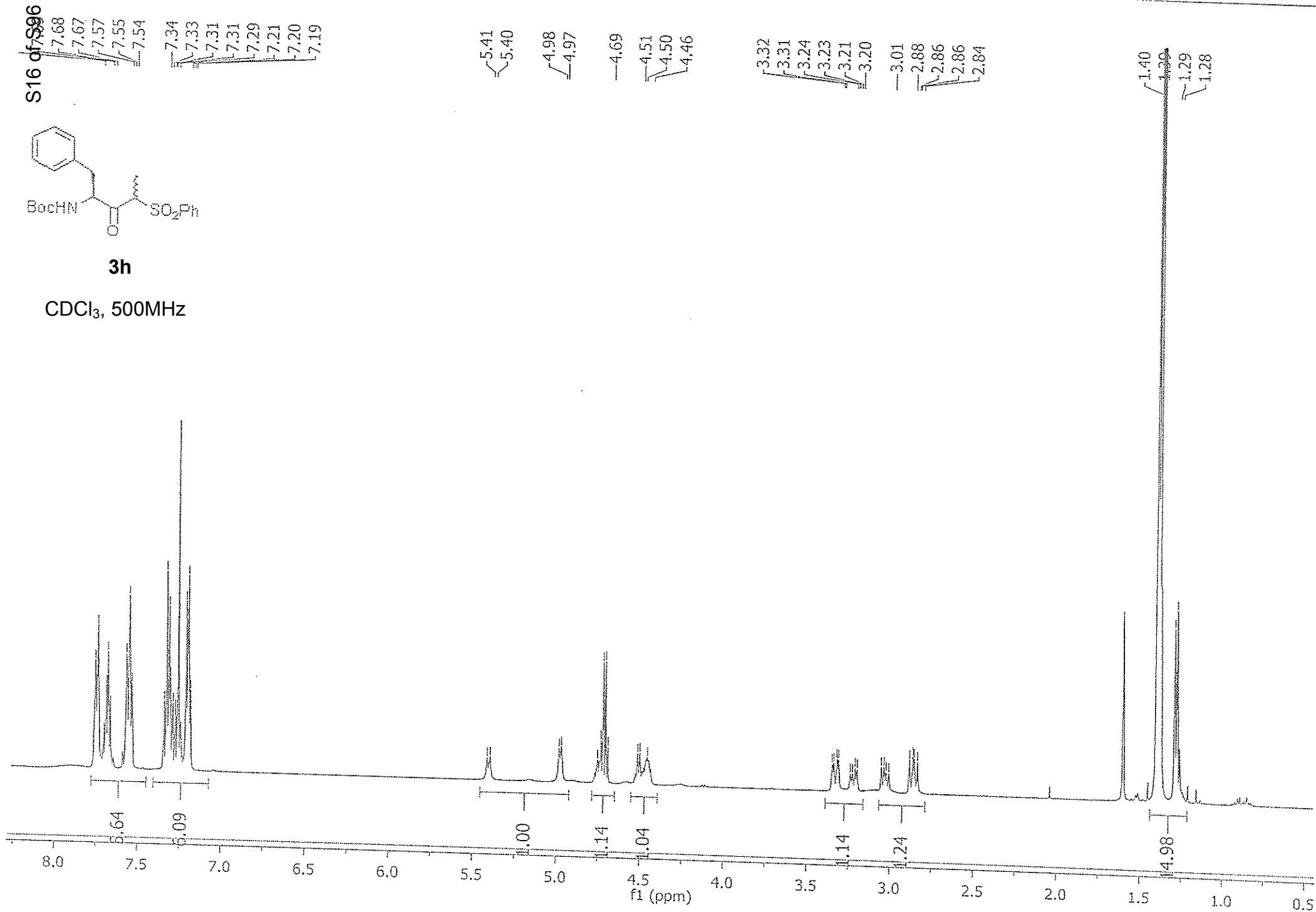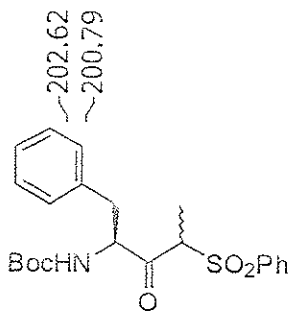
137.28
136.33
136.03
135.97
134.51
134.32
129.65
129.57
129.47
129.30
129.13
129.07
128.70
128.25
127.11
126.83

80.67
80.24

66.33
65.89
61.61

37.72
35.01

28.11
28.01

12.47
12.07

3h

CDCl₃, 125.68 MHz

BocHN

SO₂Ph

3h
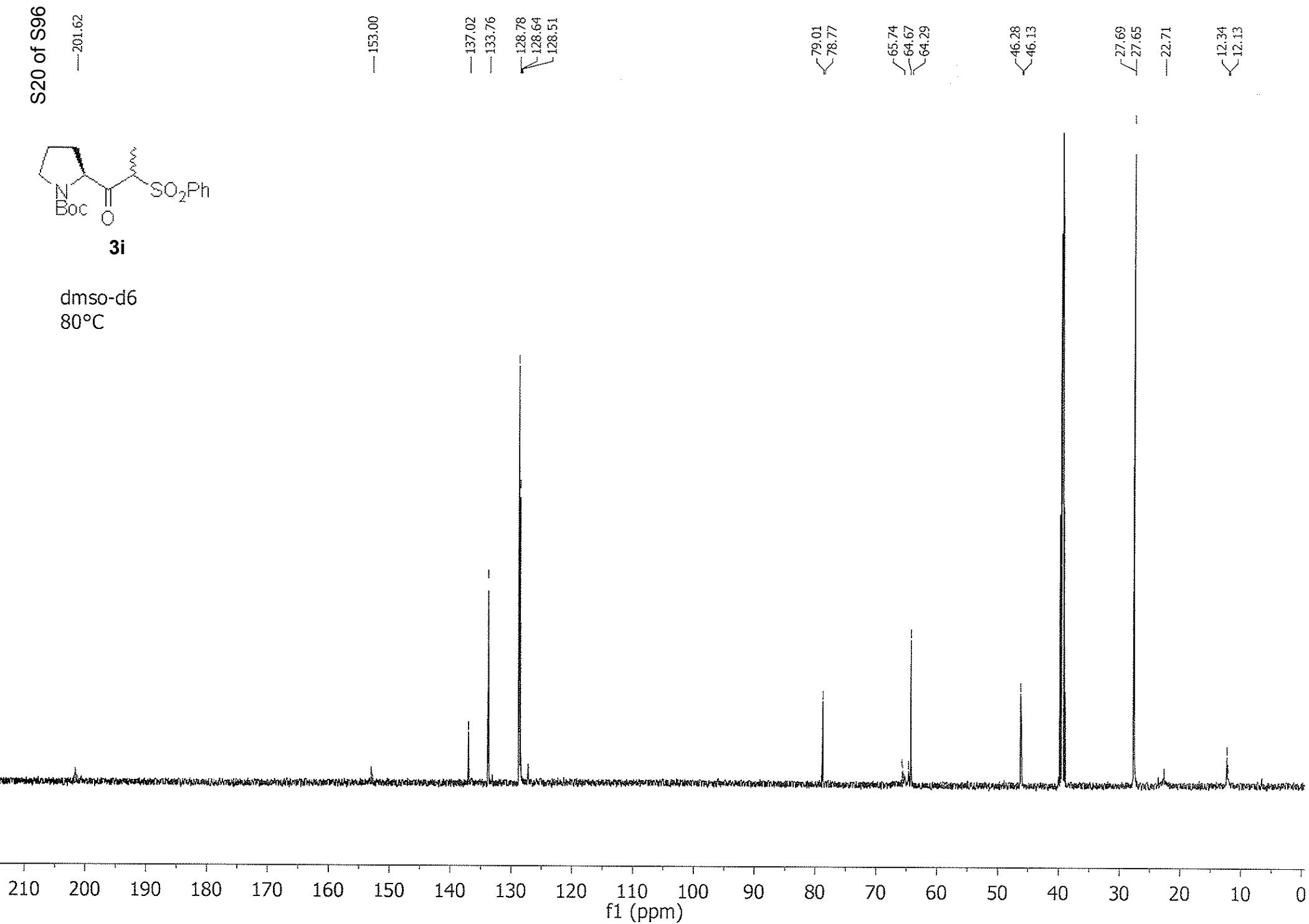
CDCl₃

7.86
7.78
7.66

4.85
4.72
4.67
4.54

3.43
3.41
3.39
3.38
3.33
3.32
3.30

2.20
2.12
2.02
1.88
1.80
1.66

1.35
1.34
1.31
1.30

dmso-d6
80°C

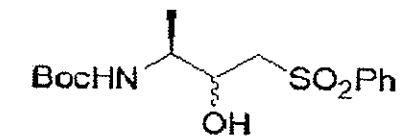**3i**

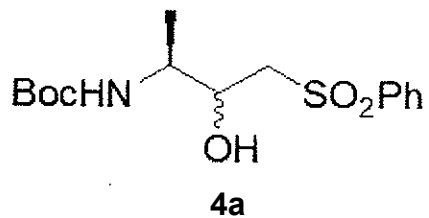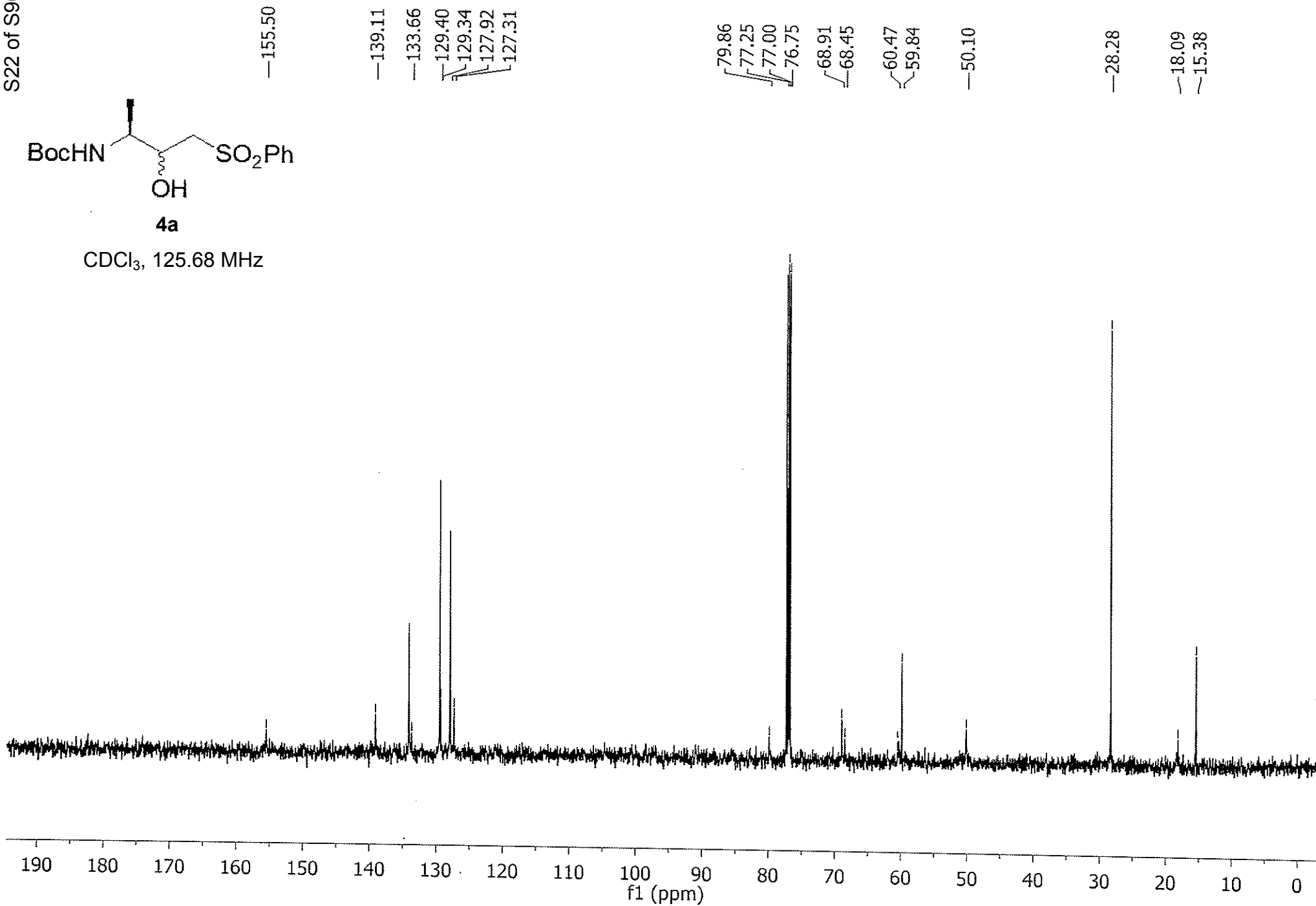mixture of diastereoisomers and rotamers

f1 (ppm)

f1 (ppm)

201.62
153.00
137.02
133.76
128.78
128.64
128.51
79.01
78.77
65.74
64.67
64.29
46.28
46.13
27.69
27.65
22.71
12.34
12.13

3i

dmso-d6
80°C

7.93
7.91
7.69
7.61
7.57
7.19
7.08

4.73

4.09

3.66

3.27
3.20

1.58
1.56
1.39
1.21
1.20
1.14
1.12

BocHN—SO₂Ph
OH

4a

CDCl₃, 500MHz

1.21

1.20

1.14

1.12

1.00

3.23

1.22 1.20 1.18 1.16 1.14 1.12 1.10
f1 (ppm)

2.00

0.93
1.89

0.69

1.03

1.67

1.68

2.39
8.20

0.61
1.97

8.0   7.5   7.0   6.5   6.0   5.5   5.0   4.5   4.0   3.5   3.0   2.5   2.0   1.5   1.0   0.5   0.0
f1 (ppm)

155.50

139.11
133.66
129.40
129.34
127.92
127.31

79.86
77.25
77.00
76.75
68.91
68.45
60.47
59.84
50.10

28.28

18.09
15.38

BocHN ⟍⟍N⟍⟍SO₂Ph
         OH
         4a

CDCl₃, 125.68 MHz

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

7.93, 7.91, 7.70, 7.69, 7.67, 7.61, 7.59, 7.58

4.43, 4.41, 4.14, 4.13, 4.11

3.57, 3.56, 3.50, 3.48, 3.46, 3.35, 3.32, 3.26, 3.23, 3.21

2.20, 2.18, 2.17

1.40

0.91, 0.90, 0.87, 0.86

BocHN ... SO₂Ph ... OH

4b

CDCl₃, 500MHz

3.57, 3.56, 3.50, 3.48, 3.46

3.35, 3.32, 3.26, 3.24, 3.23, 3.21

4.43, 4.41 / 4.14, 4.13, 4.11

0.96 ... 0.99

3.7 3.6 3.5 3.4 3.3 3.2
f1 (ppm)

4.4 4.3 4.2 4.1
f1 (ppm)
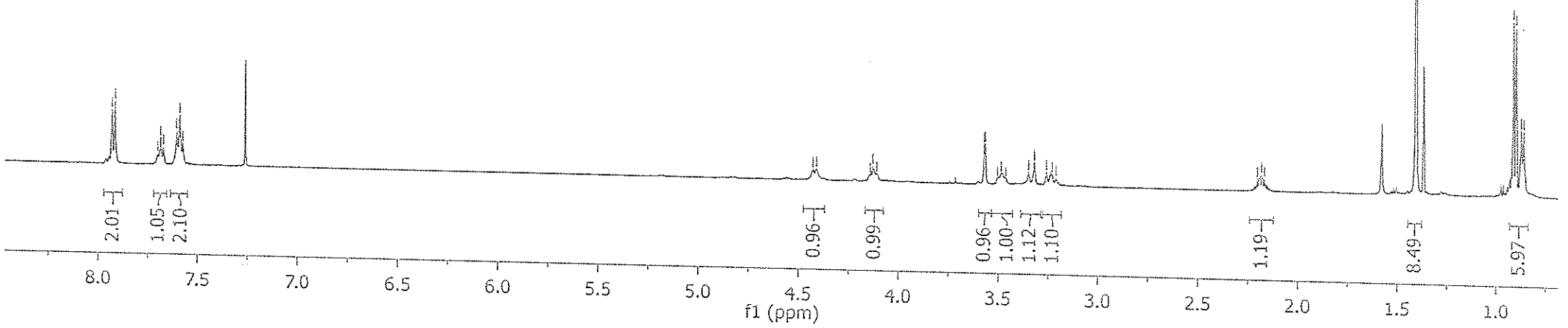
0.96, 1.00, 1.12, 1.10

2.01 / 1.05, 2.10 / 0.96 / 0.99 / 0.96, 1.00, 1.12, 1.10 / 1.19 / 8.49 / 5.97

8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
f1 (ppm)

156.54

139.46
134.20
129.55
127.94
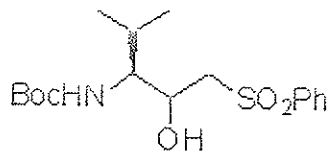
79.93

67.22

60.21
58.33

28.15
27.48

19.80
15.56

BocHN SO$_2$Ph
OH

4b

CDCl$_3$, 125.68 MHz

160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
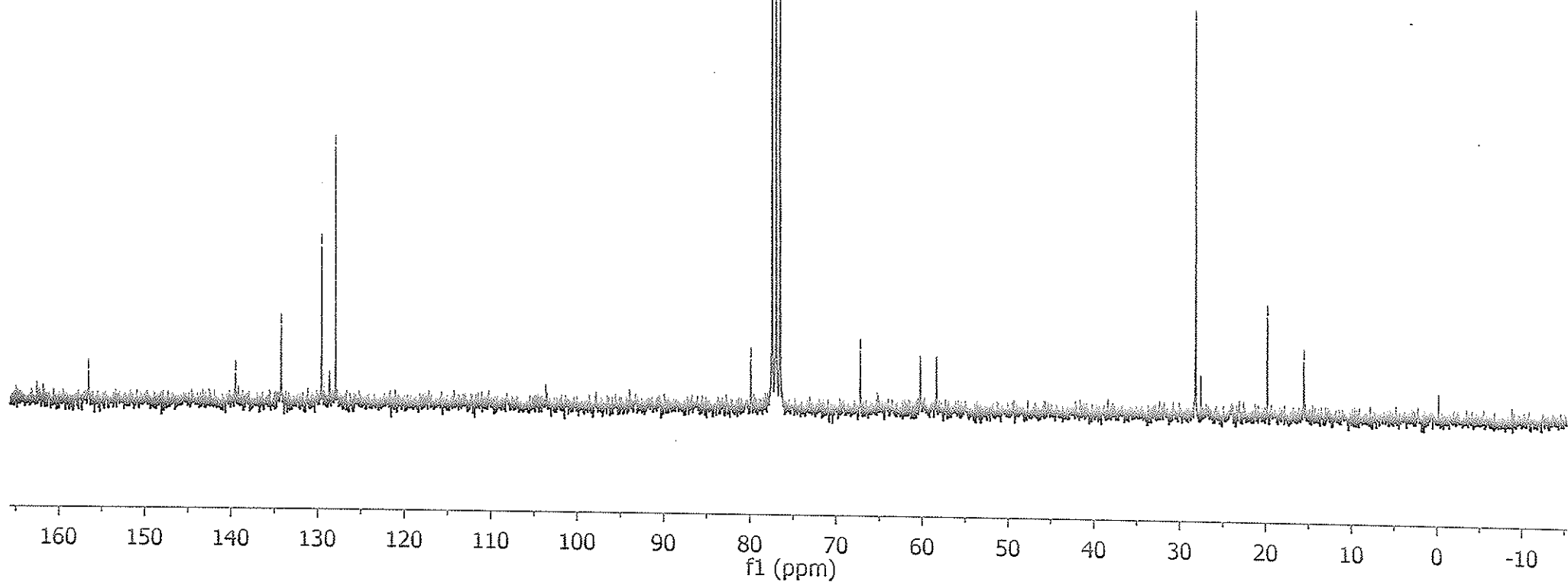
7.94, 7.93, 7.93, 7.92, 7.92, 7.91, 7.69, 7.69, 7.69, 7.68, 7.67, 7.67, 7.66, 7.66, 7.66, 7.61, 7.60, 7.59, 7.58, 7.57, 7.57

4.56, 4.54

4.09

3.72, 3.64, 3.60, 3.28, 3.27, 3.27, 3.24, 3.24

1.63, 1.62, 1.61, 1.44, 1.43, 1.40, 1.39, 1.34, 1.33, 1.32, 1.31, 1.30, 1.28, 1.27

0.91, 0.90, 0.88, 0.87, 0.87, 0.86
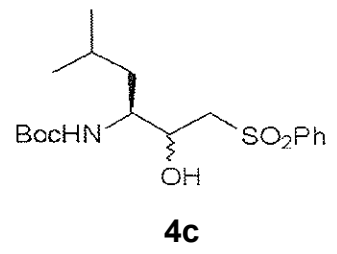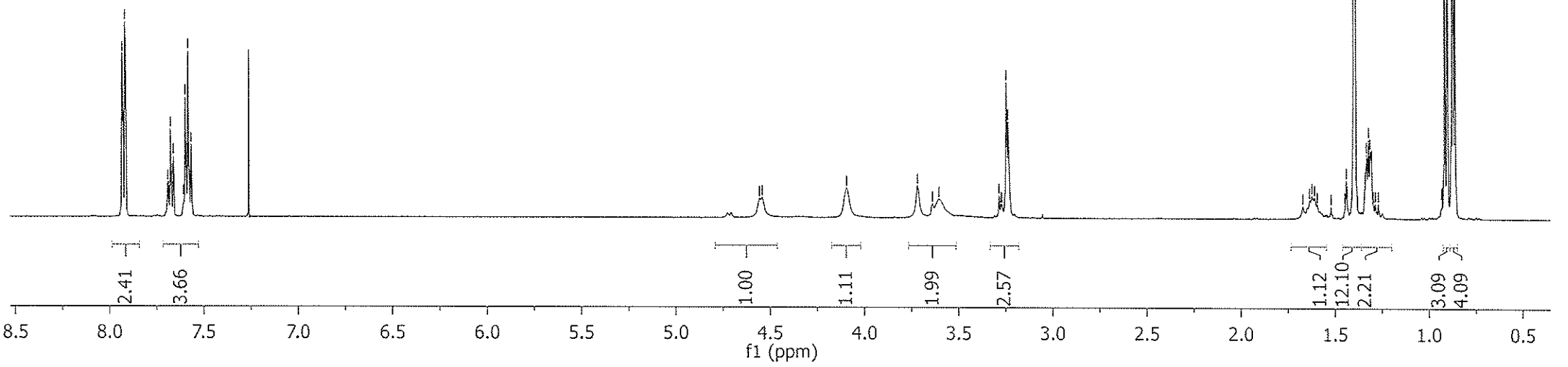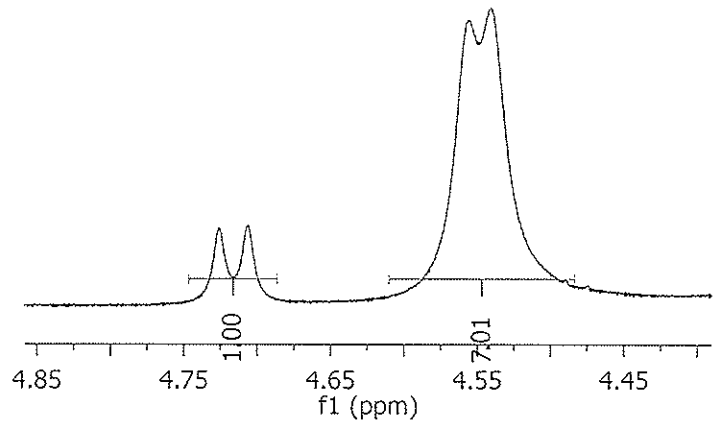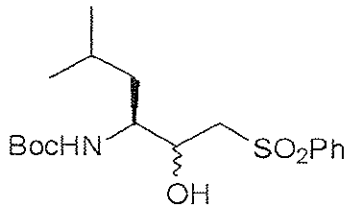
4c

CDCl$_3$, 500MHz

BocHN / OH / SO$_2$Ph

f1 (ppm)

4c

CDCl$_3$, 125.68 MHz

7.92
7.67
7.59
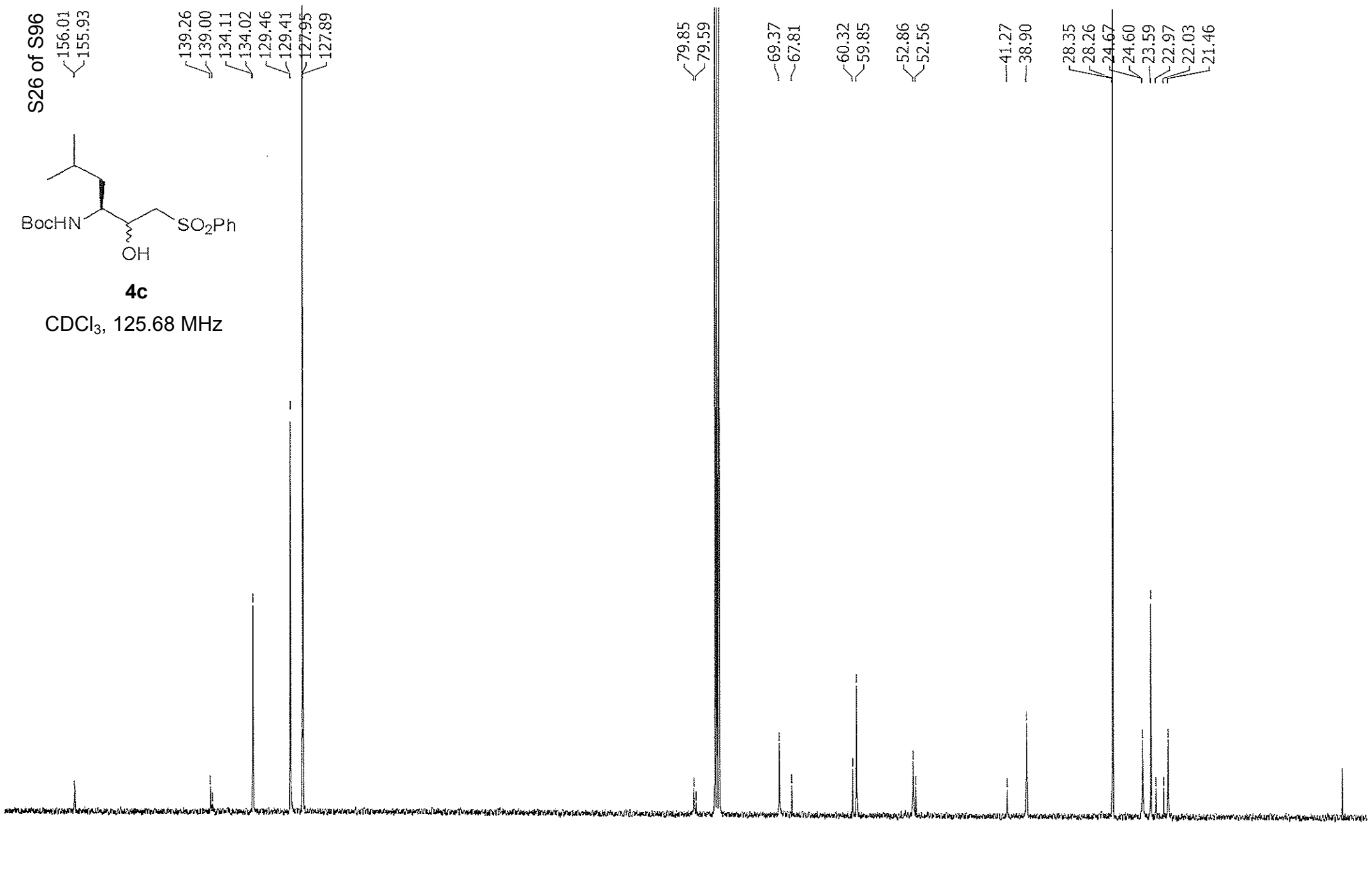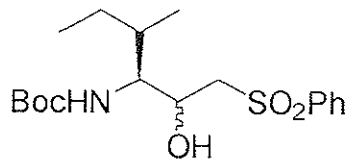
4.41
4.19

3.59
3.49
3.30
3.23

1.88
1.61
1.54
1.40

0.93
0.92
0.90
0.89

BocHN

SO_2Ph

OH

4d

$CDCl_3$, 500MHz

4.41
4.19

4.5 4.4 4.3 4.2 4.1
f1 (ppm)

1.00
0.96

3.59
3.49
3.30
3.23

3.5 3.2
f1 (ppm)

2.04
2.16

2.67
3.96

1.00
0.96

2.04
2.16

1.12
2.89
11.36

8.66

8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
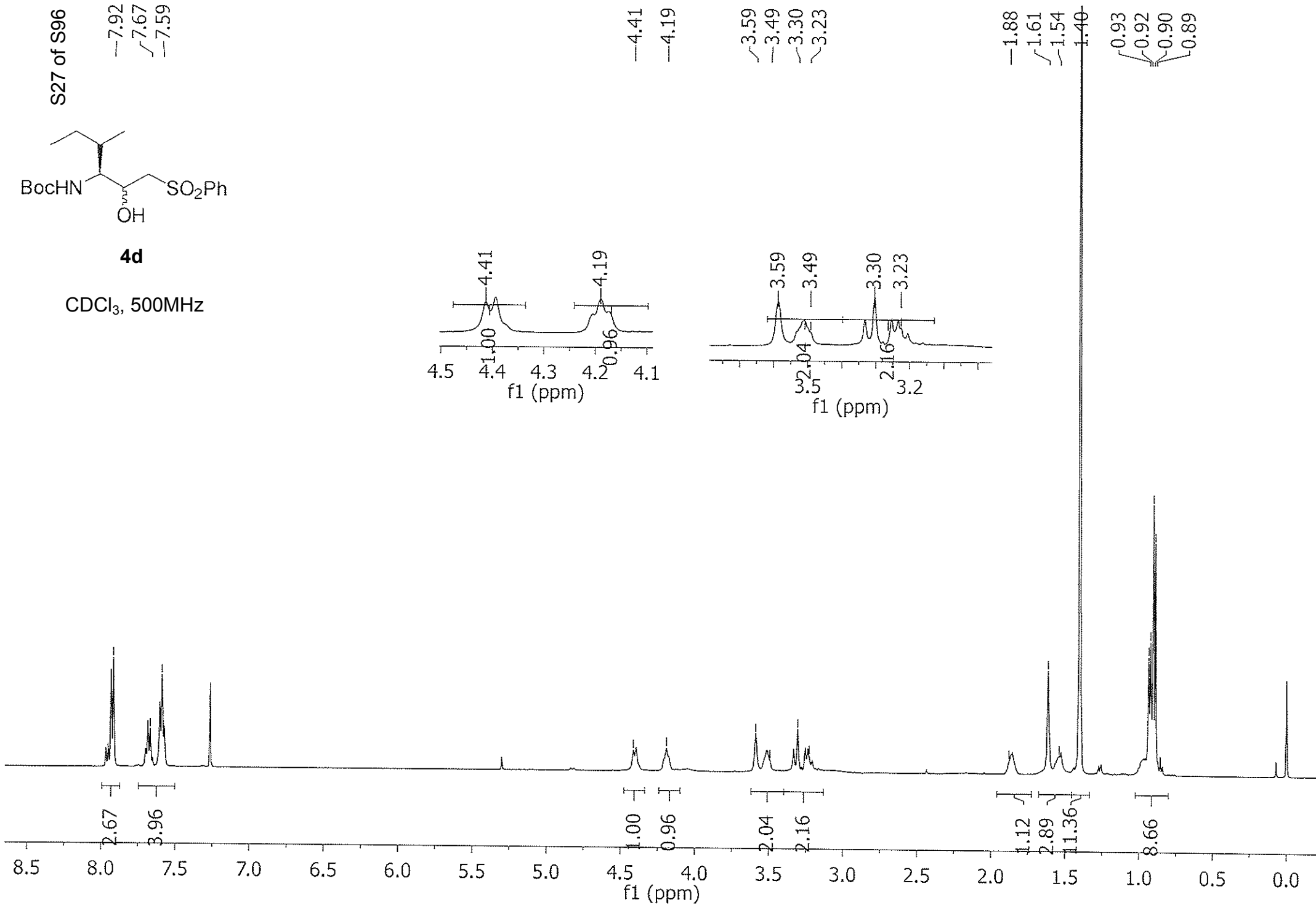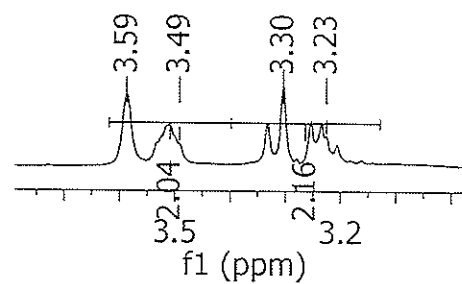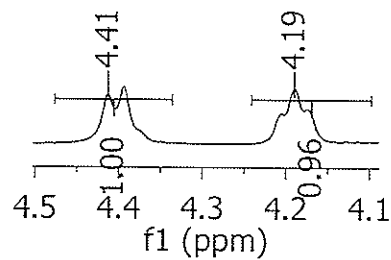
156.47

140.63
139.46
134.04
133.69
129.42
129.37
127.88
127.36

79.97
77.27
77.02
76.76

67.10
65.29
60.64
60.31
58.83
58.67

36.24
34.38
30.95
28.41
25.67
23.07

16.28
15.41
11.83
10.95

BocHN— ... —SO$_2$Ph

OH

4d

CDCl$_3$, 125.68 MHz

160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

7.66
7.64
7.62
7.62
7.59
7.57
7.57
7.56
7.54
7.52
7.52
7.50
7.26
7.22
7.21
7.18
7.16
7.16

4.94
4.92
4.55
4.55
3.93
3.81
3.70
3.31
3.24
3.00
2.95
2.87
2.77
1.35
1.35
1.30
1.30

4e

CDCl$_3$, 500MHz

6.03
10.65
16.09
0.35
1.84
2.46
0.99
1.79
0.35
5.04
4.00
2.53
14.78

f1 (ppm)
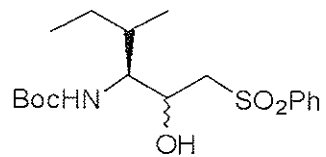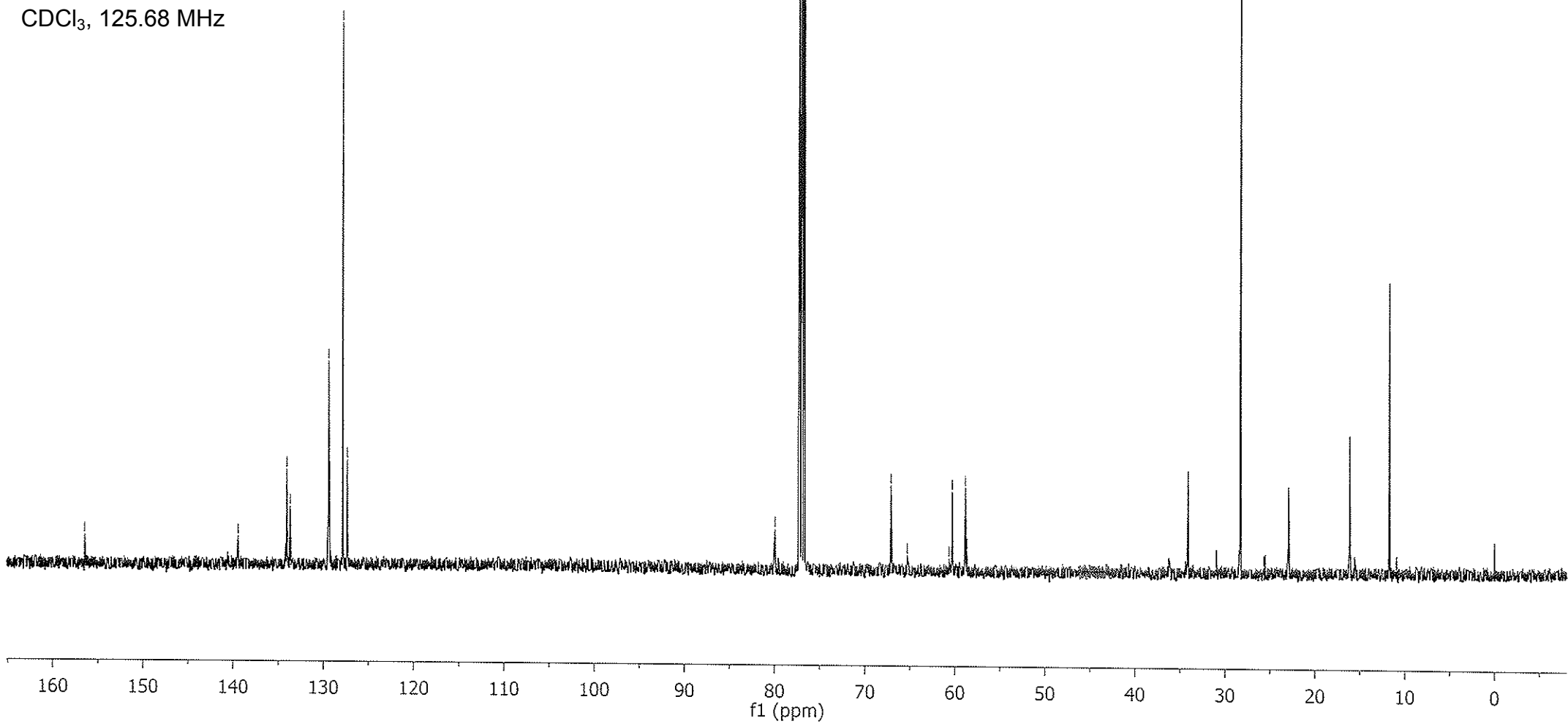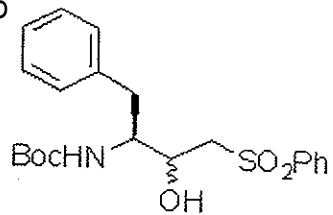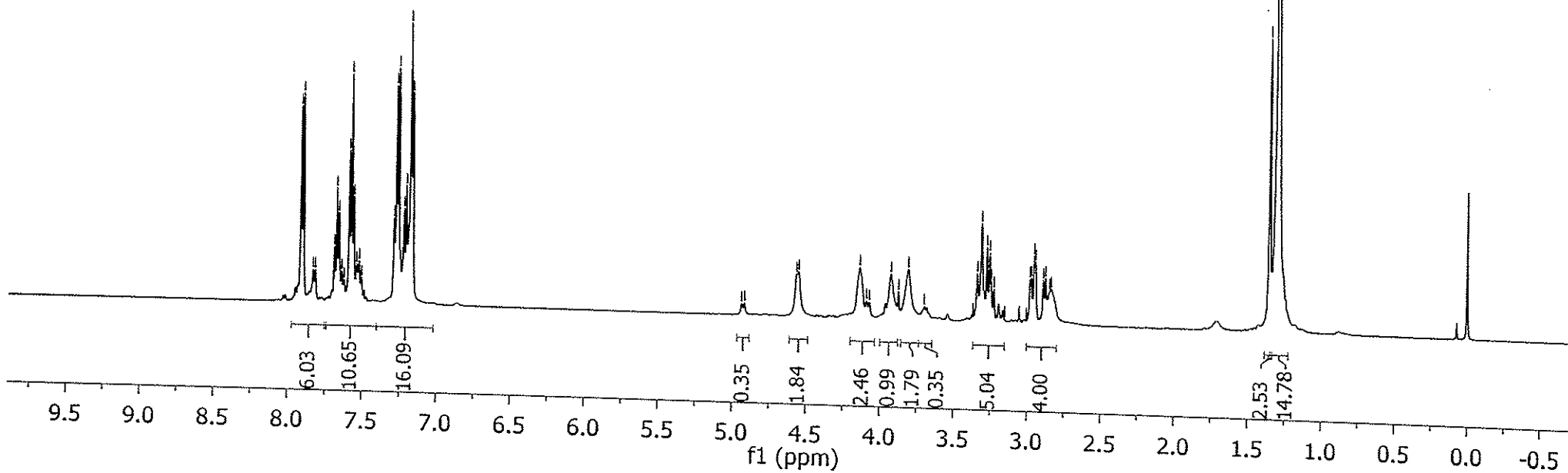
155.66

139.10
136.94
134.07

129.44
129.39
128.52
128.46
127.89
127.79
126.61
126.50

79.97
79.79

68.12
65.70

60.35
59.84

38.26
35.78

28.22
28.15

Ph

BocHN

SO₂Ph

OH

4e

CDCl₃, 125.68 MHz

155   145   135   125   115   105   95   90   85   80   75   70   65   60   55   50   45   40   35   30   25
f1 (ppm)

Ph

BocHN — SO$_2$Ph

OH

4e
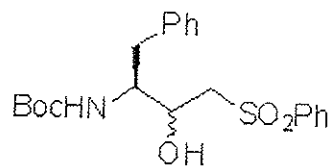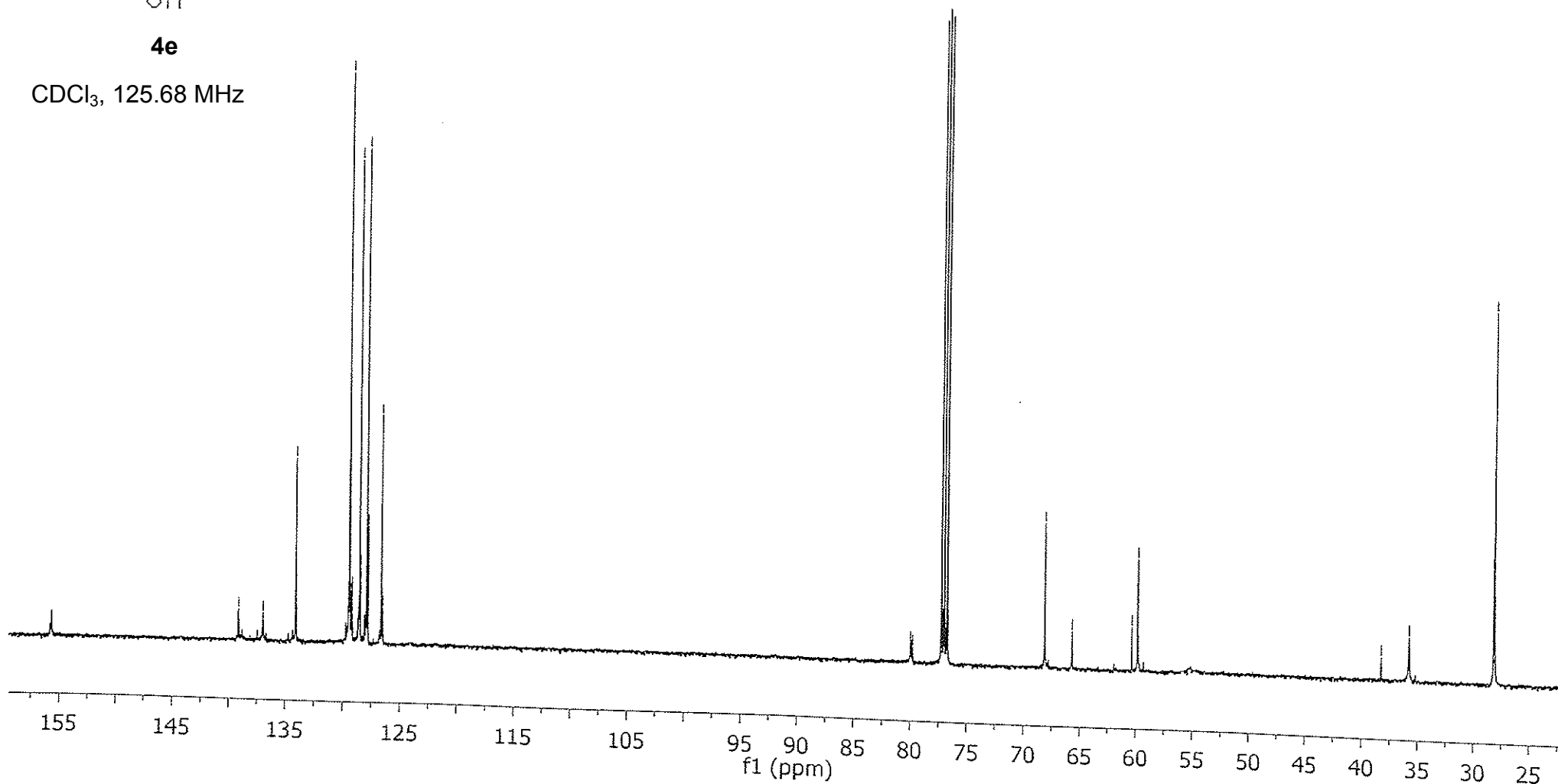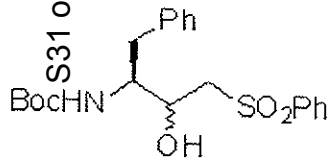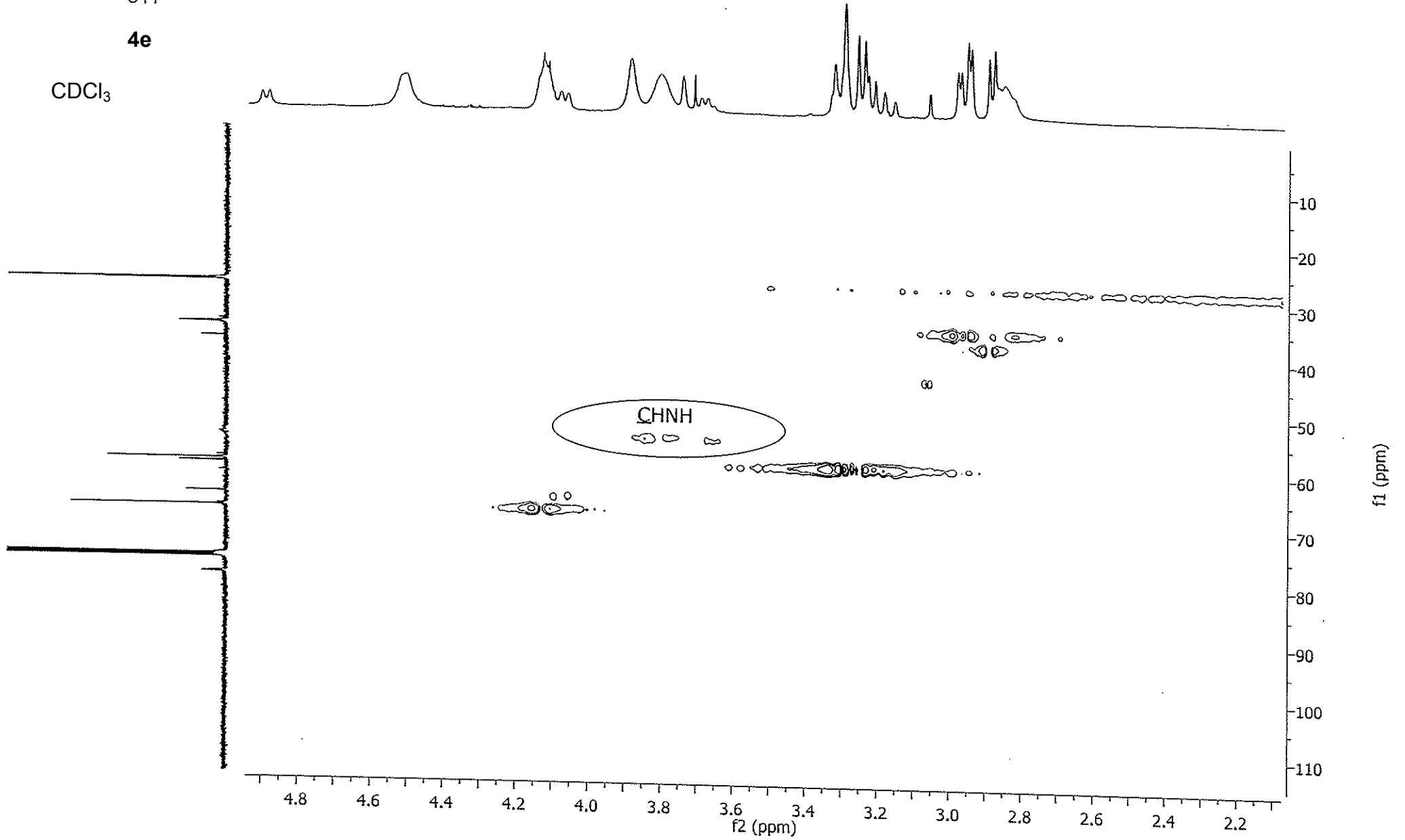
CDCl$_3$

7.66
7.63
7.58
7.55
7.52
7.03
7.00
6.76
6.74
6.72
6.70
4.89
4.52
4.11
4.05
3.89
3.73
3.62
3.28
2.87
2.79
1.36
1.32
0.98
0.97
0.17
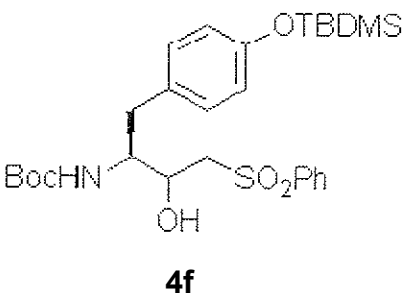
4f

CDCl$_3$, 500MHz

2.00 3.10 2.00 1.94 0.37 2.35 1.77 1.73 9.35 9.36 6.00

f1 (ppm)

155.71
154.40
139.14
134.07
127.82
120.12
120.03
79.75
68.13
65.66
59.88
55.95
55.34
37.45
34.98
28.26
28.20
25.66
18.16
4.43
4.45

OTBDS

BocHN

SO₂Ph

OH

4f

CDCl₃, 125.68 MHz

160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

4g
dmso-d6
80°C

4g

dmso-d6
80°C

ok
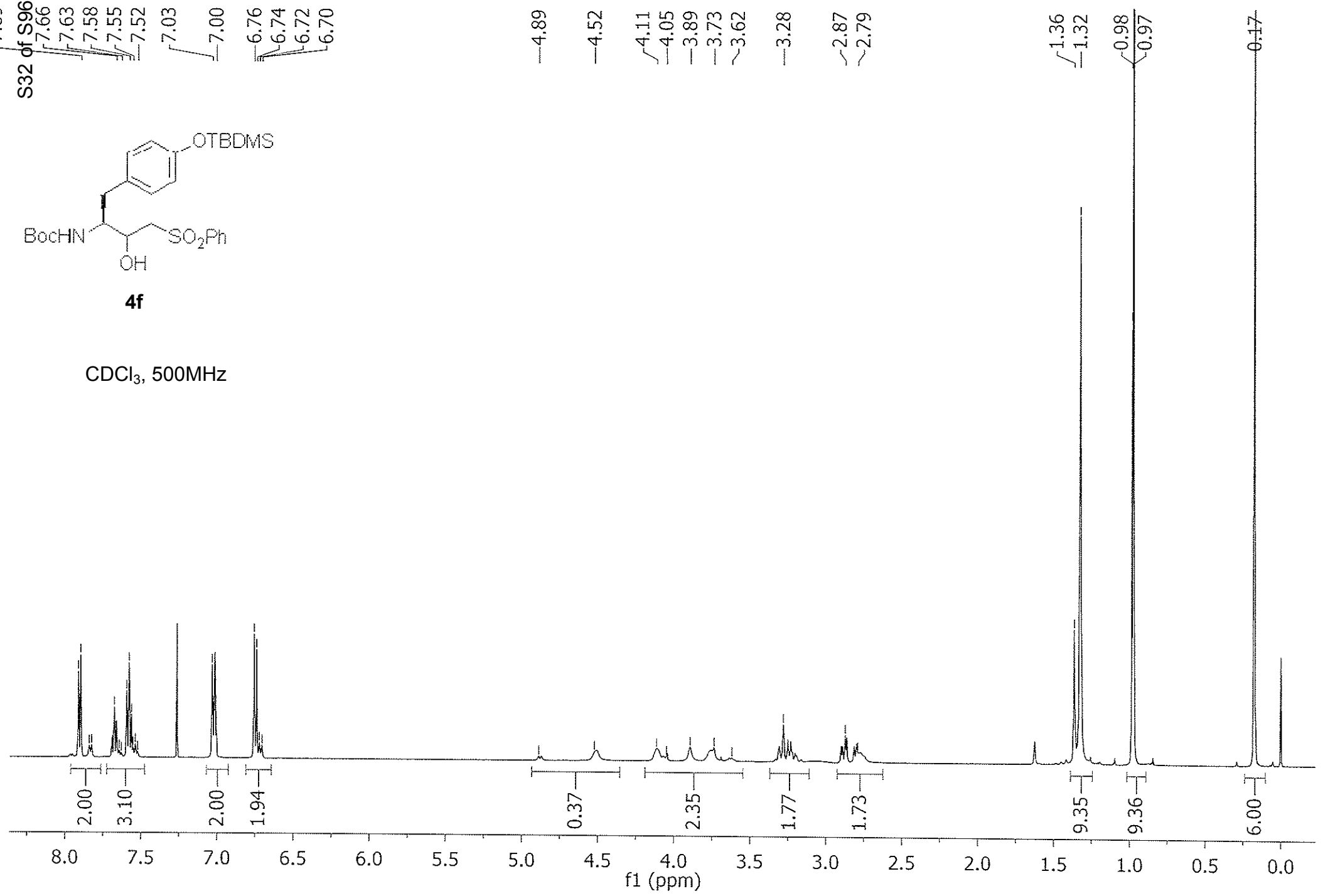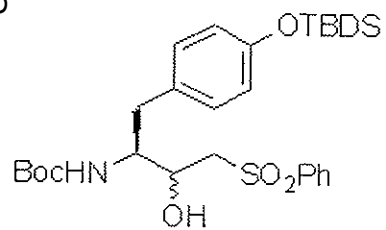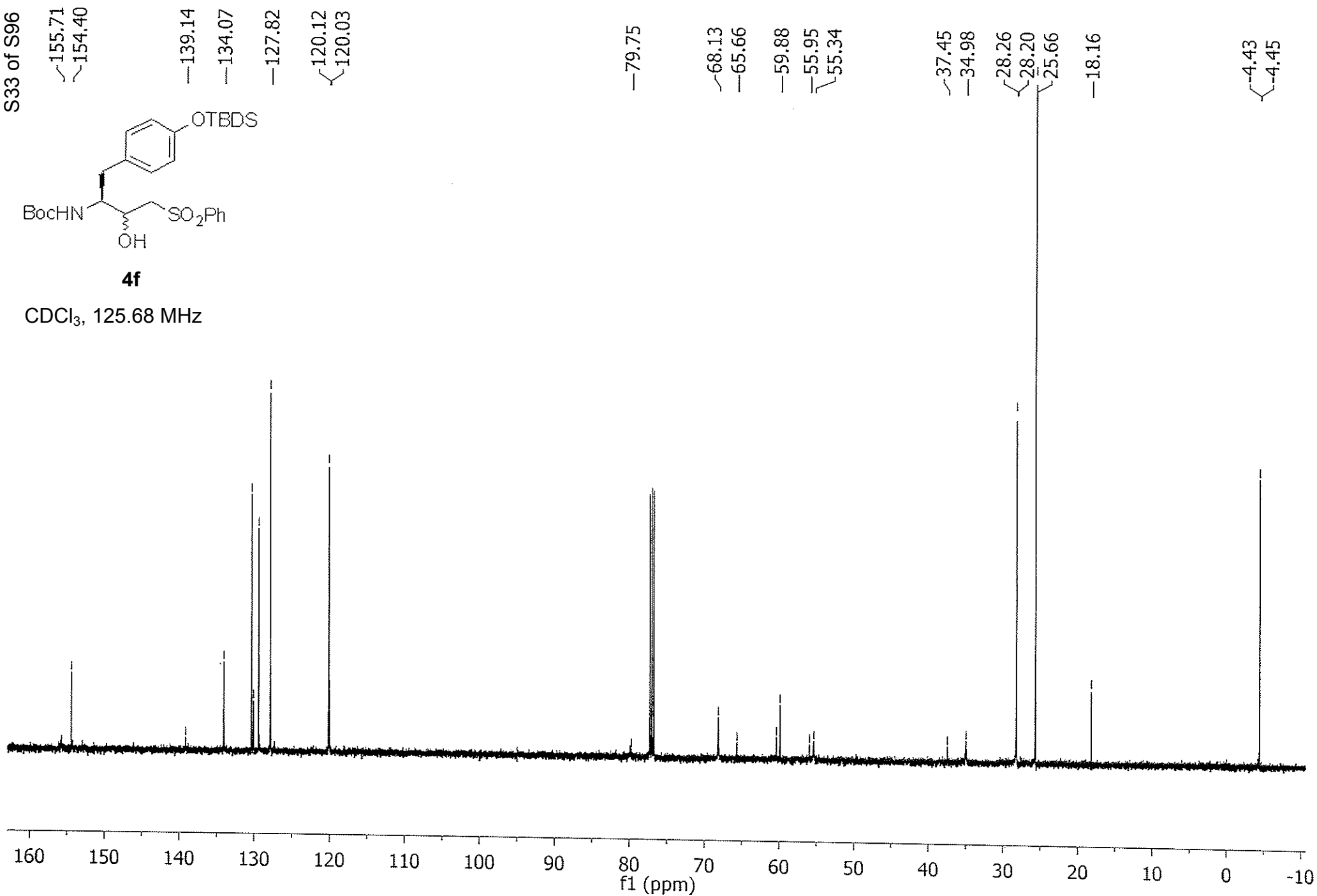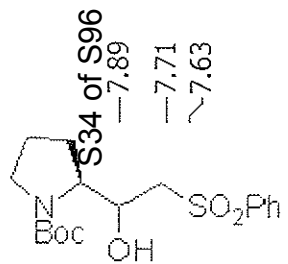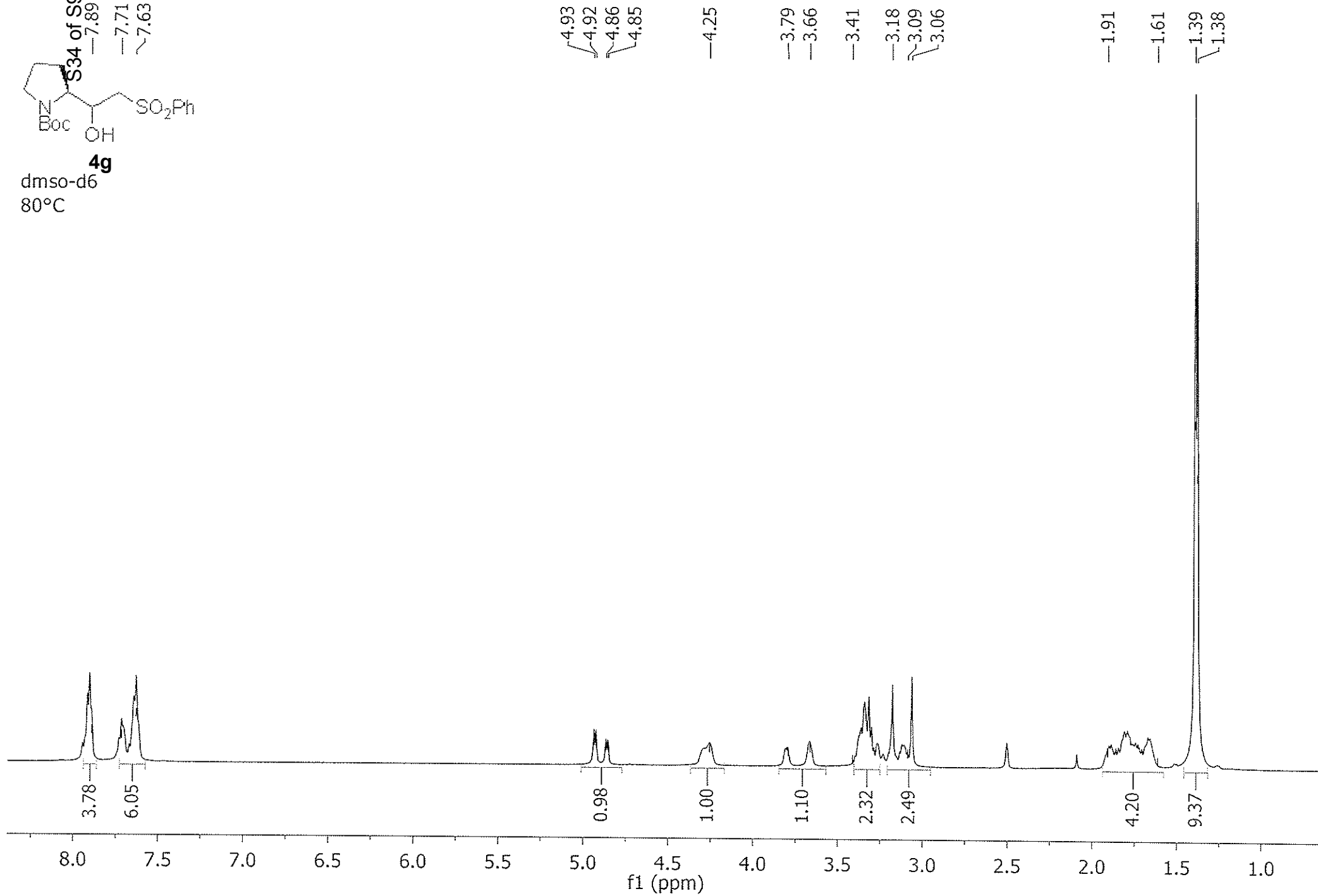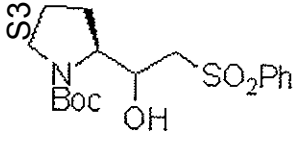
153.84
153.46

140.27
140.26
132.87
132.83
128.84
128.63
128.59
127.14
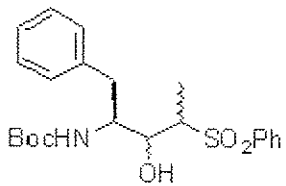127.08
126.27

78.33
78.10

66.79
65.58
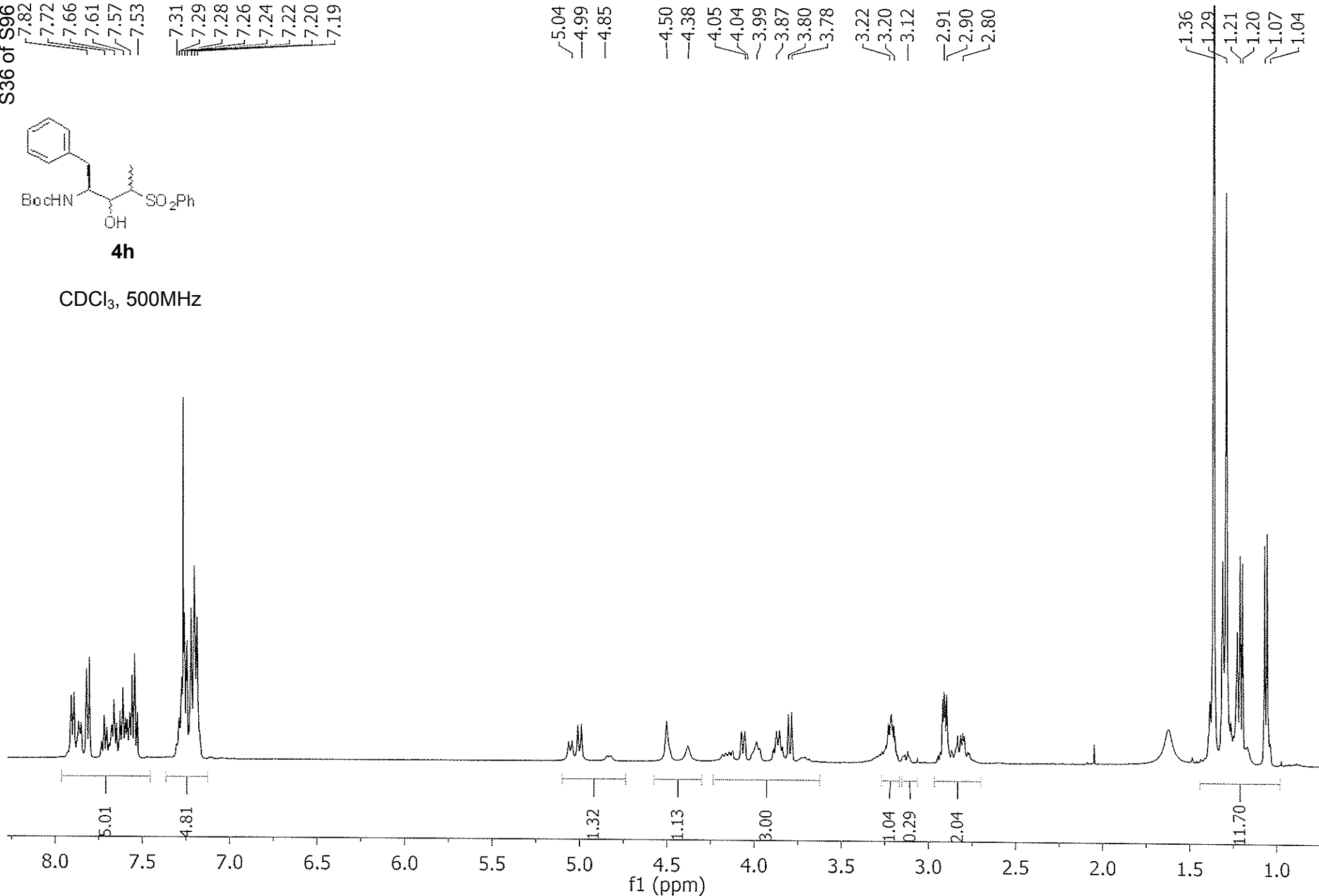60.76

59.79
58.79
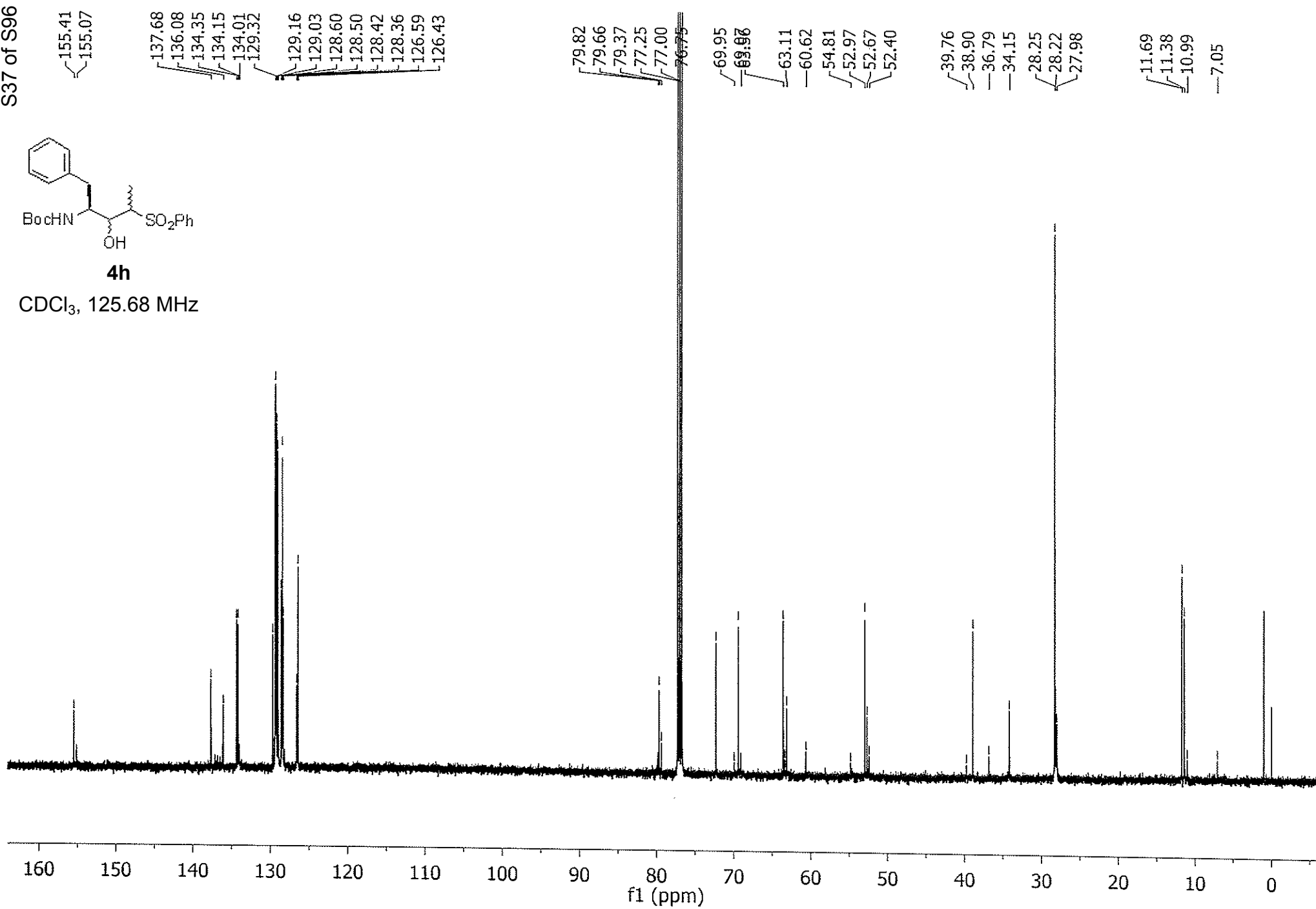
46.74
46.24

30.00
27.75
27.64
26.19
24.73
22.89
22.81

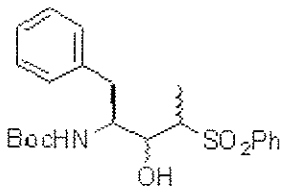7.82, 7.72, 7.66, 7.61, 7.57, 7.53
7.31, 7.29, 7.28, 7.26, 7.24, 7.22, 7.20, 7.19
5.04, 4.99, 4.85
4.50, 4.38
4.05, 4.04, 3.99, 3.87, 3.80, 3.78
3.22, 3.20, 3.12
2.91, 2.90, 2.80
1.36, 1.29, 1.21, 1.20, 1.07, 1.04

BocHN, OH, SO_2Ph

4h

CDCl$_3$, 500MHz

5.01 4.81 1.32 1.13 3.00 1.04 0.29 2.04 11.70

8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
f1 (ppm)

155.41
155.07

137.68
136.08
134.35
134.15
134.01
129.32
129.16
129.03
128.60
128.50
128.42
128.36
126.59
126.43

79.82
79.66
79.37
77.25
77.00
76.75

69.95
69.07
69.06
63.11
60.62
54.81
52.97
52.67
52.40

39.76
38.90
36.79
34.15

28.25
28.22
27.98

11.69
11.38
10.99

7.05

BocHN—CH(CH$_2$Ph)—CH(OH)—CH(CH$_3$)—SO$_2$Ph

4h

CDCl$_3$, 125.68 MHz

f1 (ppm)

4h

CDCl₃

4i

CDCl$_3$, 500MHz

7.93
7.91
7.63
7.61
7.56
7.54
7.52
5.18
4.40
3.73
3.50
3.37
3.30
2.19
1.82
1.48
1.38

2.00
3.89
0.82
0.79
1.10
0.89
2.11
1.21
2.92
16.36

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

157.94

138.46
133.53
129.29
128.83

80.46
77.01
76.75
65.26
63.96
60.37
59.68

47.87

28.39
23.81
17.47
14.63
13.21

-0.02

4i

CDCl$_3$, 125.68 MHz

170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

4i

CDCl₃

OK

BocHN—SO₂Ph
OAc

5a

CDCl₃, 500MHz

169.83
155.05
139.20
133.87
129.36
129.34
128.32
128.26
80.02
70.21
69.55
57.77
56.75
49.37
48.34
28.27
20.62
18.12
16.32

BocHN

SO₂Ph

OAc

5a

CDCl₃, 125.68 MHz

170   160   150   140   130   120   110   100   90   80   70   60   50   40   30   20   10   0
f1 (ppm)

7.94
7.90
7.88
7.88
7.65
7.58
7.58
7.57
7.56
7.55
7.54

5.31
5.30
5.29
5.28

4.36
4.33

3.64
3.61
3.46
3.44
3.42
3.40
3.33
3.30

1.75

1.39

0.91
0.89
0.86
0.85

BocHN

SO$_2$Ph

OAc

5b

CDCl$_3$, 500MHz

5.31
5.30
5.29
5.28

4.36
4.33

3.64
3.61
3.42
3.40
3.33
3.30

5.5   5.0   4.5   4.0   3.5
f1 (ppm)

1.88
1.29
2.69

1.00

0.84

0.87
2.06

2.44
0.75
8.51

6.56

8.5   8.0   7.5   7.0   6.5   6.0   5.5   5.0   4.5   4.0   3.5   3.0   2.5   2.0   1.5   1.0   0.5   0.0
f1 (ppm)

169.68
155.64
139.14
133.91
129.12
128.34
127.35
79.86
68.08
57.31
56.58
28.27
20.60
19.59
17.07

5b

CDCl$_3$, 125.68 MHz

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

5c

CDCl₃, 500MHz

169.74
155.44
139.25
133.82
129.31
129.27
128.33
128.26
127.34
79.94
79.80
70.12
69.14
57.89
56.35
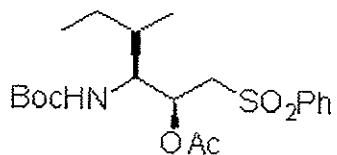52.01
50.58
41.13
39.56
28.29
24.68
23.20
21.77
21.56
20.59

BocHN
SO$_2$Ph
OAc

5c

CDCl$_3$, 125.68 MHz

f1 (ppm)
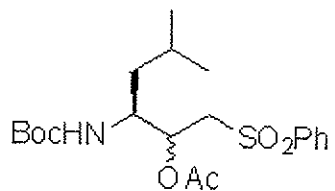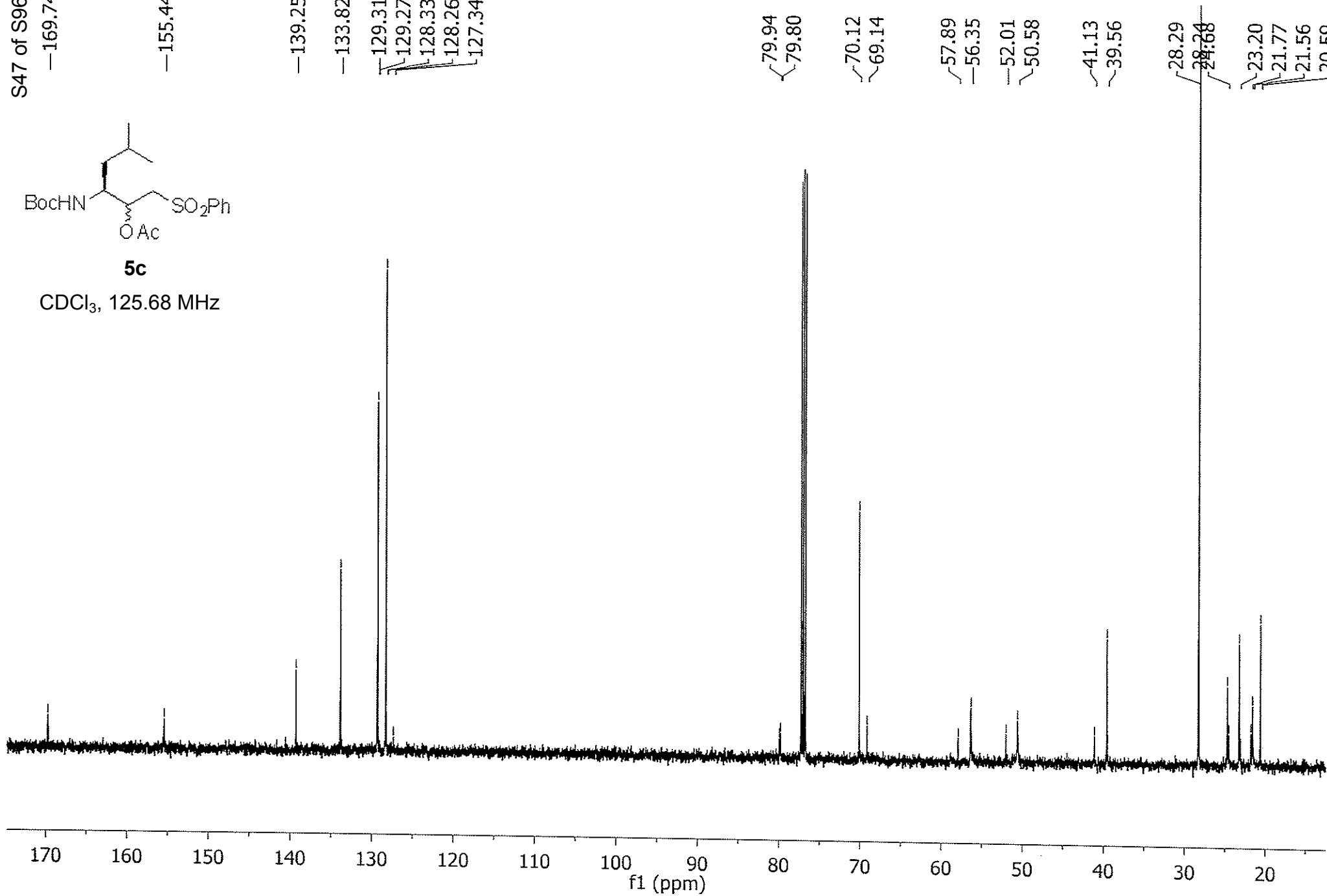
7.89
7.66
7.57
5.35
4.30
3.68
3.45
3.42
3.41
3.31
3.28
1.73
1.41
1.30
0.90
0.89
0.84

BocHN—SO₂Ph

OAc

5d

CDCl₃, 500MHz

H2O

2nd diastereoisomer

2nd diastereoisomer

1.99
1.24
2.55
1.00
0.82
1.10
1.20
1.12
0.18
3.08
1.33
10.67
1.44
3.53
3.66

f1 (ppm)

169.66
155.48
139.04
133.62
129.19
128.11
127.04
80.64
67.95
56.52
56.27
35.15
27.89
24.22
20.62
15.50
10.79

BocHN SO₂Ph
OAc

5d

CDCl₃, 125.68 MHz

170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

7.90
7.89
7.88
7.56
7.27
7.26
7.26
7.25
7.21
7.12
5.32
5.31
5.30
5.30
5.30
5.29
5.29
5.28
4.40
4.08
3.43
3.42
3.41
3.39
3.38
2.85
2.83
2.61
1.84
1.57
1.28

5e
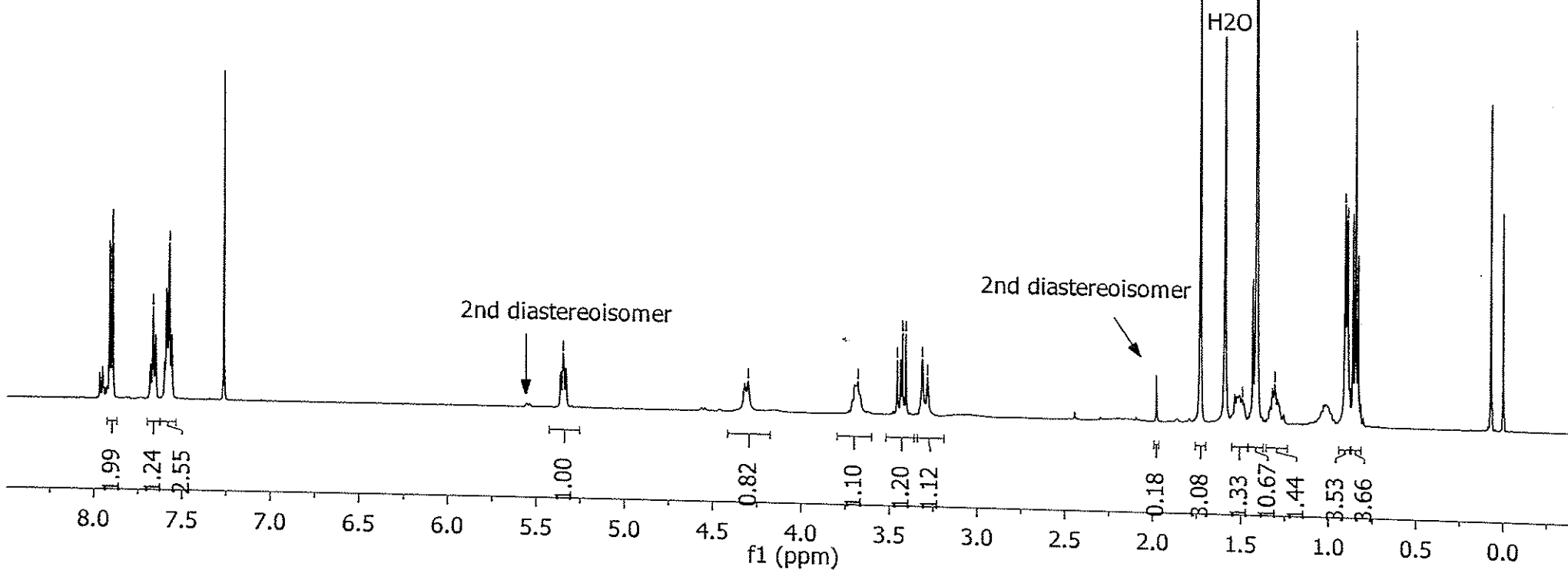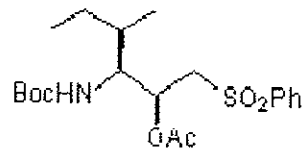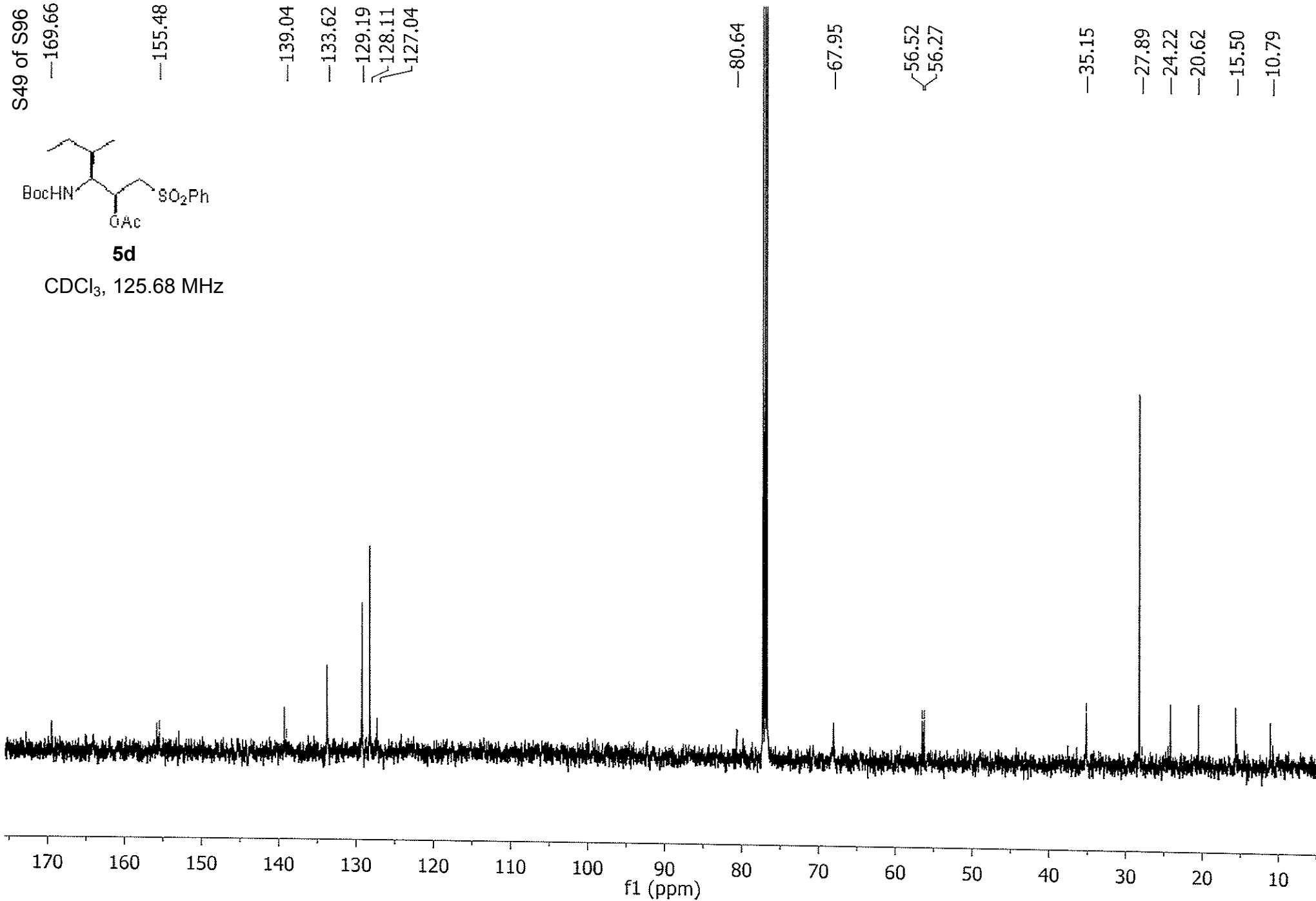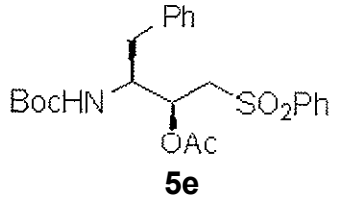
1 diastereoisomer

CDCl₃, 500MHz

1.86
0.92
0.93
6.78
0.88
2.08
1.00
0.67
1.08
2.11
1.03
0.97
2.91
10.33

9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
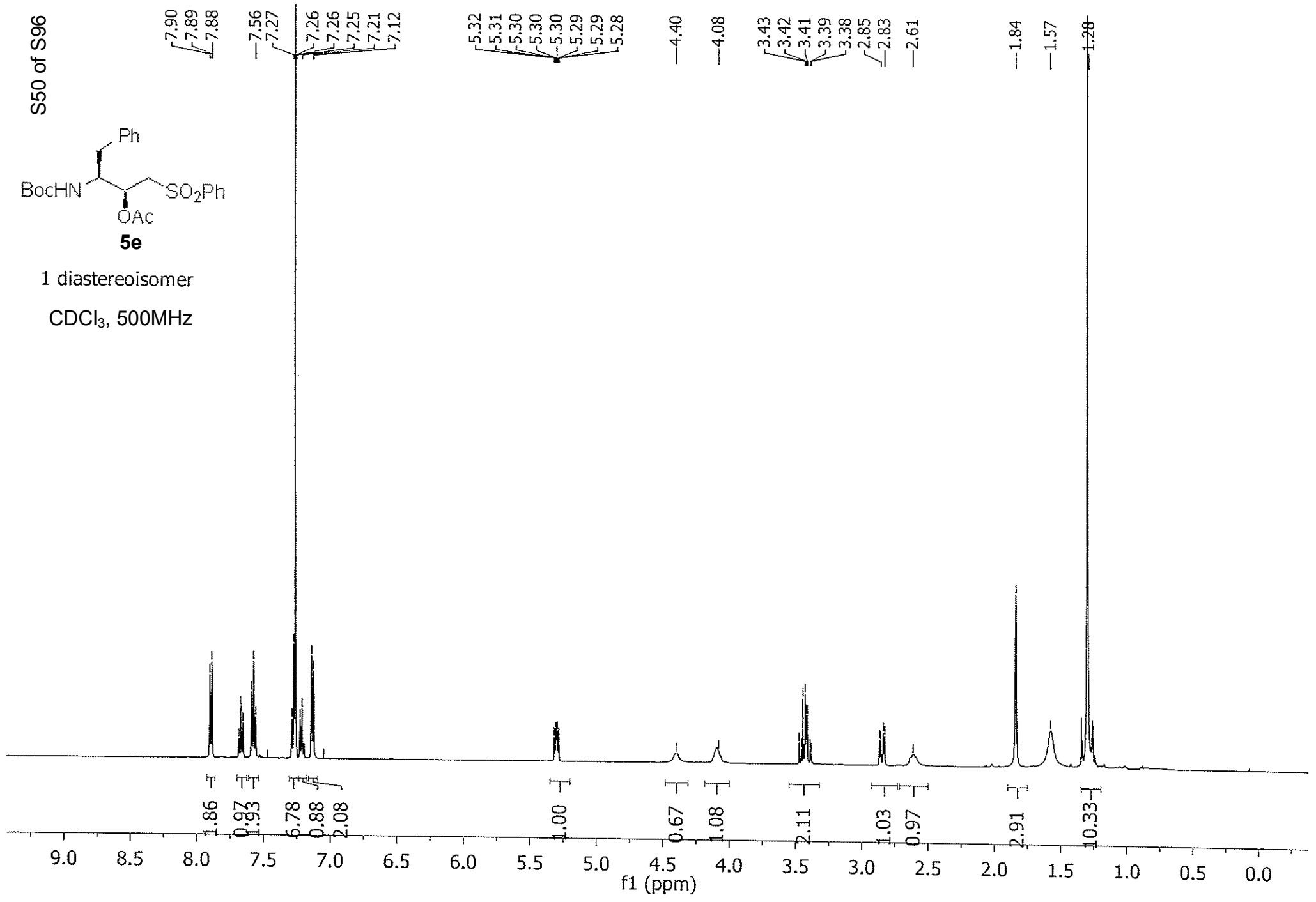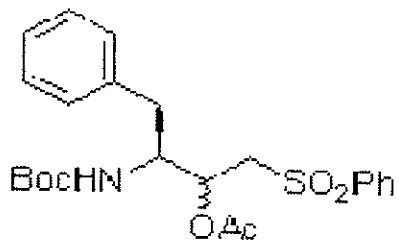
5e

CDCl₃, 125.68 MHz

169.84
169.84
155.26
155.26
139.26
136.52
133.84
129.29
129.01
128.57
128.22
126.76
79.99
79.95
77.25
77.00
76.75
69.75
56.66
56.64
53.56
53.50
36.82
36.82
28.12
20.63
20.63

7.90, 7.88, 7.83, 7.82, 7.66, 7.56, 7.14, 6.99, 6.97, 6.74, 6.72, 5.34, 5.29, 4.59, 4.57, 4.49, 4.48, 4.04, 3.96, 3.43, 2.84, 2.45, 2.00, 1.82, 1.35, 1.31, 0.96, 0.16

5f

CDCl$_3$, 500MHz

2nd diastereoisomer

2nd diastereoisomer

1.92, 3.02, 2.03, 1.85, 1.00, 0.94, 0.99, 2.26, 0.94, 1.08, 3.07, 9.73, 8.79, 5.88

f1 (ppm)
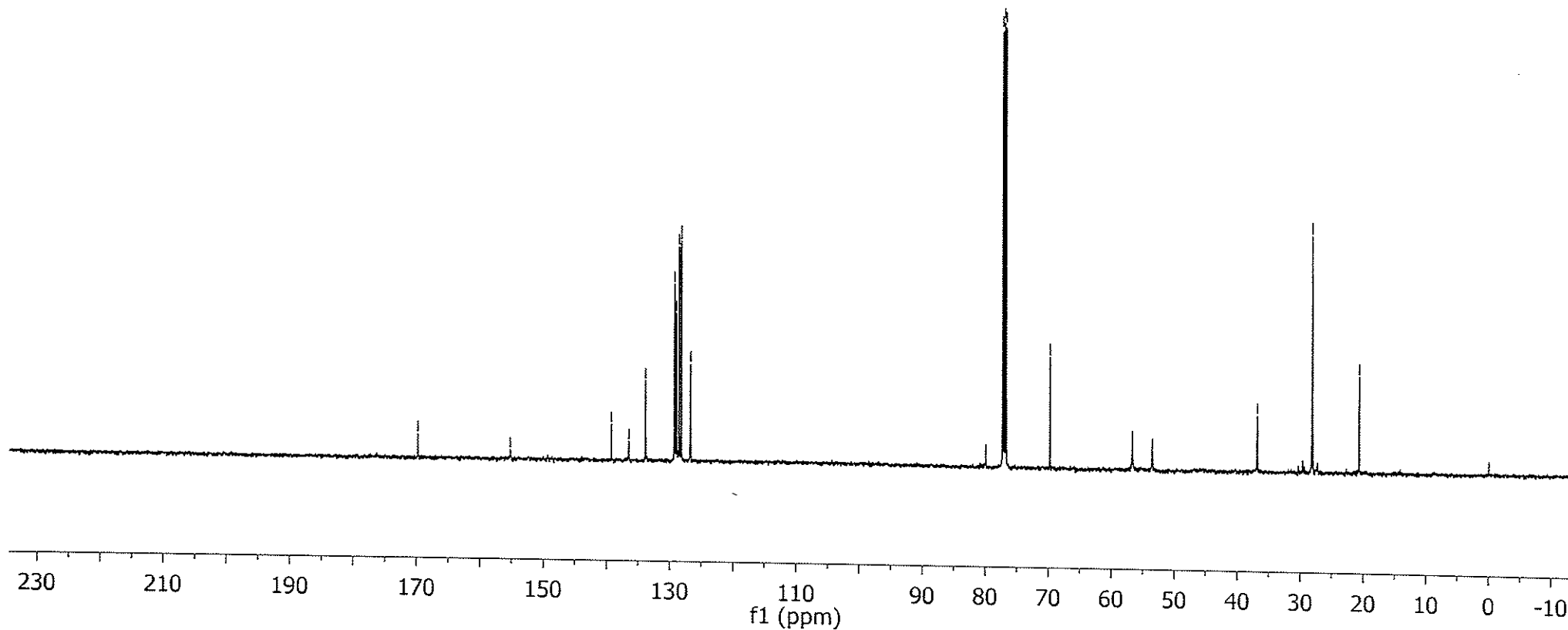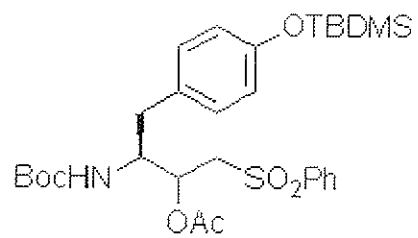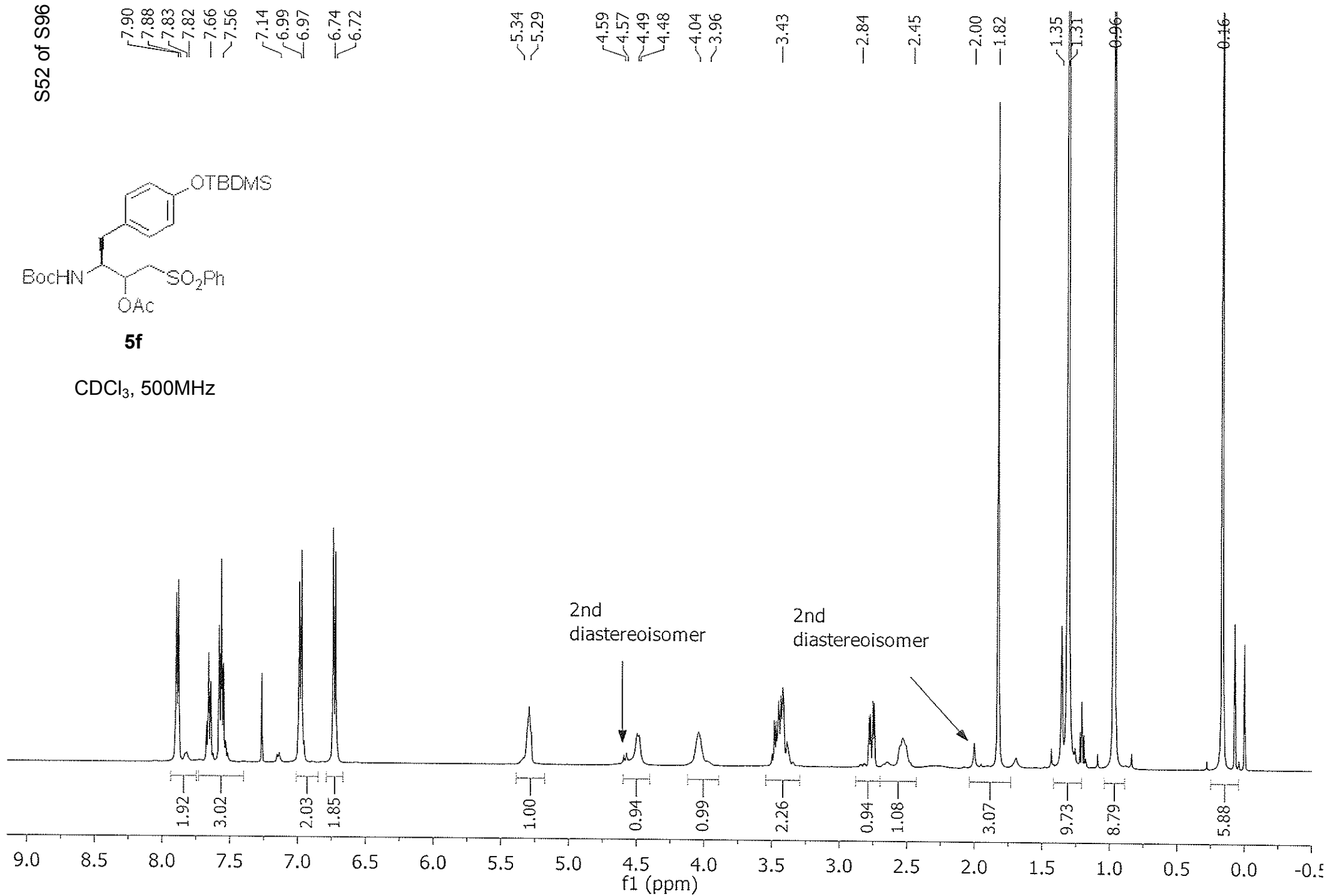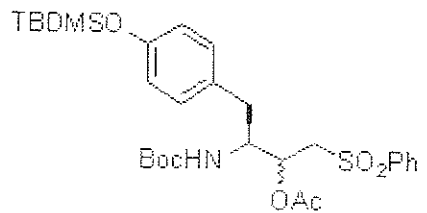
169.80

155.27
154.42

139.25
133.79
129.90
129.26
128.19

120.12

79.86

69.66
68.16

56.64
53.52

35.92

28.14
25.63
20.55
18.15

4.49
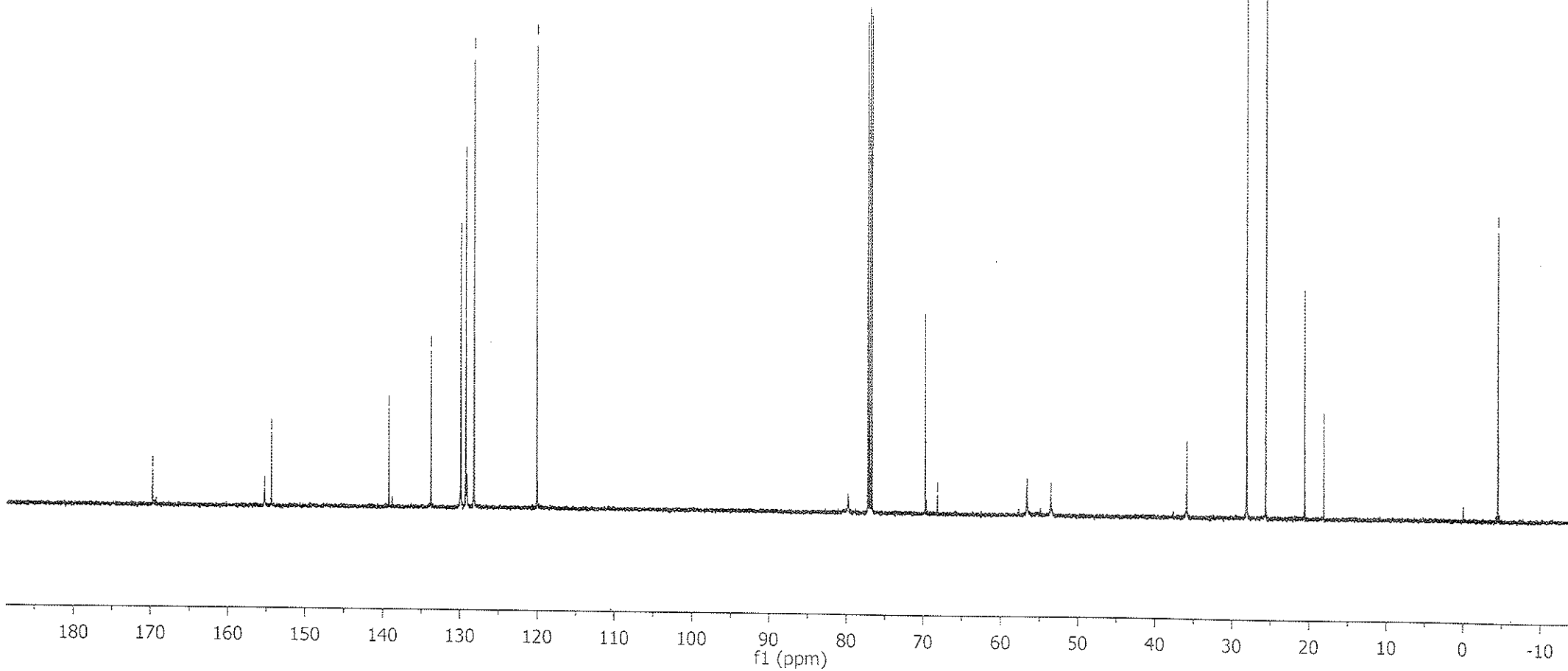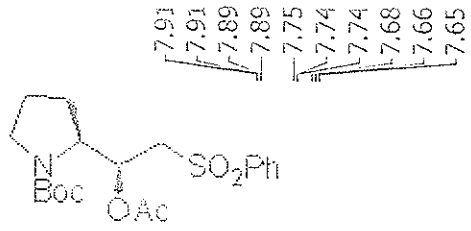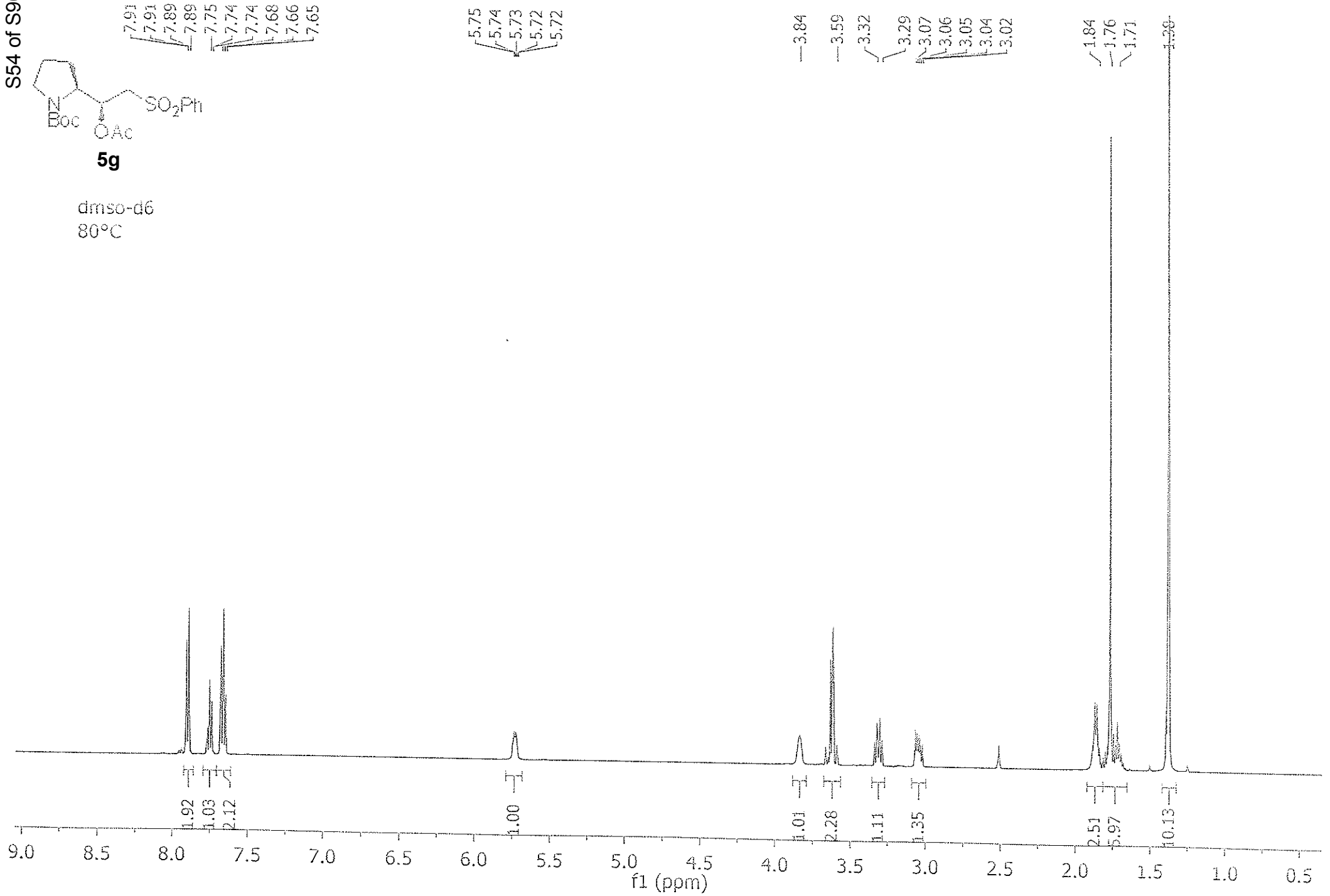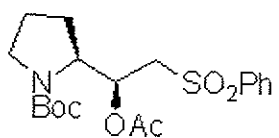
TBDMSO

BocHN

SO$_2$Ph

OAc

5f

CDCl$_3$, 125.68 MHz

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

5g

dmso-d6
80°C

5g

dmso-d6
80°C

1 diastereoisomer

7.83, 7.79, 7.76, 7.59, 7.56, 7.51, 7.48, 7.45
7.19, 7.16, 7.12, 7.09, 7.04
5.42, 5.40, 5.32, 5.31, 5.30, 5.29, 5.27, 5.25
4.65, 4.63, 4.26
4.10, 4.08, 4.07, 4.00
3.93
3.38
2.70, 2.69, 2.68, 2.66
2.47
2.28, 2.15, 2.02, 1.92
1.45, 1.43, 1.24
1.17, 1.17, 1.16, 1.12
0.00, -0.07

5h

CDCl₃, 500MHz

f1 (ppm)

2.35 3.70 6.50 1.22 1.00 1.44 1.21 2.50 4.43 16.92

170.34
170.12
170.05
155.31
155.17
137.49
136.88
133.86
133.82
129.21
129.15
128.65
128.60
128.52
126.80
126.75
80.55
80.08
79.82
71.93
71.72
70.47
61.06
60.17
52.38
40.74
39.79
37.56
36.39
28.01
21.17
20.94
14.24
12.98
11.96
10.79

BocHN

SO$_2$Ph

OAc

5h

CDCl$_3$, 125.68 MHz

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

f1 (ppm)

5i

CDCl₃, 500MHz

7.91, 7.89, 7.64, 7.62, 7.57, 7.56, 7.54, 5.08, 4.32, 4.24, 3.61, 3.49, 3.37, 3.17, 1.77, 1.43, 1.40, 1.39

2.01 3.48 1.00 1.06 2.38 1.17 7.16 12.86
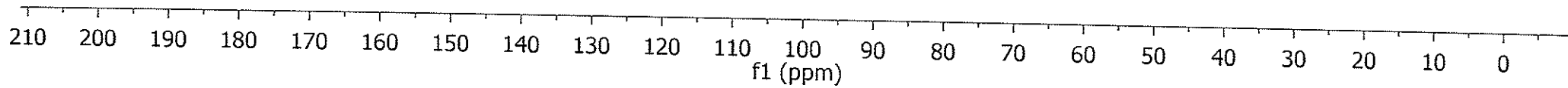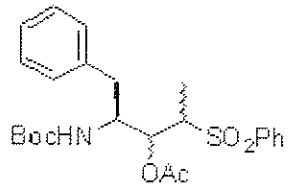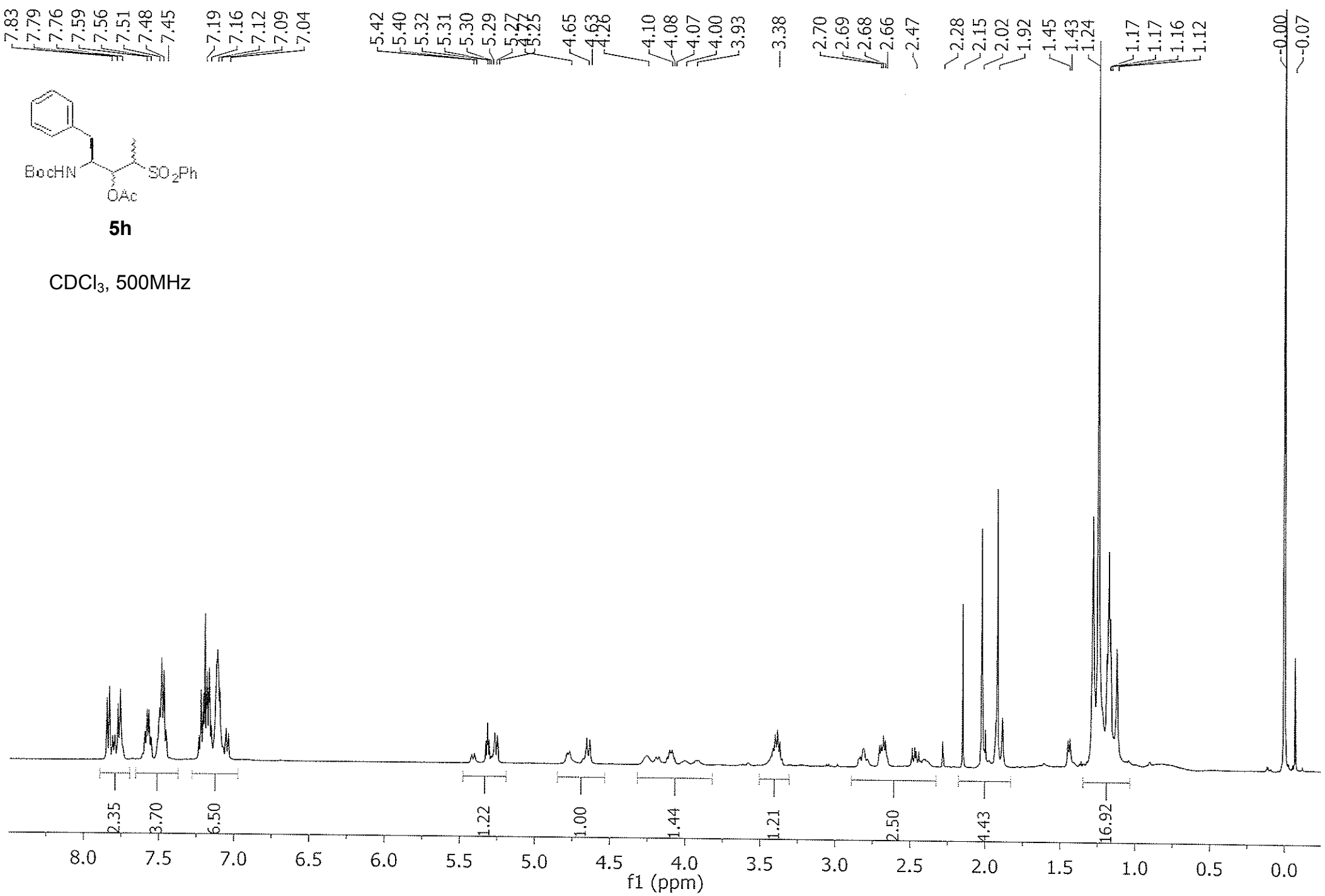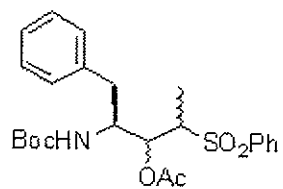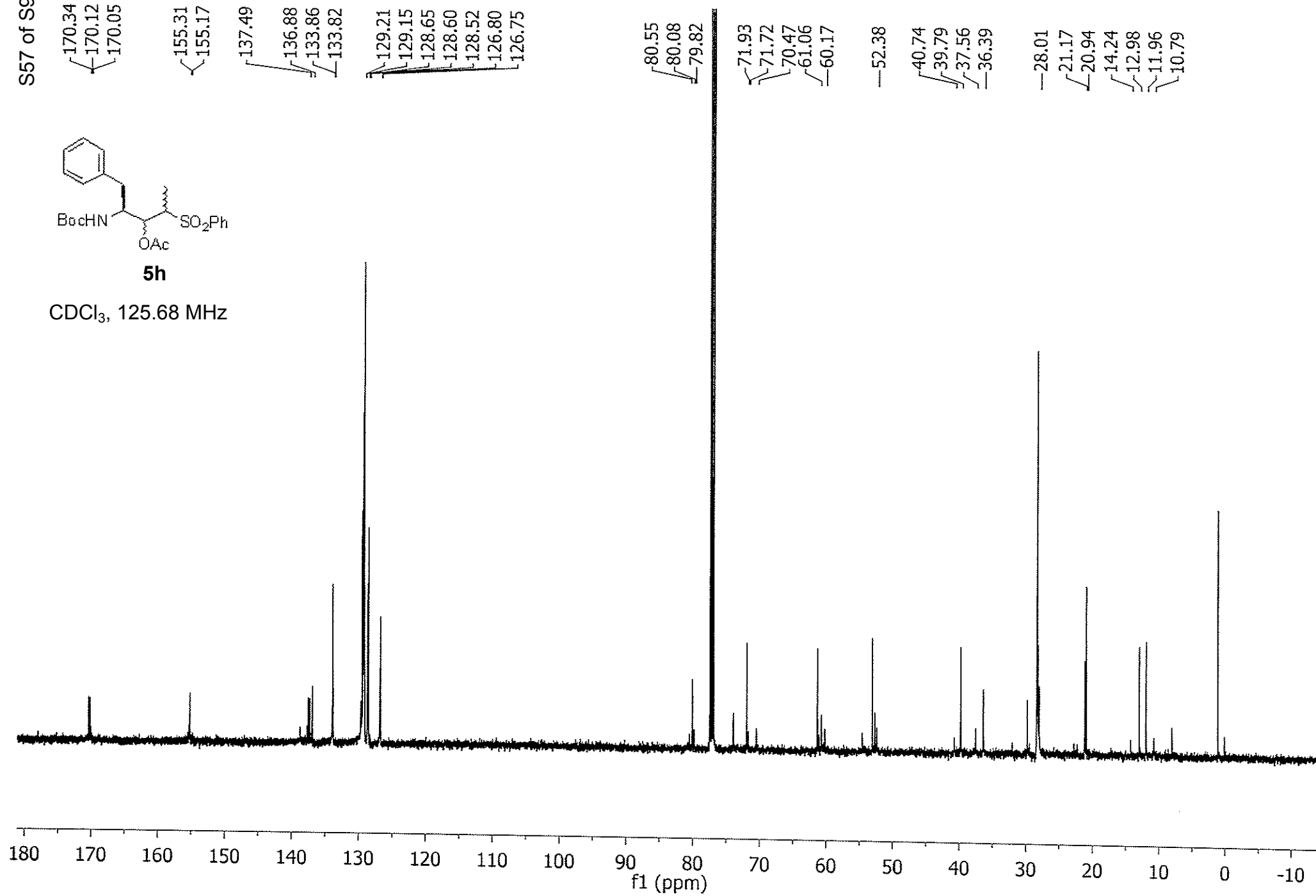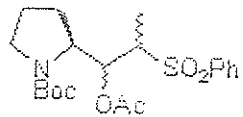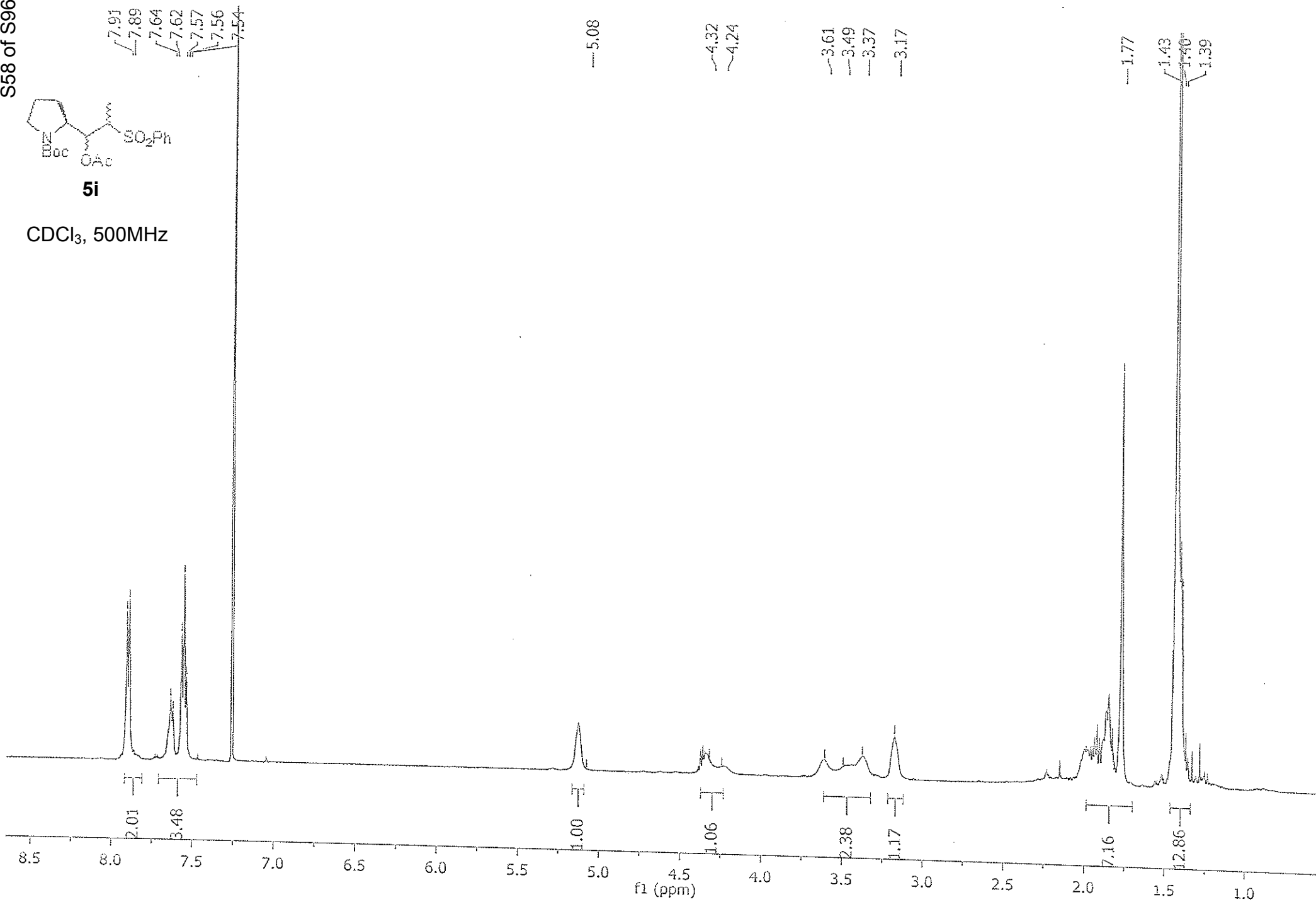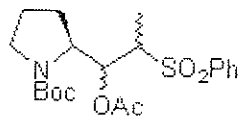
169.72
155.37
138.42
133.49
128.97
128.88
79.69
73.49
61.99
57.31
46.93
29.24
28.42
23.26
20.70
11.72
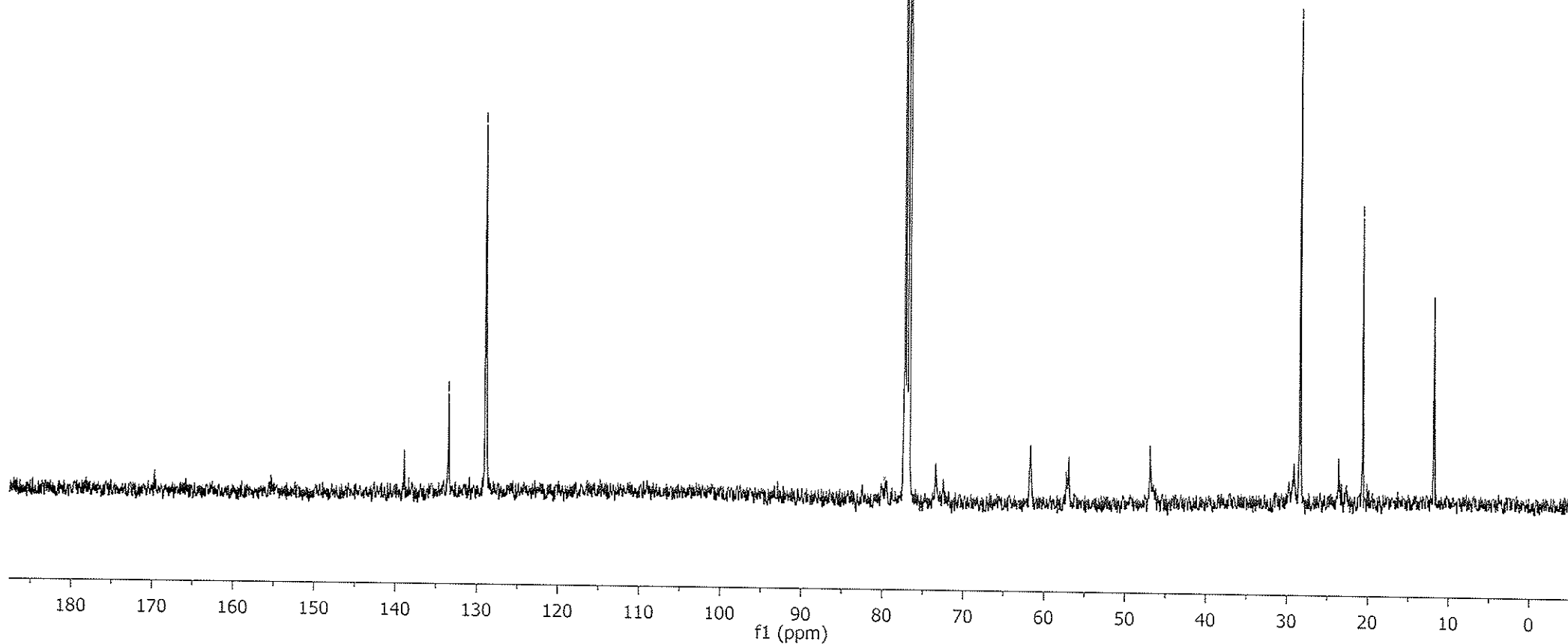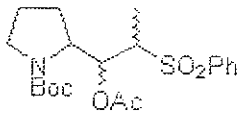
5i

CDCl$_3$, 125.68 MHz
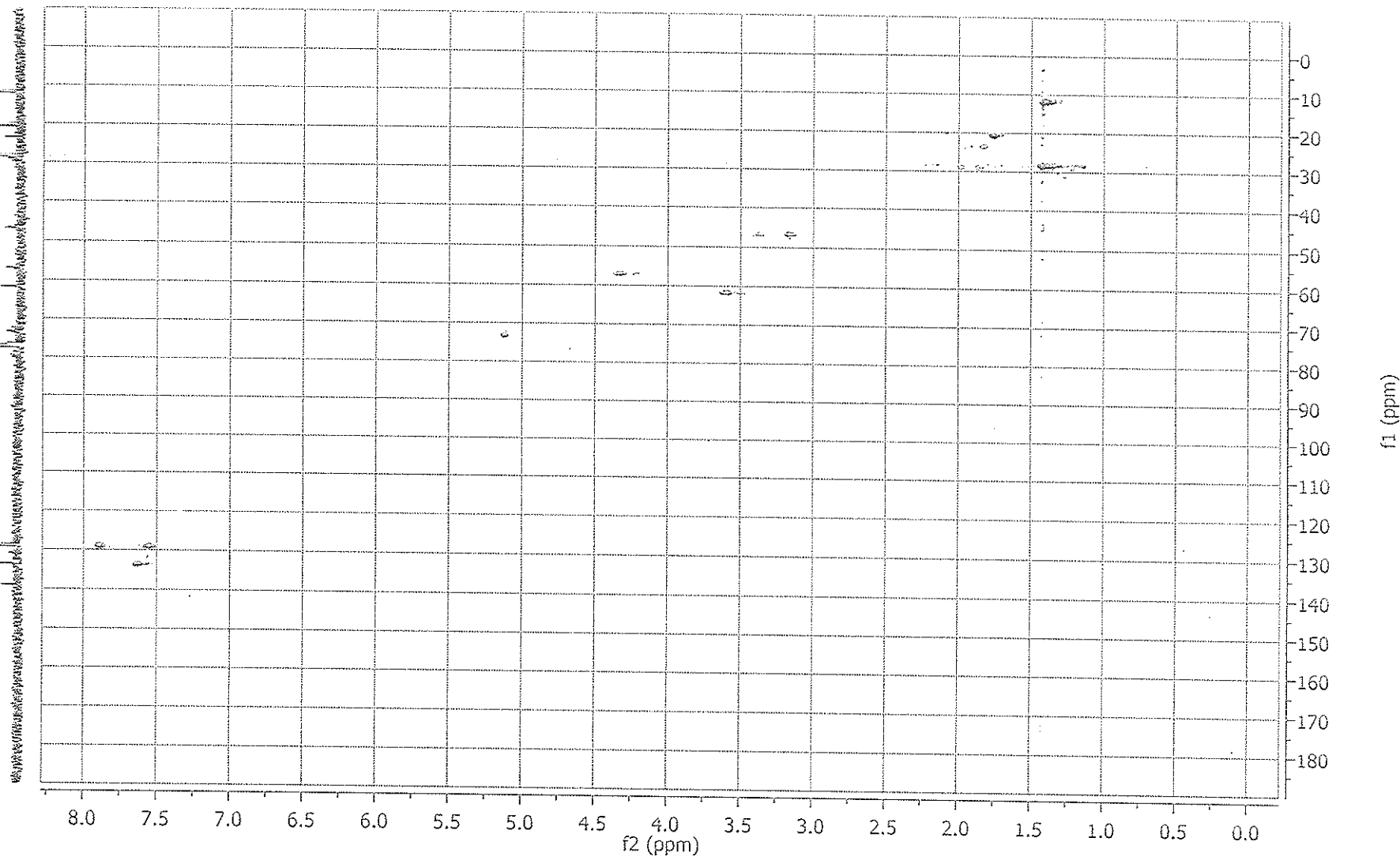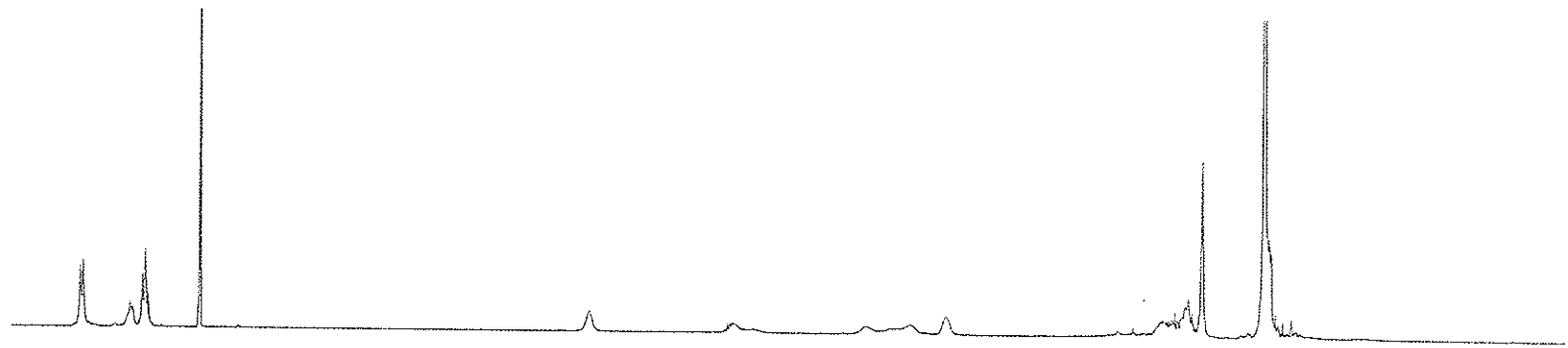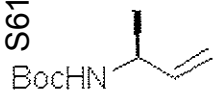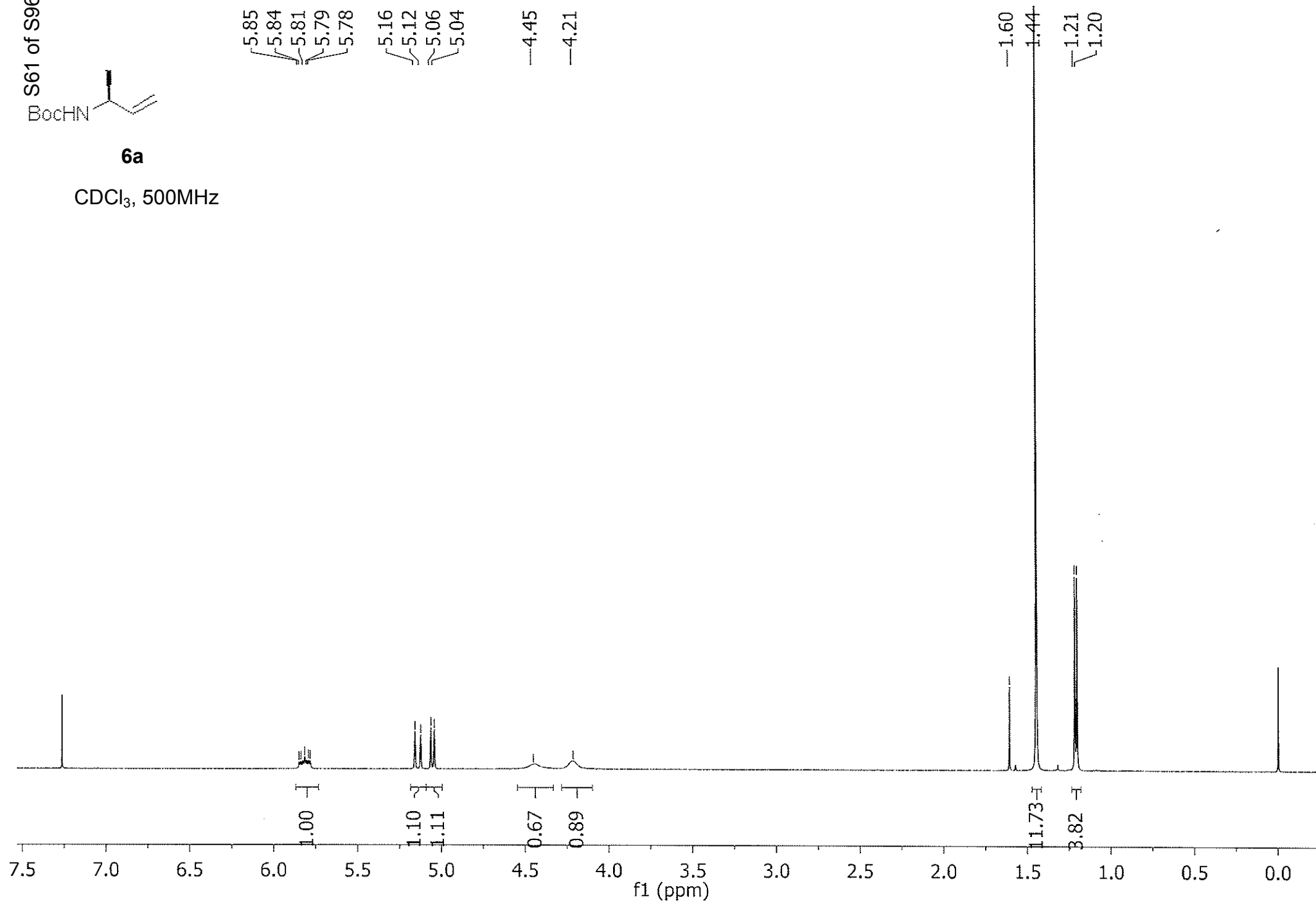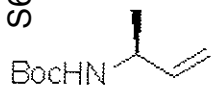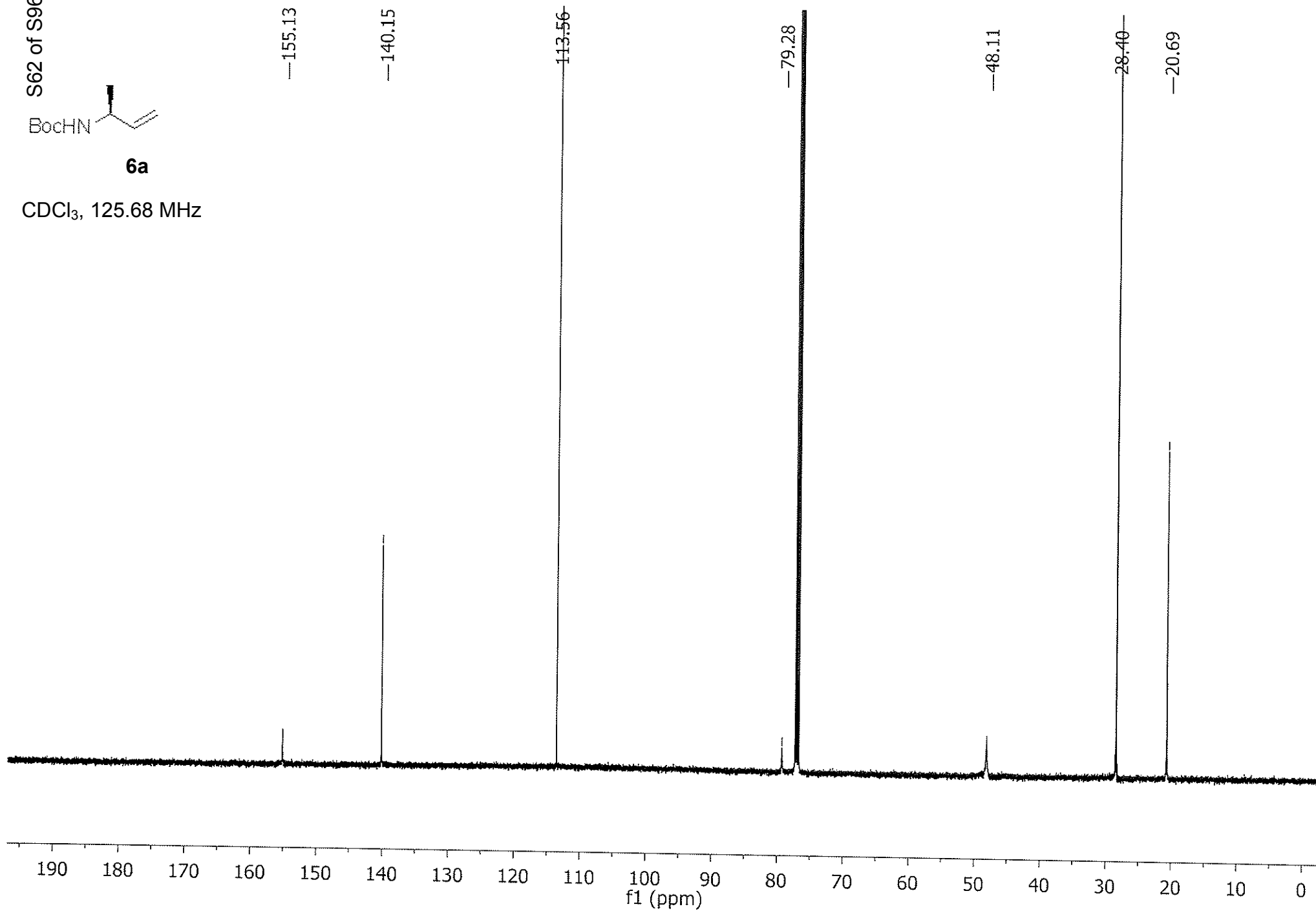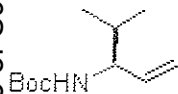
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

5i

CDCl$_3$

6a

CDCl₃, 500MHz

6a

CDCl₃, 125.68 MHz

¹³C NMR peaks at: 155.13, 140.15, 113.56, 79.28, 48.11, 28.40, 20.69 ppm

6b

CDCl₃, 500MHz

155.74
137.47
115.21
79.33
58.11
32.38
28.56
18.80
18.15

BocHN

6b

CDCl$_3$, 125.68 MHz

160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

f1 (ppm)

6c

CDCl$_3$, 500MHz

5.74
5.73
5.72
5.71
5.69
5.68
5.67

5.14
5.11
5.04
5.02

4.42

4.12

1.69
1.68
1.66
1.65
1.64
1.62
1.61

1.29

0.91
0.90
0.89
0.89

1.00

1.03
1.06

0.81

1.00

1.08

9.80
2.34

6.64

7.5   7.0   6.5   6.0   5.5   5.0   4.5   4.0   3.5   3.0   2.5   2.0   1.5   1.0   0.5
f1 (ppm)

—155.08

—139.37

—113.95

—79.18

—51.06

—44.73

—28.06

—24.45

22.66
22.04

6c

CDCl$_3$, 125.68 MHz

155 145 135 125 115 105 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10
f1 (ppm)

BocHN

6d

CDCl$_3$, 500MHz

—5.73

5.16
5.13
5.11

—4.56

—4.08

—1.54
—1.46

—1.09

0.93
0.91
0.90
0.87
0.86

0.93
0.91
0.90
0.87
0.86

3.00
2.94

1.05 1.00 0.95 0.90 0.85 0.80 0.75 0.70 0.65
f1 (ppm)

1.00

2.12

0.73

0.73

3.09

1.20
7.24

7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

6d
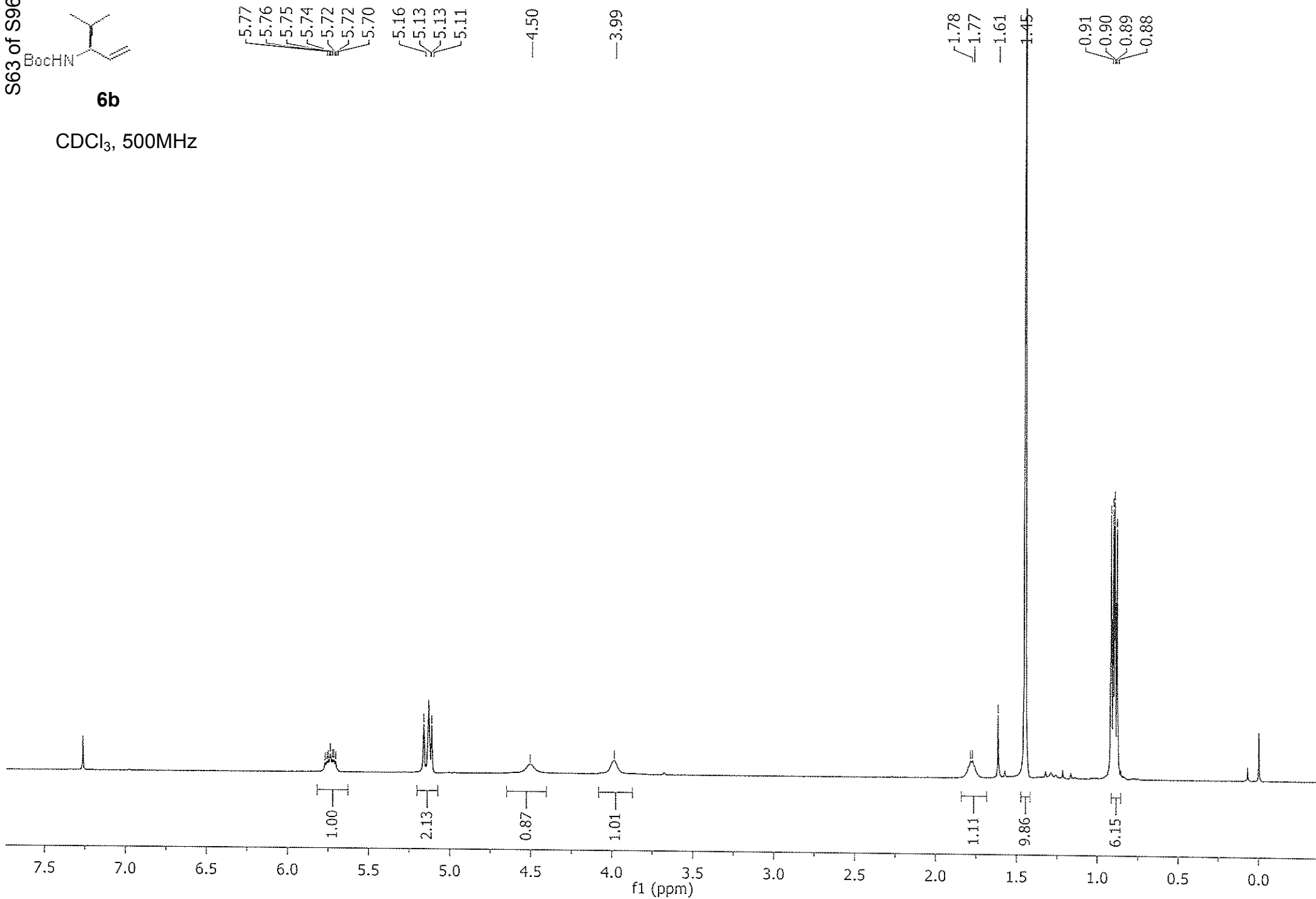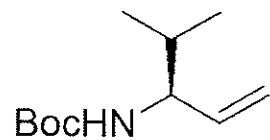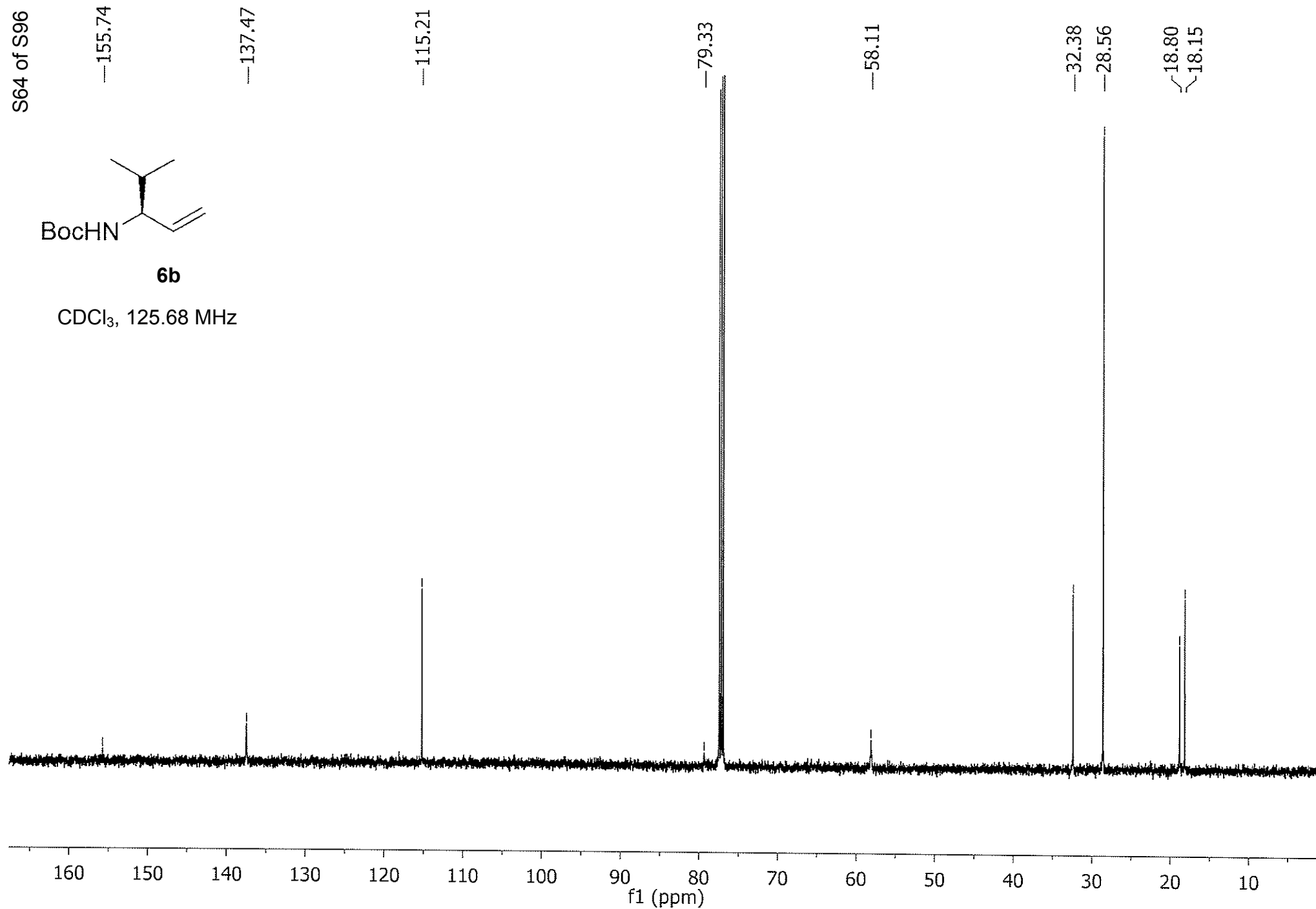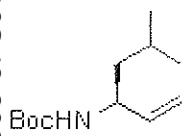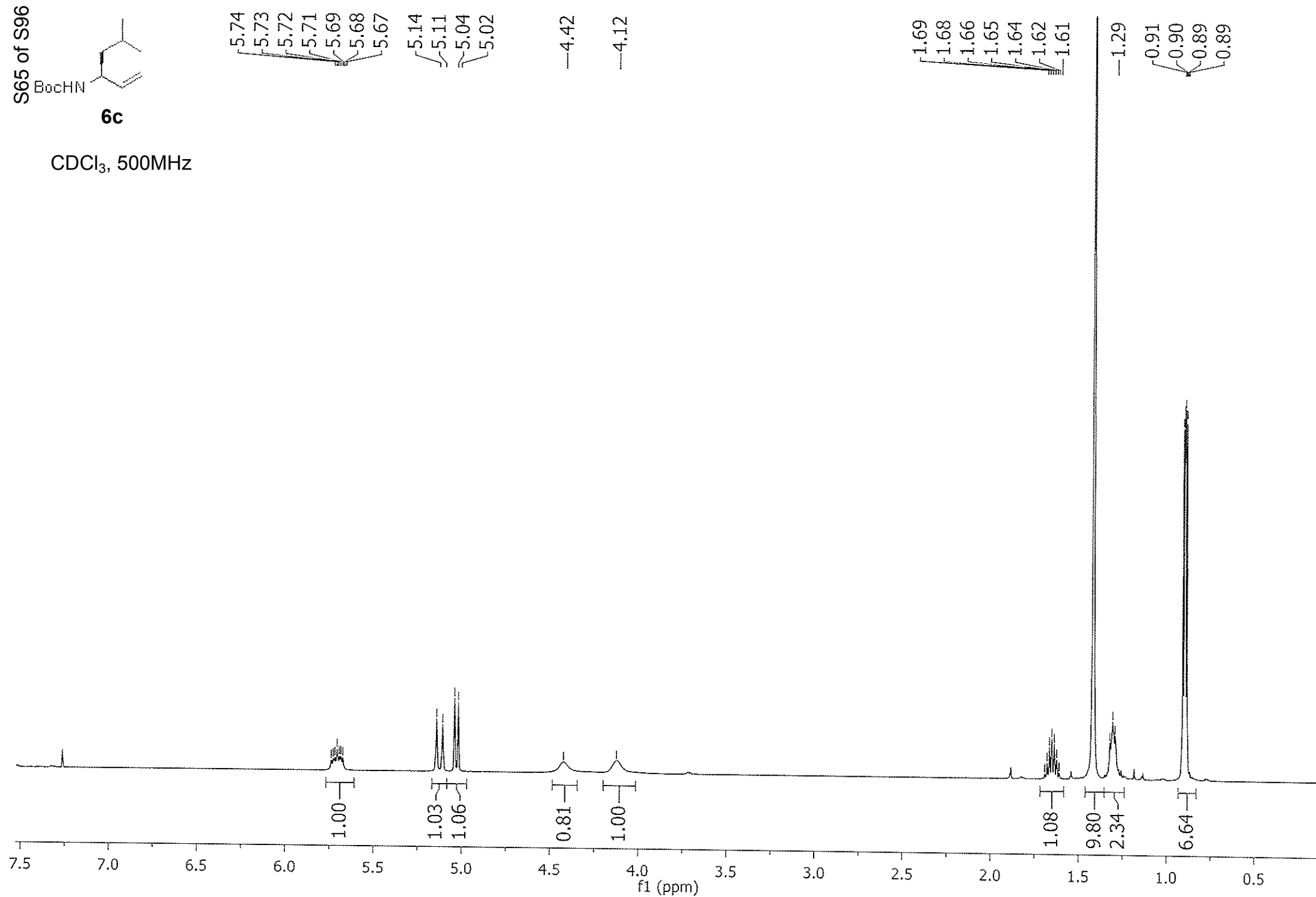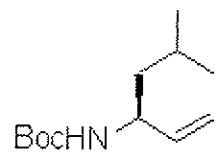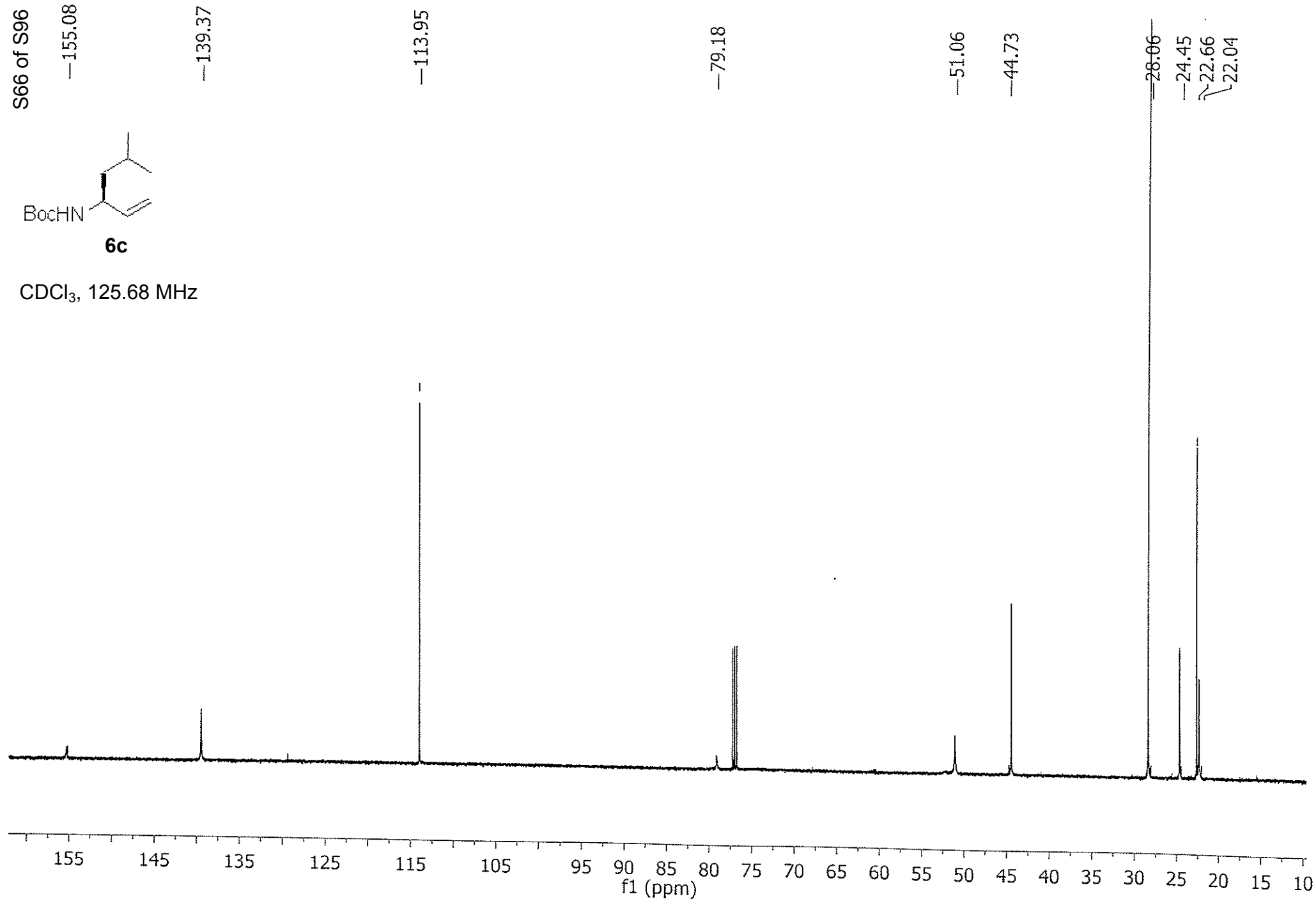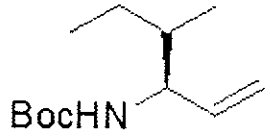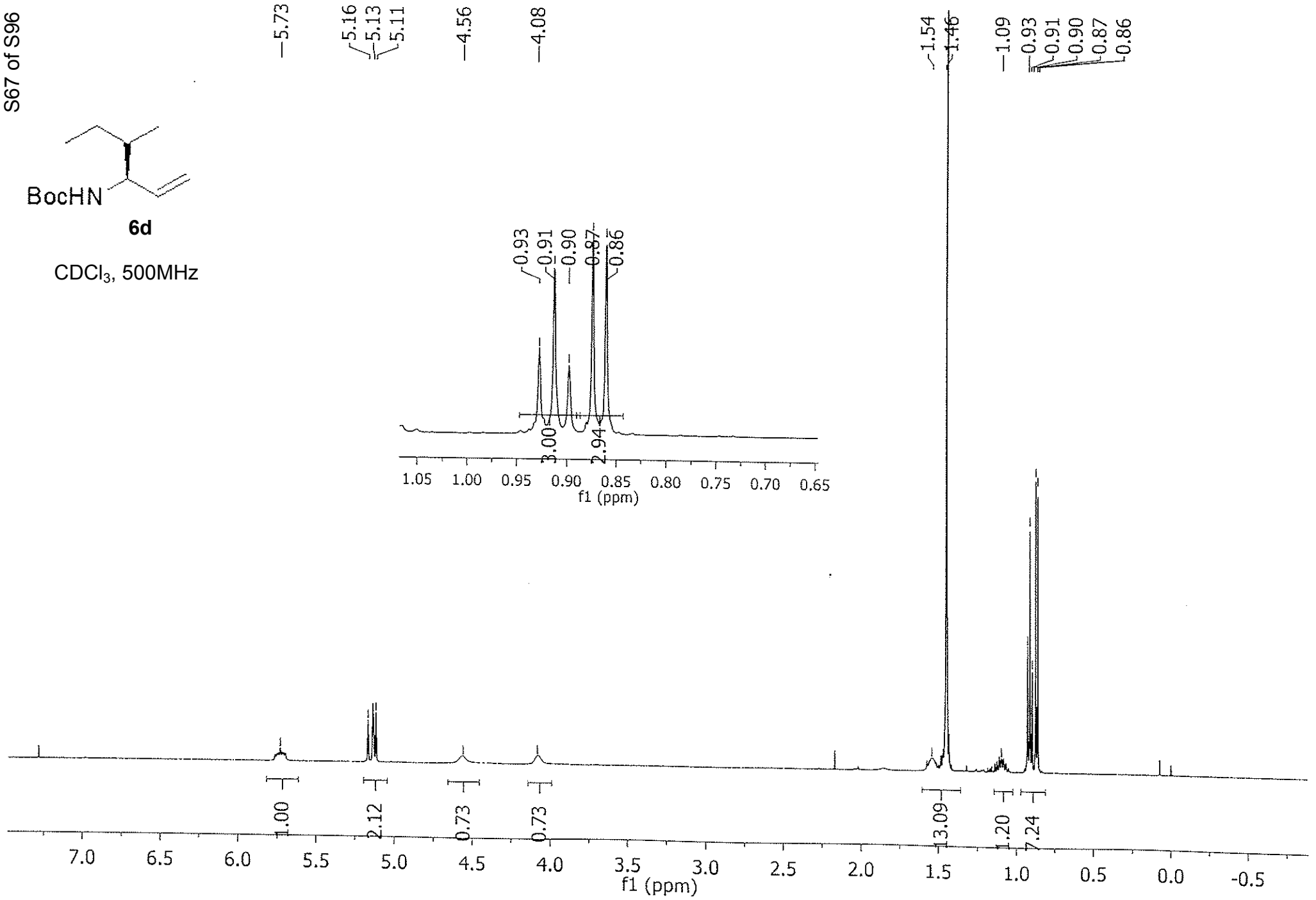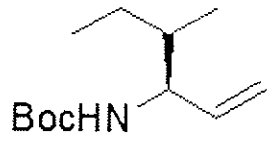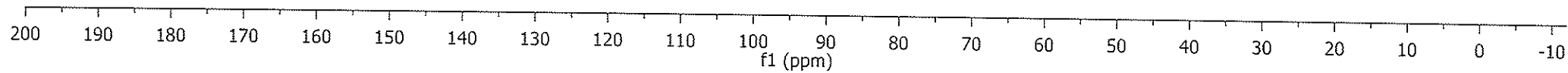
CDCl₃, 125.68 MHz

7.23, 7.23, 7.22, 7.22, 7.20, 7.20, 7.19, 7.19, 7.18, 7.18
5.84, 5.82, 5.81, 5.80, 5.80, 5.79, 5.78, 5.77, 5.13, 5.13, 5.09, 5.08, 5.07, 5.07
4.50, 4.43
2.85, 2.83
1.74
1.54, 1.41, 1.28, 1.27
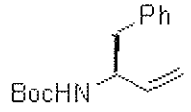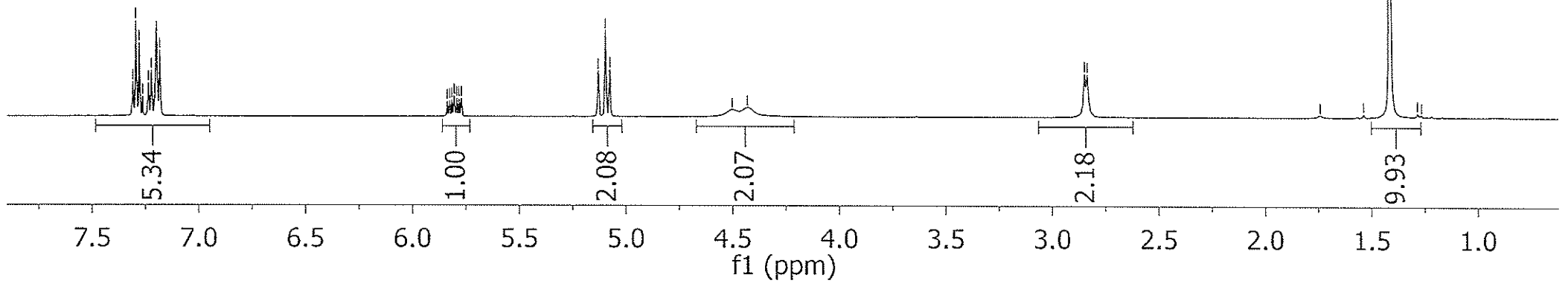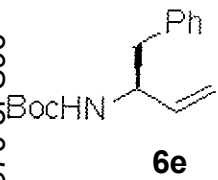
6e

CDCl$_3$, 500MHz

5.34 1.00 2.08 2.07 2.18 9.93

f1 (ppm)
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0

CDCl₃, 125.68 MHz

7.03
7.02
6.76
6.75
5.78
5.77
5.76
5.75
5.74
5.10
5.07
5.06
5.05
4.47
4.36
2.75
1.41
0.98
0.19

OTBDMS

BocHN

6f

CDCl$_3$, 500MHz

2.09
2.05
1.00
2.05
1.68
2.05
9.38
9.48
6.11

8.0   7.5   7.0   6.5   6.0   5.5   5.0   4.5   4.0   3.5   3.0   2.5   2.0   1.5   1.0   0.5   0.0   -0.5
f1 (ppm)
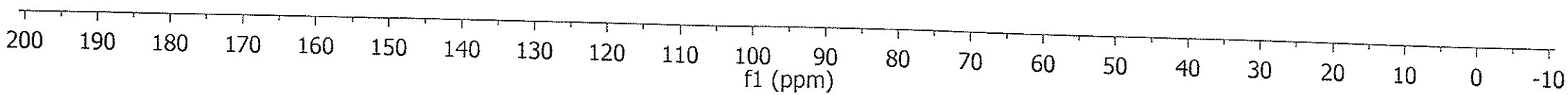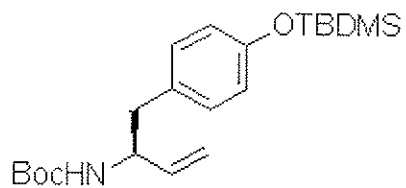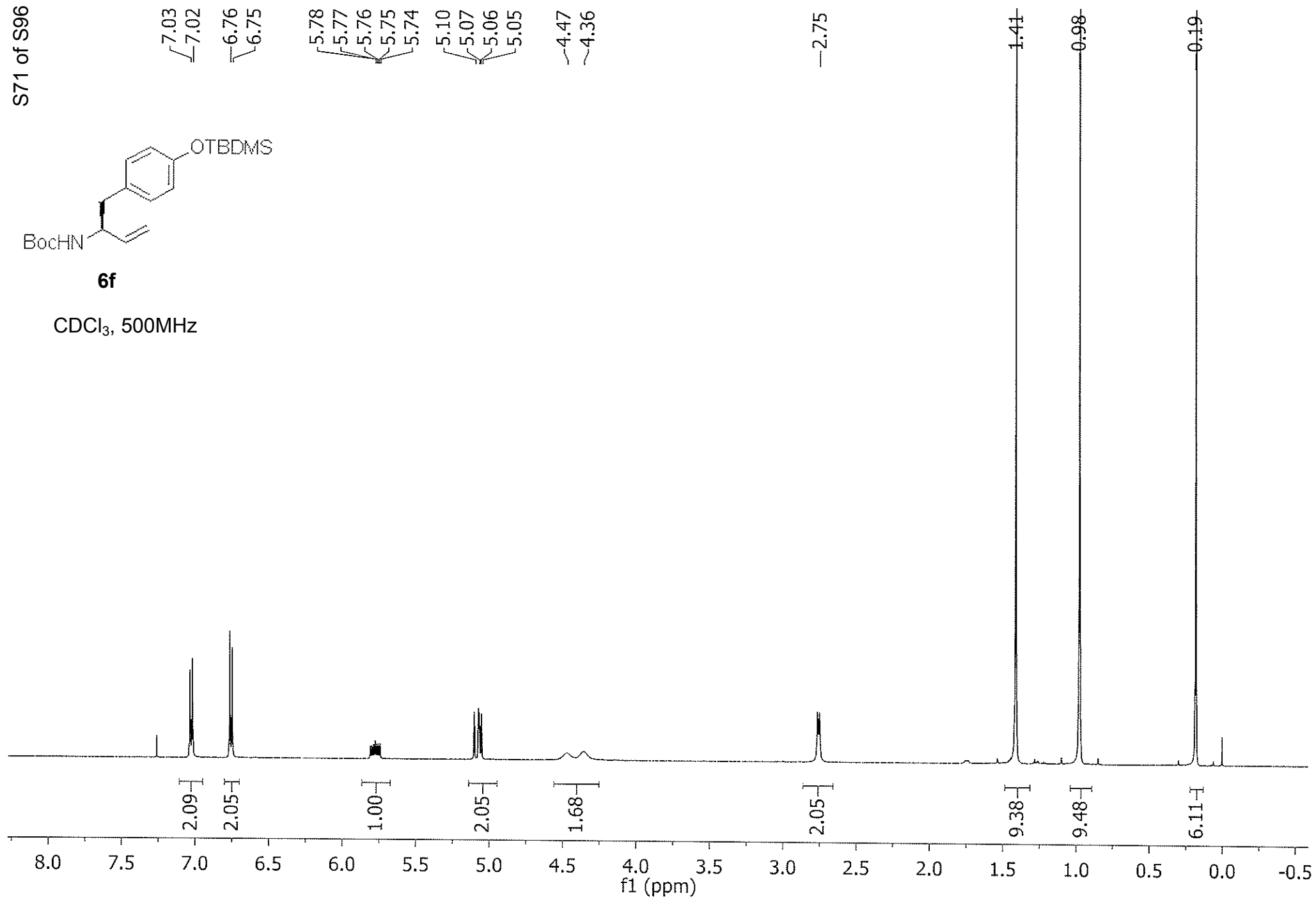
155.16
154.40
138.28
130.52
129.76
119.92
114.61
79.01
53.34
40.57
27.94
25.37
18.03
-4.80

OTBDMS

BocHN

6f

CDCl$_3$, 125.68 MHz

170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)
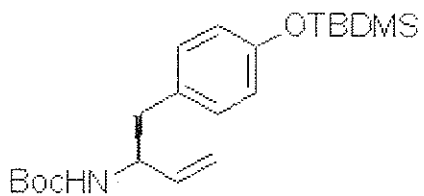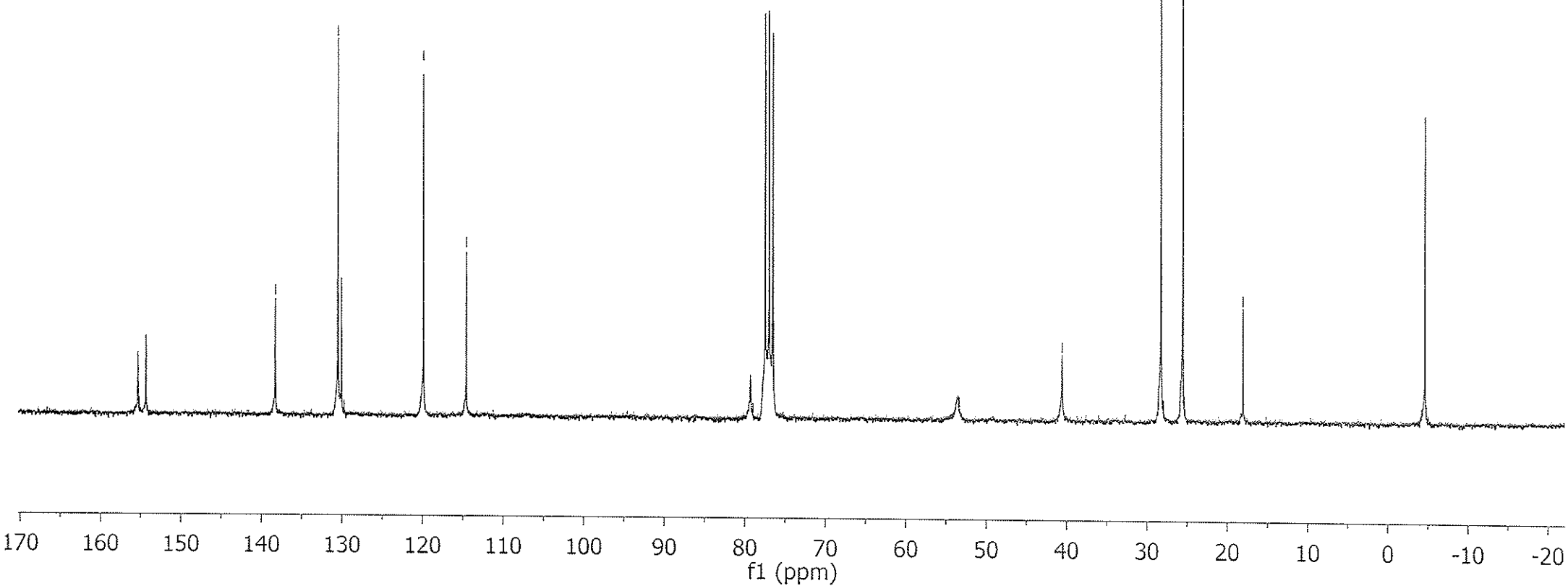
—7.01
—6.75
—5.78
—5.09
—4.50
—4.35
—2.75
1.41

OH

BocHN

6f'

CDCl$_3$, 500MHz

2.09
2.08
1.00
0.66
2.13
1.97
2.04
8.35

7.2 6.8 6.4 6.0 5.6 5.2 4.8 4.4 4.0 3.6 3.2 2.8 2.4 2.0 1.6 1.2
f1 (ppm)
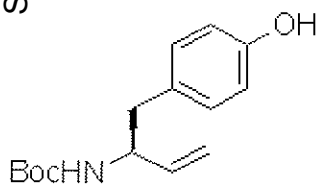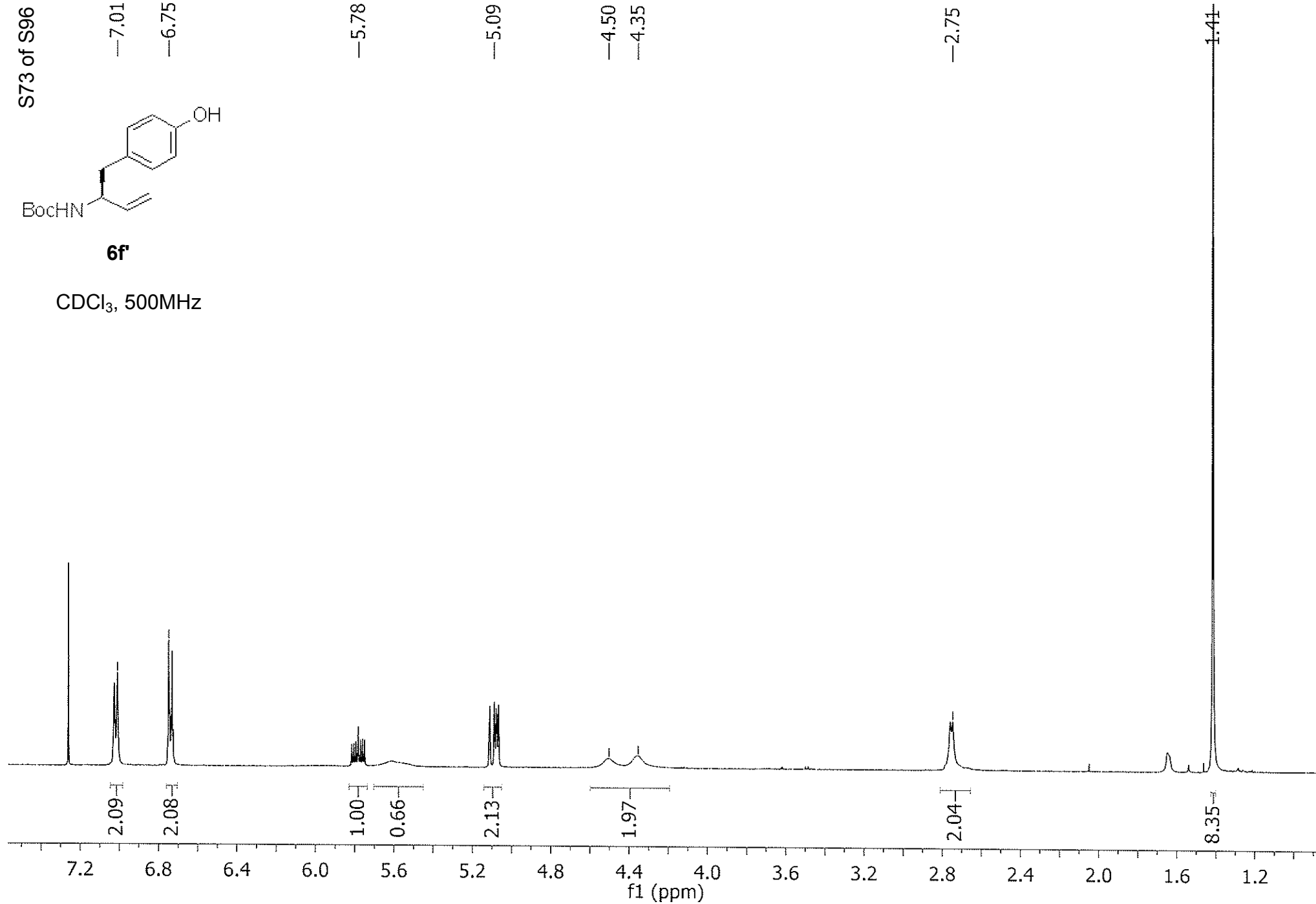
155.51
154.62
138.06
130.54
129.03
115.26
114.66
79.67
53.70
40.65
28.36
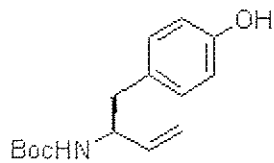
6f'

CDCl$_3$, 125.68 MHz
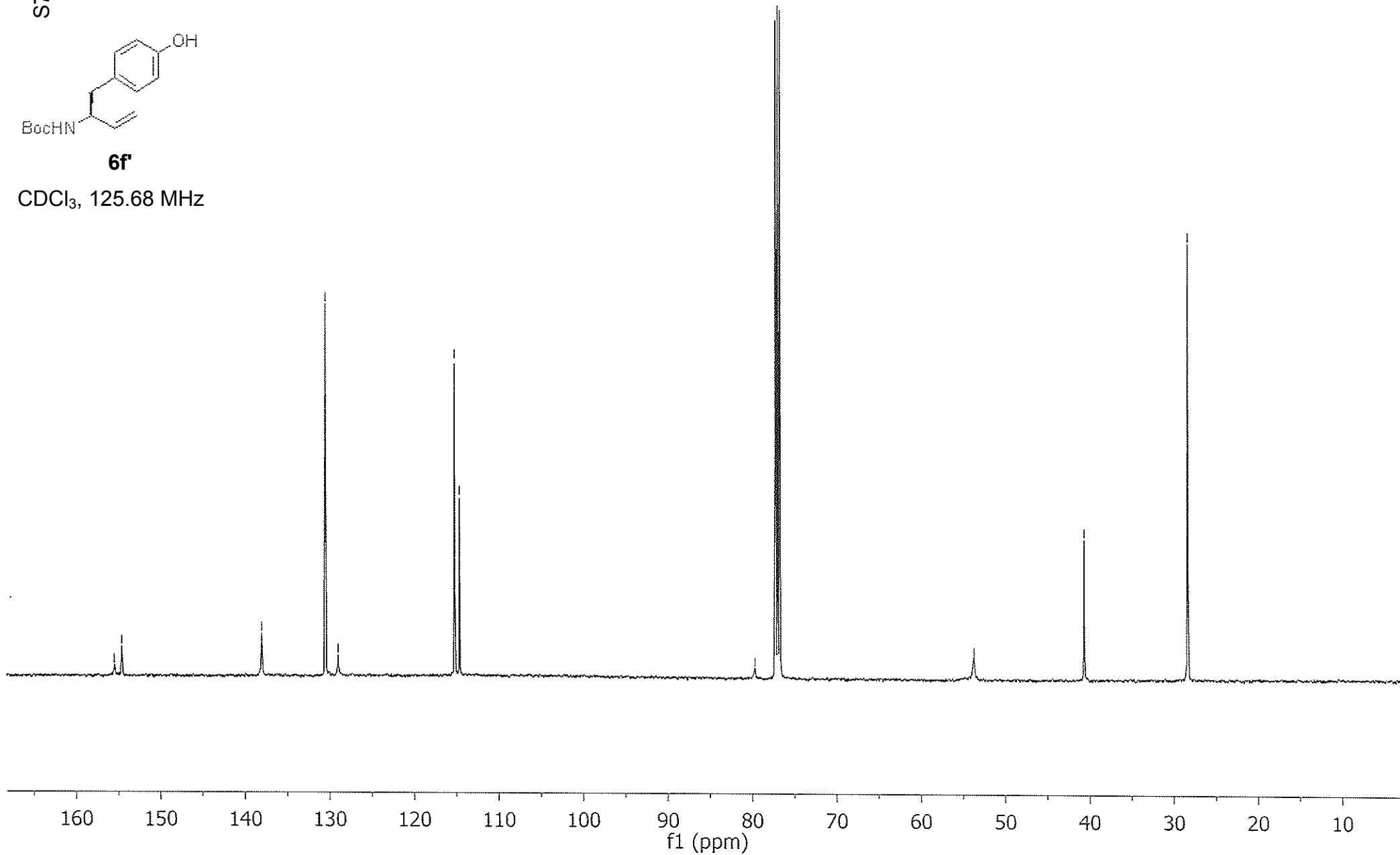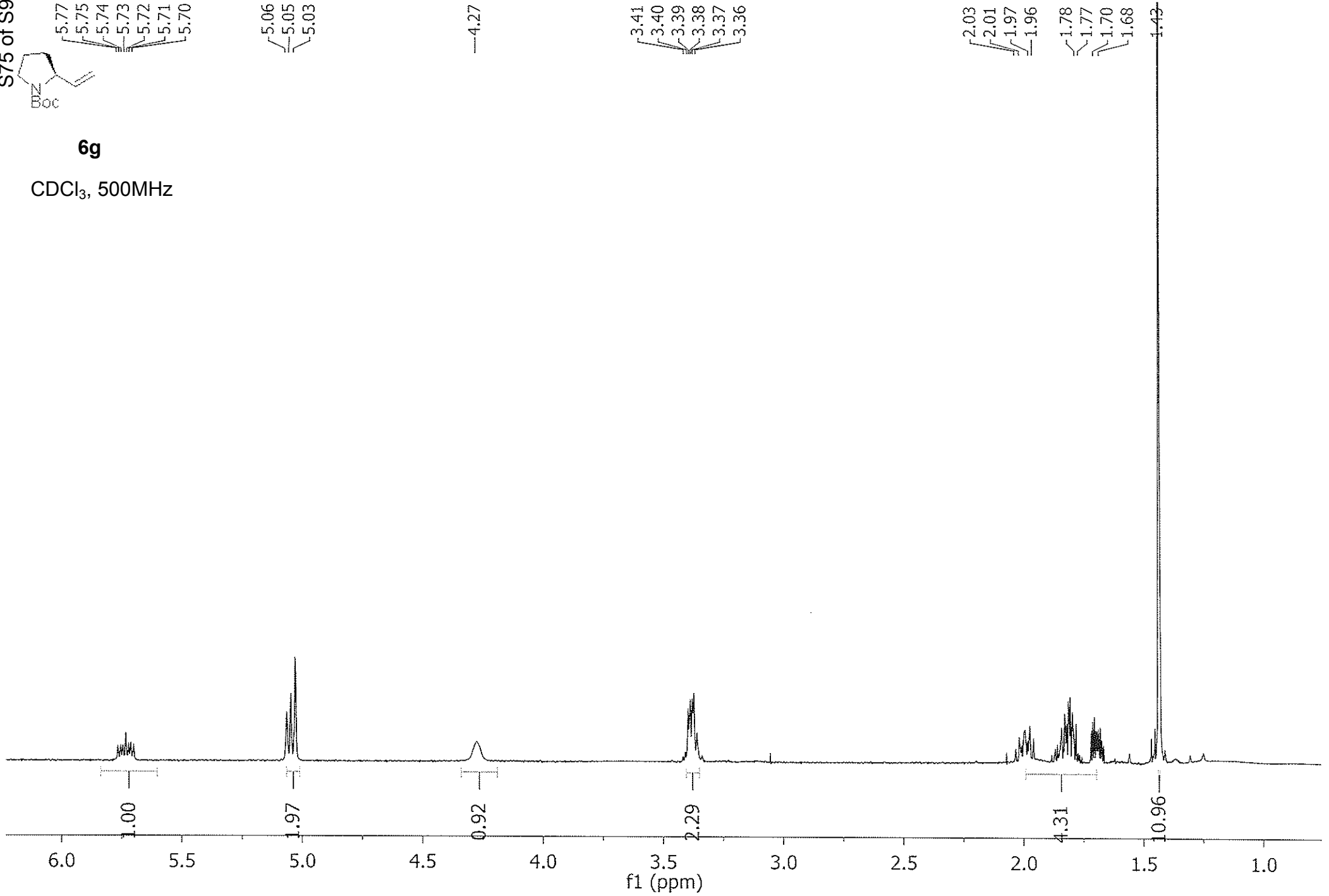
f1 (ppm)

5.77
5.75
5.74
5.73
5.72
5.71
5.70

5.06
5.05
5.03

4.27

3.41
3.40
3.39
3.38
3.37
3.36

2.03
2.01
1.97
1.96

1.78
1.77
1.70
1.68

1.43

6g

CDCl$_3$, 500MHz

1.00
1.97
0.92
2.29
4.31
10.96

f1 (ppm)

6g

CDCl₃, 125.68 MHz

7.29, 7.28, 7.26, 7.26, 7.22, 7.21, 7.20, 7.19, 7.18, 7.16

5.51, 5.50, 5.49, 5.41, 5.41, 5.40, 5.39, 5.38, 5.37, 5.24, 5.22, 5.20

4.61, 4.43, 4.33

2.90, 2.81, 2.80, 2.73, 2.72, 2.71, 2.69

1.65, 1.64, 1.62, 1.49, 1.48, 1.47, 1.47, 1.42, 1.40

6h

CDCl$_3$, 500MHz

4.28 1.97 2.07 2.00 3.42 7.40

f1 (ppm)

155.14
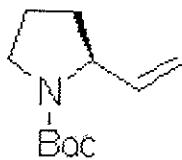
130.88
129.70
129.58
128.20
128.17
126.28
126.15
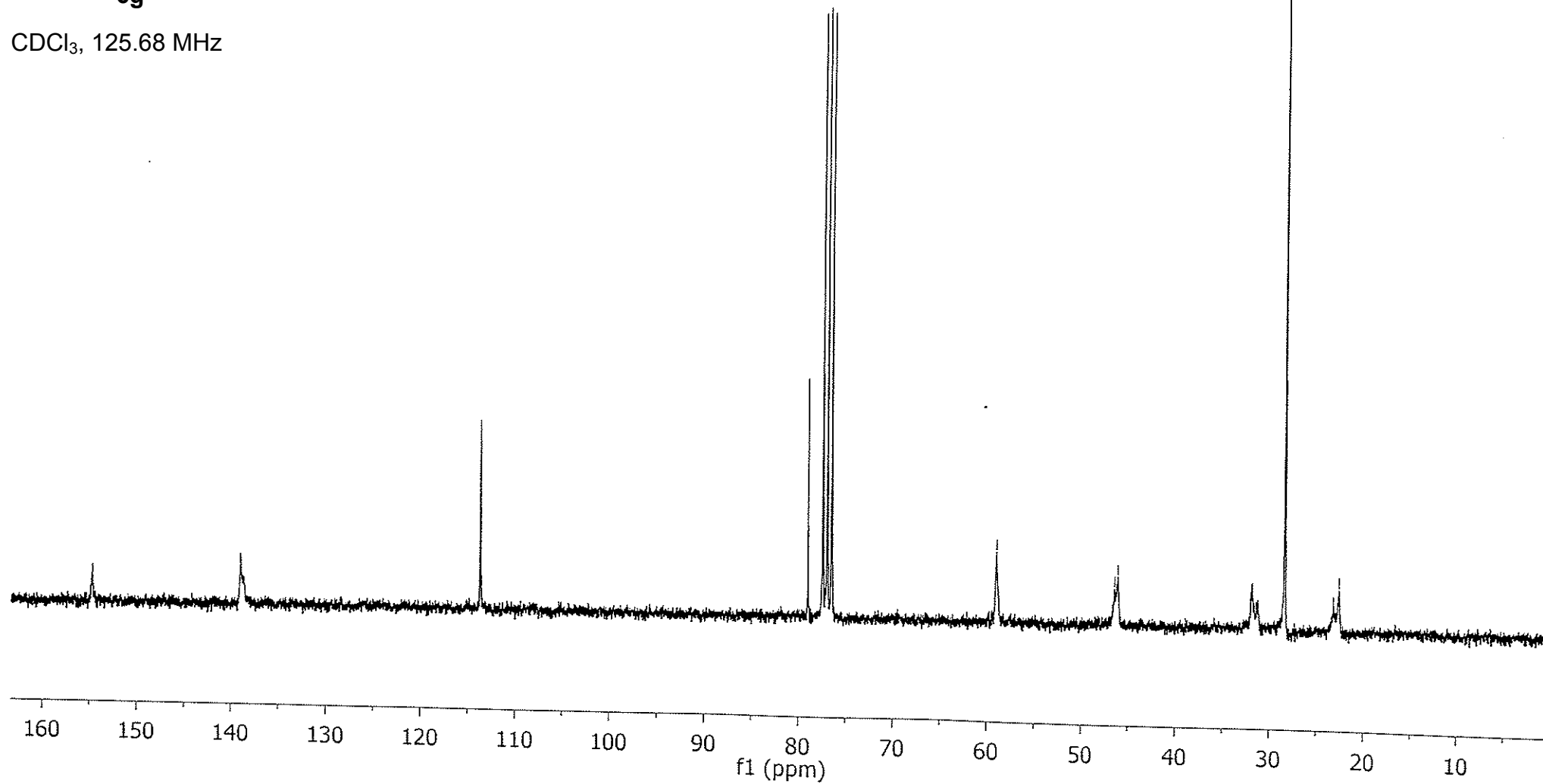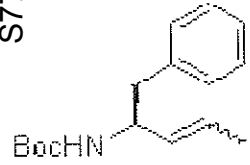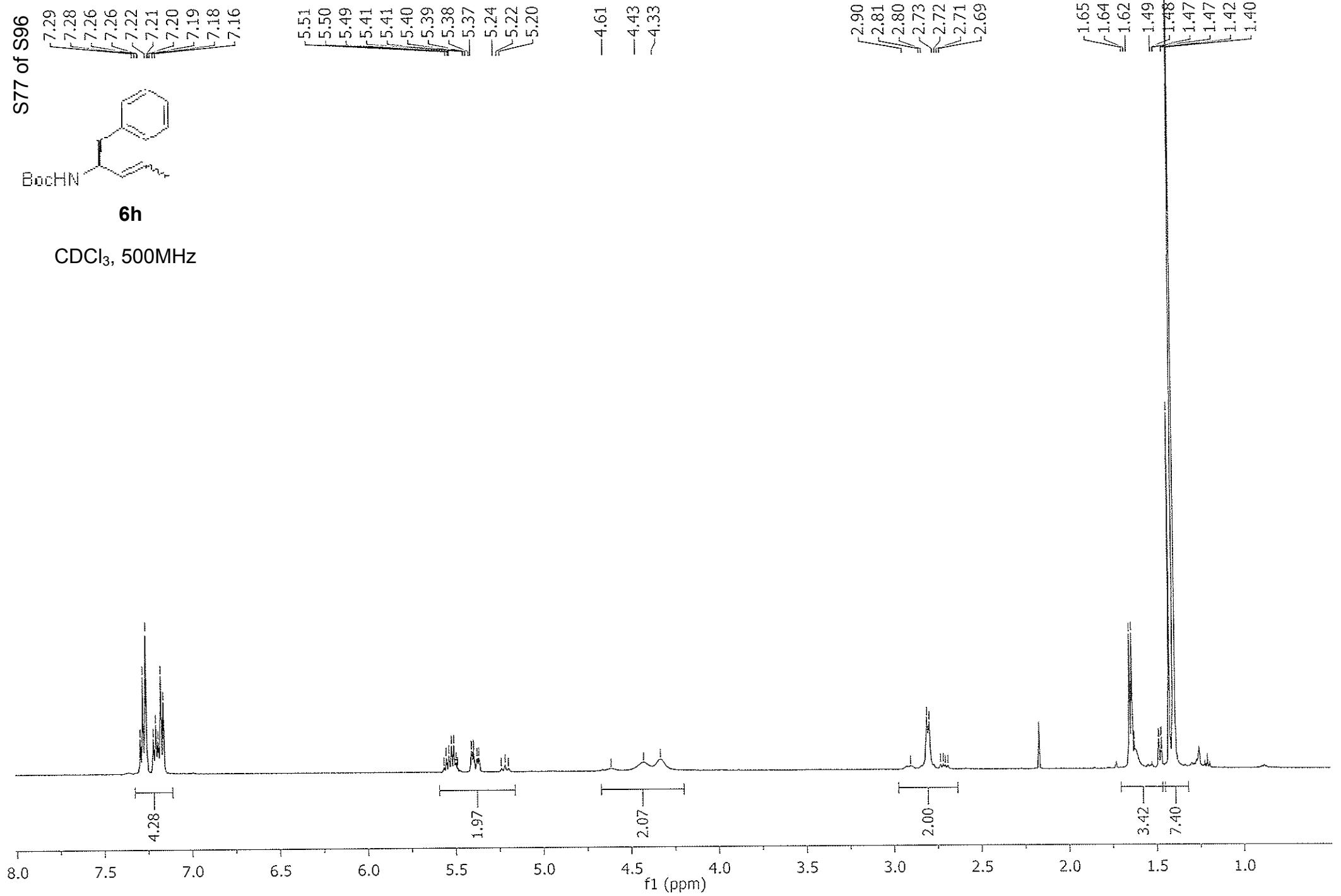
79.25
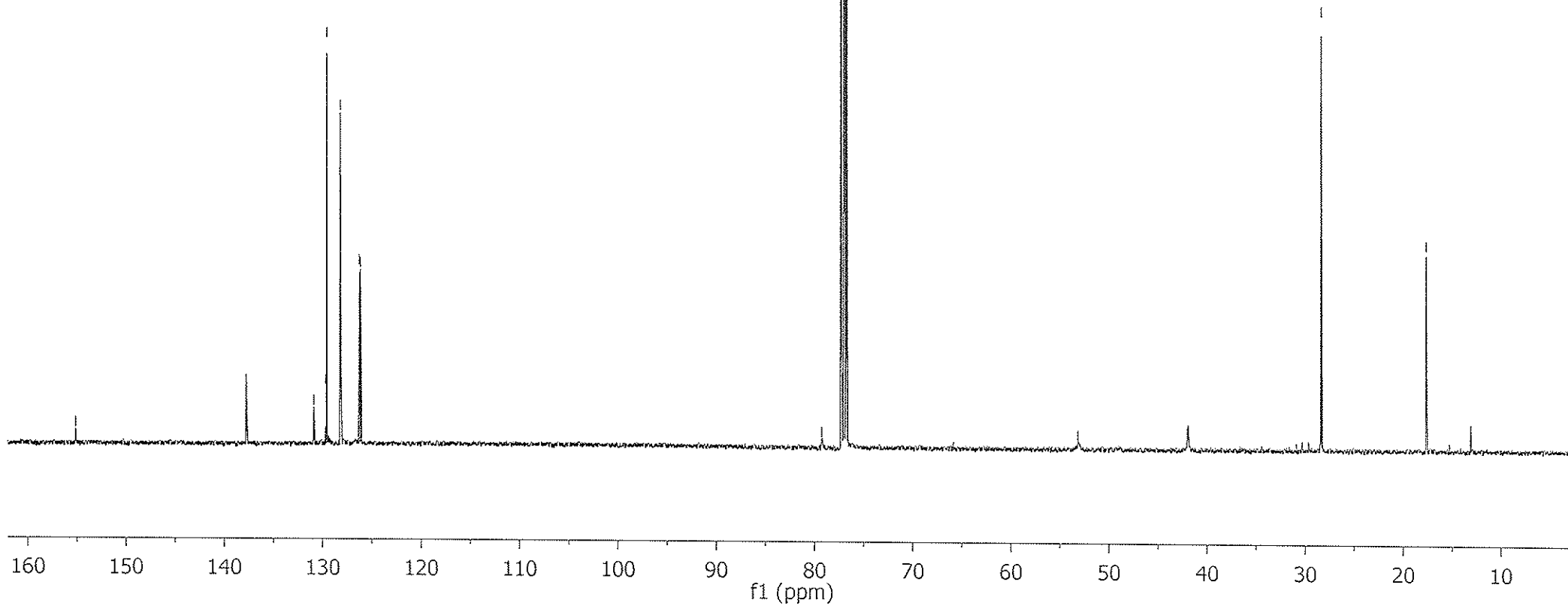
53.18

42.03
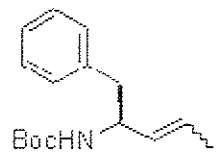
28.40
28.36

17.65
13.09

6h

CDCl$_3$, 125.68 MHz

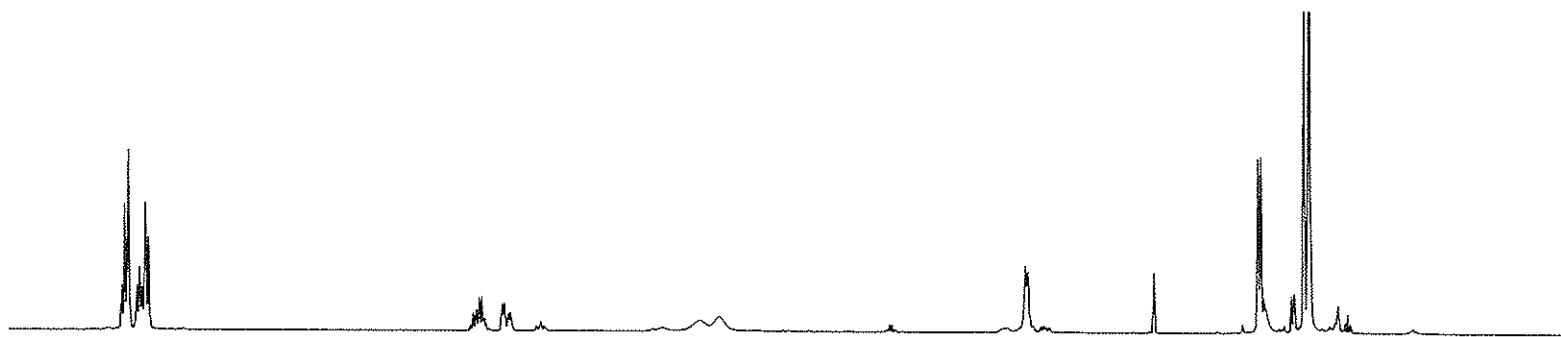160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

f1 (ppm)

6h
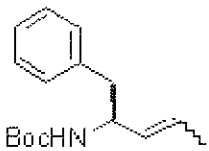
CDCl$_3$, 500MHz
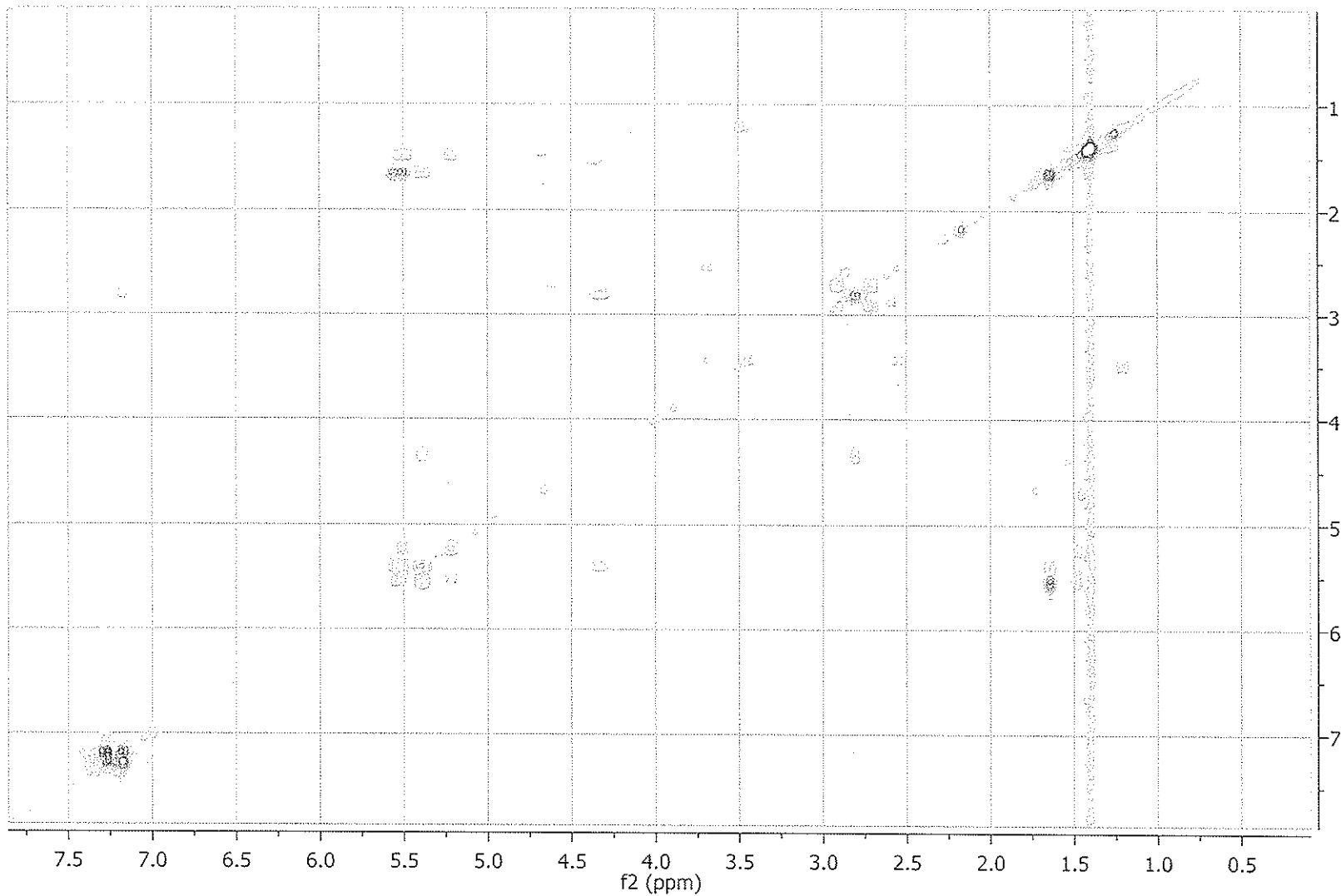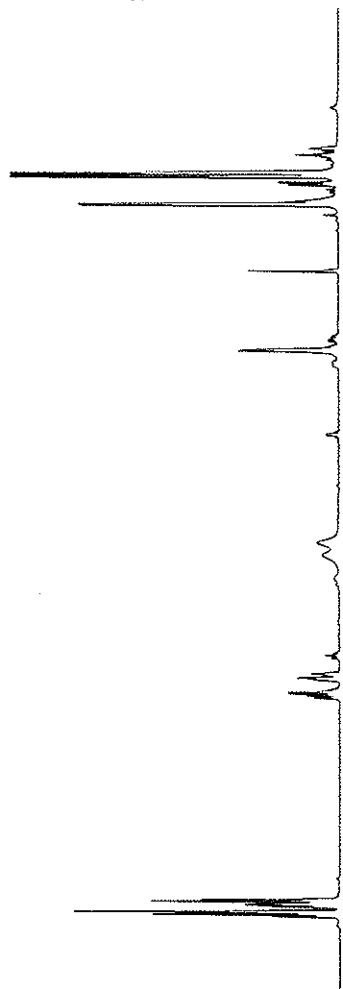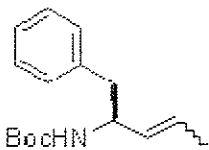
6h

CDCl₃

6i

d6-DMSO, 80°C

H2O

DMSO

6i
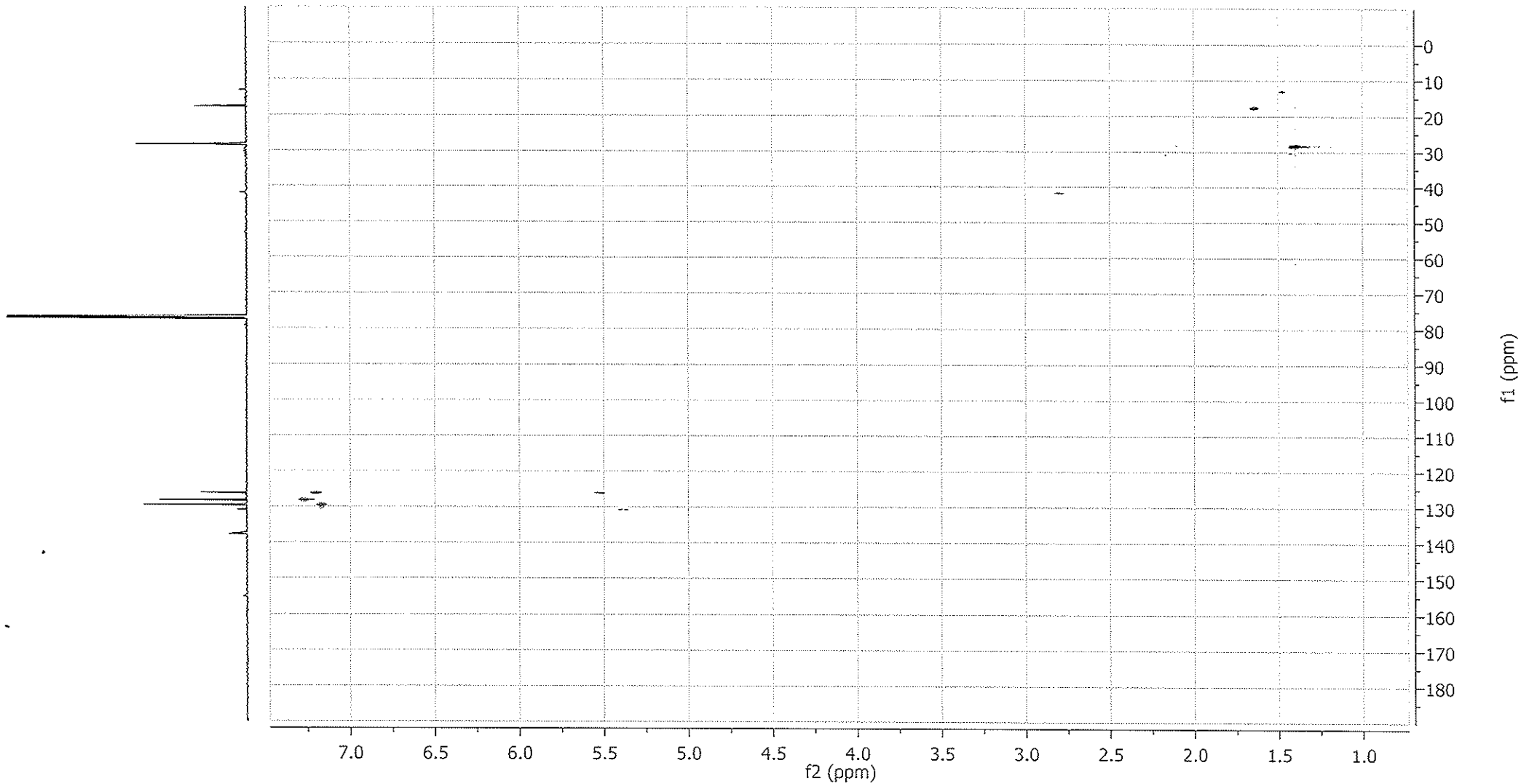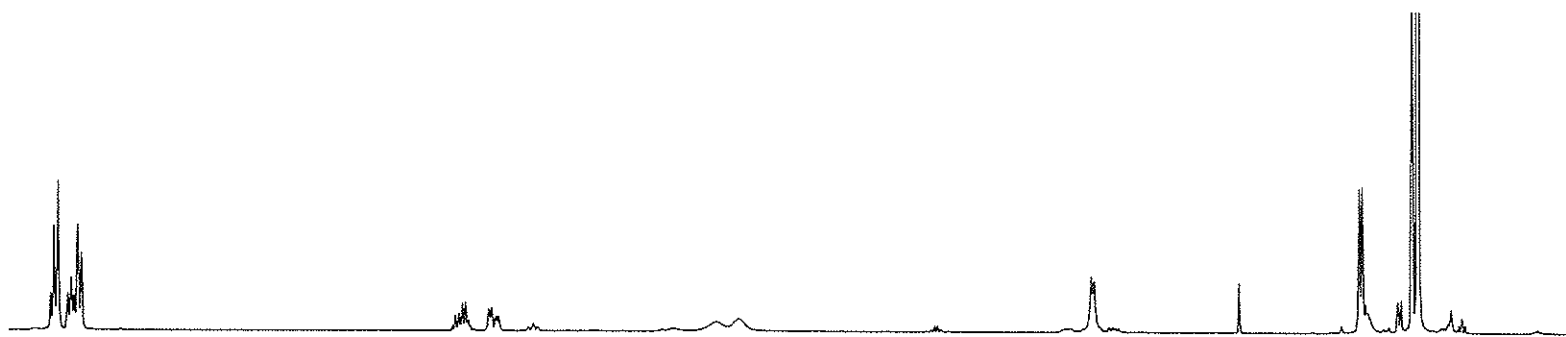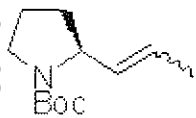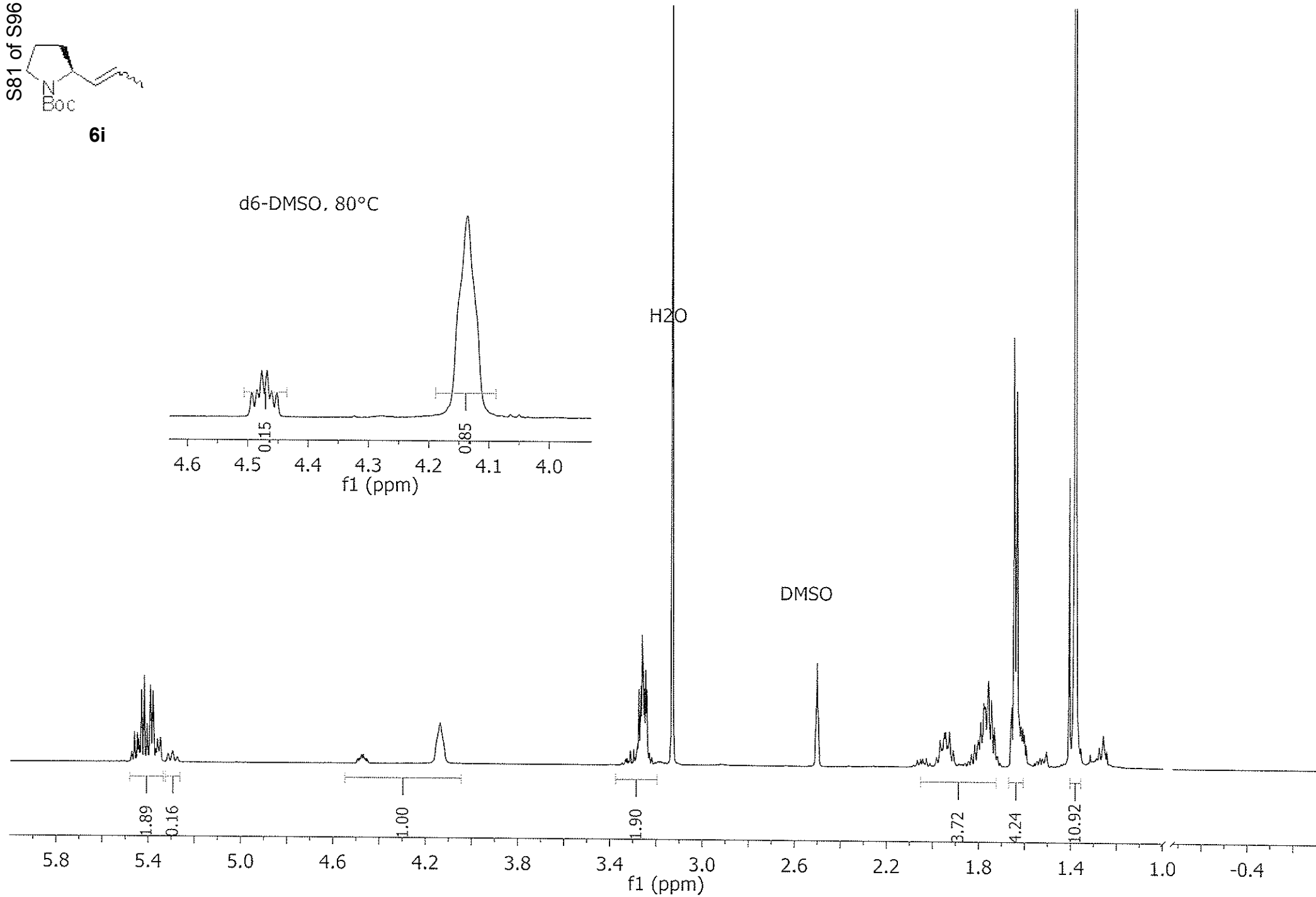
d6-DMSO, 80 °C, 125.68 MHz

153.16
132.54
131.87
123.59
77.59
77.53
57.65
56.15
53.26
45.62
45.51
31.26
27.83
22.34
18.38
16.61
13.41
12.17

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

6i

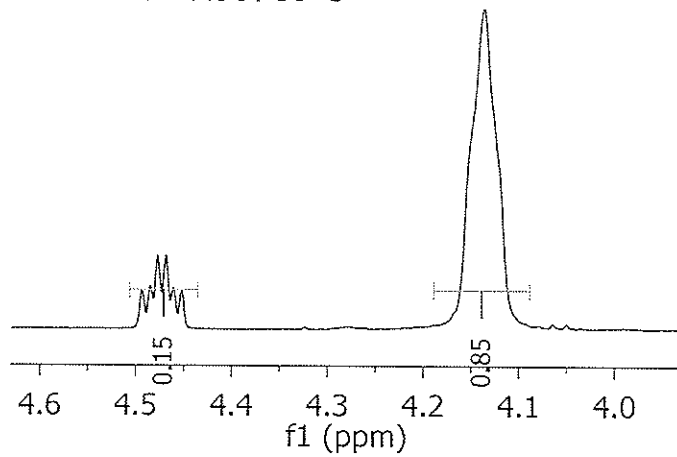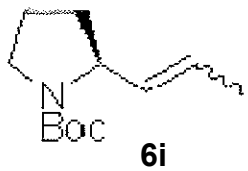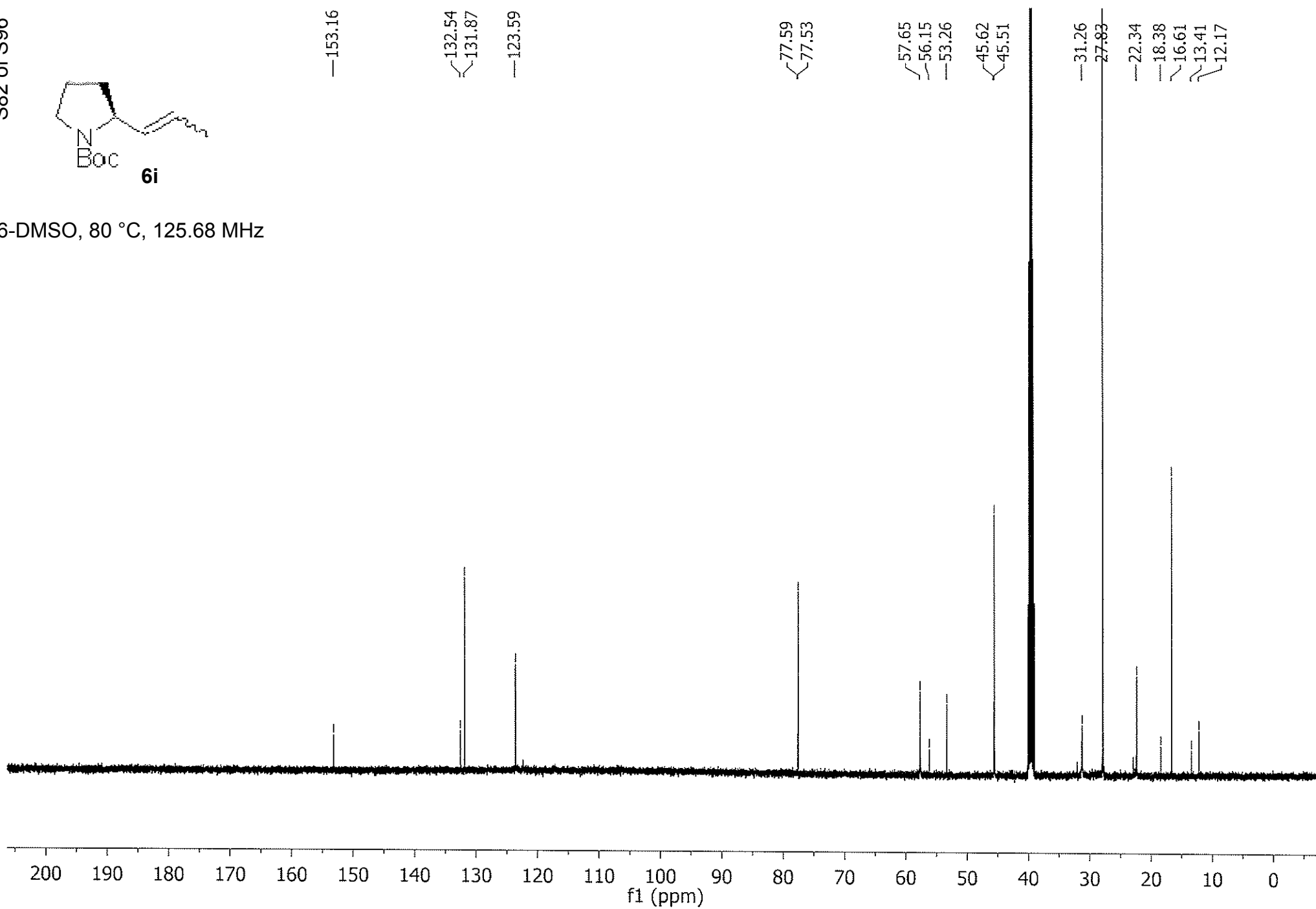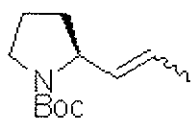d6-DMSO, 80 °C, 125.68 MHz
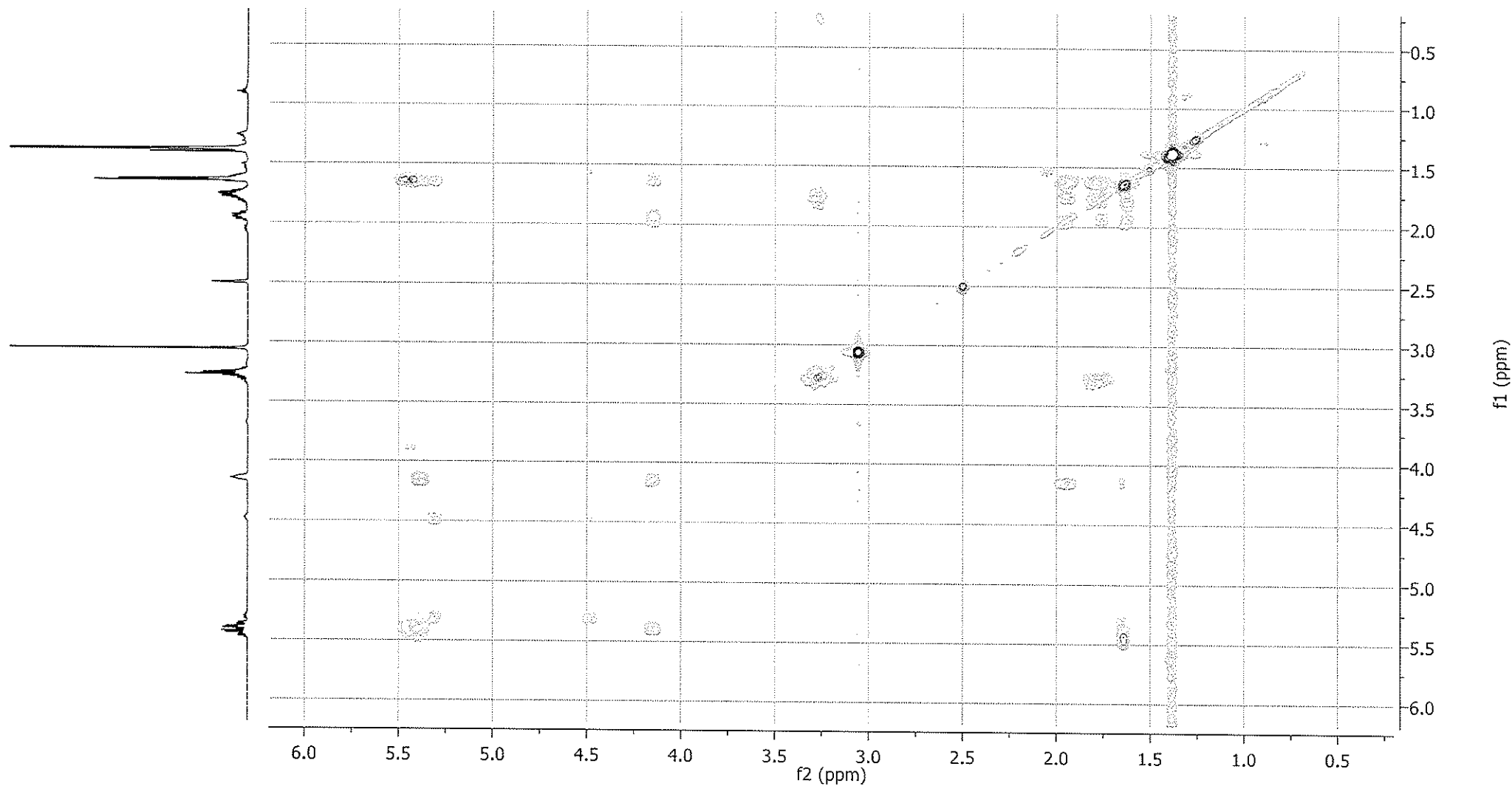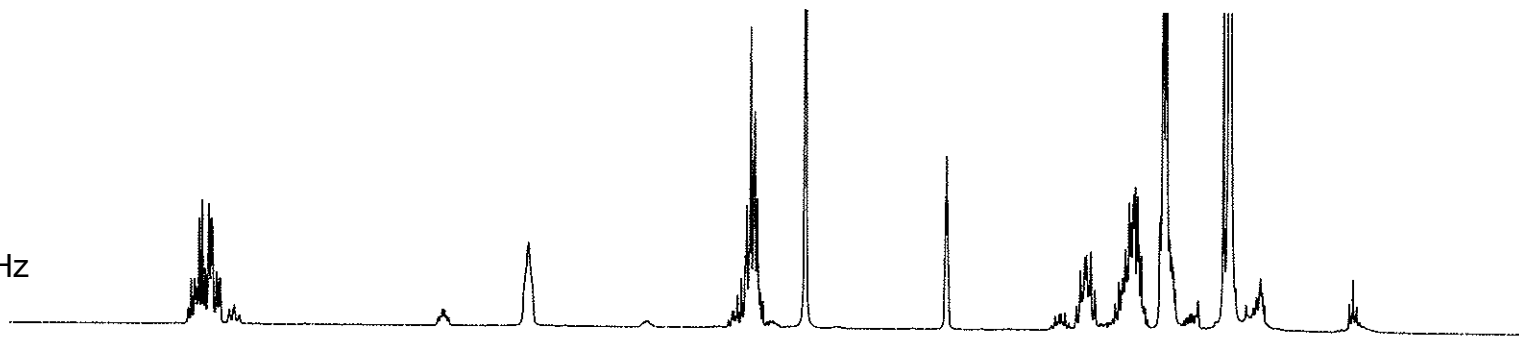
BocAllylAla-Racemo
g-cdx
90°C x 5' --> 1°C/min --> 150°C
File   : c:\class-vp\chrom\lixa2
Method   : c:\class-vp\methods\Fulvia2.met
Sample ID   : LIX162-BocAllylAla
Acquired   : May 22, 2012 04:02:06
Printed   : Jul 31, 2012 01:50:24

BocHN

rac-6a

c:\class-vp\chrom\lixa2 -- Channel A

c:\class-vp\chrom\lixa2 -- Channel A
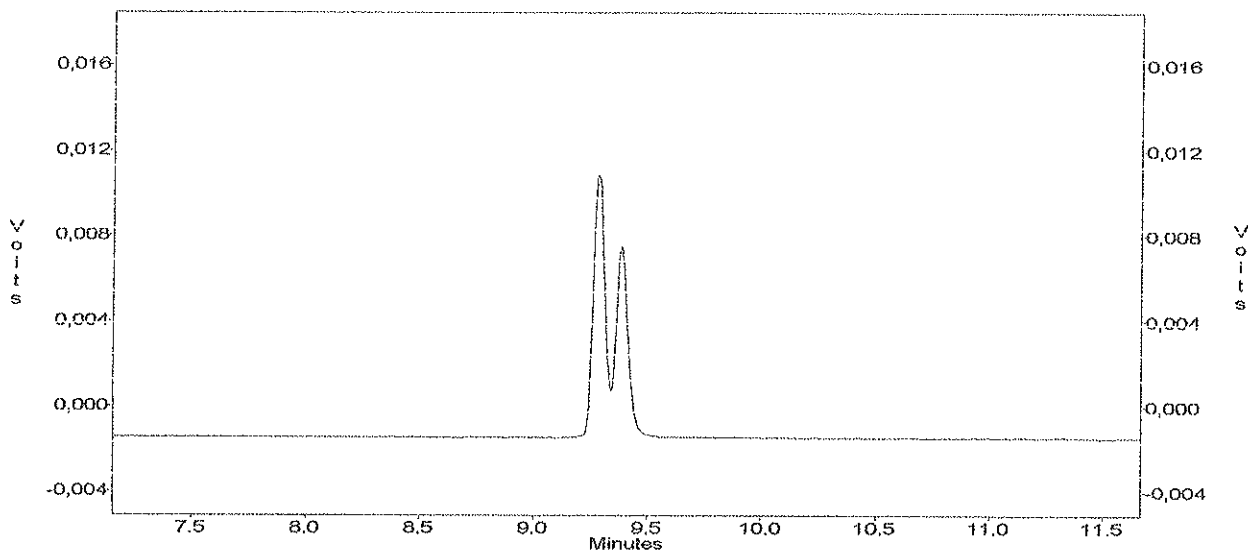
Channel A Results

Peak	Time	Area	Area %
1	9,28	41662	57,271
2	9,38	31083	42,729

Totals :

72745   100,000

BocHN (S)-6a

BocAllylAla
g-cdx
90°C x 5' --> 1°C/min --> 150°C
File   : c:\class-vp\chrom\lixa8
Method   : c:\class-vp\methods\Fulvia2.met
Sample ID : LIX142
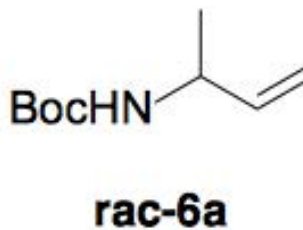Acquired : May 23, 2012 04:11:49
Printed   : Jul 31, 2012 01:45:06

c:\class-vp\chrom\lixa8 -- Channel A

c:\class-vp\chrom\lixa8 -- Channel A
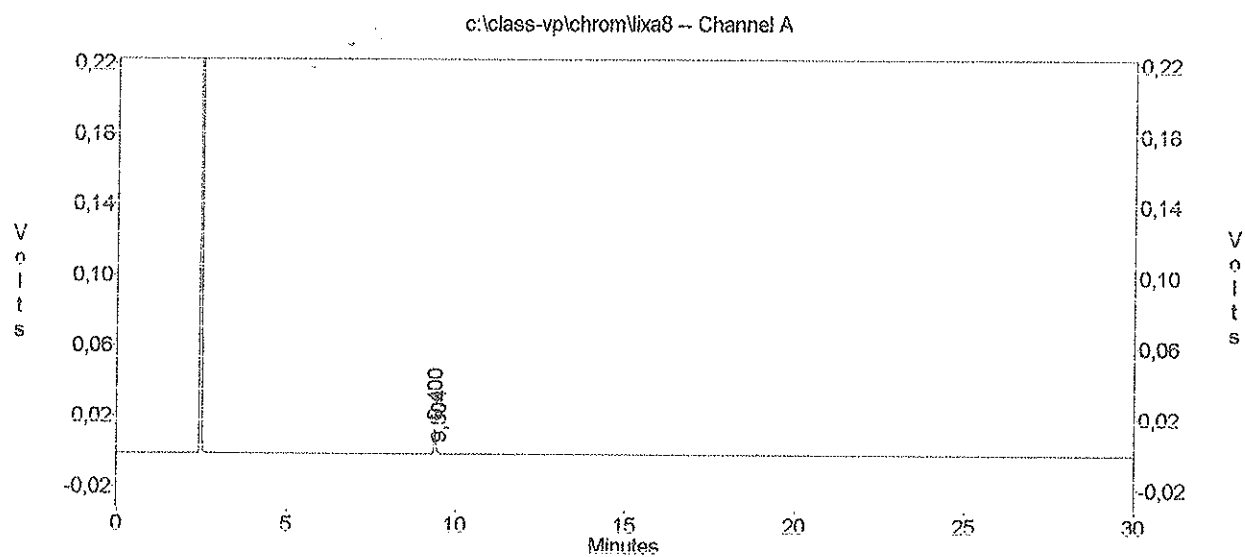
Channel A Results

Peak	Time	Area	Area %
1	9,40	44491	96,778
2	9,50	1481	3,222

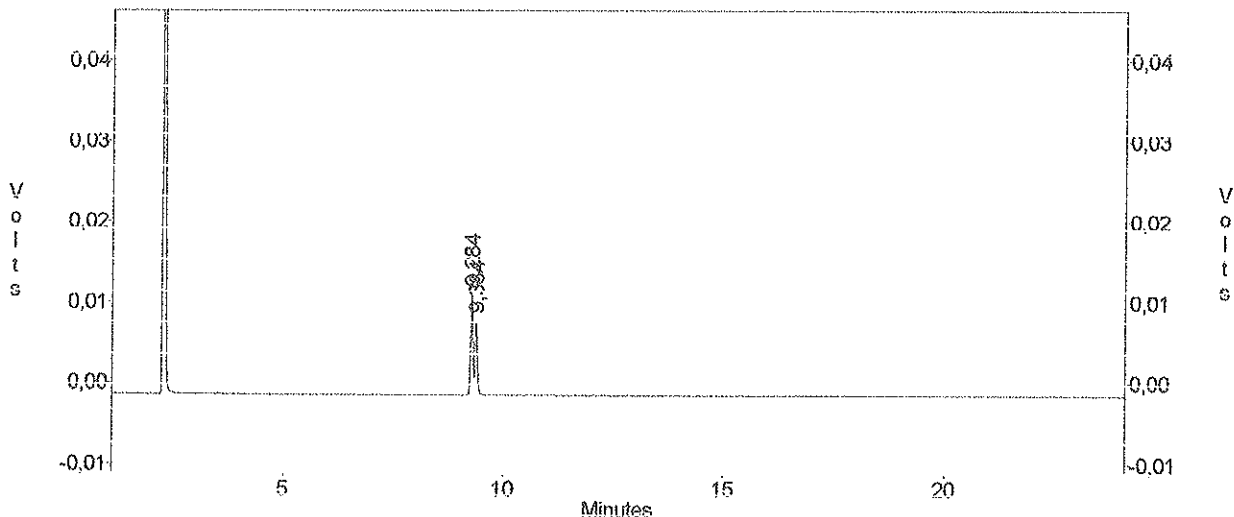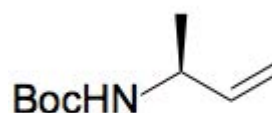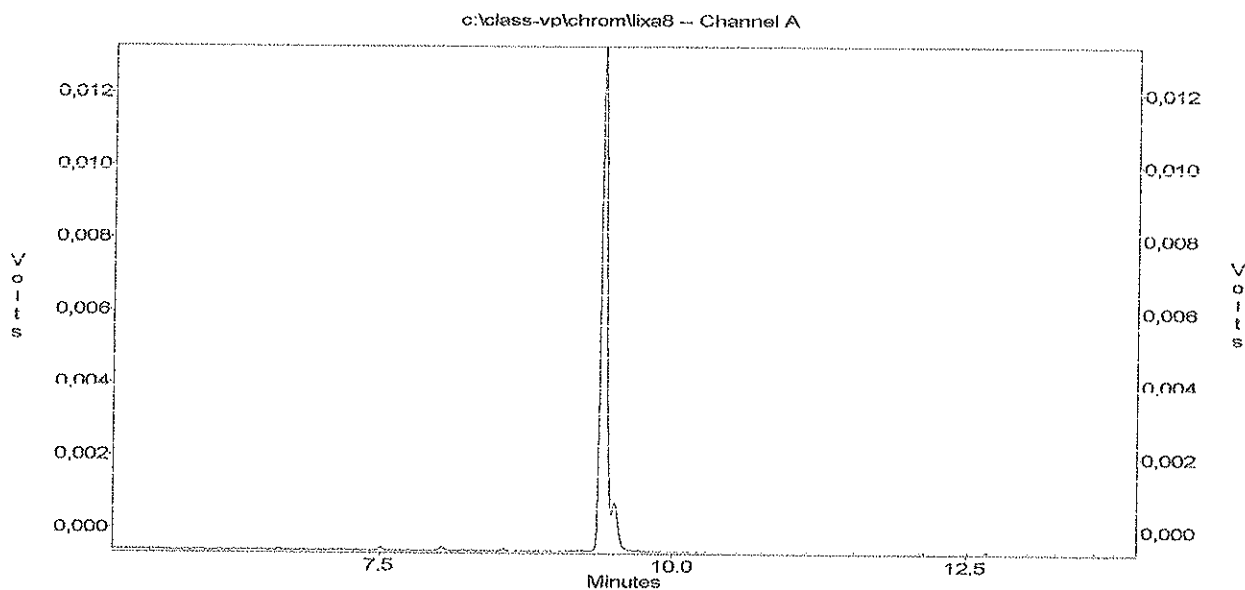Totals :

| | | 45972 | 100,000 |

DL-BocAllylVal

β s-cdx 100°C x 5 min, 1°C/min -> 150°C

File : c:\class-vp\chrom\Mg11
Method : c:\class-vp\methods\Fulvia2.met
Sample ID : DL-BocAllylVal
Acquired : Jan 04, 1980 21:05:25
Printed : Jan 04, 1980 21:38:54

rac-6b

c:\class-vp\chrom\Mg11 -- Channel B
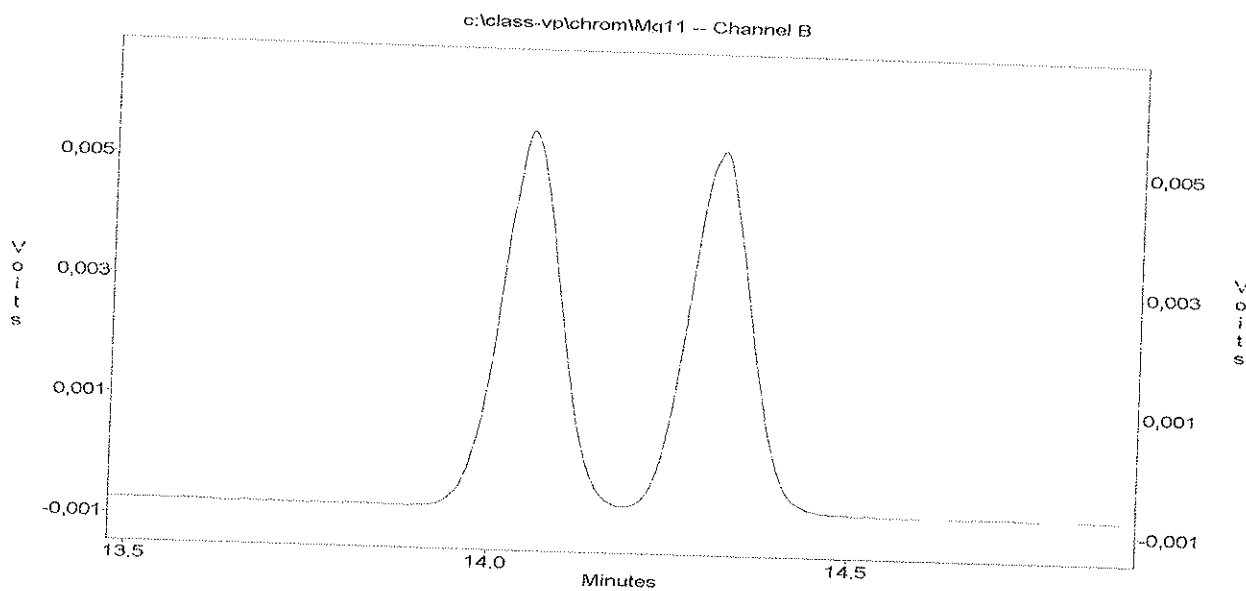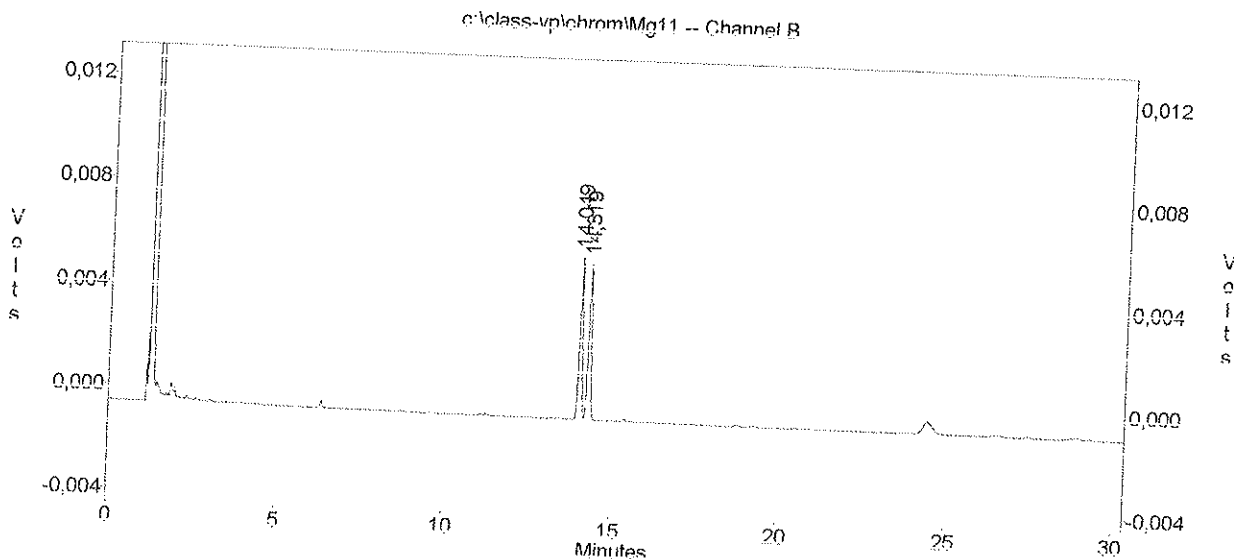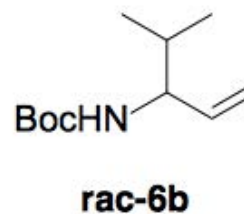
c:\class-vp\chrom\Mg11 -- Channel B

Channel B Results

Peak	Time	Area	Area %
1	14,05	35472	50,097
2	14,32	35334	49,903

Totals :

| | 70806 | 100,000 |

L-BocAllylVal

β-cdx 100°C x 5 min, 1°C/min -> 150 °C

File   : c:\class-vp\chrom\Mg12
Method   : c:\class-vp\methods\Fulvia2.met
Sample ID   : L-BocAllylVal
Acquired   : Jan 04, 1980 21:44:26
Printed   : Jan 04, 1980 22:31:05

BocHN

(S)-6b

c:\class-vp\chrom\Mg12 -- Channel B

c:\class-vp\chrom\Mg12 -- Channel B

Channel B Results

Peak	Time	Area	Area %
1	14,10	129031	99,847
2	14,36	198	0,153

Totals :

129229   100,000

DL-BocAllylLeu

β▪-cdx 100°C x 5 min, 1°C/min -> 150°C

File : c:\class-vp\chrom\Mg13
Method : c:\class-vp\methods\Fulvia2.met
Sample ID : DL-BocAllylLeu
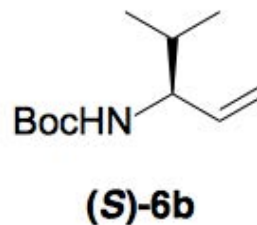Acquired : Jan 04, 1980 22:33:48
Printed : Jan 04, 1980 22:59:46

rac-6c

c:\class-vp\chrom\Mg13 -- Channel B

c:\class-vp\chrom\Mg13 -- Channel B

Channel B Results

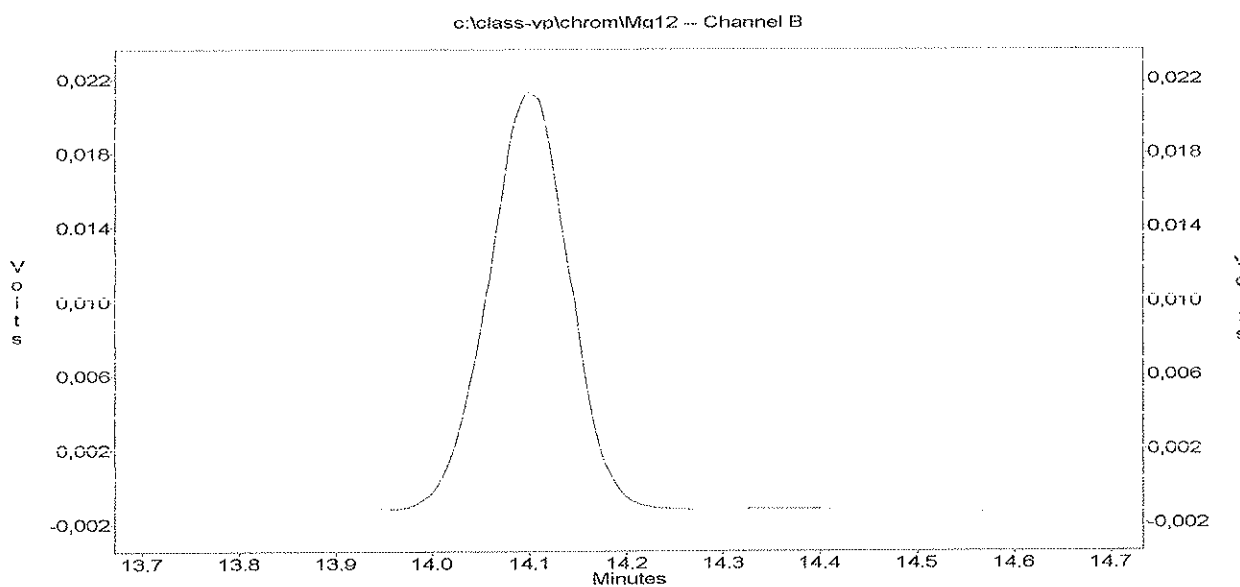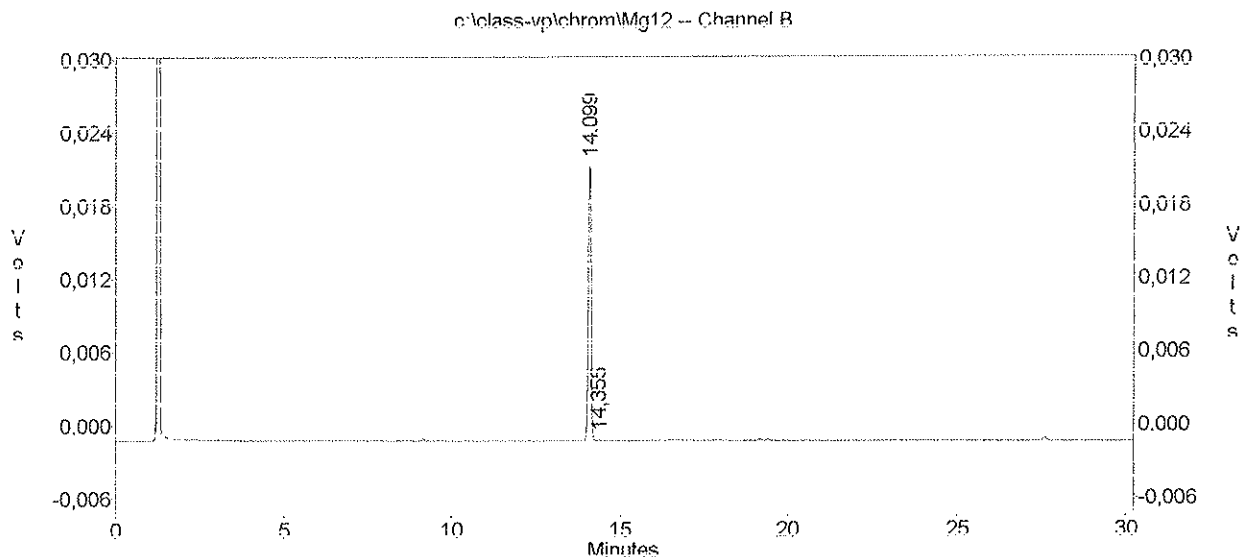| Peak	Time	Area	Area %
1	17,60	354	0,059
2	17,87	466	0,078
3	18,93	296546	49,821
4	19,39	297851	50,041

Continued...

L-BocAllylLeu

βg-cdx 100°C x 5 min, 1°C/min -> 150°C

File   : c:\class-vp\chrom\Mg14
Method   : c:\class-vp\methods\Fulvia2.met
Sample ID   : L-BocAllylLeu
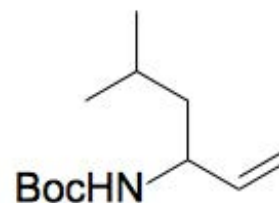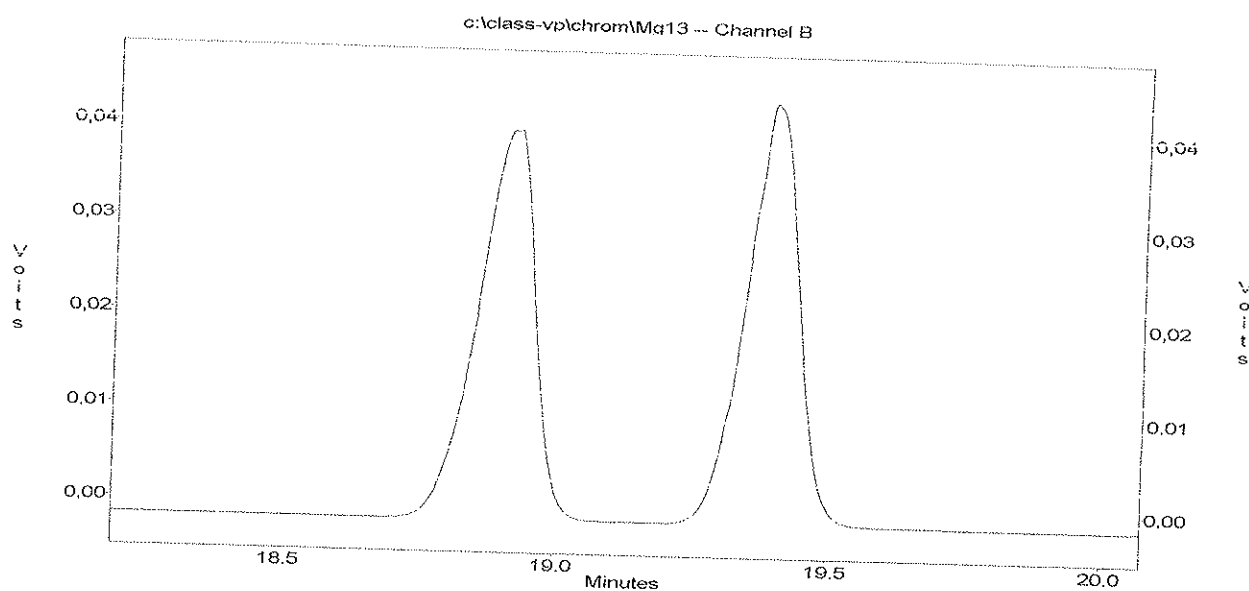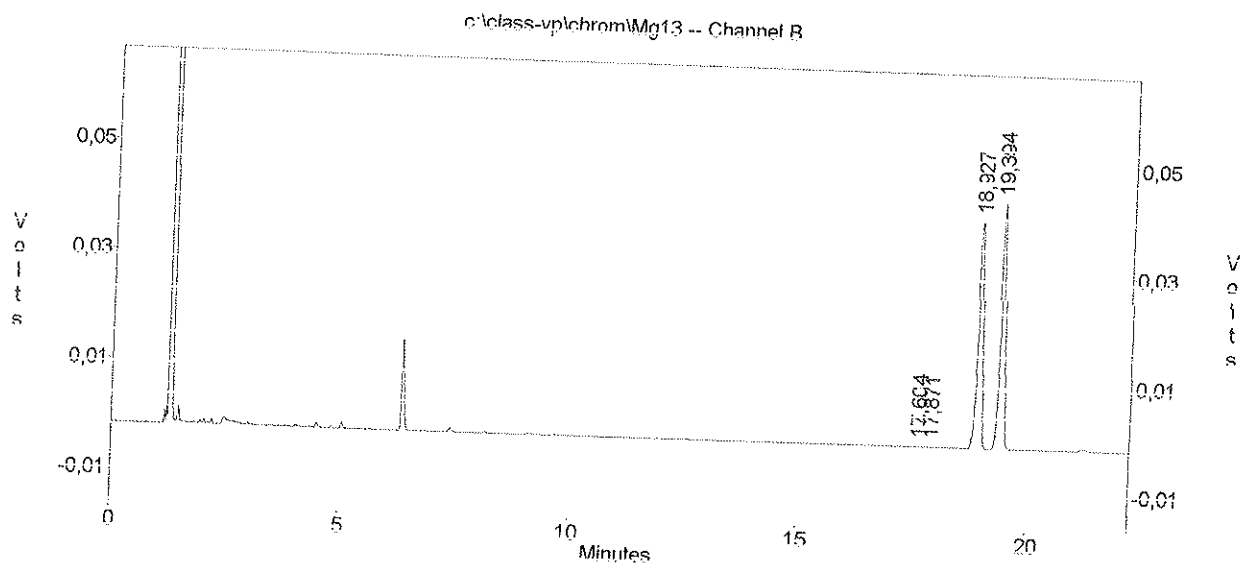Acquired   : Jan 04, 1980 23:04:34
Printed   : Jan 05, 1980 00:32:41

(S)-6c
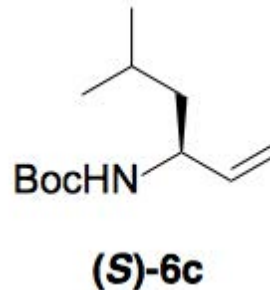
c:\class-vp\chrom\Mg14 -- Channel B

c:\class-vp\chrom\Mg14 -- Channel B

Channel B Results

Peak	Time	Area	Area %
1	18,90	96838	99,620
2	33,25	369	0,380

Totals :

| | | 97207 | 100,000 |

DL-BocAllyllic

β 4-cdx 100°C x 5 min, 1°C/min ->150°C

File	:	c:\class-vp\chrom\Mg9
Method	:	c:\class-vp\methods\Fulvia2.met
Sample ID	:	
Acquired	:	Jan 04, 1980 20:02:01
Printed	:	Jan 04, 1980 20:32:02

rac-6d

c:\class-vp\chrom\Mg9 -- Channel B

c:\class-vp\chrom\Mg9 -- Channel B

Channel B Results

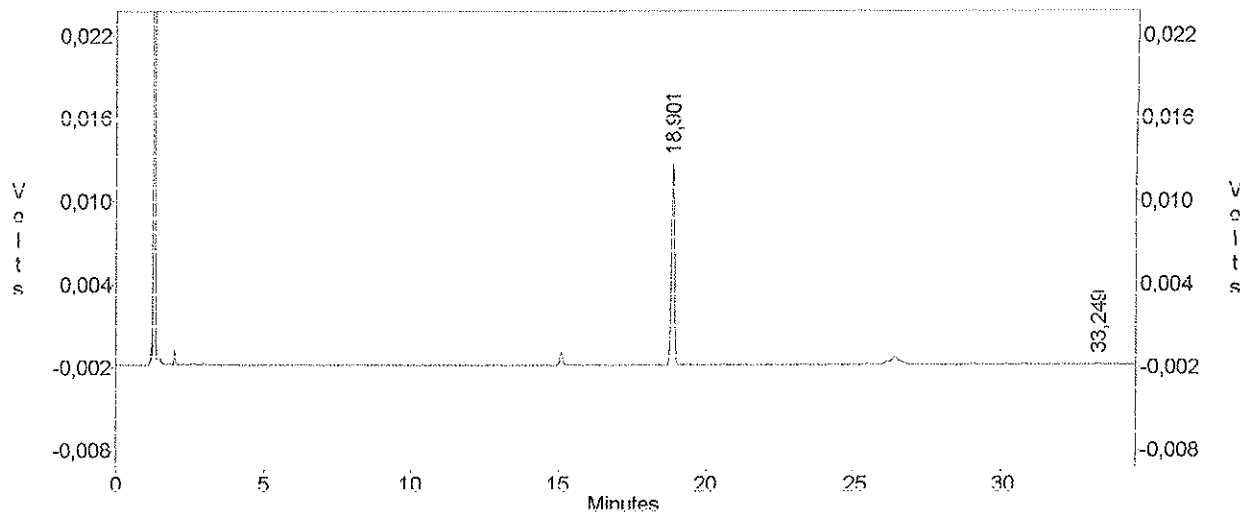| Peak	Time	Area	Area %
1	0,10	419	1,029
2	19,27	19240	47,231
3	20,39	21077	51,740

Totals :

40736   100,000

L-BocAllylIle
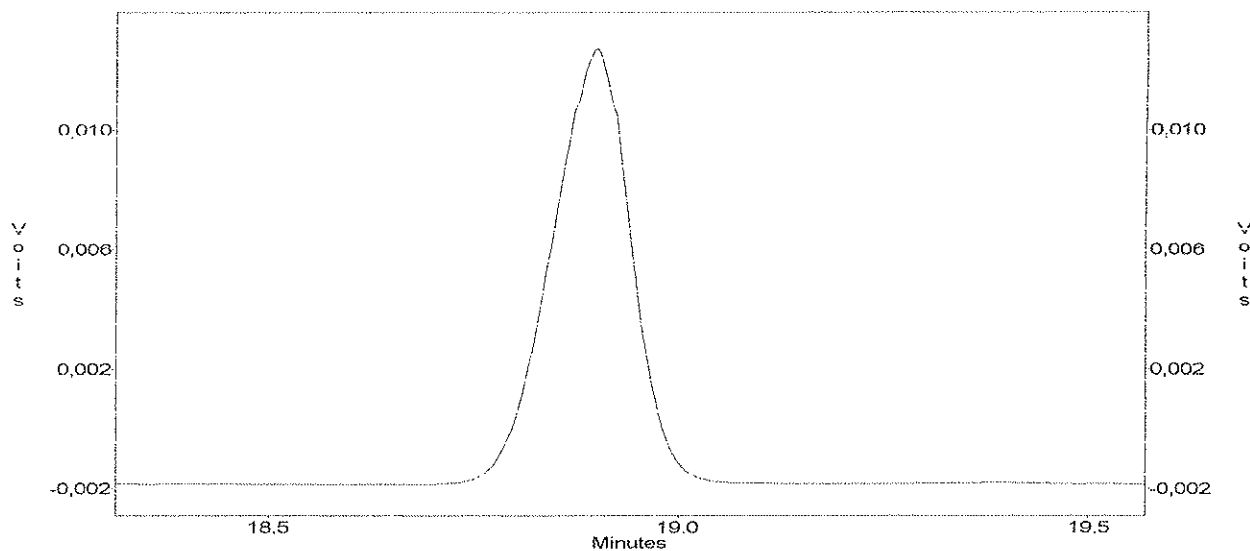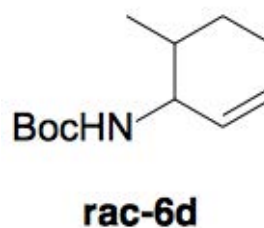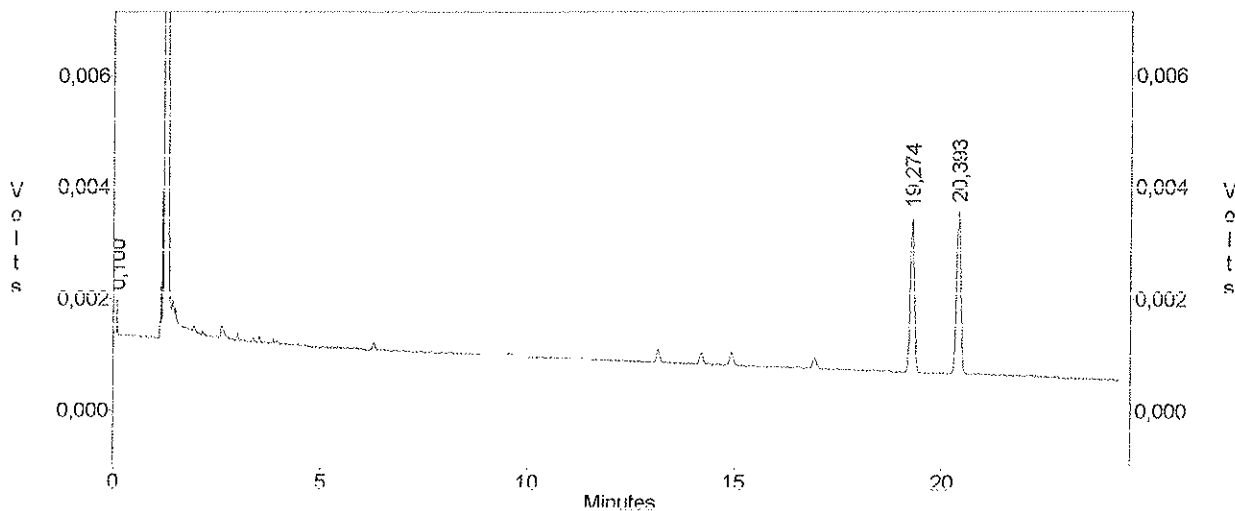
β s-cdx 100°C x 5 min, 1°C/min -> 150°C

File : c:\class-vp\chrom\Mg10
Method : c:\class-vp\methods\Fulvia2.met
Sample ID : L-BocAllylIle
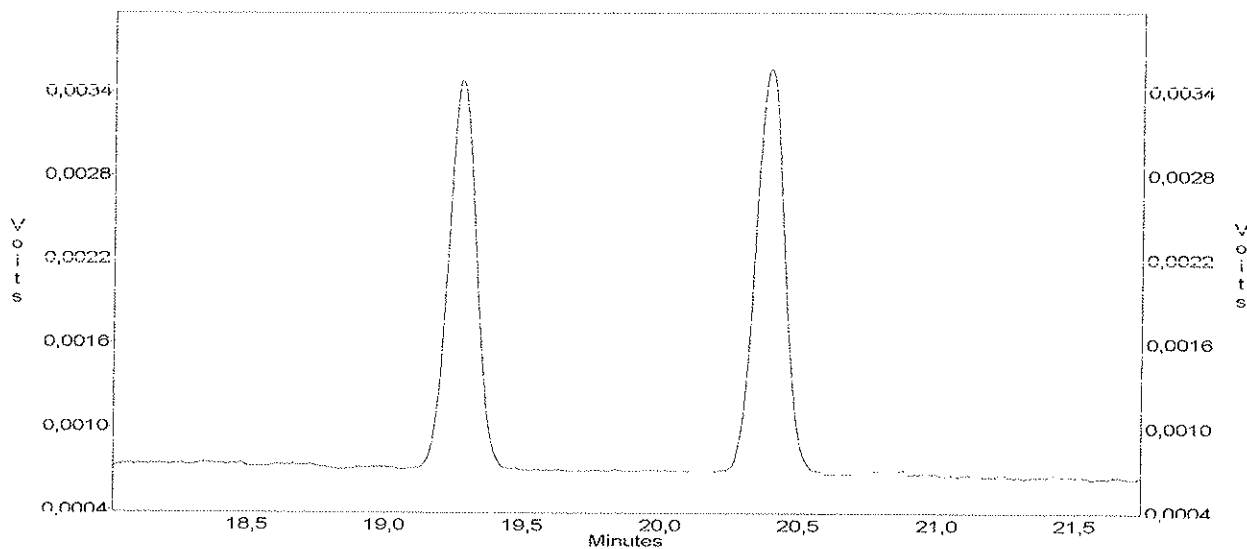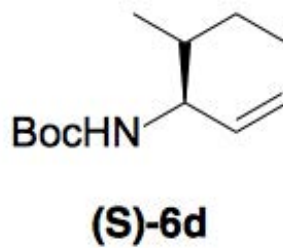Acquired : Jan 04, 1980 20:35:40
Printed : Jan 04, 1980 21:01:16

BocHN

(S)-6d

c:\class-vp\chrom\Mg10 -- Channel B

c:\class-vp\chrom\Mg10 -- Channel B
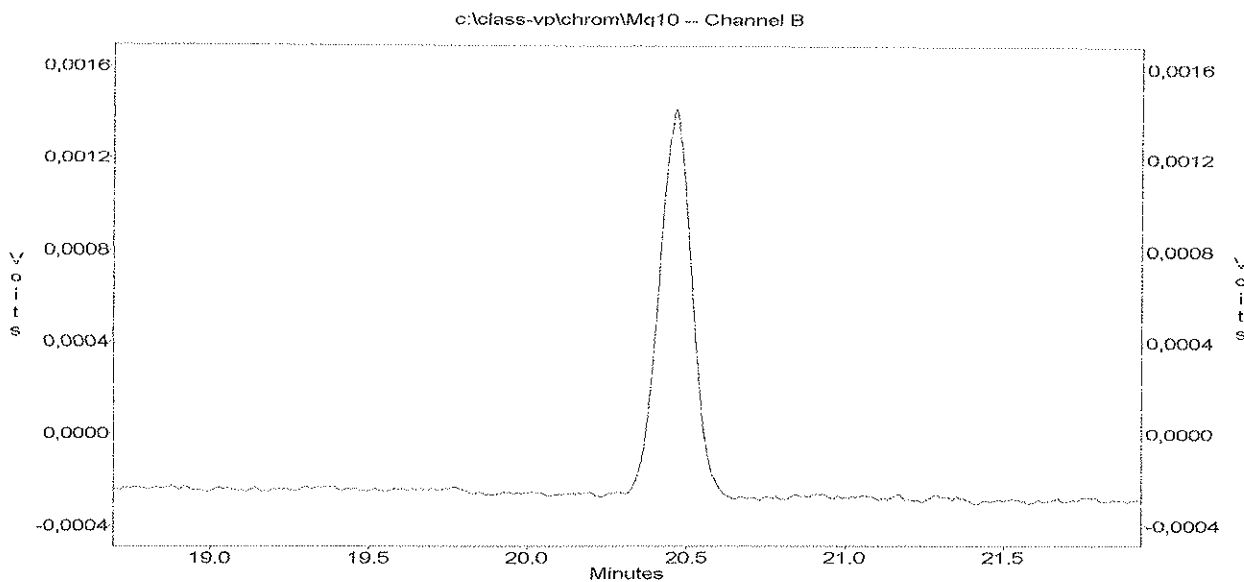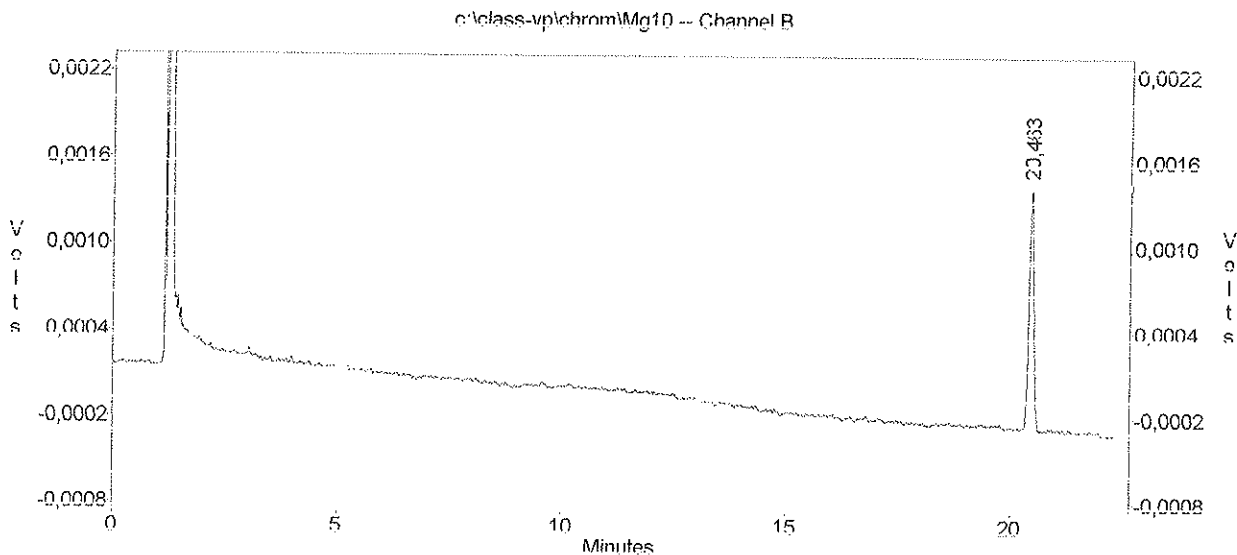
Channel B Results

Peak	Time	Area	Area %
1	20,46	12041	100,000

Totals :

| | | 12041 | 100,000 |

DL-BocAllylPhe
g-cdx isoterma 150°C
File   : c:\class-vp\chrom\Mg7
Method   : c:\class-vp\methods\Fulvia2.met
Sample ID   :

rac-6e

c:\class-vp\chrom\Mg7 -- Channel A

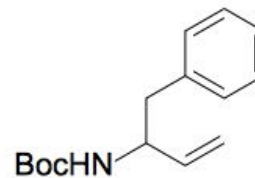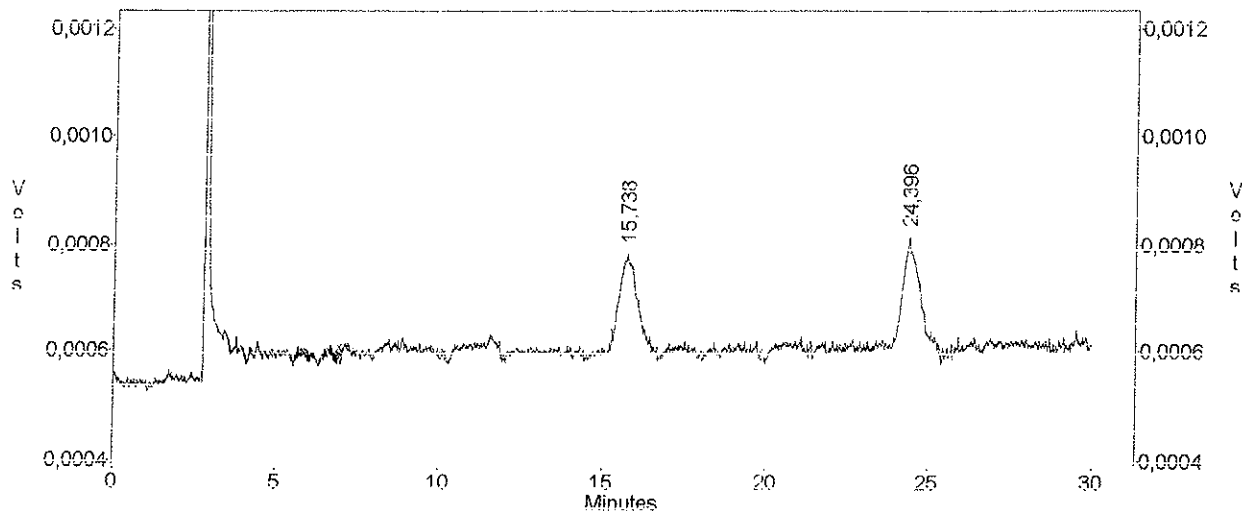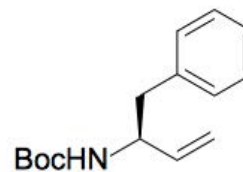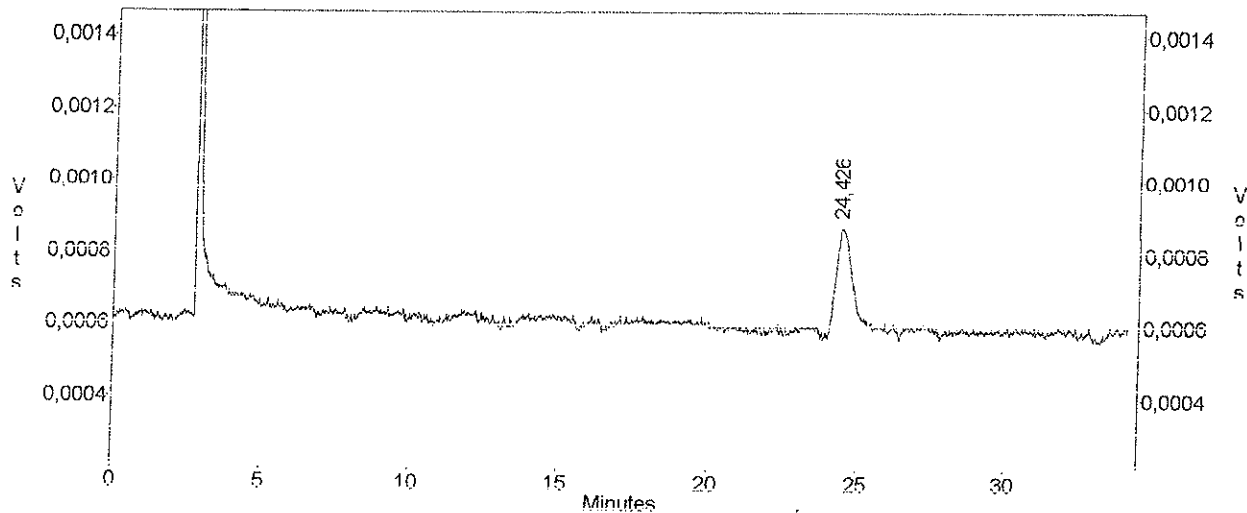**L-BocAllylPhe**
g-cdx isoterma 150°C
File   : c:\class-vp\chrom\Mg8
Method   : c:\class-vp\methods\Fulvia2.met
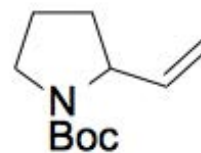Sample ID   : L-AllylPhe

(S)-6e

c:\class-vp\chrom\Mg8 -- Channel A

DL-BocAllylPro
b-cdx isoterma 100°C
File   : c:\class-vp\chrom\Mg5
Method   : c:\class-vp\methods\Fulvia2.met
Sample ID   : DL-BocAllylPro bcd
Acquired   : Aug 09, 2012 09:42:00
Printed   : Aug 09, 2012 10:21:35

rac-6g

c:\class-vp\chrom\Mg5 -- Channel B

c:\class-vp\chrom\Mg5 -- Channel B

Channel B Results

Peak	Time	Area	Area %
1	0,38	107	0,084
2	19,44	66321	52,134
3	19,67	60785	47,782

Totals :

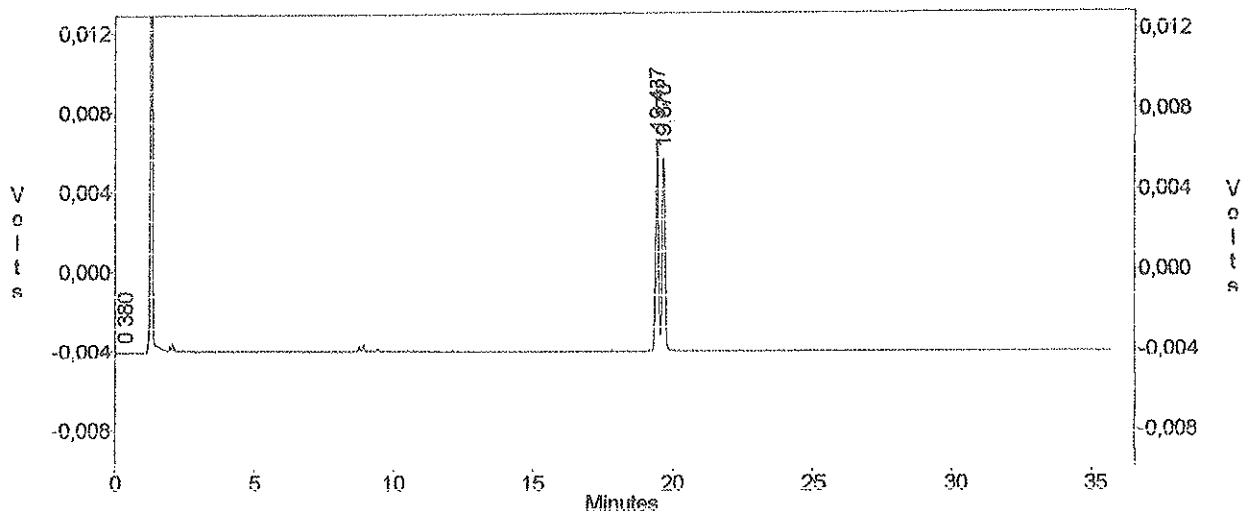|		127213	100,000

L-BocAllylPro

b-cdx isoterma 100°C

File   : c:\class-vp\chrom\Mg6
Method   : c:\class-vp\methods\Fulvia2.met
Sample ID   : L-BocAllylPro
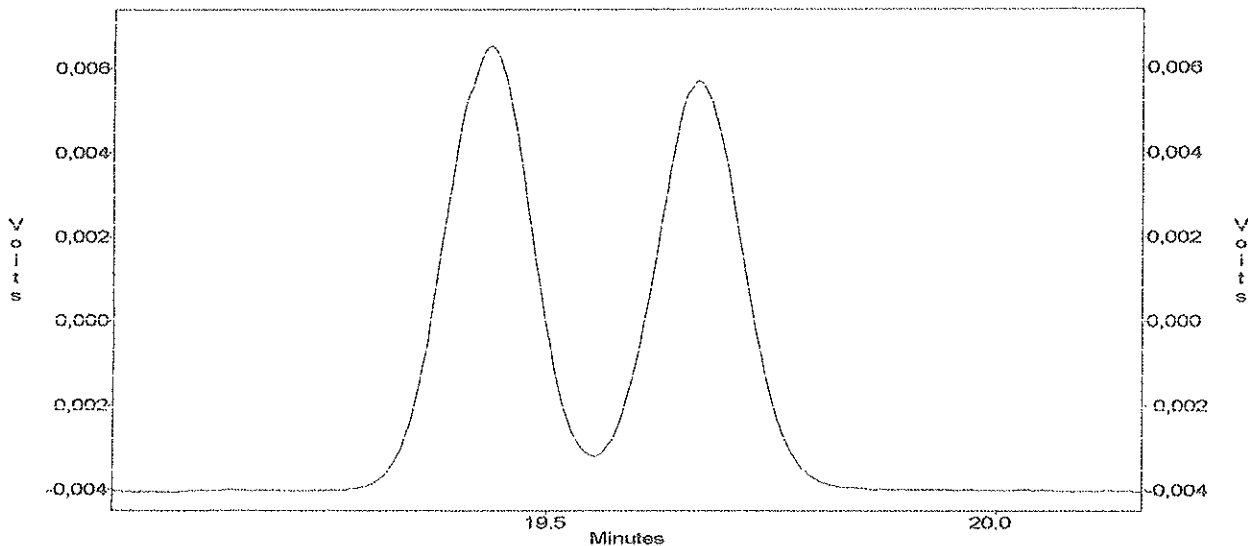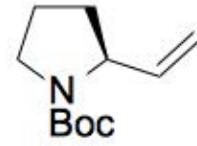Acquired   : Aug 09, 2012 10:25:15
Printed   : Aug 09, 2012 10:58:15
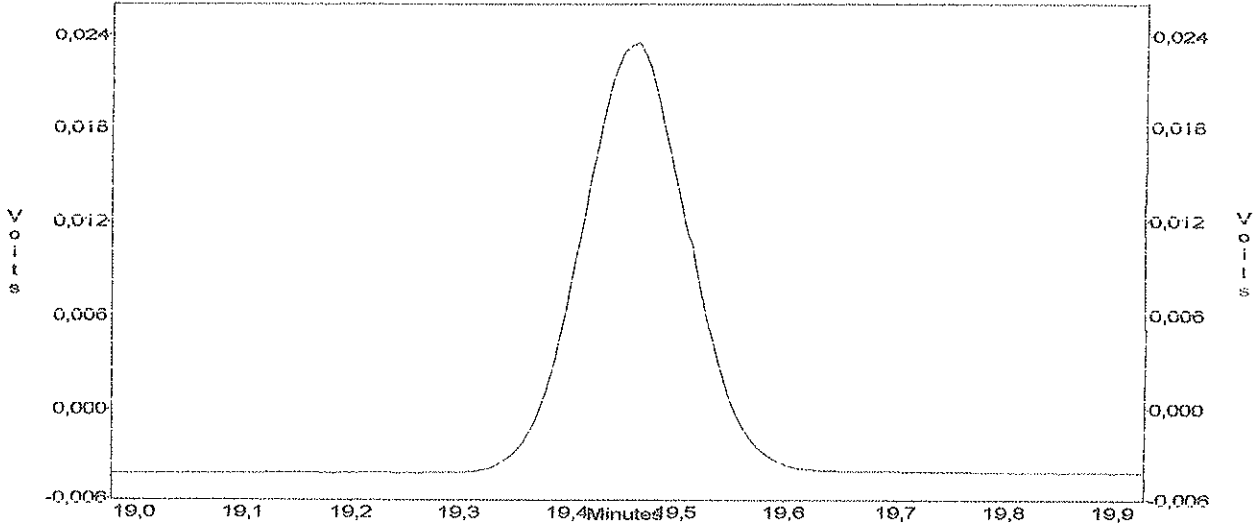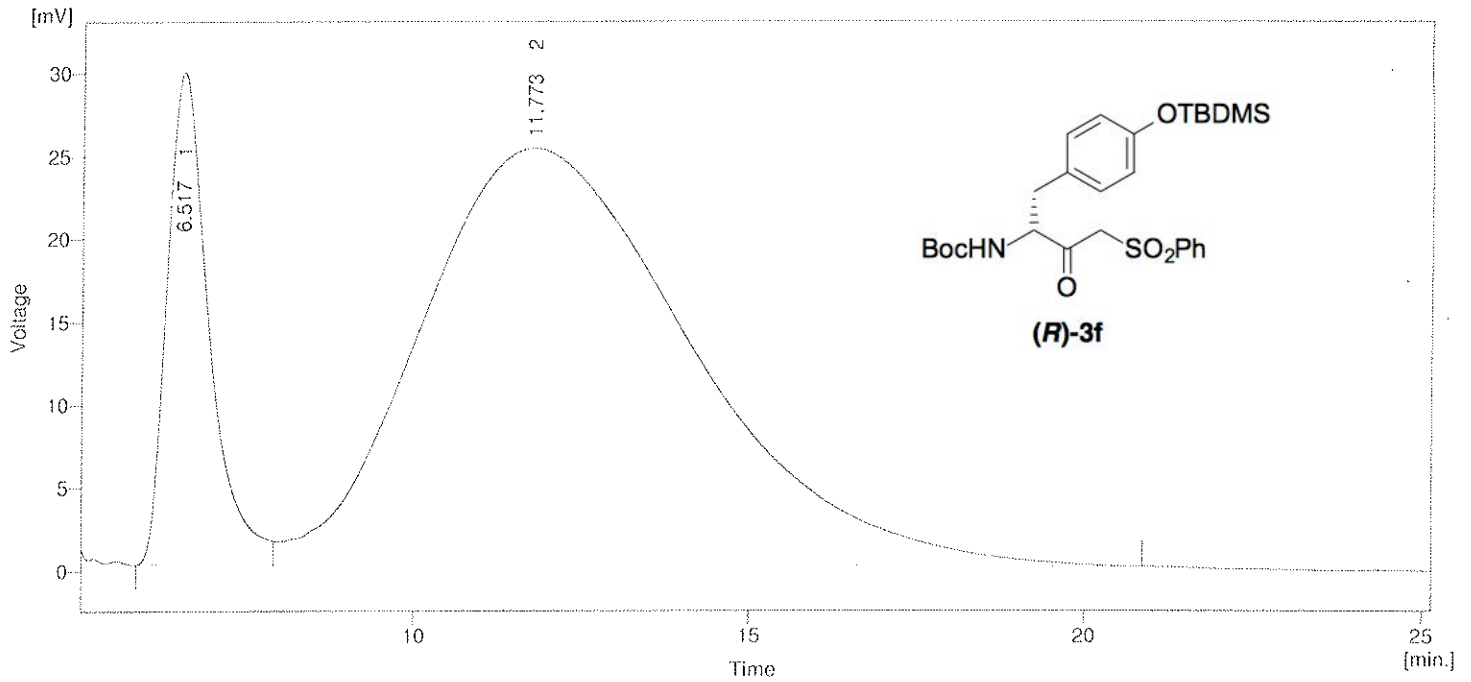
(S)-6g

c:\class-vp\chrom\Mg6 -- Channel B
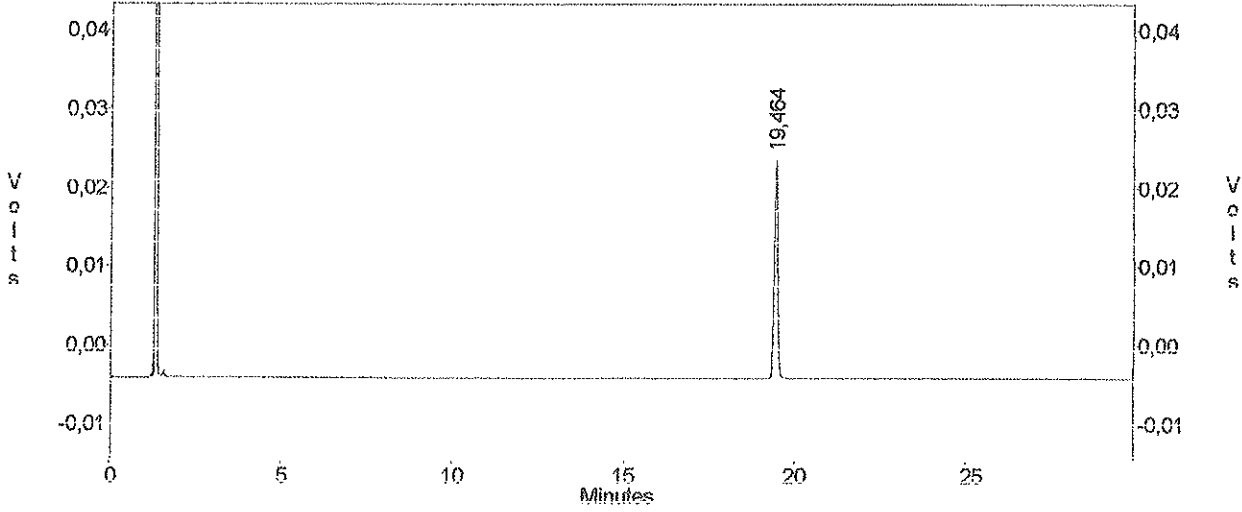
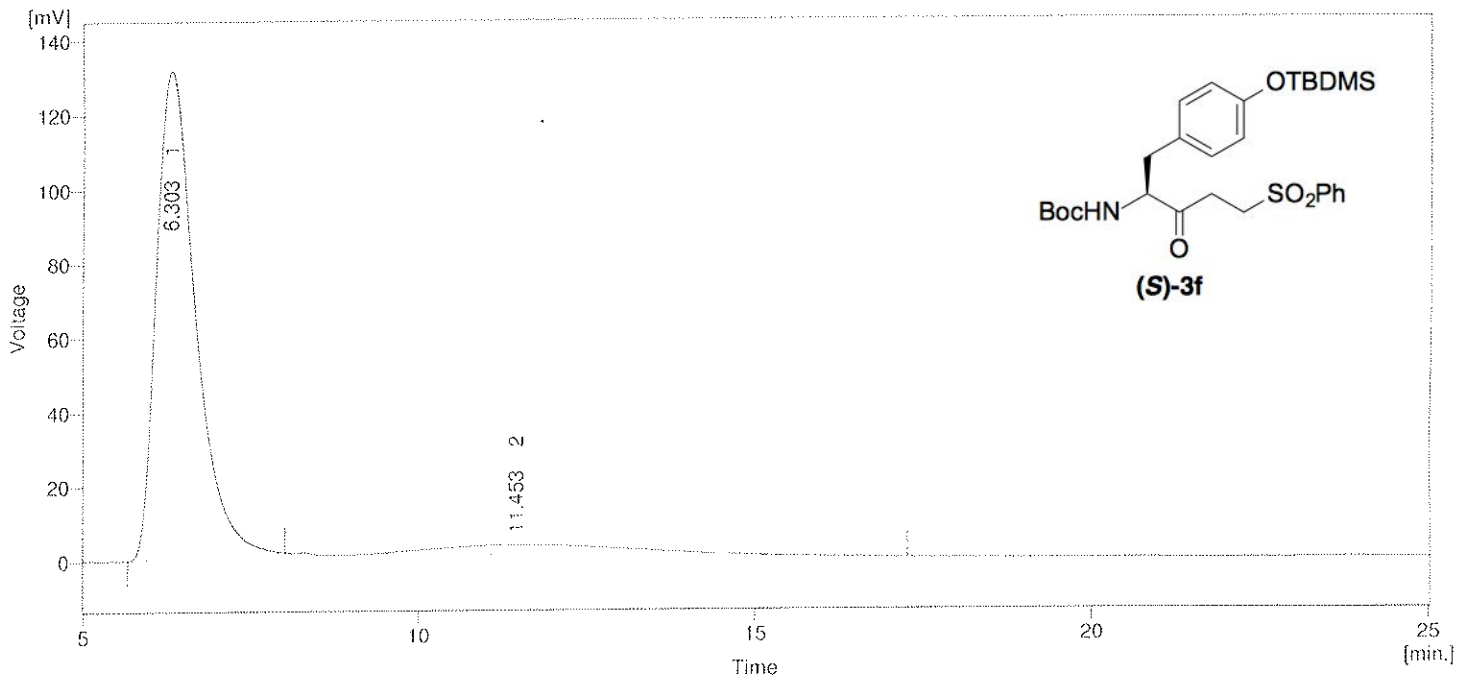c:\class-vp\chrom\Mg6 -- Channel B

Channel B Results

Peak	Time	Area	Area %
1	19,46	188661	100,000

Totals :

| | | 188661 | 100,000 |

Result Table

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	6.517	1293.477	29.654	15.5	54.1	0.64
2	11.773	7034.776	25.134	84.5	45.9	4.32
	Total	8328.253	54.788	100.0	100.0	

Result Table

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	6.303	5242.714	130.997	90.7	98.1	0.61
2	11.453	536.721	2.527	9.3	1.9	3.24
	Total	5779.435	133.524	100.0	100.0	
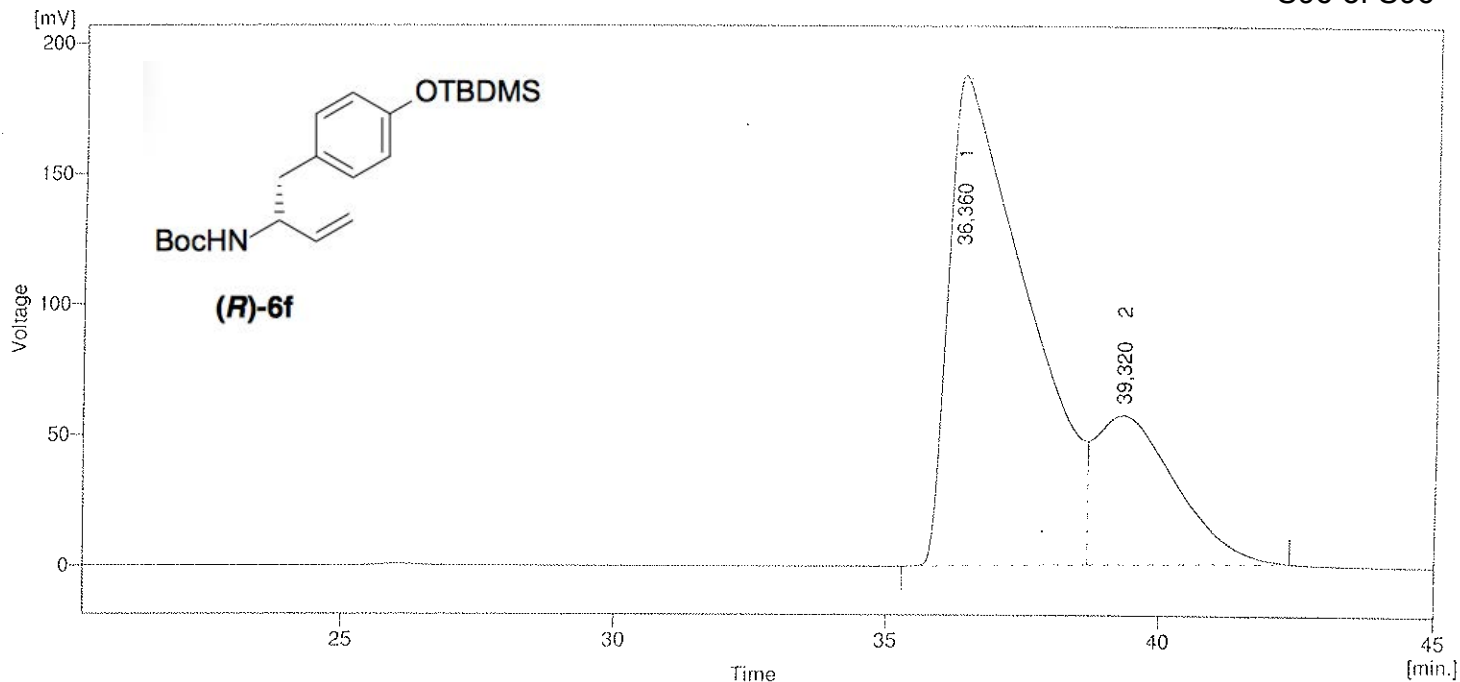
Result Table

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	36,360	18656,150	187,571	76,0	76,7	1,62
2	39,320	5896,603	57,075	24,0	23,3	1,69
	Total	24552,753	244,646	100,0	100,0	
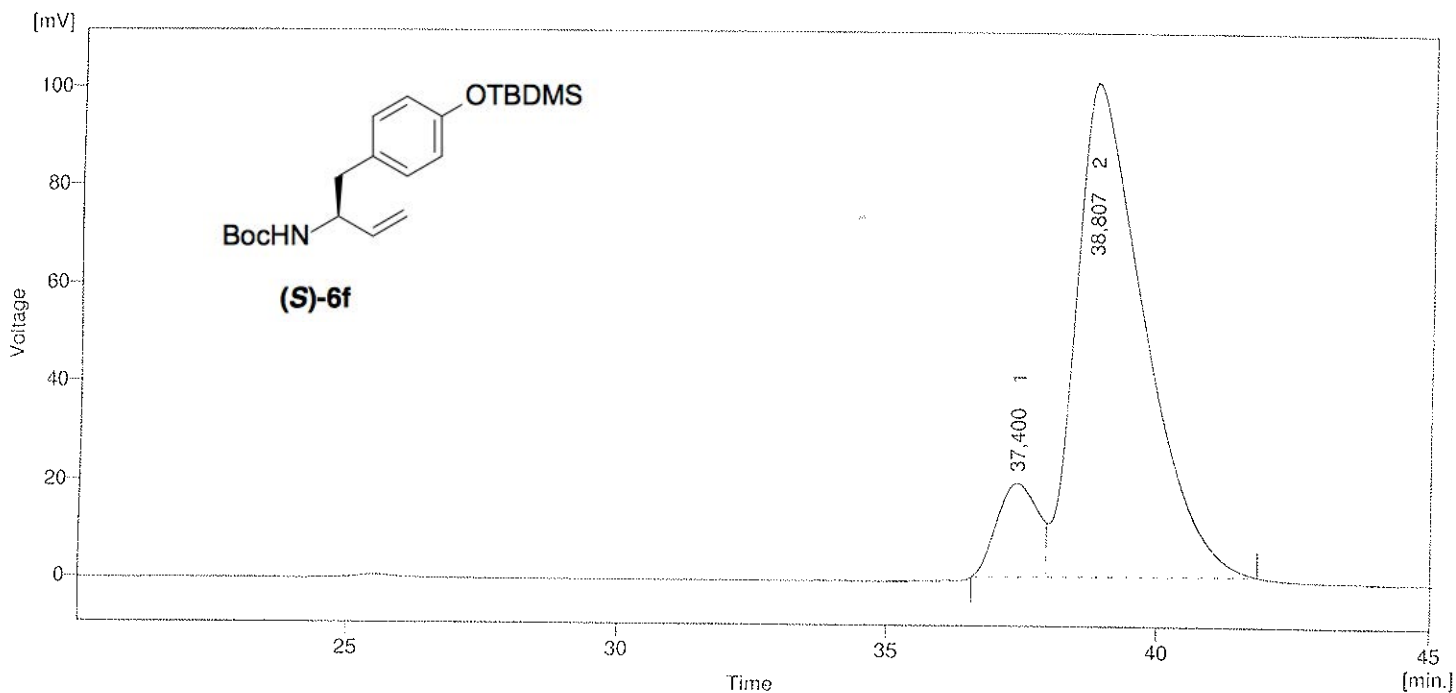
Result Table

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	37,400	1004,754	19,350	10,2	16,1	0,97
2	38,807	8819,375	100,597	89,8	83,9	1,34
	Total	9824,129	119,947	100,0	100,0	