Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Stereoselective Synthesis of (–)-Desethyleburnamonine, (–)- Vindeburnol and (–)-3-Epitacamonine: Observation of a Substrate Dependent Diastereoselectivity Reversal of an Aldol Reaction†

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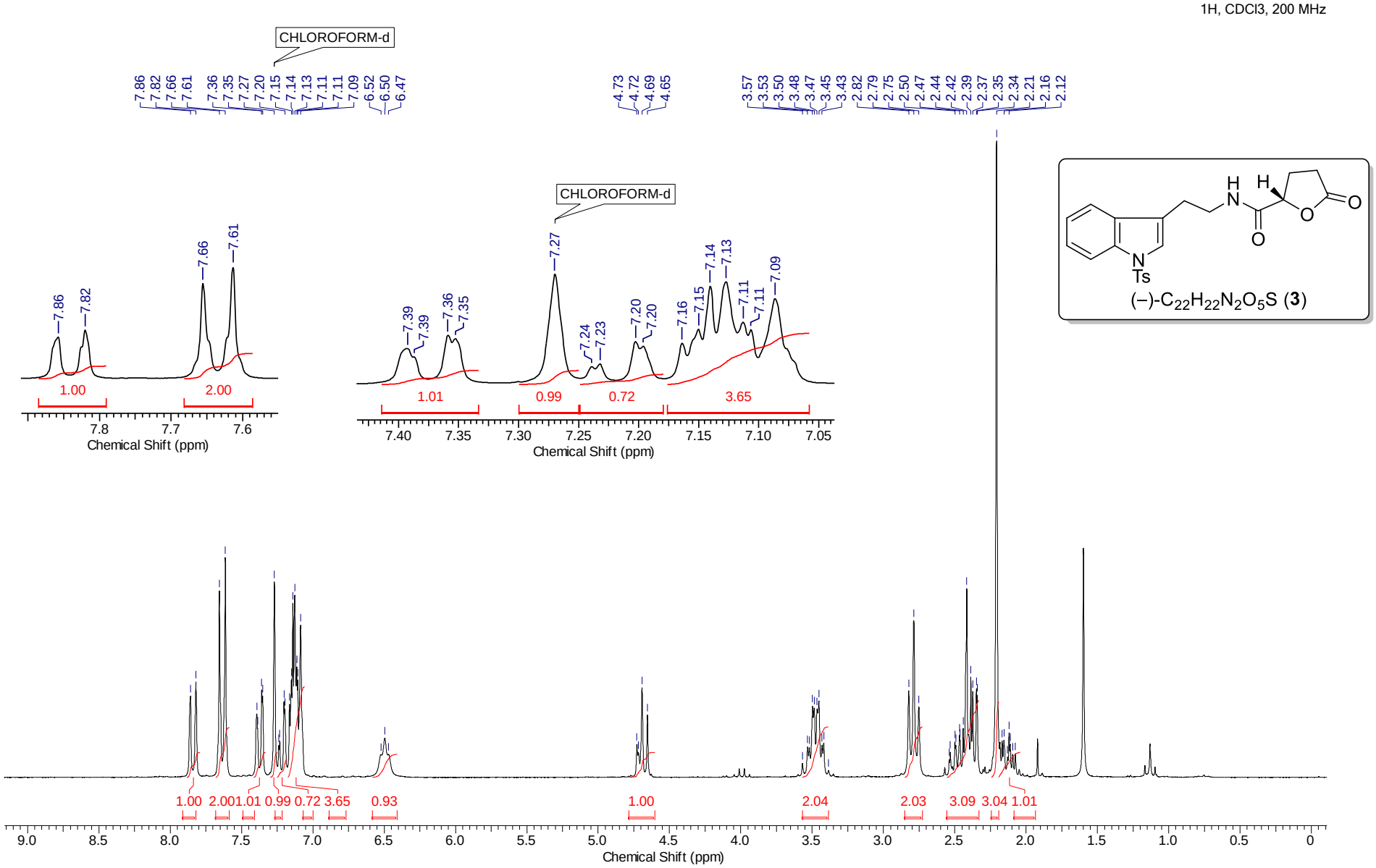
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4.73 4.72 4.69 4.65
3.57 3.53 3.50 3.48 3.47 3.45 3.43 2.82 2.79 2.75 2.50 2.47 2.44 2.42 2.39 2.37 2.35 2.34 2.21 2.16 2.12
1.00 2.00
Chemical Shift (ppm)
1.01 0.99 0.72 3.65
Chemical Shift (ppm)
(−)-C22H22N2O5S (3)
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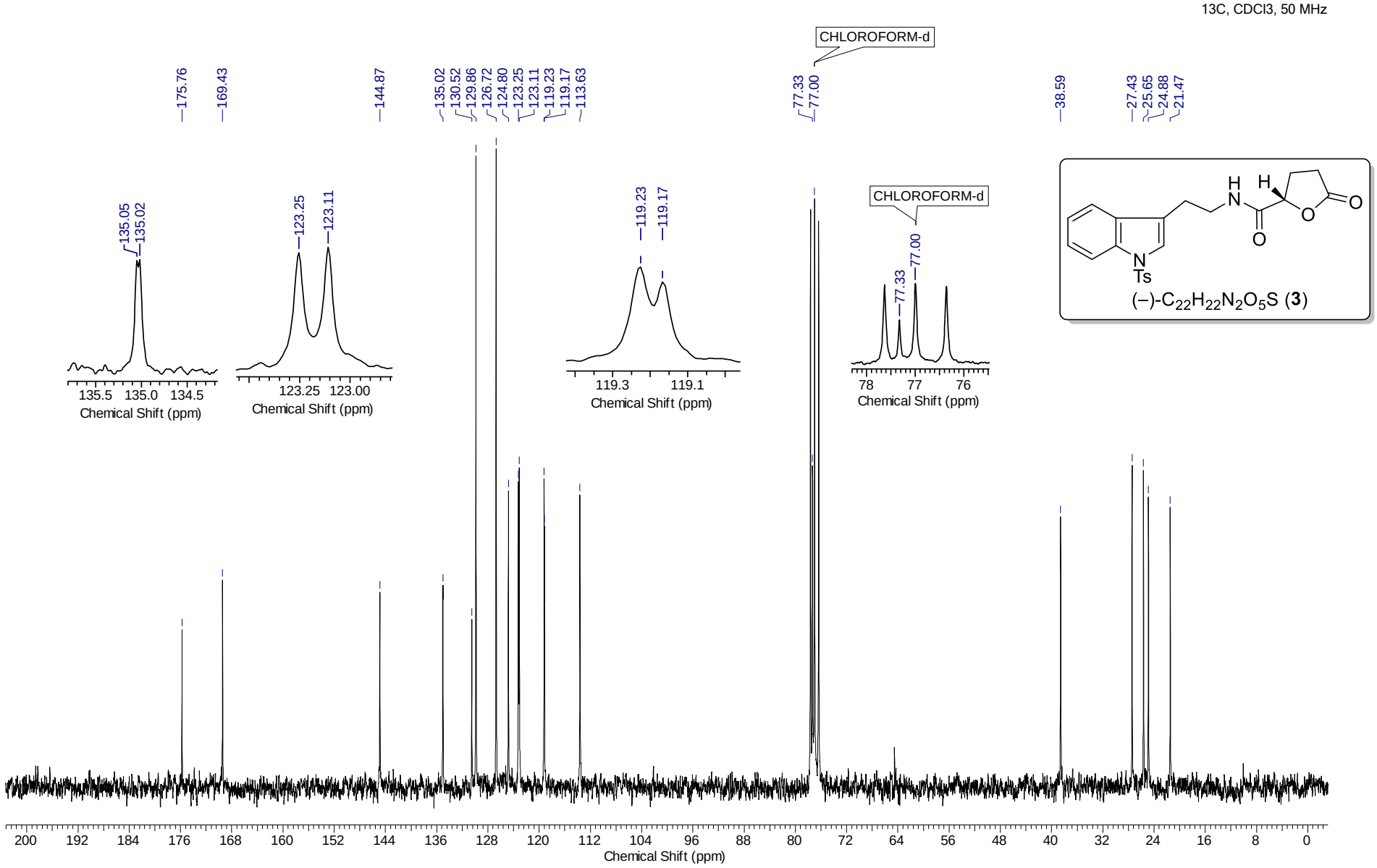
Chemical Shift (ppm)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169.43
144.87
135.02
130.52
129.86
126.72
124.80
123.25
123.11
119.23
119.17
113.63
77.33
77.00
38.59
27.43
25.65
24.88
21.47
135.05
135.02
135.5 135.0 134.5
Chemical Shift (ppm)
123.25
123.11
123.25 123.00
Chemical Shift (ppm)
119.23
119.17
119.3 119.1
Chemical Shift (ppm)
CHLOROFORM-d
77.33
77.00
78 77 76
Chemical Shift (ppm)
H
H
N
O
O
O
N
Ts
(–)-C22H22N2O5S (3)
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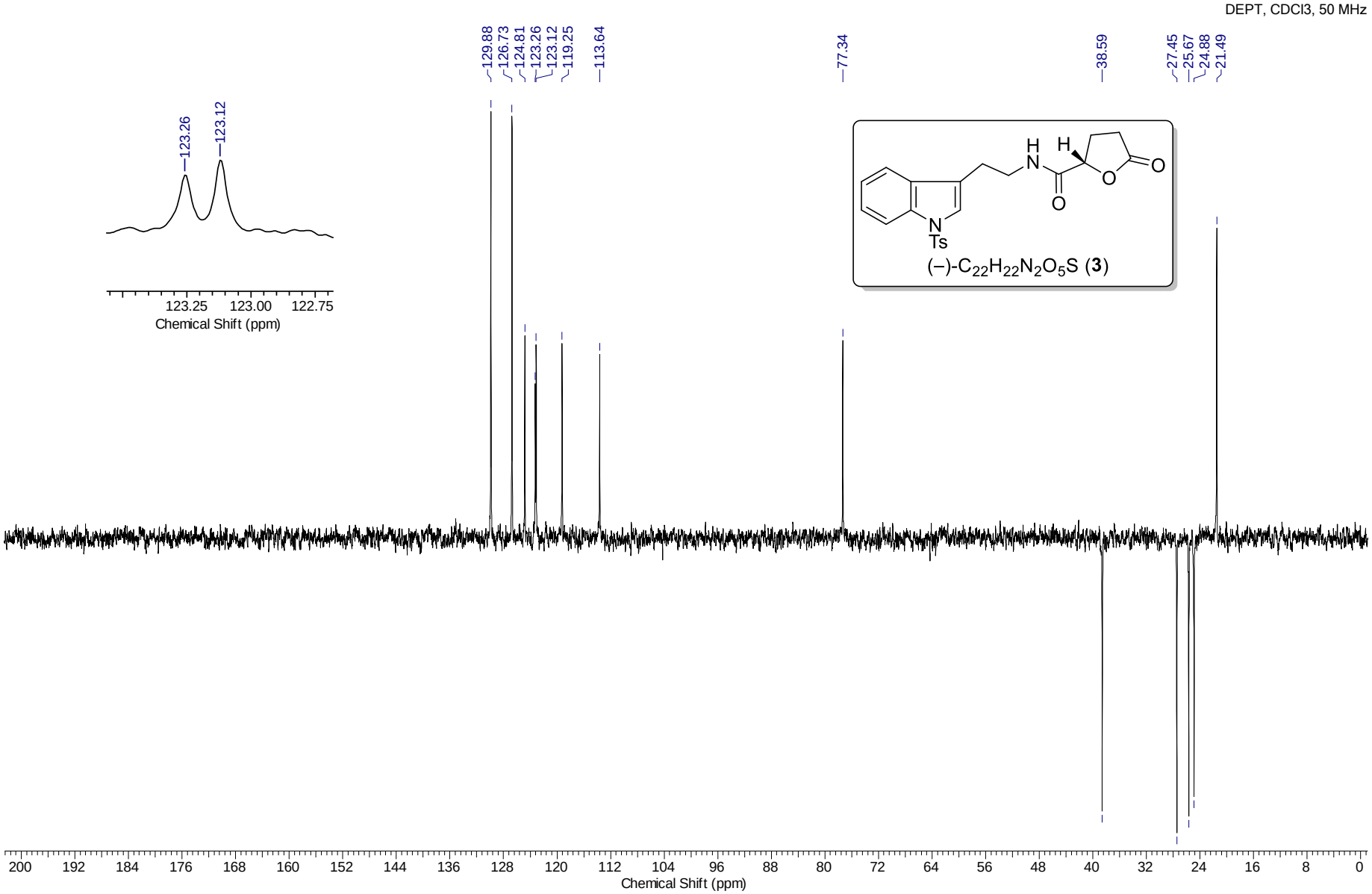
Chemical Shift (ppm)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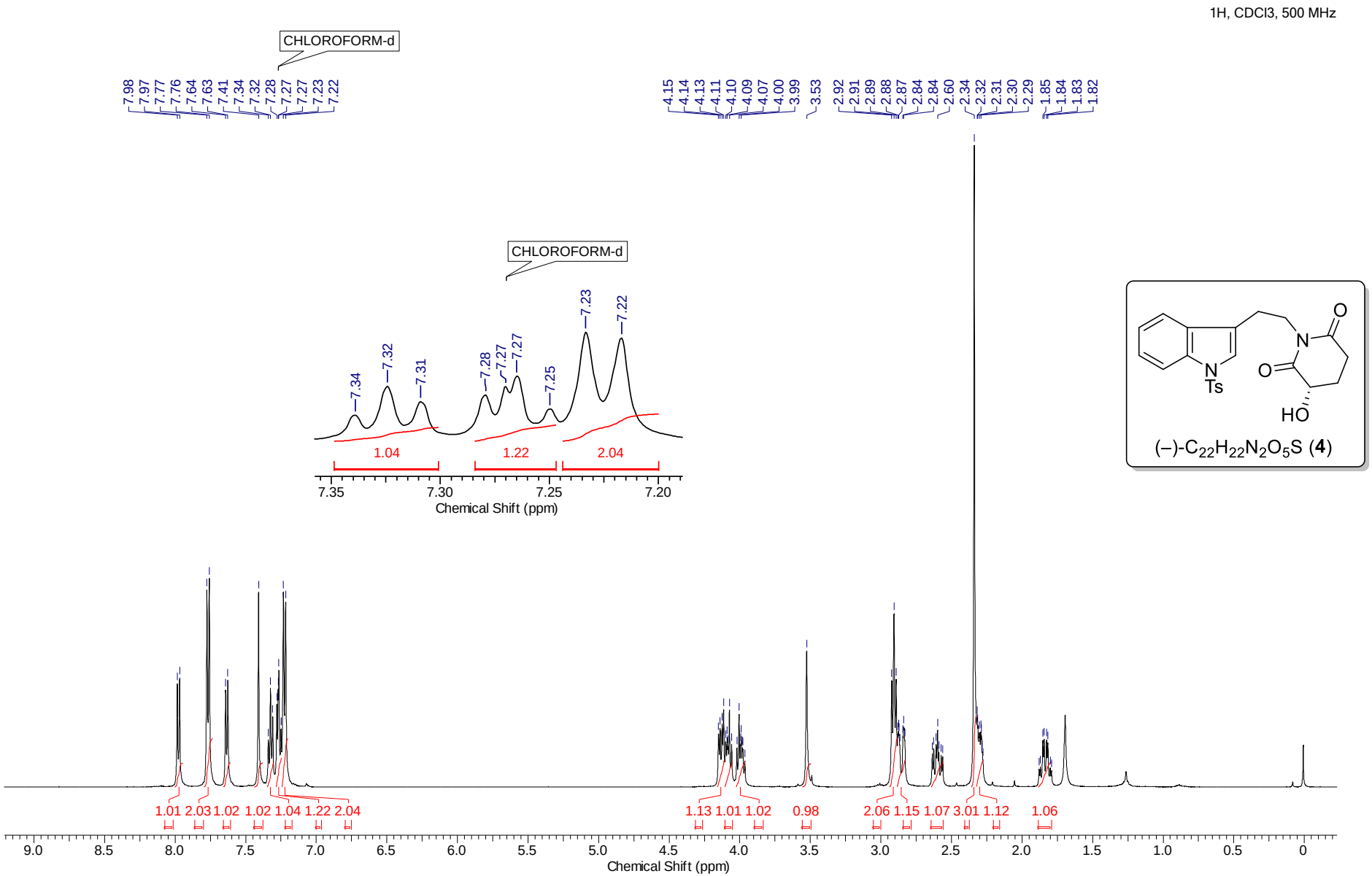
126.73
124.81
123.26
123.12
119.25
113.64
77.34
38.59
27.45
25.67
24.88
21.49
123.26
123.12
123.25
123.00
122.75
Chemical Shift (ppm)
H
N
H
O
O
O
N
Ts
(–)-C22H22N2O5S (3)
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)


1H, CDCl3, 500 MHz
CHLOROFORM-d
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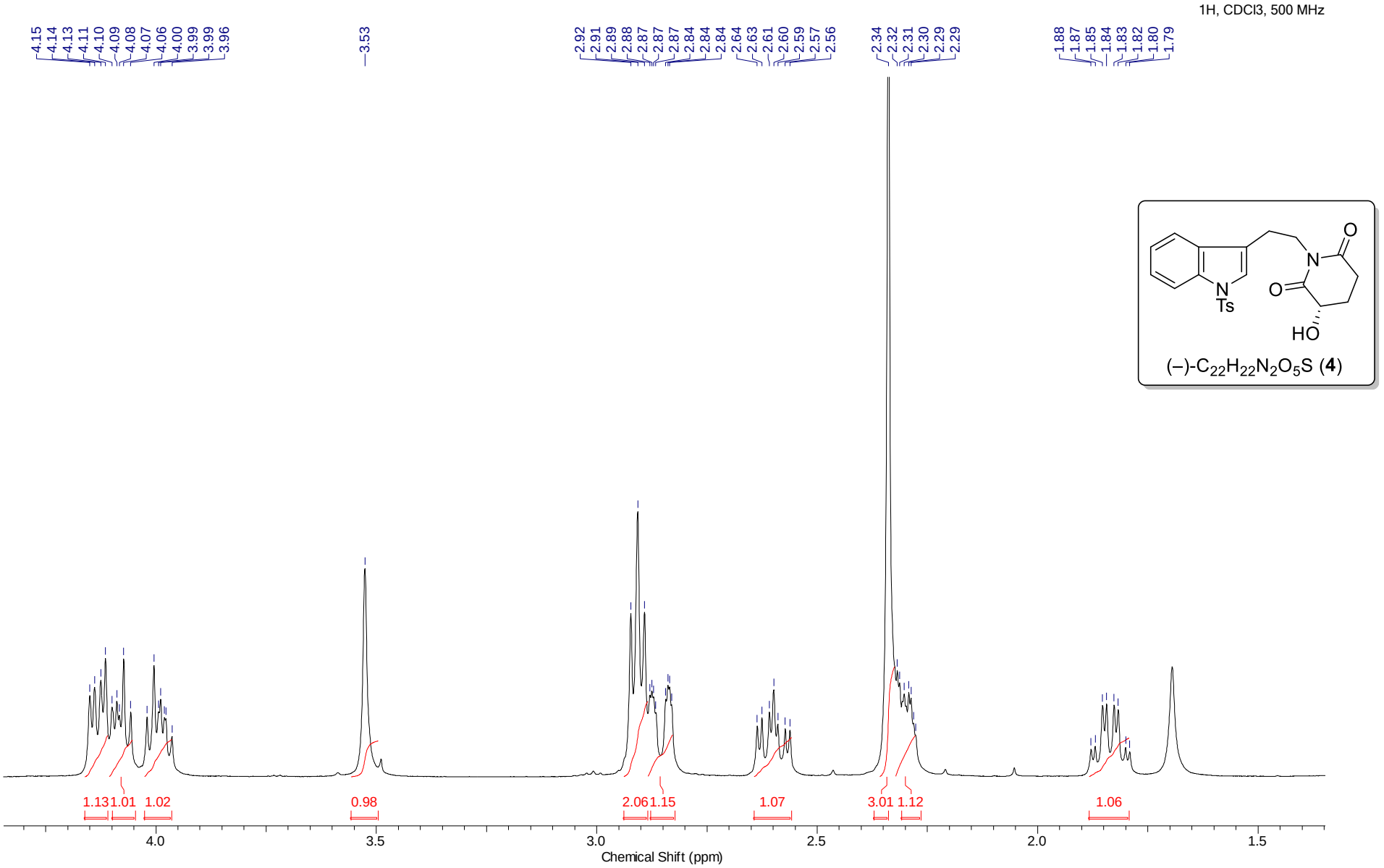
4.15 4.14 4.13 4.11 4.10 4.09 4.07 4.00 3.99 3.53 2.92 2.91 2.89 2.88 2.87 2.84 2.84 2.60 2.34 2.32 2.31 2.30 2.29 1.85 1.84 1.83 1.82
CHLOROFORM-d
7.34 7.32 7.31 7.28 7.27 7.27 7.25 7.23 7.22
1.04 1.22 2.04
7.35 7.30 7.25 7.20
Chemical Shift (ppm)
N
Ts
N
O
O
HO
(–)-C22H22N2O5S (4)
1.01 2.03 1.02 1.02 1.04 1.22 2.04
1.13 1.01 1.02 0.98 2.06 1.15 1.07 3.01 1.12 1.06
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
Chemical Shift (ppm)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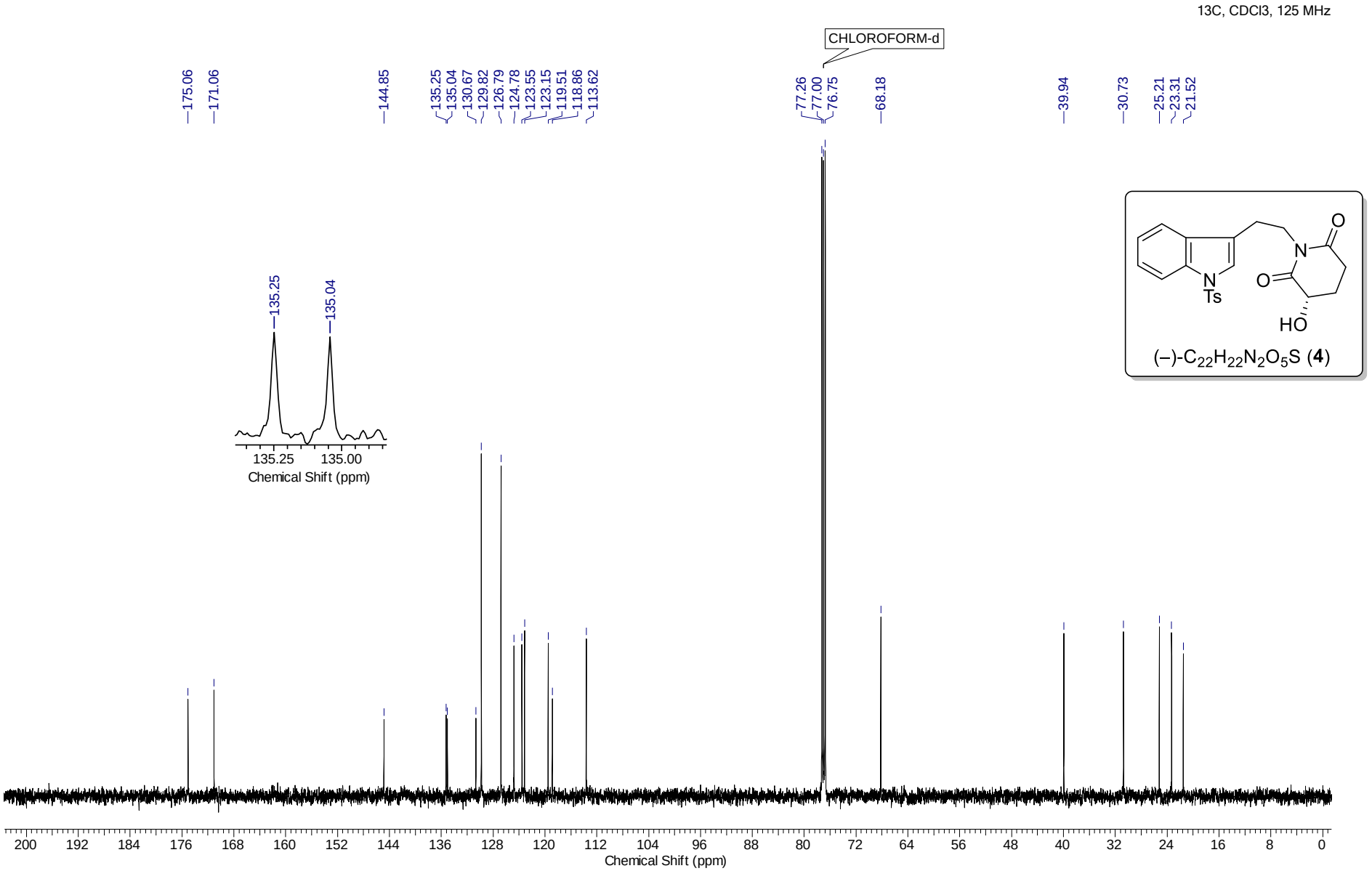
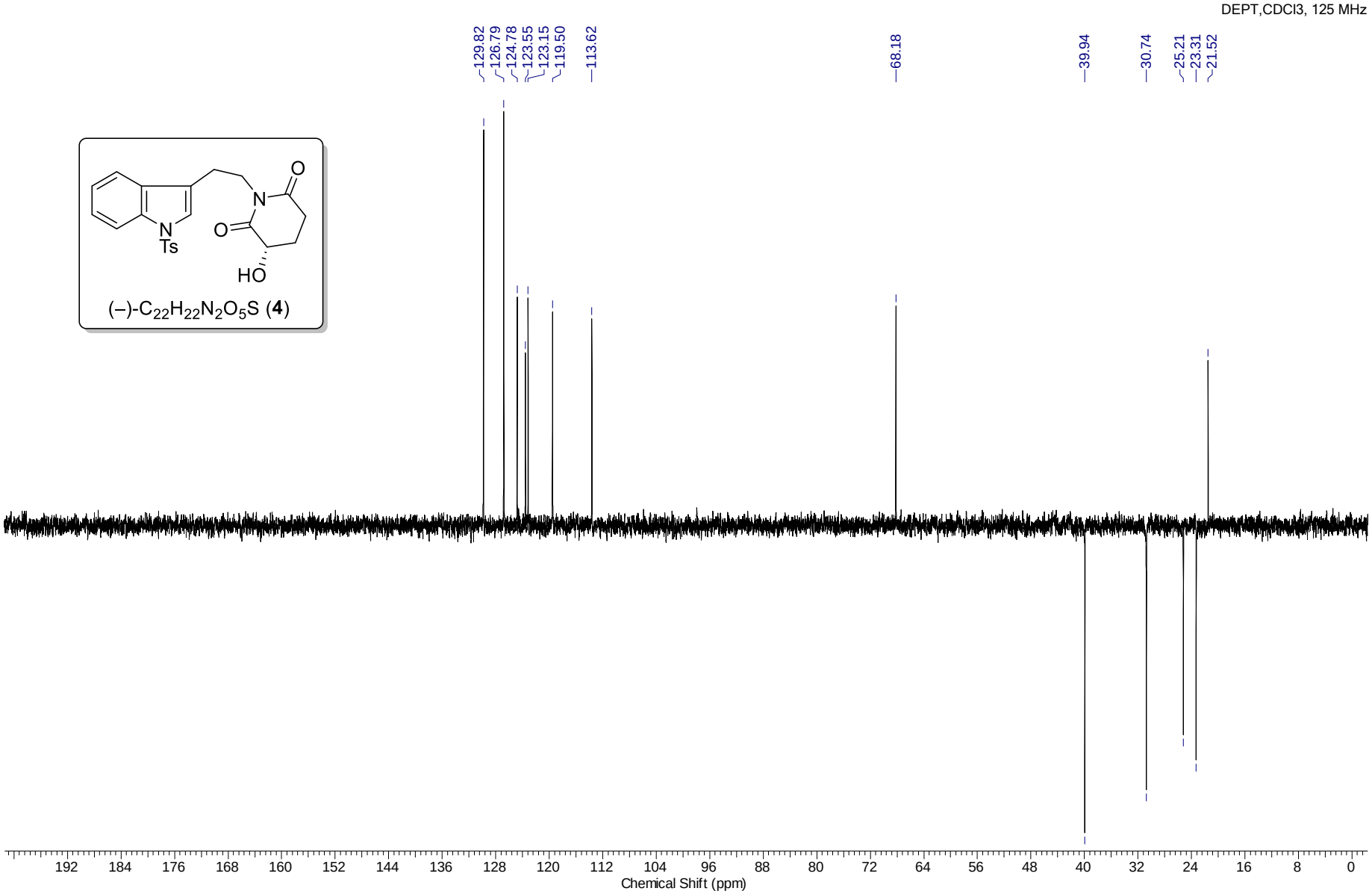
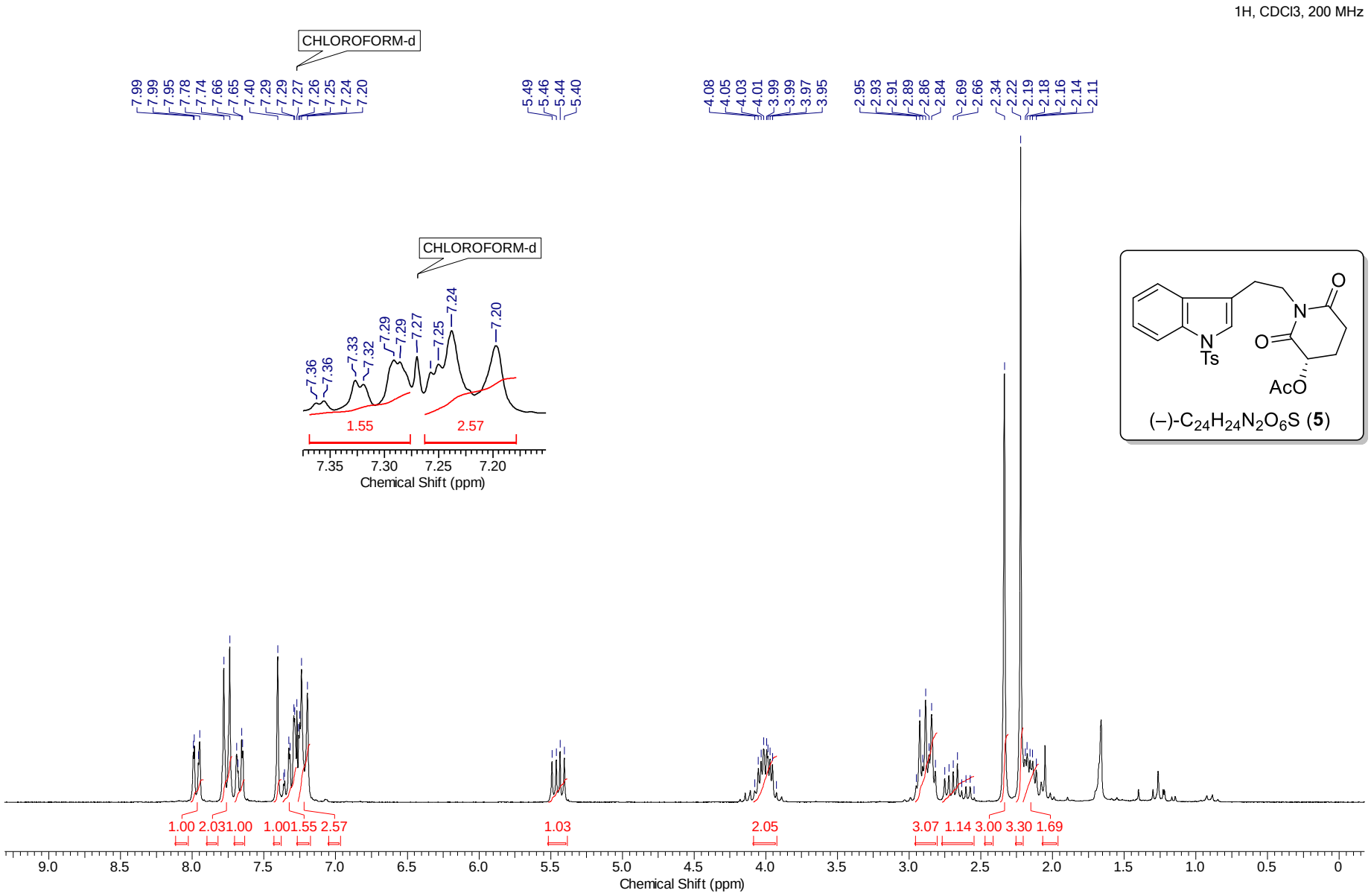
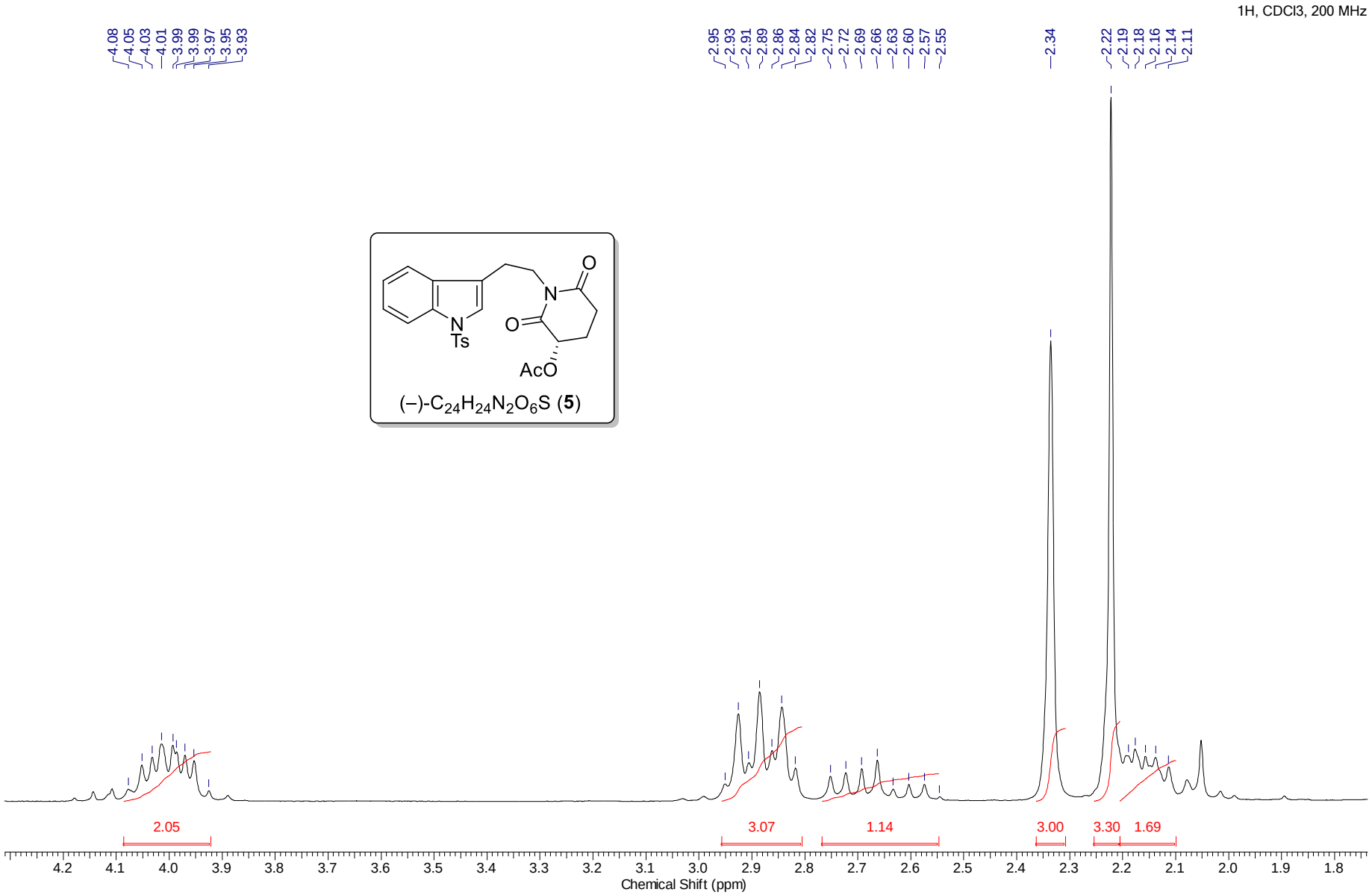
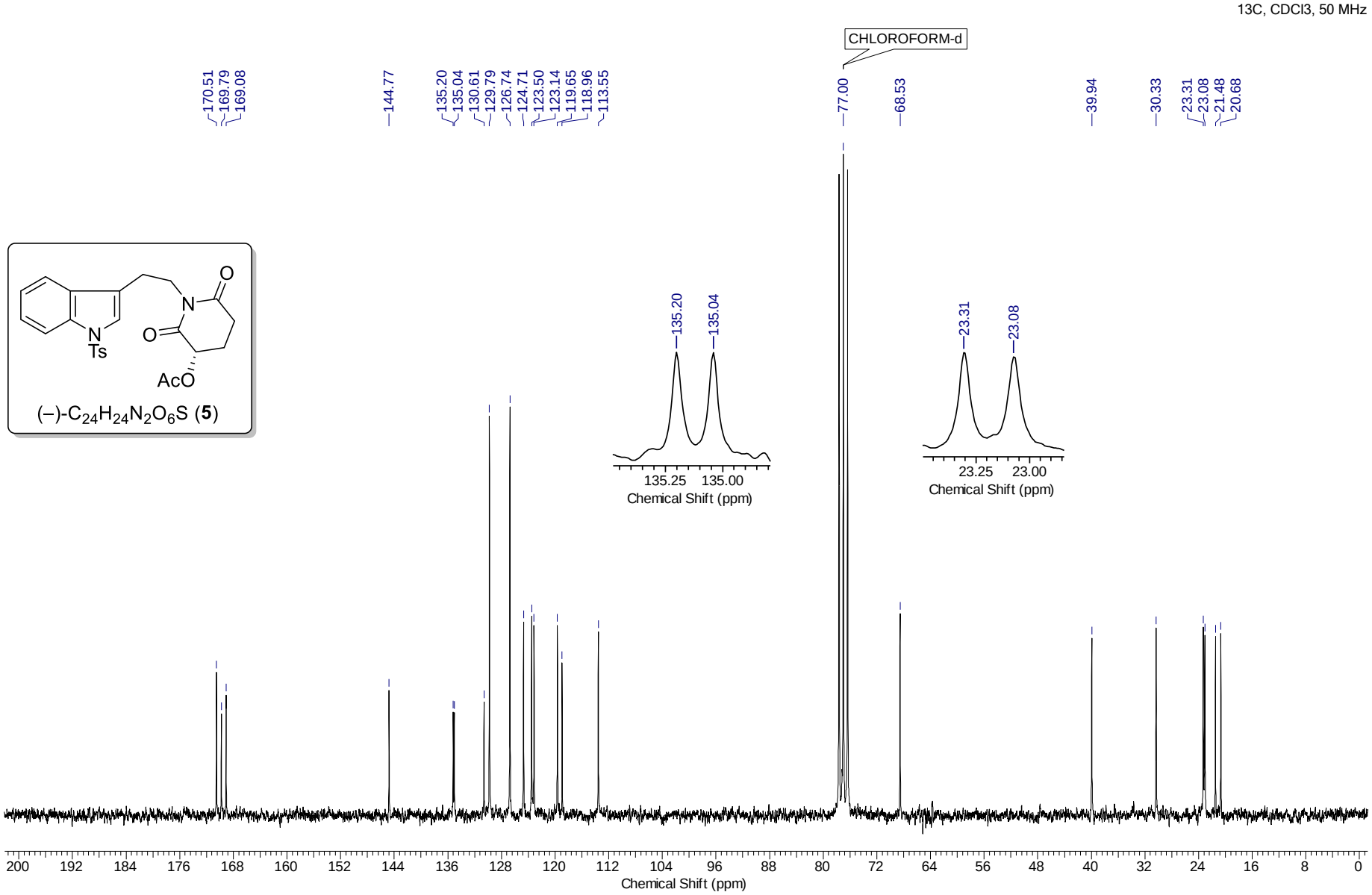
4.15 4.14 4.13 4.11 4.10 4.09 4.08 4.07 4.06 4.00 3.99 3.99 3.96
3.53
2.92 2.91 2.89 2.88 2.87 2.87 2.87 2.84 2.84 2.84 2.64 2.63 2.61 2.60 2.59 2.57 2.56
2.34 2.32 2.31 2.30 2.29 2.29
1.88 1.87 1.85 1.84 1.83 1.82 1.80 1.79
1.13 1.01 1.02
0.98
2.06 1.15
1.07
3.01 1.12
1.06
4.0
3.5
3.0
2.5
2.0
1.5
Chemical Shift (ppm)
N
Ts
O
N
O
HO
(–)-C22H22N2O5S (4)

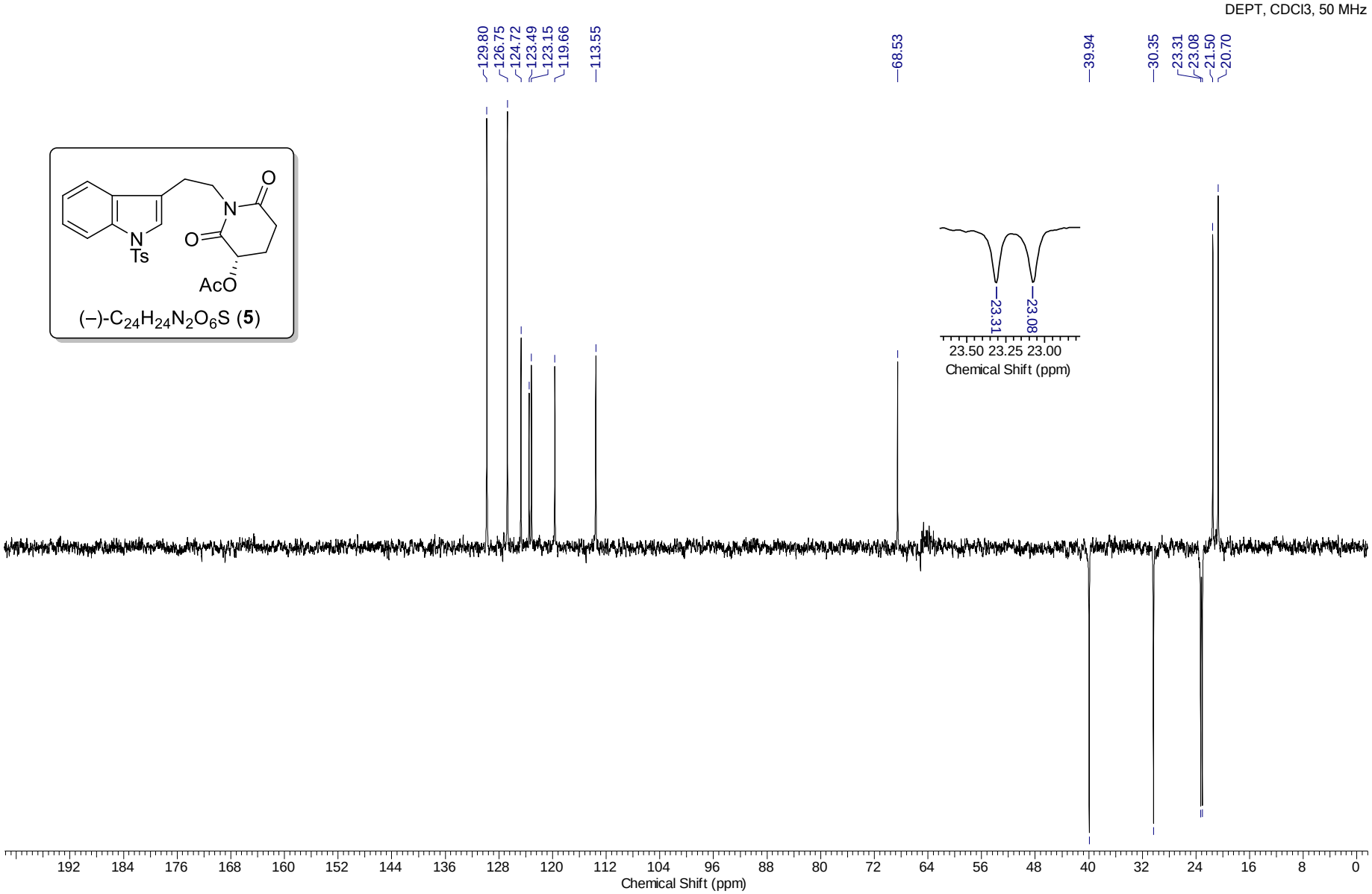

13C, CDCl3, 125 MHz
CHLOROFORM-d
175.06
171.06
144.85
135.25
135.04
130.67
129.82
126.79
124.78
123.55
123.15
119.51
118.86
113.62
77.26
77.00
76.75
68.18
39.94
30.73
25.21
23.31
21.52
135.25
135.04
135.25
135.00
Chemical Shift (ppm)
N
Ts
O
N
O
HO
(–)-C22H22N2O5S (4)
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

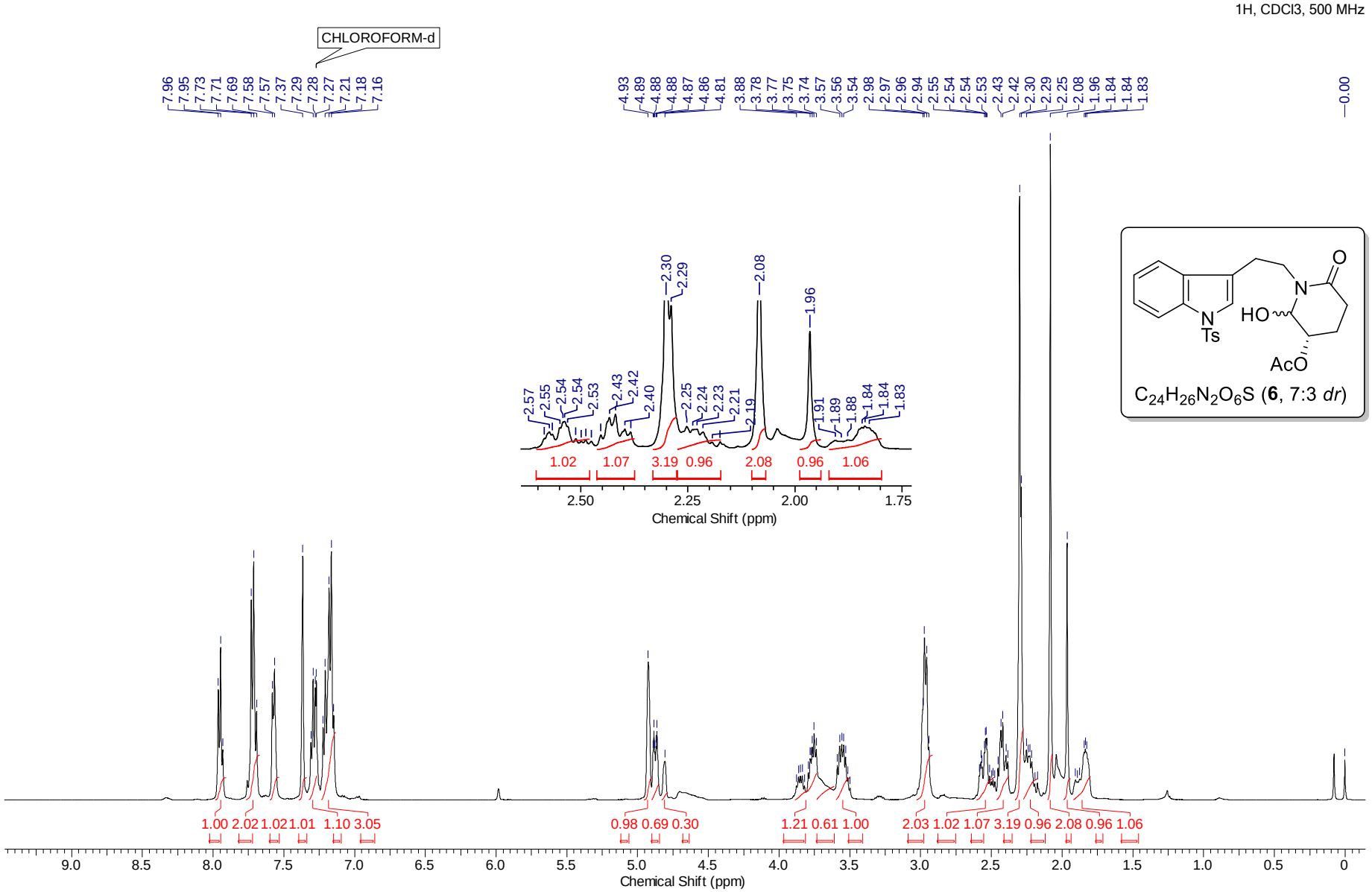

DEPT,CDCl3, 125 MHz
129.82
126.79
124.78
123.55
123.15
119.50
113.62
68.18
39.94
30.74
25.21
23.31
21.52
O
N
O
N
Ts
HO
(–)-C22H22N2O5S (4)
192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

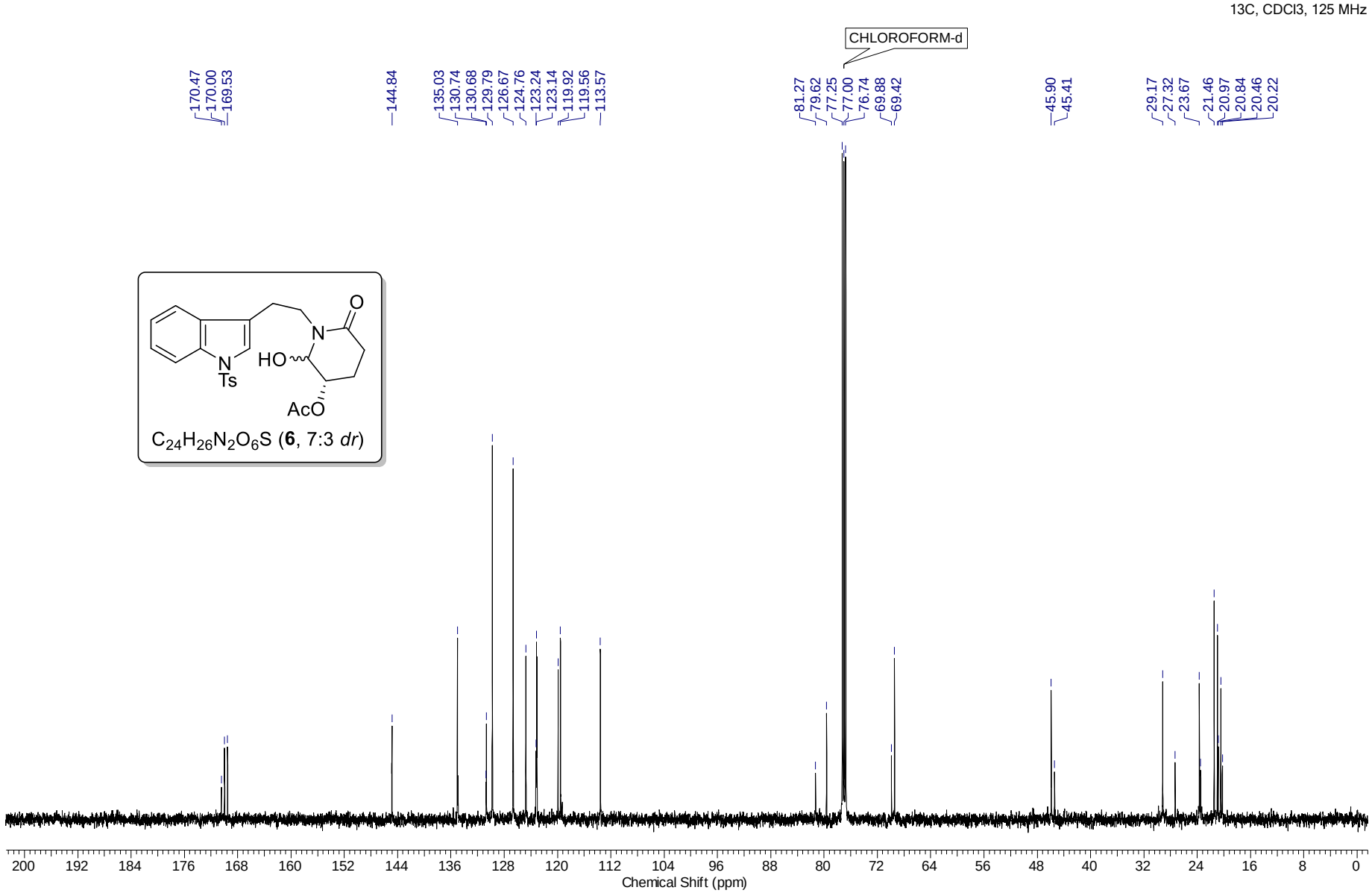

1H, CDCl3, 200 MHz
CHLOROFORM-d
7.99 7.99 7.95 7.78 7.74 7.66 7.65 7.40 7.29 7.29 7.27 7.26 7.25 7.24 7.20
5.49 5.46 5.44 5.40
4.08 4.05 4.03 4.01 3.99 3.99 3.97 3.95
2.95 2.93 2.91 2.89 2.86 2.84 2.69 2.66 2.34 2.22 2.19 2.18 2.16 2.14 2.11
CHLOROFORM-d
7.36 7.36 7.33 7.32 7.29 7.29 7.27 7.25 7.24 7.20
1.55 2.57
7.35 7.30 7.25 7.20
Chemical Shift (ppm)
N
Ts
O
N
O
AcO
(–)-C24H24N2O6S (5)
1.00 2.03 1.00 1.00 1.55 2.57 1.03 2.05 3.07 1.14 3.00 3.30 1.69
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
Chemical Shift (ppm)

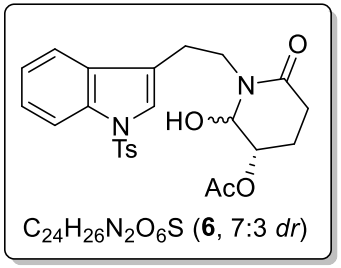

1H, CDCl3, 200 MHz
4.08
4.05
4.03
4.01
3.99
3.99
3.97
3.95
3.93
2.95
2.93
2.91
2.89
2.86
2.84
2.82
2.75
2.72
2.69
2.66
2.63
2.60
2.57
2.55
2.34
2.22
2.19
2.18
2.16
2.14
2.11
N
Ts
O
N
O
AcO
(–)-C24H24N2O6S (5)
2.05
3.07
1.14
3.00
3.30
1.69
4.2
4.1
4.0
3.9
3.8
3.7
3.6
3.5
3.4
3.3
3.2
3.1
3.0
2.9
2.8
2.7
2.6
2.5
2.4
2.3
2.2
2.1
2.0
1.9
1.8
Chemical Shift (ppm)


13C, CDCl3, 50 MHz
CHLOROFORM-d
170.51
169.79
169.08
144.77
135.20
135.04
130.61
129.79
126.74
124.71
123.50
123.14
119.65
118.96
113.55
77.00
68.53
39.94
30.33
23.31
23.08
21.48
20.68
(–)-C24H24N2O6S (5)
135.25 135.00
Chemical Shift (ppm)
23.25 23.00
Chemical Shift (ppm)
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)


DEPT, CDCl3, 50 MHz
129.80
126.75
124.72
123.49
123.15
119.66
113.55
68.53
39.94
30.35
23.31
23.08
21.50
20.70
N
Ts
O
N
O
AcO
(–)-C24H24N2O6S (5)
23.31
23.08
23.50 23.25 23.00
Chemical Shift (ppm)
192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)


1H, CDCl3, 500 MHz
CHLOROFORM-d
7.96 7.95 7.73 7.71 7.69 7.58 7.57 7.37 7.29 7.28 7.27 7.21 7.18 7.16
4.93 4.89 4.88 4.88 4.87 4.86 4.81 3.88 3.78 3.77 3.75 3.74 3.57 3.56 3.54 2.98 2.97 2.96 2.94 2.55 2.54 2.54 2.53 2.43 2.42 2.30 2.29 2.25 2.08 1.96 1.84 1.84 1.83
0.00
2.57 2.55 2.54 2.54 2.53 2.43 2.42 2.40 2.30 2.29 2.25 2.24 2.23 2.21 2.19 2.08 1.96 1.91 1.89 1.88 1.84 1.84 1.83
1.02 1.07 3.19 0.96 2.08 0.96 1.06
2.50 2.25 2.00 1.75
Chemical Shift (ppm)
O
N
HO
N
Ts
AcO
C24H26N2O6S (6, 7:3 dr)
1.00 2.02 1.02 1.01 1.10 3.05
0.98 0.69 0.30
1.21 0.61 1.00
2.03 1.02 1.07 3.19 0.96 2.08 0.96 1.06
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
Chemical Shift (ppm)


13C, CDCl3, 125 MHz
CHLOROFORM-d
170.47
170.00
169.53
144.84
135.03
130.74
130.68
129.79
126.67
124.76
123.24
123.14
119.92
119.56
113.57
81.27
79.62
77.25
77.00
76.74
69.88
69.42
45.90
45.41
29.17
27.32
23.67
21.46
20.97
20.84
20.46
20.22
O
N
HO
N
Ts
AcO
C24H26N2O6S (6, 7:3 dr)
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)



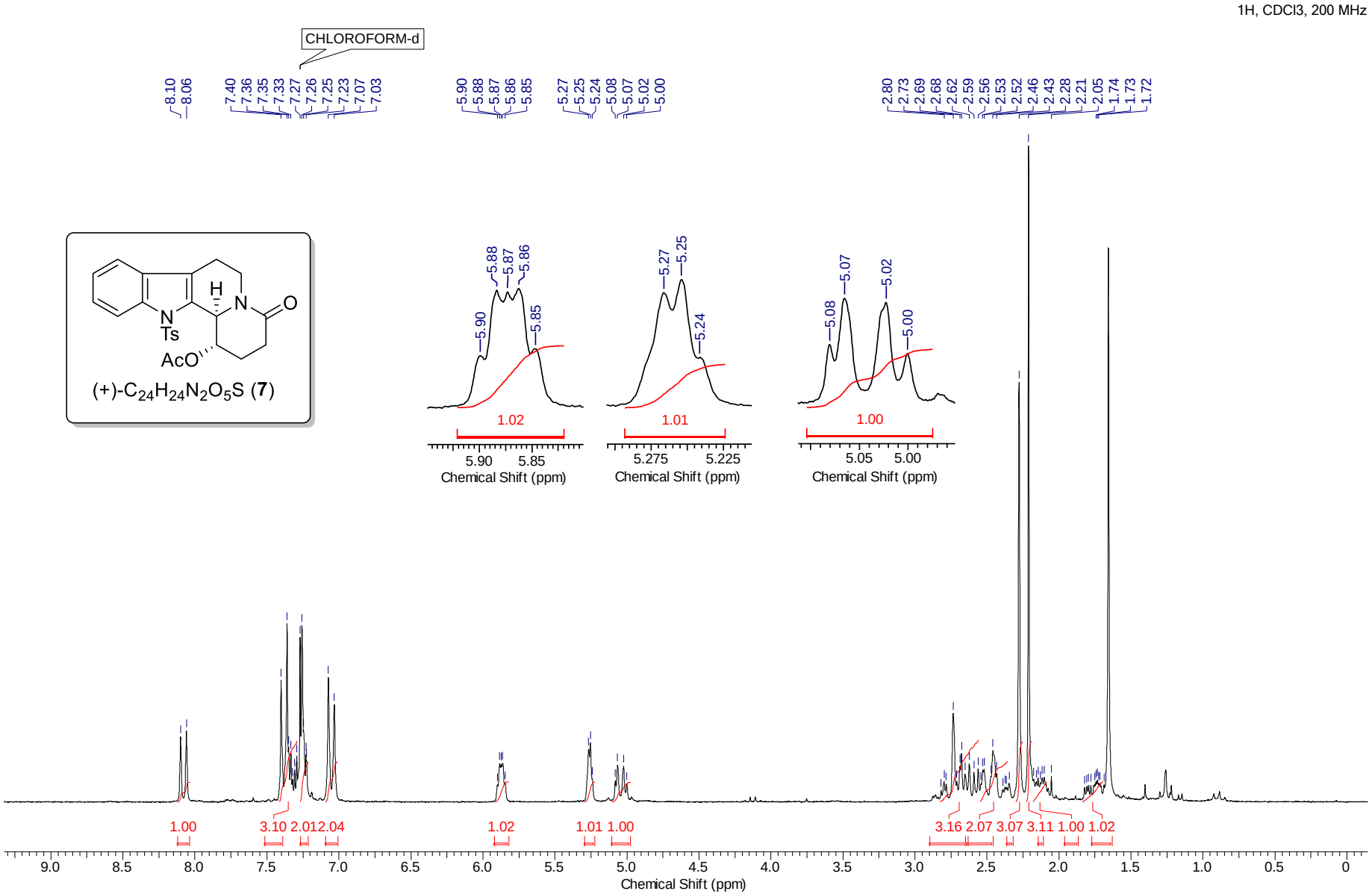
$C_{24}H_{26}N_2O_6S$ (**6**, 7:3 *dr*)

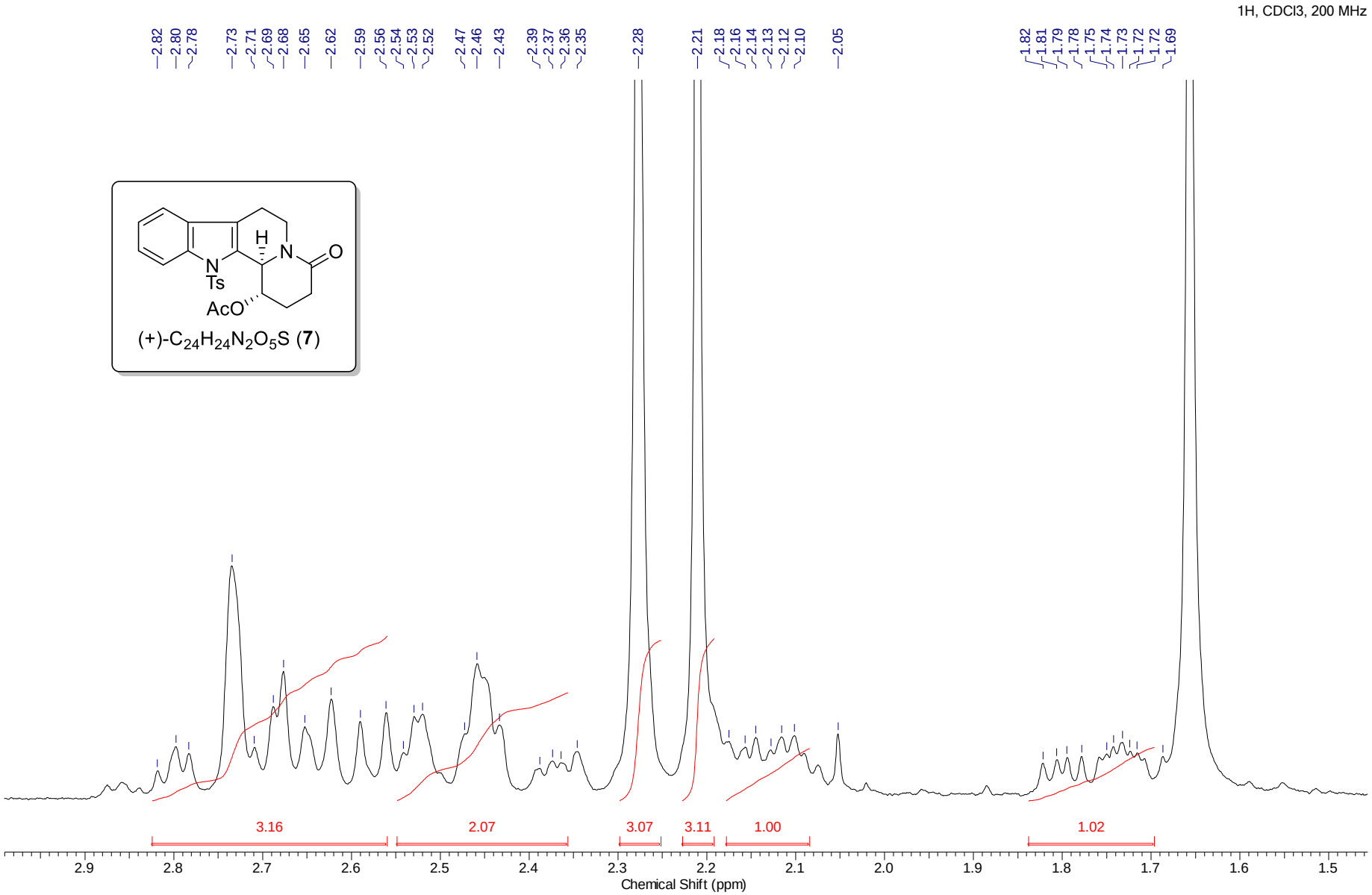
DEPT, CDCl3, 125 MHz

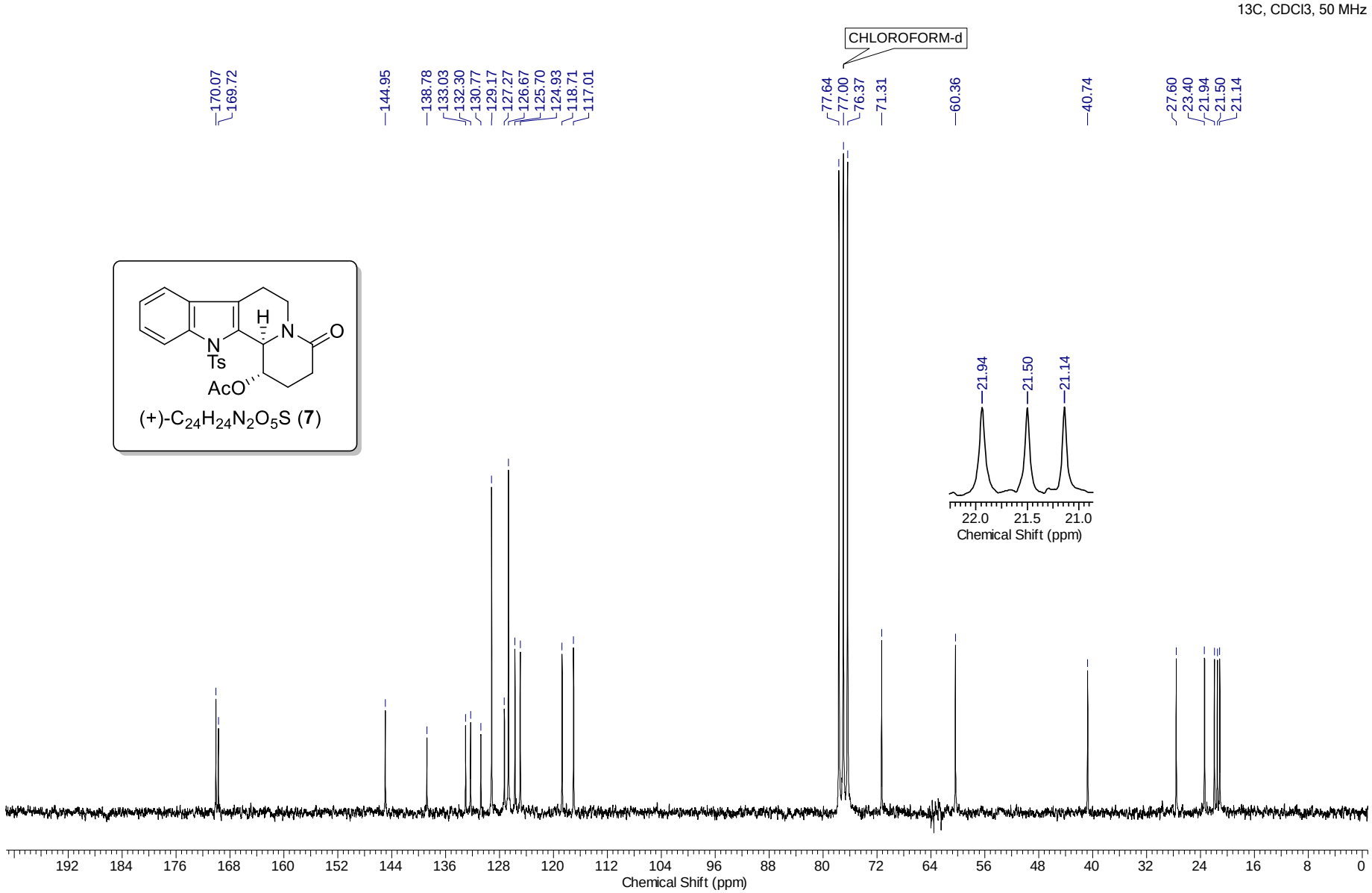
129.78
126.67
124.76
123.22
123.13
119.55
113.56
81.27
79.61
69.87
69.42
45.90
45.40
29.17
23.67
23.47
21.45
20.97
20.85
20.45
20.20

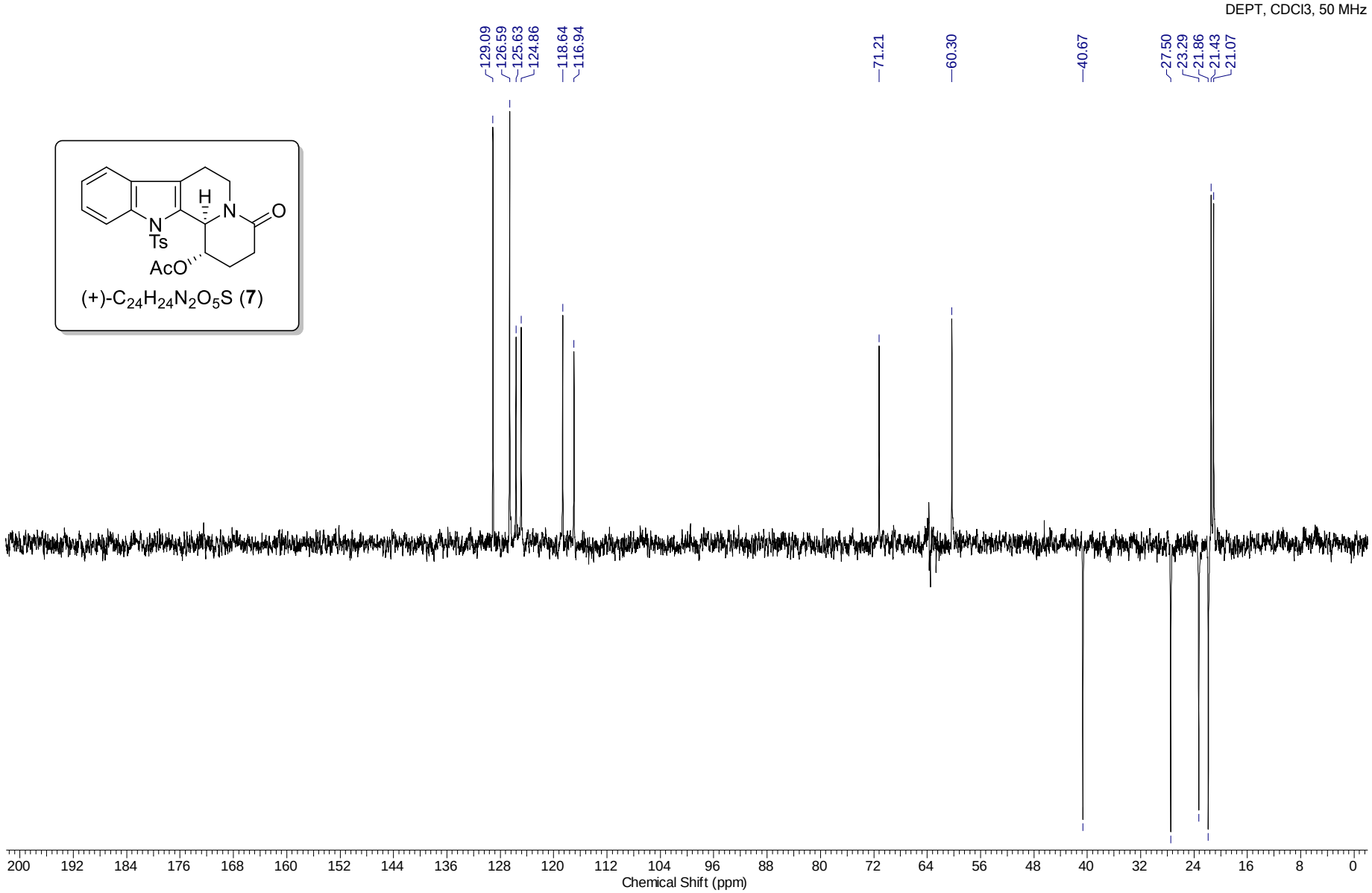
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0

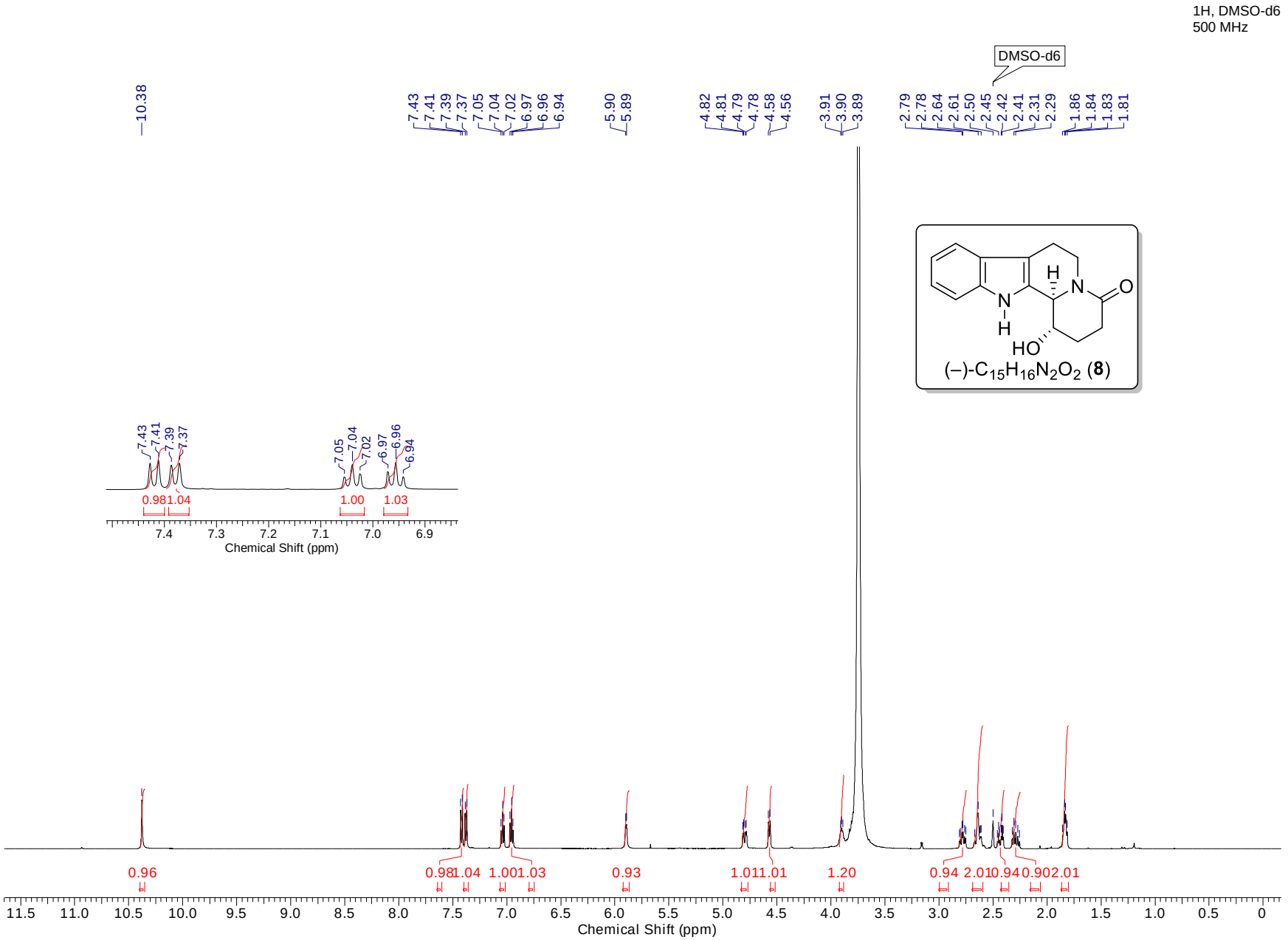
Chemical Shift (ppm)

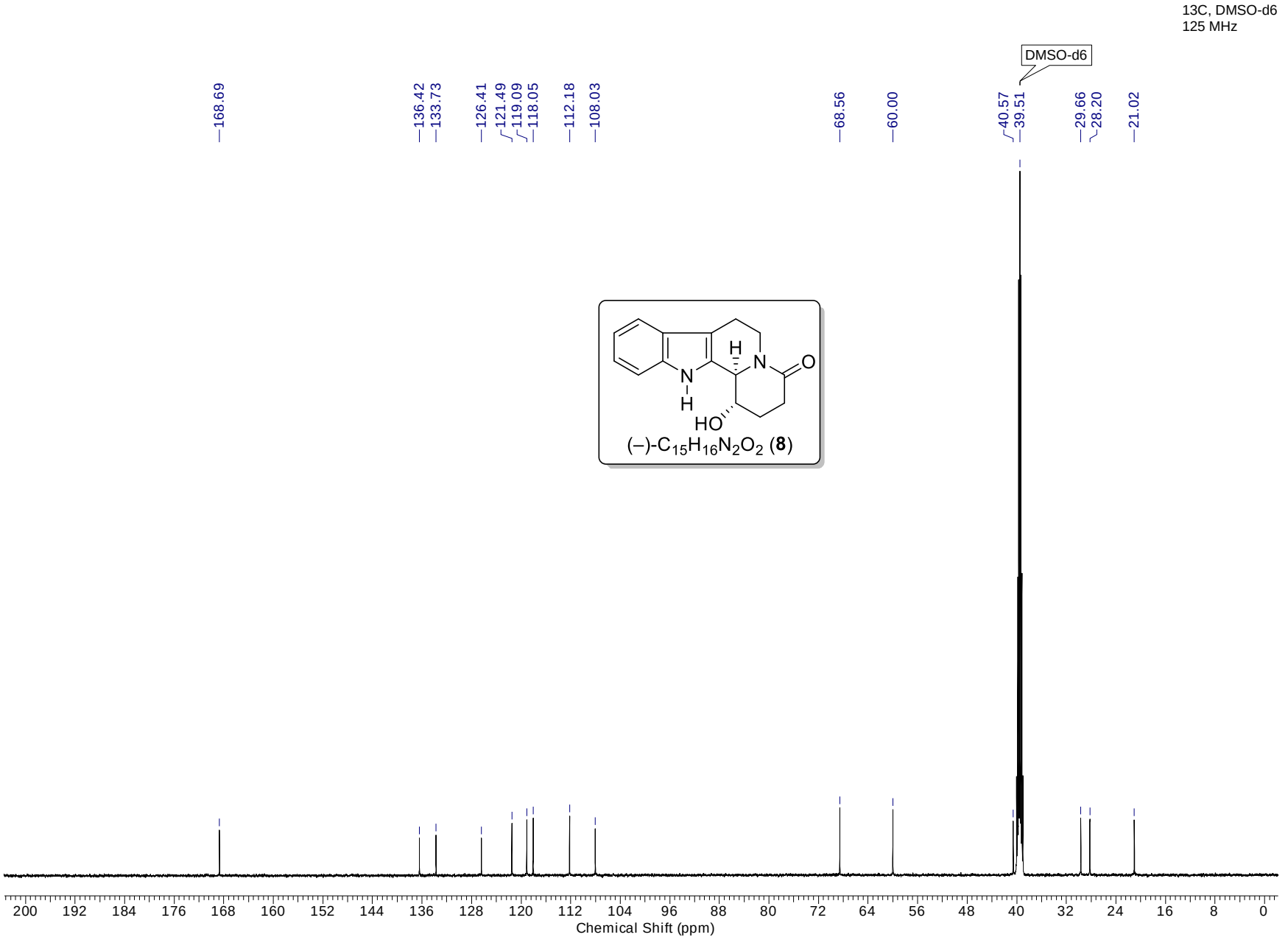

1H, CDCl3, 200 MHz
CHLOROFORM-d
8.10 8.06 7.40 7.36 7.35 7.33 7.27 7.26 7.25 7.23 7.07 7.03 5.90 5.88 5.87 5.86 5.85 5.27 5.25 5.24 5.08 5.07 5.02 5.00 2.80 2.73 2.69 2.68 2.62 2.59 2.56 2.53 2.52 2.46 2.43 2.28 2.21 2.05 1.74 1.73 1.72
(+)-C24H24N2O5S (7)
1.02
1.01
1.00
Chemical Shift (ppm)
1.00 3.10 2.01 2.04 1.02 1.01 1.00 3.16 2.07 3.07 3.11 1.00 1.02
Chemical Shift (ppm)

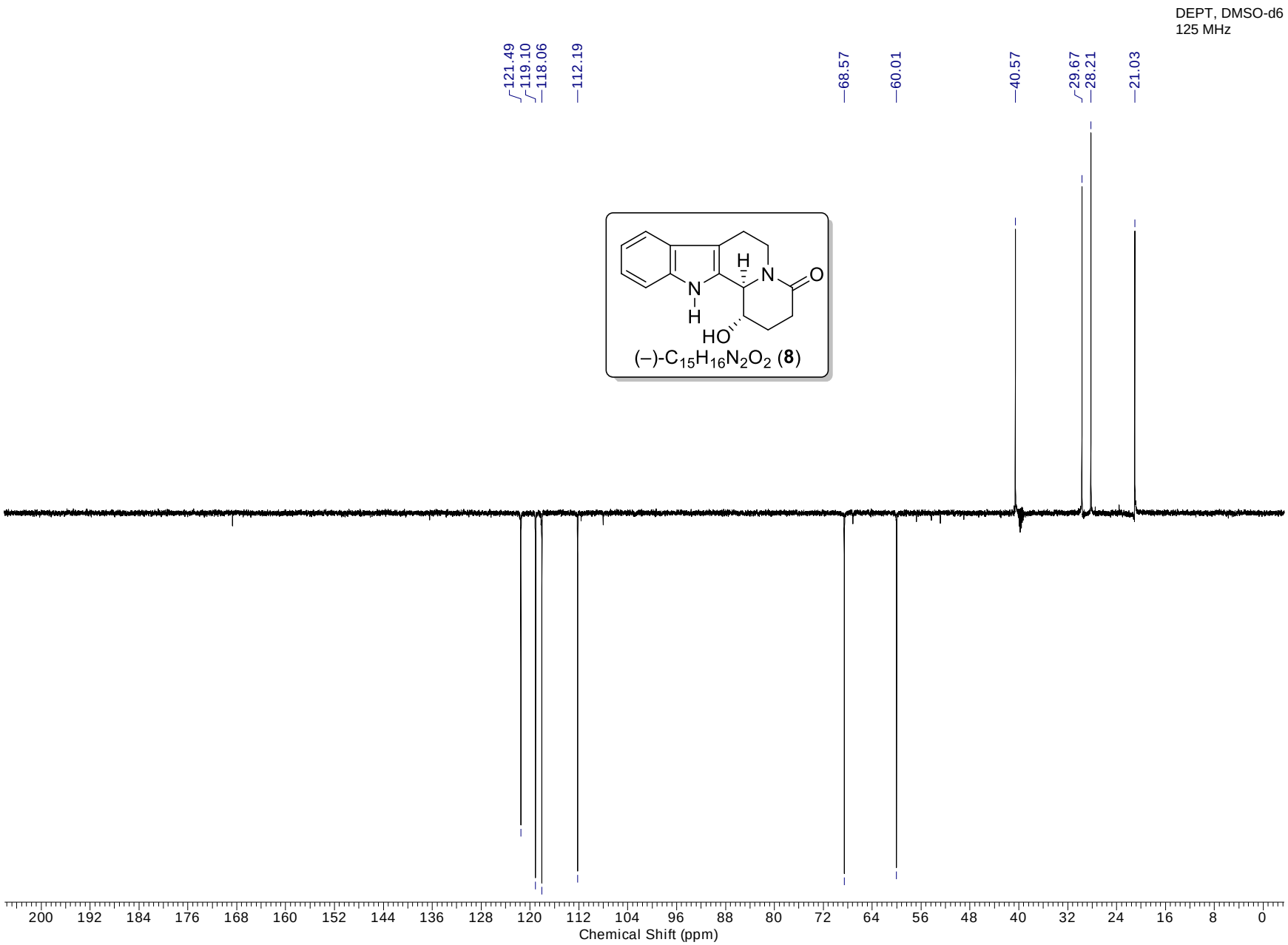

1H, CDCl3, 200 MHz
2.82
2.80
2.78
2.73
2.71
2.69
2.68
2.65
2.62
2.59
2.56
2.54
2.53
2.52
2.47
2.46
2.43
2.39
2.37
2.36
2.35
2.28
2.21
2.18
2.16
2.14
2.13
2.12
2.10
2.05
1.82
1.81
1.79
1.78
1.75
1.74
1.73
1.72
1.72
1.69
H
N
N
O
Ts
AcO
(+)-C24H24N2O5S (7)
3.16
2.07
3.07
3.11
1.00
1.02
2.9
2.8
2.7
2.6
2.5
2.4
2.3
2.2
2.1
2.0
1.9
1.8
1.7
1.6
1.5
Chemical Shift (ppm)

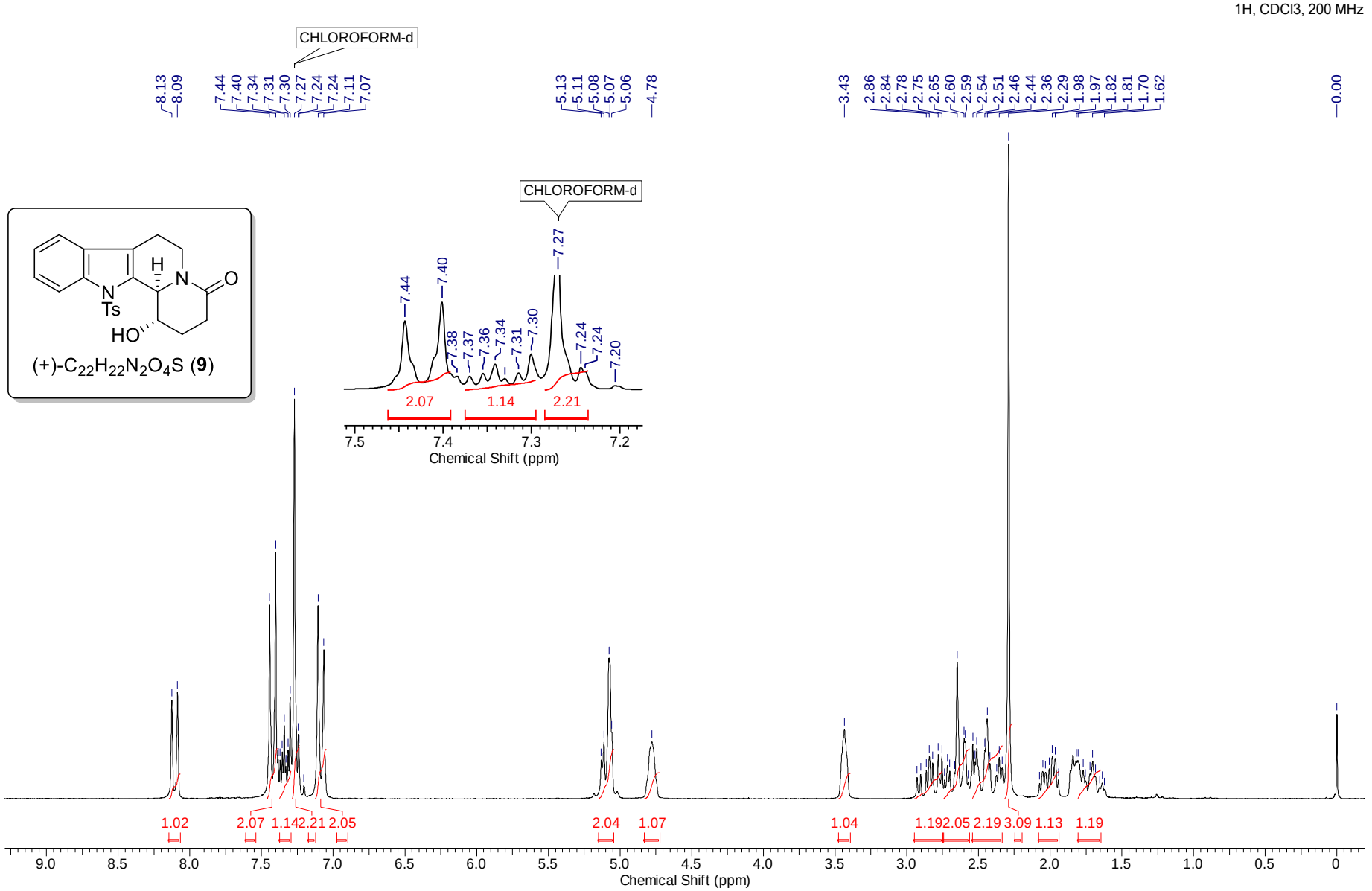

13C, CDCl3, 50 MHz
CHLOROFORM-d
170.07
169.72
144.95
138.78
133.03
132.30
130.77
129.17
127.27
126.67
125.70
124.93
118.71
117.01
77.64
77.00
76.37
71.31
60.36
40.74
27.60
23.40
21.94
21.50
21.14
Ts
AcO
(+)-C24H24N2O5S (7)
21.94
21.50
21.14
22.0
21.5
21.0
Chemical Shift (ppm)
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

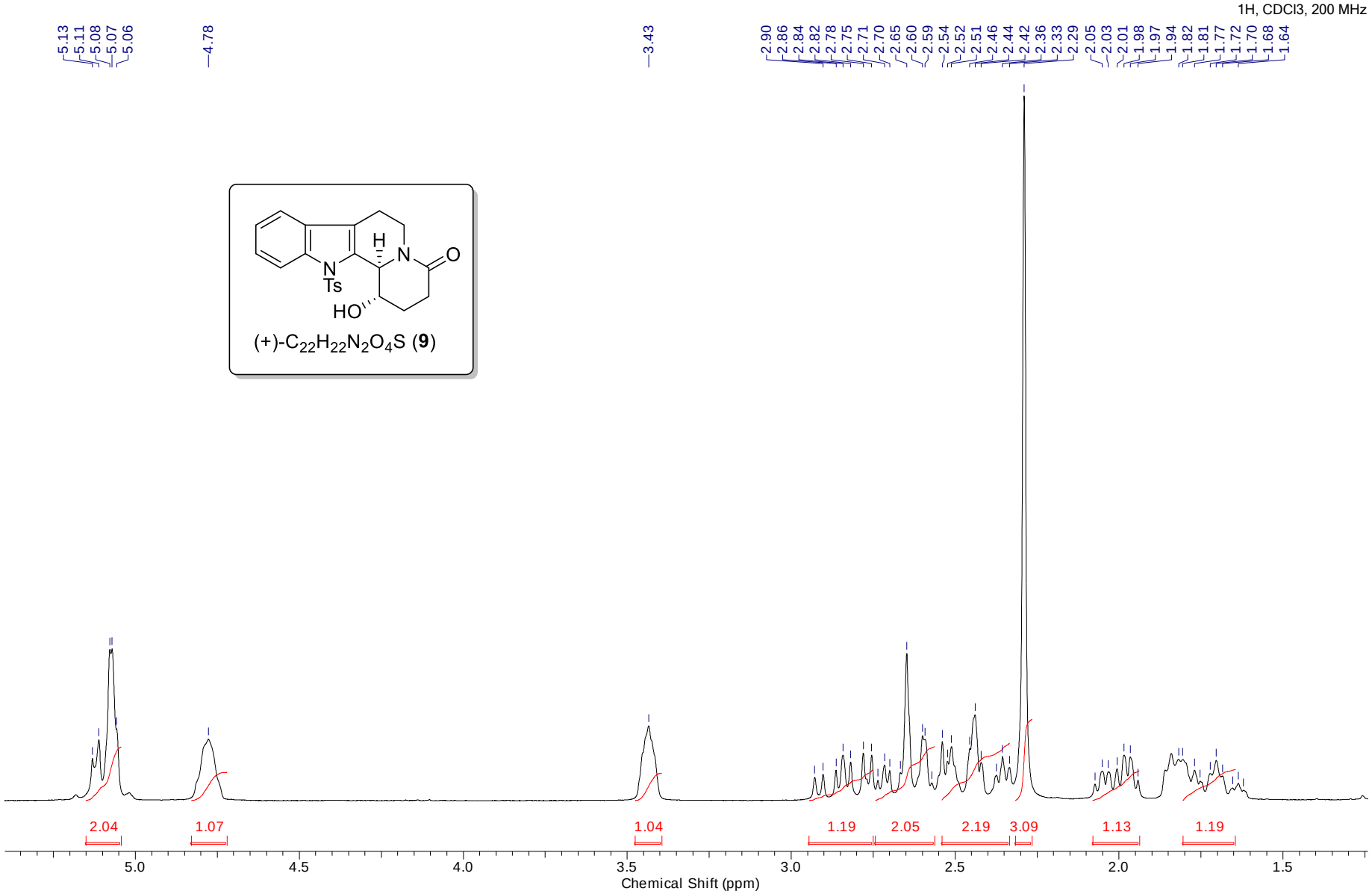

DEPT, CDCl3, 50 MHz
129.09
126.59
125.63
124.86
118.64
116.94
71.21
60.30
40.67
27.50
23.29
21.86
21.43
21.07
H
N
O
N
Ts
AcO
(+)-C24H24N2O5S (7)
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

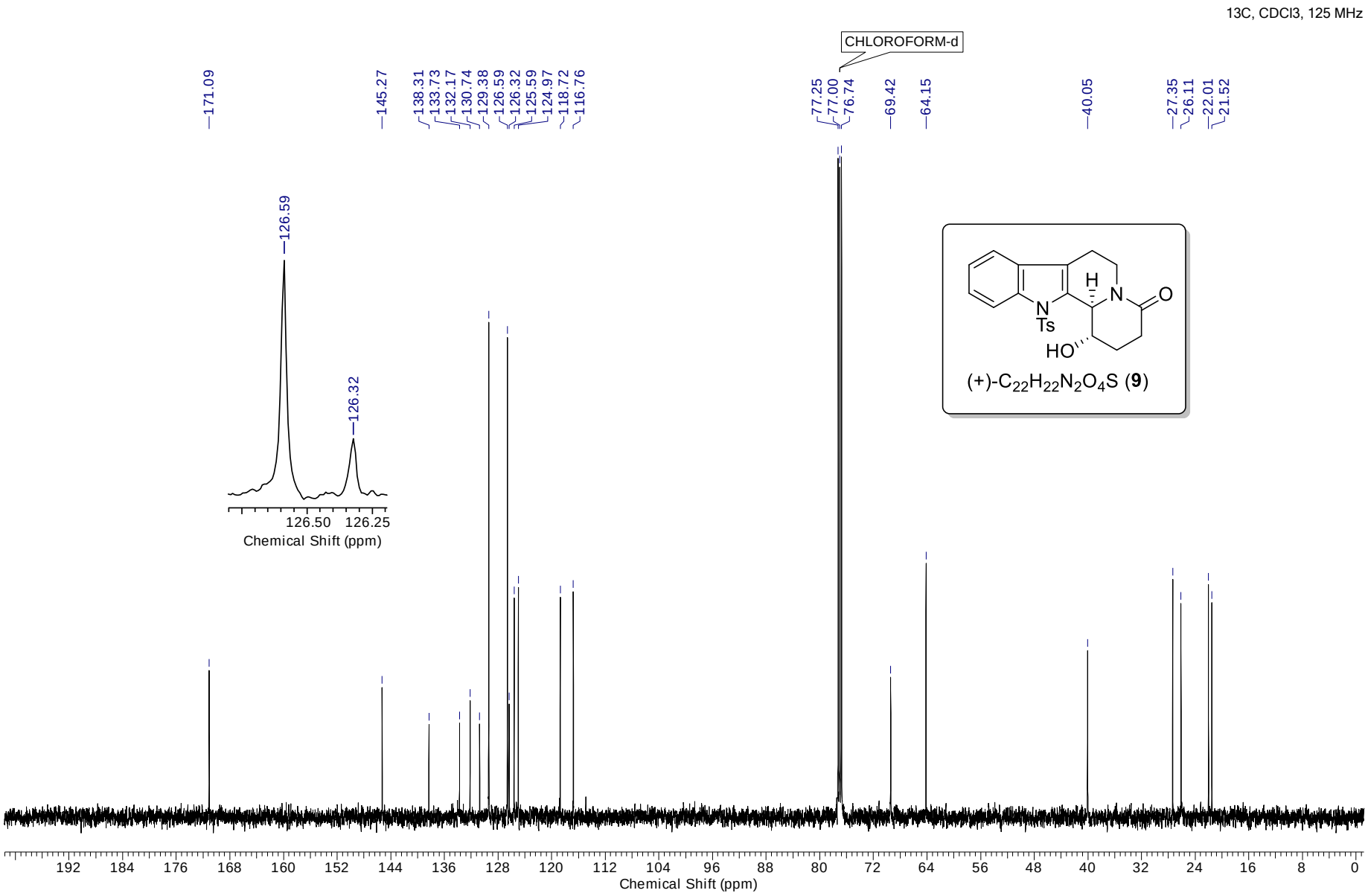

1H, DMSO-d6
500 MHz
DMSO-d6
10.38
7.43 7.41 7.39 7.37 7.05 7.04 7.02 6.97 6.96 6.94
5.90 5.89
4.82 4.81 4.79 4.78 4.58 4.56
3.91 3.90 3.89
2.79 2.78 2.64 2.61 2.50 2.45 2.42 2.41 2.31 2.29
1.86 1.84 1.83 1.81
H
N
O
N
H
HO
(–)-C15H16N2O2 (8)
0.96 0.98 1.04 1.00 1.03 0.93 1.01 1.01 1.20 0.94 2.01 0.94 0.90 2.01
11.5 11.0 10.5 10.0 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
Chemical Shift (ppm)
0.98 1.04 1.00 1.03
7.4 7.3 7.2 7.1 7.0 6.9
Chemical Shift (ppm)

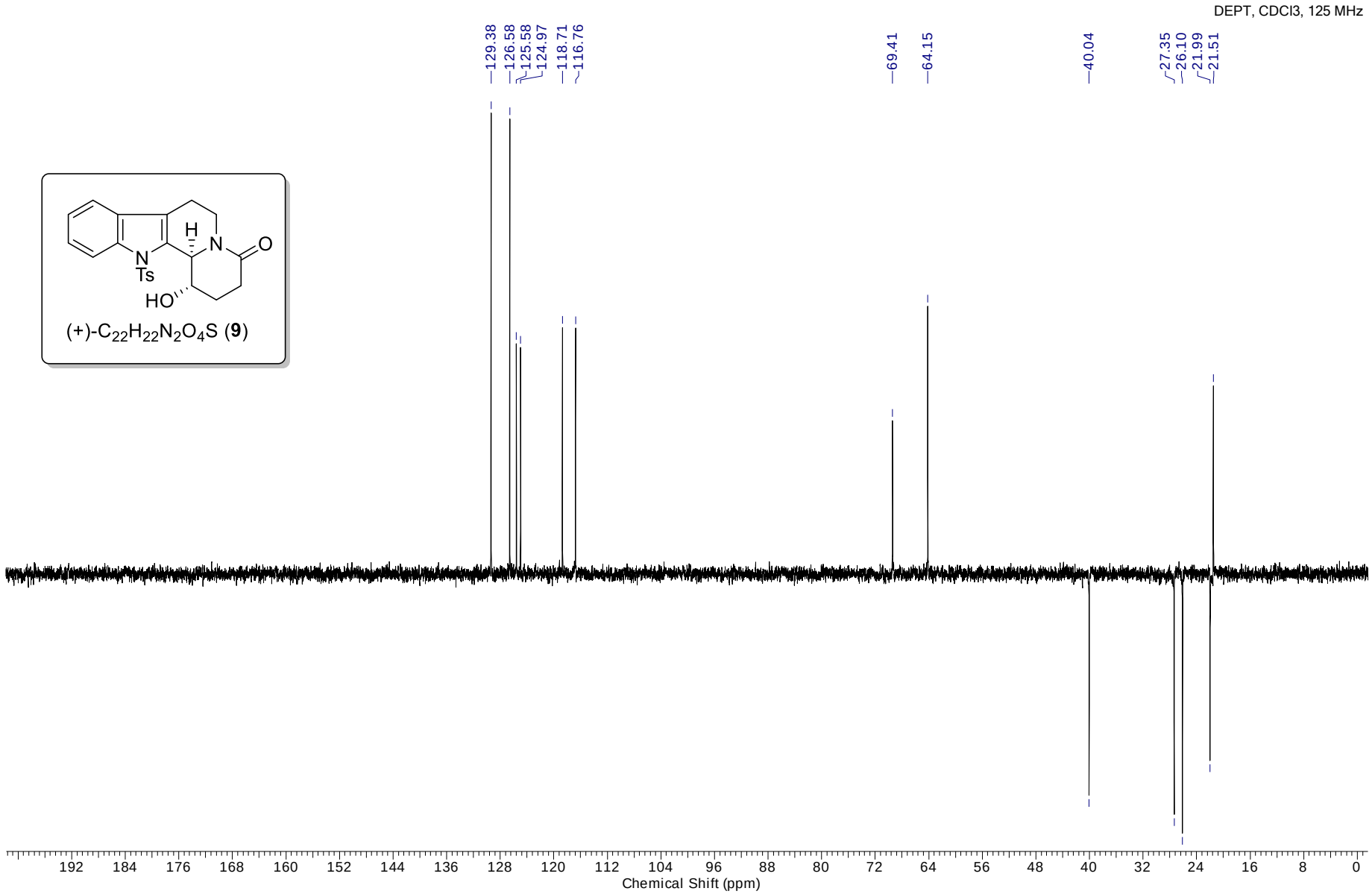

13C, DMSO-d6
125 MHz
DMSO-d6
168.69
136.42
133.73
126.41
121.49
119.09
118.05
112.18
108.03
68.56
60.00
40.57
39.51
29.66
28.20
21.02
(–)-C15H16N2O2 (8)
Chemical Shift (ppm)

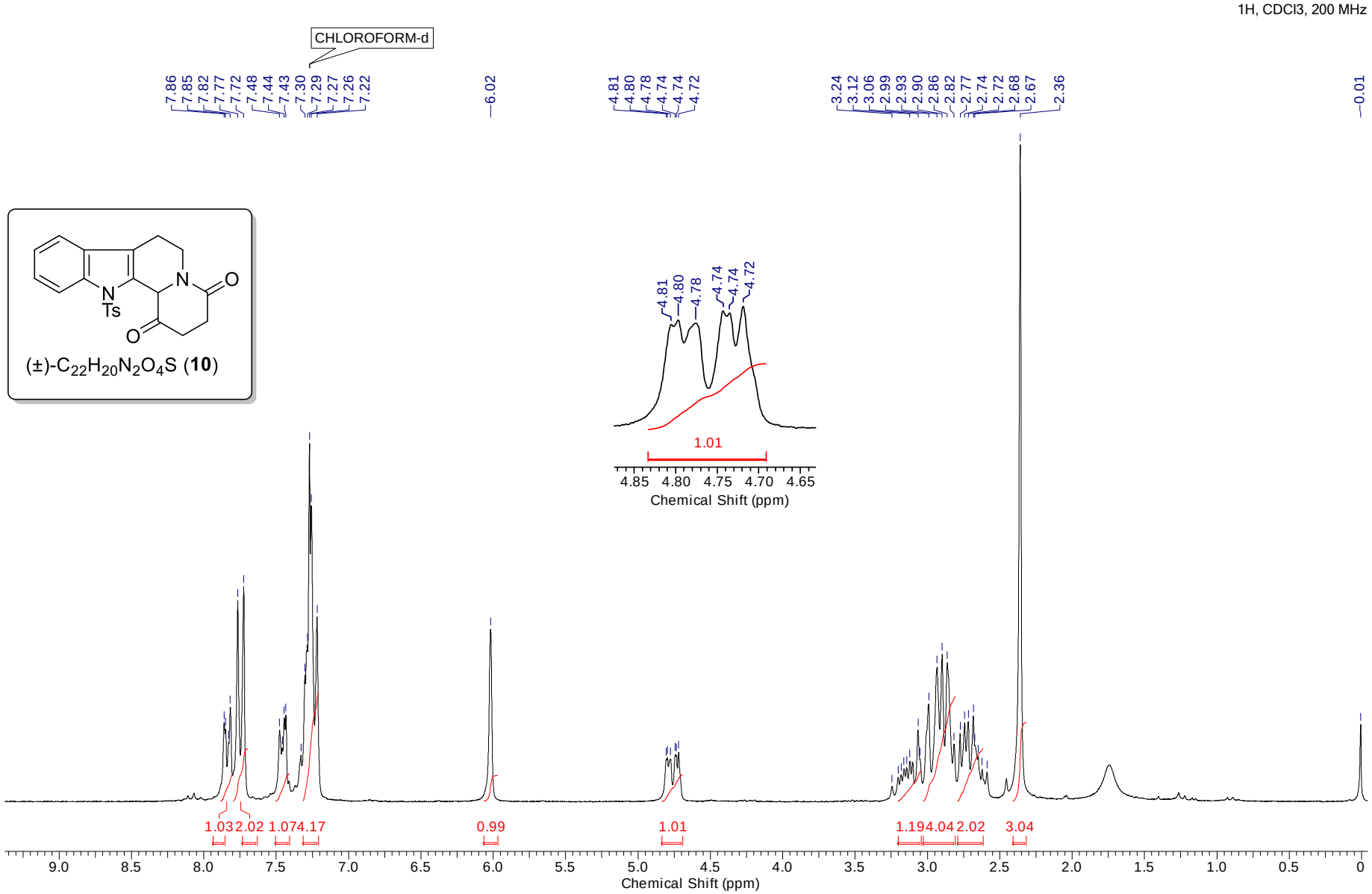

DEPT, DMSO-d6
125 MHz
121.49
119.10
118.06
112.19
68.57
60.01
40.57
29.67
28.21
21.03
(–)-C15H16N2O2 (8)
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

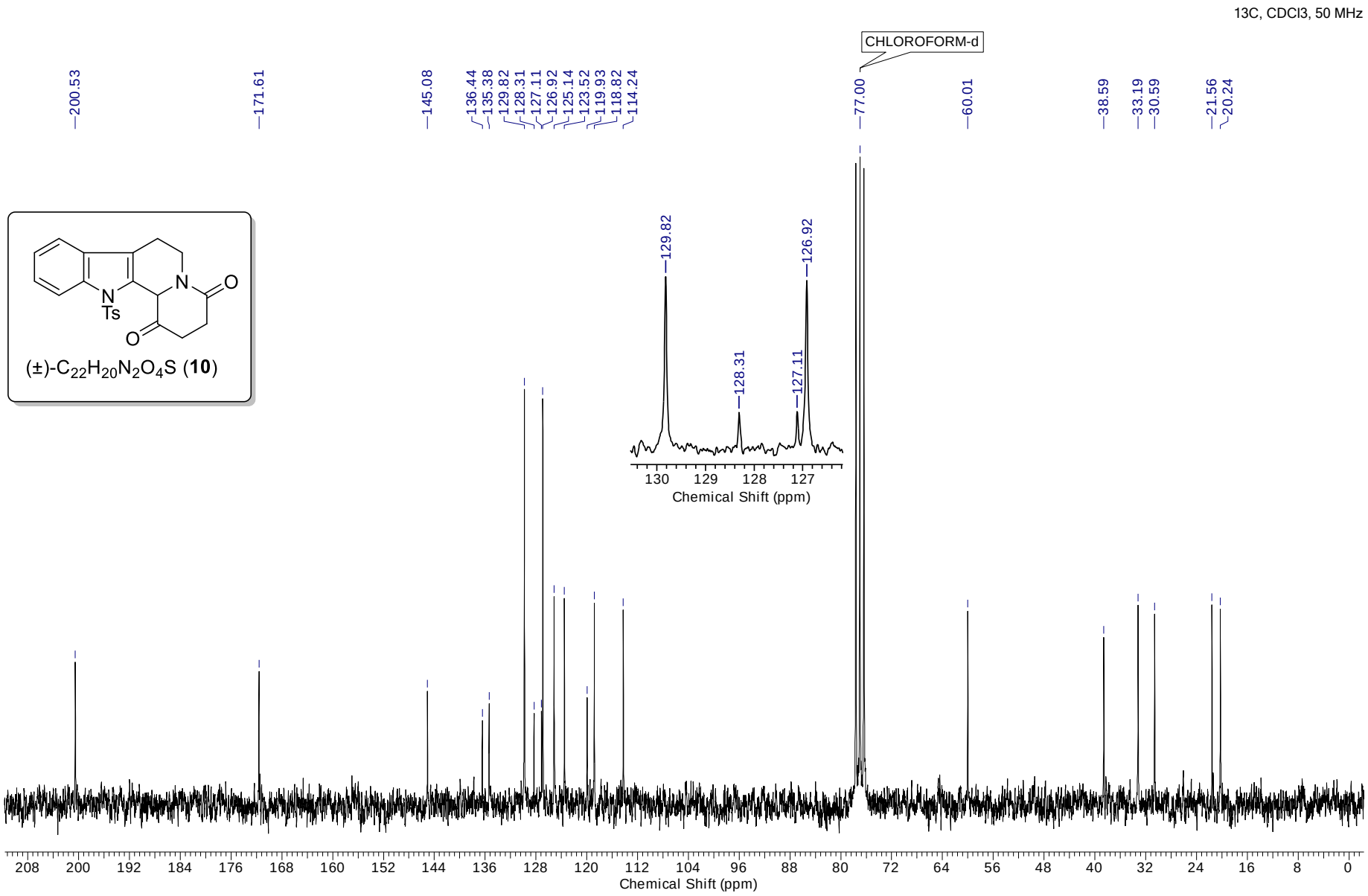

1H, CDCl3, 200 MHz
CHLOROFORM-d
8.13
8.09
7.44
7.40
7.34
7.31
7.30
7.27
7.24
7.24
7.11
7.07
5.13
5.11
5.08
5.07
5.06
4.78
3.43
2.86
2.84
2.78
2.75
2.65
2.60
2.59
2.54
2.51
2.46
2.44
2.36
2.29
1.98
1.97
1.82
1.81
1.70
1.62
0.00
(+)-C22H22N2O4S (9)
CHLOROFORM-d
7.44
7.40
7.38
7.37
7.36
7.34
7.31
7.30
7.27
7.24
7.24
7.20
2.07
1.14
2.21
Chemical Shift (ppm)
1.02
2.07
1.14
2.21
2.05
2.04
1.07
1.04
1.19
2.05
2.19
3.09
1.13
1.19
Chemical Shift (ppm)

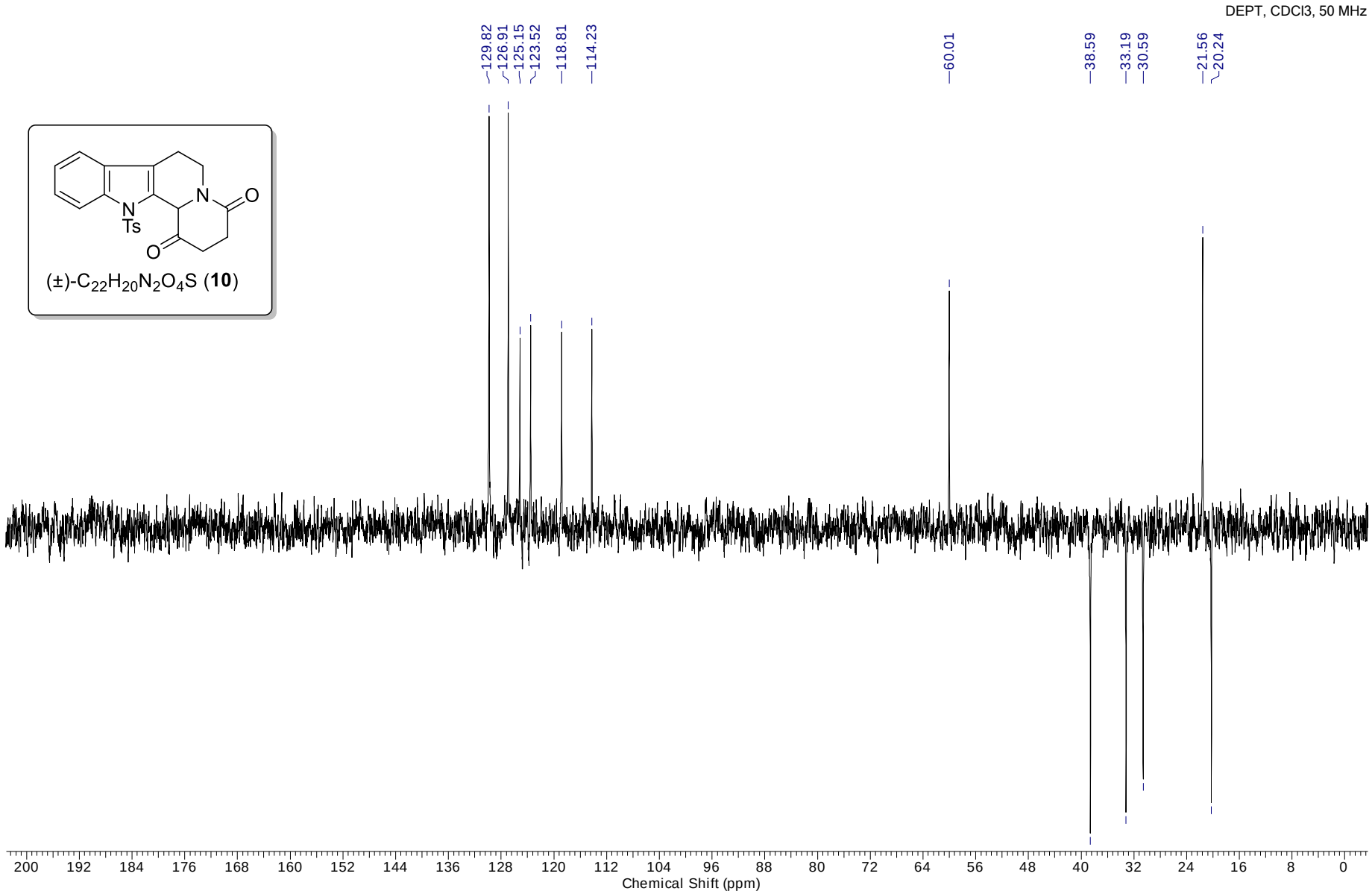

1H, CDCl3, 200 MHz
5.13
5.11
5.08
5.07
5.06
4.78
3.43
2.90
2.86
2.84
2.82
2.78
2.75
2.71
2.70
2.65
2.60
2.59
2.54
2.52
2.51
2.46
2.44
2.42
2.36
2.33
2.29
2.05
2.03
2.01
1.98
1.97
1.94
1.82
1.81
1.77
1.72
1.70
1.68
1.64
H
N
O
N
Ts
HO
(+)-C22H22N2O4S (9)
2.04
1.07
1.04
1.19
2.05
2.19
3.09
1.13
1.19
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
Chemical Shift (ppm)

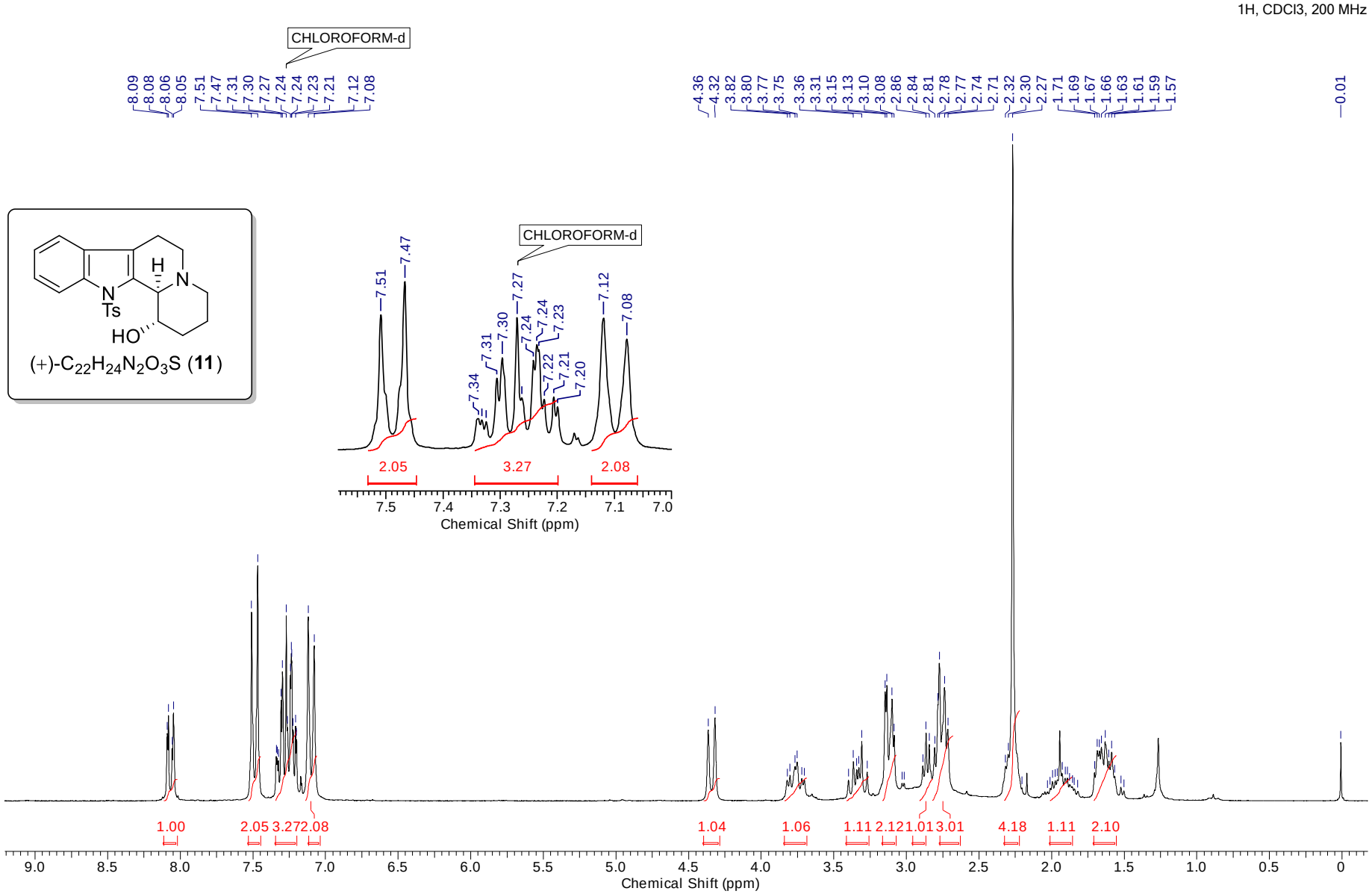

13C, CDCl3, 125 MHz
CHLOROFORM-d
171.09
145.27
138.31
133.73
132.17
130.74
129.38
126.59
126.32
125.59
124.97
118.72
116.76
77.25
77.00
76.74
69.42
64.15
40.05
27.35
26.11
22.01
21.52
126.59
126.32
126.50
126.25
Chemical Shift (ppm)
(+)-C22H22N2O4S (9)
Chemical Shift (ppm)

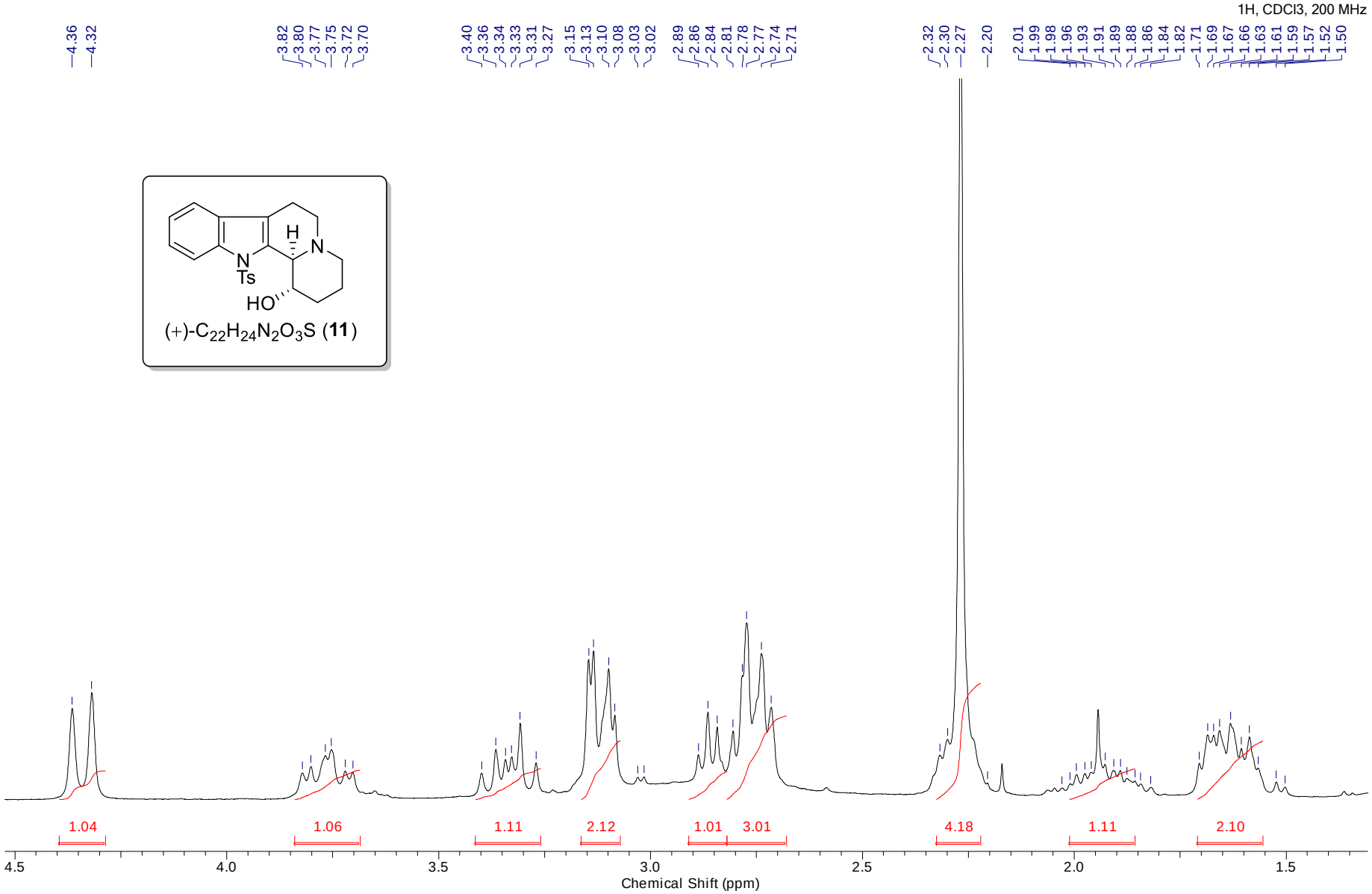

DEPT, CDCl3, 125 MHz
129.38
126.58
125.58
124.97
118.71
116.76
69.41
64.15
40.04
27.35
26.10
21.99
21.51
H
N
O
N
Ts
HO
(+)-C22H22N2O4S (9)
192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

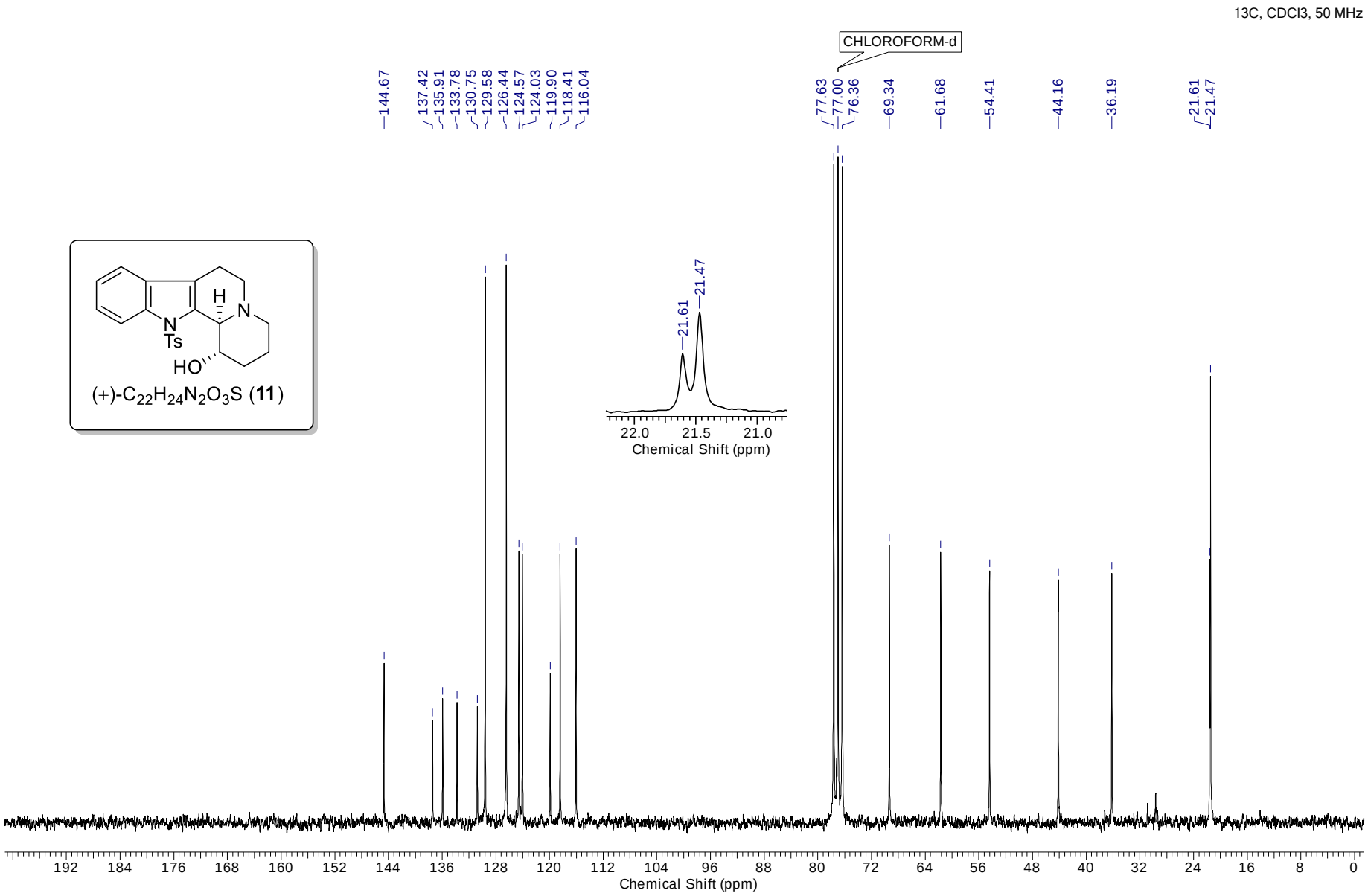

1H, CDCl3, 200 MHz
CHLOROFORM-d
7.86 7.85 7.82 7.77 7.72 7.48 7.44 7.43 7.30 7.29 7.27 7.26 7.22
6.02
4.81 4.80 4.78 4.74 4.74 4.72
3.24 3.12 3.06 2.99 2.93 2.90 2.86 2.82 2.77 2.74 2.72 2.68 2.67
2.36
0.01
N
Ts
N
O
O
(±)-C22H20N2O4S (10)
1.01
4.85 4.80 4.75 4.70 4.65
Chemical Shift (ppm)
1.03 2.02 1.07 4.17
0.99
1.01
1.19 4.04 2.02
3.04
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
Chemical Shift (ppm)

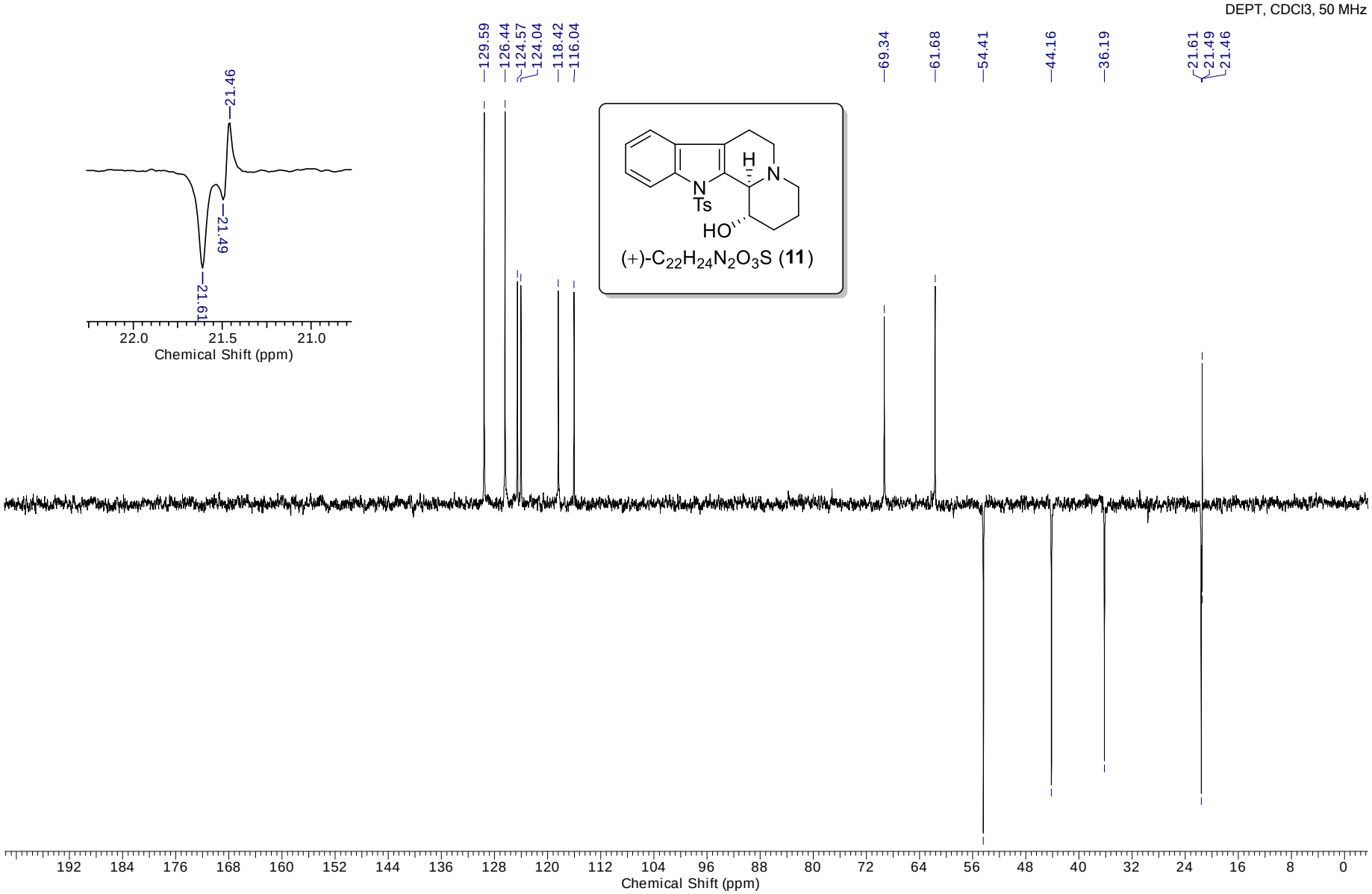

13C, CDCl3, 50 MHz
CHLOROFORM-d
200.53
171.61
145.08
136.44
135.38
129.82
128.31
127.11
126.92
125.14
123.52
119.93
118.82
114.24
77.00
60.01
38.59
33.19
30.59
21.56
20.24
N
Ts
N
O
O
(±)-C22H20N2O4S (10)
129.82
128.31
127.11
126.92
130 129 128 127
Chemical Shift (ppm)
208 200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

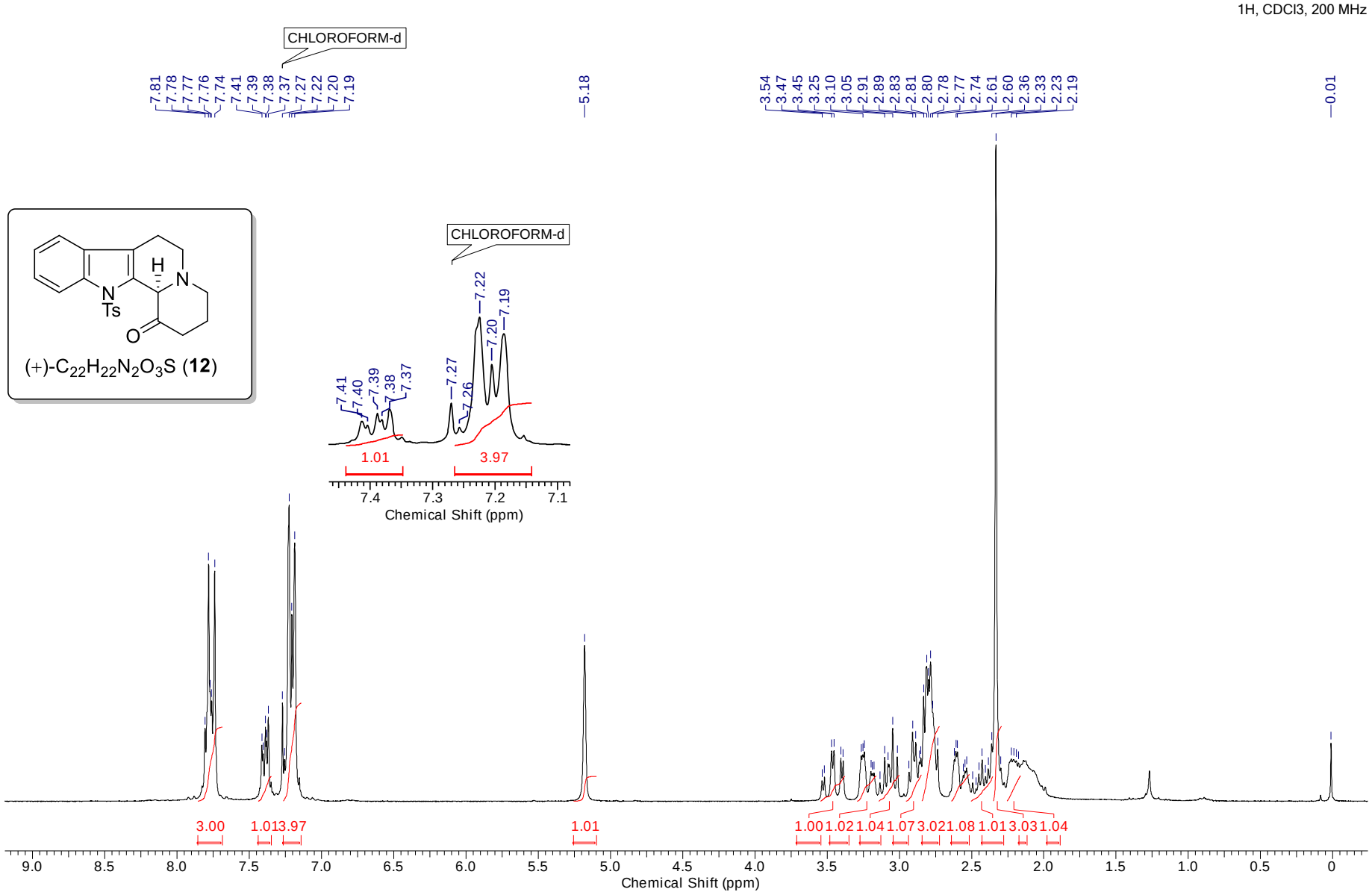

DEPT, CDCl3, 50 MHz
129.82
126.91
125.15
123.52
118.81
114.23
60.01
38.59
33.19
30.59
21.56
20.24
N
Ts
N
O
O
(±)-C22H20N2O4S (10)
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

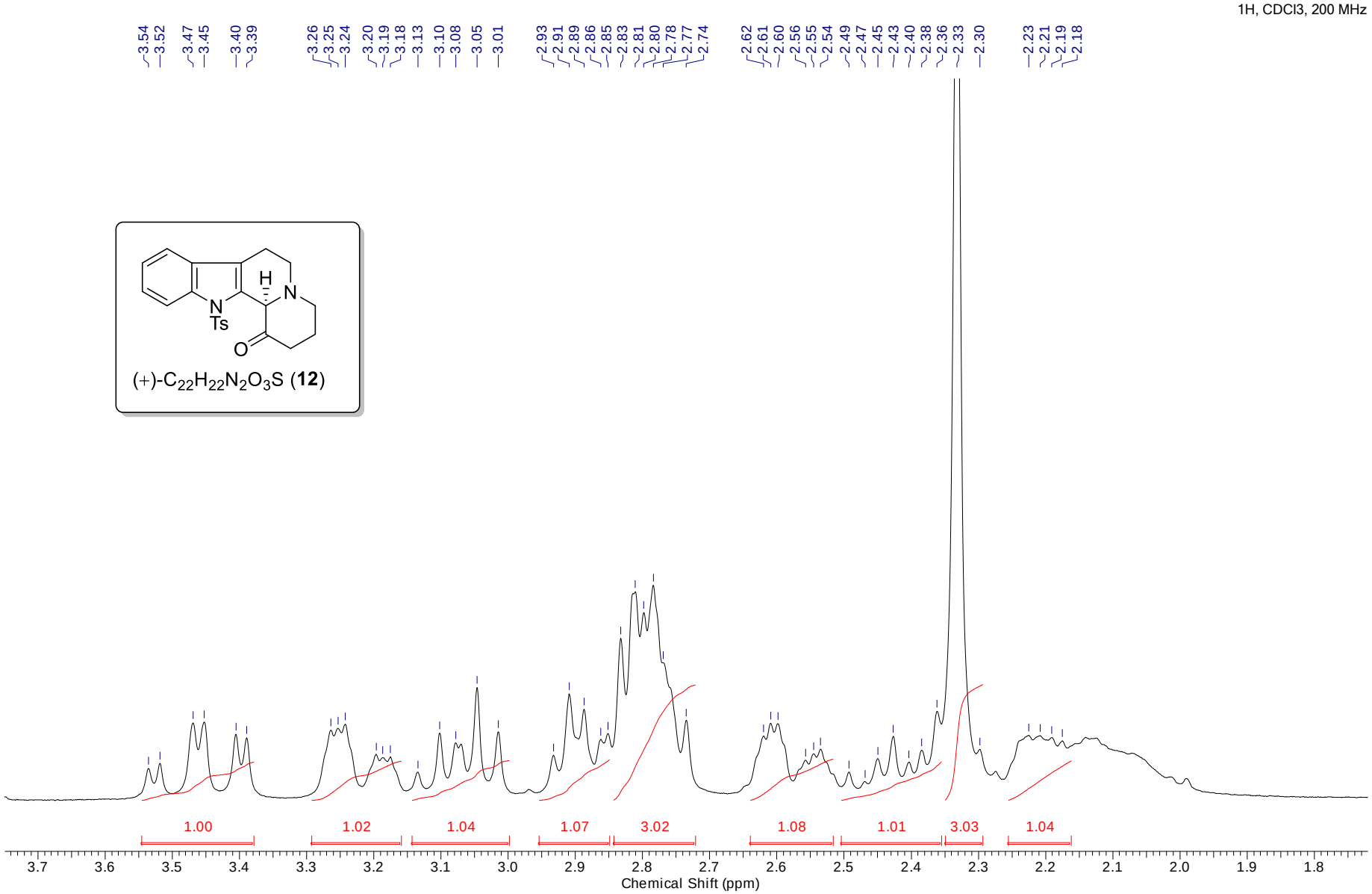

1H, CDCl3, 200 MHz
CHLOROFORM-d
8.09
8.08
8.06
8.05
7.51
7.47
7.31
7.30
7.27
7.24
7.24
7.23
7.21
7.12
7.08
4.36
4.32
3.82
3.80
3.77
3.75
3.36
3.31
3.15
3.13
3.10
3.08
2.86
2.84
2.81
2.78
2.77
2.74
2.71
2.32
2.30
2.27
1.71
1.69
1.67
1.66
1.63
1.61
1.59
1.57
0.01
(+)-C22H24N2O3S (11)
CHLOROFORM-d
7.51
7.47
7.34
7.31
7.30
7.27
7.24
7.24
7.23
7.22
7.21
7.20
7.12
7.08
2.05
3.27
2.08
7.5
7.4
7.3
7.2
7.1
7.0
Chemical Shift (ppm)
1.00
2.05
3.27
2.08
1.04
1.06
1.11
2.12
1.01
3.01
4.18
1.11
2.10
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
Chemical Shift (ppm)

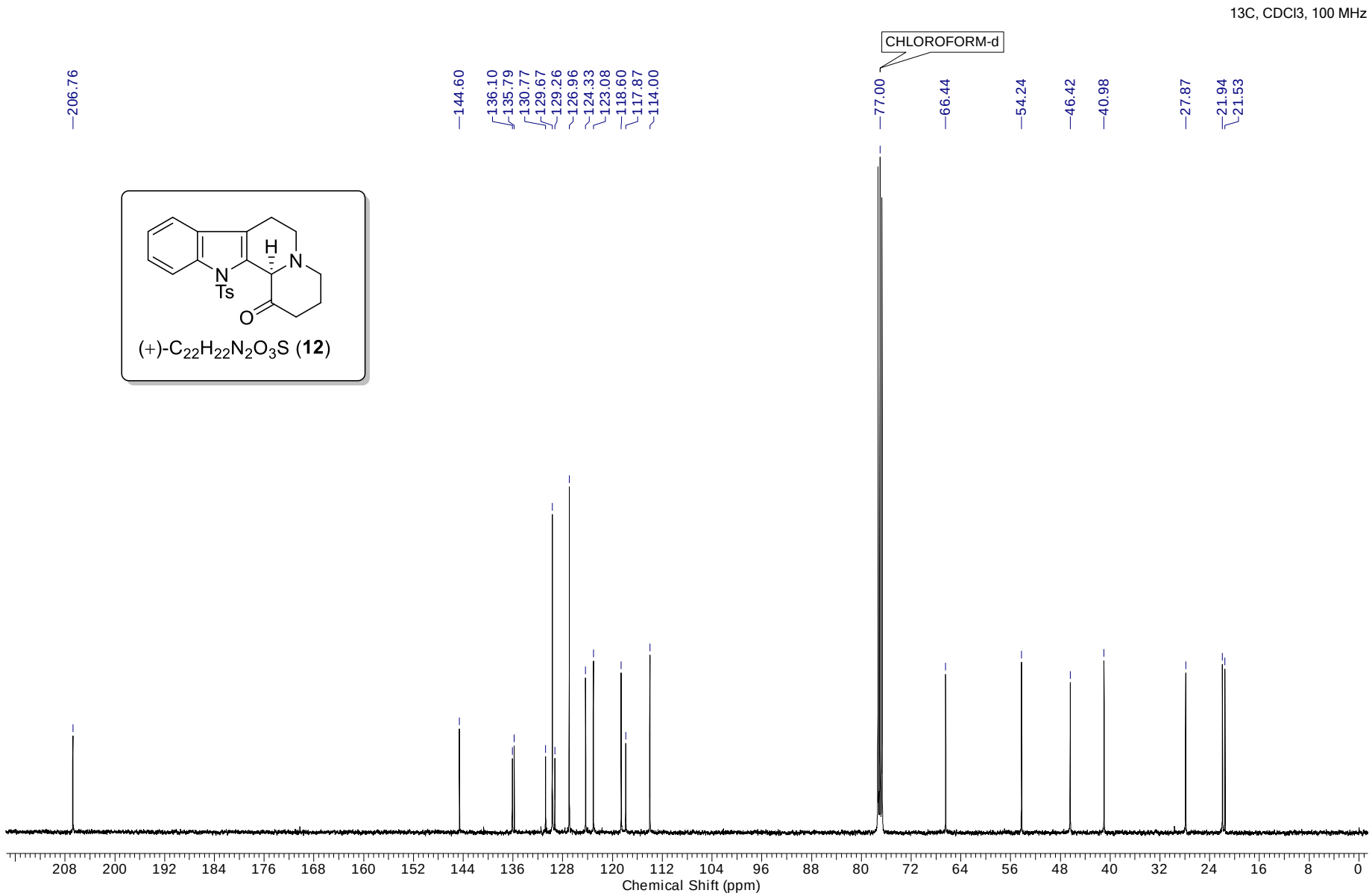

1H, CDCl3, 200 MHz
4.36
4.32
3.82
3.80
3.77
3.75
3.72
3.70
3.40
3.36
3.34
3.33
3.31
3.27
3.15
3.13
3.10
3.08
3.03
3.02
2.89
2.86
2.84
2.81
2.78
2.77
2.74
2.71
2.32
2.30
2.27
2.20
2.01
1.99
1.98
1.96
1.93
1.91
1.89
1.88
1.86
1.84
1.82
1.71
1.69
1.67
1.66
1.63
1.61
1.59
1.57
1.52
1.50
H
N
N
Ts
HO
(+)-C22H24N2O3S (11)
1.04
1.06
1.11
2.12
1.01
3.01
4.18
1.11
2.10
4.5
4.0
3.5
3.0
2.5
2.0
1.5
Chemical Shift (ppm)

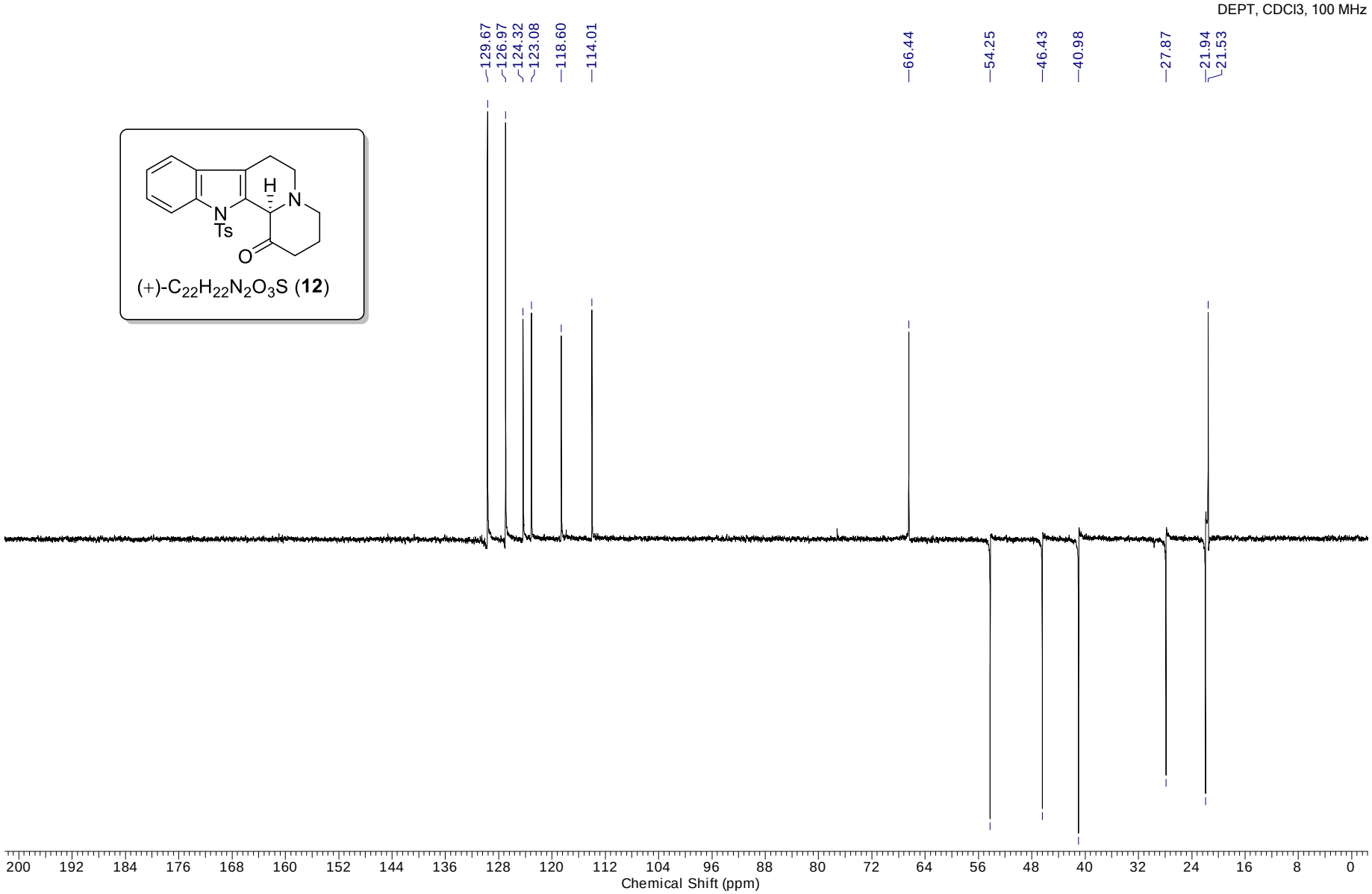

13C, CDCl3, 50 MHz
CHLOROFORM-d
144.67
137.42
135.91
133.78
130.75
129.58
126.44
124.57
124.03
119.90
118.41
116.04
77.63
77.00
76.36
69.34
61.68
54.41
44.16
36.19
21.61
21.47
Ts
HO
(+)-C22H24N2O3S (11)
22.0
21.5
21.0
Chemical Shift (ppm)
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

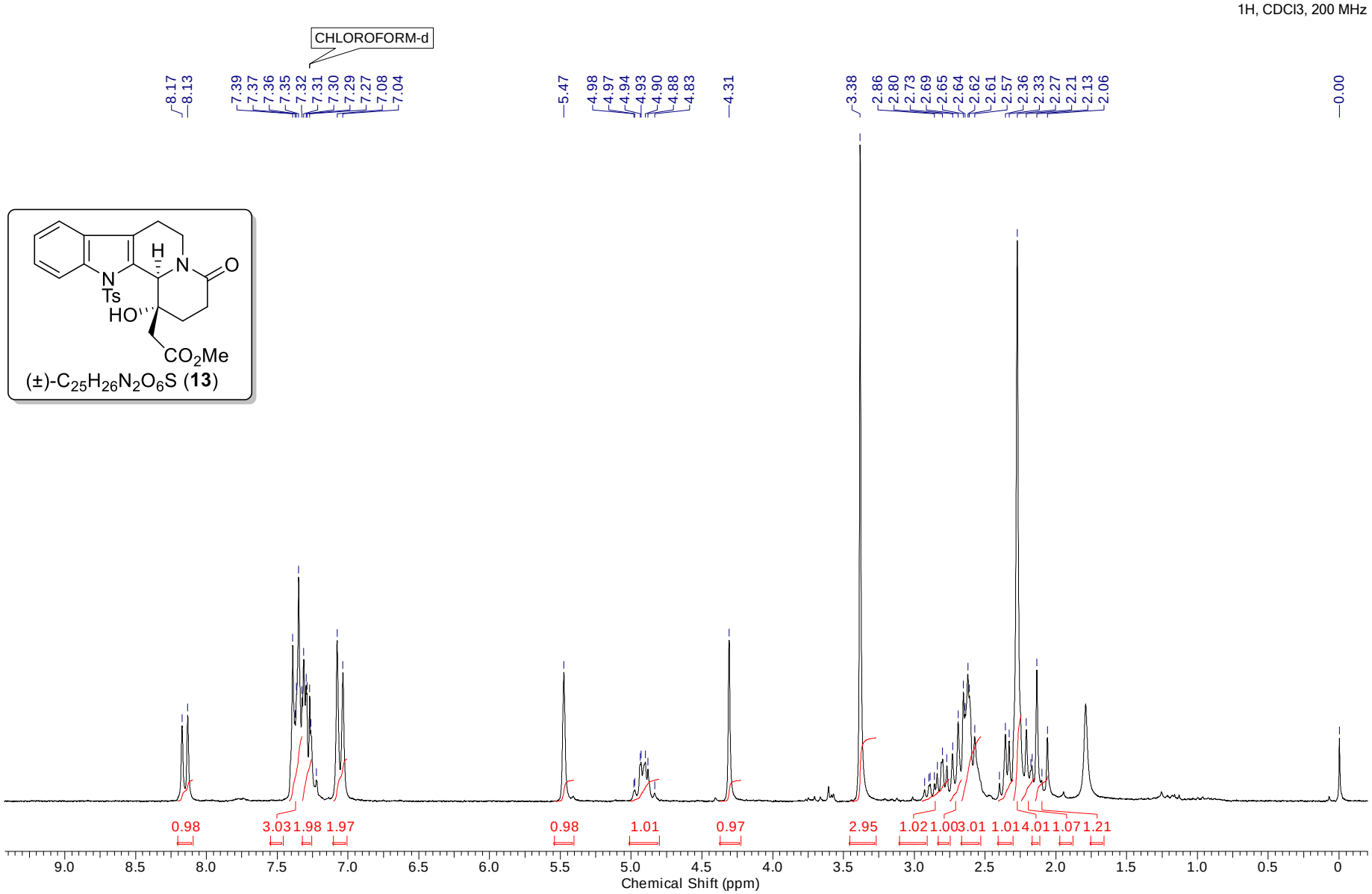

DEPT, CDCl3, 50 MHz
129.59
126.44
124.57
124.04
118.42
116.04
69.34
61.68
54.41
44.16
36.19
21.61
21.49
21.46
21.46
21.49
21.61
22.0
21.5
21.0
Chemical Shift (ppm)
H
N
N
Ts
HO
(+)-C22H24N2O3S (11)
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

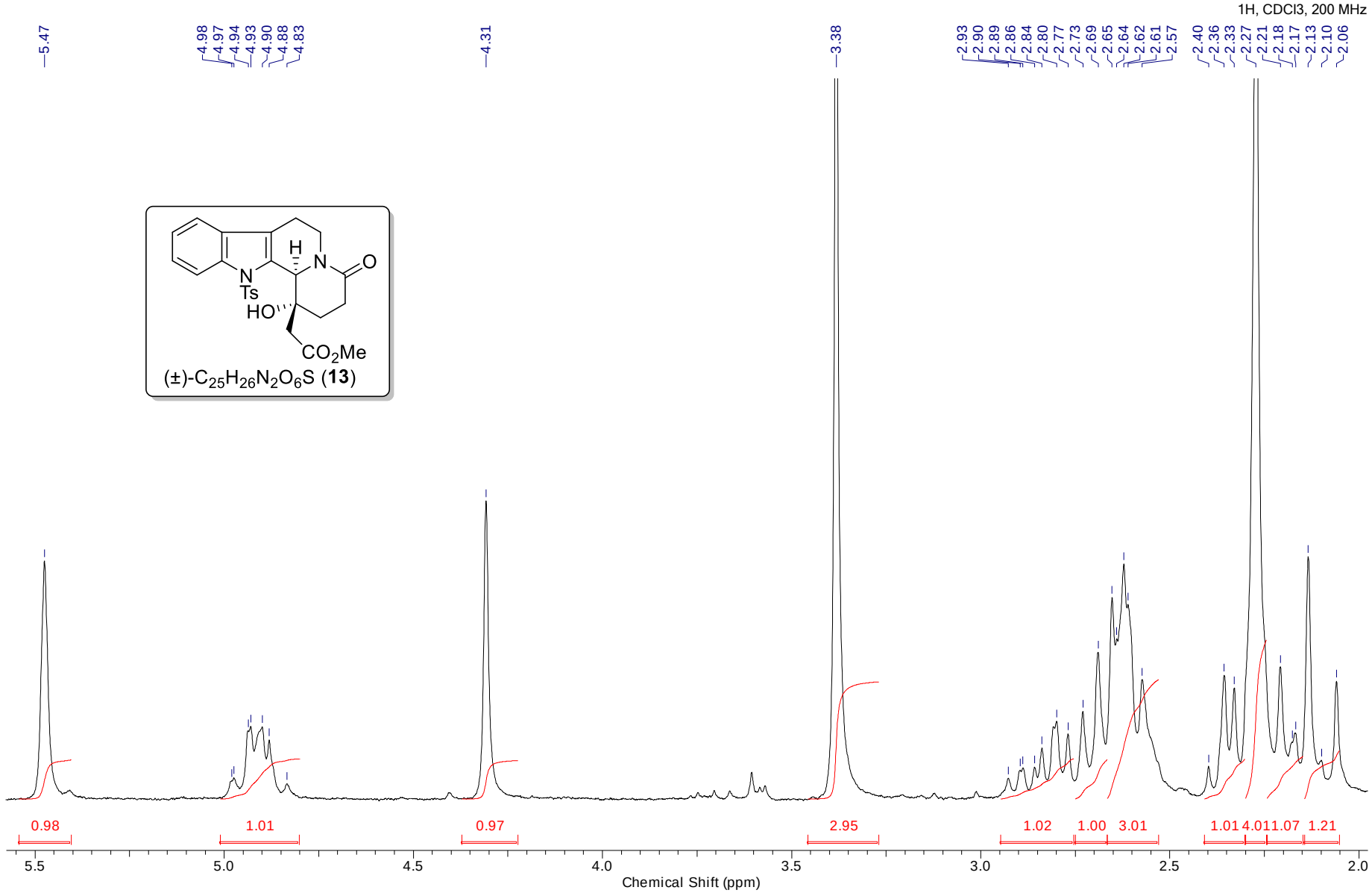

1H, CDCl3, 200 MHz
CHLOROFORM-d
7.81
7.78
7.77
7.76
7.74
7.41
7.39
7.38
7.37
7.27
7.22
7.20
7.19
5.18
3.54
3.47
3.45
3.25
3.10
3.05
2.91
2.89
2.83
2.81
2.80
2.78
2.77
2.74
2.61
2.60
2.36
2.33
2.23
2.19
0.01
(+)-C22H22N2O3S (12)
CHLOROFORM-d
7.41
7.40
7.39
7.38
7.37
7.27
7.26
7.22
7.20
7.19
1.01
3.97
Chemical Shift (ppm)
3.00
1.01
3.97
1.01
1.00
1.02
1.04
1.07
3.02
1.08
1.01
3.03
1.04
Chemical Shift (ppm)

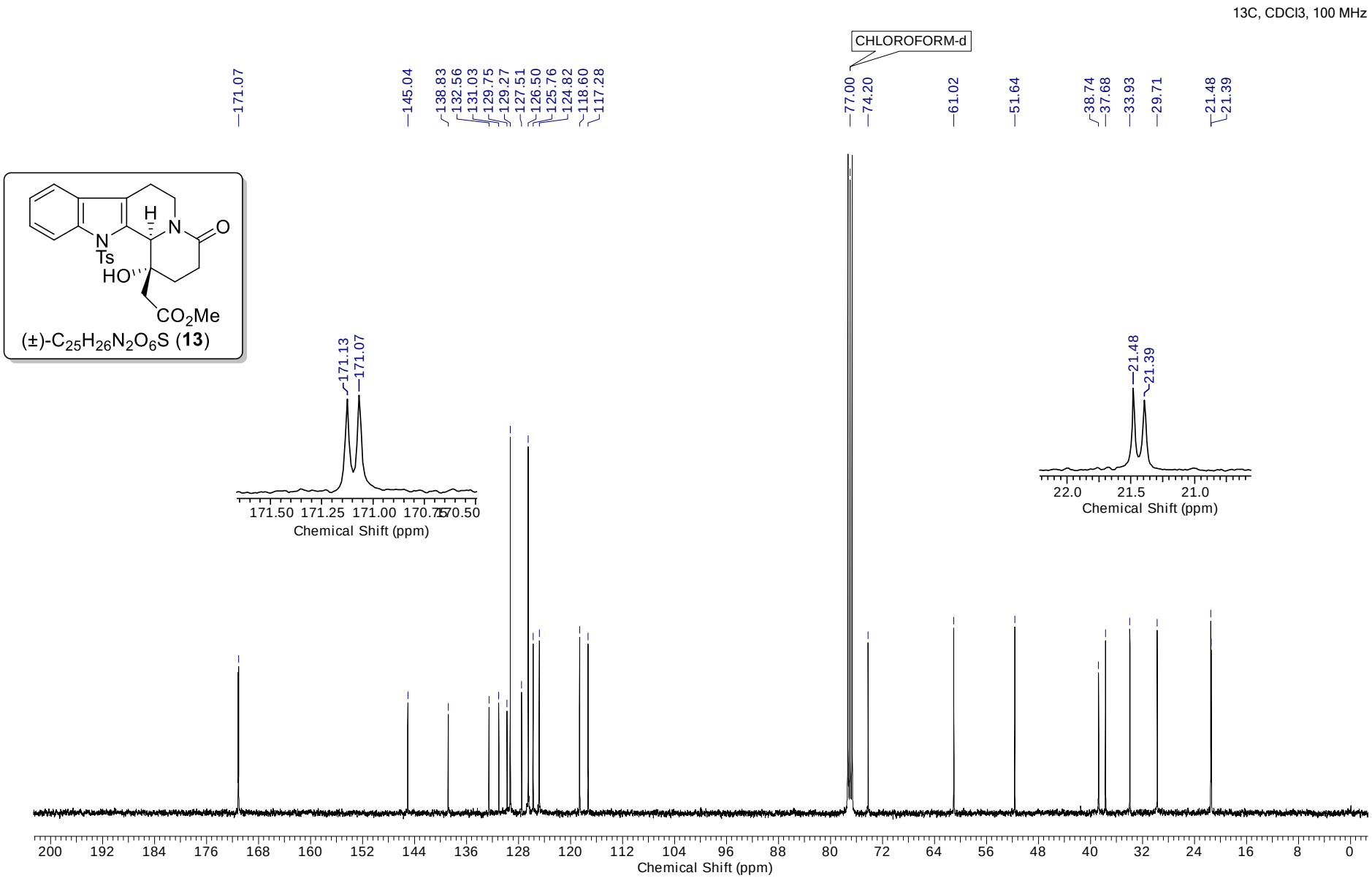

1H, CDCl3, 200 MHz
3.54
3.52
3.47
3.45
3.40
3.39
3.26
3.25
3.24
3.20
3.19
3.18
3.13
3.10
3.08
3.05
3.01
2.93
2.91
2.89
2.86
2.85
2.83
2.81
2.80
2.78
2.77
2.74
2.62
2.61
2.60
2.56
2.55
2.54
2.49
2.47
2.45
2.43
2.40
2.38
2.36
2.33
2.30
2.23
2.21
2.19
2.18
H
N
N
Ts
O
(+)-C22H22N2O3S (12)
1.00
1.02
1.04
1.07
3.02
1.08
1.01
3.03
1.04
3.7
3.6
3.5
3.4
3.3
3.2
3.1
3.0
2.9
2.8
2.7
2.6
2.5
2.4
2.3
2.2
2.1
2.0
1.9
1.8
Chemical Shift (ppm)

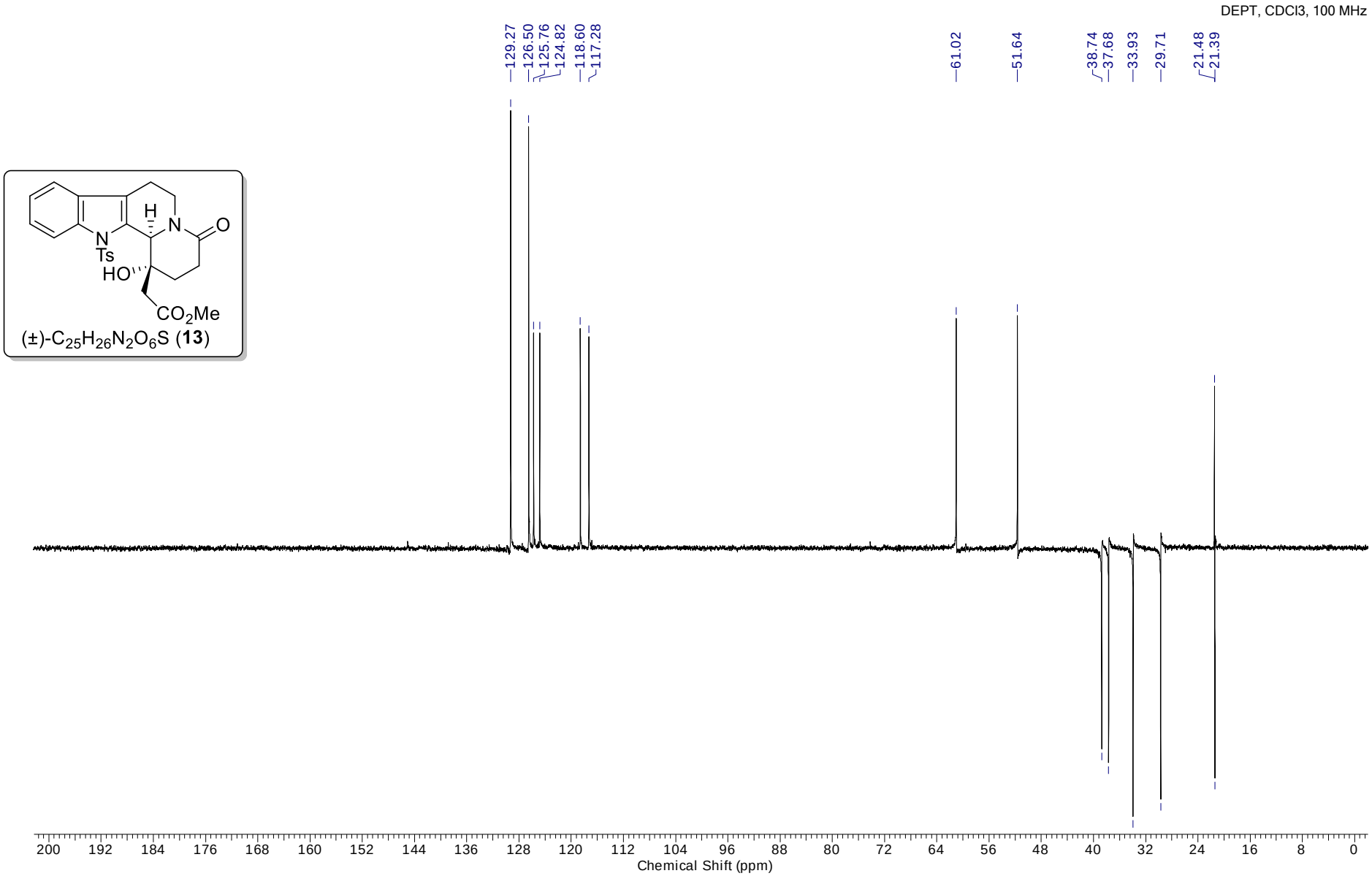

13C, CDCl3, 100 MHz
CHLOROFORM-d
206.76
144.60
136.10
135.79
130.77
129.67
129.26
126.96
124.33
123.08
118.60
117.87
114.00
77.00
66.44
54.24
46.42
40.98
27.87
21.94
21.53
H
N
N
Ts
O
(+)-C22H22N2O3S (12)
208 200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

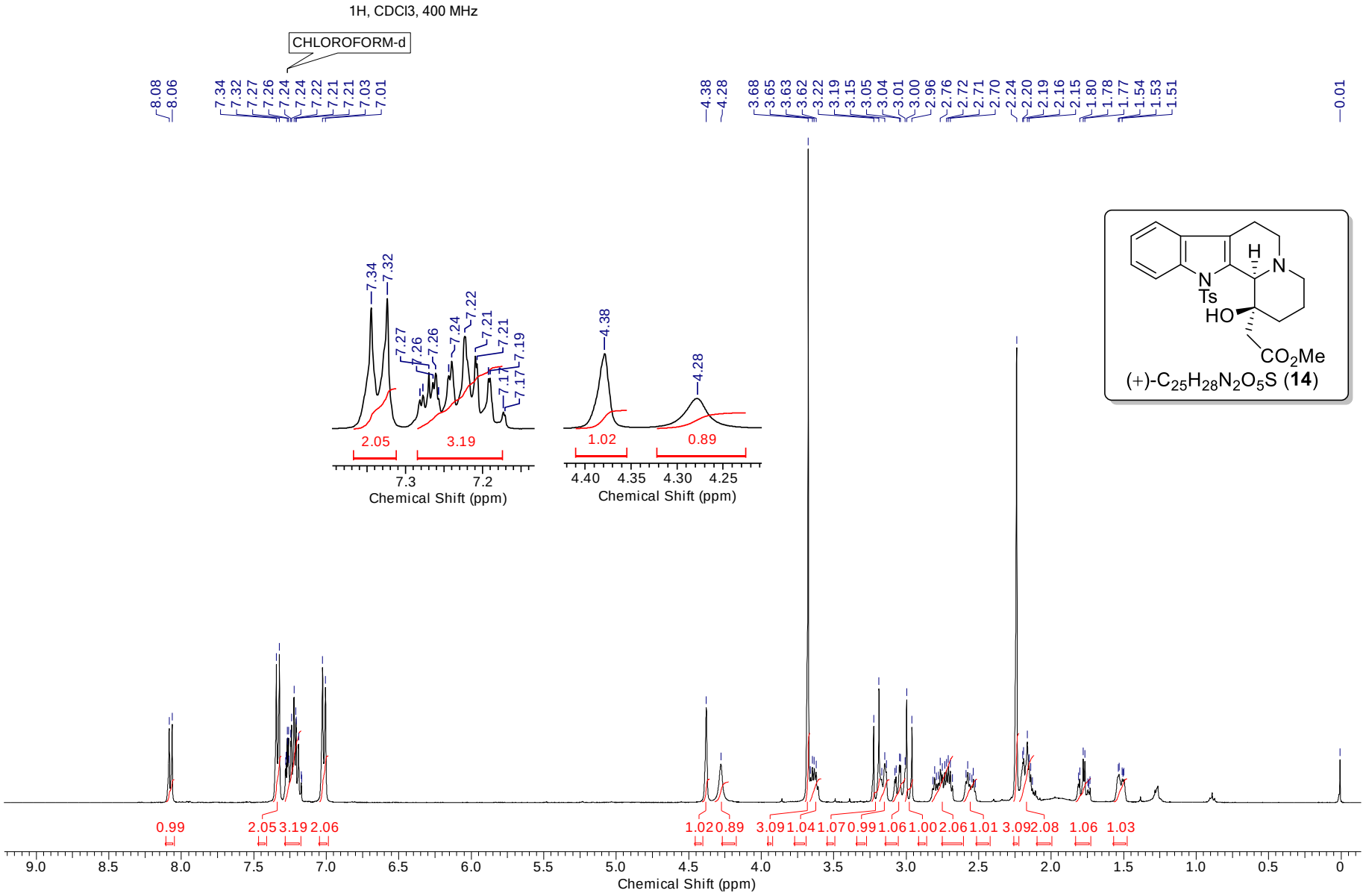

DEPT, CDCl3, 100 MHz
129.67
126.97
124.32
123.08
118.60
114.01
66.44
54.25
46.43
40.98
27.87
21.94
21.53
H
N
N
Ts
O
(+)-C22H22N2O3S (12)
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

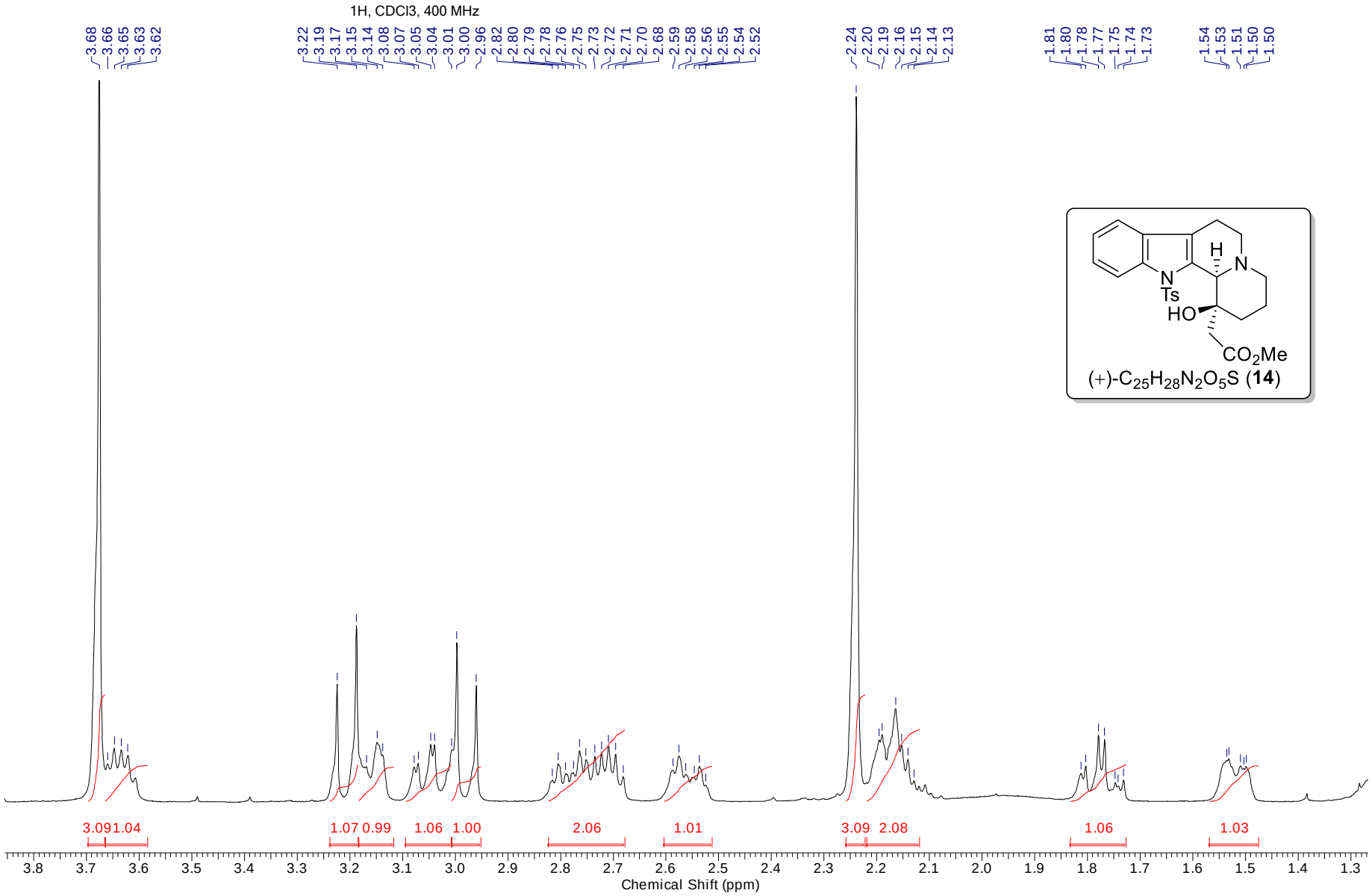

1H, CDCl3, 200 MHz
CHLOROFORM-d
8.17
8.13
7.39
7.37
7.36
7.35
7.32
7.31
7.30
7.29
7.27
7.08
7.04
5.47
4.98
4.97
4.94
4.93
4.90
4.88
4.83
4.31
3.38
2.86
2.80
2.73
2.69
2.65
2.64
2.62
2.61
2.57
2.36
2.33
2.27
2.21
2.13
2.06
0.00
H
N
O
N
Ts
HO
CO2Me
(±)-C25H26N2O6S (13)
0.98
3.03
1.98
1.97
0.98
1.01
0.97
2.95
1.02
1.00
3.01
1.01
4.01
1.07
1.21
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
Chemical Shift (ppm)

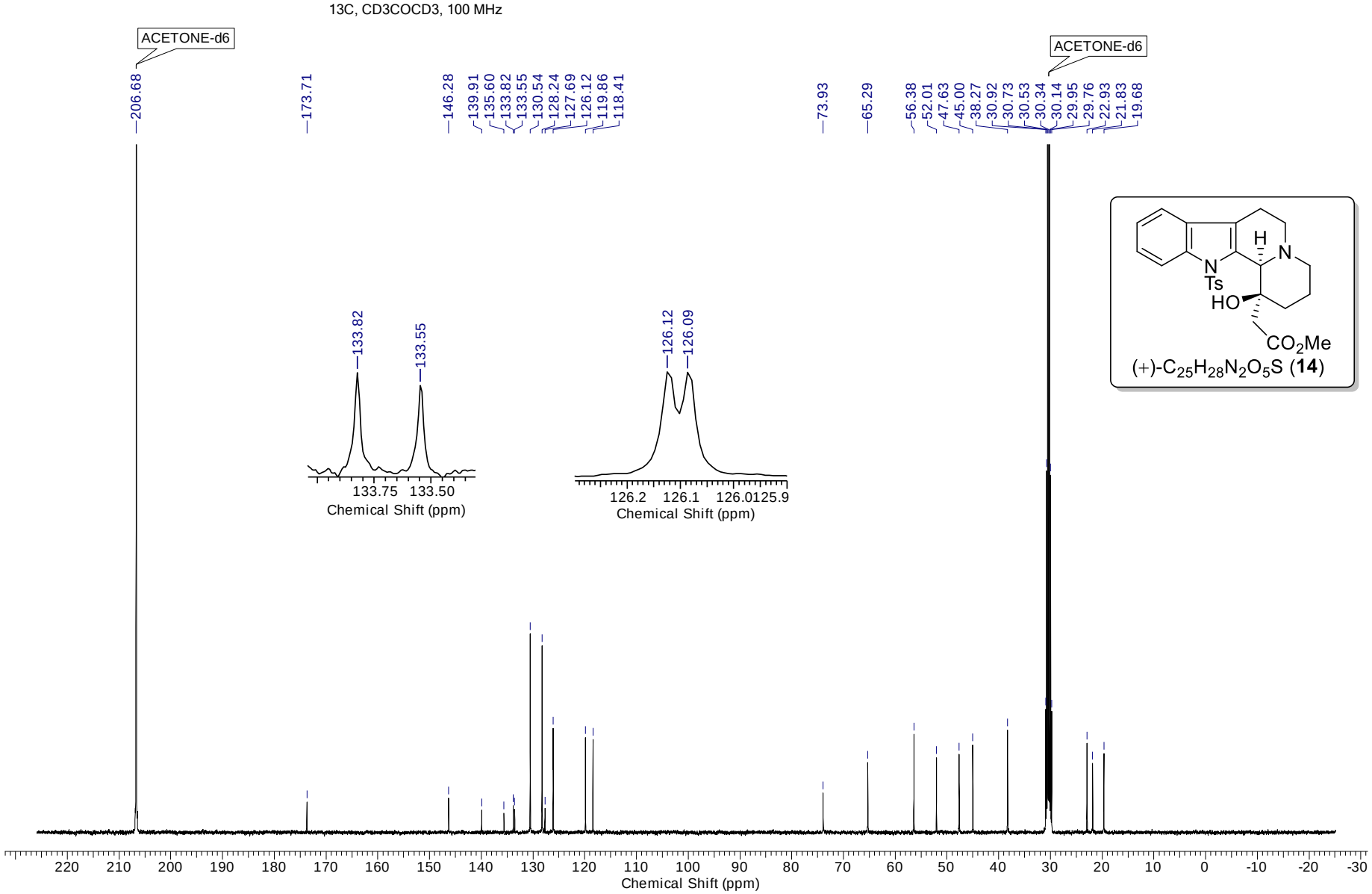

1H, CDCl3, 200 MHz
5.47
4.98
4.97
4.94
4.93
4.90
4.88
4.83
4.31
3.38
2.93
2.90
2.89
2.86
2.84
2.80
2.77
2.73
2.69
2.65
2.64
2.62
2.61
2.57
2.40
2.36
2.33
2.27
2.21
2.18
2.17
2.13
2.10
2.06
H
N
O
N
Ts
HO
CO2Me
(±)-C25H26N2O6S (13)
0.98
1.01
0.97
2.95
1.02
1.00
3.01
1.01
4.01
1.07
1.21
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
Chemical Shift (ppm)

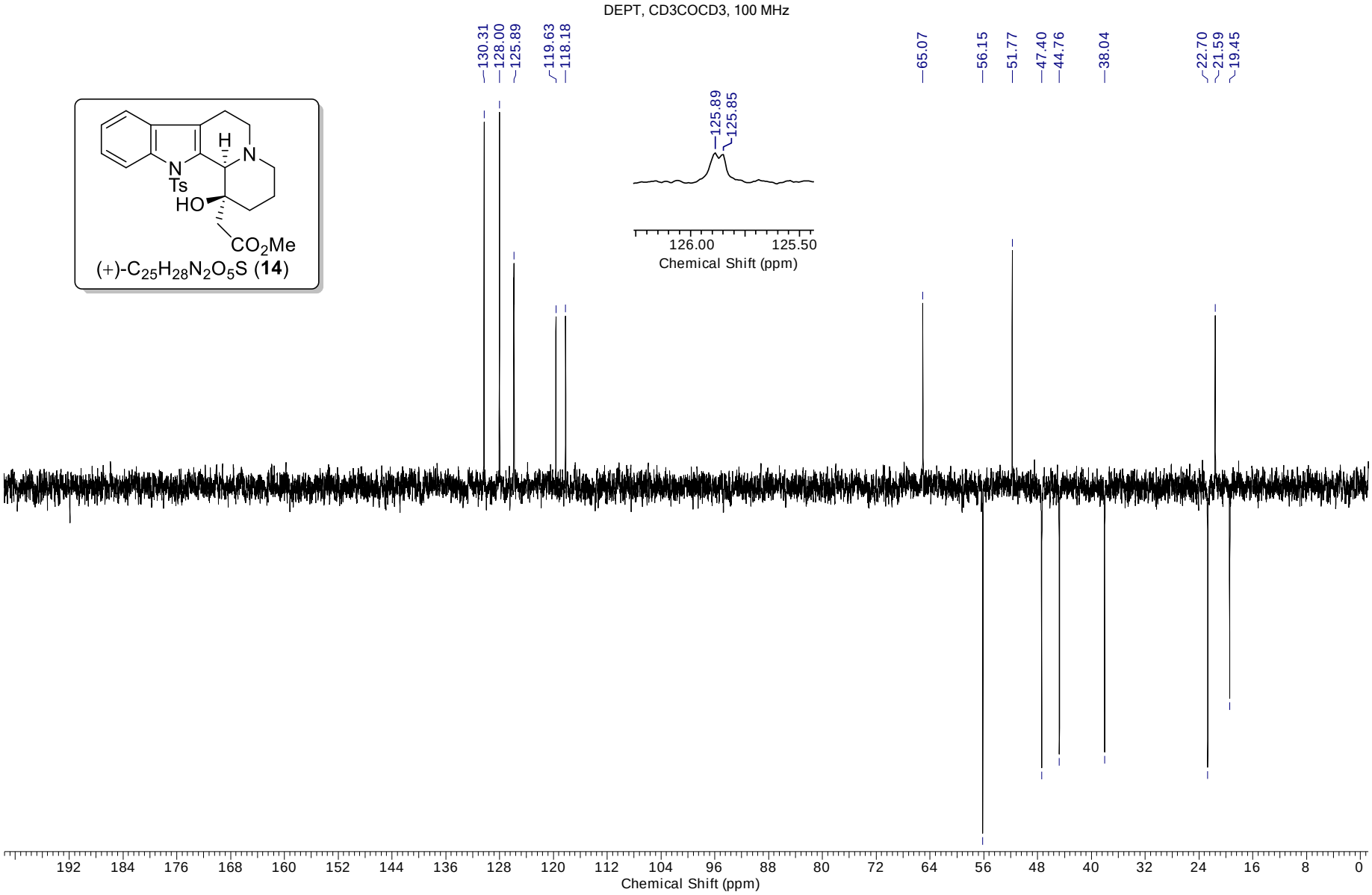

13C, CDCl3, 100 MHz
CHLOROFORM-d
171.07
145.04
138.83
132.56
131.03
129.75
129.27
127.51
126.50
125.76
124.82
118.60
117.28
77.00
74.20
61.02
51.64
38.74
37.68
33.93
29.71
21.48
21.39
Ts
HO
CO2Me
(±)-C25H26N2O6S (13)
171.13
171.07
171.50 171.25 171.00 170.75 170.50
Chemical Shift (ppm)
21.48
21.39
22.0 21.5 21.0
Chemical Shift (ppm)
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

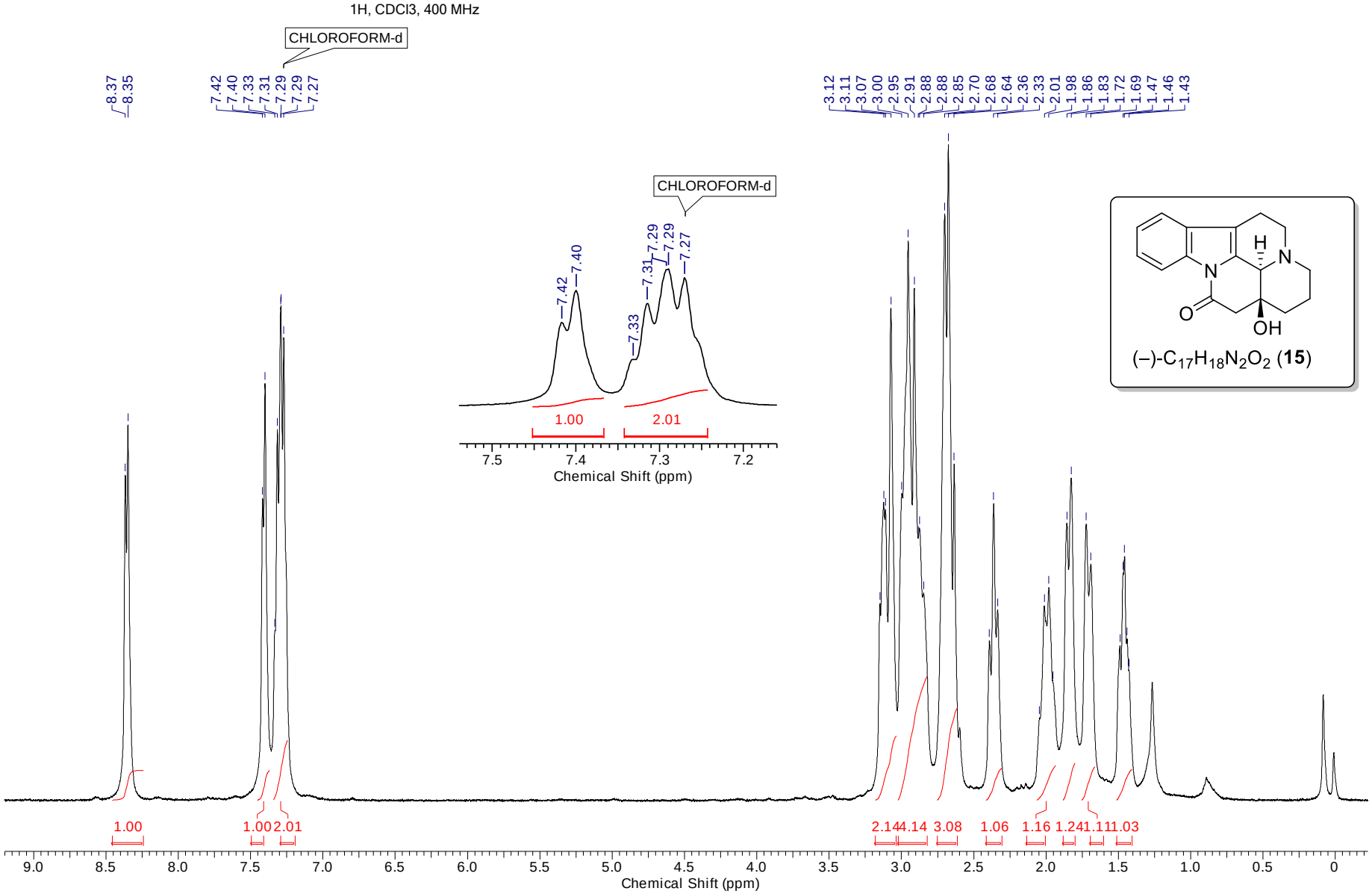

DEPT, CDCl3, 100 MHz
129.27
126.50
125.76
124.82
118.60
117.28
61.02
51.64
38.74
37.68
33.93
29.71
21.48
21.39
Ts
HO
CO2Me
(±)-C25H26N2O6S (13)
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

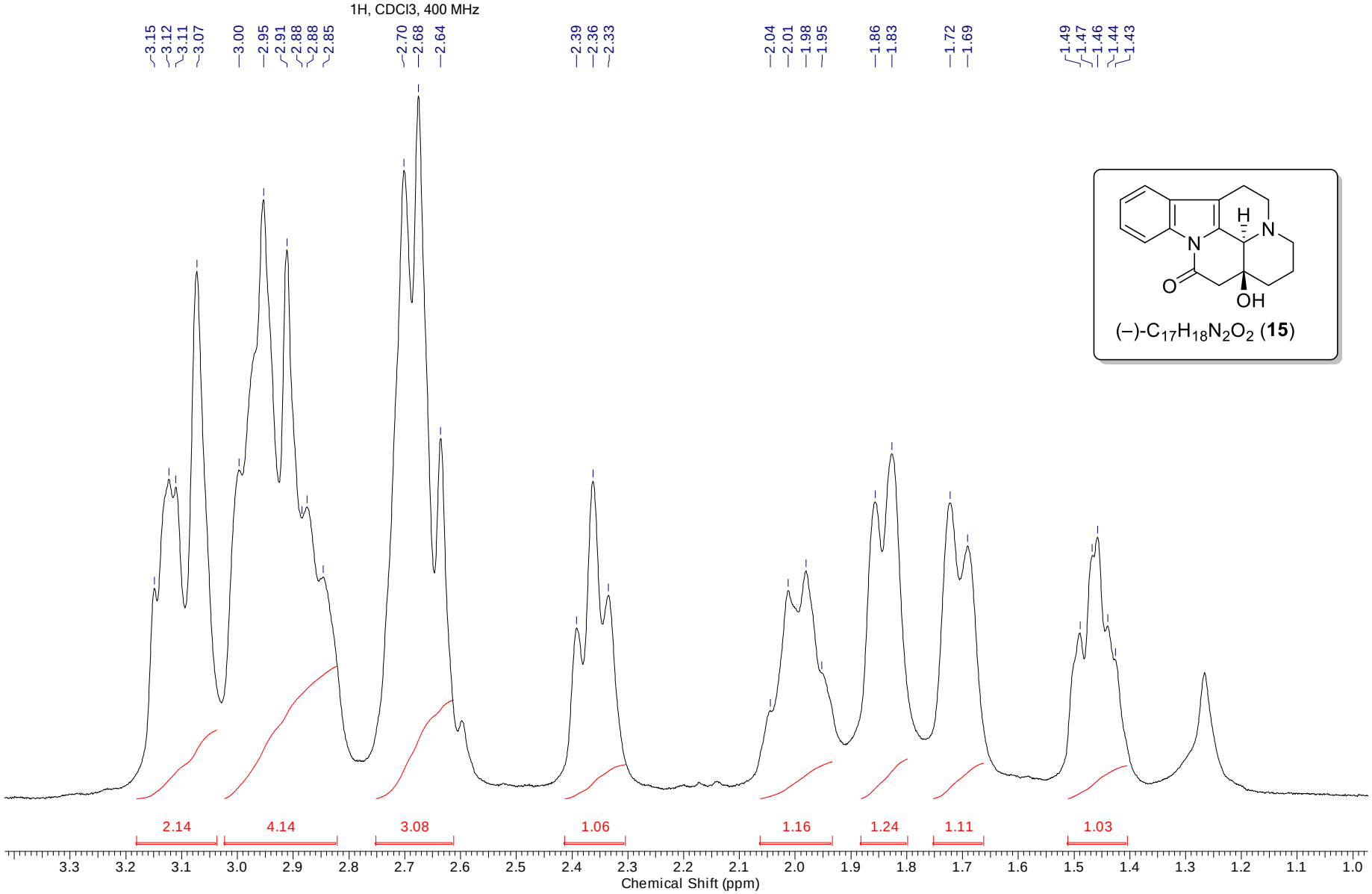

1H, CDCl3, 400 MHz
CHLOROFORM-d
8.08 8.06 7.34 7.32 7.27 7.26 7.24 7.24 7.22 7.21 7.21 7.03 7.01 4.38 4.28 3.68 3.65 3.63 3.62 3.22 3.19 3.15 3.05 3.04 3.01 3.00 2.96 2.76 2.72 2.71 2.70 2.24 2.20 2.19 2.16 2.15 1.80 1.78 1.77 1.54 1.53 1.51 0.01
7.34 7.32 7.27 7.26 7.24 7.22 7.21 7.21 7.19 7.17 7.17
2.05 3.19
7.3 7.2
Chemical Shift (ppm)
4.38 4.28
1.02 0.89
4.40 4.35 4.30 4.25
Chemical Shift (ppm)
H N N Ts HO CO2Me
(+)-C25H28N2O5S (14)
0.99 2.05 3.19 2.06 1.02 0.89 3.09 1.04 1.07 0.99 1.06 1.00 2.06 1.01 3.09 2.08 1.06 1.03
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
Chemical Shift (ppm)

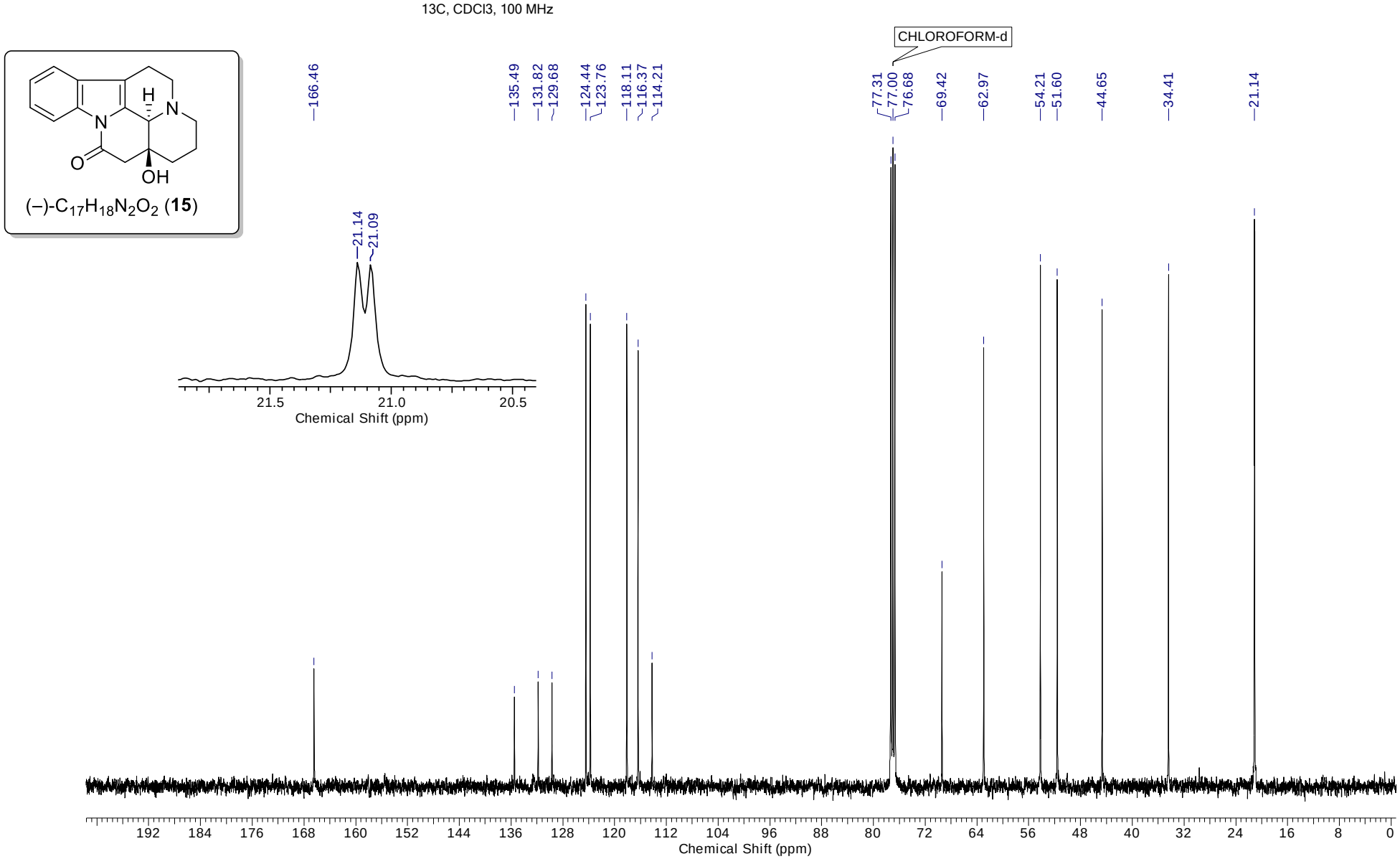

1H, CDCl3, 400 MHz
3.68 3.66 3.65 3.63 3.62
3.22 3.19 3.17 3.15 3.14 3.08 3.07 3.05 3.04 3.01 3.00 2.96 2.82 2.80 2.79 2.78 2.76 2.75 2.73 2.72 2.71 2.70 2.68 2.59 2.58 2.56 2.55 2.54 2.52
2.24 2.20 2.19 2.16 2.15 2.14 2.13
1.81 1.80 1.78 1.77 1.75 1.74 1.73
1.54 1.53 1.51 1.50 1.50
3.09 1.04
1.07 0.99
1.06 1.00
2.06
1.01
3.09 2.08
1.06
1.03
3.8 3.7 3.6 3.5 3.4 3.3 3.2 3.1 3.0 2.9 2.8 2.7 2.6 2.5 2.4 2.3 2.2 2.1 2.0 1.9 1.8 1.7 1.6 1.5 1.4 1.3
Chemical Shift (ppm)
H
N
N
Ts
HO
CO2Me
(+)-C25H28N2O5S (14)

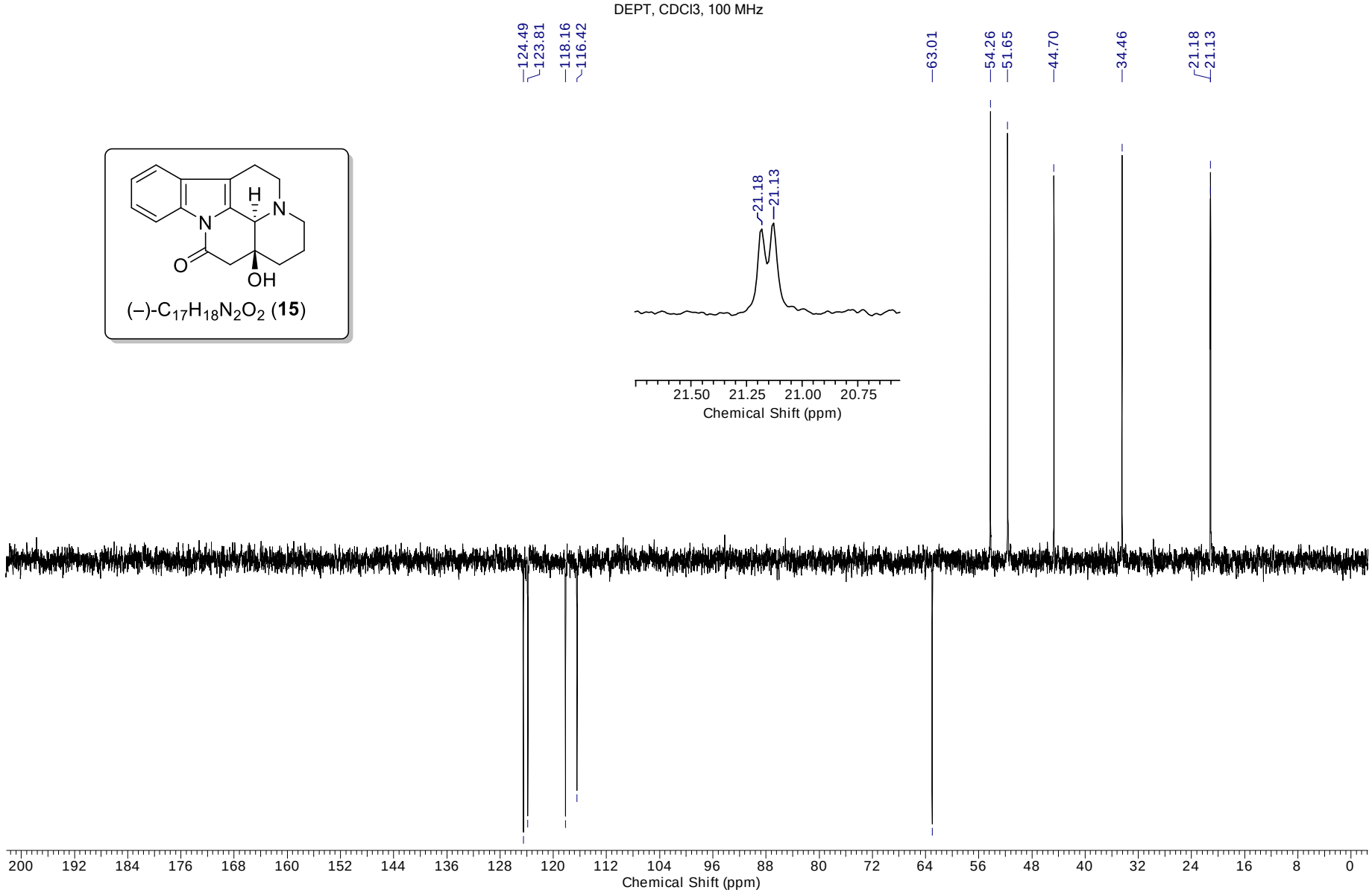

13C, CD3COCD3, 100 MHz
ACETONE-d6
206.68
173.71
146.28
139.91
135.60
133.82
133.55
130.54
128.24
127.69
126.12
119.86
118.41
73.93
65.29
56.38
52.01
47.63
45.00
38.27
30.92
30.73
30.53
30.34
30.14
29.95
29.76
22.93
21.83
19.68
ACETONE-d6
133.82
133.55
133.75 133.50
Chemical Shift (ppm)
126.12
126.09
126.2 126.1 126.0125.9
Chemical Shift (ppm)
H
N
N
Ts
HO
CO2Me
(+)-C25H28N2O5S (14)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30
Chemical Shift (ppm)

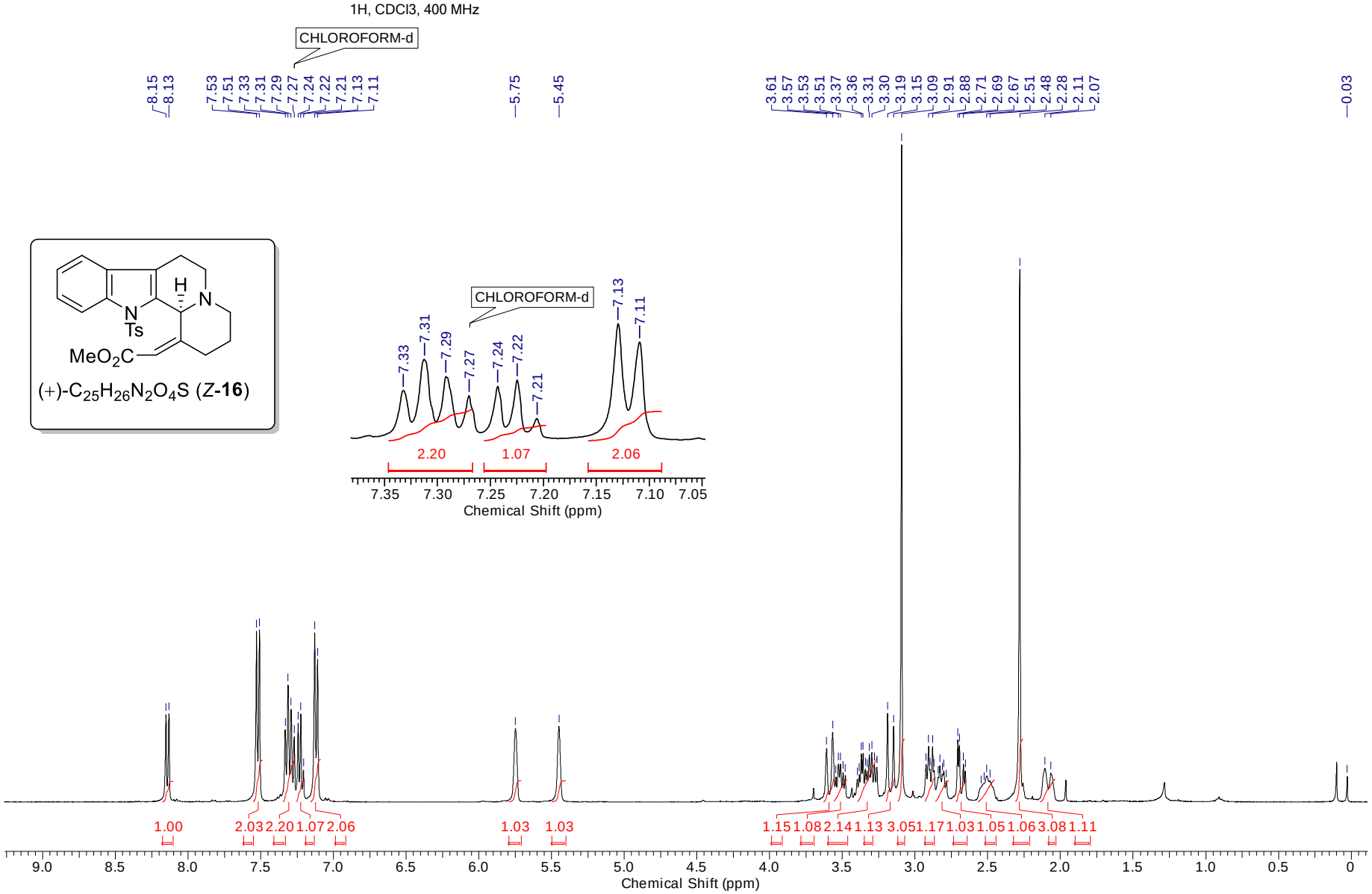

DEPT, CD3COCD3, 100 MHz
130.31
128.00
125.89
119.63
118.18
65.07
56.15
51.77
47.40
44.76
38.04
22.70
21.59
19.45
125.89
125.85
126.00
125.50
Chemical Shift (ppm)
H
N
N
Ts
HO
CO2Me
(+)-C25H28N2O5S (14)
192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

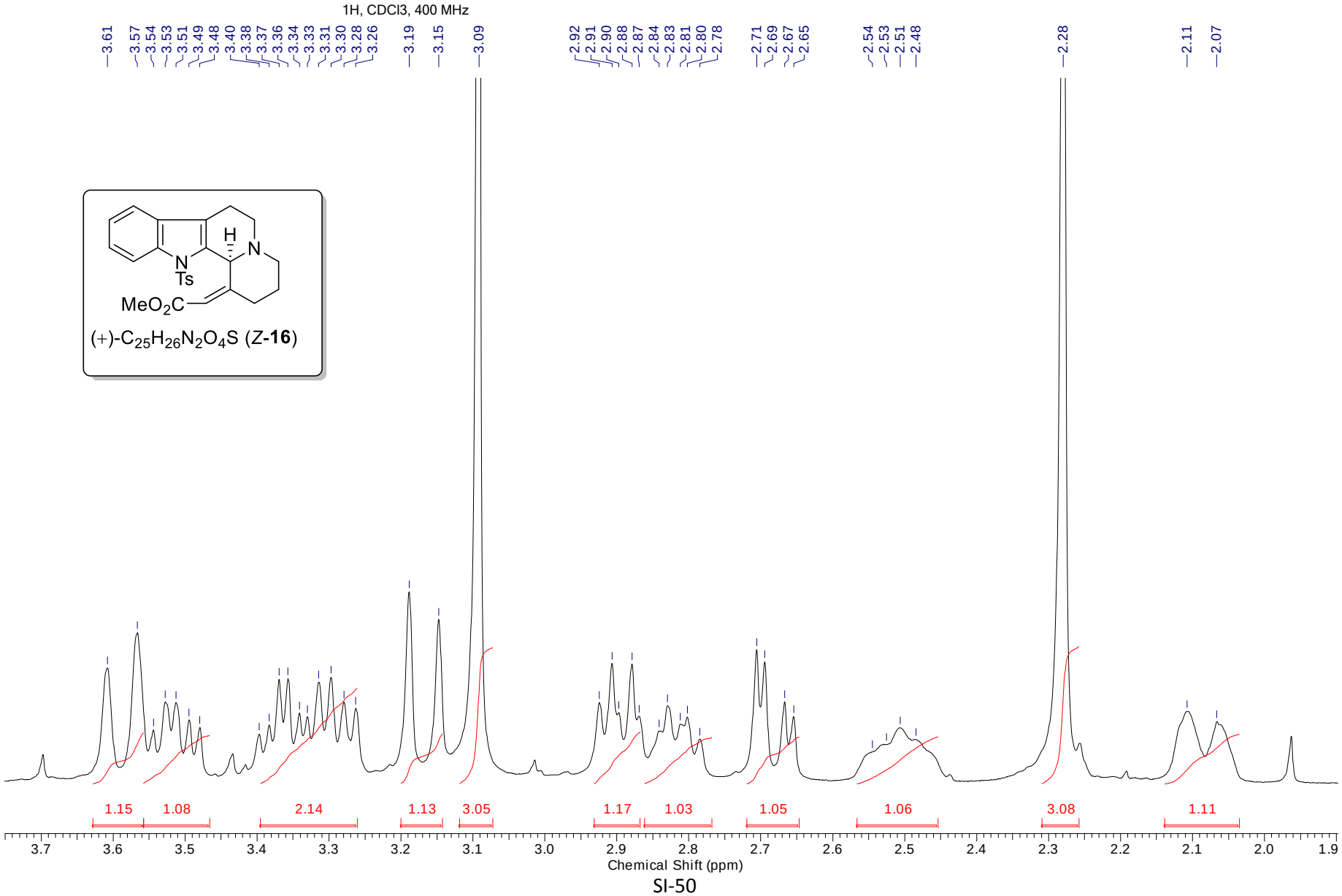

1H, CDCl3, 400 MHz
CHLOROFORM-d
8.37
8.35
7.42
7.40
7.33
7.31
7.29
7.29
7.27
3.12
3.11
3.07
3.00
2.95
2.91
2.88
2.88
2.85
2.70
2.68
2.64
2.36
2.33
2.01
1.98
1.86
1.83
1.72
1.69
1.47
1.46
1.43
1.00
1.00
2.01
2.14
4.14
3.08
1.06
1.16
1.24
1.11
1.03
Chemical Shift (ppm)
CHLOROFORM-d
1.00
2.01
Chemical Shift (ppm)
(–)-C17H18N2O2 (15)

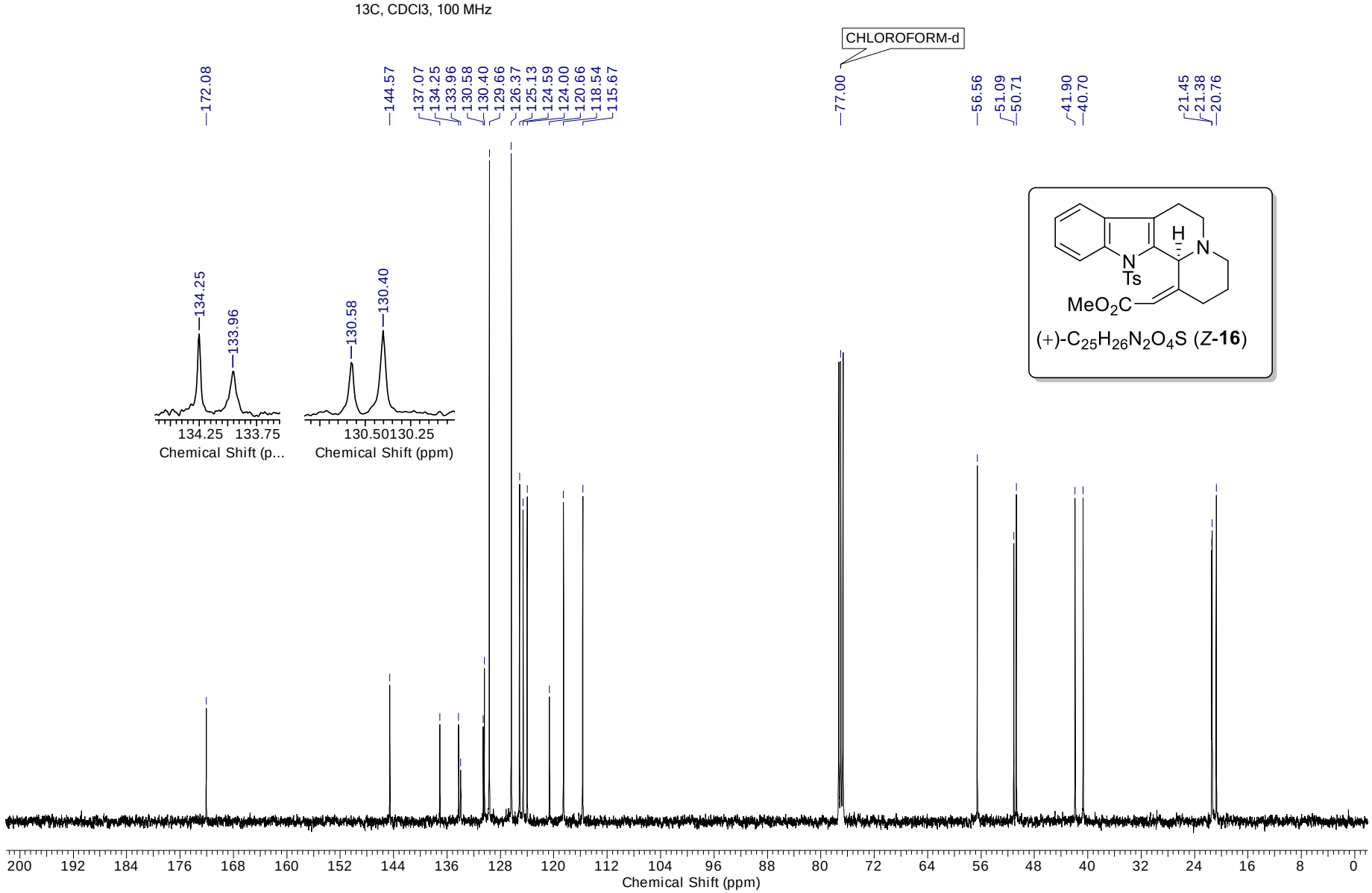

1H, CDCl3, 400 MHz
3.15
3.12
3.11
3.07
3.00
2.95
2.91
2.88
2.88
2.85
2.70
2.68
2.64
2.39
2.36
2.33
2.04
2.01
1.98
1.95
1.86
1.83
1.72
1.69
1.49
1.47
1.46
1.44
1.43
2.14
4.14
3.08
1.06
1.16
1.24
1.11
1.03
3.3
3.2
3.1
3.0
2.9
2.8
2.7
2.6
2.5
2.4
2.3
2.2
2.1
2.0
1.9
1.8
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1.0
Chemical Shift (ppm)
H
N
N
O
OH
(–)-C17H18N2O2 (15)

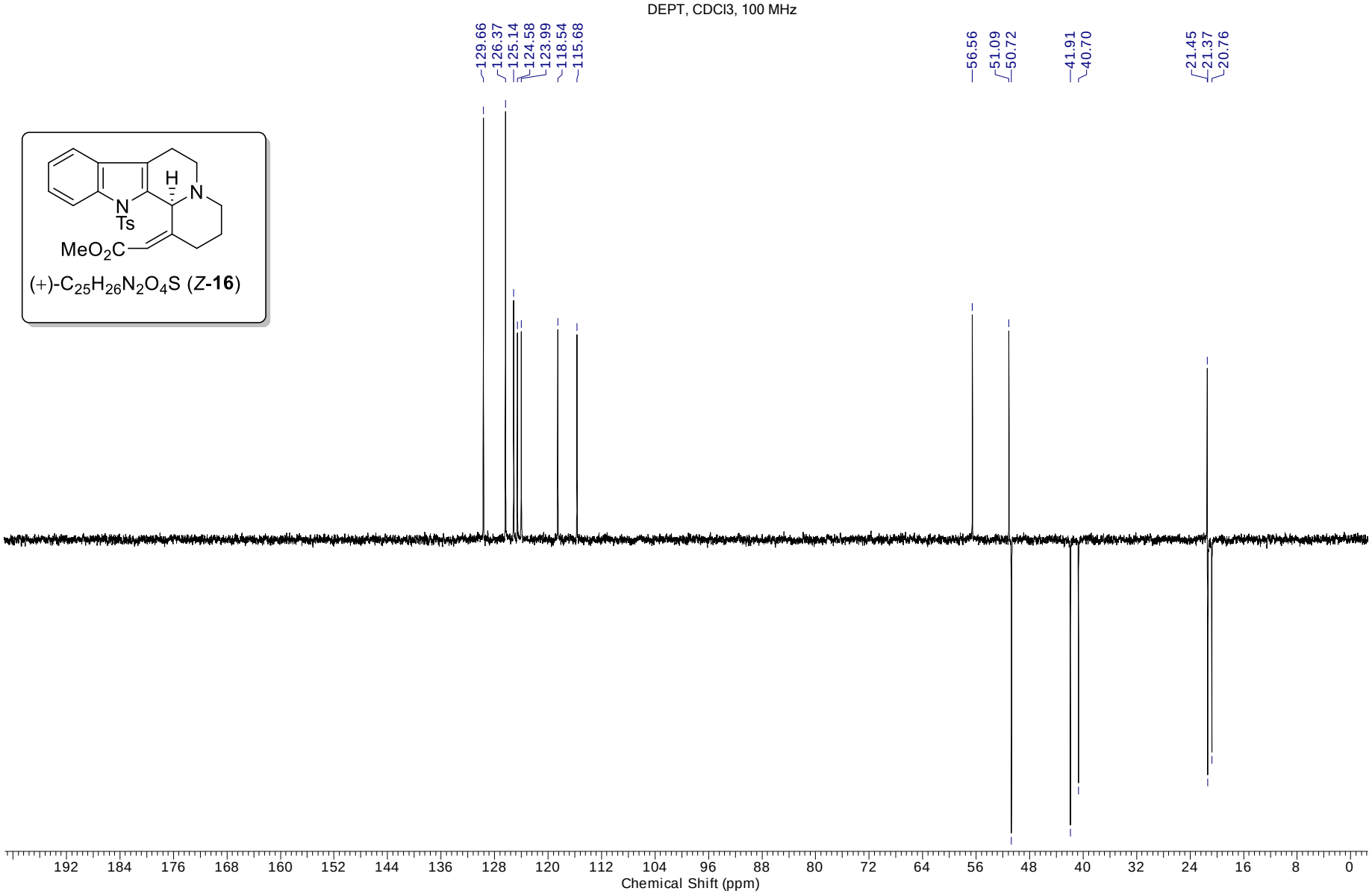

13C, CDCl3, 100 MHz
(–)-C17H18N2O2 (15)
CHLOROFORM-d
166.46
135.49
131.82
129.68
124.44
123.76
118.11
116.37
114.21
77.31
77.00
76.68
69.42
62.97
54.21
51.60
44.65
34.41
21.14
21.14
21.09
Chemical Shift (ppm)
Chemical Shift (ppm)

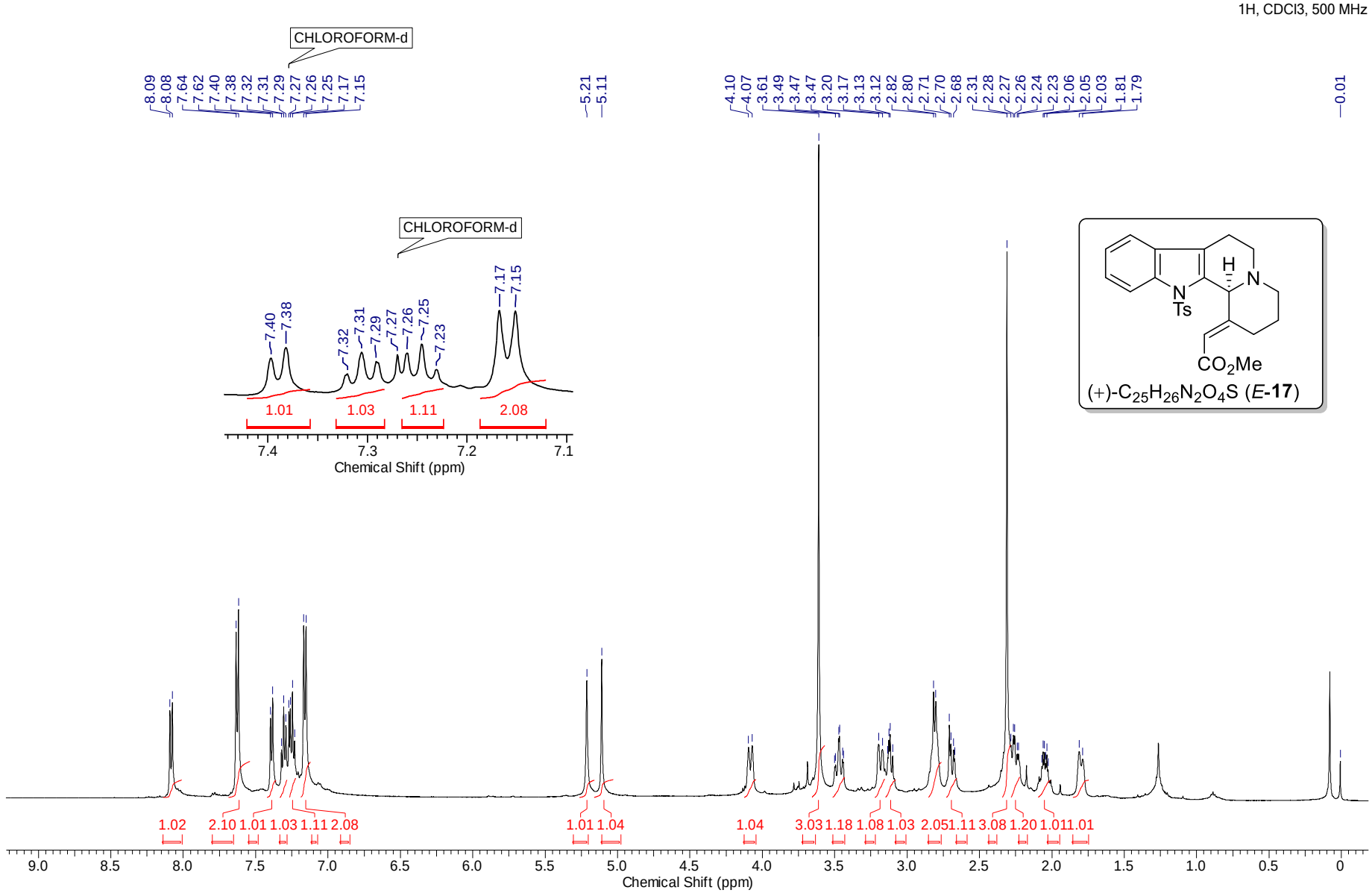

DEPT, CDCl3, 100 MHz
124.49
123.81
118.16
116.42
63.01
54.26
51.65
44.70
34.46
21.18
21.13
H
N
N
O
OH
(–)-C17H18N2O2 (15)
21.50 21.25 21.00 20.75
Chemical Shift (ppm)
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

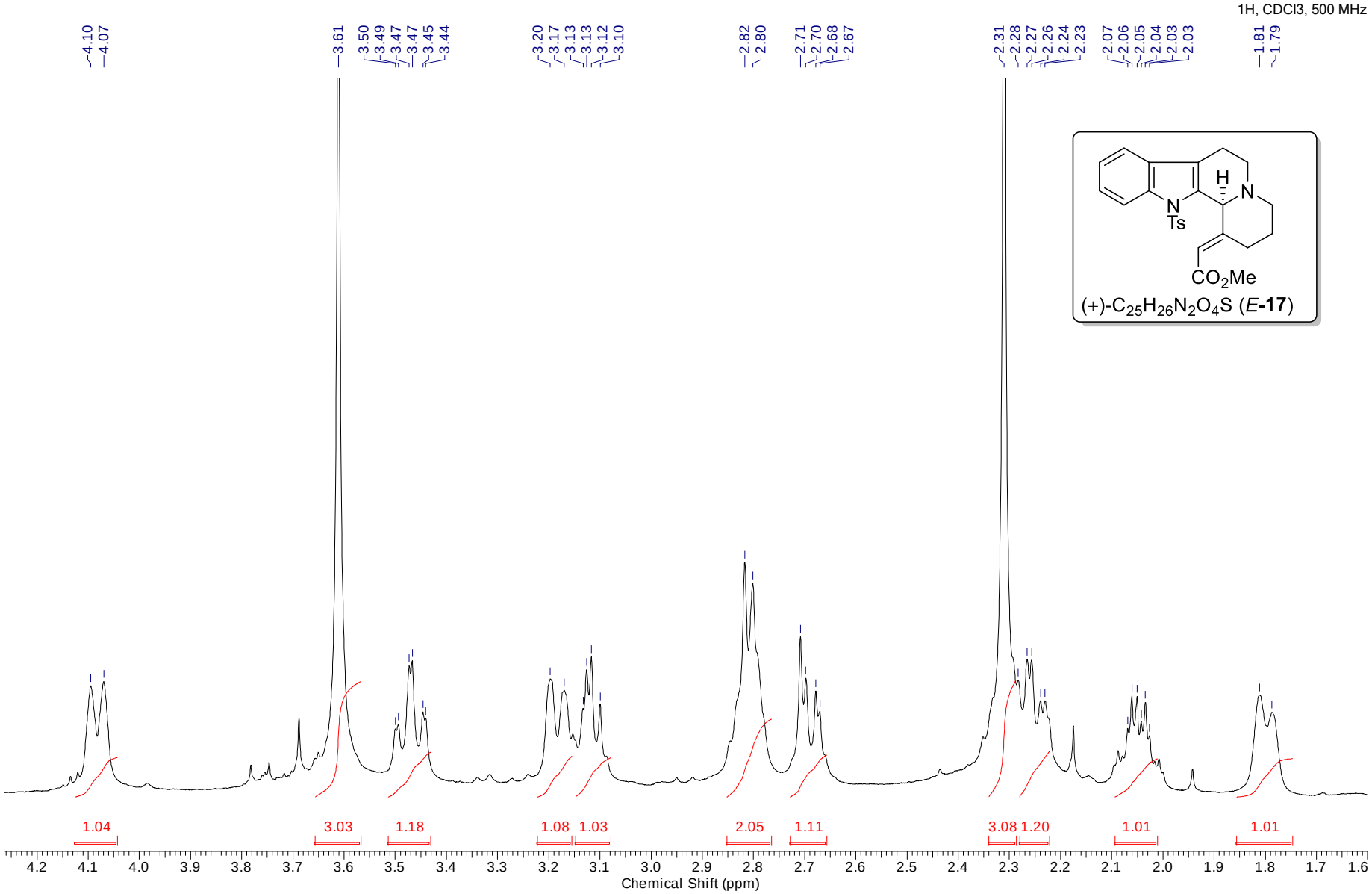

1H, CDCl3, 400 MHz
CHLOROFORM-d
8.15
8.13
7.53
7.51
7.33
7.31
7.29
7.27
7.24
7.22
7.21
7.13
7.11
5.75
5.45
3.61
3.57
3.53
3.51
3.37
3.36
3.31
3.30
3.19
3.15
3.09
2.91
2.88
2.71
2.69
2.67
2.51
2.48
2.28
2.11
2.07
0.03
H
N
N
Ts
MeO2C
(+)-C25H26N2O4S (Z-16)
CHLOROFORM-d
7.33
7.31
7.29
7.27
7.24
7.22
7.21
7.13
7.11
2.20
1.07
2.06
7.35 7.30 7.25 7.20 7.15 7.10 7.05
Chemical Shift (ppm)
1.00
2.03
2.20
1.07
2.06
1.03
1.03
1.15
1.08
2.14
1.13
3.05
1.17
1.03
1.05
1.06
3.08
1.11
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
Chemical Shift (ppm)

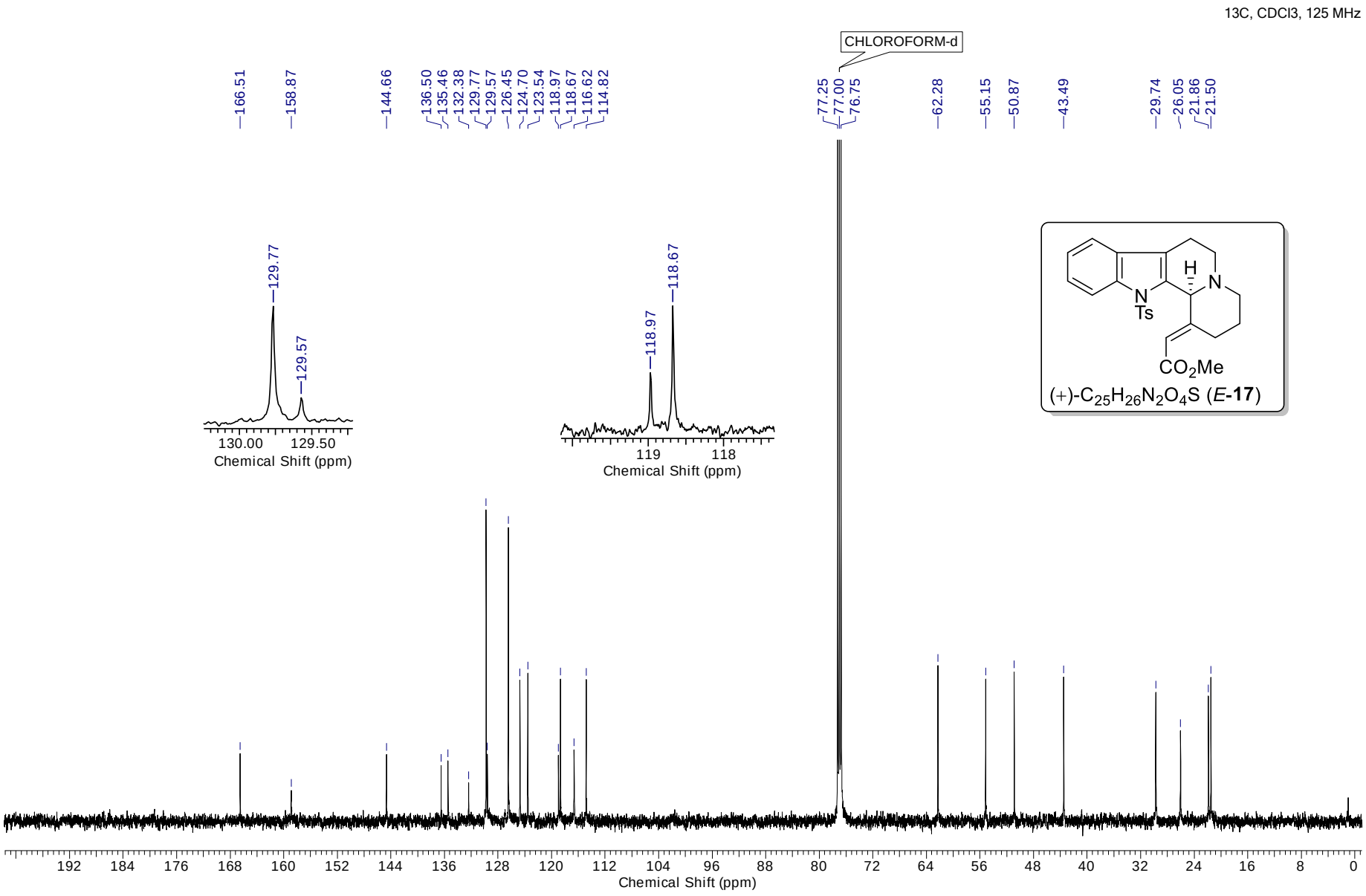

1H, CDCl3, 400 MHz
3.61
3.57
3.54
3.53
3.51
3.49
3.48
3.40
3.38
3.37
3.36
3.34
3.33
3.31
3.30
3.28
3.26
3.19
3.15
3.09
2.92
2.91
2.90
2.88
2.87
2.84
2.83
2.81
2.80
2.78
2.71
2.69
2.67
2.65
2.54
2.53
2.51
2.48
2.28
2.11
2.07
H
N
N
Ts
MeO2C
(+)-C25H26N2O4S (Z-16)
1.15
1.08
2.14
1.13
3.05
1.17
1.03
1.05
1.06
3.08
1.11
3.7
3.6
3.5
3.4
3.3
3.2
3.1
3.0
2.9
2.8
2.7
2.6
2.5
2.4
2.3
2.2
2.1
2.0
1.9
Chemical Shift (ppm)

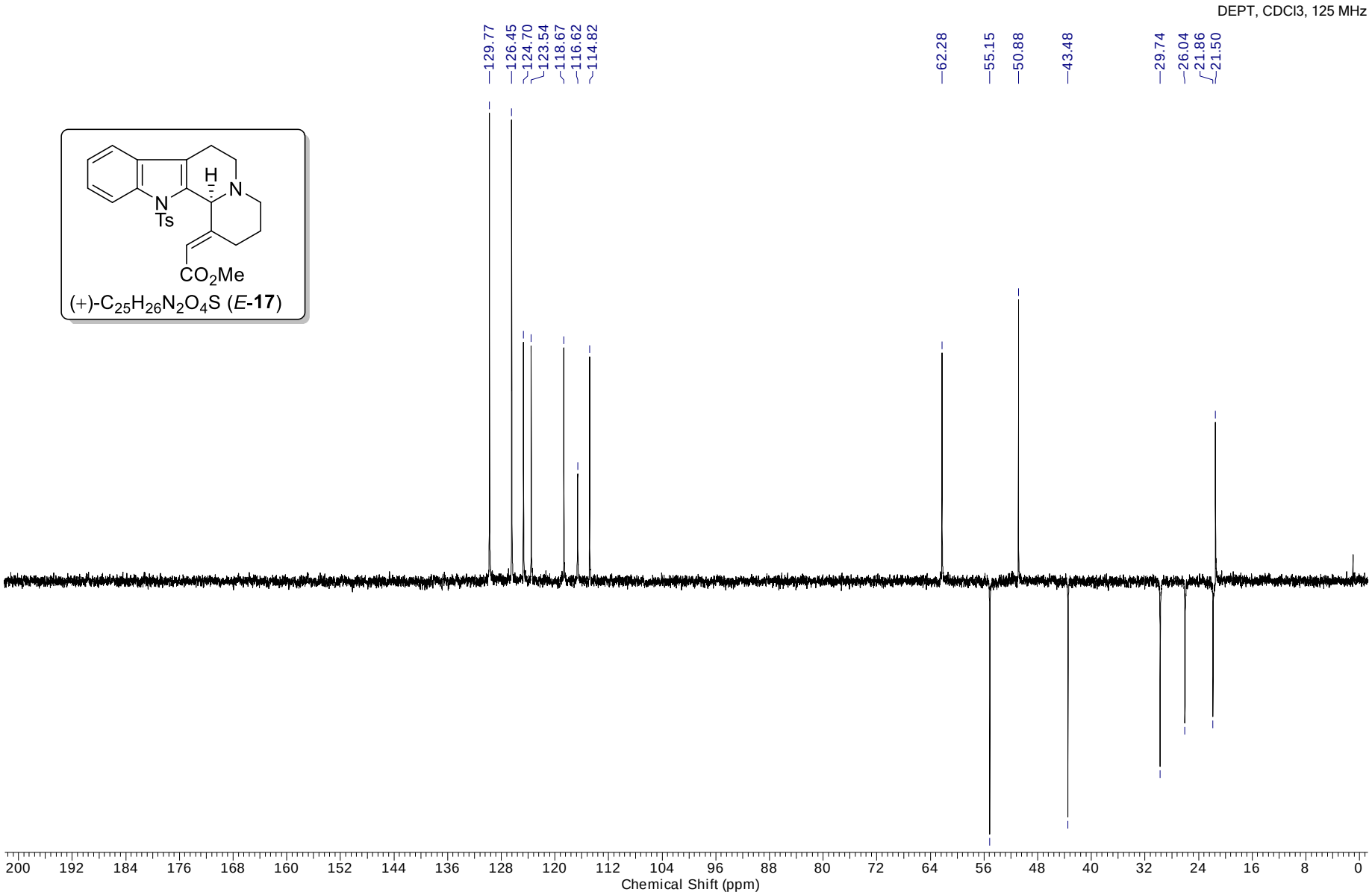

13C, CDCl3, 100 MHz
CHLOROFORM-d
172.08
144.57
137.07
134.25
133.96
130.58
130.40
129.66
126.37
125.13
124.59
124.00
120.66
118.54
115.67
77.00
56.56
51.09
50.71
41.90
40.70
21.45
21.38
20.76
134.25
133.96
130.58
130.40
134.25 133.75
Chemical Shift (p...
130.50130.25
Chemical Shift (ppm)
H
N
N
Ts
MeO2C
(+)-C25H26N2O4S (Z-16)
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

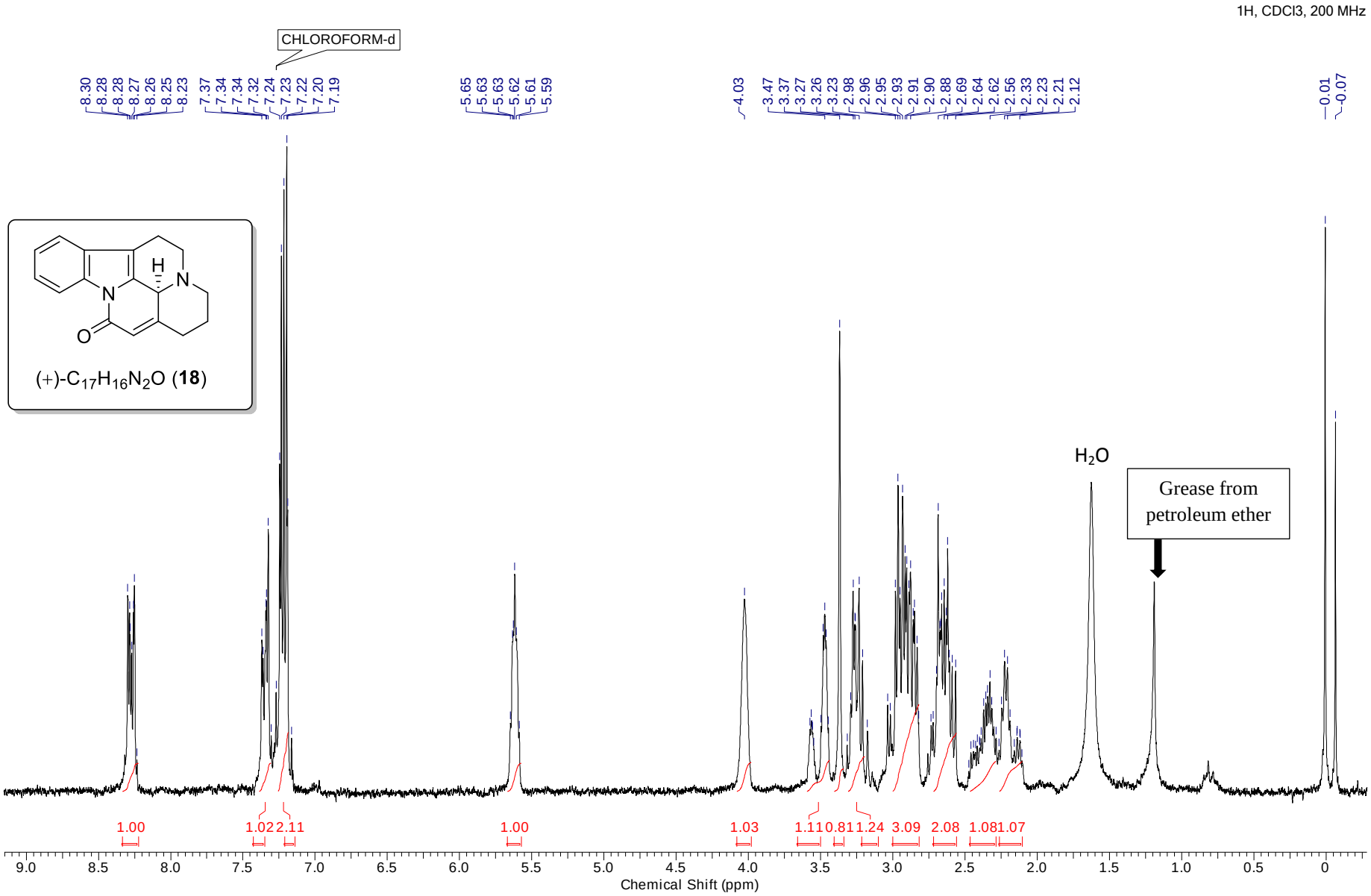

DEPT, CDCl3, 100 MHz
129.66
126.37
125.14
124.58
123.99
118.54
115.68
56.56
51.09
50.72
41.91
40.70
21.45
21.37
20.76
H
N
N
Ts
MeO2C
(+)-C25H26N2O4S (Z-16)
192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

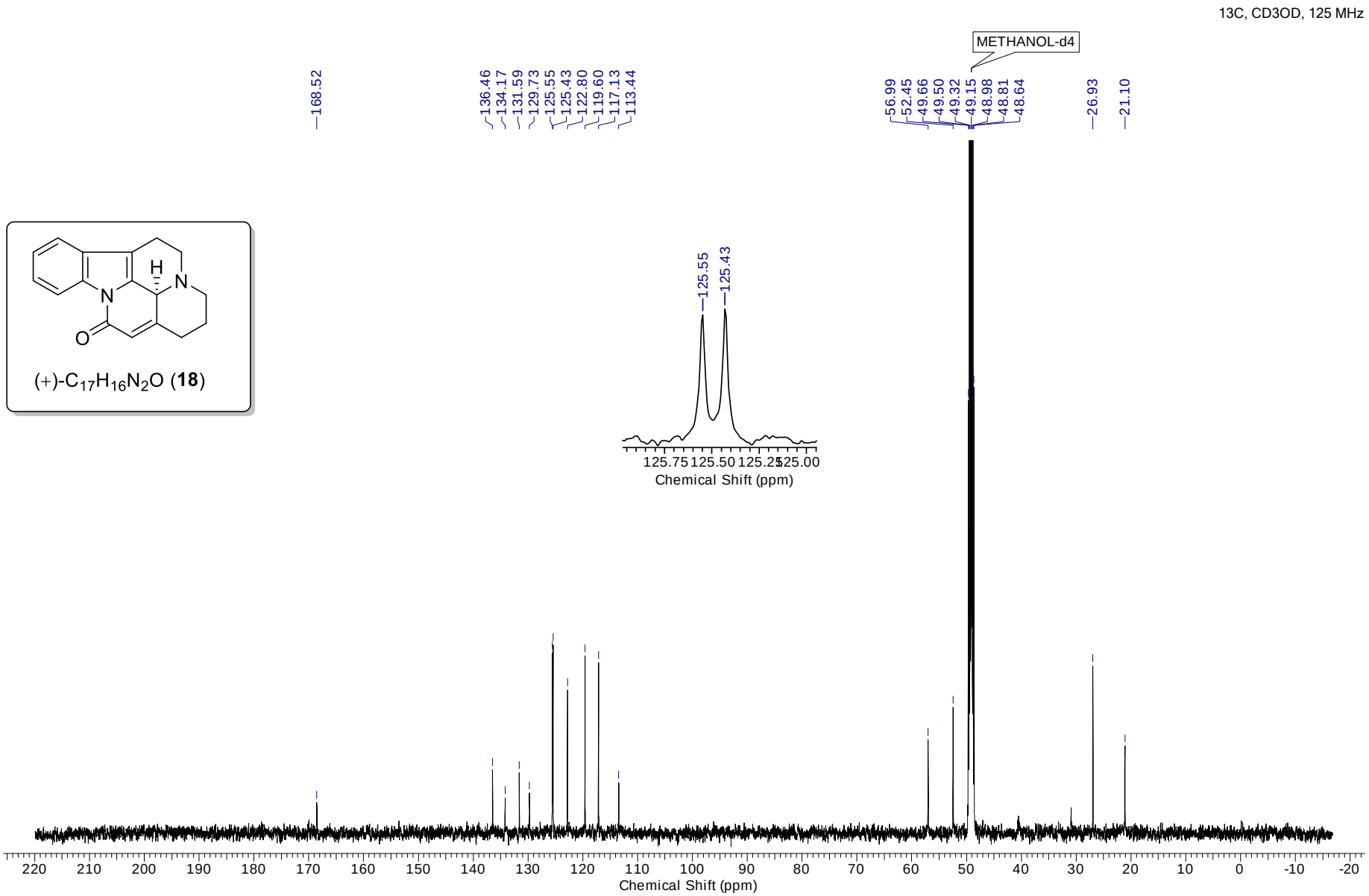

1H, CDCl3, 500 MHz
CHLOROFORM-d
8.09
8.08
7.64
7.62
7.40
7.38
7.32
7.31
7.29
7.27
7.26
7.25
7.17
7.15
5.21
5.11
4.10
4.07
3.61
3.49
3.47
3.47
3.20
3.17
3.13
3.12
2.82
2.80
2.71
2.70
2.68
2.31
2.28
2.27
2.26
2.24
2.23
2.06
2.05
2.03
1.81
1.79
0.01
CHLOROFORM-d
7.40
7.38
7.32
7.31
7.29
7.27
7.26
7.25
7.23
7.17
7.15
1.01
1.03
1.11
2.08
7.4
7.3
7.2
7.1
Chemical Shift (ppm)
H
N
N
Ts
CO2Me
(+)-C25H26N2O4S (E-17)
1.02
2.10
1.01
1.03
1.11
2.08
1.01
1.04
1.04
3.03
1.18
1.08
1.03
2.05
1.11
3.08
1.20
1.01
1.01
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
Chemical Shift (ppm)

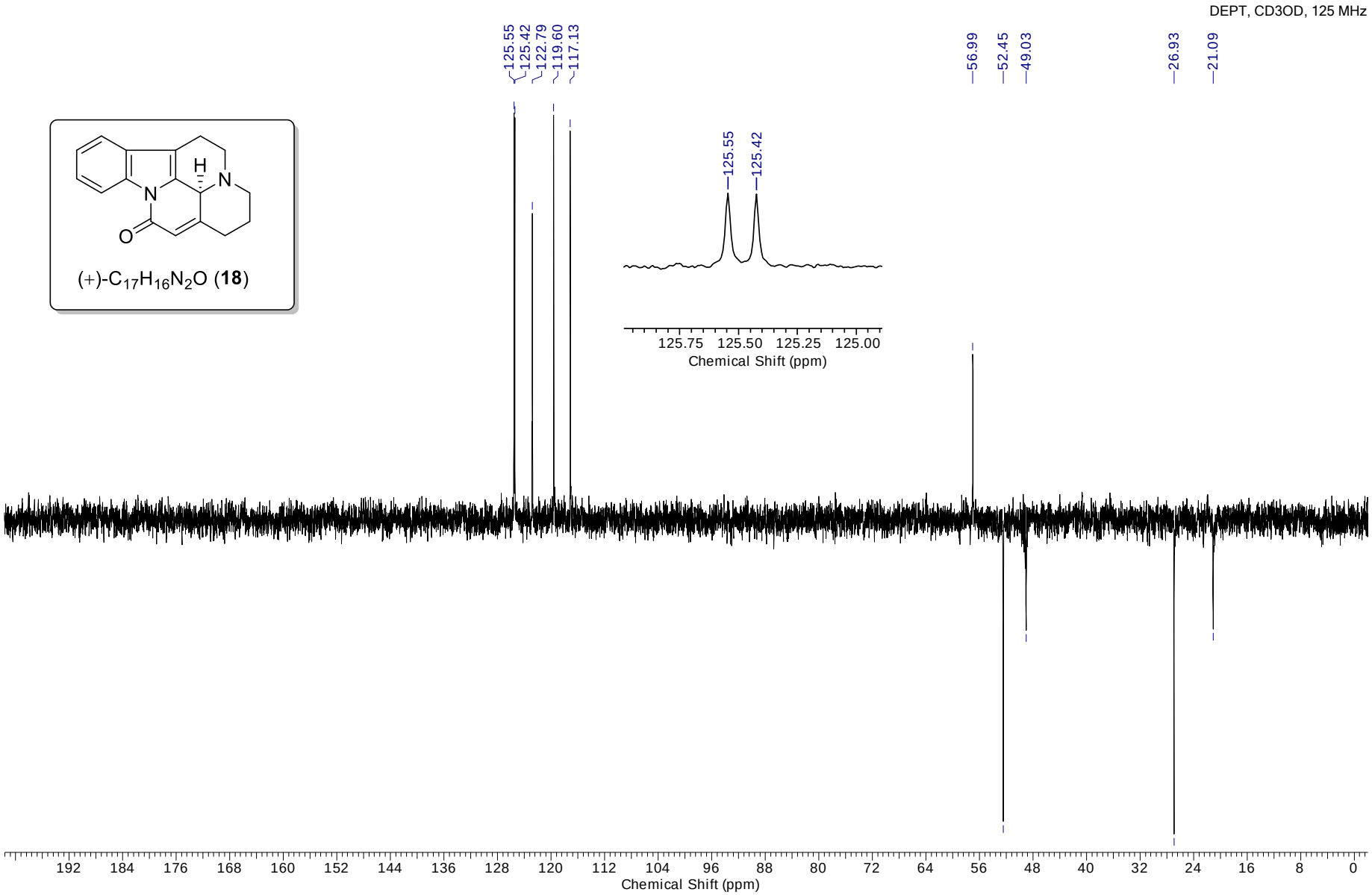

1H, CDCl3, 500 MHz
4.10
4.07
3.61
3.50
3.49
3.47
3.47
3.45
3.44
3.20
3.17
3.13
3.13
3.12
3.10
2.82
2.80
2.71
2.70
2.68
2.67
2.31
2.28
2.27
2.26
2.24
2.23
2.07
2.06
2.05
2.04
2.03
2.03
1.81
1.79
H
N
N
Ts
CO2Me
(+)-C25H26N2O4S (E-17)
1.04
3.03
1.18
1.08
1.03
2.05
1.11
3.08
1.20
1.01
1.01
4.2
4.1
4.0
3.9
3.8
3.7
3.6
3.5
3.4
3.3
3.2
3.1
3.0
2.9
2.8
2.7
2.6
2.5
2.4
2.3
2.2
2.1
2.0
1.9
1.8
1.7
1.6
Chemical Shift (ppm)

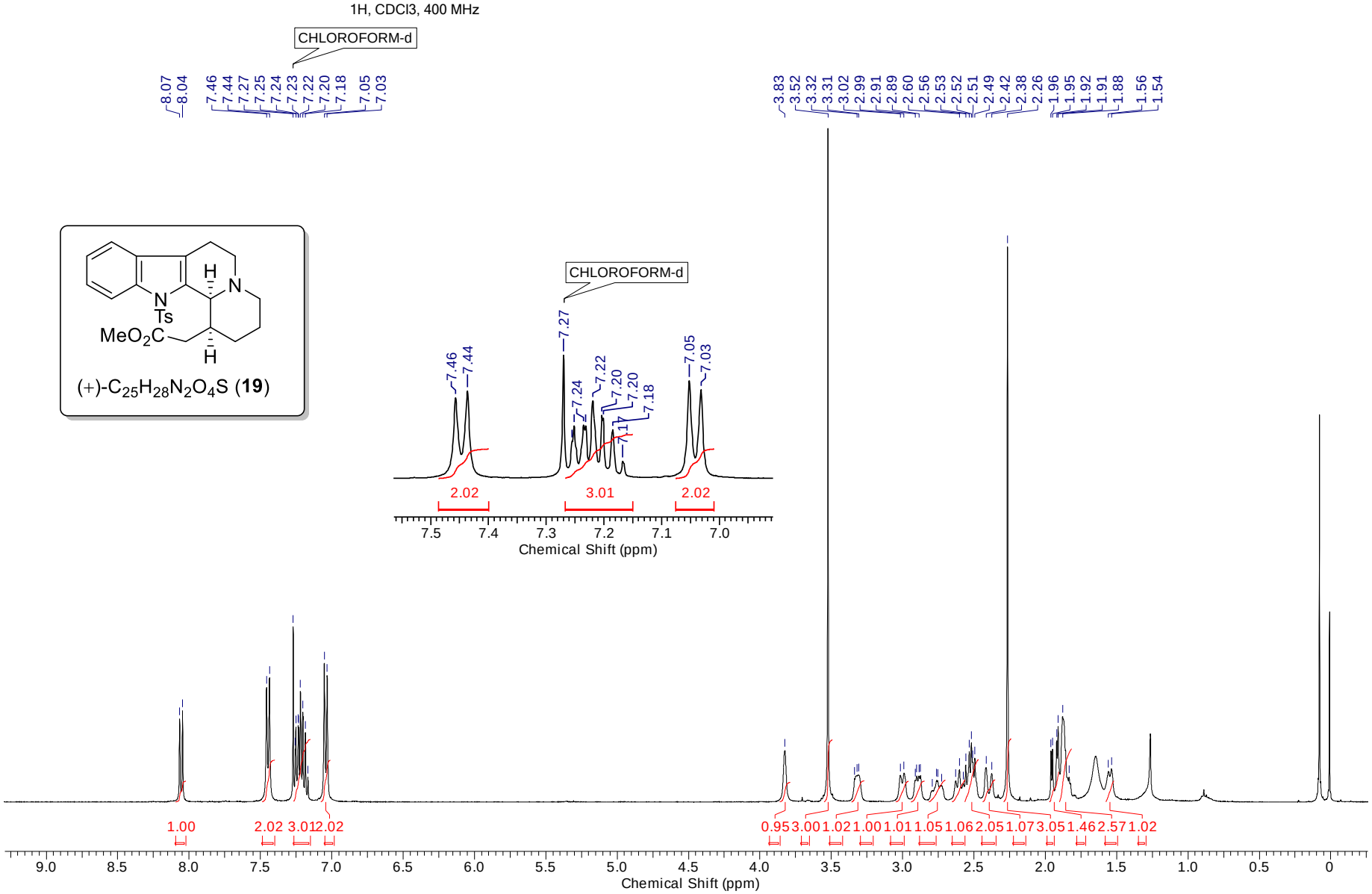

13C, CDCl3, 125 MHz
CHLOROFORM-d
166.51
158.87
144.66
136.50
135.46
132.38
129.77
129.57
126.45
124.70
123.54
118.97
118.67
116.62
114.82
77.25
77.00
76.75
62.28
55.15
50.87
43.49
29.74
26.05
21.86
21.50
129.77
129.57
130.00
129.50
Chemical Shift (ppm)
118.97
118.67
119
118
Chemical Shift (ppm)
H
N
N
Ts
CO2Me
(+)-C25H26N2O4S (E-17)
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

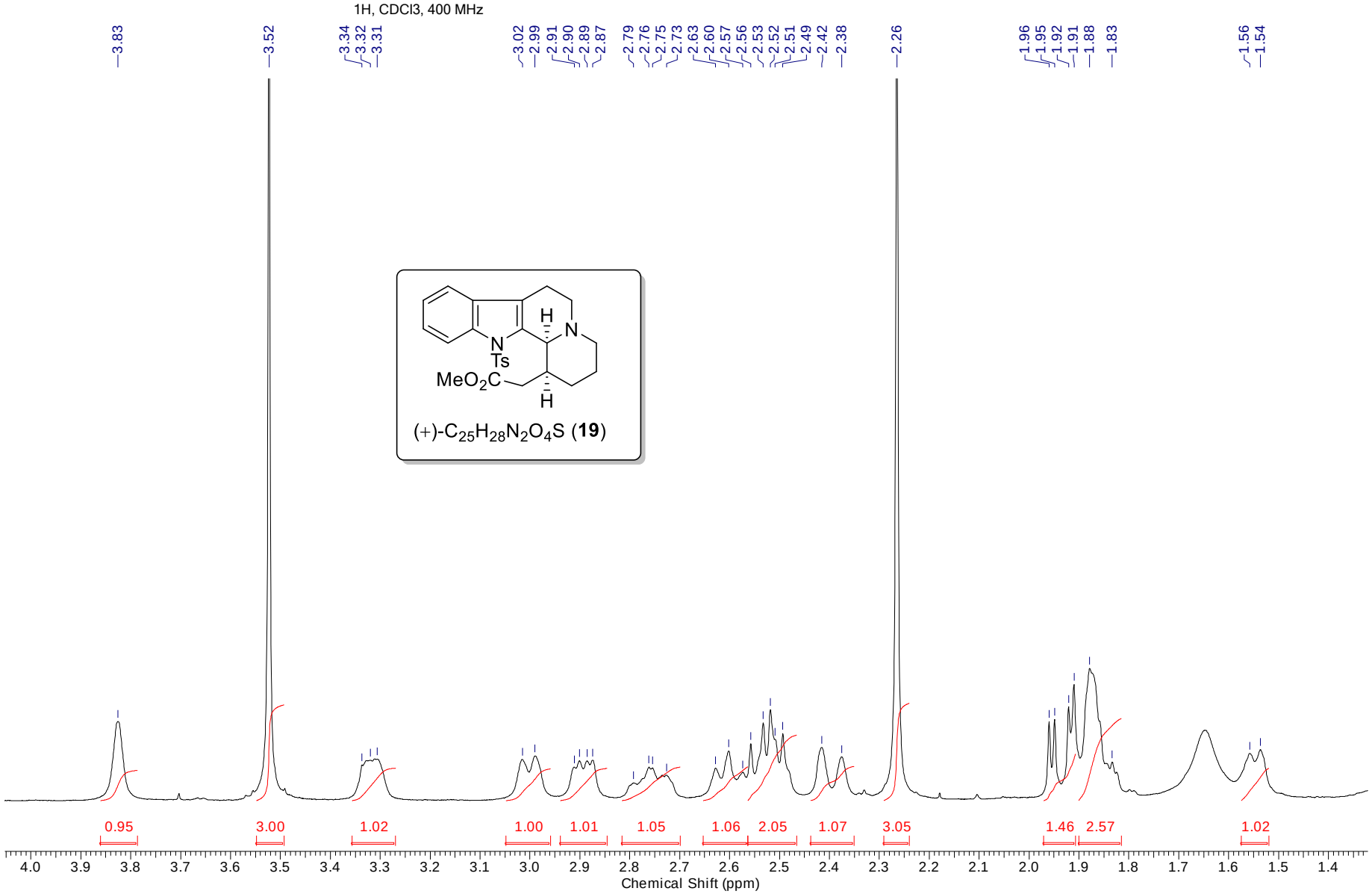

DEPT, CDCl3, 125 MHz
129.77
126.45
124.70
123.54
118.67
116.62
114.82
62.28
55.15
50.88
43.48
29.74
26.04
21.86
21.50
Ts
CO2Me
(+)-C25H26N2O4S (E-17)
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

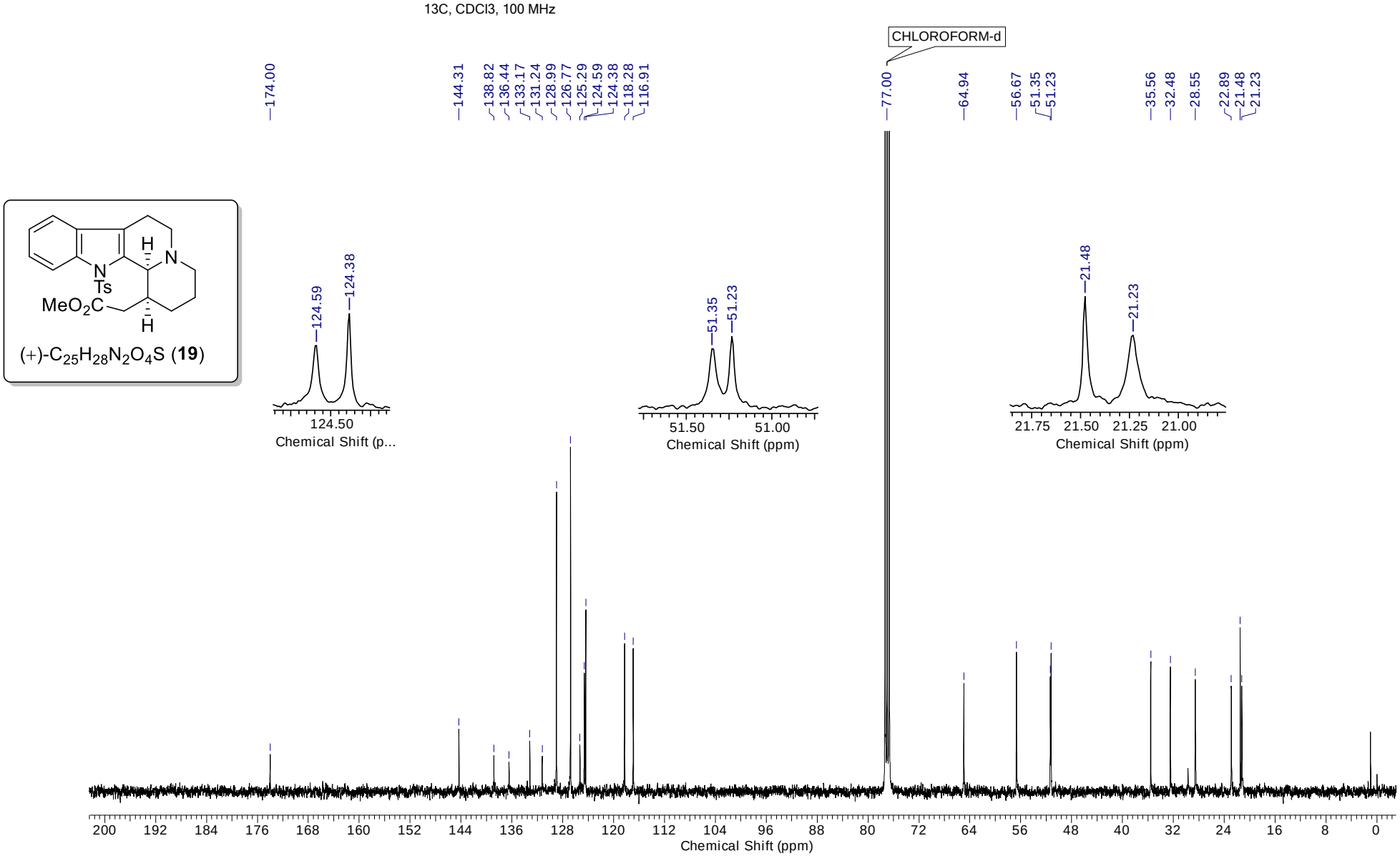

1H, CDCl3, 200 MHz
CHLOROFORM-d
8.30 8.28 8.28 8.27 8.26 8.25 8.23
7.37 7.34 7.34 7.32 7.24 7.23 7.22 7.20 7.19
5.65 5.63 5.63 5.62 5.61 5.59
4.03
3.47 3.37 3.27 3.26 3.23 2.98 2.96 2.95 2.93 2.91 2.90 2.88 2.69 2.64 2.62 2.56 2.33 2.23 2.21 2.12
0.01 -0.07
(+)-C17H16N2O (18)
H2O
Grease from petroleum ether
1.00 1.02 2.11 1.00 1.03 1.11 0.81 1.24 3.09 2.08 1.08 1.07
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
Chemical Shift (ppm)

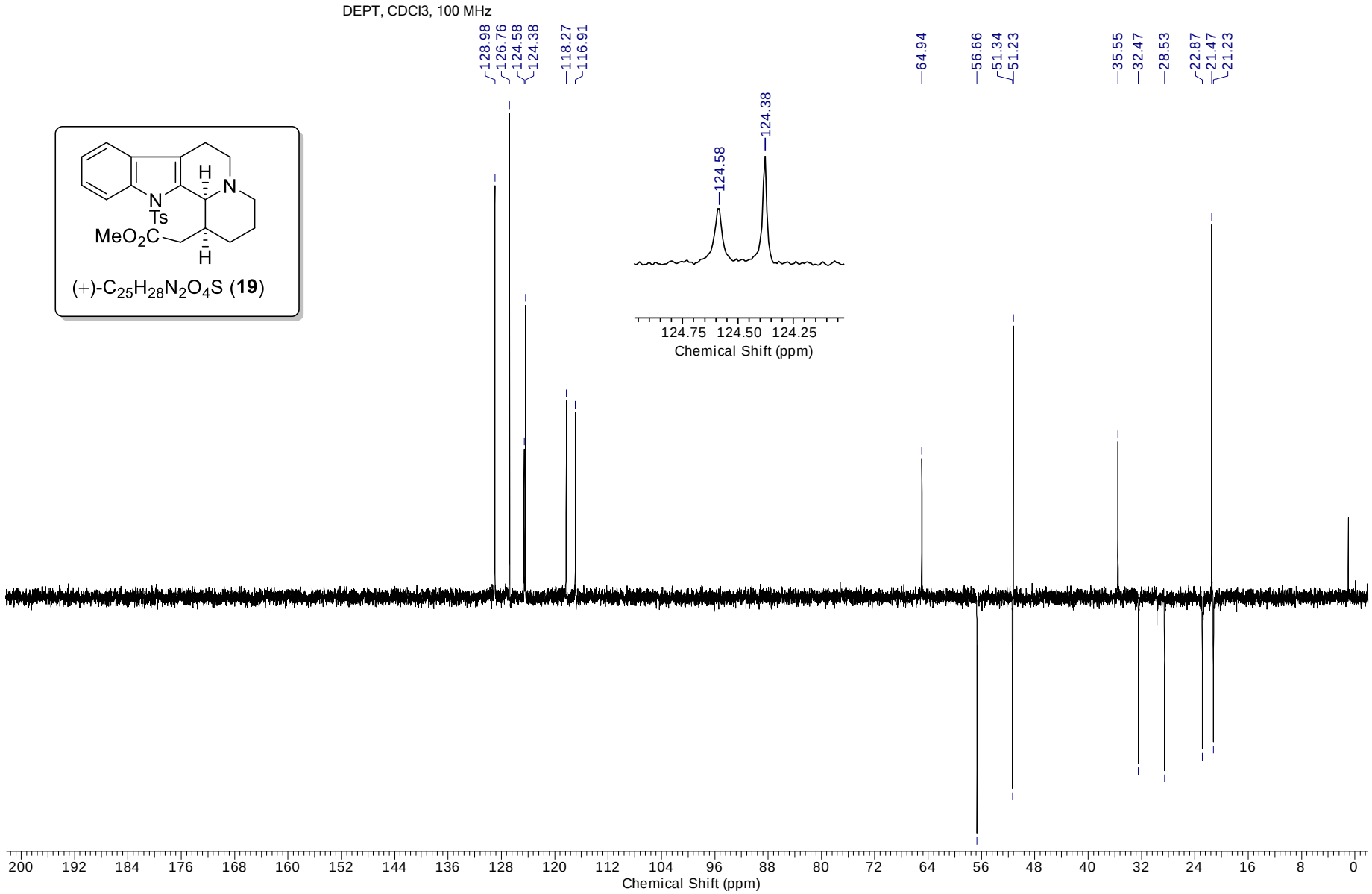

13C, CD3OD, 125 MHz
METHANOL-d4
168.52
136.46
134.17
131.59
129.73
125.55
125.43
122.80
119.60
117.13
113.44
56.99
52.45
49.66
49.50
49.32
49.15
48.98
48.81
48.64
26.93
21.10
125.55
125.43
125.75 125.50 125.25 125.00
Chemical Shift (ppm)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
Chemical Shift (ppm)
(+)-C17H16N2O (18)

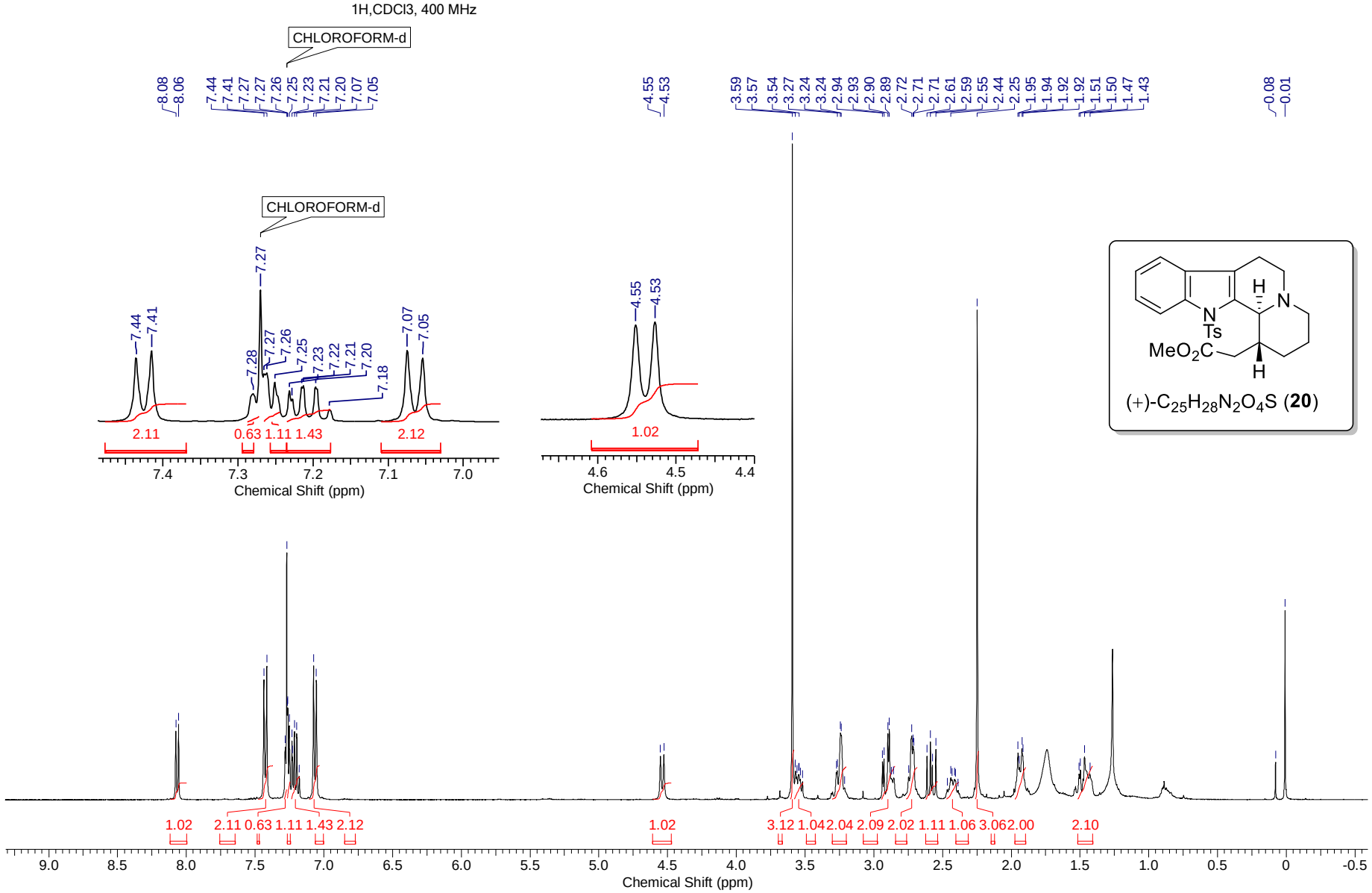

DEPT, CD3OD, 125 MHz
125.55
125.42
122.79
119.60
117.13
56.99
52.45
49.03
26.93
21.09
125.55
125.42
125.75 125.50 125.25 125.00
Chemical Shift (ppm)
H
N
N
O
(+)-C17H16N2O (18)
192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

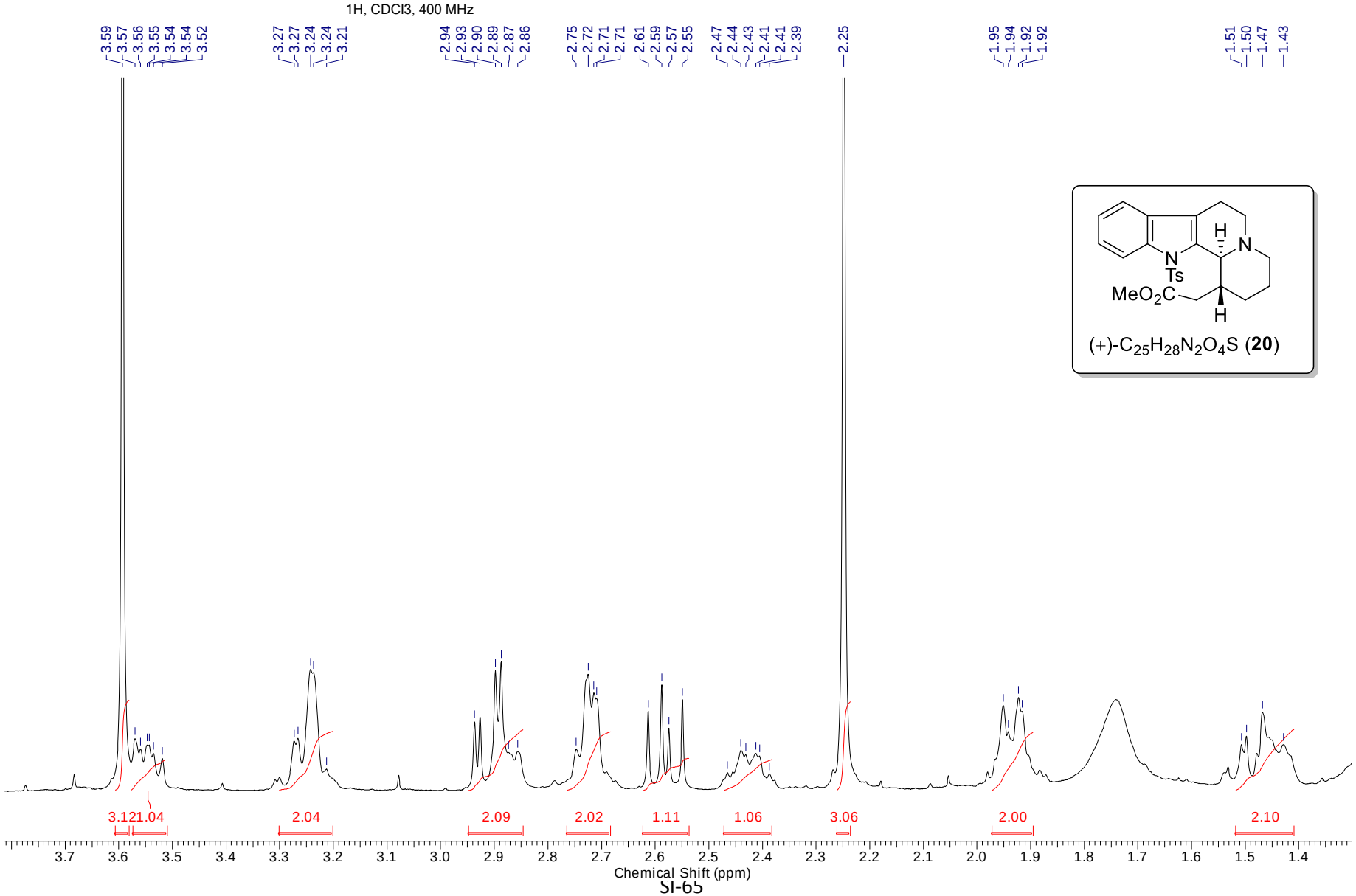

1H, CDCl3, 400 MHz
CHLOROFORM-d
8.07
8.04
7.46
7.44
7.27
7.25
7.24
7.23
7.22
7.20
7.18
7.05
7.03
3.83
3.52
3.32
3.31
3.02
2.99
2.91
2.89
2.60
2.56
2.53
2.52
2.51
2.49
2.42
2.38
2.26
1.96
1.95
1.92
1.91
1.88
1.56
1.54
(+)-C25H28N2O4S (19)
CHLOROFORM-d
2.02
3.01
2.02
Chemical Shift (ppm)
1.00
2.02
3.01
2.02
0.95
3.00
1.02
1.00
1.01
1.05
1.06
2.05
1.07
3.05
1.46
2.57
1.02
Chemical Shift (ppm)

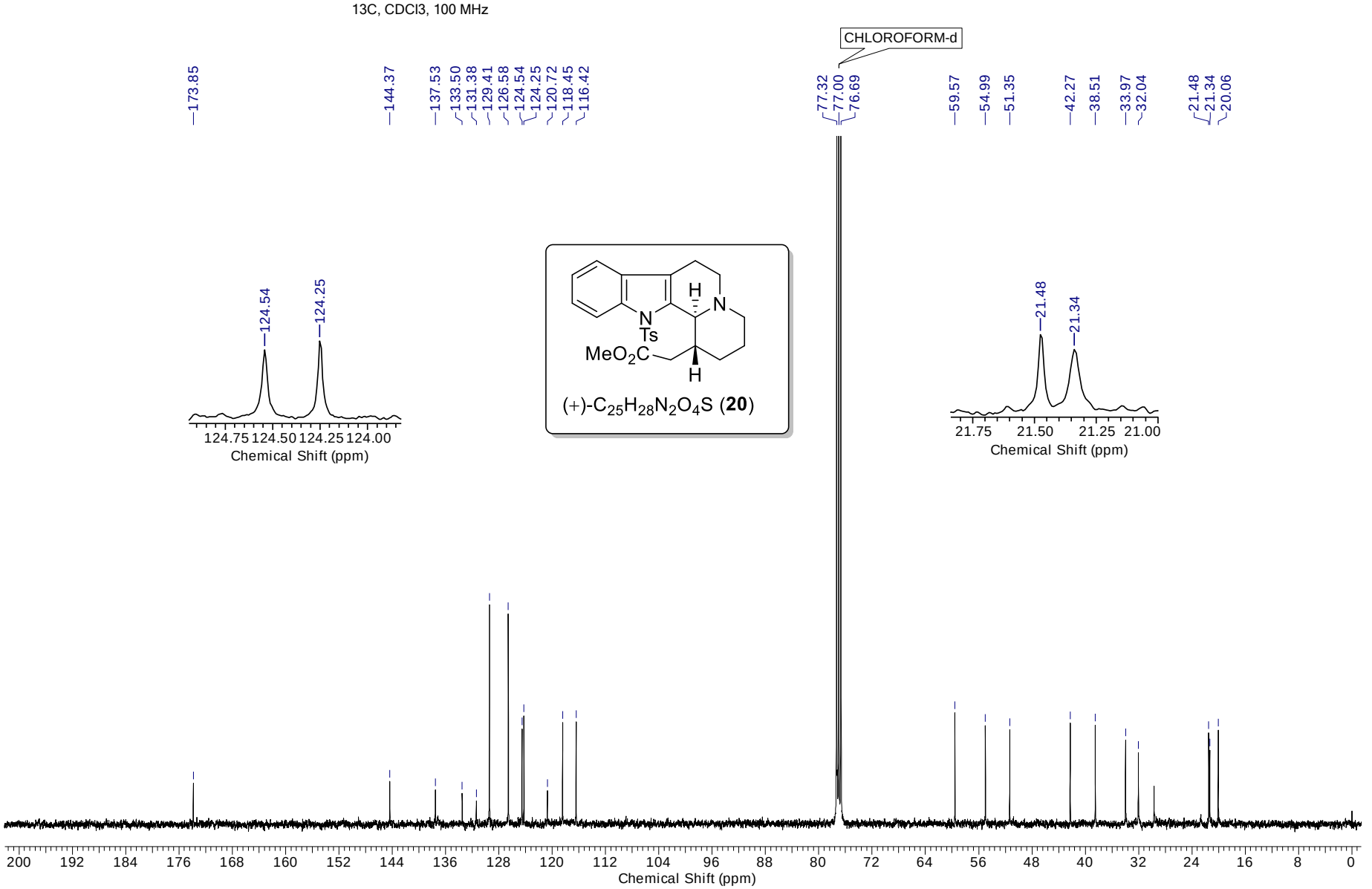

1H, CDCl3, 400 MHz
3.83
3.52
3.34
3.32
3.31
3.02
2.99
2.91
2.90
2.89
2.87
2.79
2.76
2.75
2.73
2.63
2.60
2.57
2.56
2.53
2.52
2.51
2.49
2.42
2.38
2.26
1.96
1.95
1.92
1.91
1.88
1.83
1.56
1.54
H
N
N
Ts
MeO2C
H
(+)-C25H28N2O4S (19)
0.95
3.00
1.02
1.00
1.01
1.05
1.06
2.05
1.07
3.05
1.46
2.57
1.02
4.0 3.9 3.8 3.7 3.6 3.5 3.4 3.3 3.2 3.1 3.0 2.9 2.8 2.7 2.6 2.5 2.4 2.3 2.2 2.1 2.0 1.9 1.8 1.7 1.6 1.5 1.4
Chemical Shift (ppm)

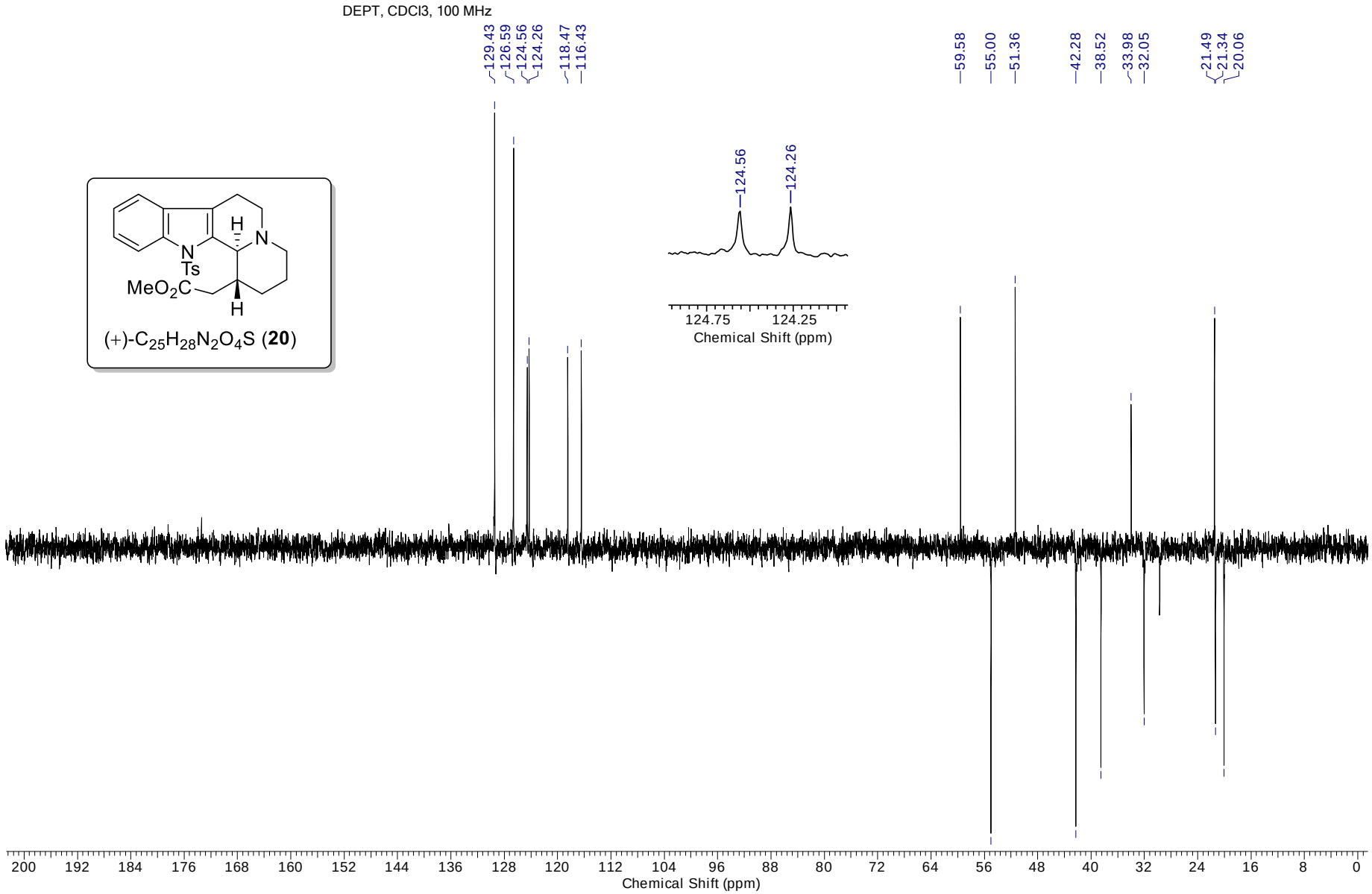

13C, CDCl3, 100 MHz
CHLOROFORM-d
174.00
144.31
138.82
136.44
133.17
131.24
128.99
126.77
125.29
124.59
124.38
118.28
116.91
77.00
64.94
56.67
51.35
51.23
35.56
32.48
28.55
22.89
21.48
21.23
(+)-C25H28N2O4S (19)
Chemical Shift (ppm)

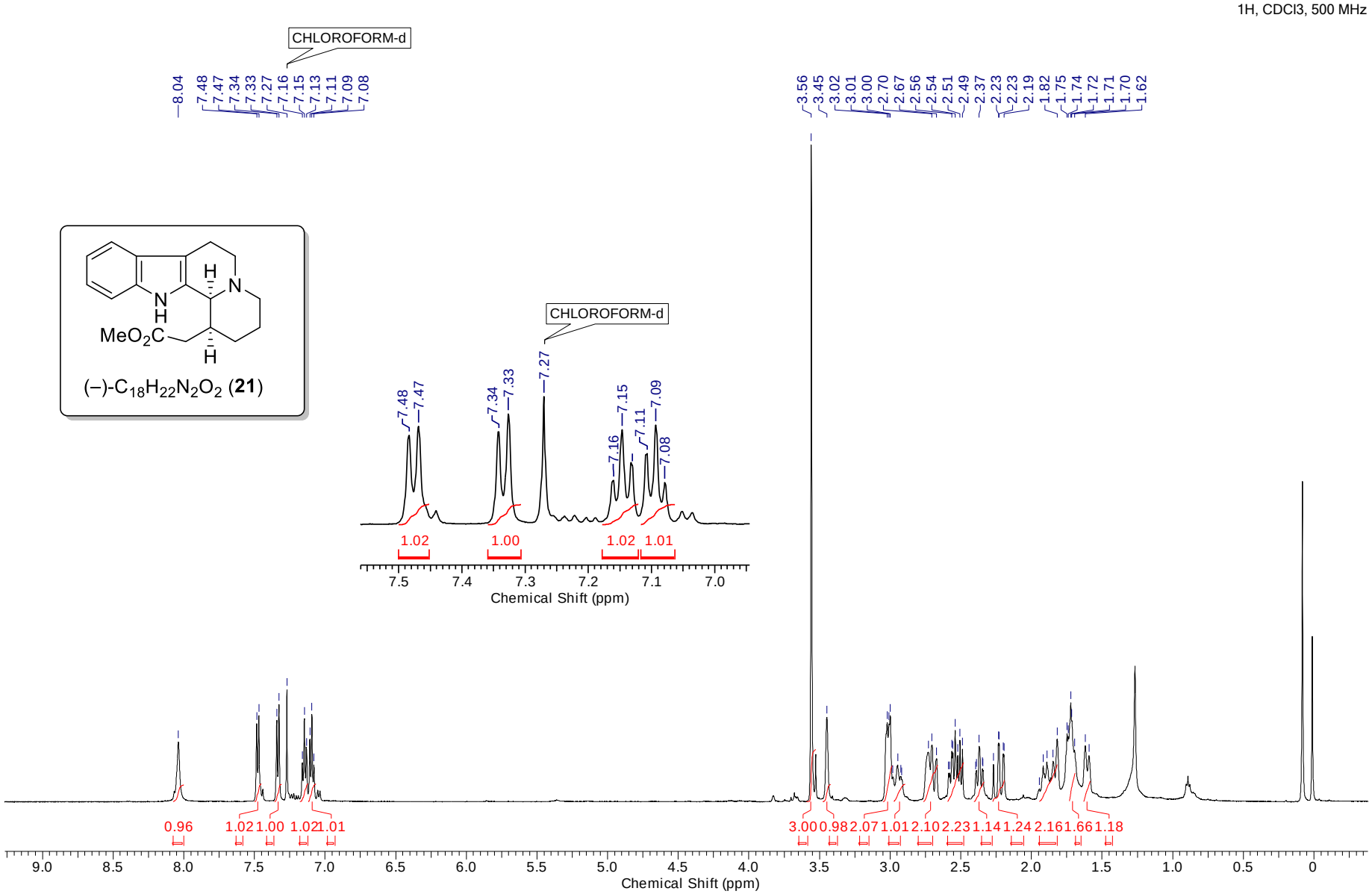

DEPT, CDCl3, 100 MHz
128.98
126.76
124.58
124.38
118.27
116.91
64.94
56.66
51.34
51.23
35.55
32.47
28.53
22.87
21.47
21.23
124.58
124.38
124.75 124.50 124.25
Chemical Shift (ppm)
(+)-C25H28N2O4S (19)
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

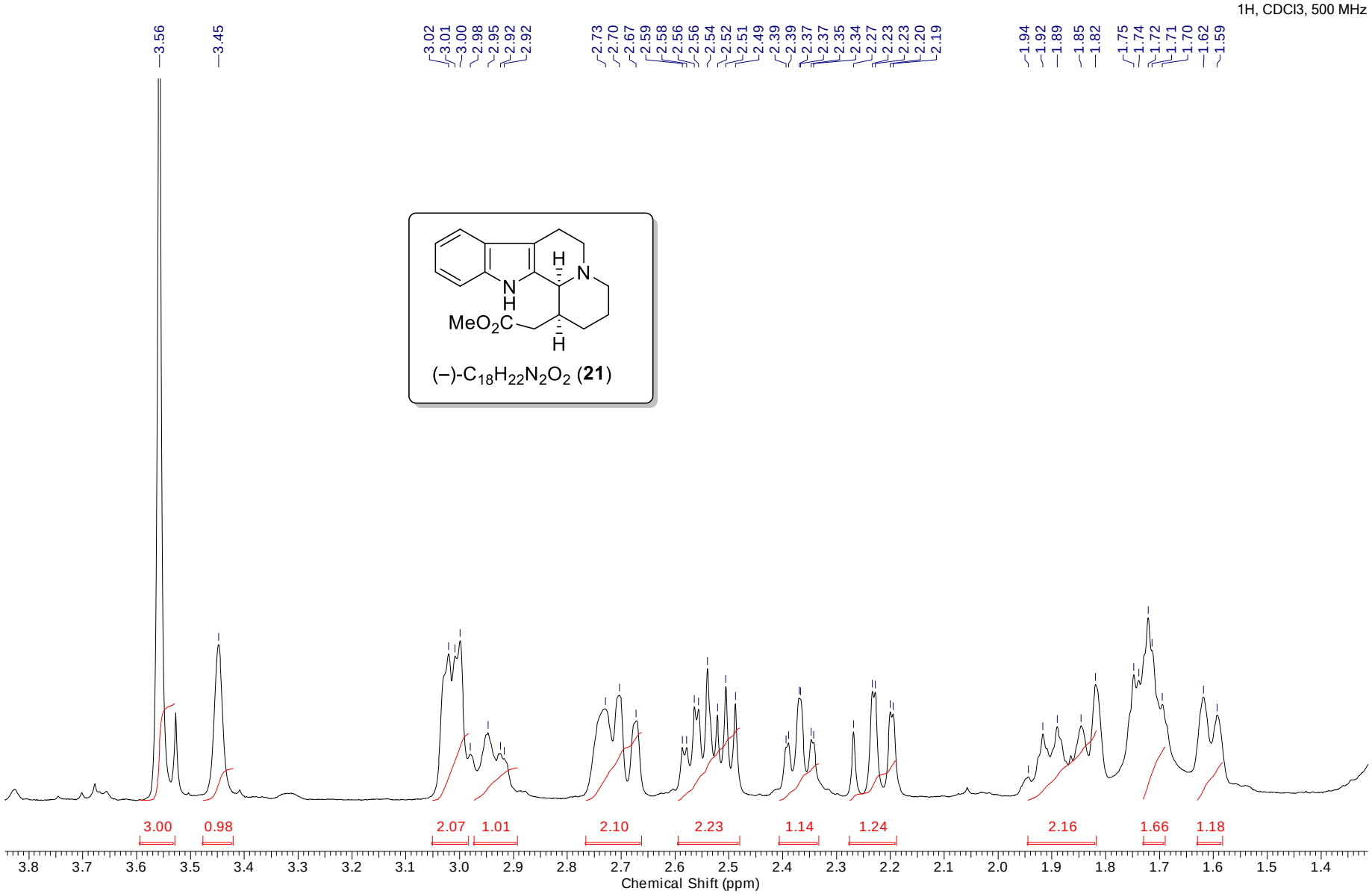

1H,CDCl3, 400 MHz
CHLOROFORM-d
8.08 8.06 7.44 7.41 7.27 7.27 7.26 7.25 7.23 7.21 7.20 7.07 7.05 4.55 4.53 3.59 3.57 3.54 3.27 3.24 3.24 2.94 2.93 2.90 2.89 2.72 2.71 2.71 2.61 2.59 2.55 2.44 2.25 1.95 1.94 1.92 1.92 1.51 1.50 1.47 1.43 0.08 0.01
CHLOROFORM-d
7.44 7.41 7.27 7.28 7.27 7.26 7.25 7.23 7.22 7.21 7.20 7.18 7.07 7.05
2.11 0.63 1.11 1.43 2.12
7.4 7.3 7.2 7.1 7.0
Chemical Shift (ppm)
4.55 4.53
1.02
4.6 4.5 4.4
Chemical Shift (ppm)
H N Ts MeO2C H
(+)-C25H28N2O4S (20)
1.02 2.11 0.63 1.11 1.43 2.12 1.02 3.12 1.04 2.04 2.09 2.02 1.11 1.06 3.06 2.00 2.10
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 -0.5
Chemical Shift (ppm)

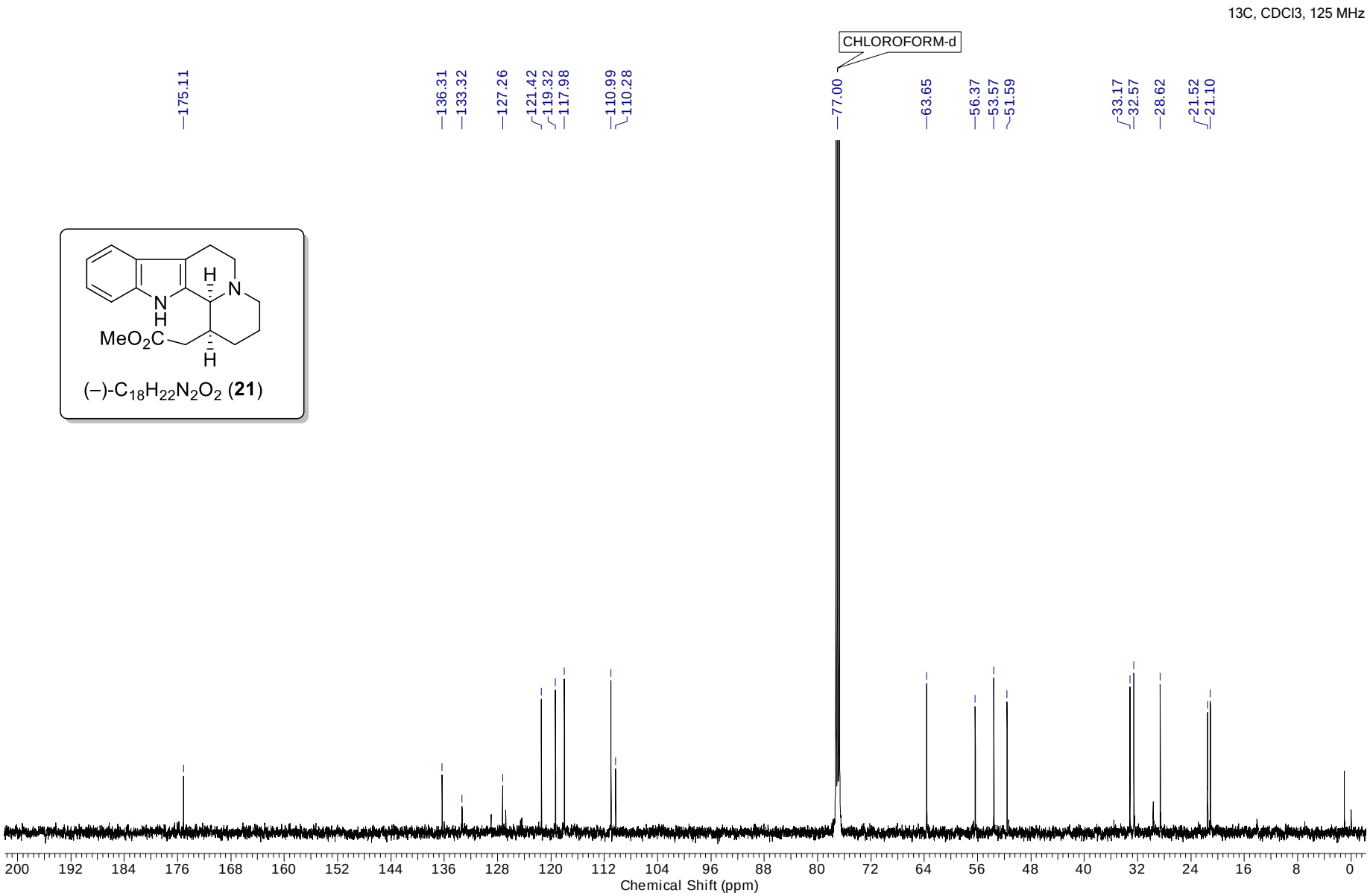

1H, CDCl3, 400 MHz
3.59
3.57
3.56
3.55
3.54
3.54
3.52
3.27
3.27
3.24
3.24
3.21
2.94
2.93
2.90
2.89
2.87
2.86
2.75
2.72
2.71
2.71
2.61
2.59
2.57
2.55
2.47
2.44
2.43
2.41
2.41
2.39
2.25
1.95
1.94
1.92
1.92
1.51
1.50
1.47
1.43
N
H
N
Ts
MeO2C
H
(+)-C25H28N2O4S (20)
3.12
1.04
2.04
2.09
2.02
1.11
1.06
3.06
2.00
2.10
Chemical Shift (ppm)


13C, CDCl3, 100 MHz
CHLOROFORM-d
173.85
144.37
137.53
133.50
131.38
129.41
126.58
124.54
124.25
120.72
118.45
116.42
77.32
77.00
76.69
59.57
54.99
51.35
42.27
38.51
33.97
32.04
21.48
21.34
20.06
124.54
124.25
124.75 124.50 124.25 124.00
Chemical Shift (ppm)
21.48
21.34
21.75 21.50 21.25 21.00
Chemical Shift (ppm)
(+)-C25H28N2O4S (20)
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

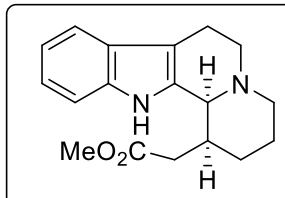

DEPT, CDCl3, 100 MHz
129.43
126.59
124.56
124.26
118.47
116.43
59.58
55.00
51.36
42.28
38.52
33.98
32.05
21.49
21.34
20.06
124.56
124.26
124.75
124.25
Chemical Shift (ppm)
(+)-C25H28N2O4S (20)
Chemical Shift (ppm)


1H, CDCl3, 500 MHz
CHLOROFORM-d
8.04
7.48
7.47
7.34
7.33
7.27
7.16
7.15
7.13
7.11
7.09
7.08
3.56
3.45
3.02
3.01
3.00
2.70
2.67
2.56
2.54
2.51
2.49
2.37
2.23
2.23
2.19
1.82
1.75
1.74
1.72
1.71
1.70
1.62
(–)-C18H22N2O2 (21)
CHLOROFORM-d
1.02
1.00
1.02
1.01
Chemical Shift (ppm)
0.96
1.02
1.00
1.02
1.01
3.00
0.98
2.07
1.01
2.10
2.23
1.14
1.24
2.16
1.66
1.18
Chemical Shift (ppm)


1H, CDCl3, 500 MHz
3.56
3.45
3.02
3.01
3.00
2.98
2.95
2.92
2.92
2.73
2.70
2.67
2.59
2.58
2.56
2.56
2.54
2.52
2.51
2.49
2.39
2.39
2.37
2.37
2.35
2.34
2.27
2.23
2.23
2.20
2.19
1.94
1.92
1.89
1.85
1.82
1.75
1.74
1.72
1.71
1.70
1.62
1.59
H
N
N
H
MeO2C
H
(–)-C18H22N2O2 (21)
3.00
0.98
2.07
1.01
2.10
2.23
1.14
1.24
2.16
1.66
1.18
3.8
3.7
3.6
3.5
3.4
3.3
3.2
3.1
3.0
2.9
2.8
2.7
2.6
2.5
2.4
2.3
2.2
2.1
2.0
1.9
1.8
1.7
1.6
1.5
1.4
Chemical Shift (ppm)


13C, CDCl3, 125 MHz
CHLOROFORM-d
175.11
136.31
133.32
127.26
121.42
119.32
117.98
110.99
110.28
77.00
63.65
56.37
53.57
51.59
33.17
32.57
28.62
21.52
21.10
(–)-C18H22N2O2 (21)
Chemical Shift (ppm)

DEPT, CDCl3, 125 MHz

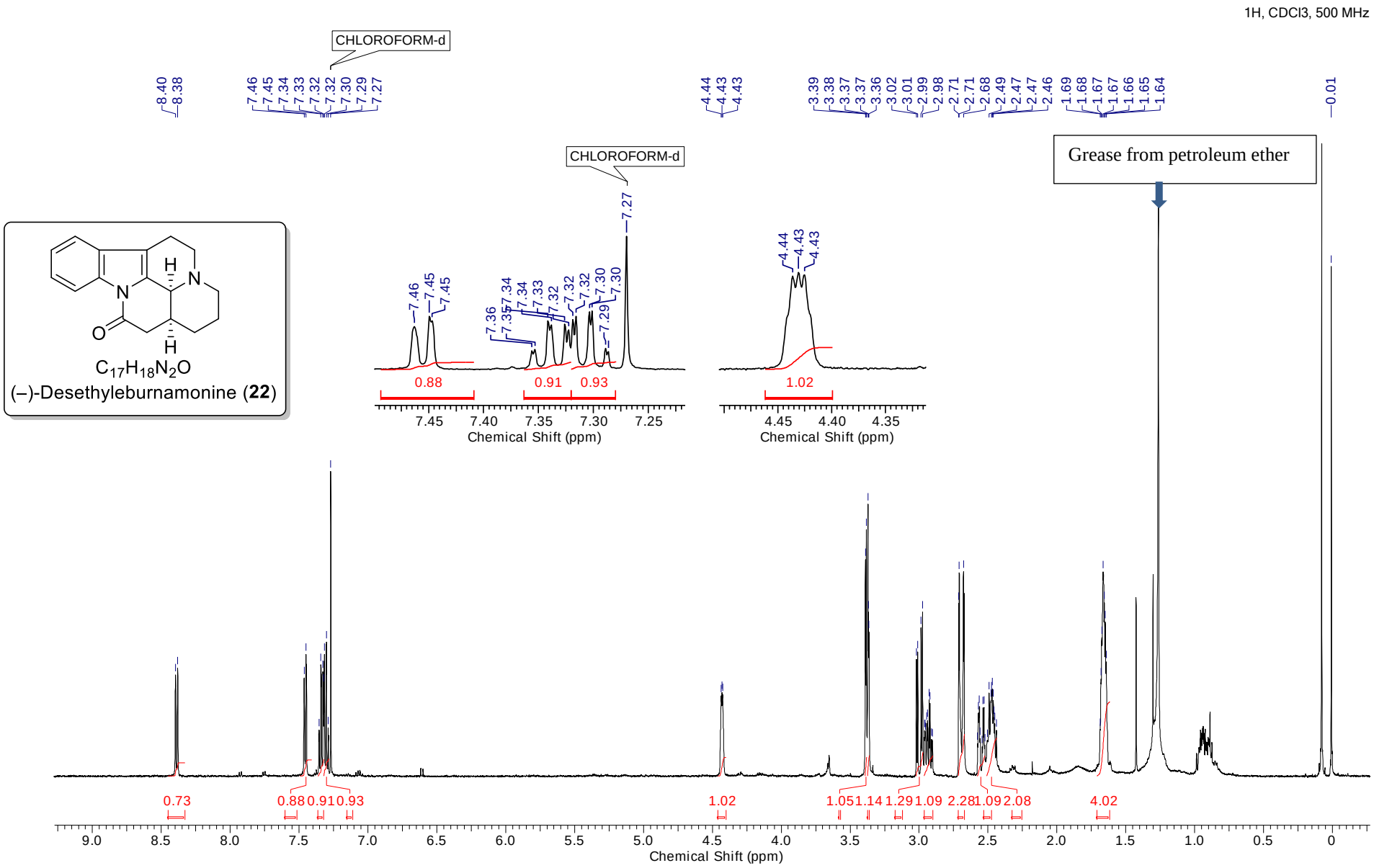


(–)-$C_{18}H_{22}N_2O_2$ (**21**)

121.42
119.31
117.98
110.99
63.65
56.36
53.57
51.58
33.16
32.57
28.61
21.52
21.08

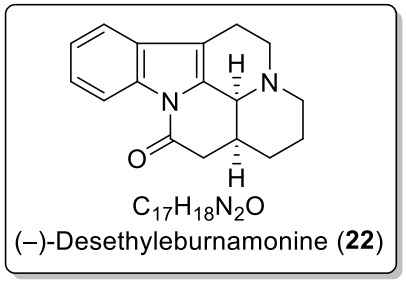
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0

Chemical Shift (ppm)


1H, CDCl3, 500 MHz
CHLOROFORM-d
8.40
8.38
7.46
7.45
7.34
7.33
7.32
7.32
7.30
7.29
7.27
4.44
4.43
4.43
3.39
3.38
3.37
3.37
3.36
3.02
3.01
2.99
2.98
2.71
2.71
2.68
2.49
2.47
2.47
2.46
1.69
1.68
1.67
1.67
1.66
1.65
1.64
0.01
Grease from petroleum ether
CHLOROFORM-d
7.27
7.46
7.45
7.45
7.36
7.35
7.34
7.34
7.33
7.32
7.32
7.32
7.30
7.30
7.29
0.88
0.91
0.93
7.45
7.40
7.35
7.30
7.25
Chemical Shift (ppm)
4.44
4.43
4.43
1.02
4.45
4.40
4.35
Chemical Shift (ppm)
H
N
N
O
H
C17H18N2O
(–)-Desethyleburnamonine (22)
0.73
0.88
0.91
0.93
1.02
1.05
1.14
1.29
1.09
2.28
1.09
2.08
4.02
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
Chemical Shift (ppm)

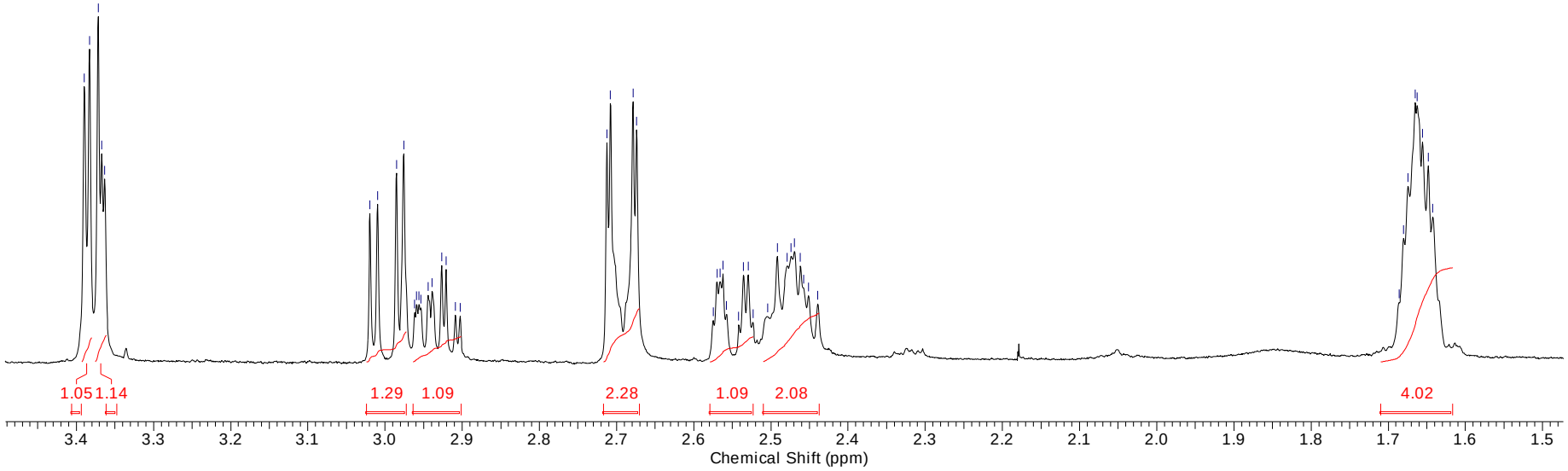
1H, CDCl3, 500 MHz

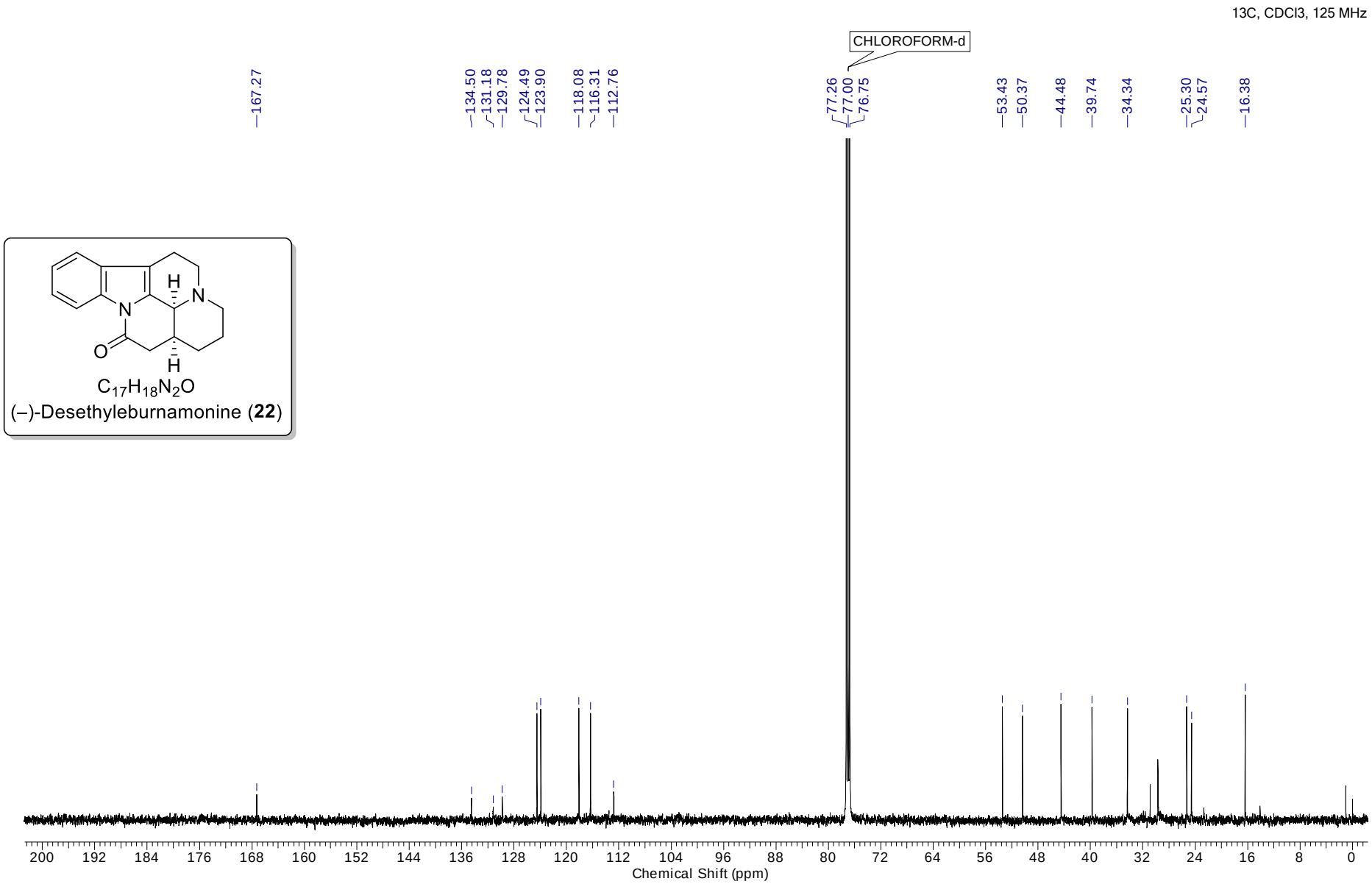
3.39 3.38 3.37 3.37 3.36 3.02 3.01 2.99 2.98 2.96 2.96 2.95 2.94 2.94 2.93 2.92 2.91 2.71 2.71 2.68 2.67 2.57 2.57 2.57 2.56 2.54 2.53 2.49 2.48 2.47 2.47 2.46 2.46 2.44 1.69 1.68 1.67 1.67 1.66 1.66 1.65 1.64

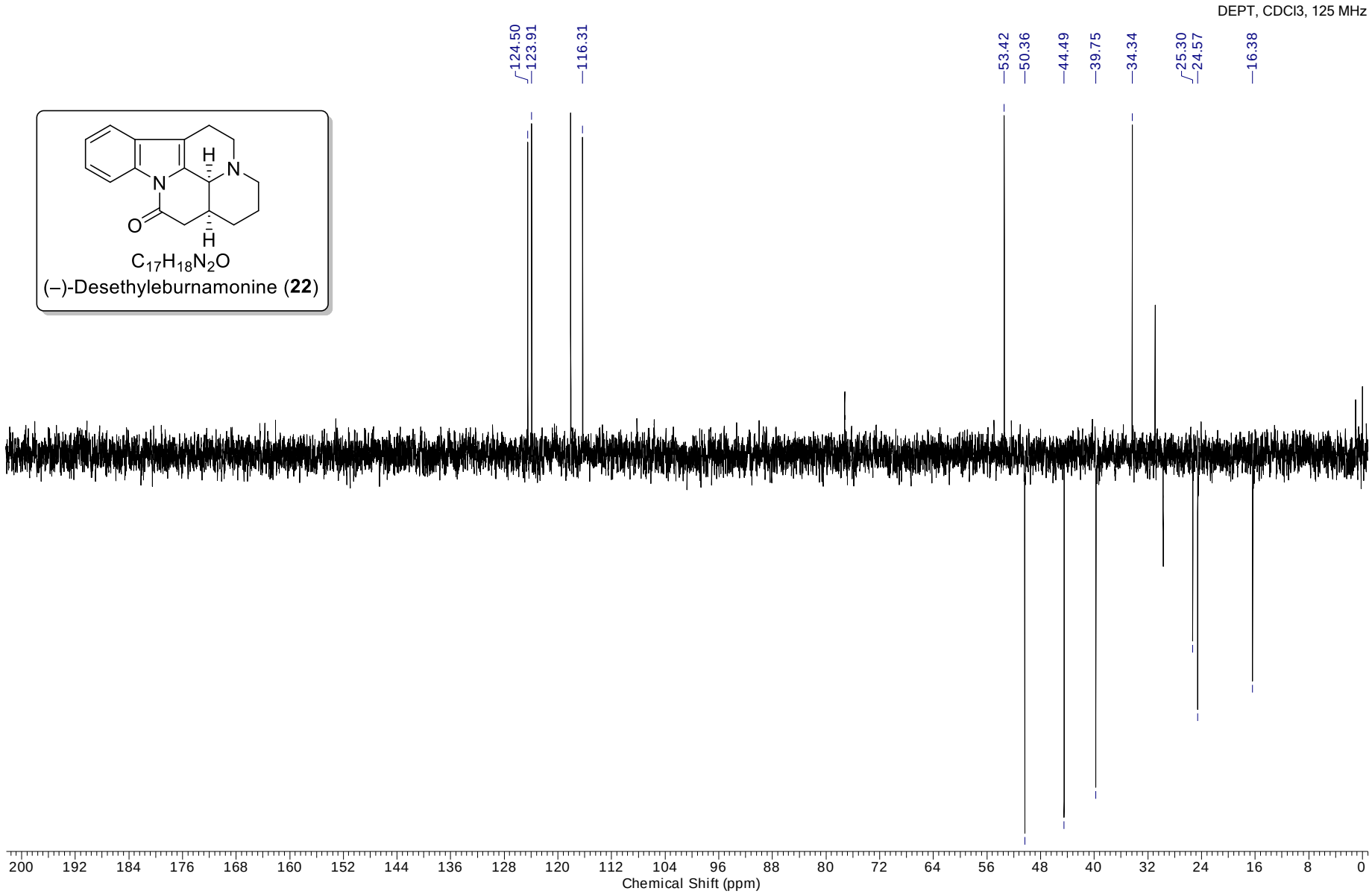


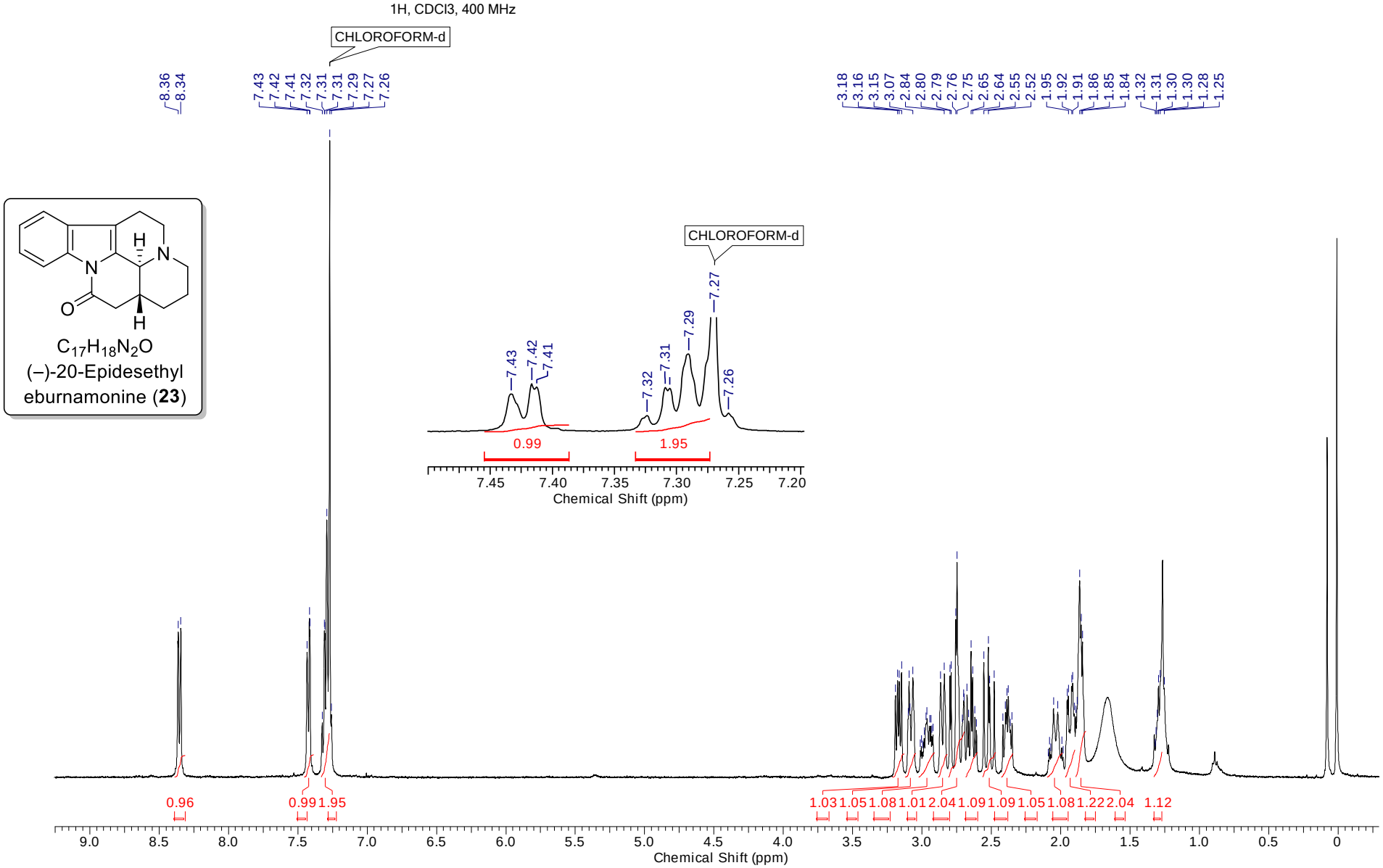
$C_{17}H_{18}N_2O$

(–)-Desethyleburnamonine (**22**)



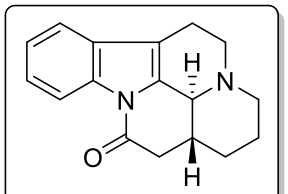

13C, CDCl3, 125 MHz
CHLOROFORM-d
167.27
134.50
131.18
129.78
124.49
123.90
118.08
116.31
112.76
77.26
77.00
76.75
53.43
50.37
44.48
39.74
34.34
25.30
24.57
16.38
H
N
N
O
H
C17H18N2O
(–)-Desethyleburnamonine (22)
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)


DEPT, CDCl3, 125 MHz
124.50
123.91
116.31
53.42
50.36
44.49
39.75
34.34
25.30
24.57
16.38
H
N
N
O
H
C17H18N2O
(–)-Desethyleburnamonine (22)
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

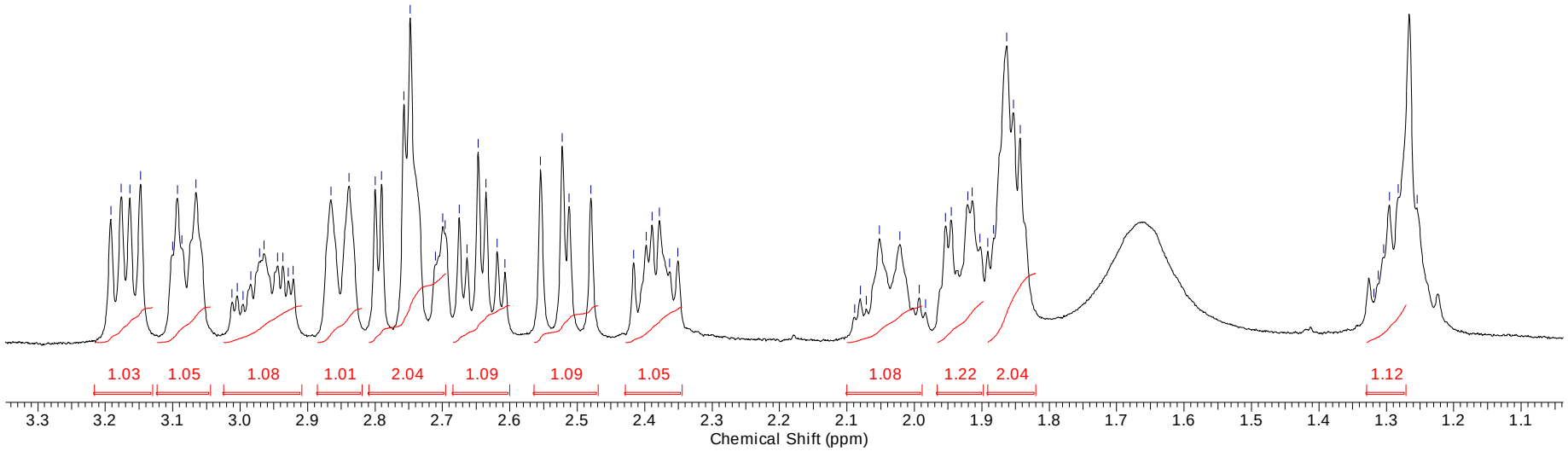

1H, CDCl3, 400 MHz
CHLOROFORM-d
8.36
8.34
7.43
7.42
7.41
7.32
7.31
7.31
7.29
7.27
7.26
3.18
3.16
3.15
3.07
2.84
2.80
2.79
2.76
2.75
2.65
2.64
2.55
2.52
1.95
1.92
1.91
1.86
1.85
1.84
1.32
1.31
1.30
1.30
1.28
1.25
H
N
N
O
H
C17H18N2O
(–)-20-Epidesethyl
eburnamonine (23)
CHLOROFORM-d
7.43
7.42
7.41
7.32
7.31
7.29
7.27
7.26
0.99
1.95
7.45
7.40
7.35
7.30
7.25
7.20
Chemical Shift (ppm)
0.96
0.99
1.95
1.03
1.05
1.08
1.01
2.04
1.09
1.09
1.05
1.08
1.22
2.04
1.12
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
Chemical Shift (ppm)

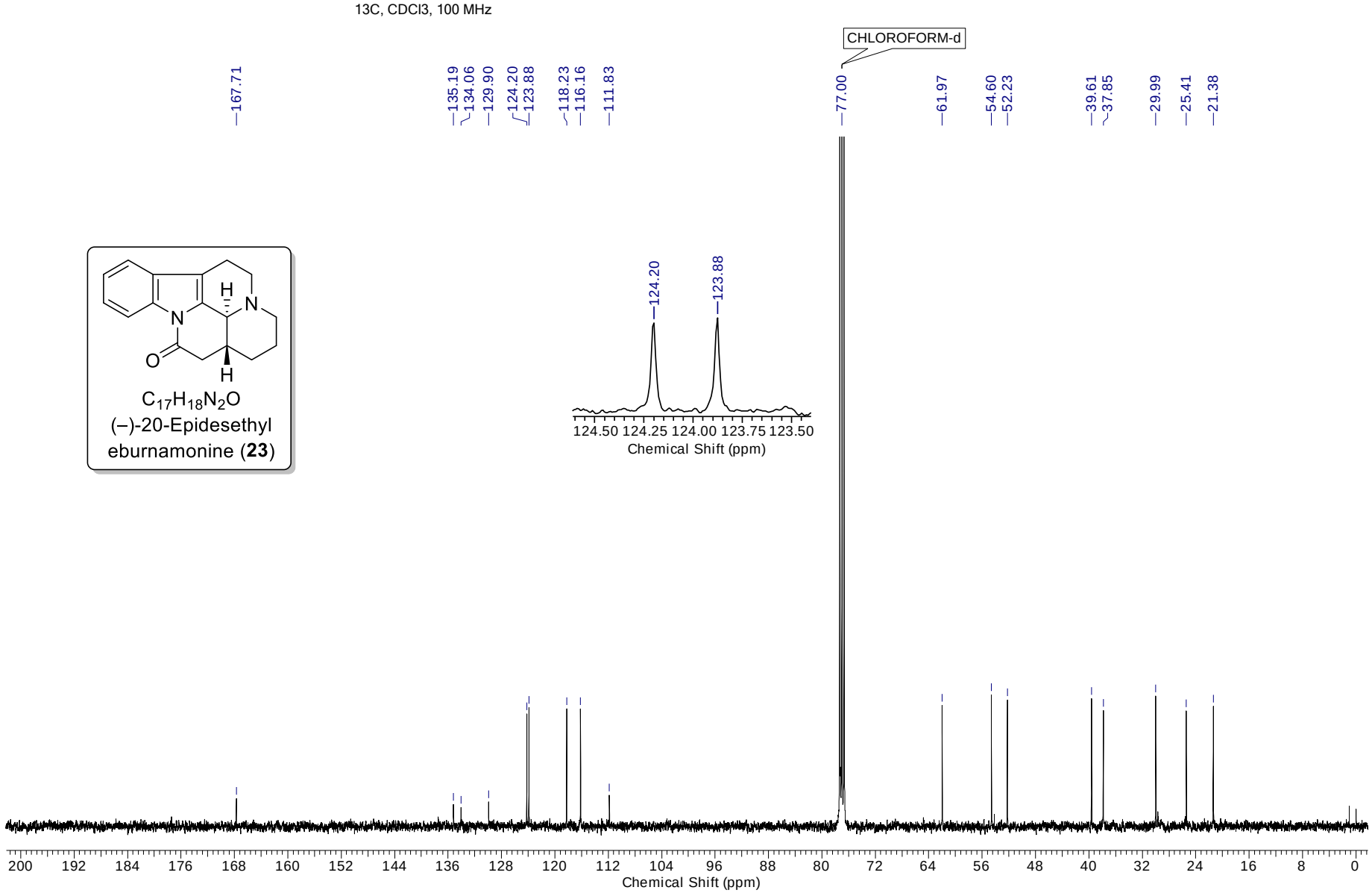
1H, CDCl3, 400 MHz

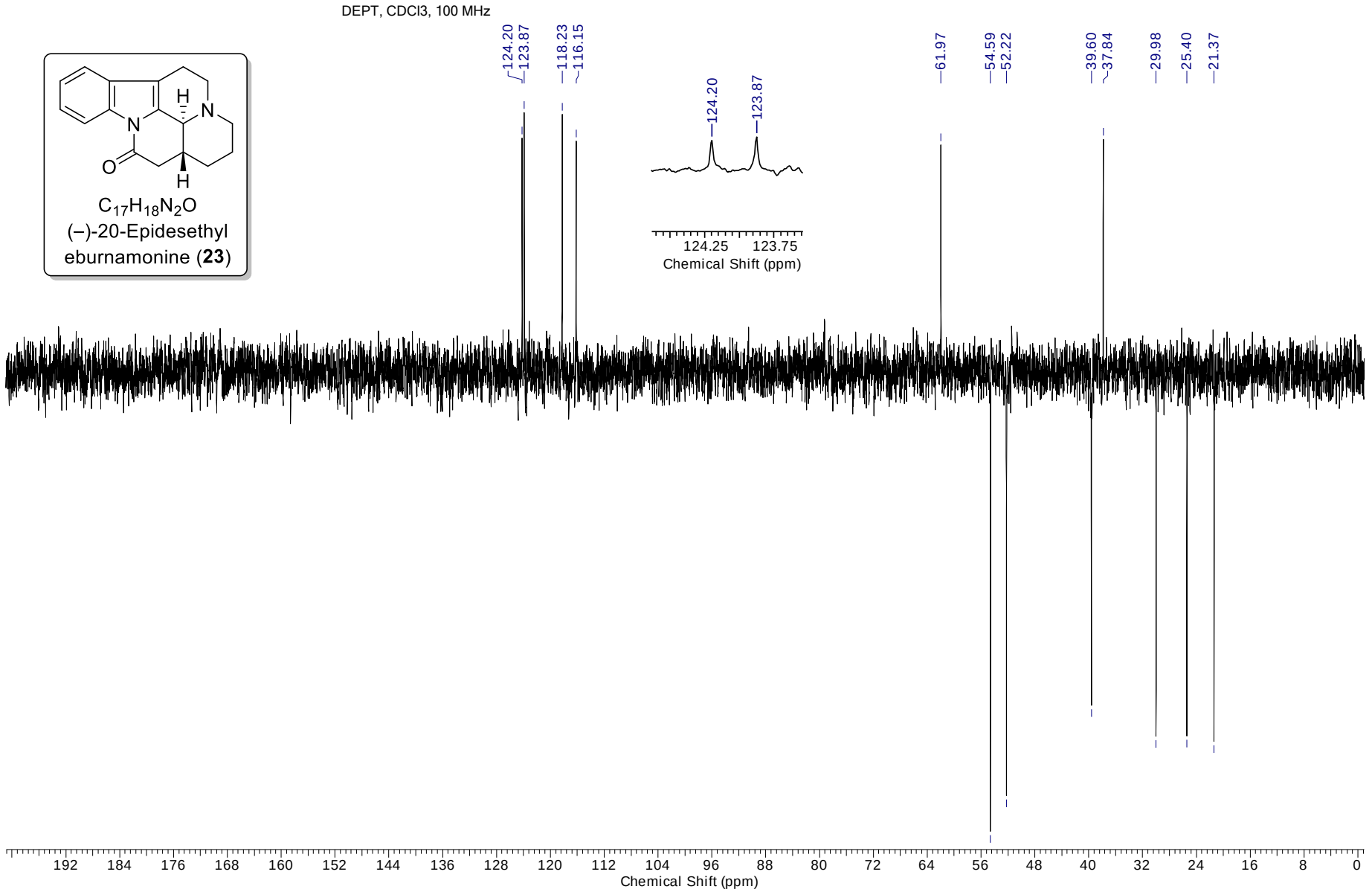
3.19 3.18 3.16 3.15 3.10 3.09 3.09 3.07 2.97 2.96 2.94 2.94 2.92 2.87 2.84 2.80 2.79 2.76 2.75 2.71 2.70 2.70 2.67 2.66 2.65 2.64 2.62 2.61 2.55 2.52 2.51 2.48 2.42 2.40 2.39 2.38 2.36 2.35 2.09 2.08 2.07 2.05 2.02 1.99 1.95 1.95 1.92 1.91 1.90 1.89 1.88 1.86 1.85 1.84 1.32 1.31 1.30 1.30 1.28 1.25

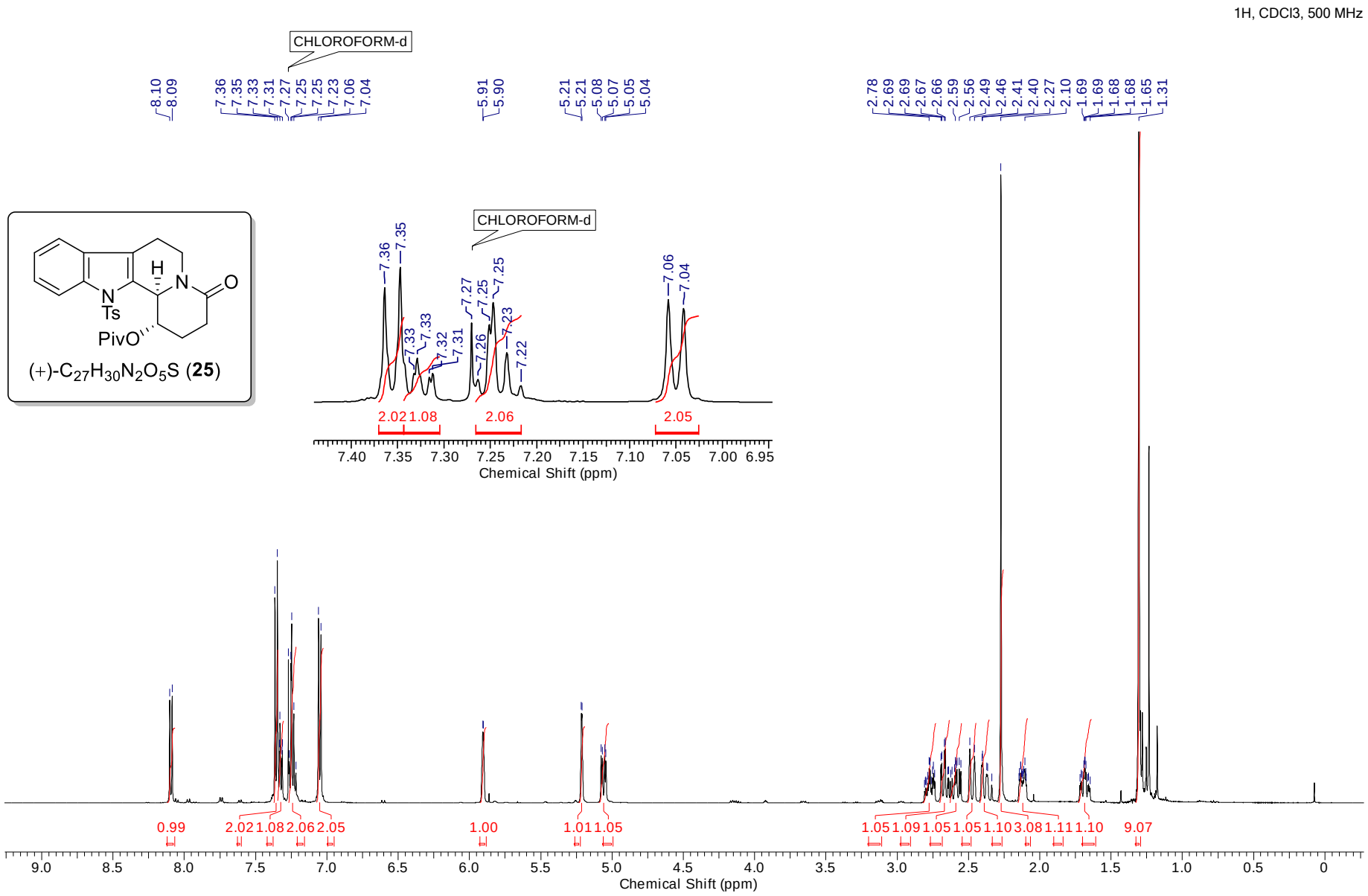


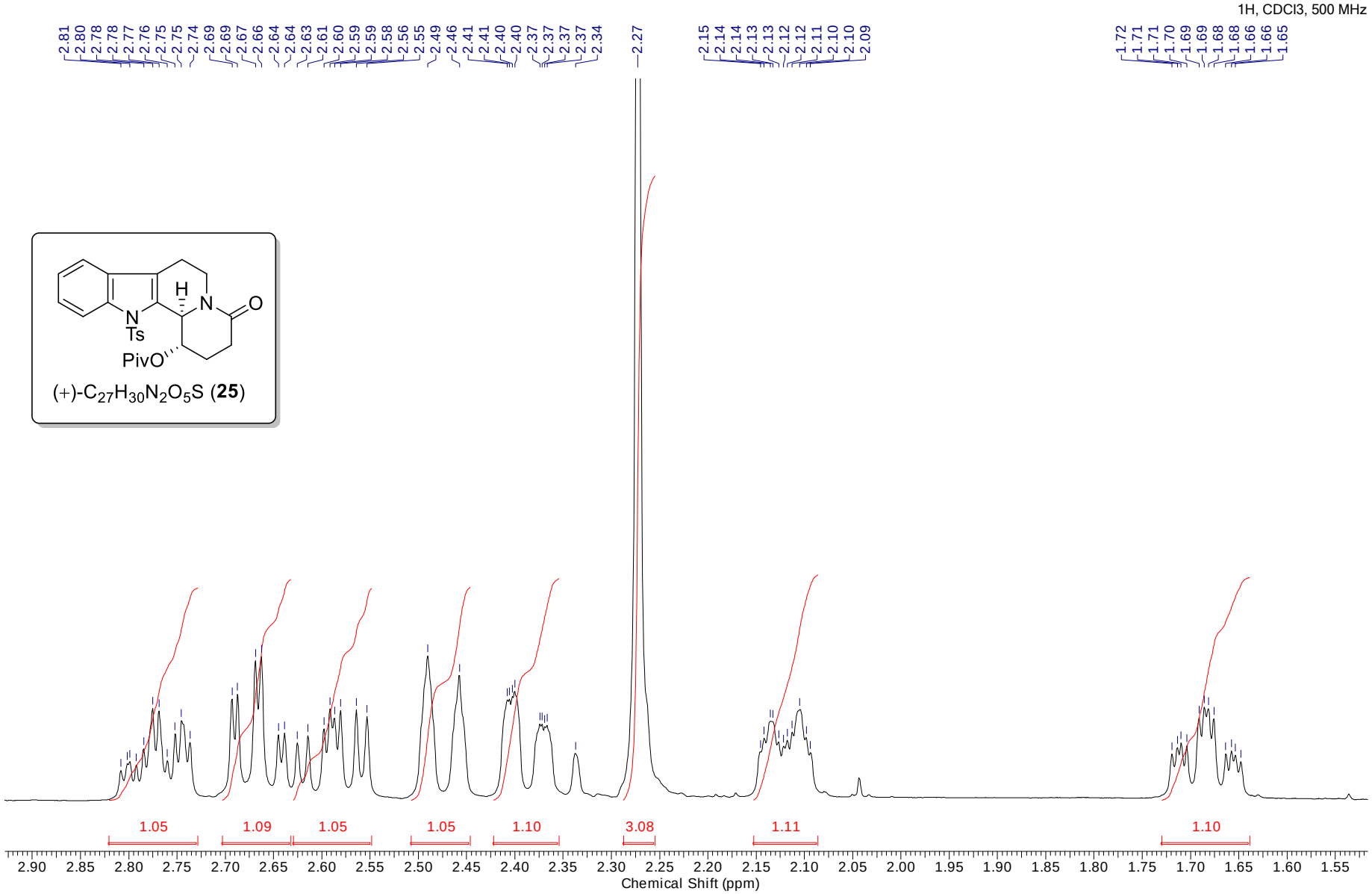
$C_{17}H_{18}N_2O$
(–)-20-Epidesethyl eburnamonine (**23**)

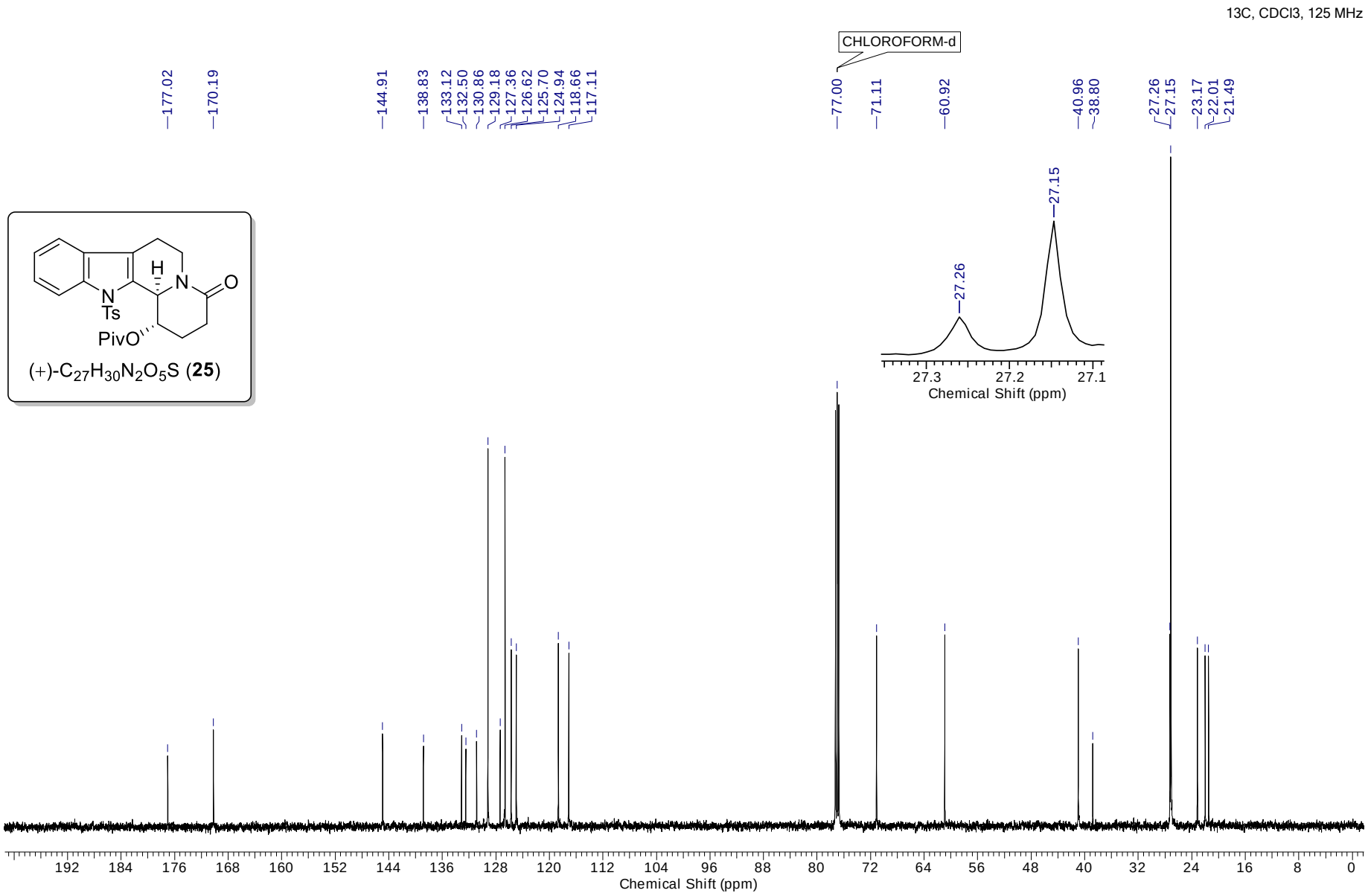


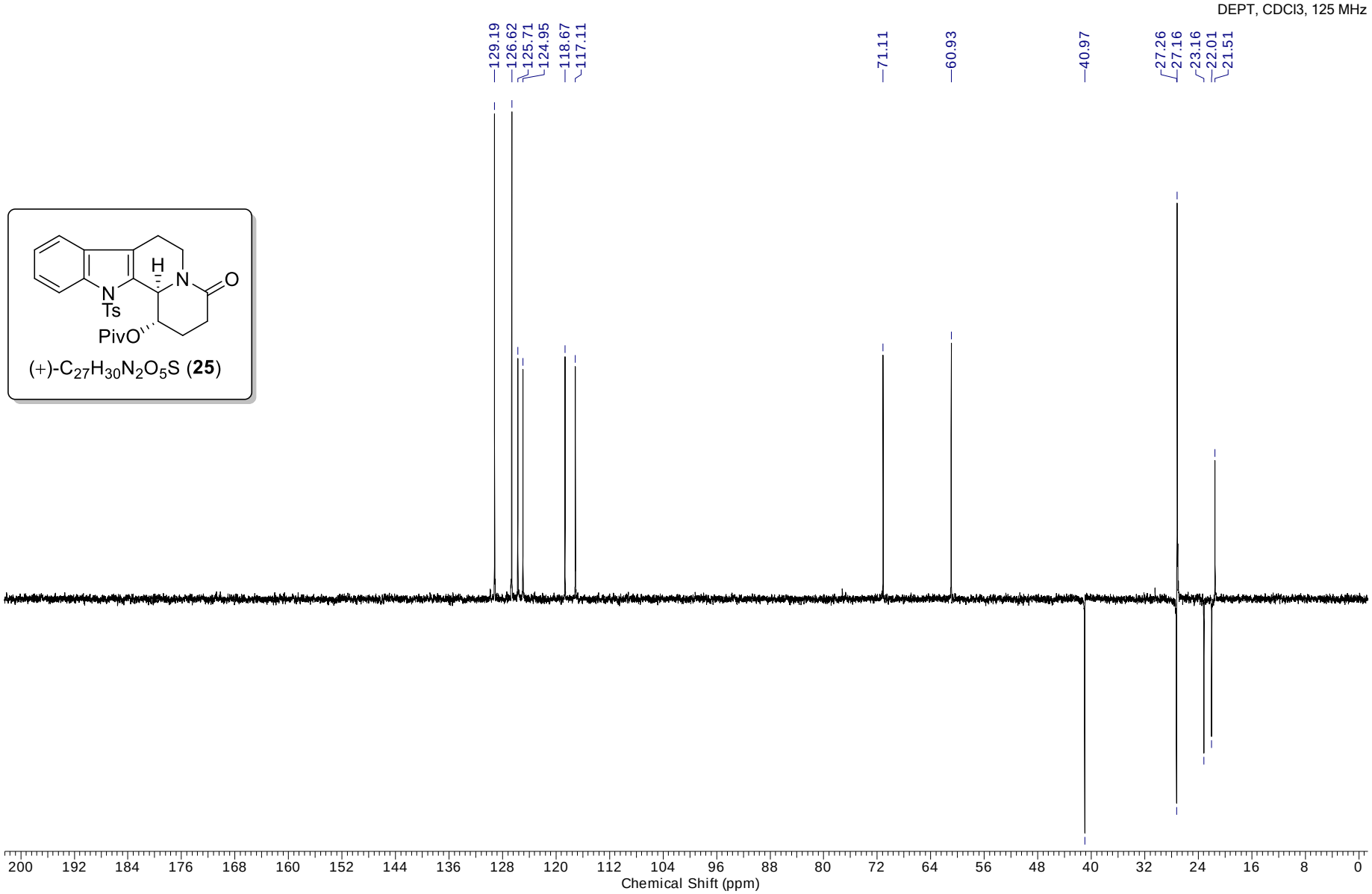

13C, CDCl3, 100 MHz
CHLOROFORM-d
167.71
135.19
134.06
129.90
124.20
123.88
118.23
116.16
111.83
77.00
61.97
54.60
52.23
39.61
37.85
29.99
25.41
21.38
C17H18N2O
(–)-20-Epidesethyl eburnamonine (23)
124.50 124.25 124.00 123.75 123.50
Chemical Shift (ppm)
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

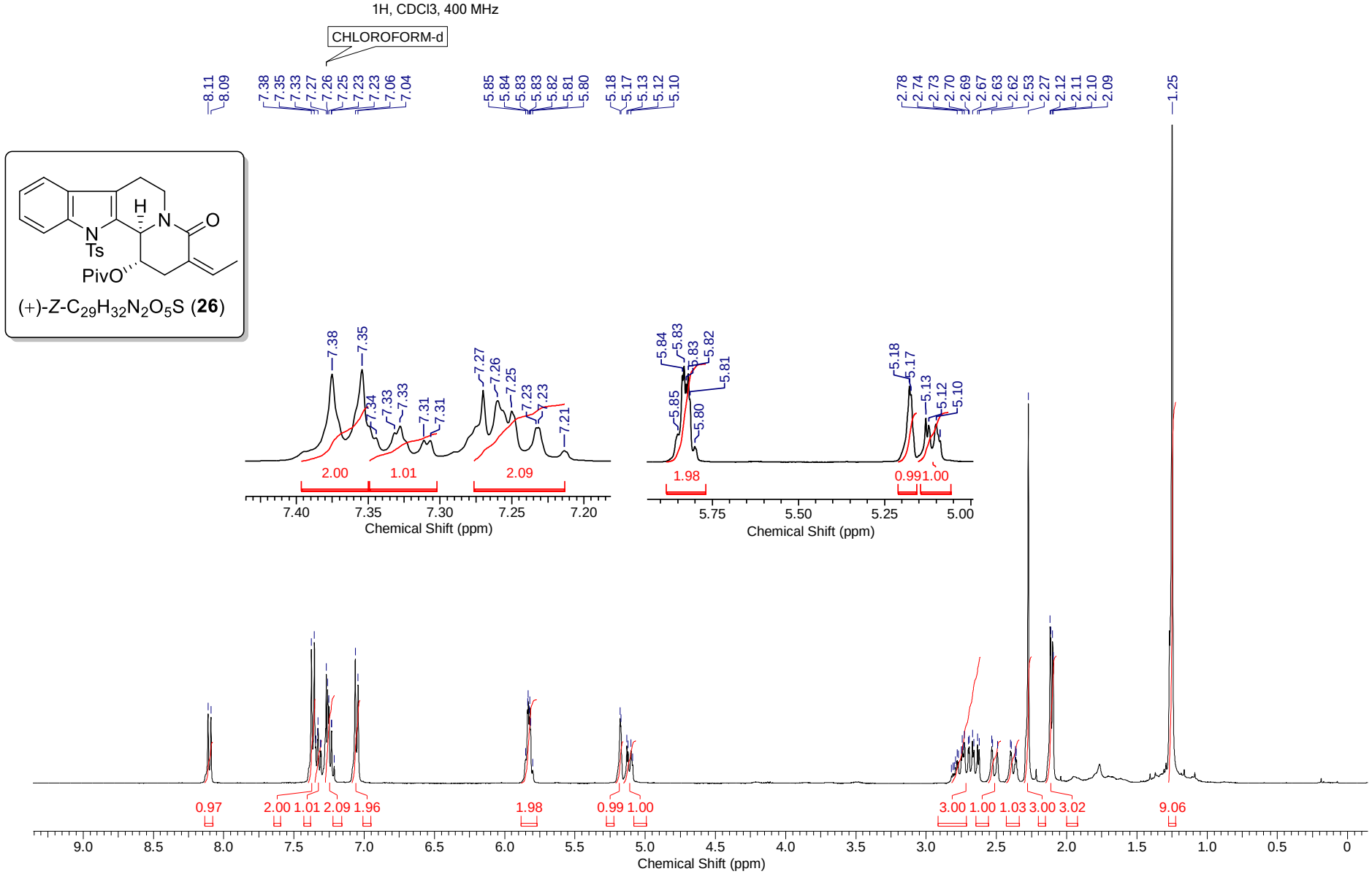

DEPT, CDCl3, 100 MHz
124.20
123.87
118.23
116.15
61.97
54.59
52.22
39.60
37.84
29.98
25.40
21.37
124.25
123.75
Chemical Shift (ppm)
$C_{17}H_{18}N_2O$
(–)-20-Epidesethyl eburnamonine (**23**)
Chemical Shift (ppm)

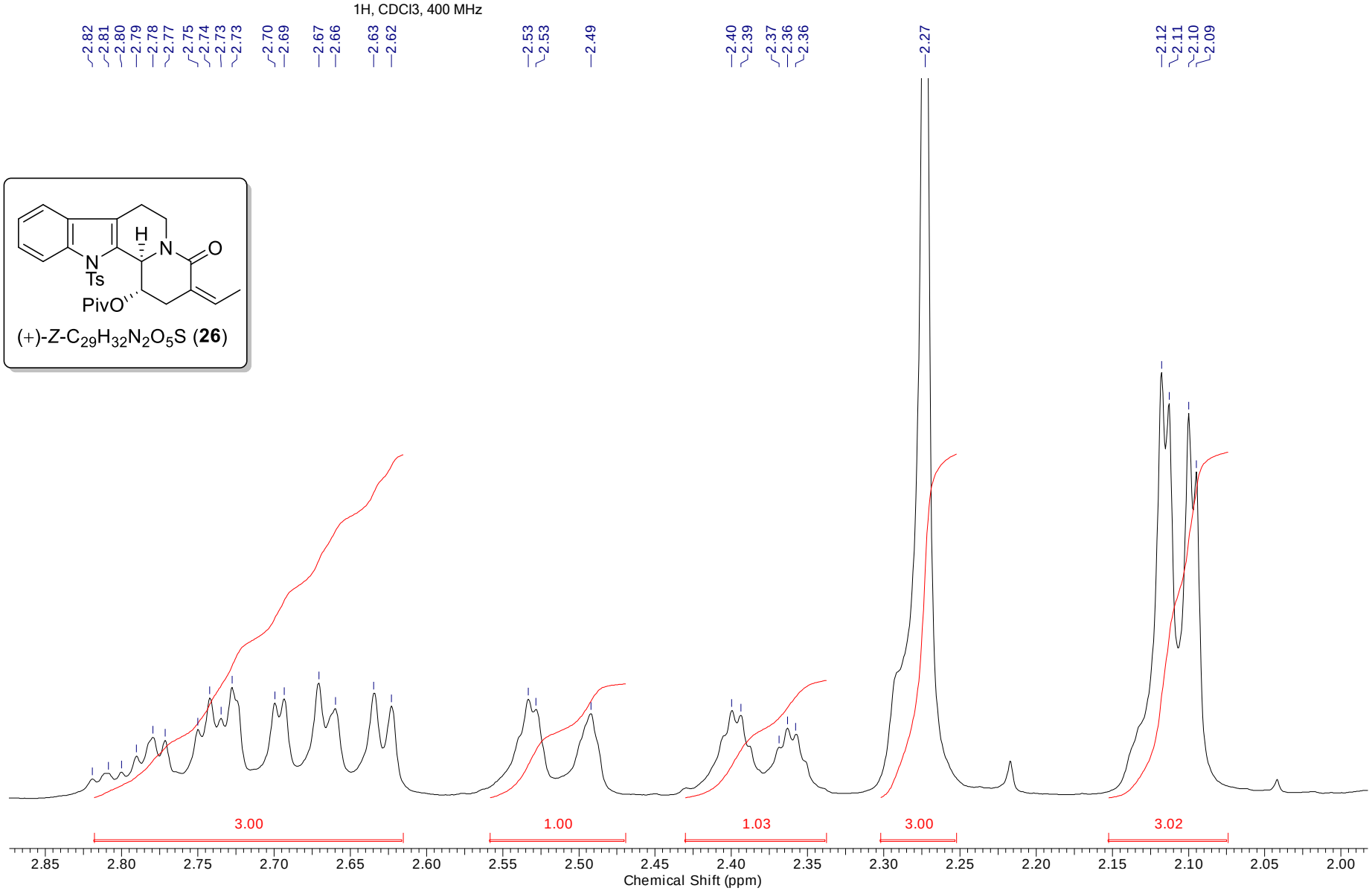

1H, CDCl3, 500 MHz
CHLOROFORM-d
8.10
8.09
7.36
7.35
7.33
7.31
7.27
7.25
7.25
7.23
7.06
7.04
5.91
5.90
5.21
5.21
5.08
5.07
5.05
5.04
2.78
2.69
2.69
2.67
2.66
2.59
2.56
2.49
2.46
2.41
2.40
2.27
2.10
1.69
1.69
1.68
1.68
1.65
1.31
N
H
N
O
Ts
PivO
(+)-C27H30N2O5S (25)
CHLOROFORM-d
7.36
7.35
7.33
7.33
7.32
7.31
7.27
7.25
7.26
7.25
7.23
7.22
7.06
7.04
2.02
1.08
2.06
2.05
7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95
Chemical Shift (ppm)
0.99
2.02
1.08
2.06
2.05
1.00
1.01
1.05
1.05
1.09
1.05
1.05
1.10
3.08
1.11
1.10
9.07
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
Chemical Shift (ppm)

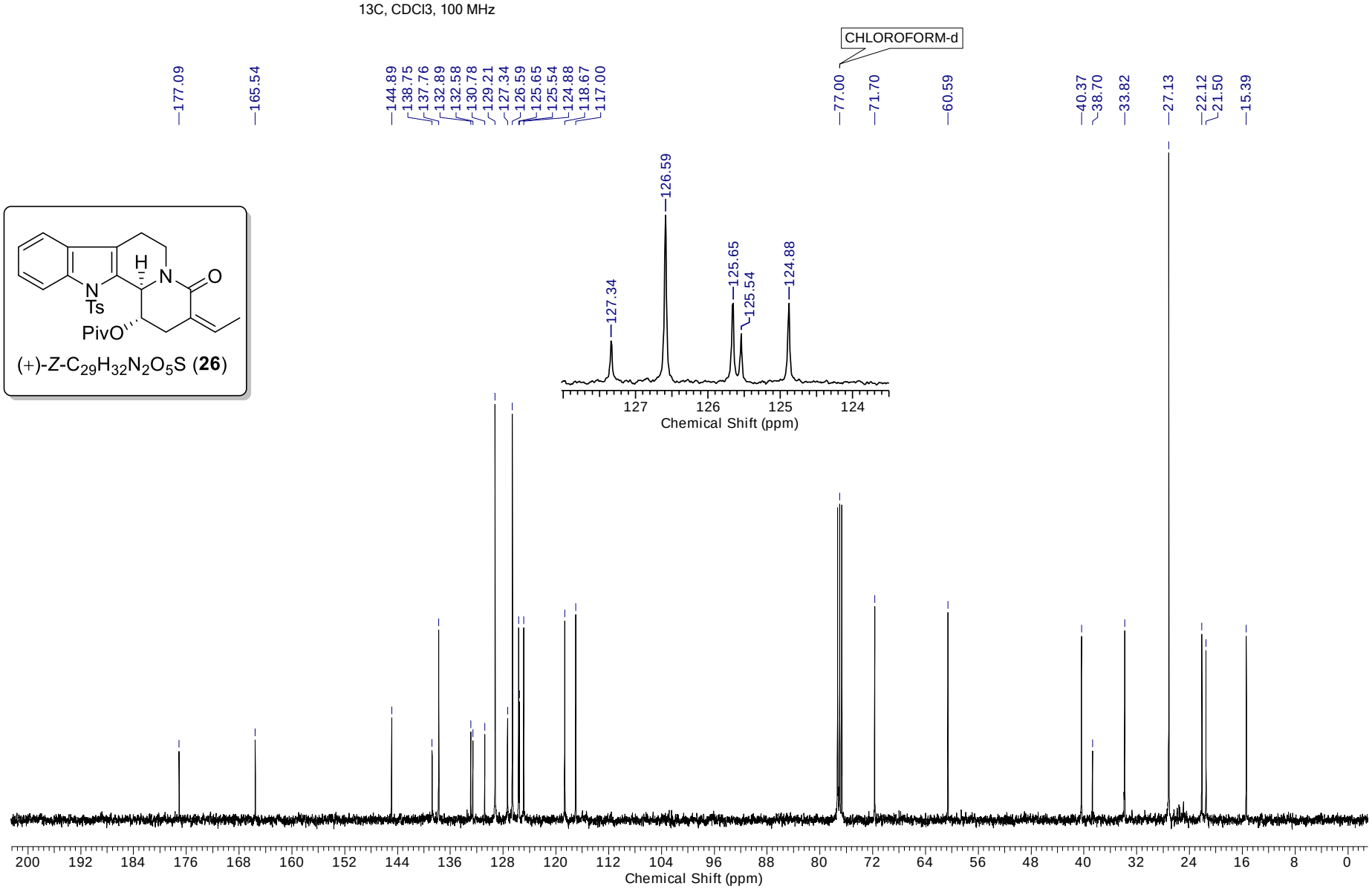

1H, CDCl3, 500 MHz
2.81 2.80 2.78 2.78 2.77 2.76 2.75 2.75 2.74 2.69 2.69 2.67 2.66 2.64 2.64 2.63 2.61 2.60 2.59 2.59 2.58 2.56 2.55 2.49 2.46 2.41 2.41 2.40 2.40 2.37 2.37 2.37 2.37 2.34
2.27
2.15 2.14 2.14 2.13 2.13 2.12 2.12 2.11 2.10 2.10 2.09
1.72 1.71 1.71 1.70 1.69 1.69 1.68 1.68 1.66 1.66 1.65
H
N
O
N
Ts
PivO
(+)-C27H30N2O5S (25)
1.05
1.09
1.05
1.05
1.10
3.08
1.11
1.10
2.90 2.85 2.80 2.75 2.70 2.65 2.60 2.55 2.50 2.45 2.40 2.35 2.30 2.25 2.20 2.15 2.10 2.05 2.00 1.95 1.90 1.85 1.80 1.75 1.70 1.65 1.60 1.55
Chemical Shift (ppm)


13C, CDCl3, 125 MHz
CHLOROFORM-d
177.02
170.19
144.91
138.83
133.12
132.50
130.86
129.18
127.36
126.62
125.70
124.94
118.66
117.11
77.00
71.11
60.92
40.96
38.80
27.26
27.15
23.17
22.01
21.49
27.26
27.15
27.3
27.2
27.1
Chemical Shift (ppm)
H
N
O
N
Ts
PivO
(+)-C27H30N2O5S (25)
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)


DEPT, CDCl3, 125 MHz
129.19
126.62
125.71
124.95
118.67
117.11
71.11
60.93
40.97
27.26
27.16
23.16
22.01
21.51
(+)-C27H30N2O5S (25)
Chemical Shift (ppm)

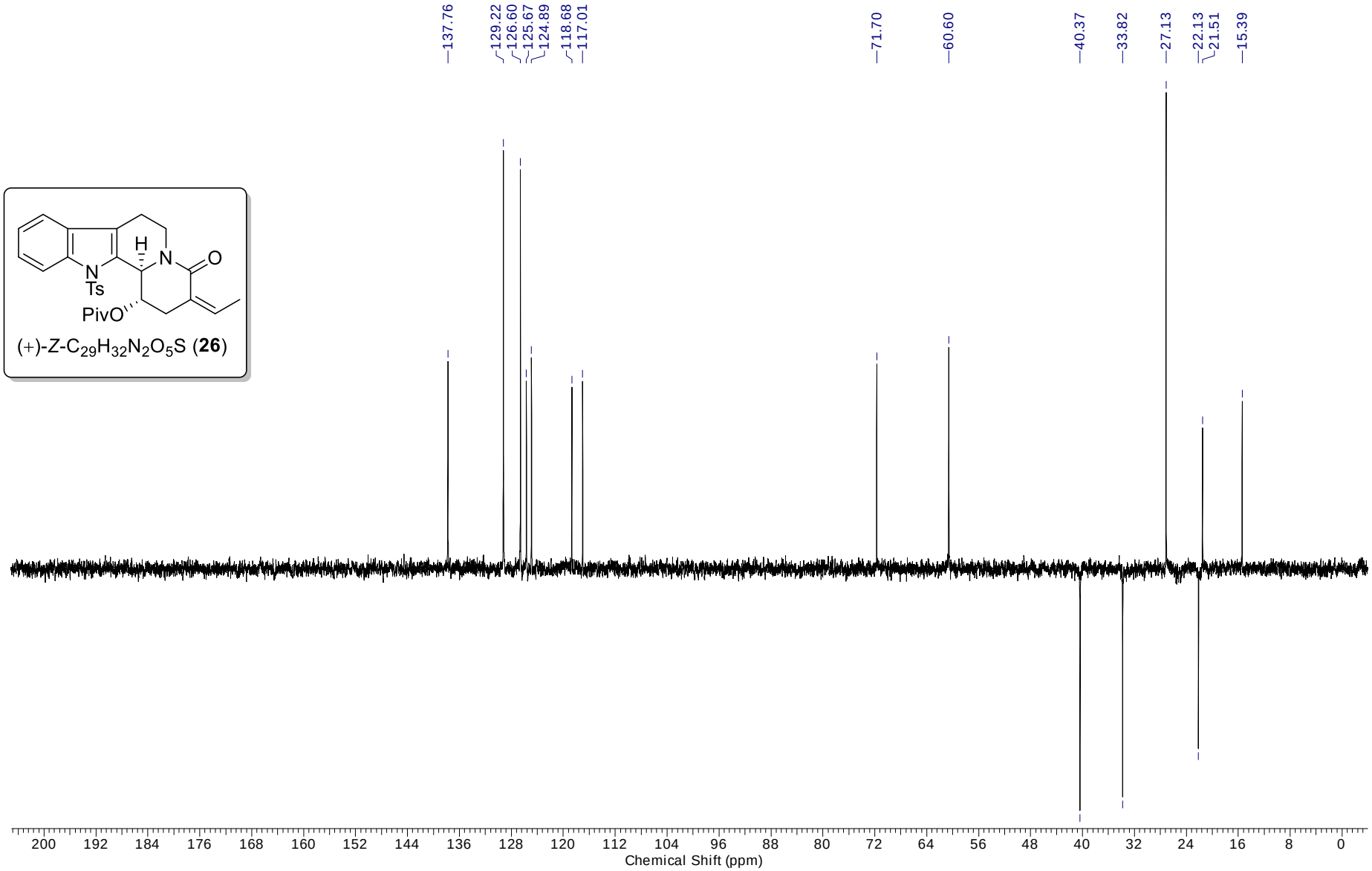

1H, CDCl3, 400 MHz
CHLOROFORM-d
(+)-Z-C29H32N2O5S (26)
Chemical Shift (ppm)
Chemical Shift (ppm)
Chemical Shift (ppm)

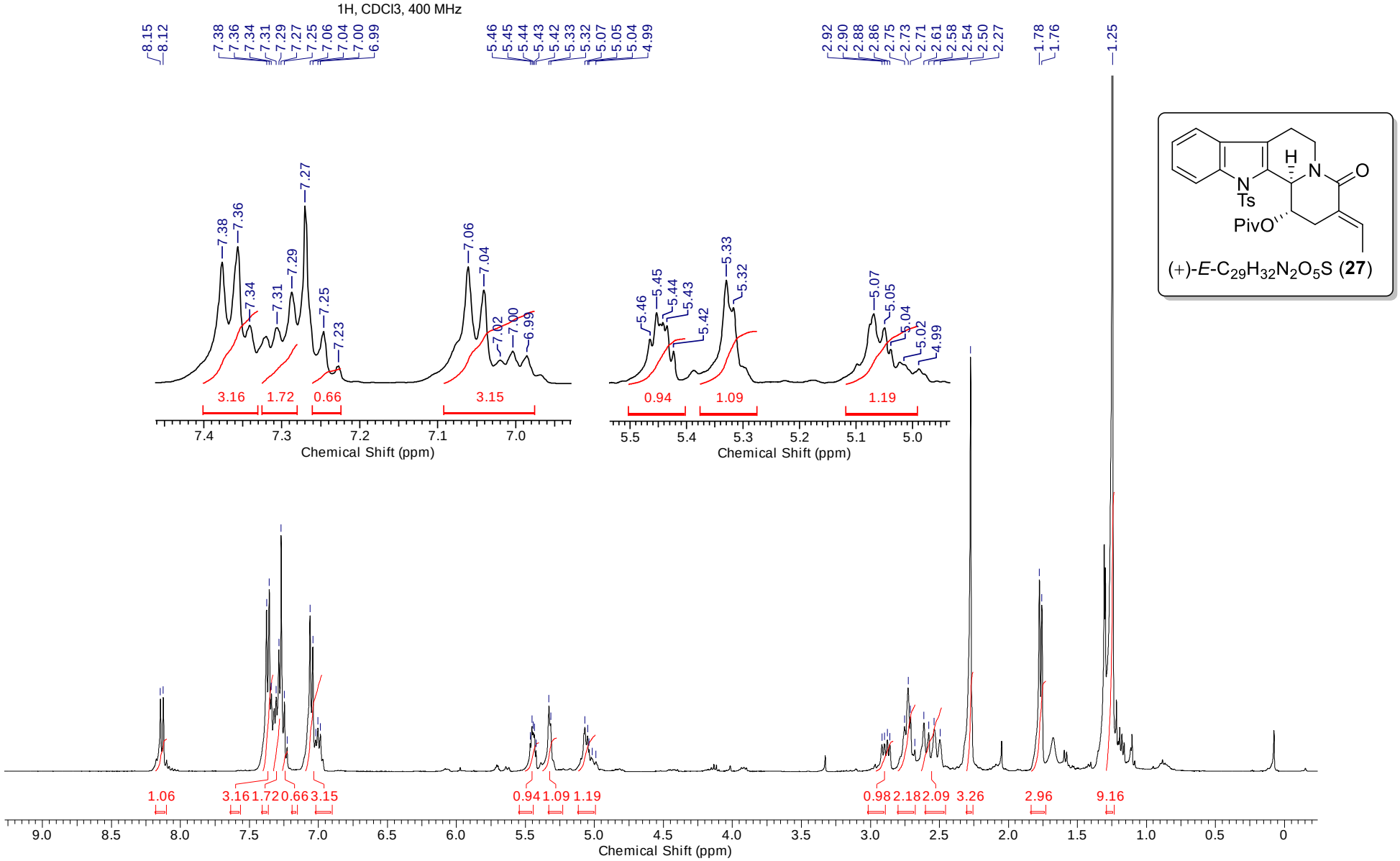

1H, CDCl3, 400 MHz
2.82
2.81
2.80
2.79
2.78
2.77
2.75
2.74
2.73
2.73
2.70
2.69
2.67
2.66
2.63
2.62
2.53
2.53
2.49
2.40
2.39
2.37
2.36
2.36
2.27
2.12
2.11
2.10
2.09
H
N
O
N
Ts
PivO
(+)-Z-C29H32N2O5S (26)
3.00
1.00
1.03
3.00
3.02
2.85
2.80
2.75
2.70
2.65
2.60
2.55
2.50
2.45
2.40
2.35
2.30
2.25
2.20
2.15
2.10
2.05
2.00
Chemical Shift (ppm)

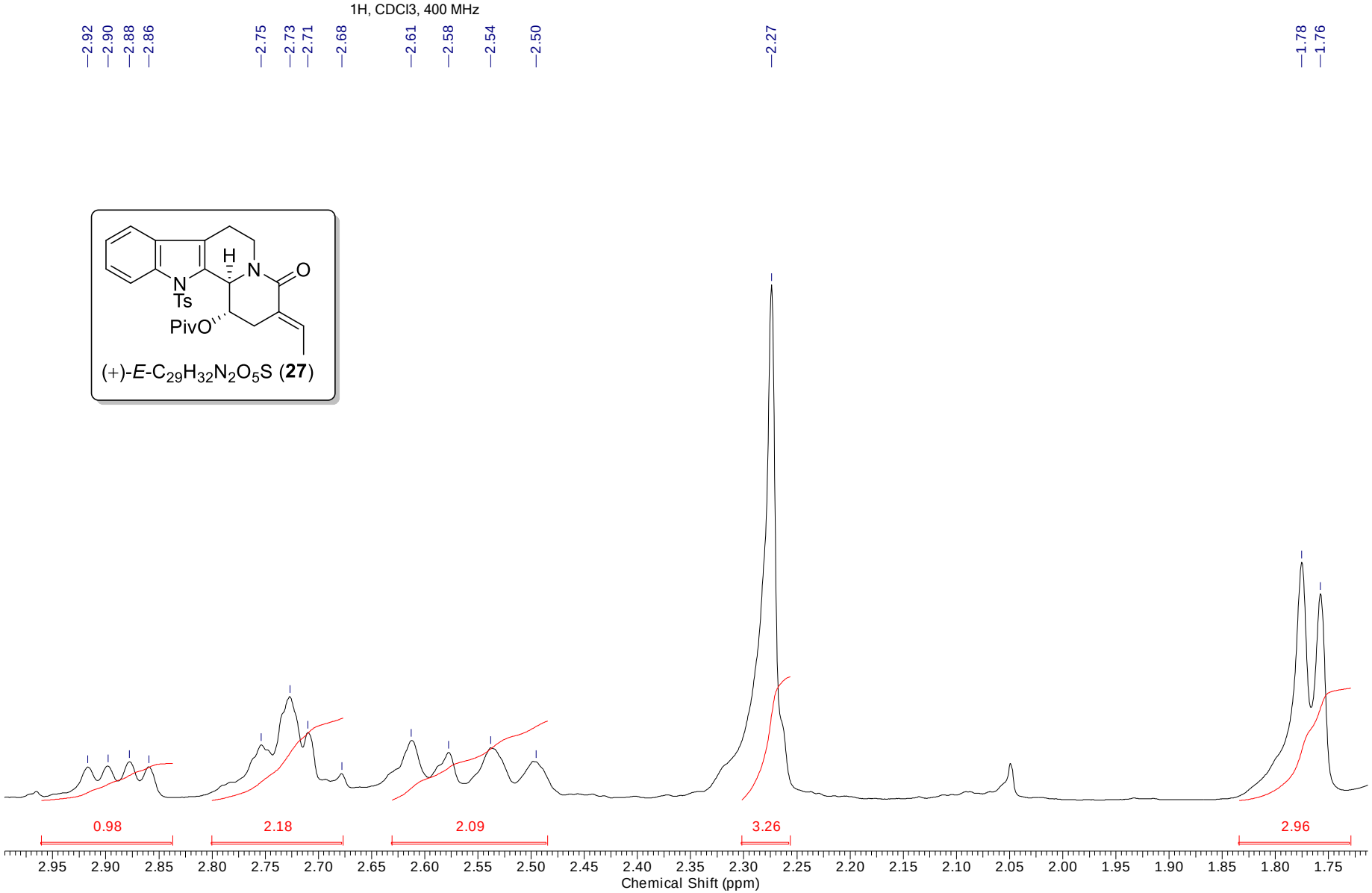

13C, CDCl3, 100 MHz
CHLOROFORM-d
177.09
165.54
144.89
138.75
137.76
132.89
132.58
130.78
129.21
127.34
126.59
125.65
125.54
124.88
118.67
117.00
77.00
71.70
60.59
40.37
38.70
33.82
27.13
22.12
21.50
15.39
Ts
PivO
(+)-Z-C29H32N2O5S (26)
Chemical Shift (ppm)
Chemical Shift (ppm)

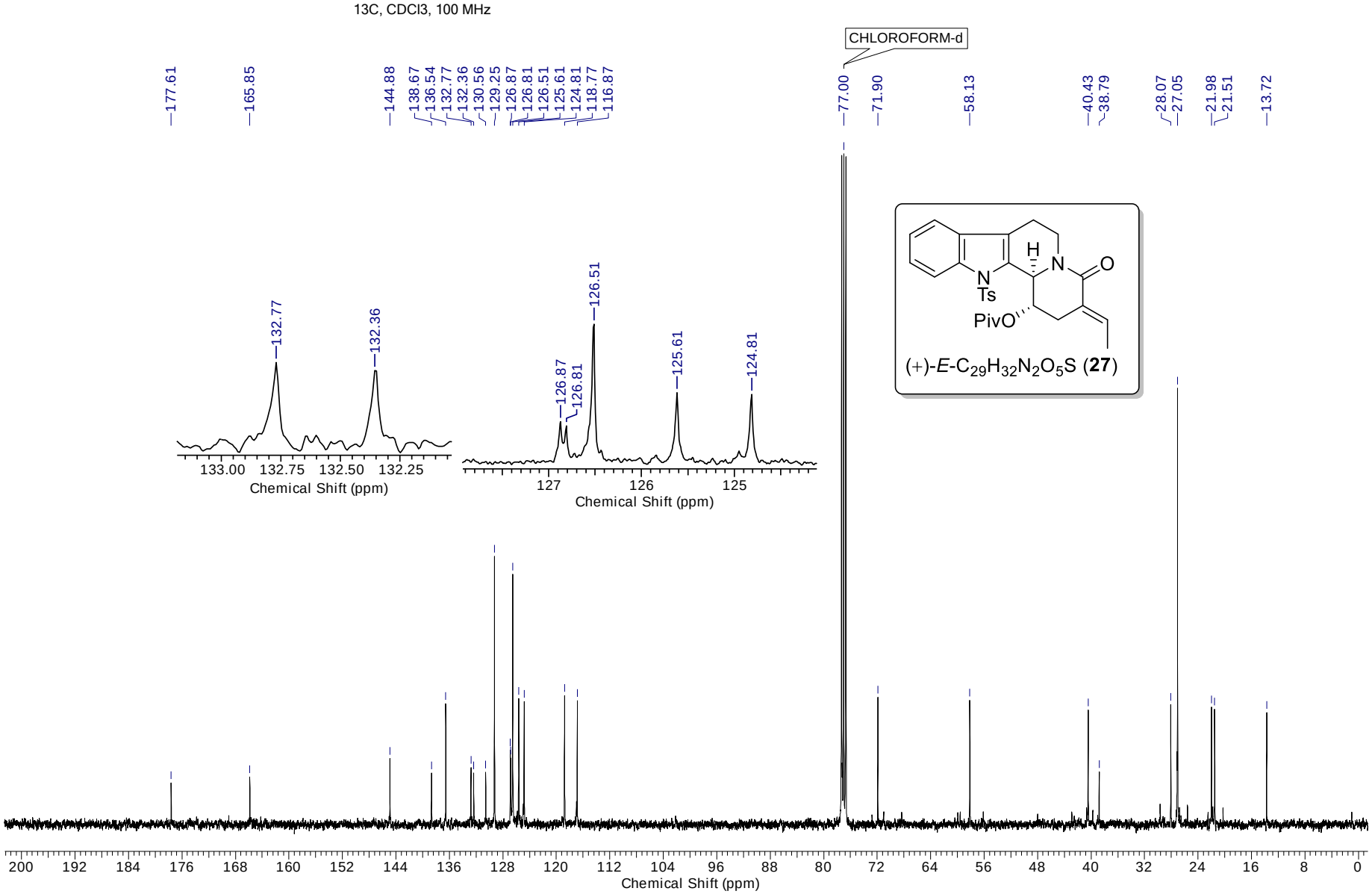
DEPT, CDCl3, 100 MHz

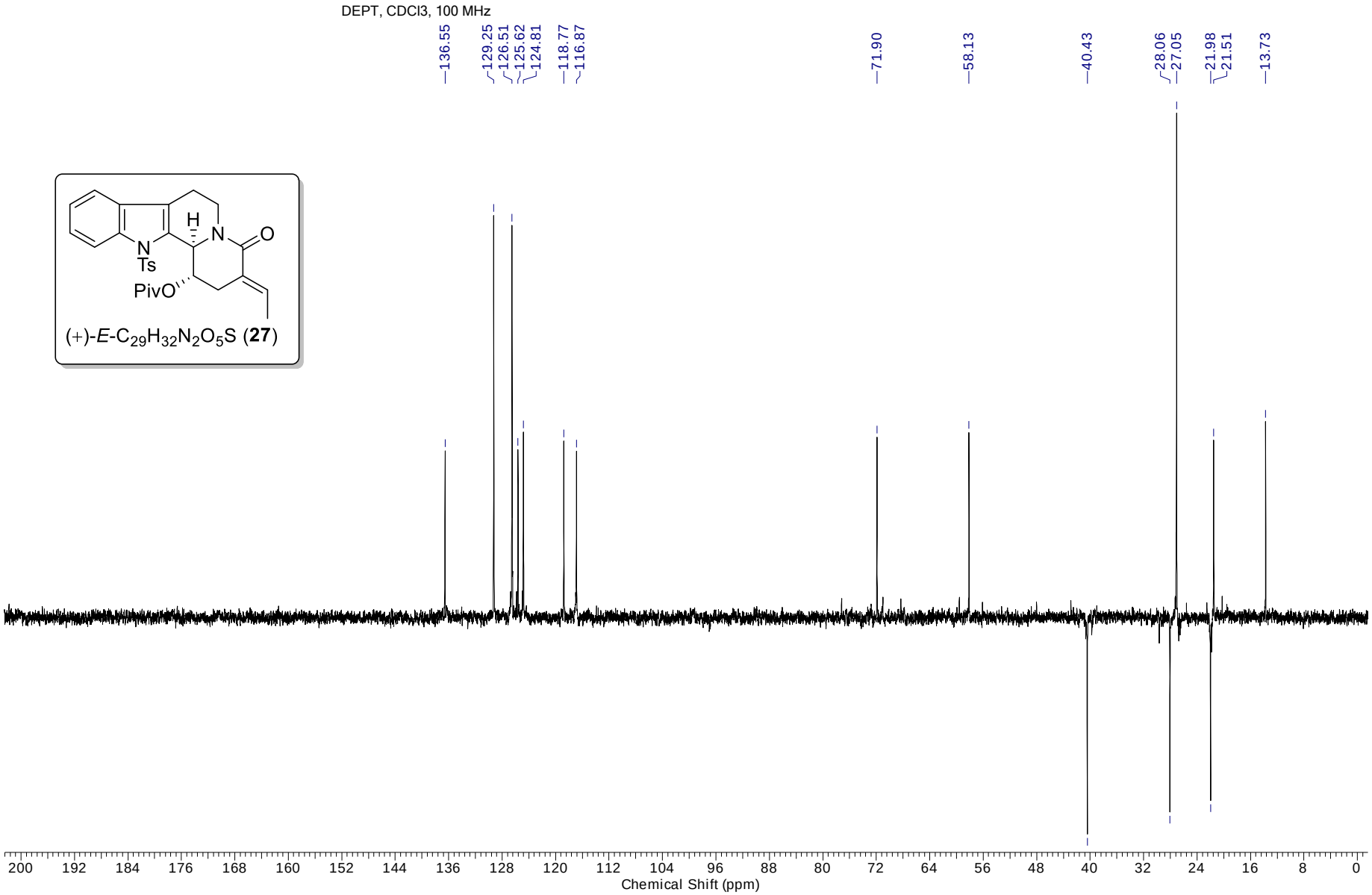
(+)-Z-$C_{29}H_{32}N_2O_5S$ (**26**)

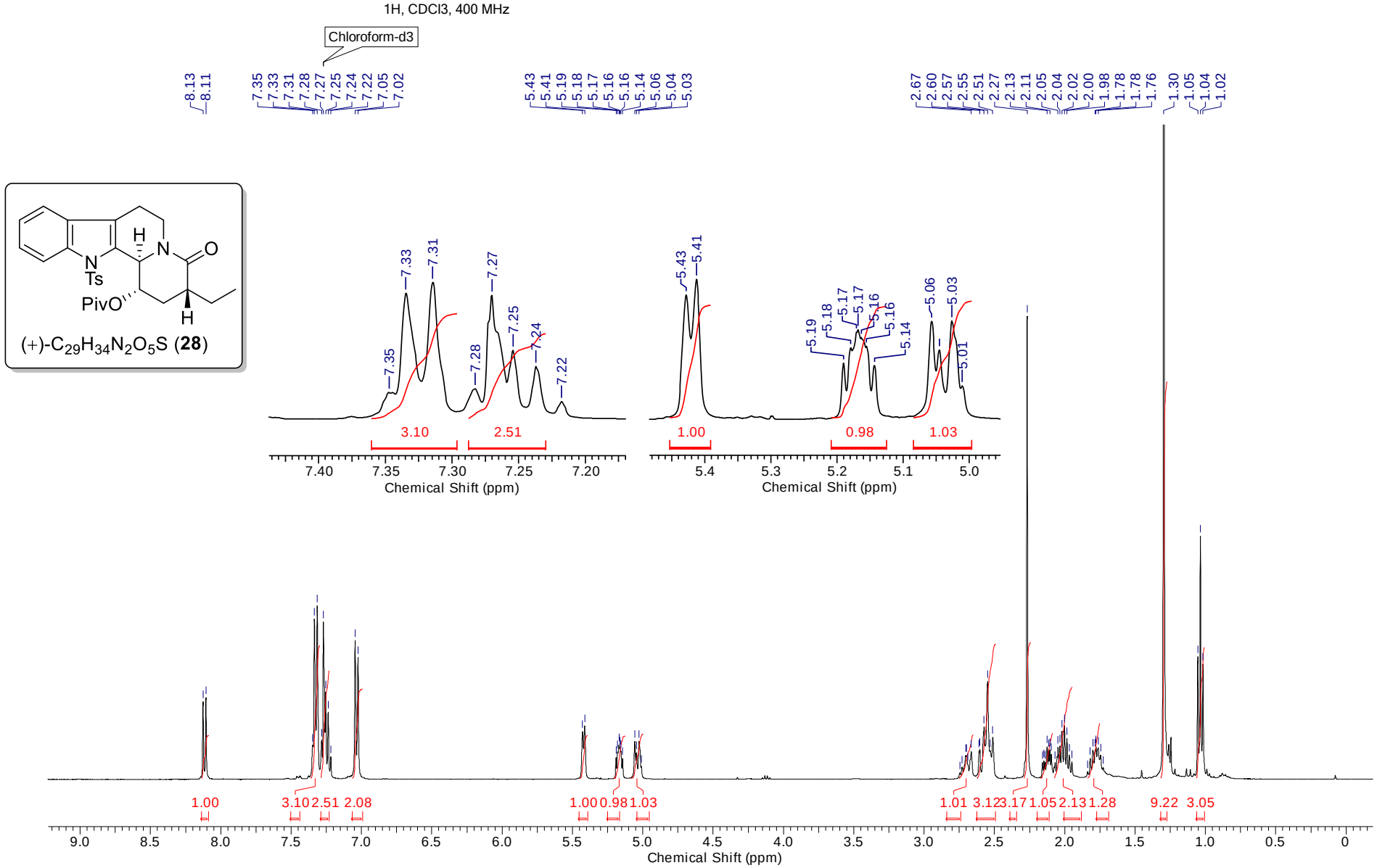


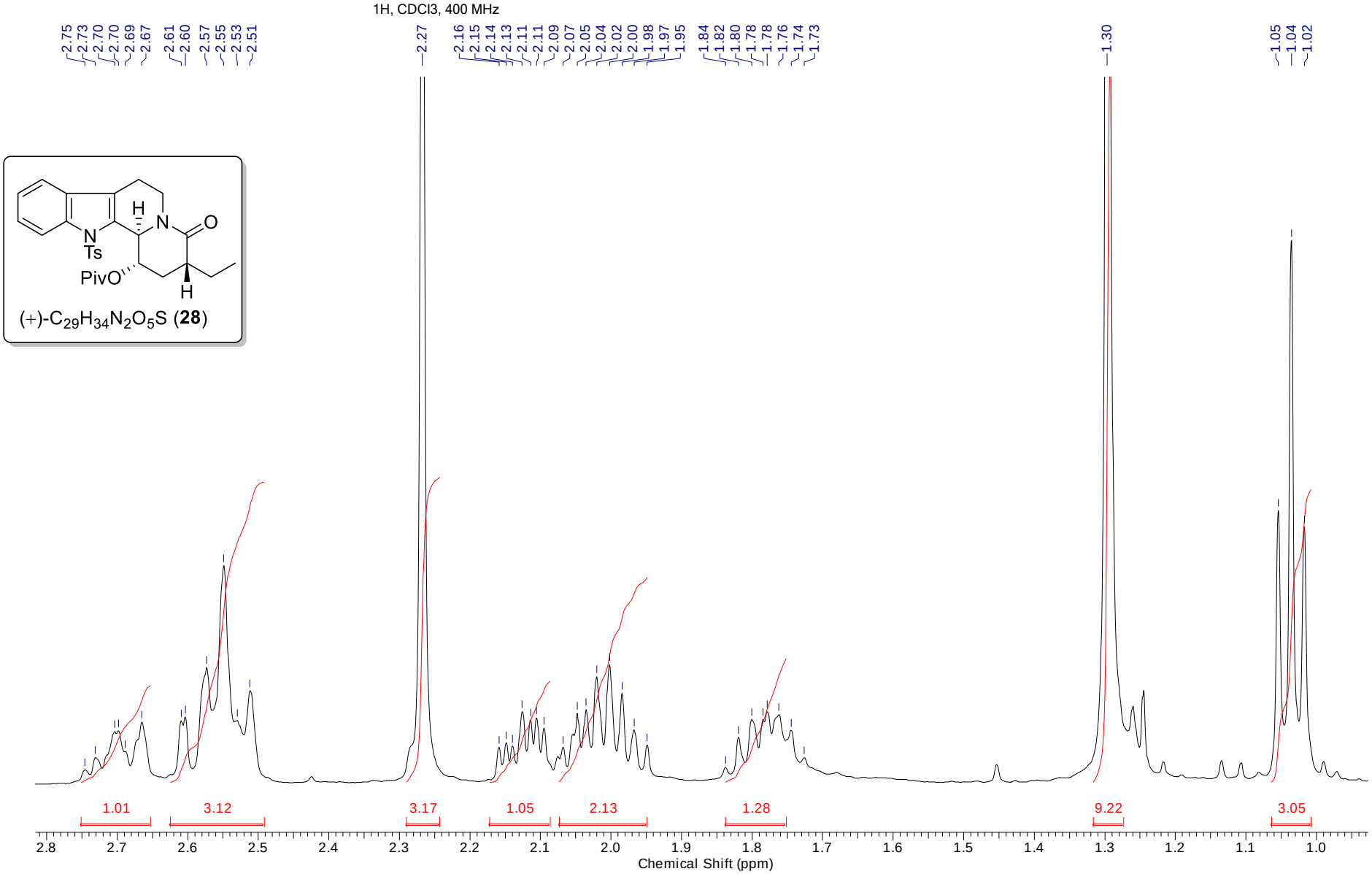

1H, CDCl3, 400 MHz
8.15
8.12
7.38
7.36
7.34
7.31
7.29
7.27
7.25
7.06
7.04
7.00
6.99
5.46
5.45
5.44
5.43
5.42
5.33
5.32
5.07
5.05
5.04
4.99
2.92
2.90
2.88
2.86
2.75
2.73
2.71
2.61
2.58
2.54
2.50
2.27
1.78
1.76
1.25
3.16
1.72
0.66
3.15
0.94
1.09
1.19
Chemical Shift (ppm)
1.06
3.16
1.72
0.66
3.15
0.94
1.09
1.19
0.98
2.18
2.09
3.26
2.96
9.16
Chemical Shift (ppm)
(+)-E-C29H32N2O5S (27)

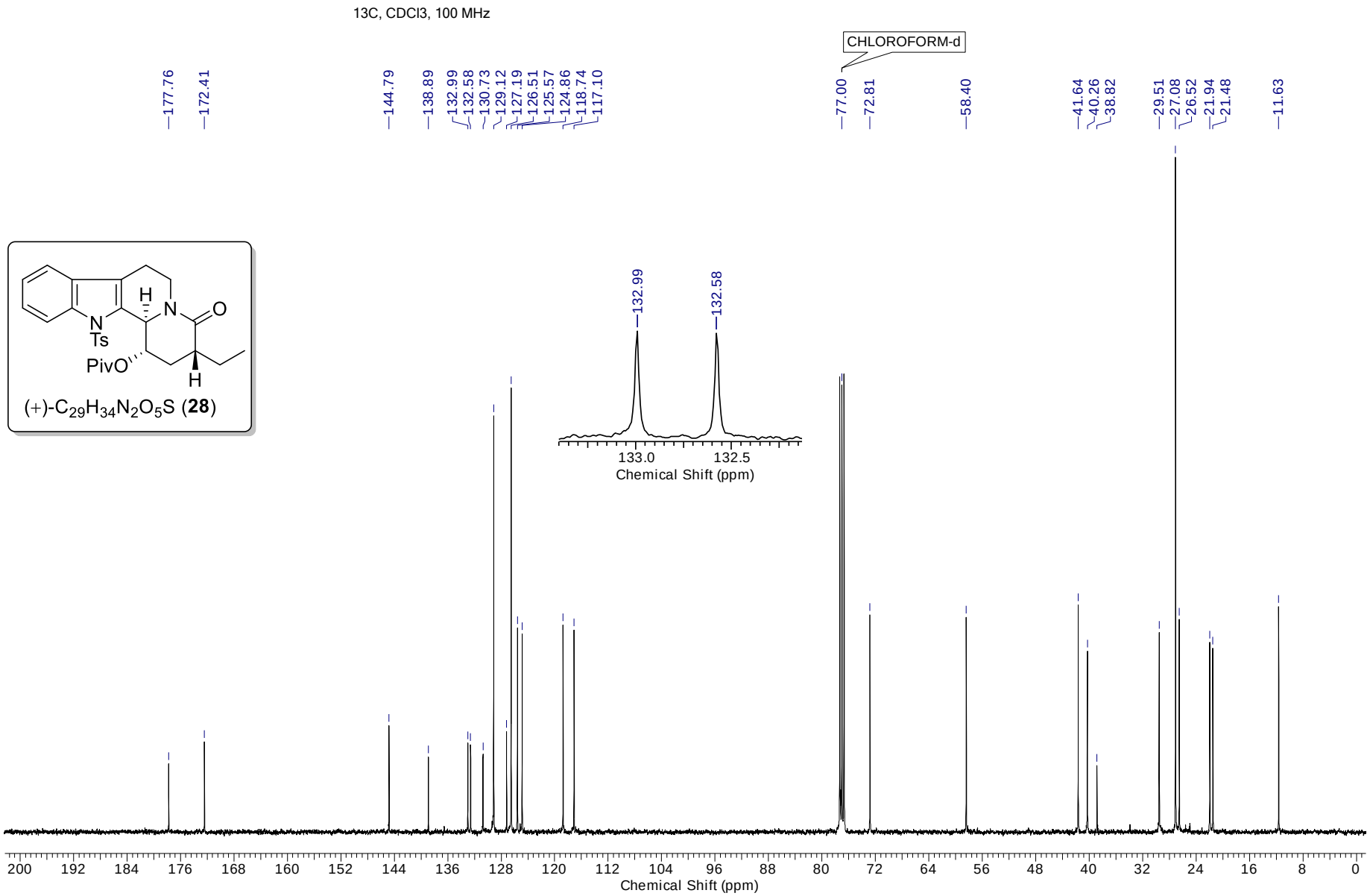

1H, CDCl3, 400 MHz
2.92
2.90
2.88
2.86
2.75
2.73
2.71
2.68
2.61
2.58
2.54
2.50
2.27
1.78
1.76
H
N
O
N
Ts
PivO
(+)-E-C29H32N2O5S (27)
0.98
2.18
2.09
3.26
2.96
2.95 2.90 2.85 2.80 2.75 2.70 2.65 2.60 2.55 2.50 2.45 2.40 2.35 2.30 2.25 2.20 2.15 2.10 2.05 2.00 1.95 1.90 1.85 1.80 1.75
Chemical Shift (ppm)


13C, CDCl3, 100 MHz
CHLOROFORM-d
177.61
165.85
144.88
138.67
136.54
132.77
132.36
130.56
129.25
126.87
126.81
126.51
125.61
124.81
118.77
116.87
77.00
71.90
58.13
40.43
38.79
28.07
27.05
21.98
21.51
13.72
132.77
132.36
133.00 132.75 132.50 132.25
Chemical Shift (ppm)
126.51
126.87
126.81
125.61
124.81
127 126 125
Chemical Shift (ppm)
H
N
O
N
Ts
PivO
(+)-E-C29H32N2O5S (27)
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

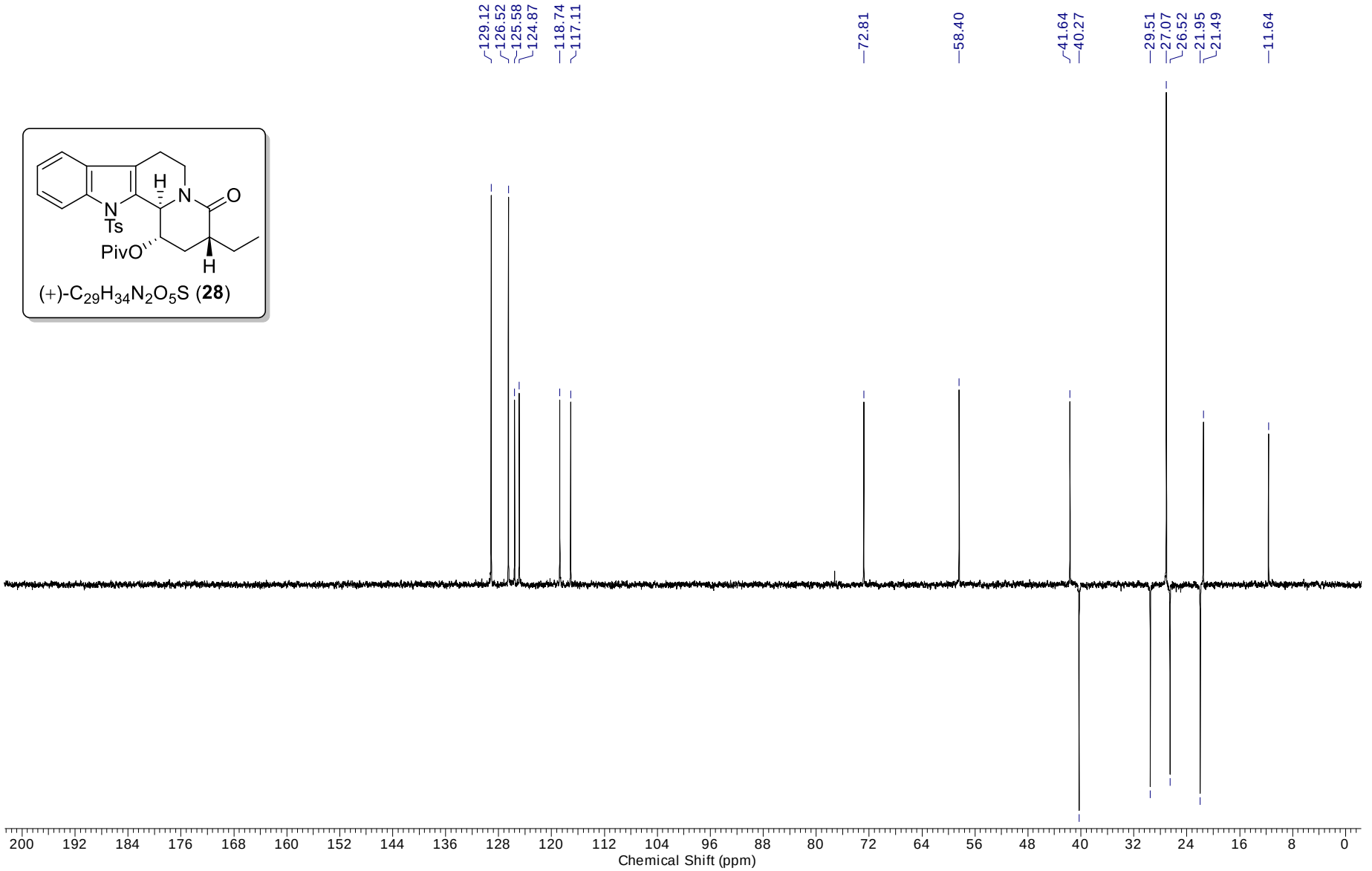

DEPT, CDCl3, 100 MHz
136.55
129.25
126.51
125.62
124.81
118.77
116.87
71.90
58.13
40.43
28.06
27.05
21.98
21.51
13.73
H
N
O
N
Ts
PivO
(+)-E-C29H32N2O5S (27)
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)


1H, CDCl3, 400 MHz
Chloroform-d3
(+)-C29H34N2O5S (28)
Chemical Shift (ppm)
Chemical Shift (ppm)
Chemical Shift (ppm)

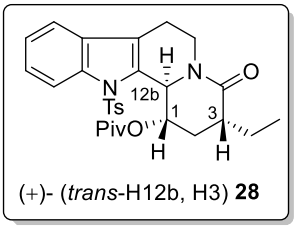

1H, CDCl3, 400 MHz
2.75
2.73
2.70
2.70
2.69
2.67
2.61
2.60
2.57
2.55
2.53
2.51
2.27
2.16
2.15
2.14
2.13
2.11
2.11
2.09
2.07
2.05
2.04
2.02
2.00
1.98
1.97
1.95
1.84
1.82
1.80
1.78
1.78
1.76
1.74
1.73
1.30
1.05
1.04
1.02
H
N
O
N
Ts
PivO
H
(+)-C29H34N2O5S (28)
1.01
3.12
3.17
1.05
2.13
1.28
9.22
3.05
2.8
2.7
2.6
2.5
2.4
2.3
2.2
2.1
2.0
1.9
1.8
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1.0
Chemical Shift (ppm)

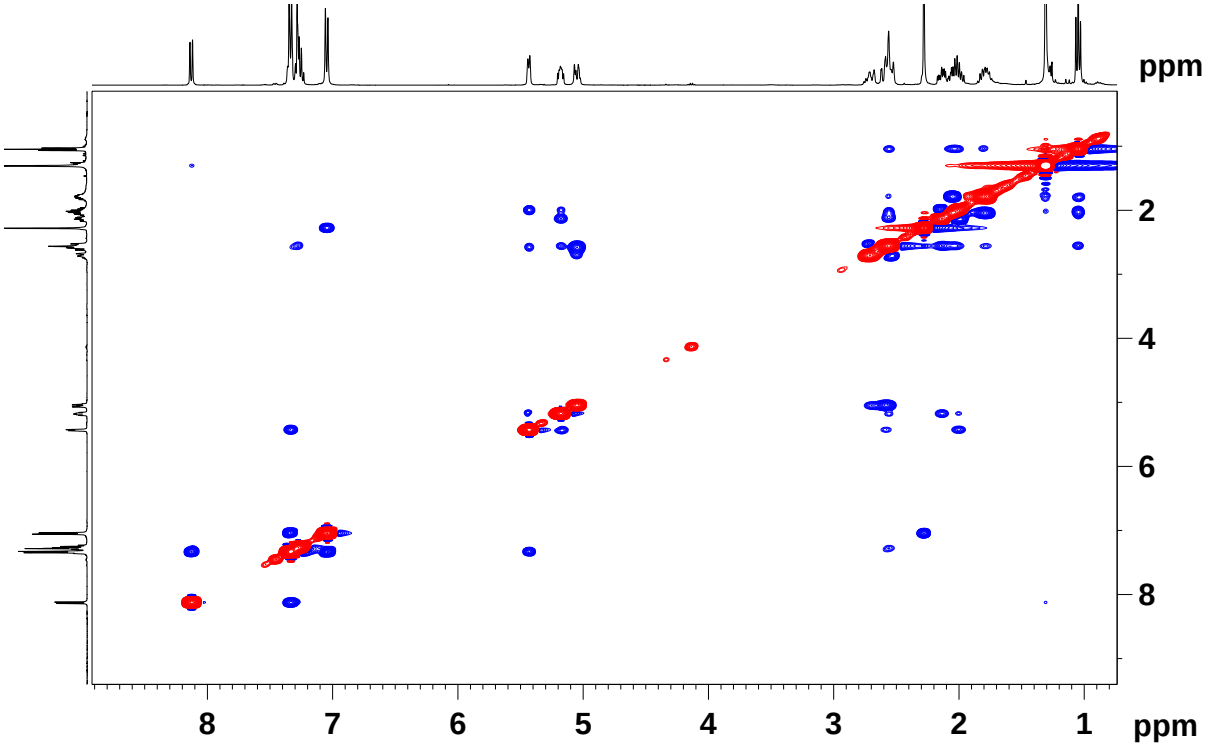

13C, CDCl3, 100 MHz
CHLOROFORM-d
177.76
172.41
144.79
138.89
132.99
132.58
130.73
129.12
127.19
126.51
125.57
124.86
118.74
117.10
77.00
72.81
58.40
41.64
40.26
38.82
29.51
27.08
26.52
21.94
21.48
11.63
(+)-C29H34N2O5S (28)
Chemical Shift (ppm)

DEPT, CDCl3, 100 MHz

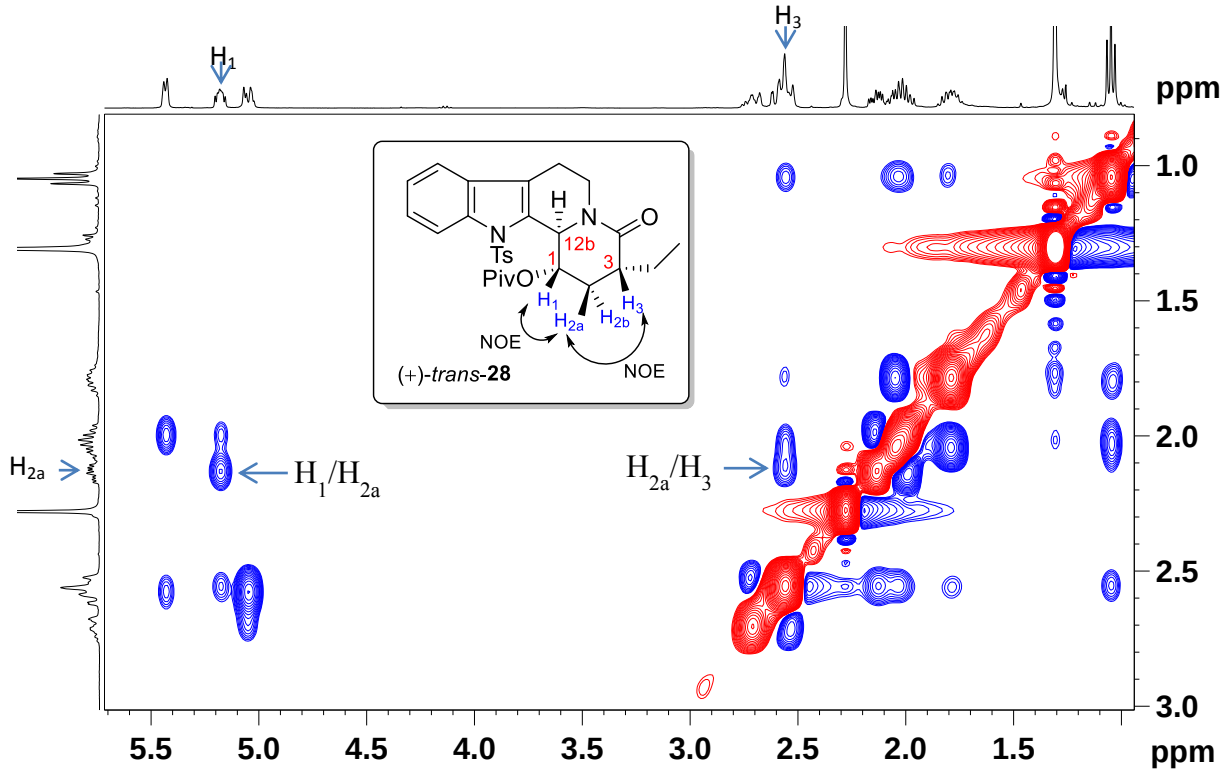


NOESY spectra of (+)-*trans*-**28**

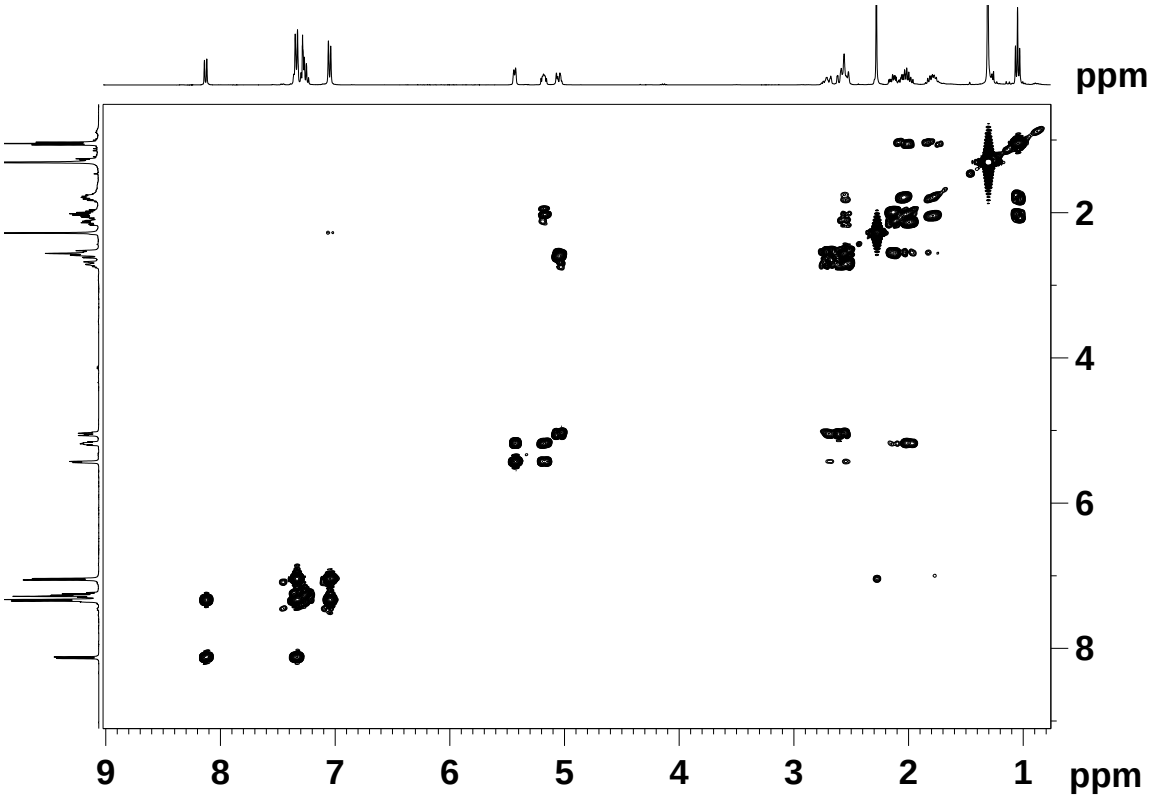




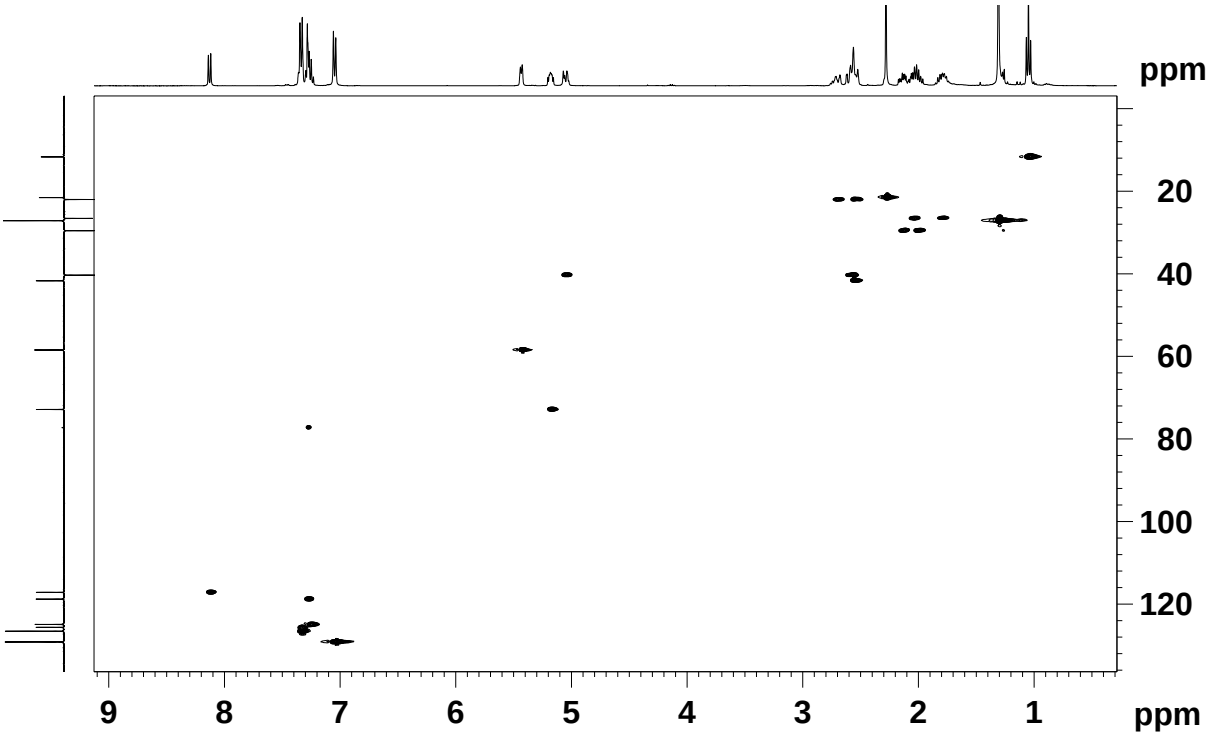
NOESY spectra of (+)-*trans*-**28**



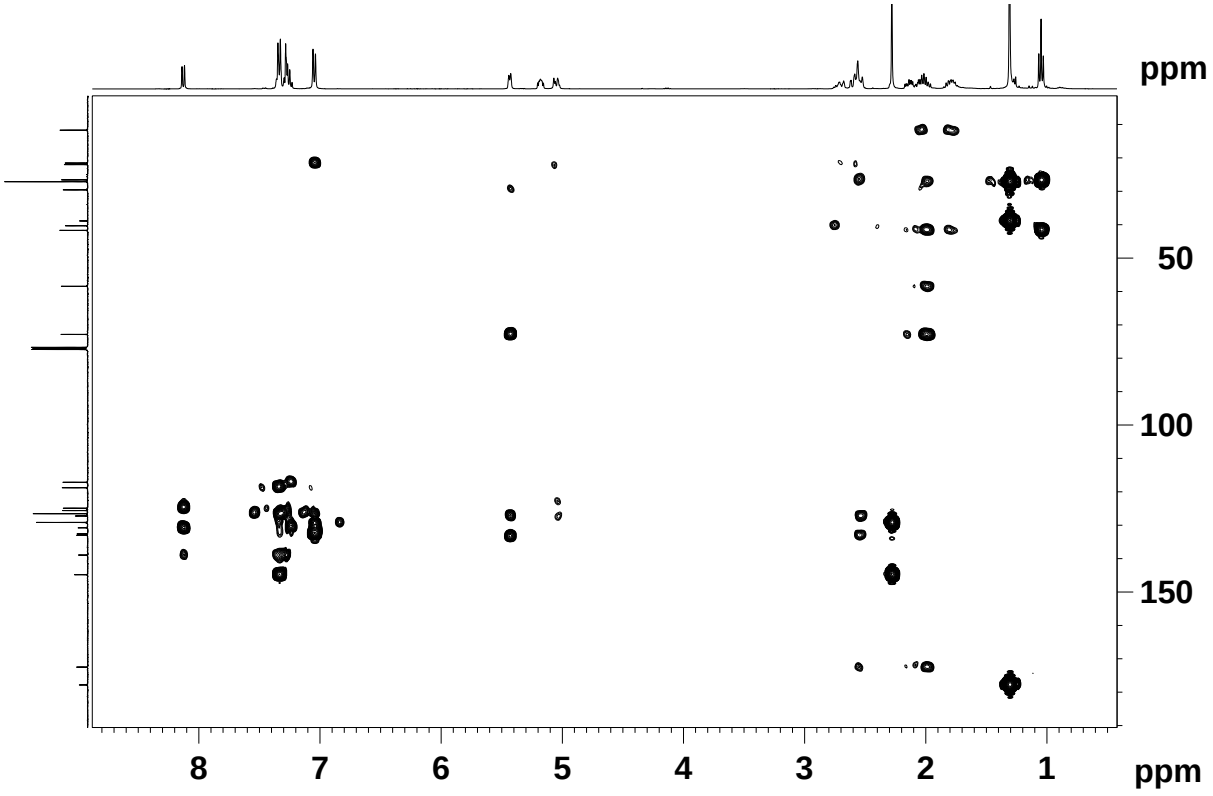
COSY spectra of (+)-*trans*-**28**

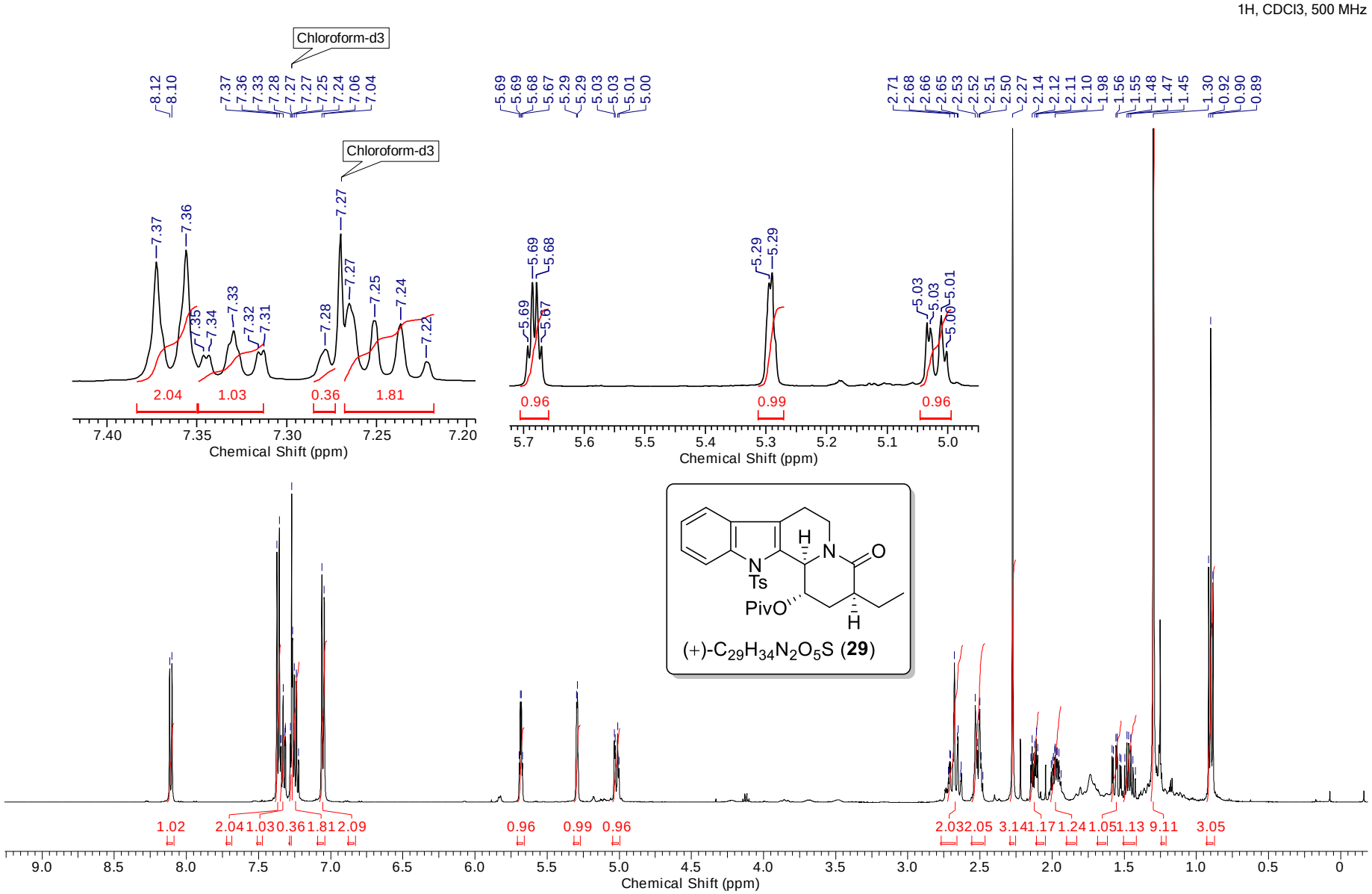


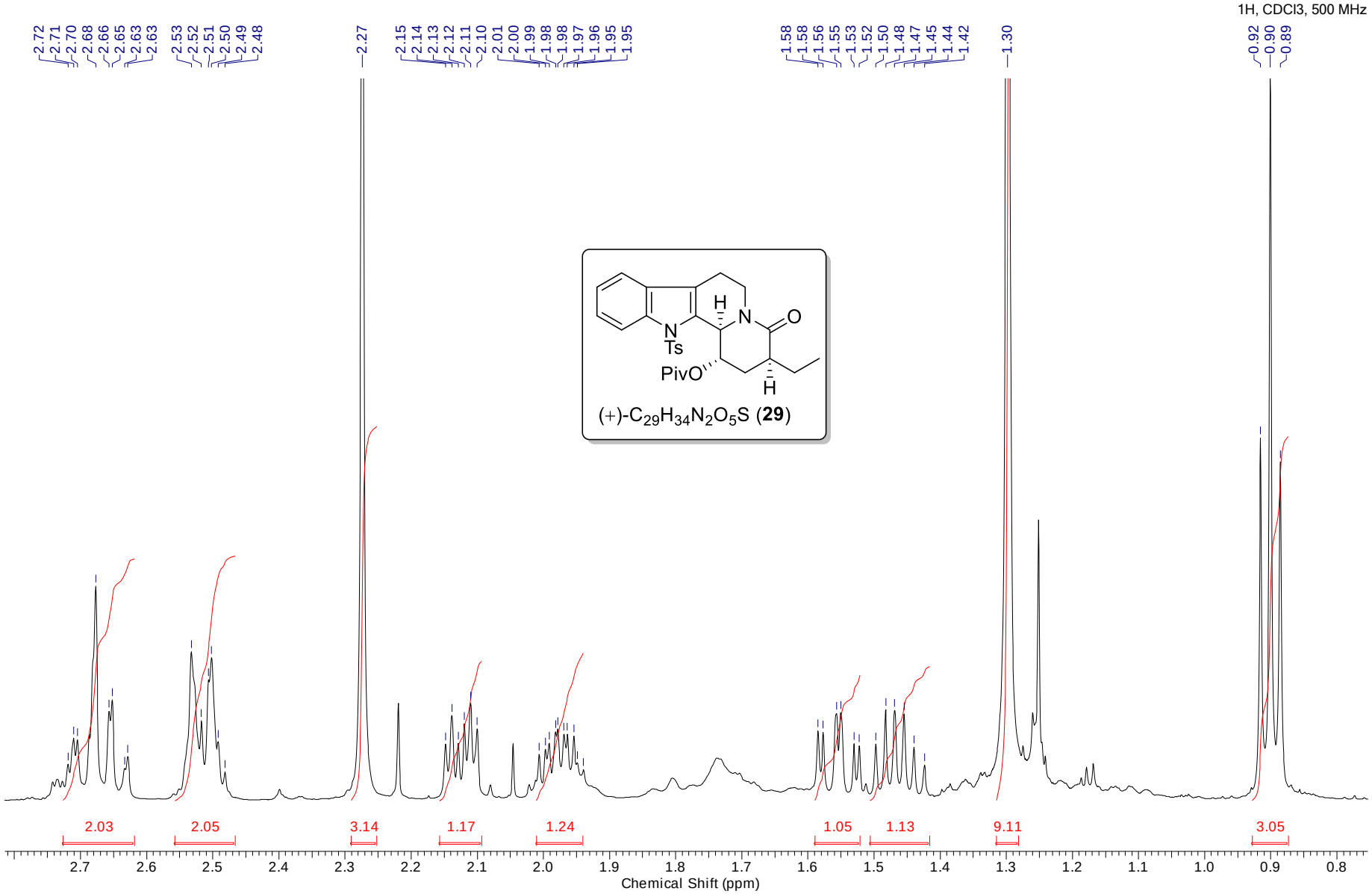
HSQC spectra of (+)-*trans*-**28**

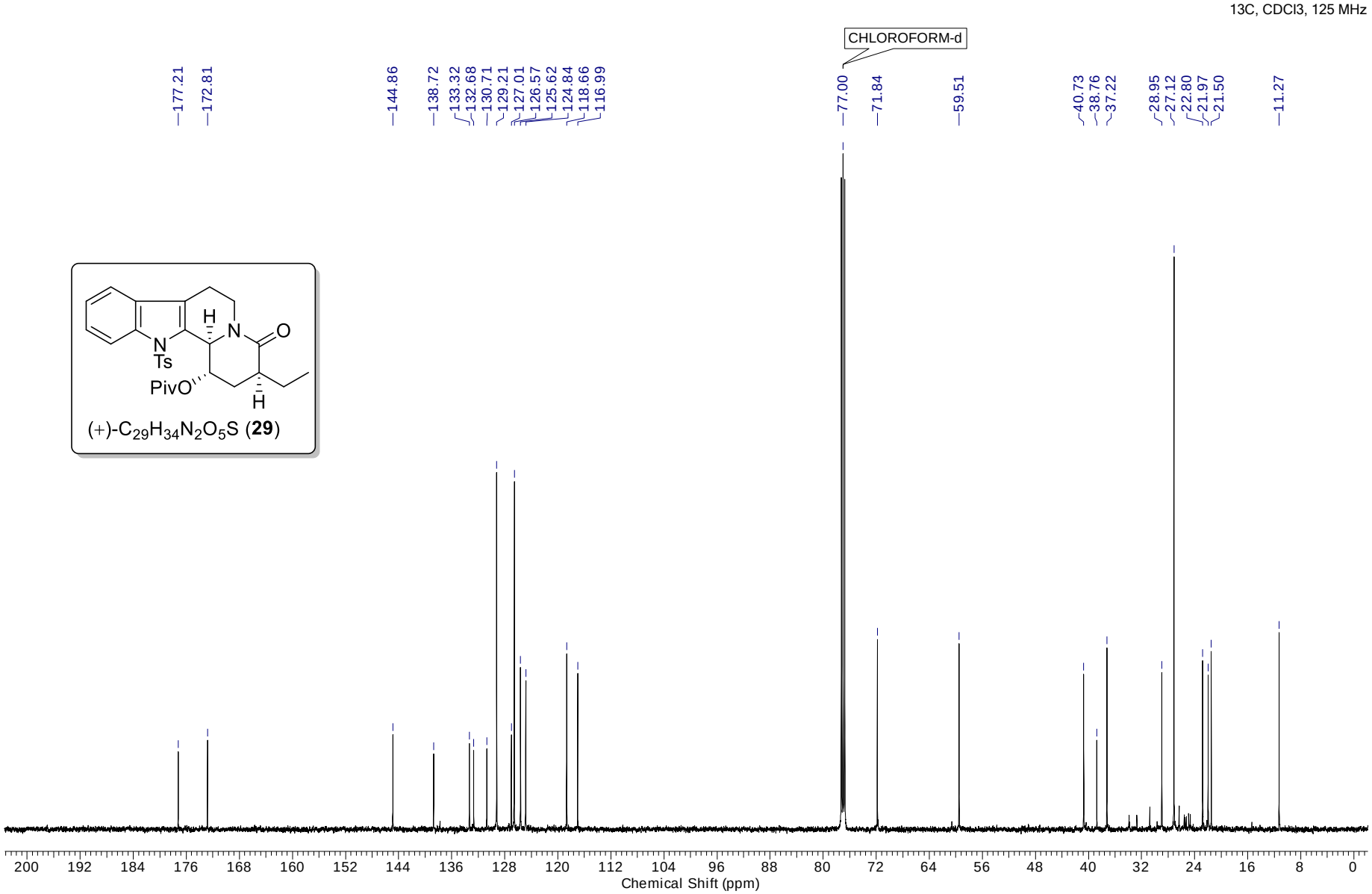


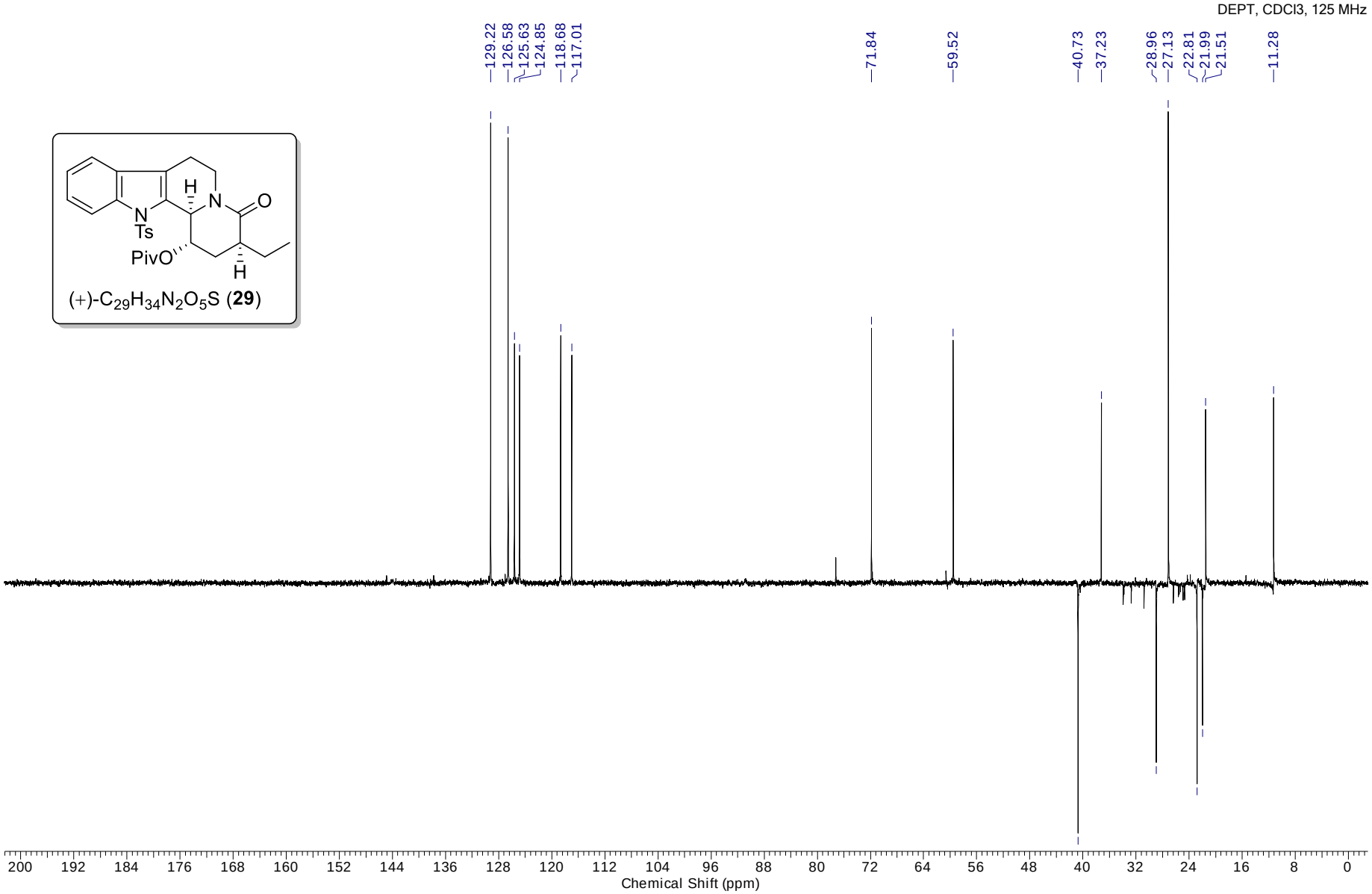
HMBC spectra of (+)-*trans*-**28**



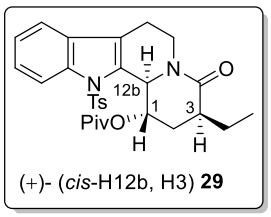

1H, CDCl3, 500 MHz
Chloroform-d3
Chloroform-d3
Chemical Shift (ppm)
Chemical Shift (ppm)
Chemical Shift (ppm)
Ts
PivO
(+)-C29H34N2O5S (29)

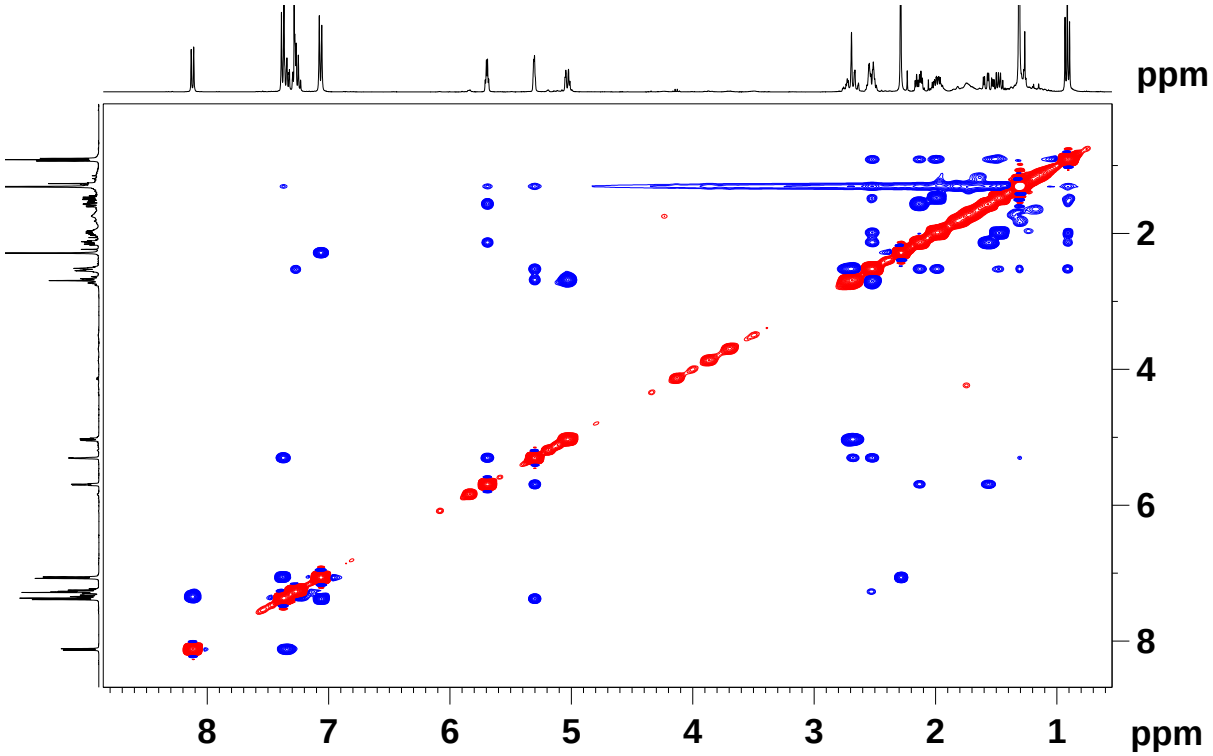

1H, CDCl3, 500 MHz
2.72 2.71 2.70 2.68 2.66 2.65 2.63 2.63 2.53 2.52 2.51 2.50 2.49 2.48
2.27
2.15 2.14 2.13 2.12 2.11 2.10 2.01 2.00 1.99 1.98 1.98 1.97 1.96 1.95 1.95
1.58 1.58 1.56 1.55 1.53 1.52 1.50 1.48 1.47 1.45 1.44 1.42
1.30
0.92 0.90 0.89
H
N
O
N
Ts
PivO
H
(+)-C29H34N2O5S (29)
2.03 2.05 3.14 1.17 1.24 1.05 1.13 9.11 3.05
2.7 2.6 2.5 2.4 2.3 2.2 2.1 2.0 1.9 1.8 1.7 1.6 1.5 1.4 1.3 1.2 1.1 1.0 0.9 0.8
Chemical Shift (ppm)


13C, CDCl3, 125 MHz
CHLOROFORM-d
177.21
172.81
144.86
138.72
133.32
132.68
130.71
129.21
127.01
126.57
125.62
124.84
118.66
116.99
77.00
71.84
59.51
40.73
38.76
37.22
28.95
27.12
22.80
21.97
21.50
11.27
(+)-C29H34N2O5S (29)
Chemical Shift (ppm)

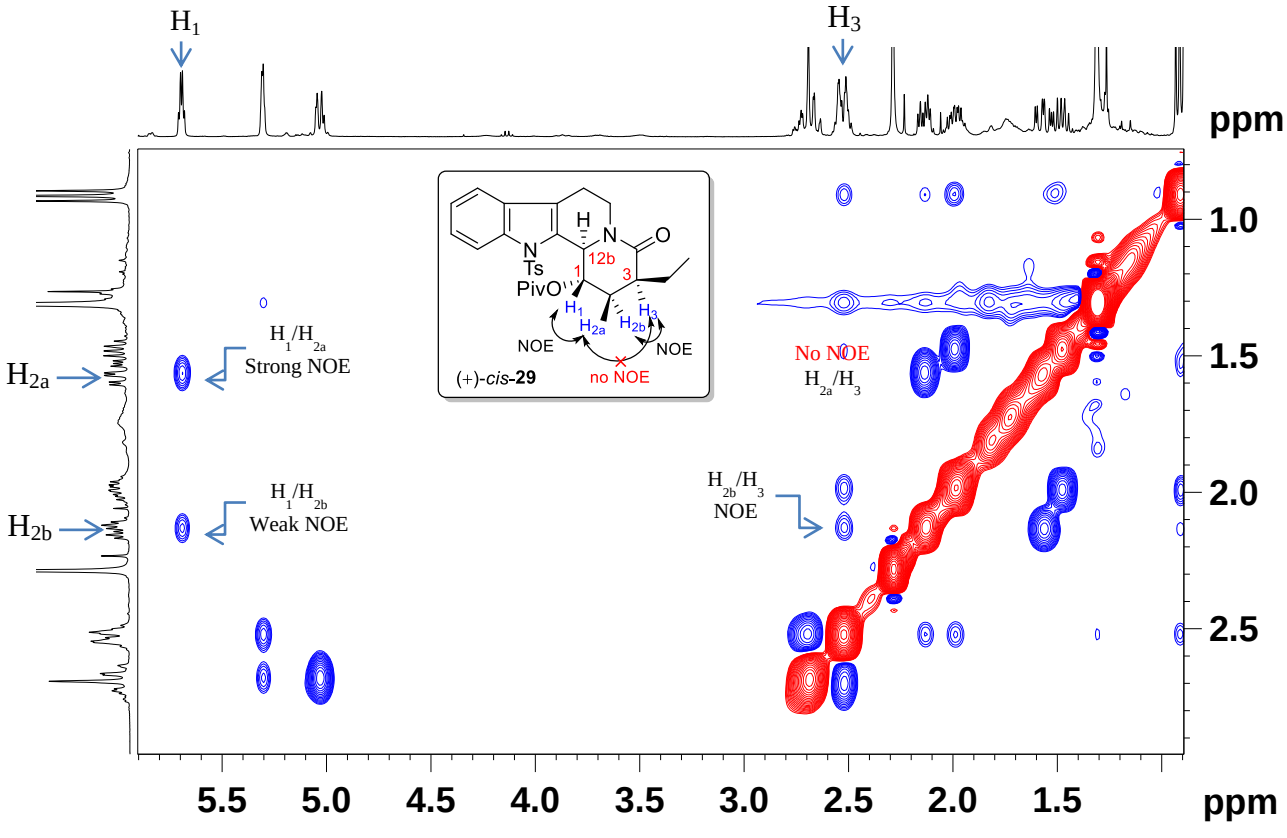

DEPT, CDCl3, 125 MHz
129.22
126.58
125.63
124.85
118.68
117.01
71.84
59.52
40.73
37.23
28.96
27.13
22.81
21.99
21.51
11.28
(+)-C29H34N2O5S (29)
Chemical Shift (ppm)

NOSEY spectra of (+)-*cis*-**29**

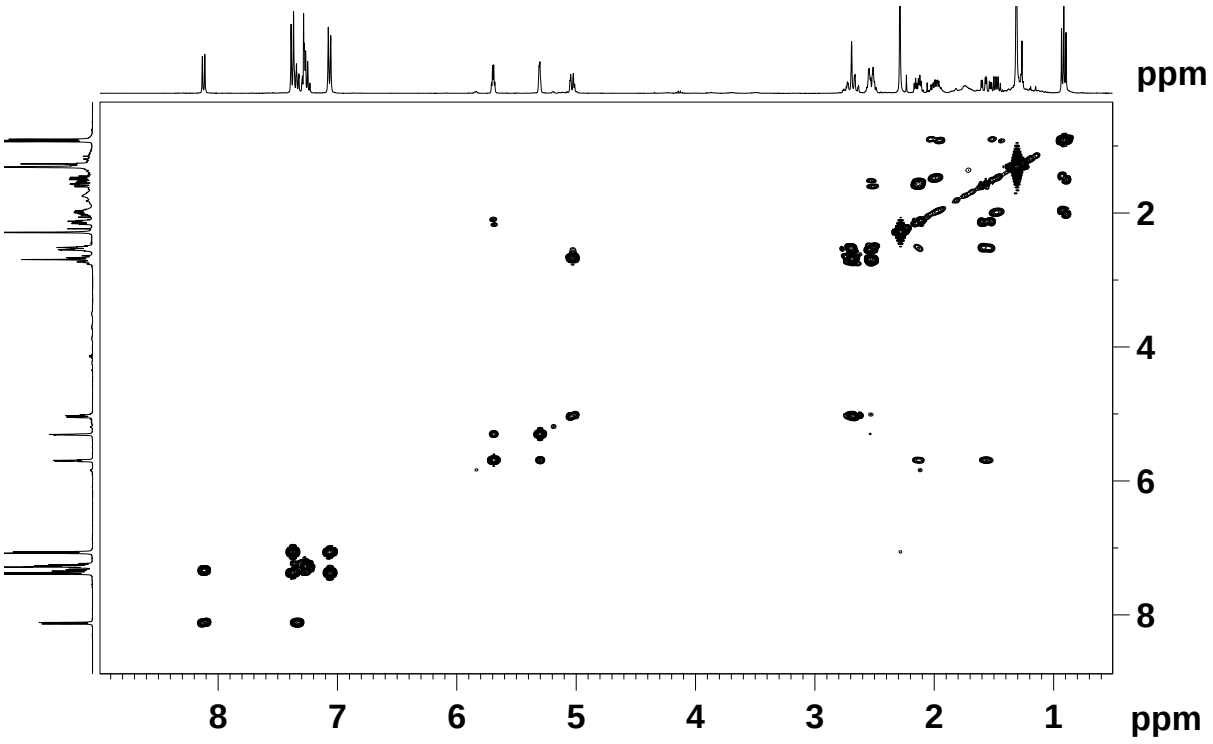




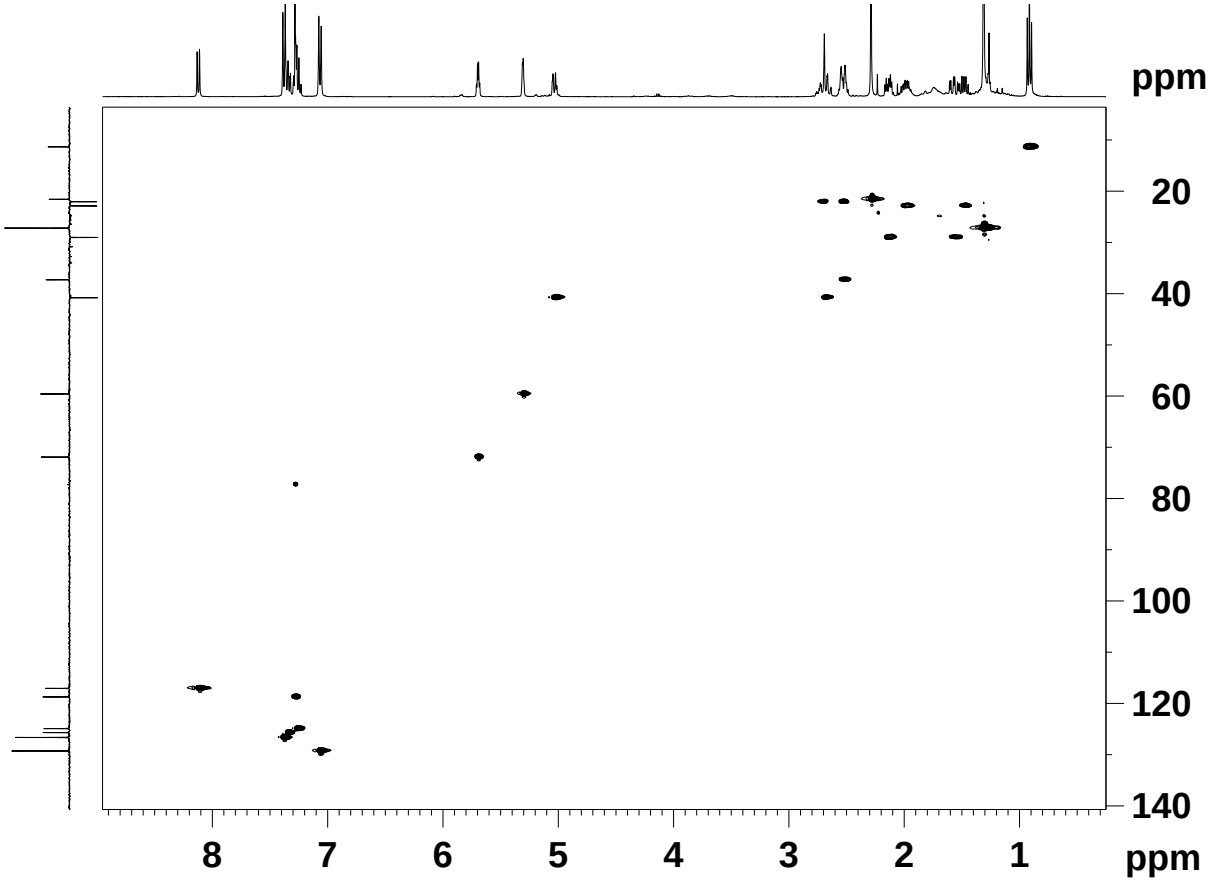
NOSEY spectra of (+)-*cis*-**29**



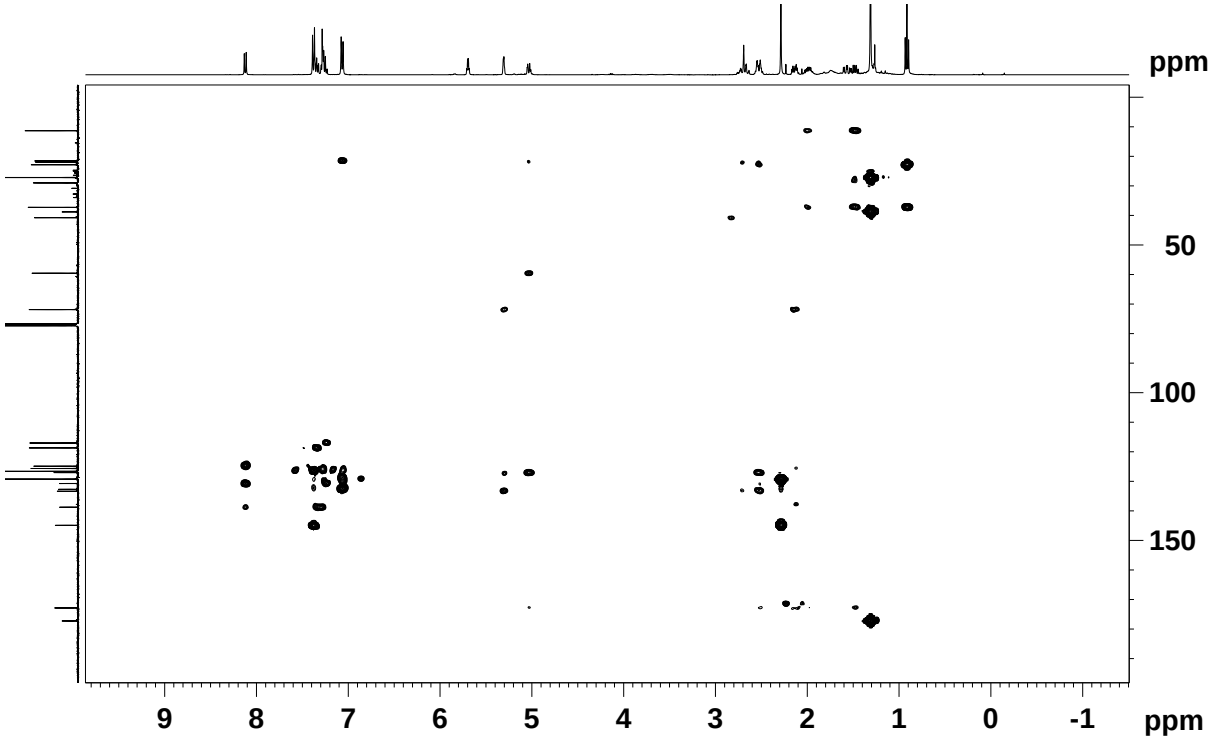
COSY spectra of (+)-*cis*-**29**

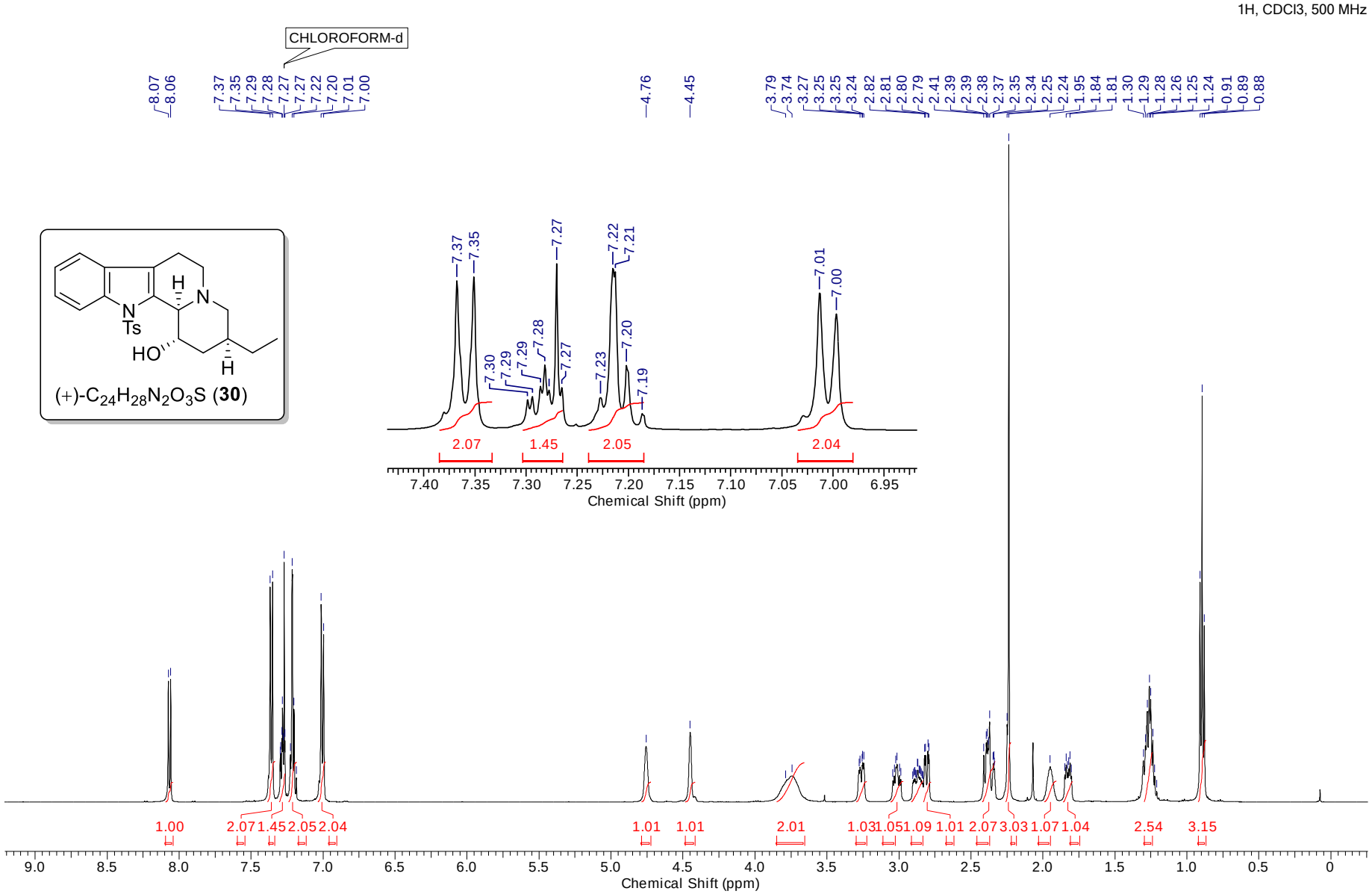


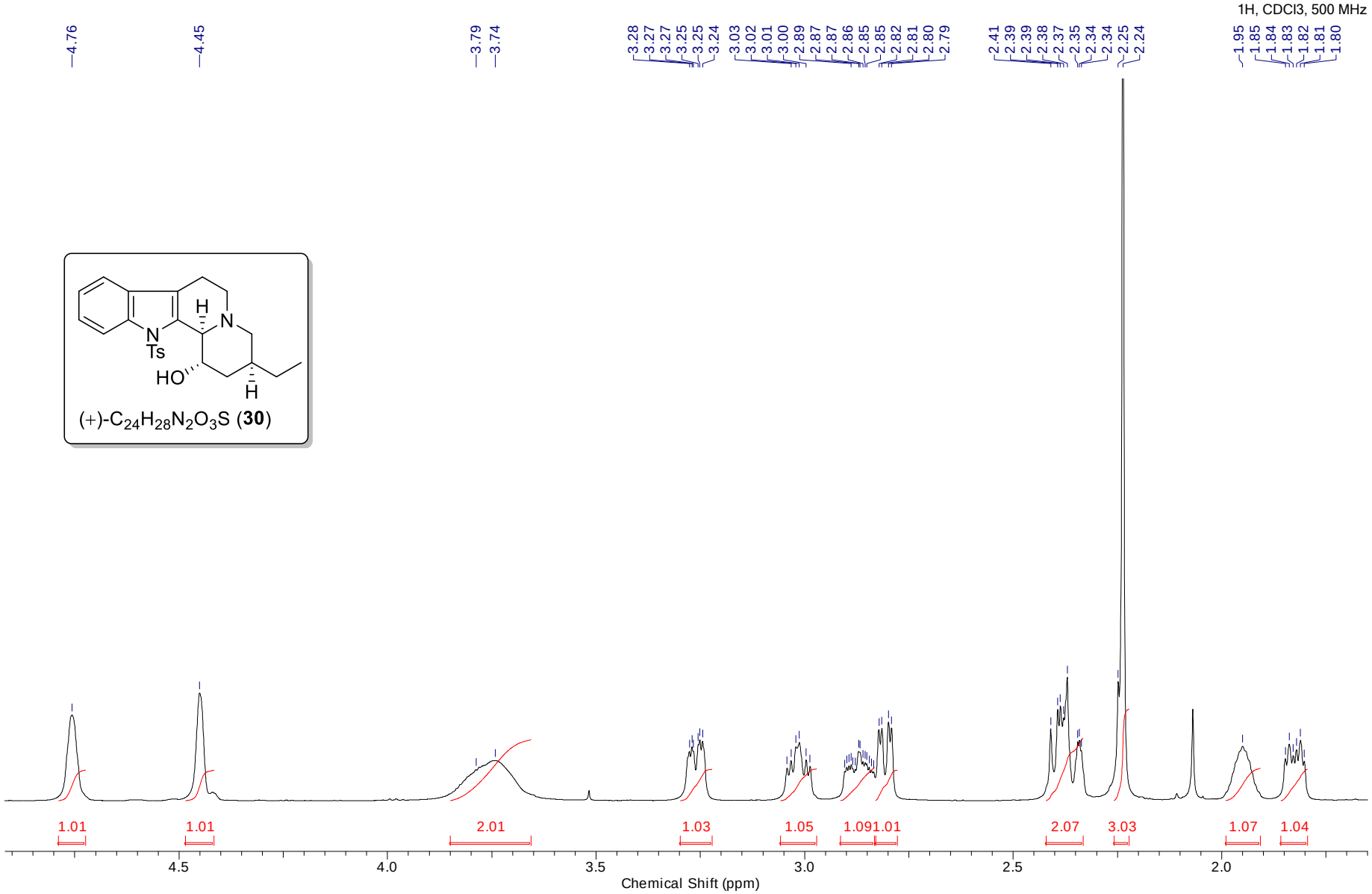
HSQC spectra of (+)-*cis*-**29**

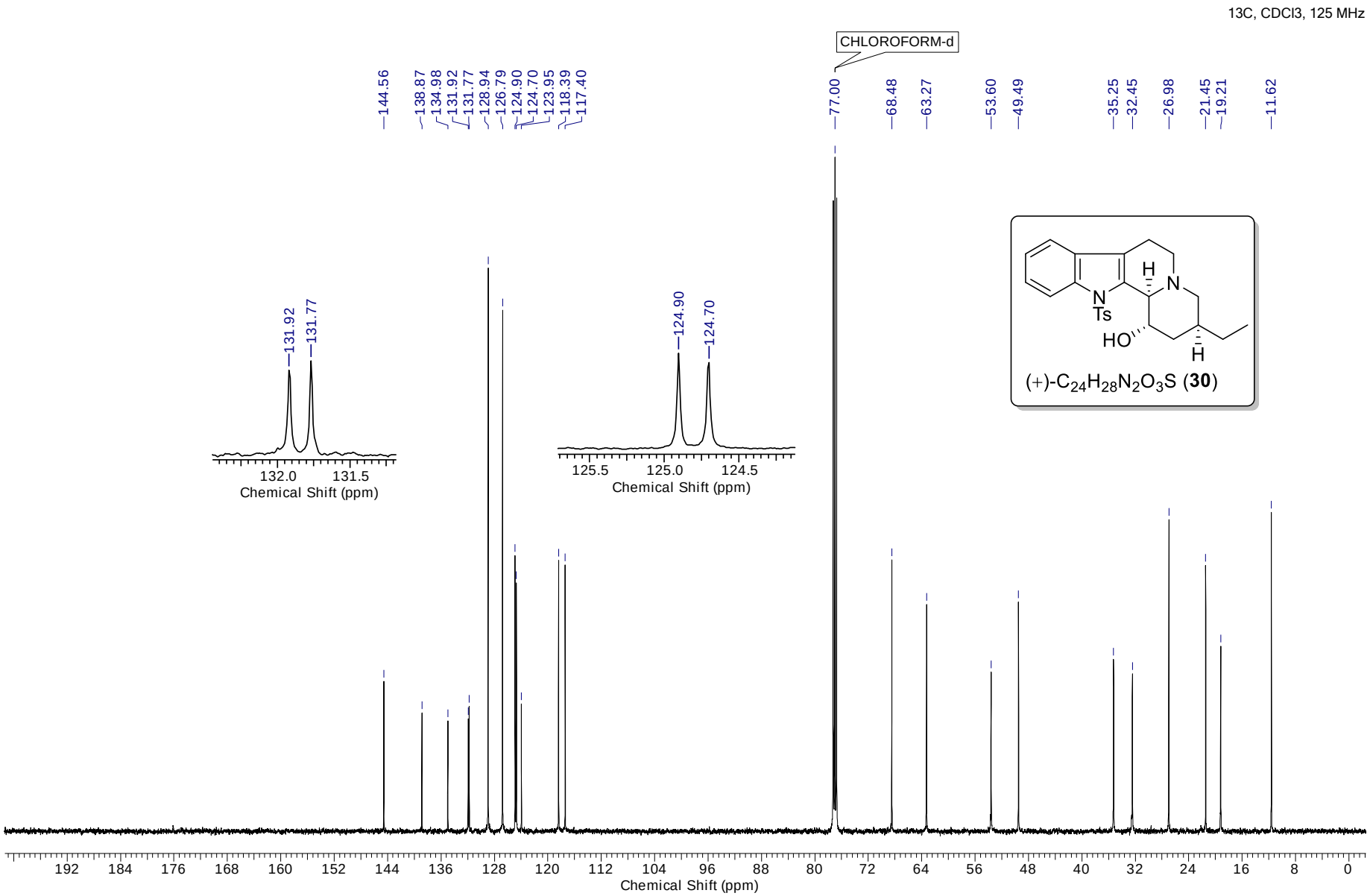


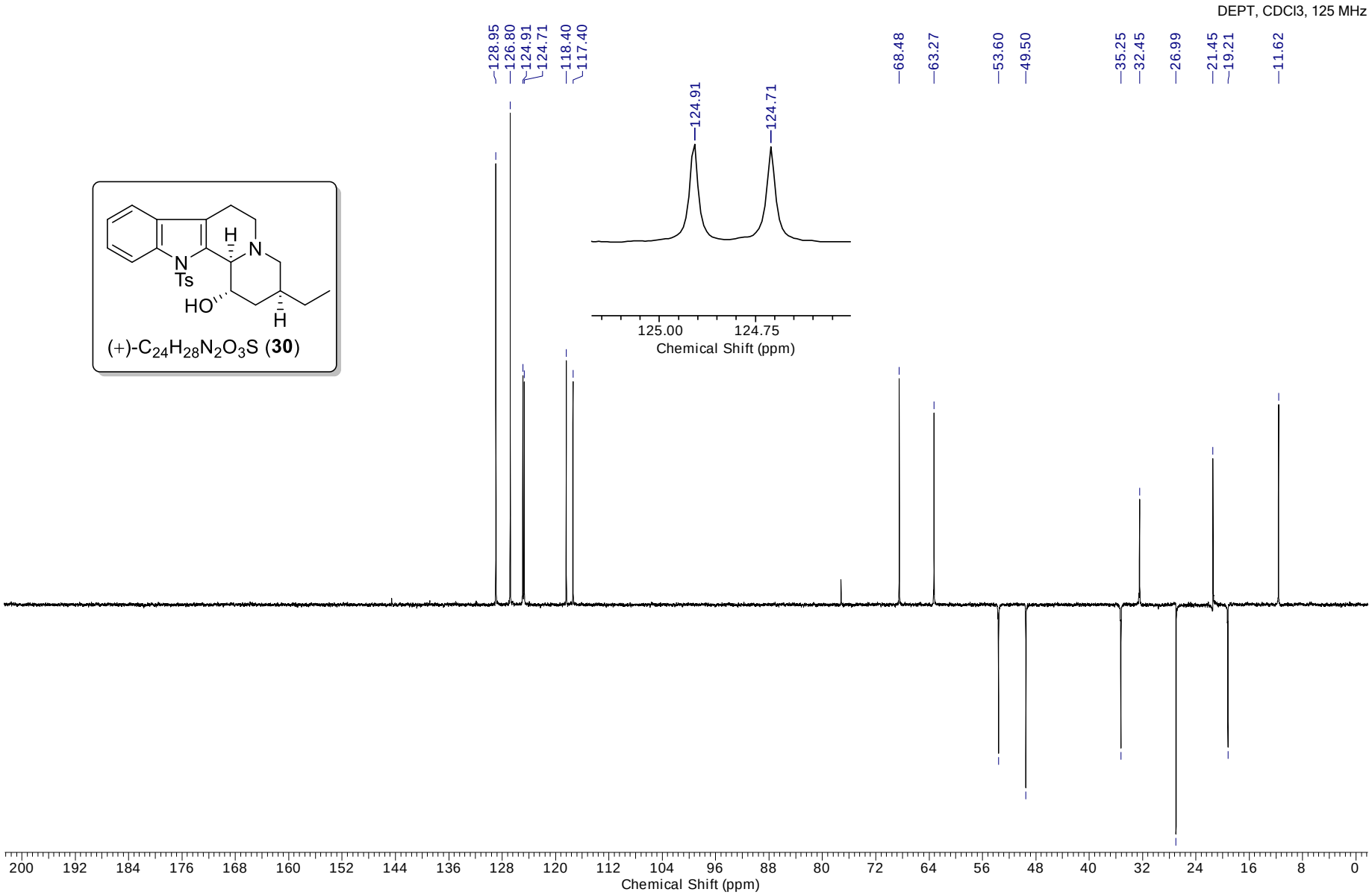
HMBC spectra of (+)-*cis*-**29**

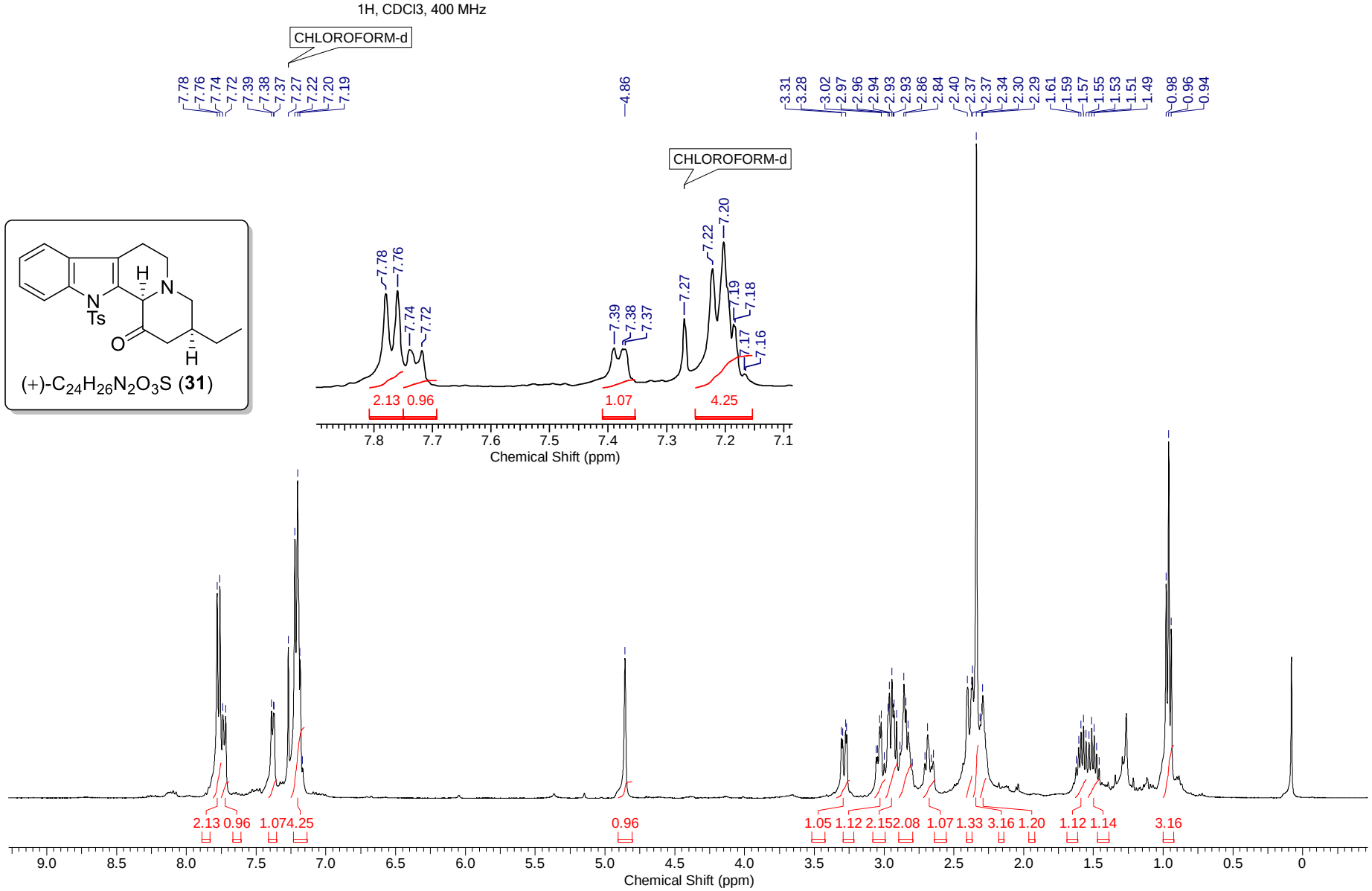


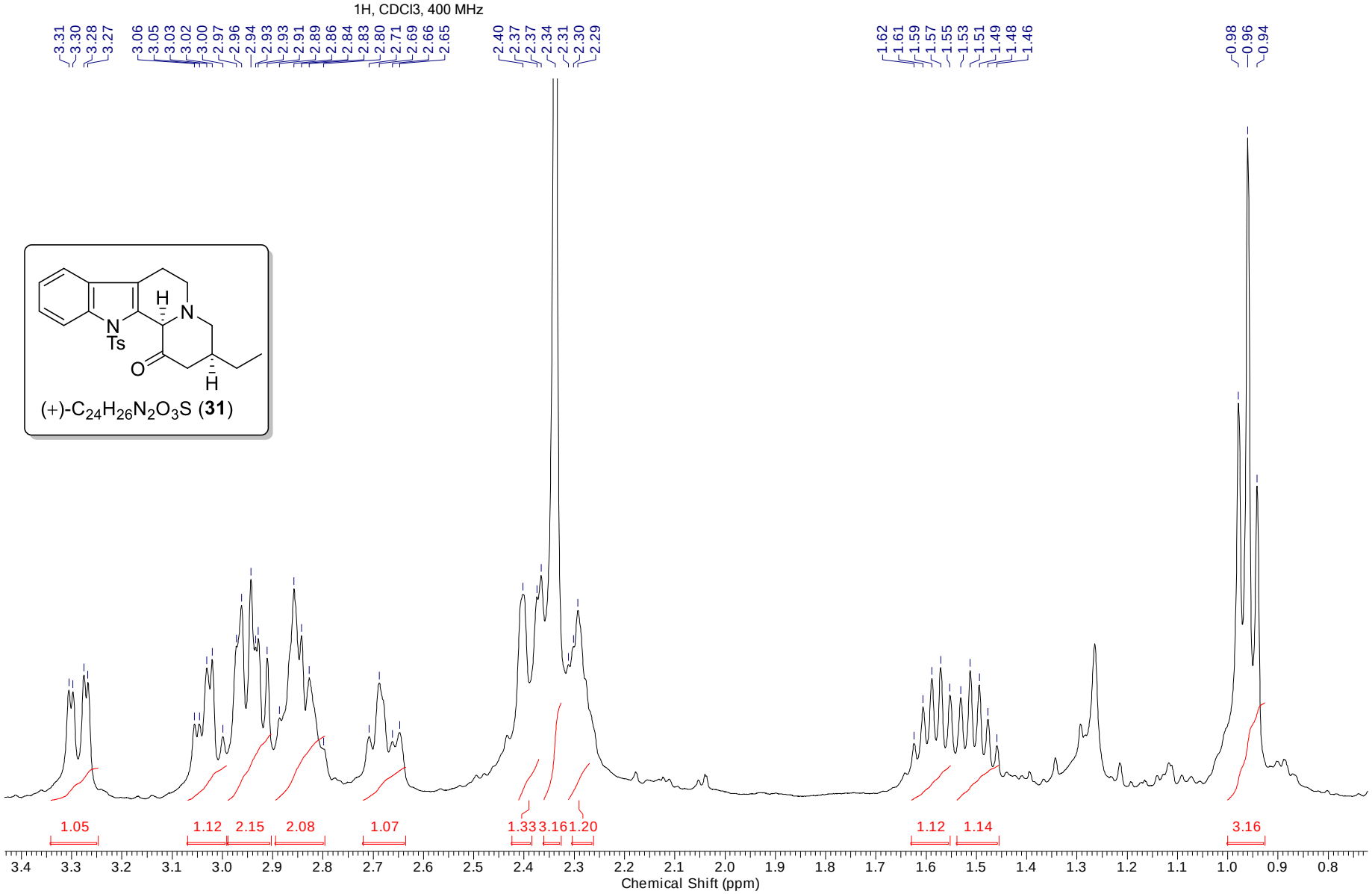

1H, CDCl3, 500 MHz
CHLOROFORM-d
8.07 8.06 7.37 7.35 7.29 7.28 7.27 7.27 7.22 7.20 7.01 7.00 4.76 4.45 3.79 3.74 3.27 3.25 3.25 3.24 2.82 2.81 2.80 2.79 2.41 2.39 2.39 2.38 2.37 2.35 2.34 2.25 2.24 1.95 1.84 1.81 1.30 1.29 1.28 1.26 1.25 1.24 0.91 0.89 0.88
(+)-C24H28N2O3S (30)
7.37 7.35 7.30 7.29 7.29 7.28 7.27 7.27 7.23 7.22 7.21 7.20 7.19 7.01 7.00
2.07 1.45 2.05 2.04
7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95
Chemical Shift (ppm)
1.00 2.07 1.45 2.05 2.04 1.01 1.01 2.01 1.03 1.05 1.09 1.01 2.07 3.03 1.07 1.04 2.54 3.15
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
Chemical Shift (ppm)

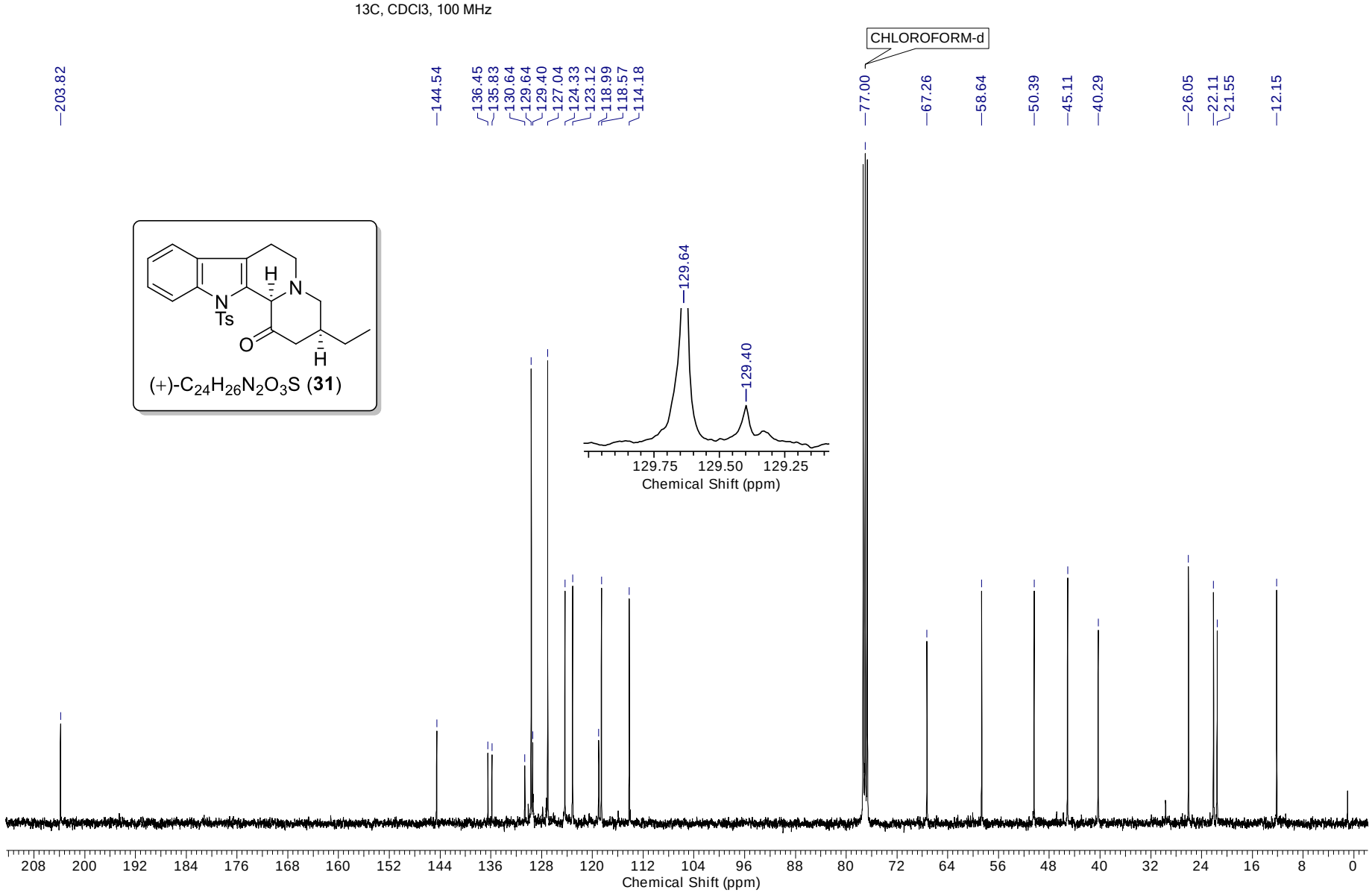

1H, CDCl3, 500 MHz
4.76
4.45
3.79
3.74
3.28
3.27
3.27
3.25
3.25
3.24
3.03
3.02
3.01
3.00
2.89
2.87
2.87
2.86
2.85
2.85
2.82
2.81
2.80
2.79
2.41
2.39
2.39
2.38
2.37
2.35
2.34
2.34
2.25
2.24
1.95
1.85
1.84
1.83
1.82
1.81
1.80
H
N
N
Ts
HO
H
(+)-C24H28N2O3S (30)
1.01
1.01
2.01
1.03
1.05
1.09
1.01
2.07
3.03
1.07
1.04
4.5
4.0
3.5
3.0
2.5
2.0
Chemical Shift (ppm)

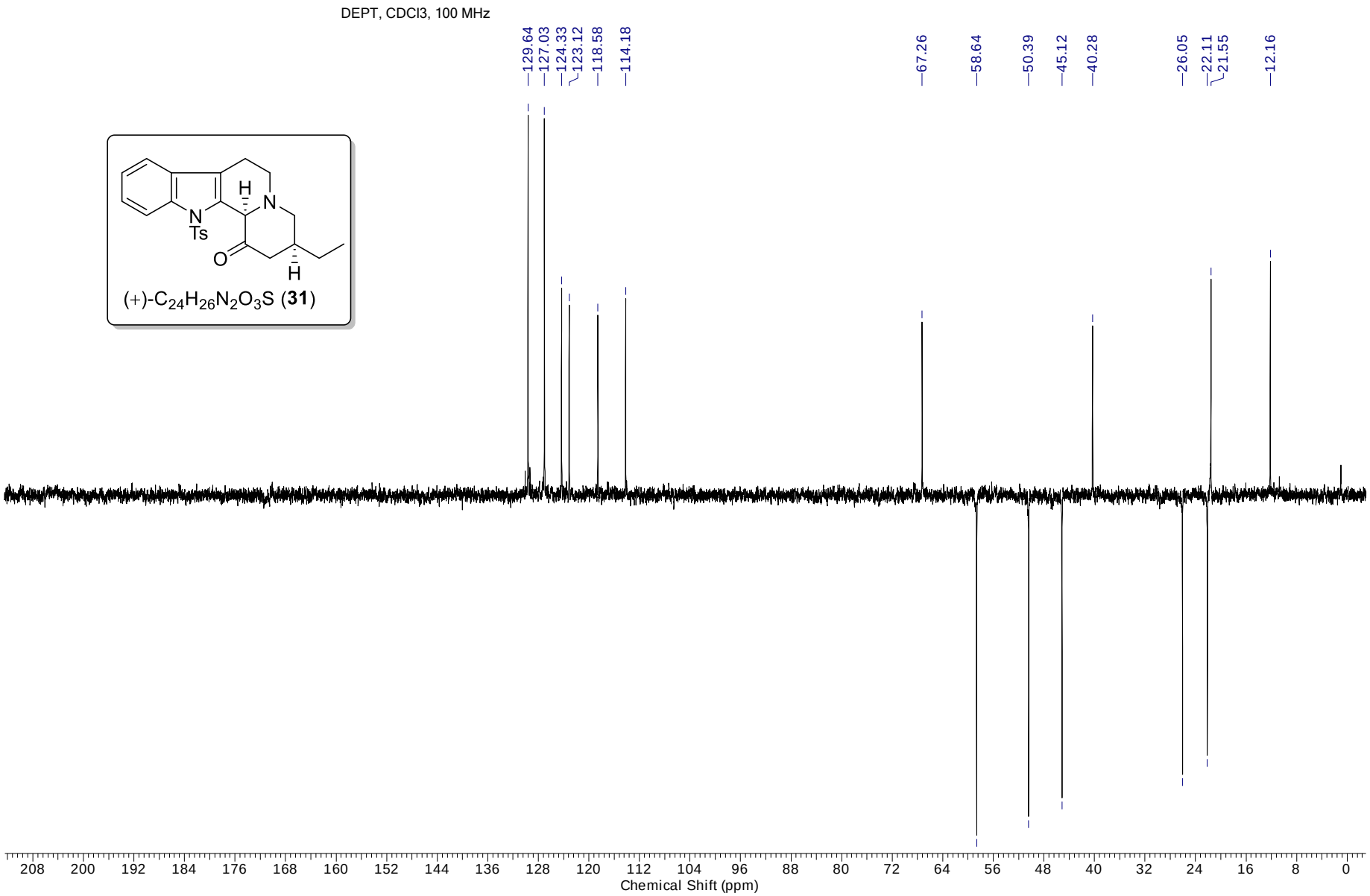

13C, CDCl3, 125 MHz
CHLOROFORM-d
144.56
138.87
134.98
131.92
131.77
128.94
126.79
124.90
124.70
123.95
118.39
117.40
77.00
68.48
63.27
53.60
49.49
35.25
32.45
26.98
21.45
19.21
11.62
131.92
131.77
132.0
131.5
Chemical Shift (ppm)
124.90
124.70
125.5
125.0
124.5
Chemical Shift (ppm)
N
Ts
H
N
HO
H
(+)-C24H28N2O3S (30)
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

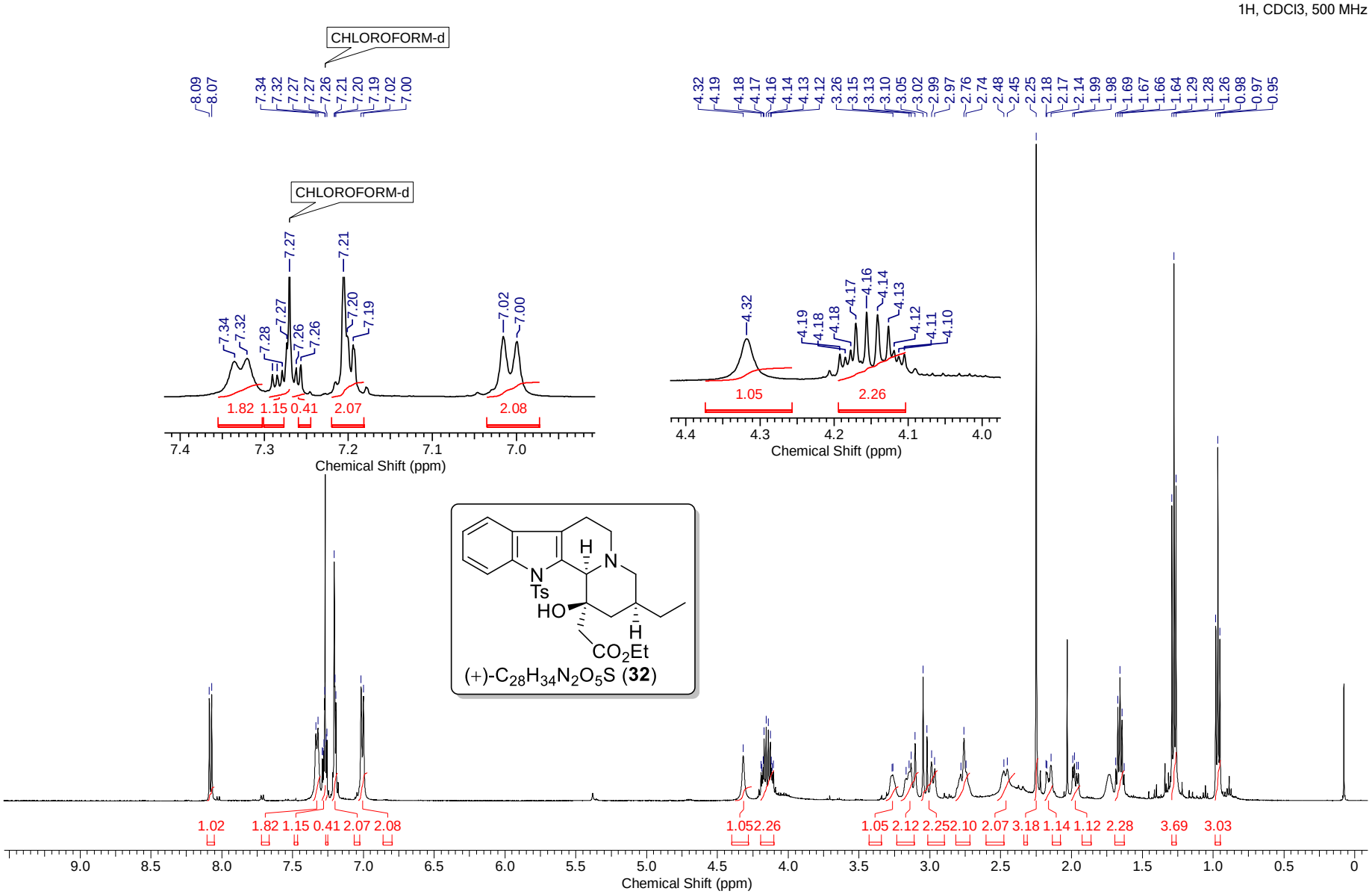

DEPT, CDCl3, 125 MHz
128.95
126.80
124.91
124.71
118.40
117.40
68.48
63.27
53.60
49.50
35.25
32.45
26.99
21.45
19.21
11.62
124.91
124.71
125.00
124.75
Chemical Shift (ppm)
(+)-C24H28N2O3S (30)
Chemical Shift (ppm)

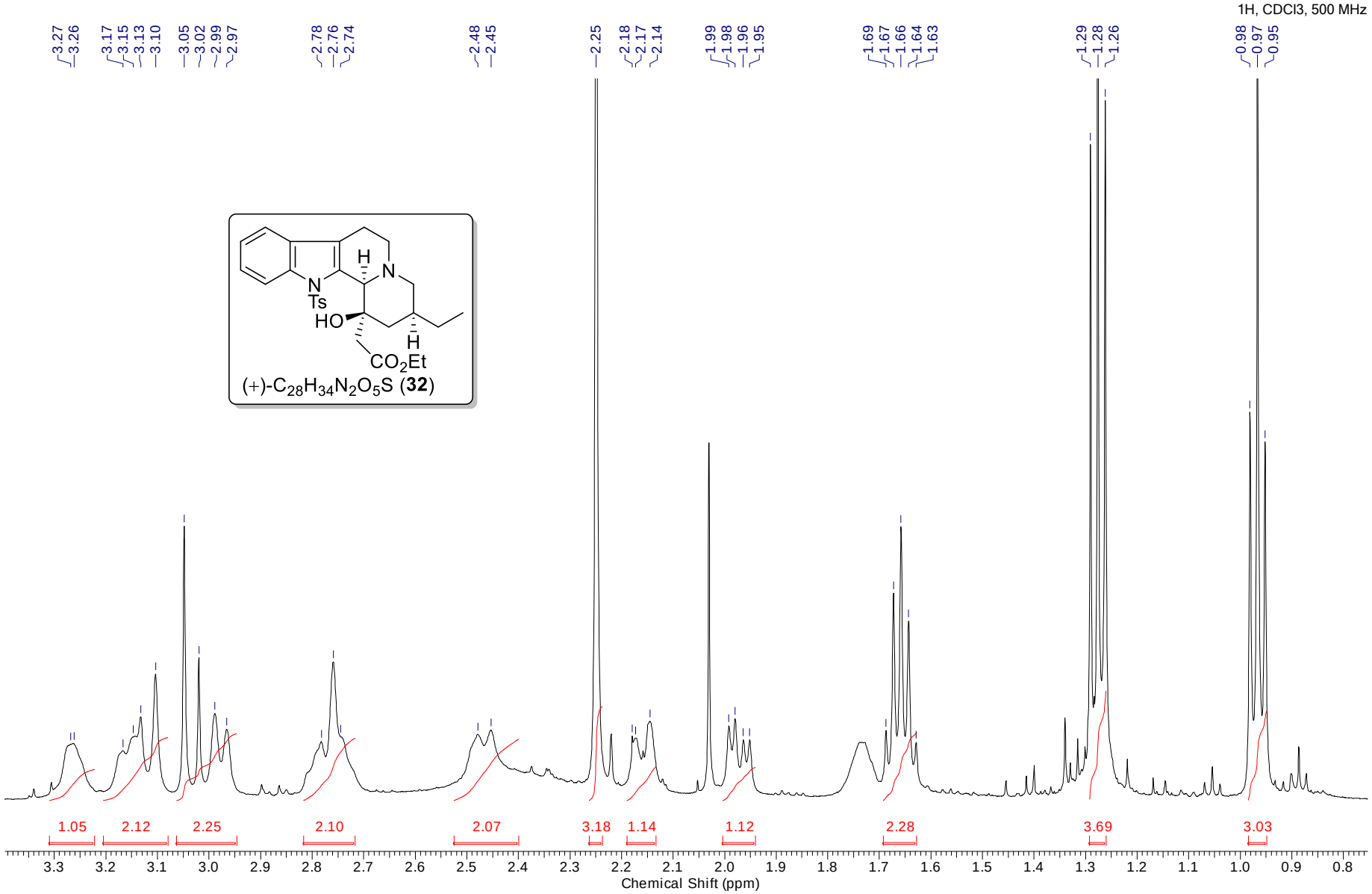

1H, CDCl3, 400 MHz
CHLOROFORM-d
7.78 7.76 7.74 7.72 7.39 7.38 7.37 7.27 7.22 7.20 7.19
4.86
3.31 3.28 3.02 2.97 2.96 2.94 2.93 2.93 2.86 2.84 2.40 2.37 2.37 2.34 2.30 2.29 1.61 1.59 1.57 1.55 1.53 1.51 1.49 0.98 0.96 0.94
CHLOROFORM-d
7.78 7.76 7.74 7.72 7.39 7.38 7.37 7.27 7.22 7.20 7.19 7.18 7.17 7.16
2.13 0.96 1.07 4.25
7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1
Chemical Shift (ppm)
(+)-C24H26N2O3S (31)
2.13 0.96 1.07 4.25
0.96
1.05 1.12 2.15 2.08 1.07 1.33 3.16 1.20 1.12 1.14 3.16
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
Chemical Shift (ppm)

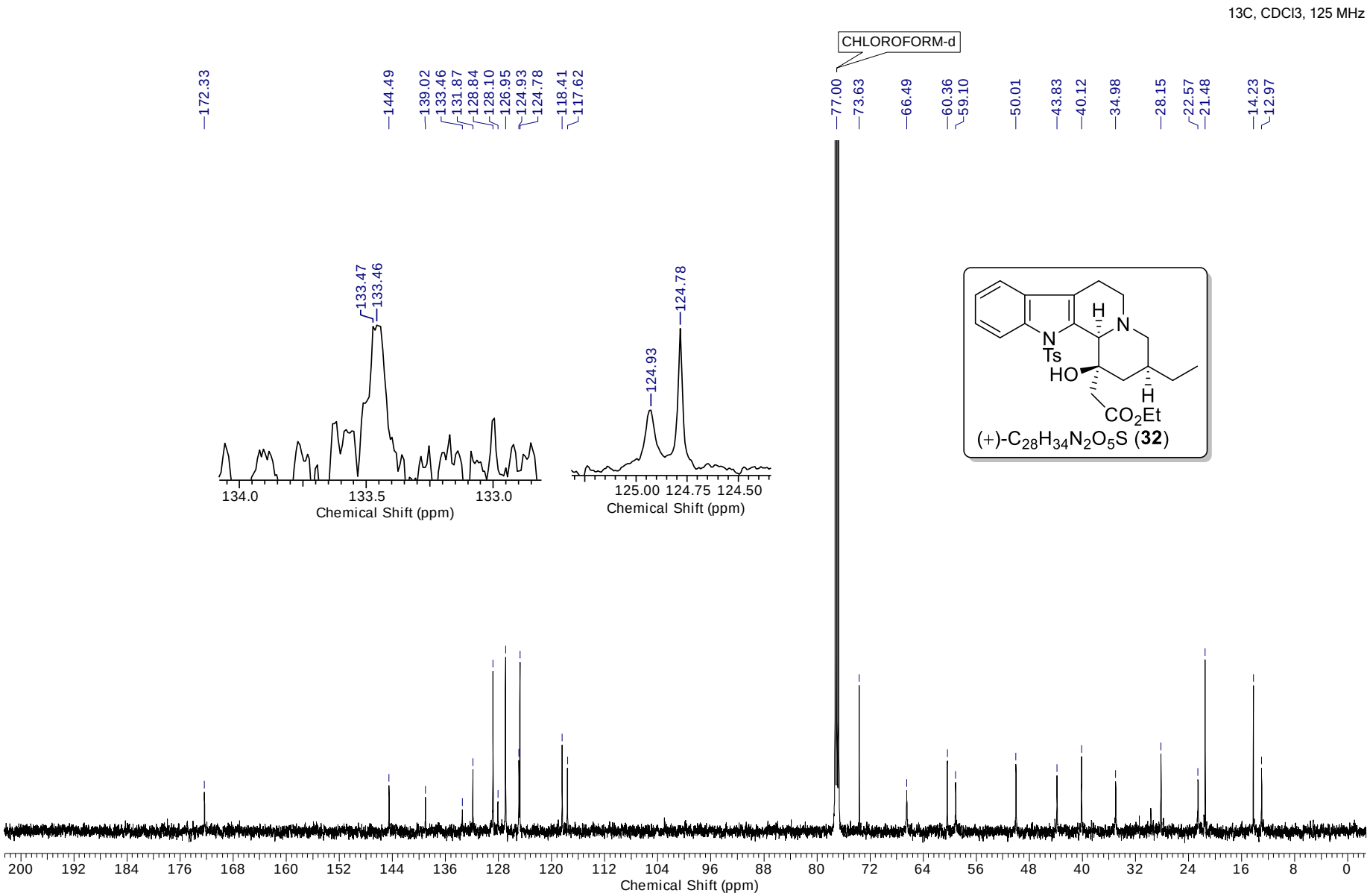

1H, CDCl3, 400 MHz
3.31
3.30
3.28
3.27
3.06
3.05
3.03
3.02
3.00
2.97
2.96
2.94
2.93
2.93
2.91
2.89
2.86
2.84
2.83
2.80
2.71
2.69
2.66
2.65
2.40
2.37
2.37
2.34
2.31
2.30
2.29
1.62
1.61
1.59
1.57
1.55
1.53
1.51
1.49
1.48
1.46
0.98
0.96
0.94
H
N
N
Ts
O
H
(+)-C24H26N2O3S (31)
1.05
1.12
2.15
2.08
1.07
1.33
3.16
1.20
1.12
1.14
3.16
3.4
3.3
3.2
3.1
3.0
2.9
2.8
2.7
2.6
2.5
2.4
2.3
2.2
2.1
2.0
1.9
1.8
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1.0
0.9
0.8
Chemical Shift (ppm)

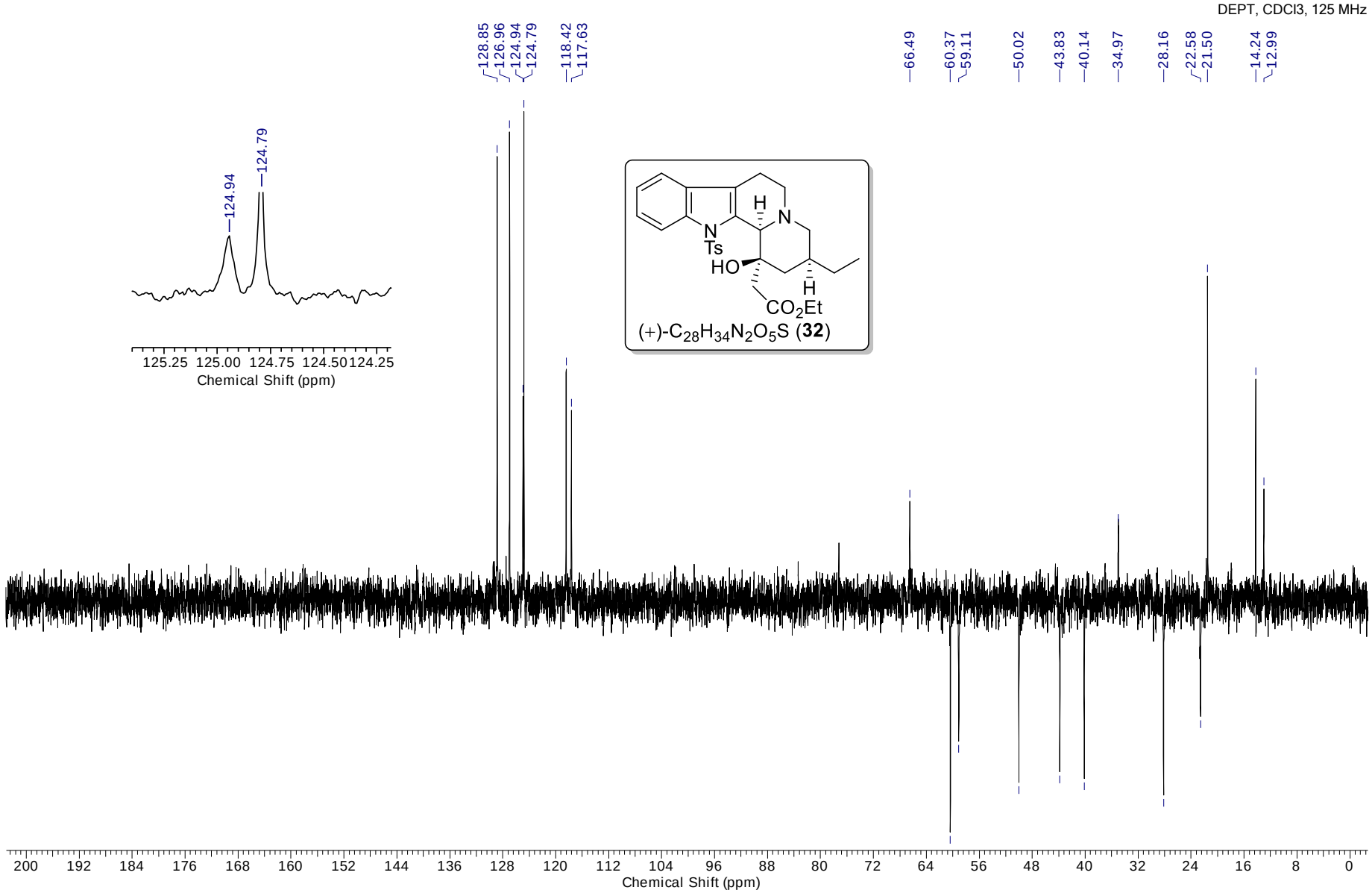

13C, CDCl3, 100 MHz
CHLOROFORM-d
203.82
144.54
136.45
135.83
130.64
129.64
129.40
127.04
124.33
123.12
118.99
118.57
114.18
77.00
67.26
58.64
50.39
45.11
40.29
26.05
22.11
21.55
12.15
(+)-C24H26N2O3S (31)
129.64
129.40
129.75 129.50 129.25
Chemical Shift (ppm)
208 200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

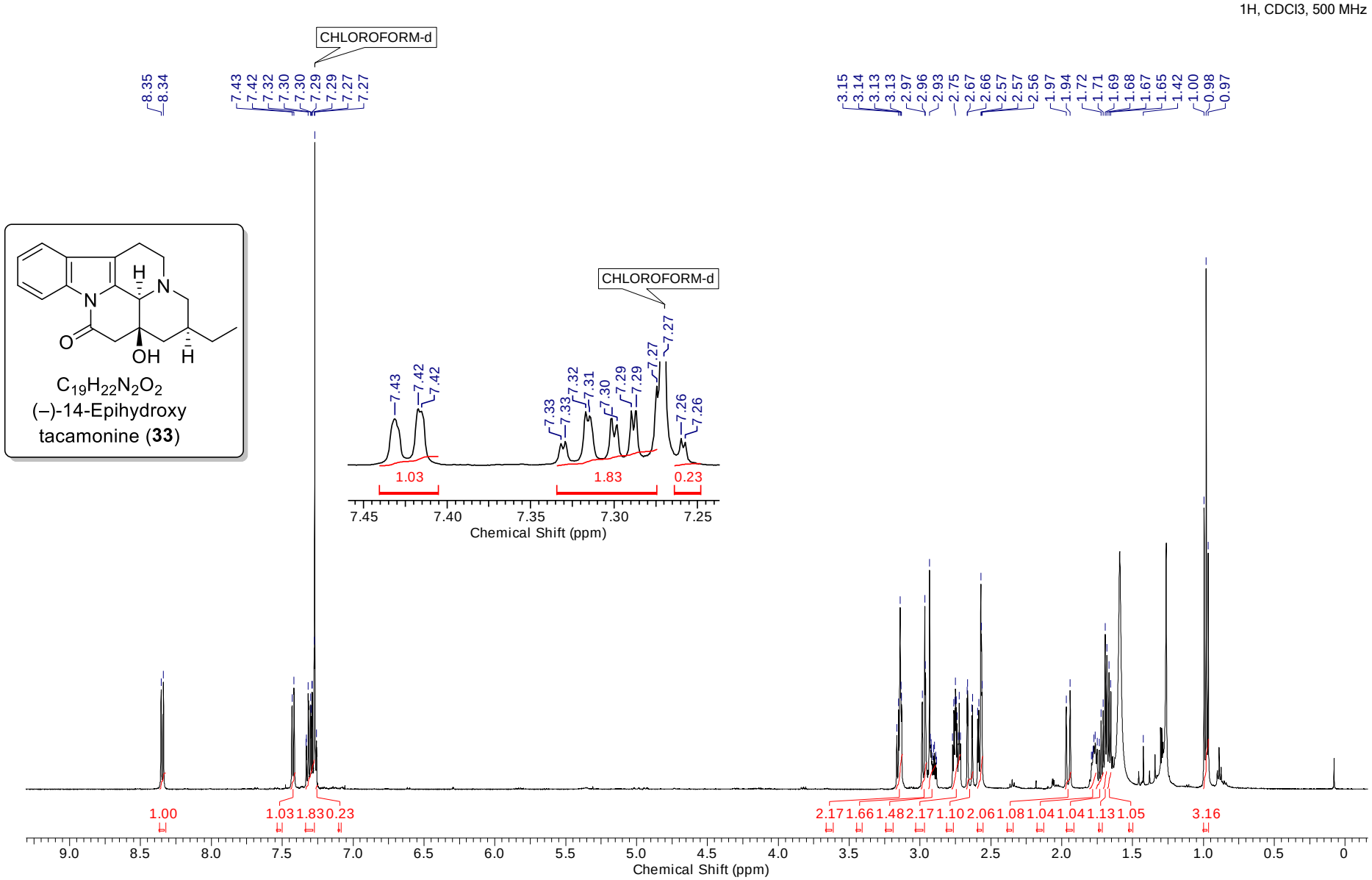

DEPT, CDCl3, 100 MHz
129.64
127.03
124.33
123.12
118.58
114.18
67.26
58.64
50.39
45.12
40.28
26.05
22.11
21.55
12.16
H
N
N
Ts
O
H
(+)-C24H26N2O3S (31)
208 200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

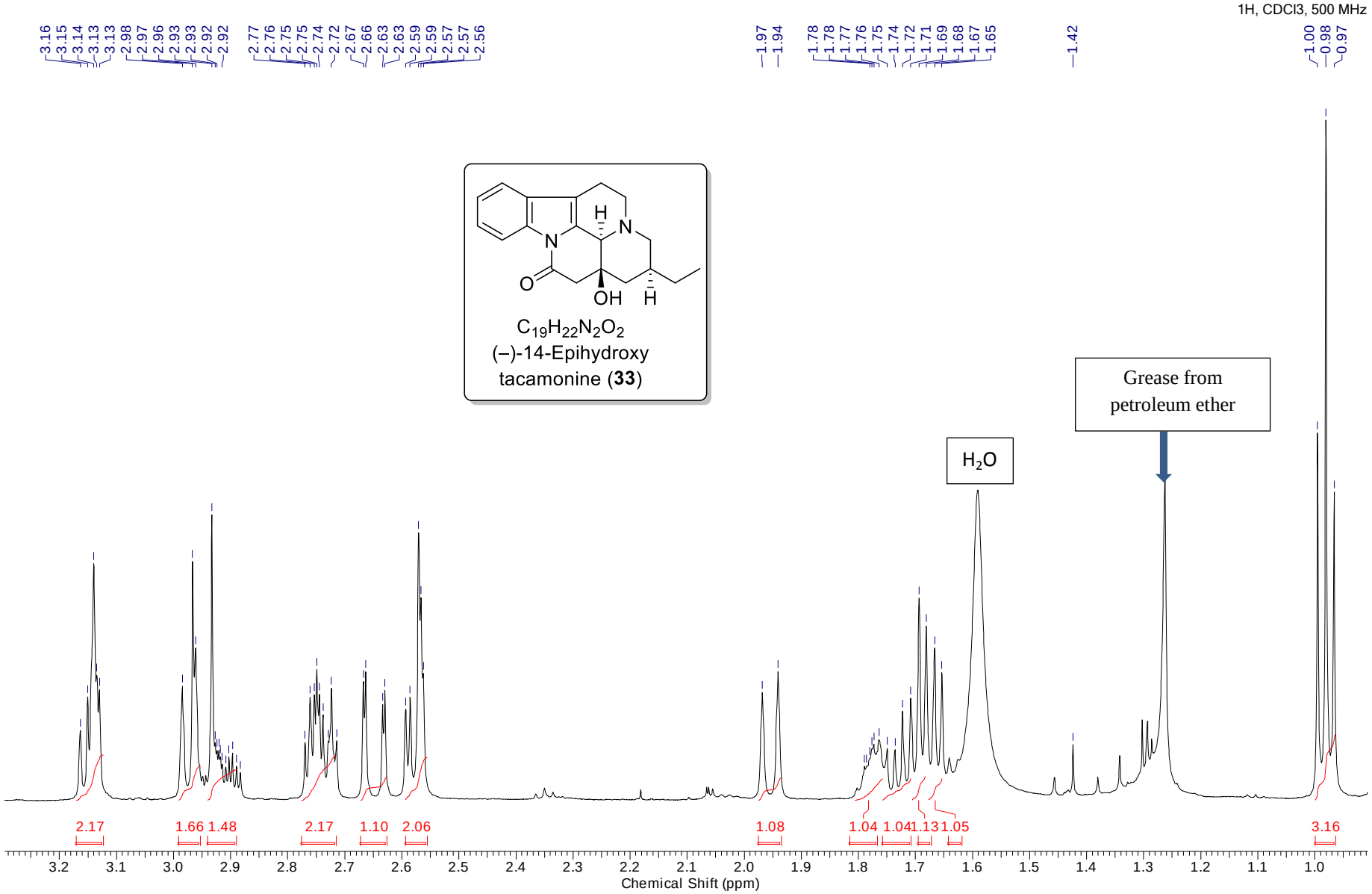

1H, CDCl3, 500 MHz
CHLOROFORM-d
8.09
8.07
7.34
7.32
7.27
7.27
7.26
7.21
7.20
7.19
7.02
7.00
4.32
4.19
4.18
4.17
4.16
4.14
4.13
4.12
3.26
3.15
3.13
3.10
3.05
3.02
2.99
2.97
2.76
2.74
2.48
2.45
2.25
2.18
2.17
2.14
1.99
1.98
1.69
1.67
1.66
1.64
1.29
1.28
1.26
0.98
0.97
0.95
1.82 1.15 0.41 2.07
2.08
1.05
2.26
Chemical Shift (ppm)
N
Ts
HO
H
N
H
CO2Et
(+)-C28H34N2O5S (32)
1.02
1.82 1.15 0.41 2.07 2.08
1.05 2.26
1.05 2.12 2.25 2.10 2.07 3.18 1.14 1.12 2.28
3.69
3.03
Chemical Shift (ppm)

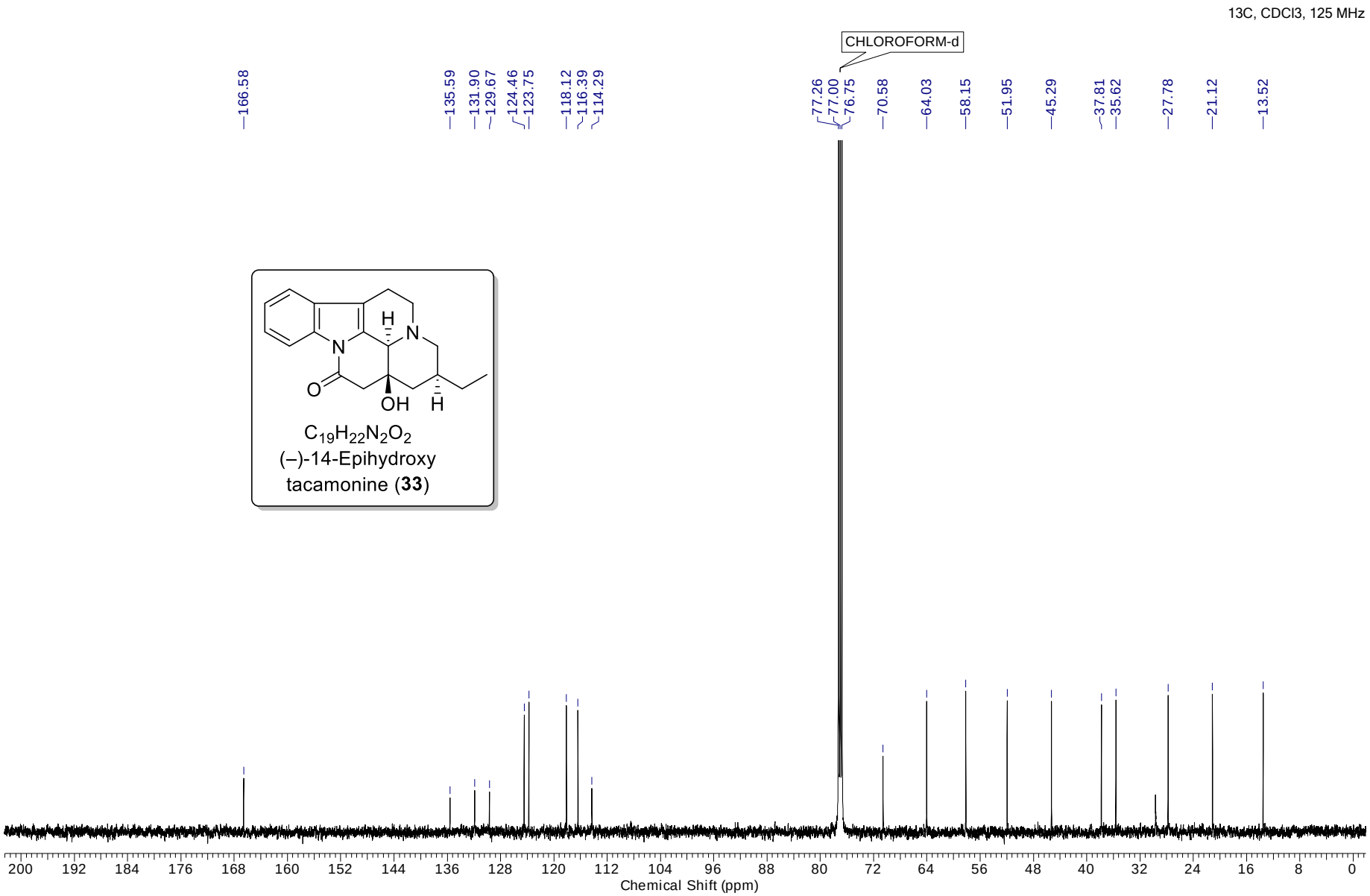

1H, CDCl3, 500 MHz
3.27
3.26
3.17
3.15
3.13
3.10
3.05
3.02
2.99
2.97
2.78
2.76
2.74
2.48
2.45
2.25
2.18
2.17
2.14
1.99
1.98
1.96
1.95
1.69
1.67
1.66
1.64
1.63
1.29
1.28
1.26
0.98
0.97
0.95
H
N
N
Ts
HO
H
CO2Et
(+)-C28H34N2O5S (32)
1.05
2.12
2.25
2.10
2.07
3.18
1.14
1.12
2.28
3.69
3.03
3.3
3.2
3.1
3.0
2.9
2.8
2.7
2.6
2.5
2.4
2.3
2.2
2.1
2.0
1.9
1.8
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1.0
0.9
0.8
Chemical Shift (ppm)

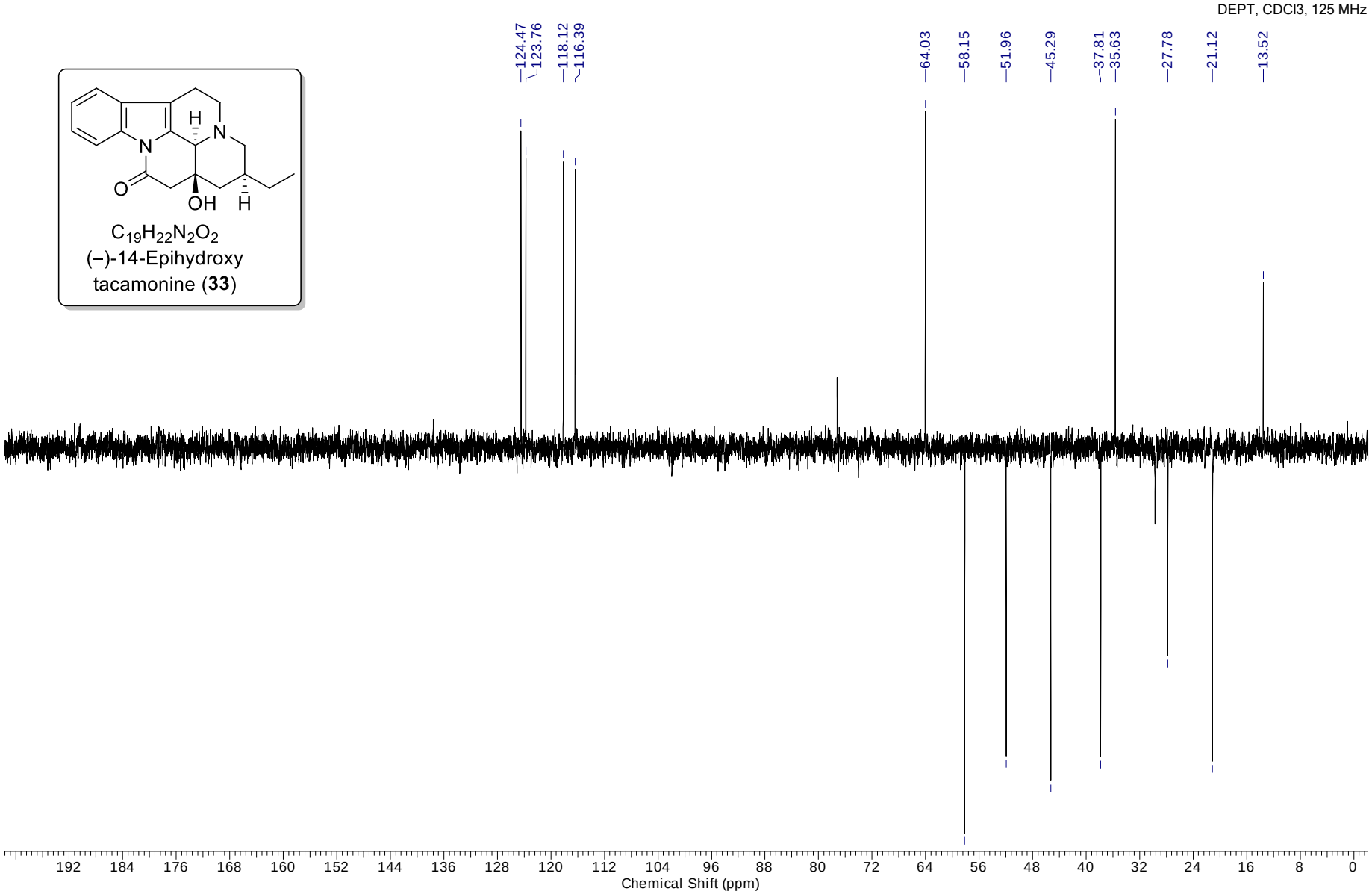

13C, CDCl3, 125 MHz
CHLOROFORM-d
172.33
144.49
139.02
133.46
131.87
128.84
128.10
126.95
124.93
124.78
118.41
117.62
77.00
73.63
66.49
60.36
59.10
50.01
43.83
40.12
34.98
28.15
22.57
21.48
14.23
12.97
133.47
133.46
134.0
133.5
133.0
Chemical Shift (ppm)
124.78
124.93
125.00 124.75 124.50
Chemical Shift (ppm)
H
N
N
Ts
HO
H
CO2Et
(+)-C28H34N2O5S (32)
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

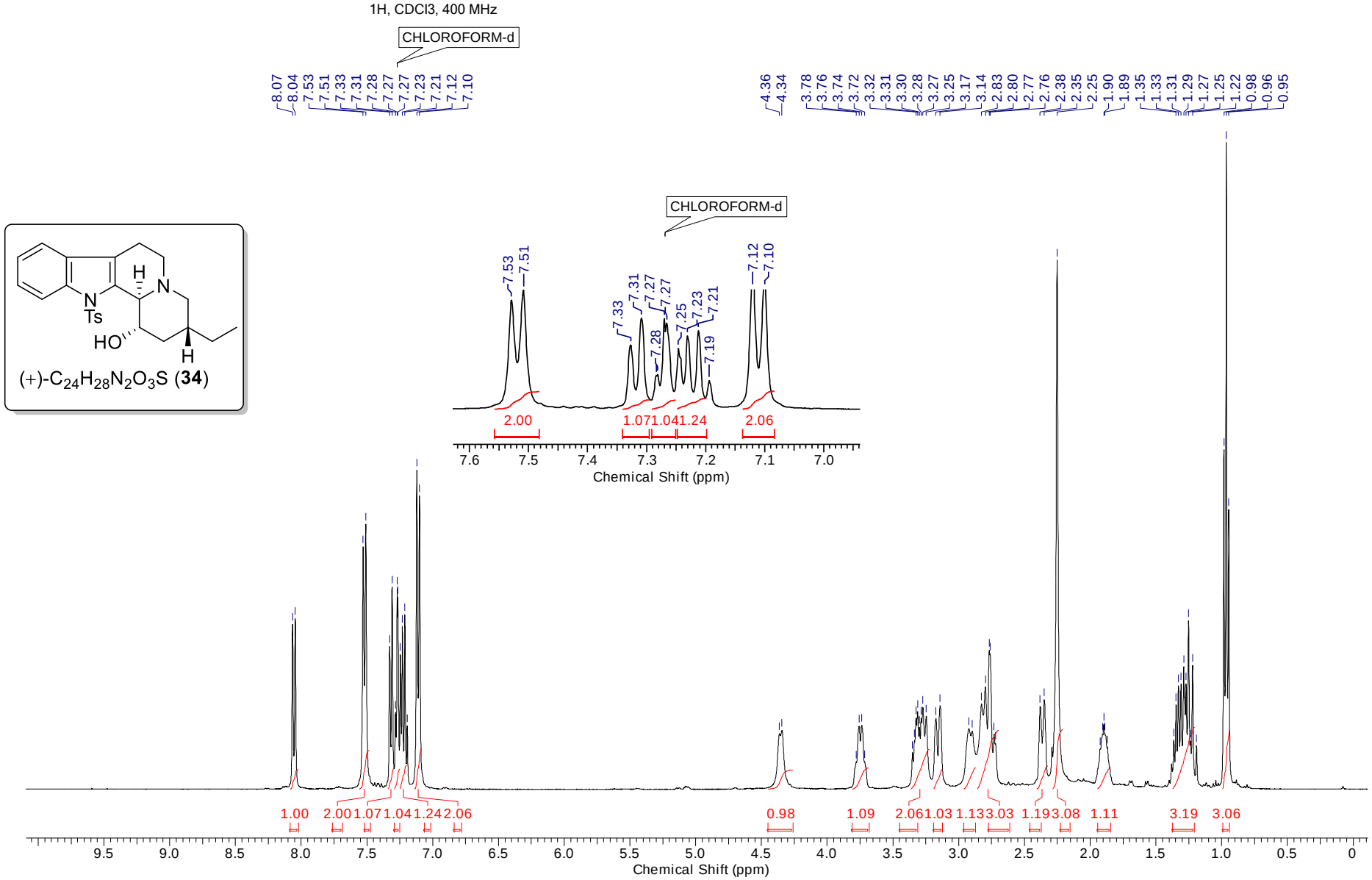

DEPT, CDCl3, 125 MHz
128.85
126.96
124.94
124.79
118.42
117.63
66.49
60.37
59.11
50.02
43.83
40.14
34.97
28.16
22.58
21.50
14.24
12.99
124.94
124.79
125.25 125.00 124.75 124.50 124.25
Chemical Shift (ppm)
H
N
N
Ts
HO
H
CO2Et
(+)-C28H34N2O5S (32)
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

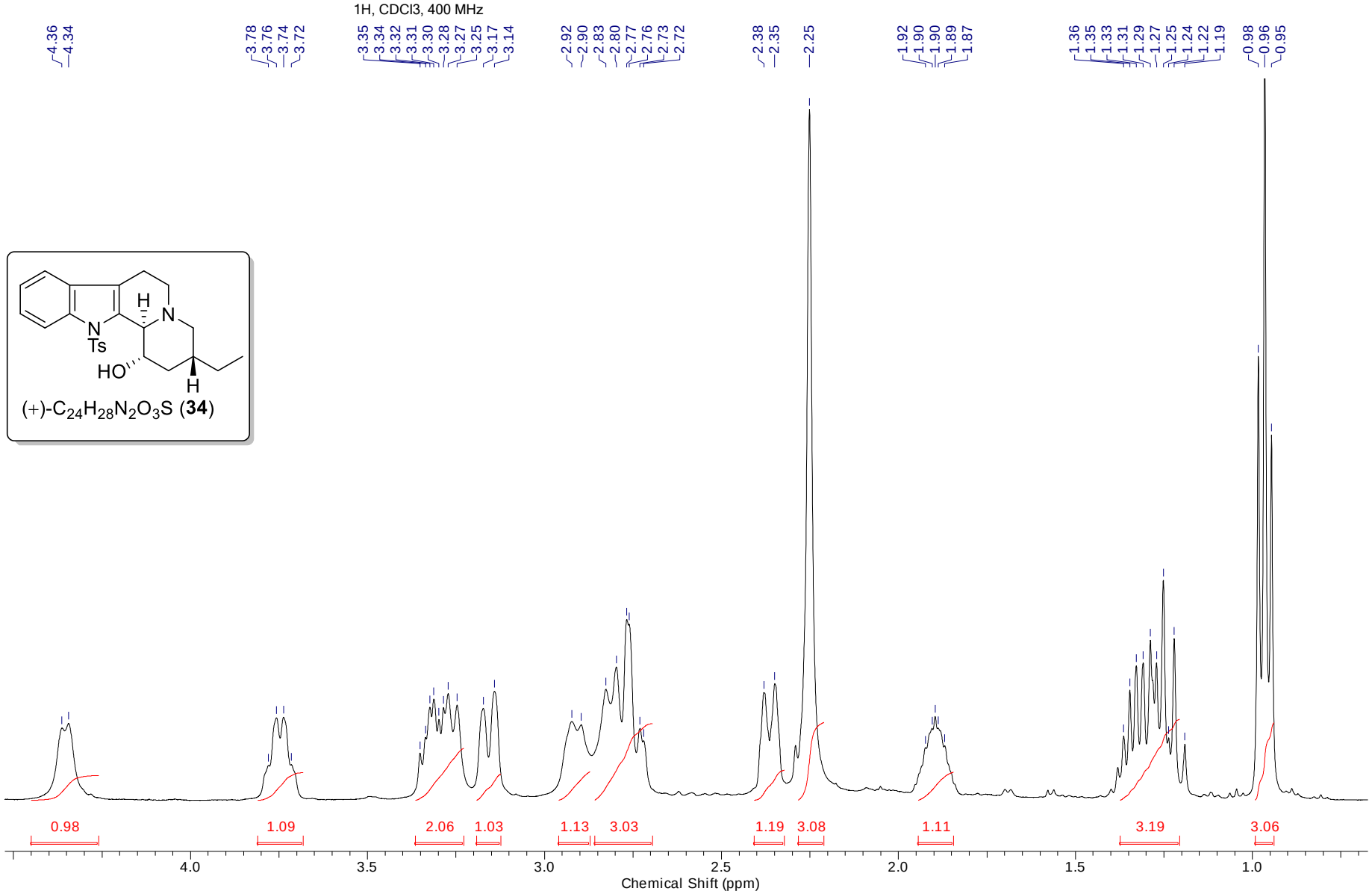

1H, CDCl3, 500 MHz
CHLOROFORM-d
8.35
8.34
7.43
7.42
7.32
7.30
7.30
7.29
7.29
7.27
7.27
3.15
3.14
3.13
3.13
2.97
2.96
2.93
2.75
2.67
2.66
2.57
2.57
2.56
1.97
1.94
1.72
1.71
1.69
1.68
1.67
1.65
1.42
1.00
0.98
0.97
H
N
N
O
OH
H
C19H22N2O2
(–)-14-Epihydroxy
tacamonine (33)
CHLOROFORM-d
7.43
7.42
7.42
7.33
7.33
7.32
7.31
7.30
7.29
7.29
7.27
7.27
7.26
7.26
1.03
1.83
0.23
7.45
7.40
7.35
7.30
7.25
Chemical Shift (ppm)
1.00
1.03
1.83
0.23
2.17
1.66
1.48
2.17
1.10
2.06
1.08
1.04
1.04
1.13
1.05
3.16
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
Chemical Shift (ppm)

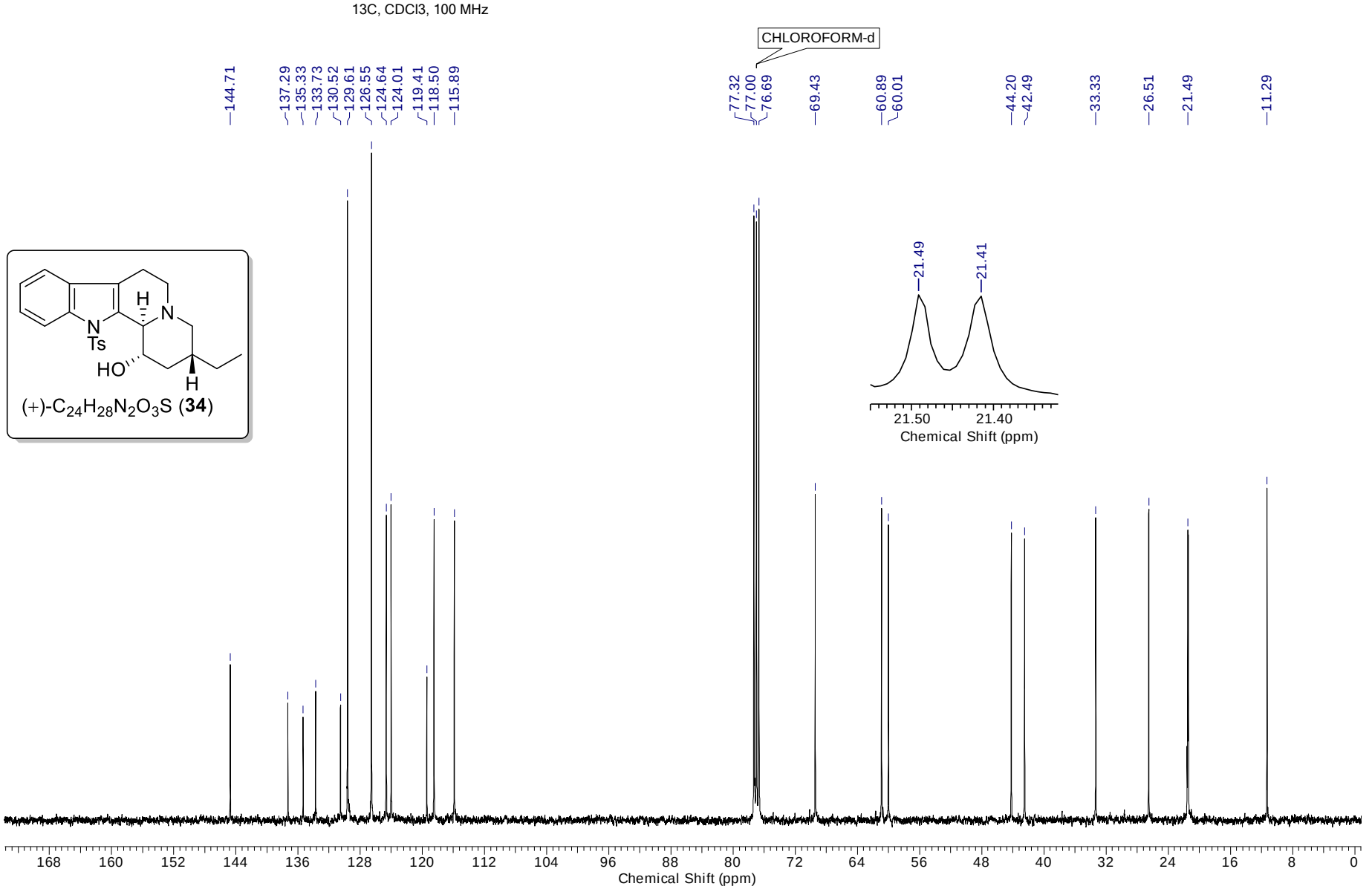

1H, CDCl3, 500 MHz
3.16 3.15 3.14 3.13 3.13 2.98 2.97 2.96 2.93 2.93 2.92 2.92 2.77 2.76 2.75 2.75 2.74 2.72 2.67 2.66 2.63 2.63 2.59 2.59 2.57 2.57 2.56
1.97 1.94 1.78 1.78 1.77 1.76 1.75 1.74 1.72 1.71 1.69 1.68 1.67 1.65 1.42 1.00 0.98 0.97
C19H22N2O2
(–)-14-Epihydroxy
tacamonine (33)
H2O
Grease from
petroleum ether
2.17 1.66 1.48 2.17 1.10 2.06 1.08 1.04 1.04 1.13 1.05 3.16
Chemical Shift (ppm)

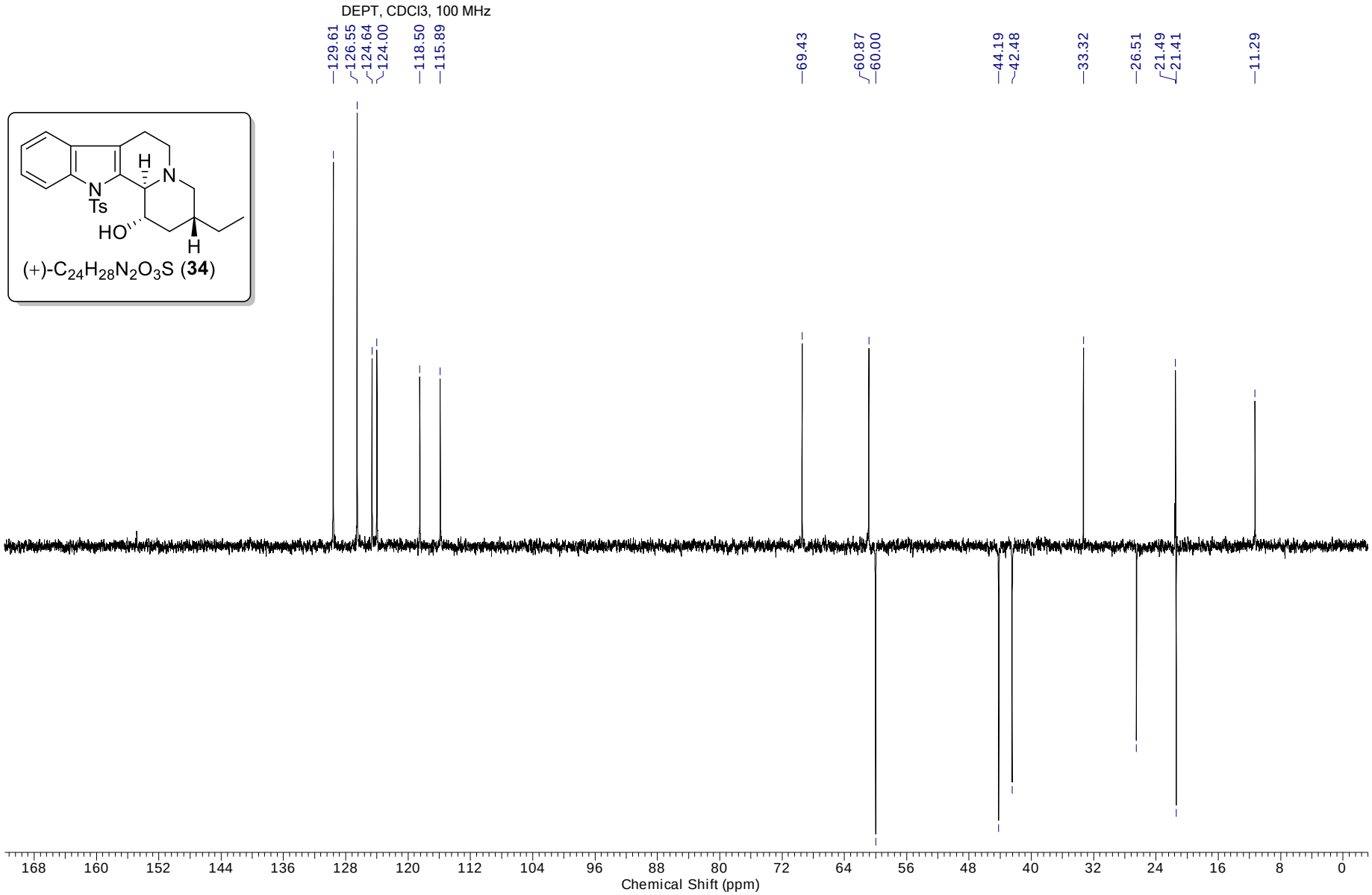

13C, CDCl3, 125 MHz
CHLOROFORM-d
166.58
135.59
131.90
129.67
124.46
123.75
118.12
116.39
114.29
77.26
77.00
76.75
70.58
64.03
58.15
51.95
45.29
37.81
35.62
27.78
21.12
13.52
H
N
N
O
OH
H
C19H22N2O2
(–)-14-Epihydroxy
tacamonine (33)
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

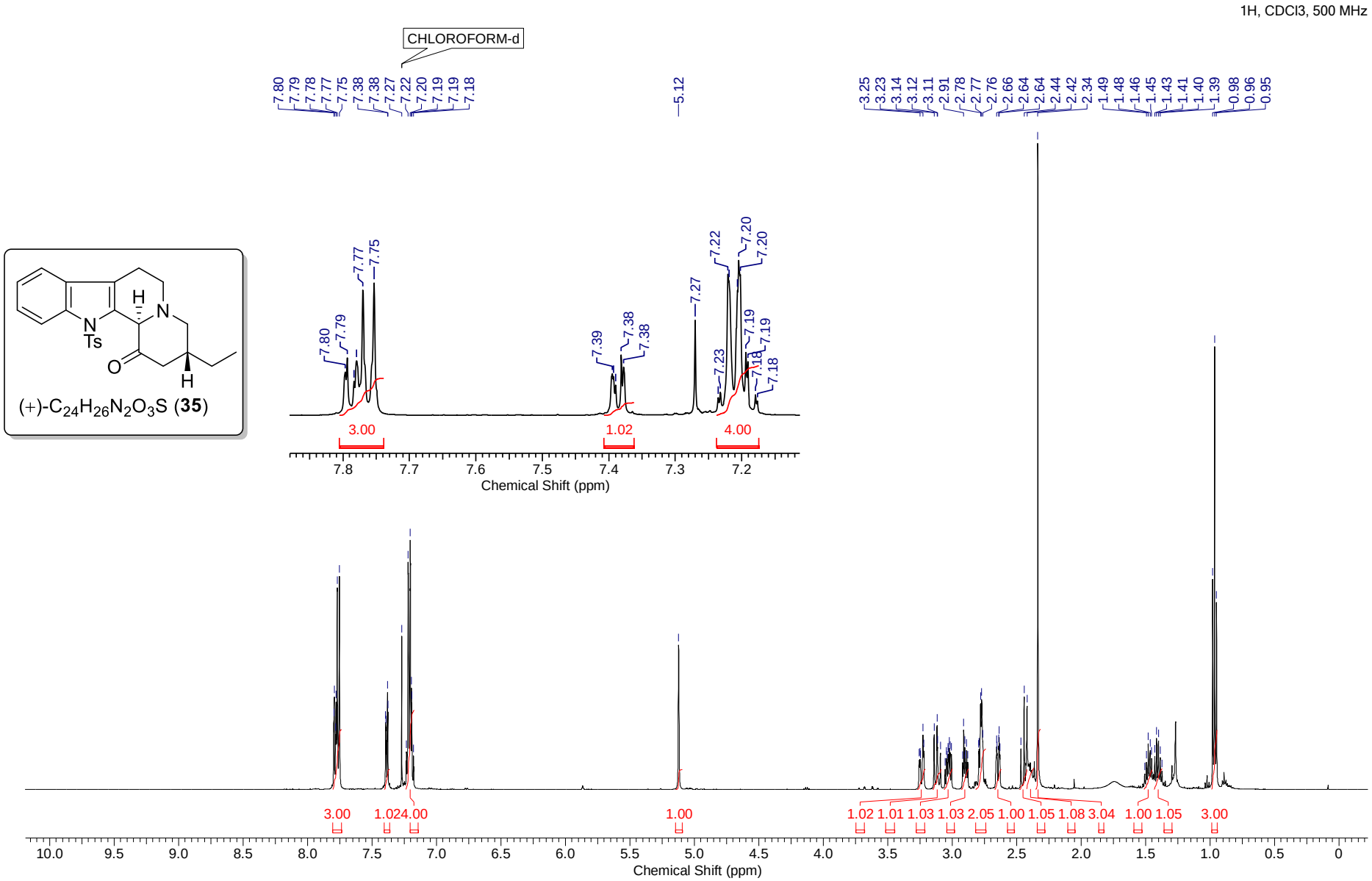

DEPT, CDCl3, 125 MHz
124.47
123.76
118.12
116.39
64.03
58.15
51.96
45.29
37.81
35.63
27.78
21.12
13.52
H
N
N
O
OH
H
C19H22N2O2
(–)-14-Epihydroxy
tacamonine (33)
192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

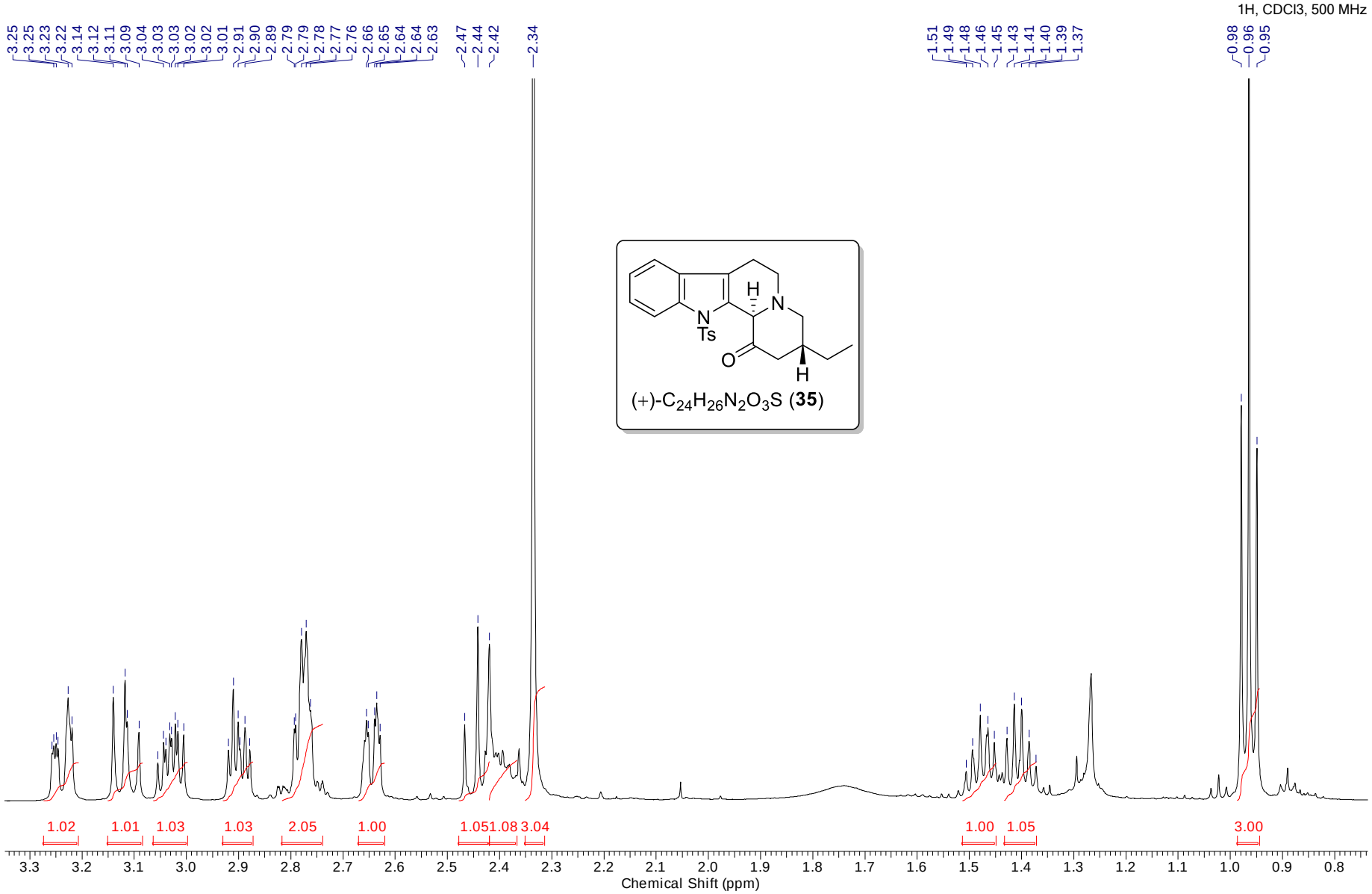

1H, CDCl3, 400 MHz
CHLOROFORM-d
8.07 8.04 7.53 7.51 7.33 7.31 7.28 7.27 7.27 7.23 7.21 7.12 7.10
4.36 4.34 3.78 3.76 3.74 3.72 3.32 3.31 3.30 3.28 3.27 3.25 3.17 3.14 2.83 2.80 2.77 2.76 2.38 2.35 2.25 1.90 1.89 1.35 1.33 1.31 1.29 1.27 1.25 1.22 0.98 0.96 0.95
(+)-C24H28N2O3S (34)
CHLOROFORM-d
7.53 7.51 7.33 7.31 7.27 7.27 7.28 7.25 7.23 7.21 7.19 7.12 7.10
2.00 1.07 1.04 1.24 2.06
7.6 7.5 7.4 7.3 7.2 7.1 7.0
Chemical Shift (ppm)
1.00 2.00 1.07 1.04 1.24 2.06 0.98 1.09 2.06 1.03 1.13 3.03 1.19 3.08 1.11 3.19 3.06
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
Chemical Shift (ppm)

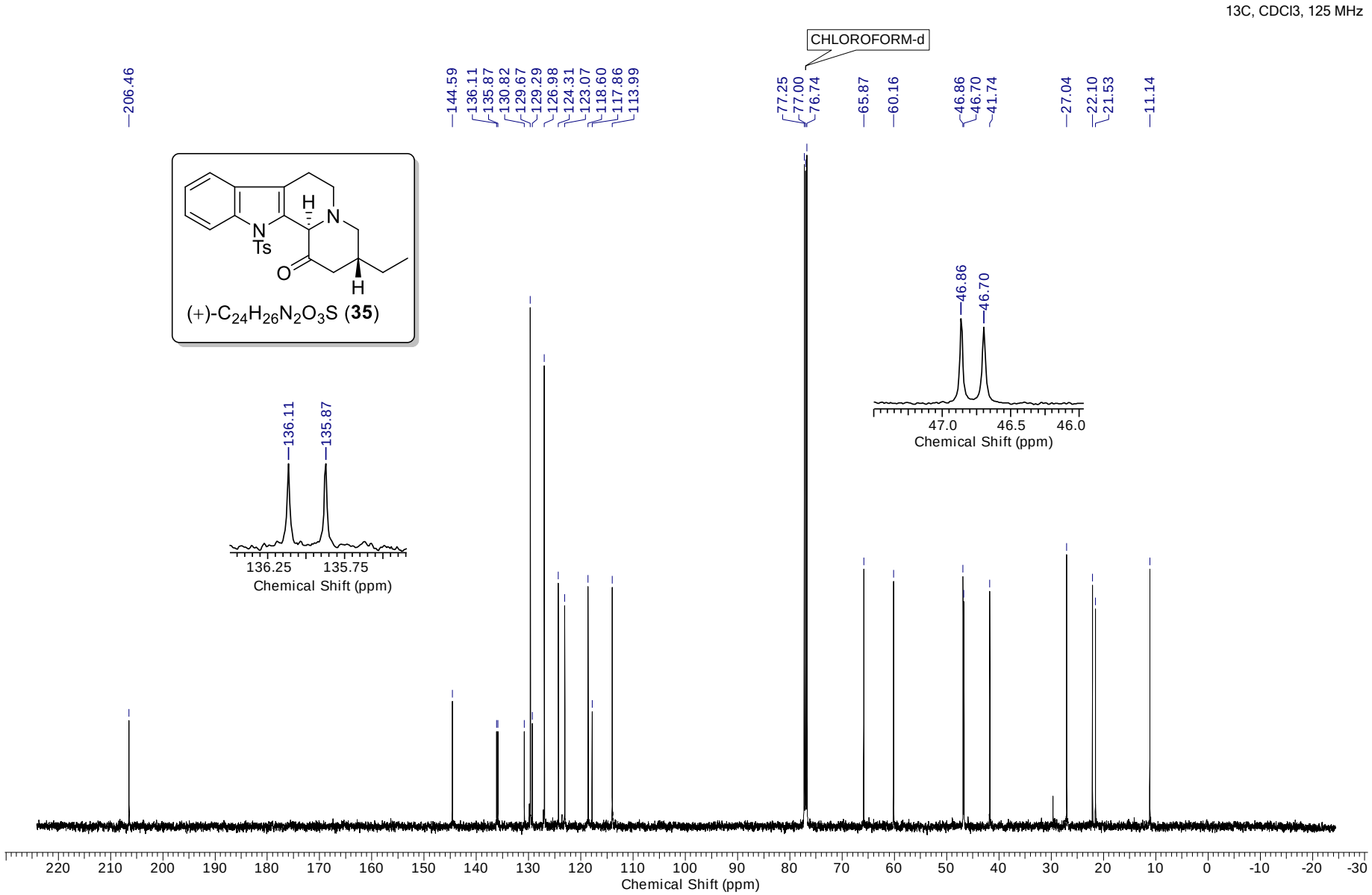

1H, CDCl3, 400 MHz
4.36
4.34
3.78
3.76
3.74
3.72
3.35
3.34
3.32
3.31
3.30
3.28
3.27
3.25
3.17
3.14
2.92
2.90
2.83
2.80
2.77
2.76
2.73
2.72
2.38
2.35
2.25
1.92
1.90
1.90
1.89
1.87
1.36
1.35
1.33
1.31
1.29
1.27
1.25
1.24
1.22
1.19
0.98
0.96
0.95
H
N
N
Ts
HO
H
(+)-C24H28N2O3S (34)
0.98
1.09
2.06
1.03
1.13
3.03
1.19
3.08
1.11
3.19
3.06
4.0
3.5
3.0
2.5
2.0
1.5
1.0
Chemical Shift (ppm)

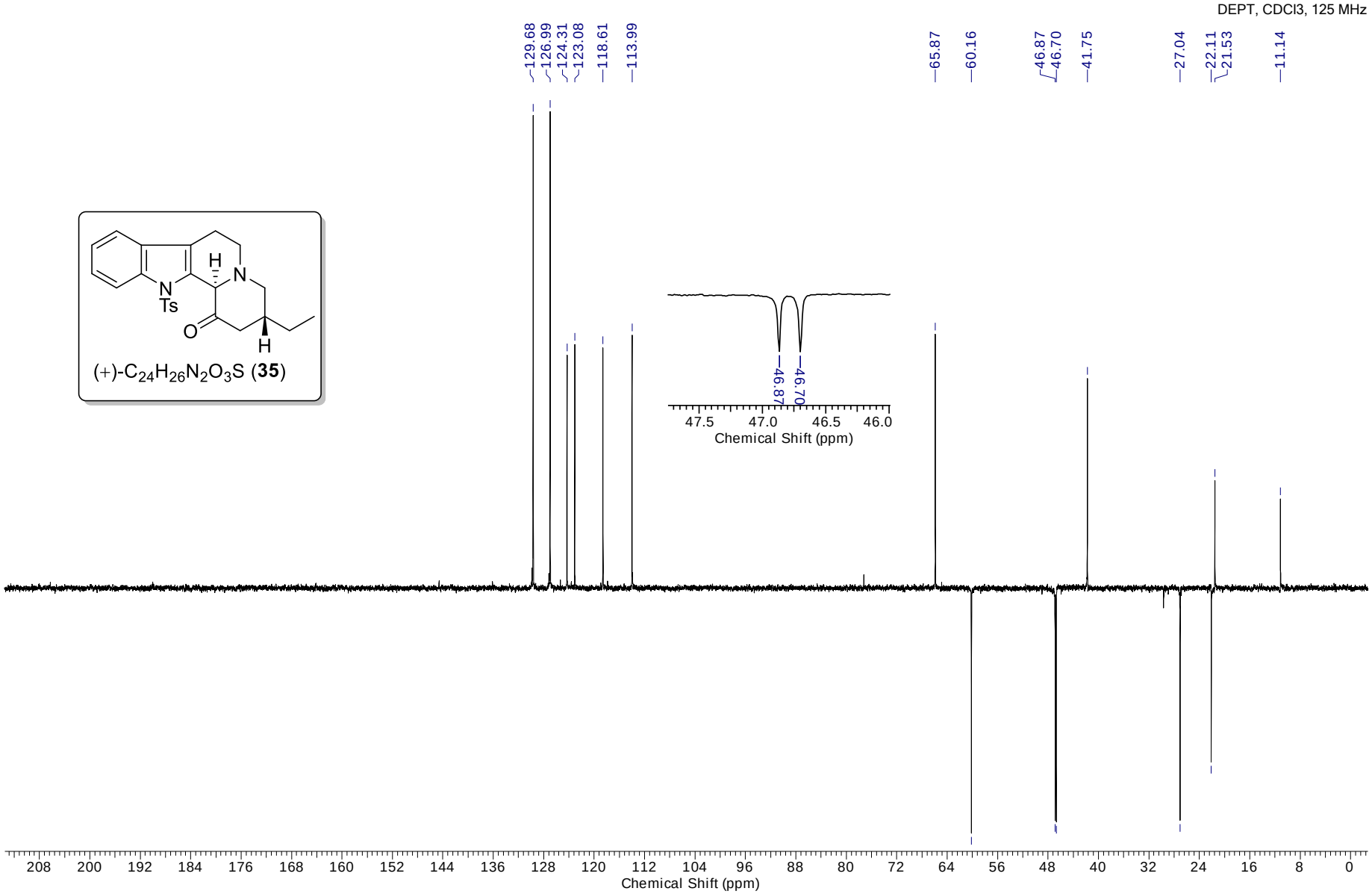

13C, CDCl3, 100 MHz
CHLOROFORM-d
144.71
137.29
135.33
133.73
130.52
129.61
126.55
124.64
124.01
119.41
118.50
115.89
77.32
77.00
76.69
69.43
60.89
60.01
44.20
42.49
33.33
26.51
21.49
11.29
21.49
21.41
21.50
21.40
Chemical Shift (ppm)
(+)-C24H28N2O3S (34)
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

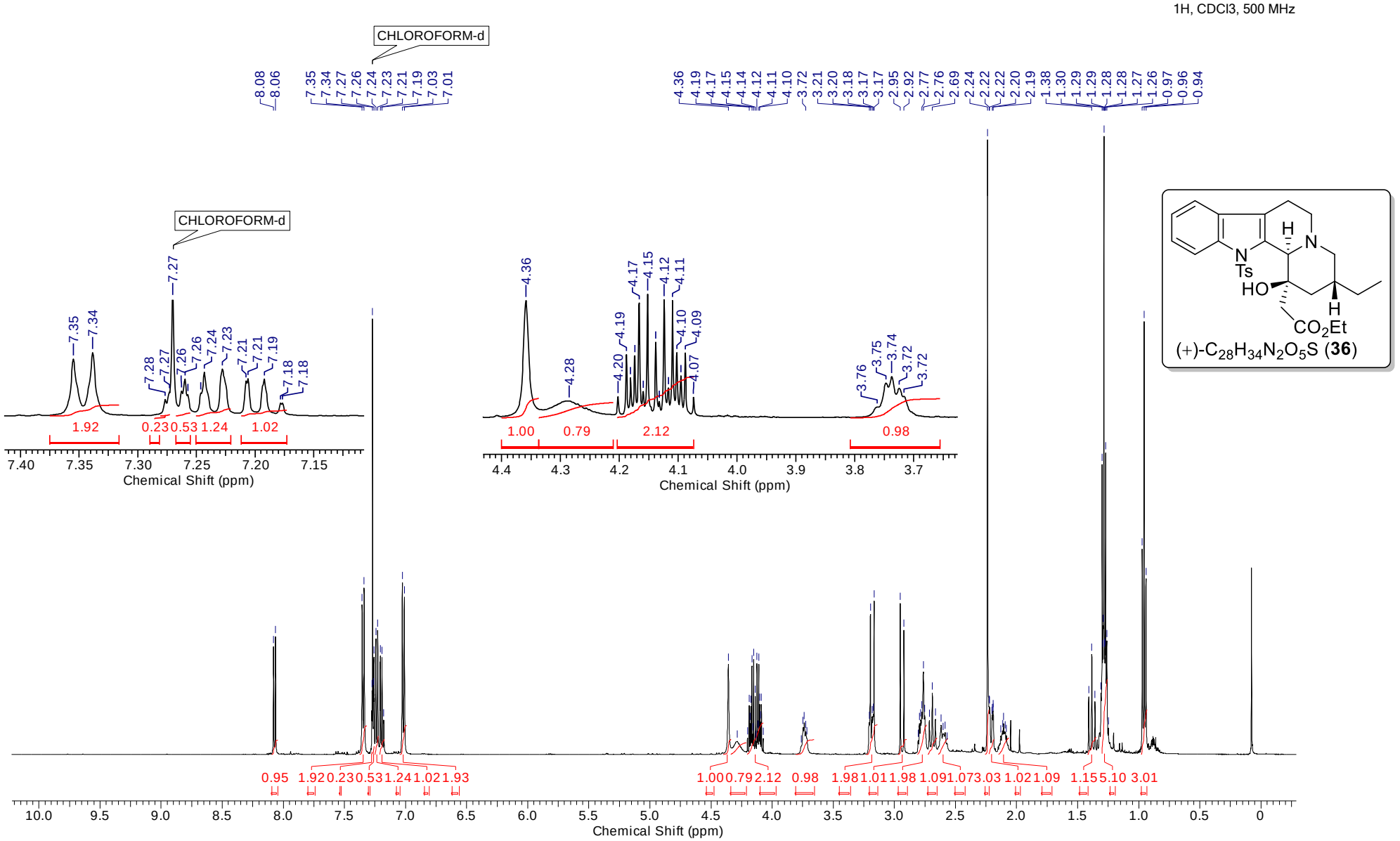

DEPT, CDCl3, 100 MHz
129.61
126.55
124.64
124.00
118.50
115.89
69.43
60.87
60.00
44.19
42.48
33.32
26.51
21.49
21.41
11.29
Ts
HO
H
H
(+)-C24H28N2O3S (34)
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

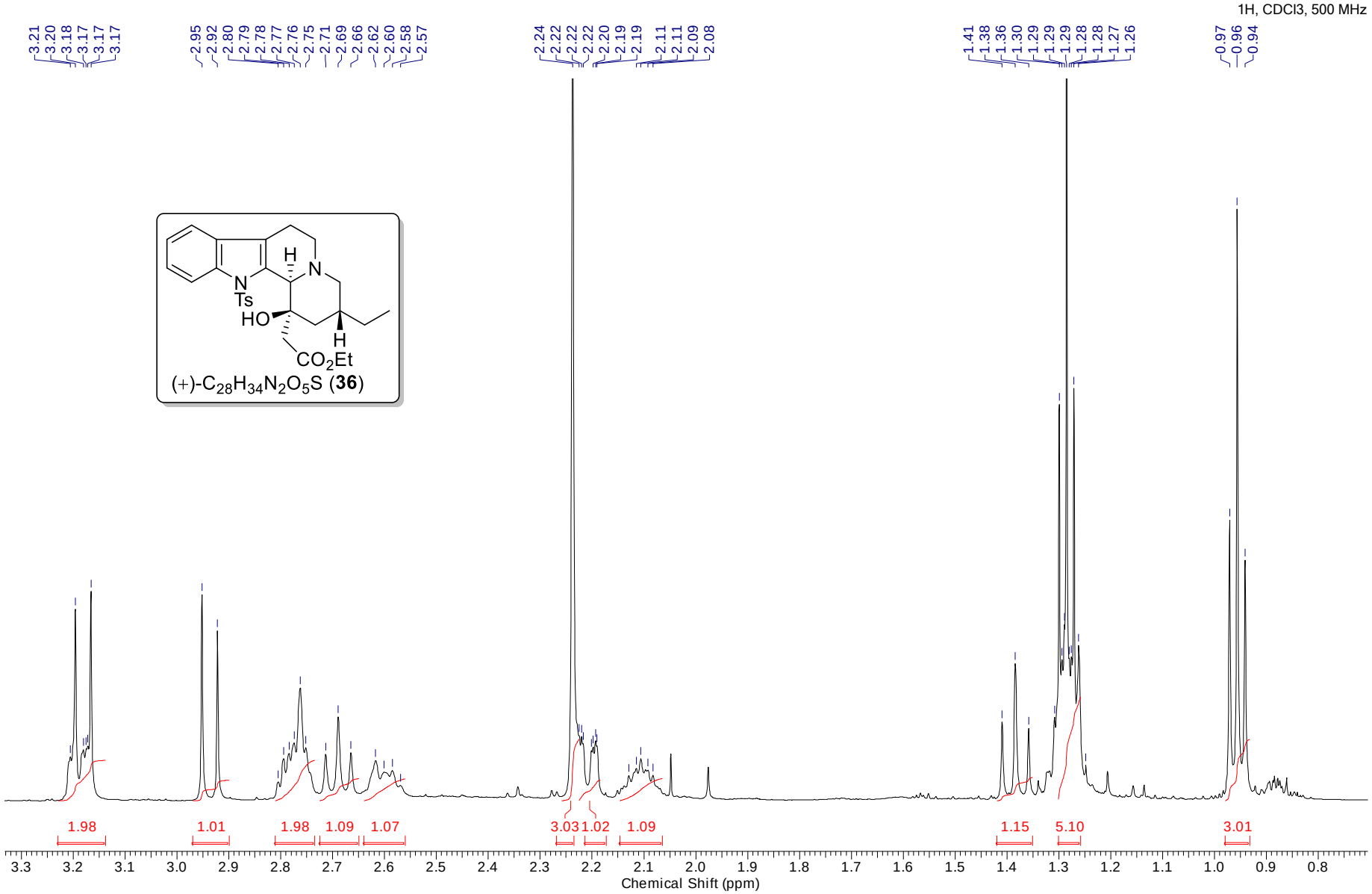

1H, CDCl3, 500 MHz
CHLOROFORM-d
7.80 7.79 7.78 7.77 7.75 7.38 7.38 7.27 7.22 7.20 7.19 7.18
5.12
3.25 3.23 3.14 3.12 3.11 2.91 2.78 2.77 2.76 2.66 2.64 2.64 2.44 2.42 2.34 1.49 1.48 1.46 1.45 1.43 1.41 1.40 1.39 0.98 0.96 0.95
(+)-C24H26N2O3S (35)
3.00 1.02 4.00
Chemical Shift (ppm)
3.00 1.02 4.00 1.00 1.02 1.01 1.03 1.03 2.05 1.00 1.05 1.08 3.04 1.00 1.05 3.00
Chemical Shift (ppm)

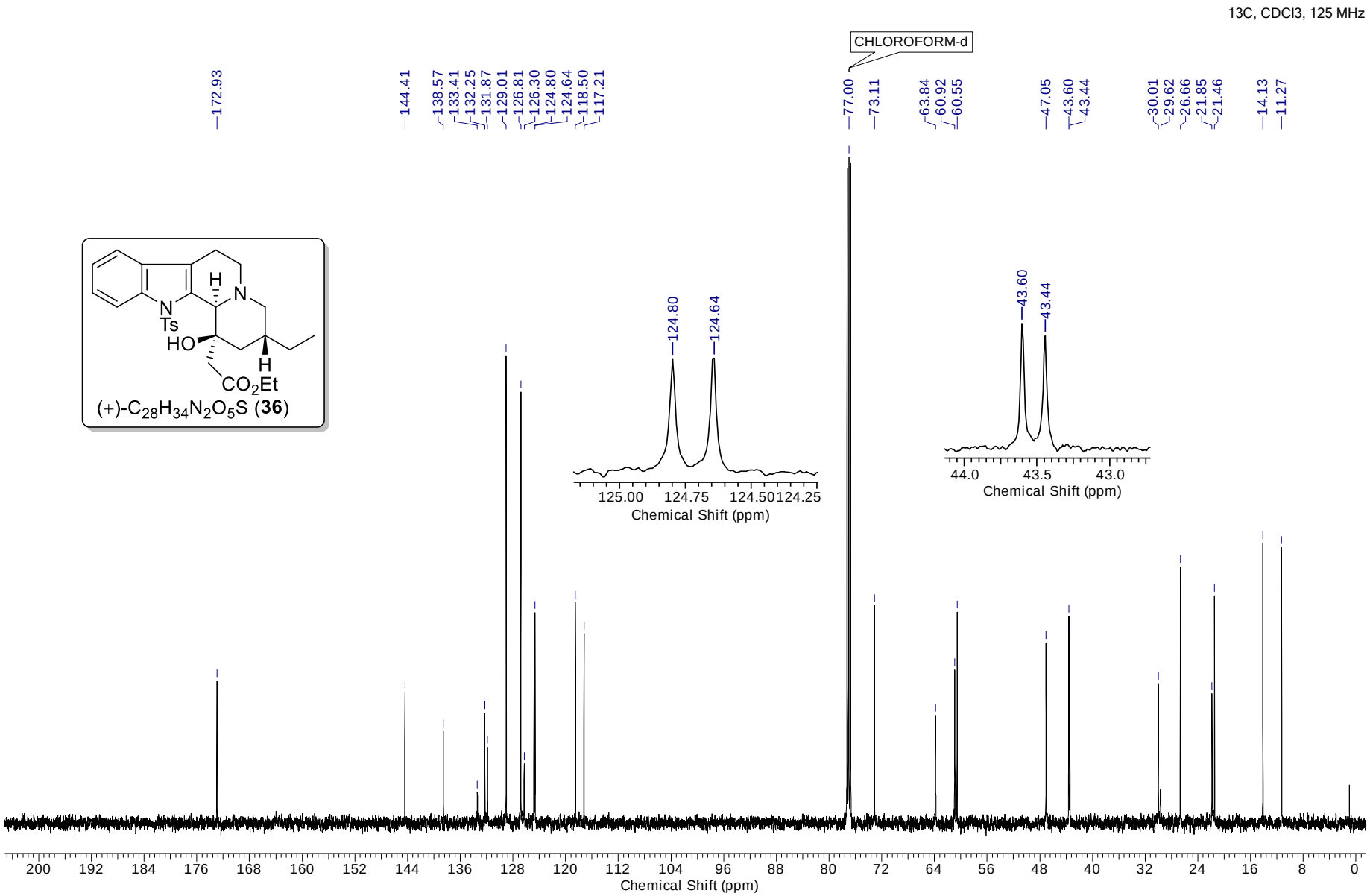

1H, CDCl3, 500 MHz
3.25
3.25
3.23
3.22
3.14
3.12
3.11
3.09
3.04
3.03
3.03
3.02
3.02
3.01
2.91
2.90
2.89
2.79
2.79
2.78
2.77
2.76
2.66
2.65
2.64
2.64
2.63
2.47
2.44
2.42
2.34
1.51
1.49
1.48
1.46
1.45
1.43
1.41
1.40
1.39
1.37
0.98
0.96
0.95
H
N
N
Ts
O
H
(+)-C24H26N2O3S (35)
1.02
1.01
1.03
1.03
2.05
1.00
1.05
1.08
3.04
1.00
1.05
3.00
3.3
3.2
3.1
3.0
2.9
2.8
2.7
2.6
2.5
2.4
2.3
2.2
2.1
2.0
1.9
1.8
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1.0
0.9
0.8
Chemical Shift (ppm)

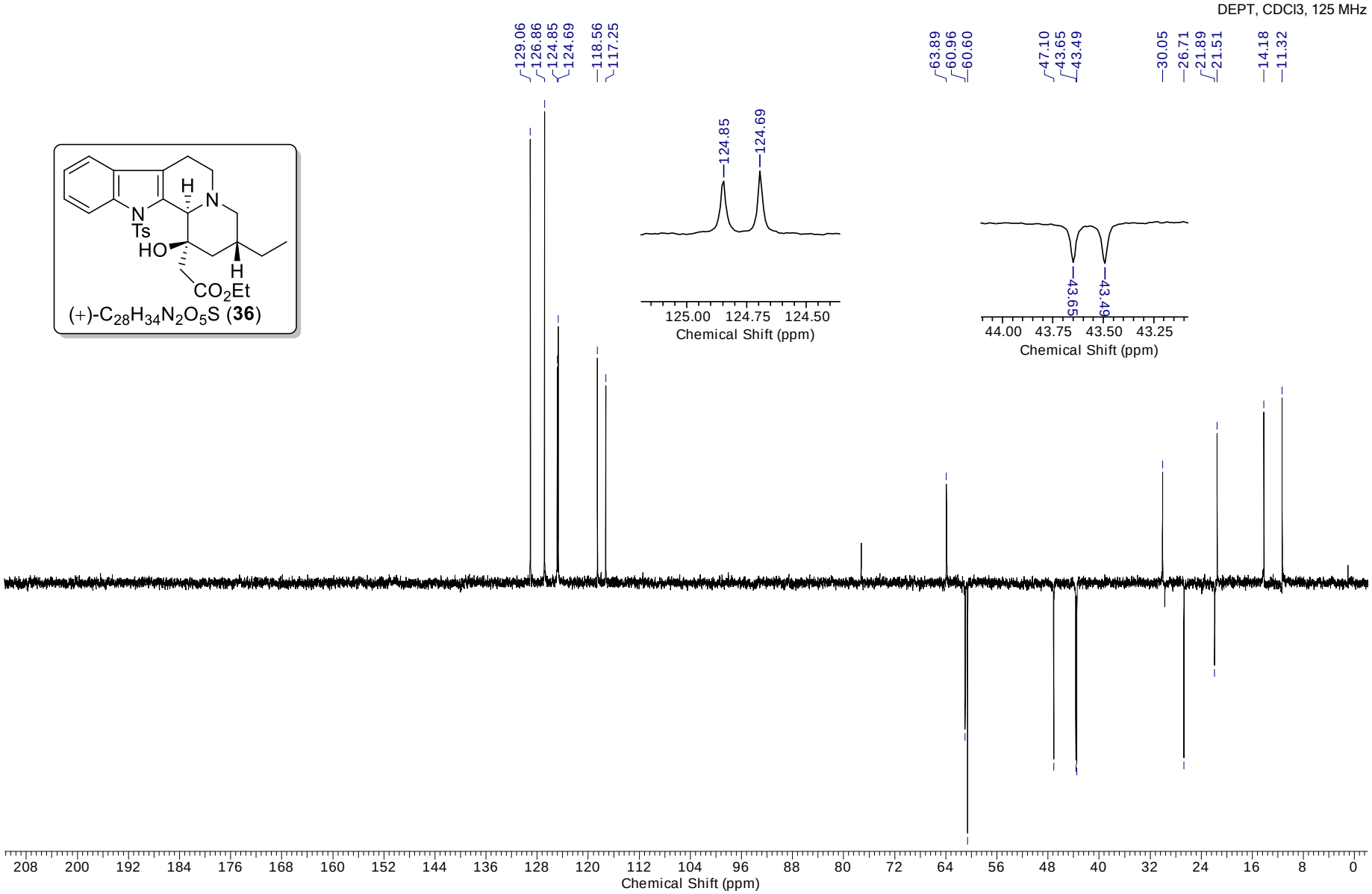

13C, CDCl3, 125 MHz
CHLOROFORM-d
206.46
144.59
136.11
135.87
130.82
129.67
129.29
126.98
124.31
123.07
118.60
117.86
113.99
77.25
77.00
76.74
65.87
60.16
46.86
46.70
41.74
27.04
22.10
21.53
11.14
(+)-C24H26N2O3S (35)
136.11
135.87
136.25
135.75
Chemical Shift (ppm)
46.86
46.70
47.0
46.5
46.0
Chemical Shift (ppm)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30
Chemical Shift (ppm)

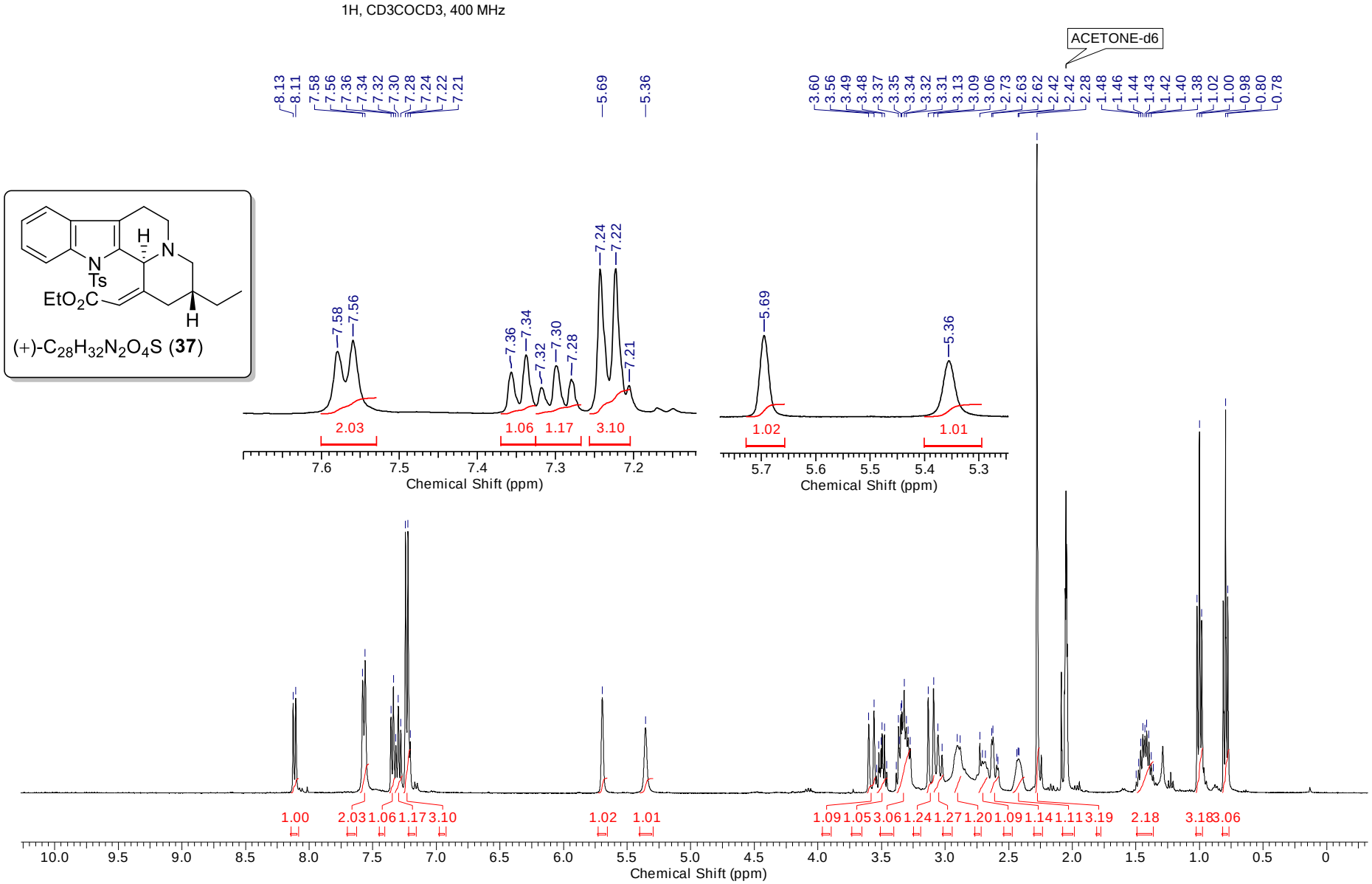

DEPT, CDCl3, 125 MHz
129.68
126.99
124.31
123.08
118.61
113.99
65.87
60.16
46.87
46.70
41.75
27.04
22.11
21.53
11.14
H
N
N
Ts
O
H
(+)-C24H26N2O3S (35)
46.87
46.70
47.5
47.0
46.5
46.0
Chemical Shift (ppm)
208
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

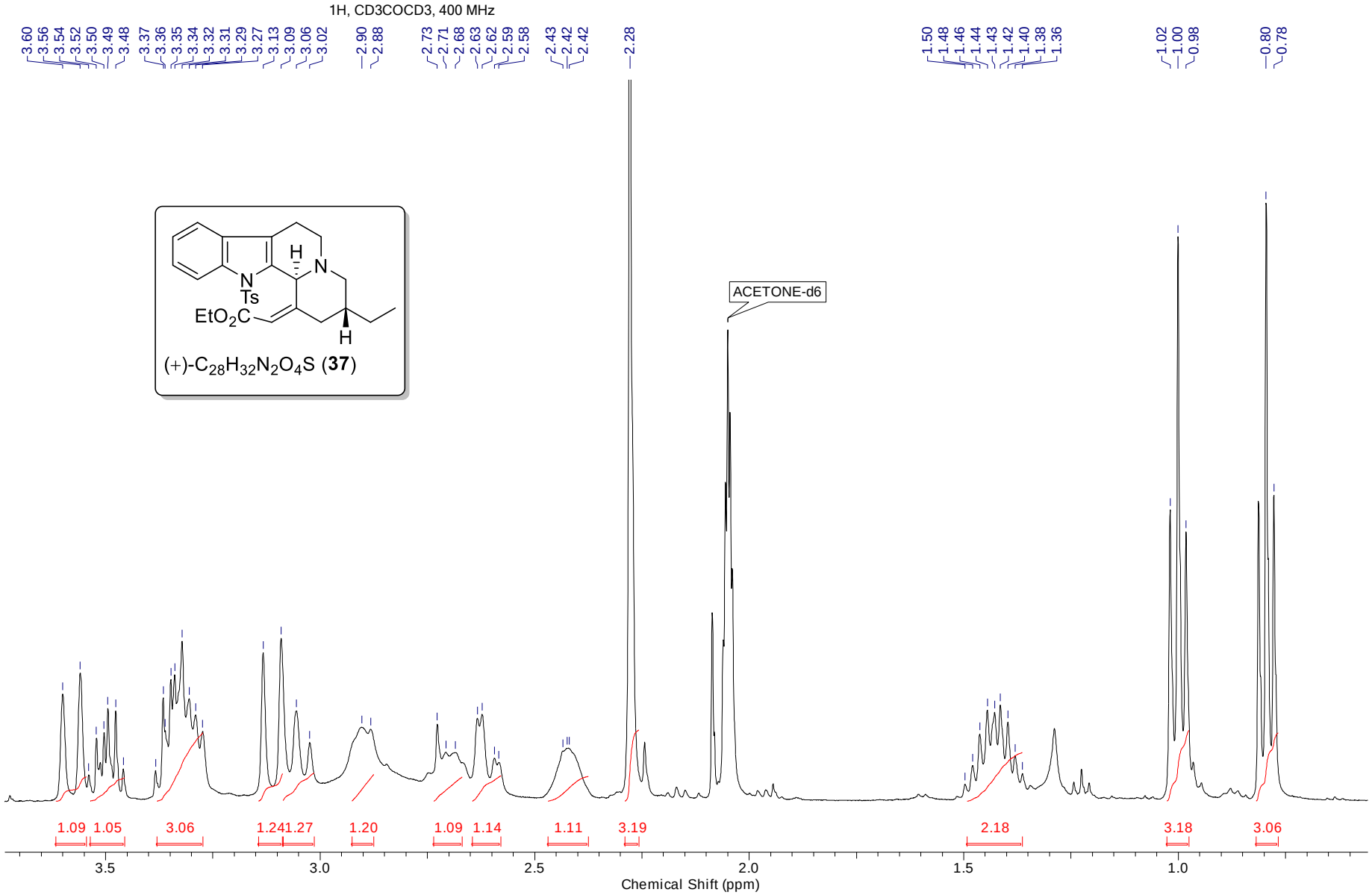

1H, CDCl3, 500 MHz
CHLOROFORM-d
8.08 8.06 7.35 7.34 7.27 7.26 7.24 7.23 7.21 7.19 7.03 7.01
4.36 4.19 4.17 4.15 4.14 4.12 4.11 4.10 3.72 3.21 3.20 3.18 3.17 3.17 2.95 2.92 2.77 2.76 2.69 2.24 2.22 2.22 2.20 2.19 1.38 1.30 1.29 1.29 1.28 1.28 1.27 1.26 0.97 0.96 0.94
0.95 1.92 0.23 0.53 1.24 1.02 1.93
1.00 0.79 2.12 0.98 1.98 1.01 1.98 1.09 1.07 3.03 1.02 1.09 1.15 5.10 3.01
Chemical Shift (ppm)
CHLOROFORM-d
7.35 7.34 7.28 7.27 7.27 7.26 7.26 7.24 7.23 7.21 7.21 7.19 7.18 7.18
1.92 0.23 0.53 1.24 1.02
Chemical Shift (ppm)
4.36 4.28 4.20 4.19 4.17 4.15 4.12 4.11 4.10 4.09 4.07 3.76 3.75 3.74 3.72 3.72
1.00 0.79 2.12 0.98
Chemical Shift (ppm)
(+)-C28H34N2O5S (36)

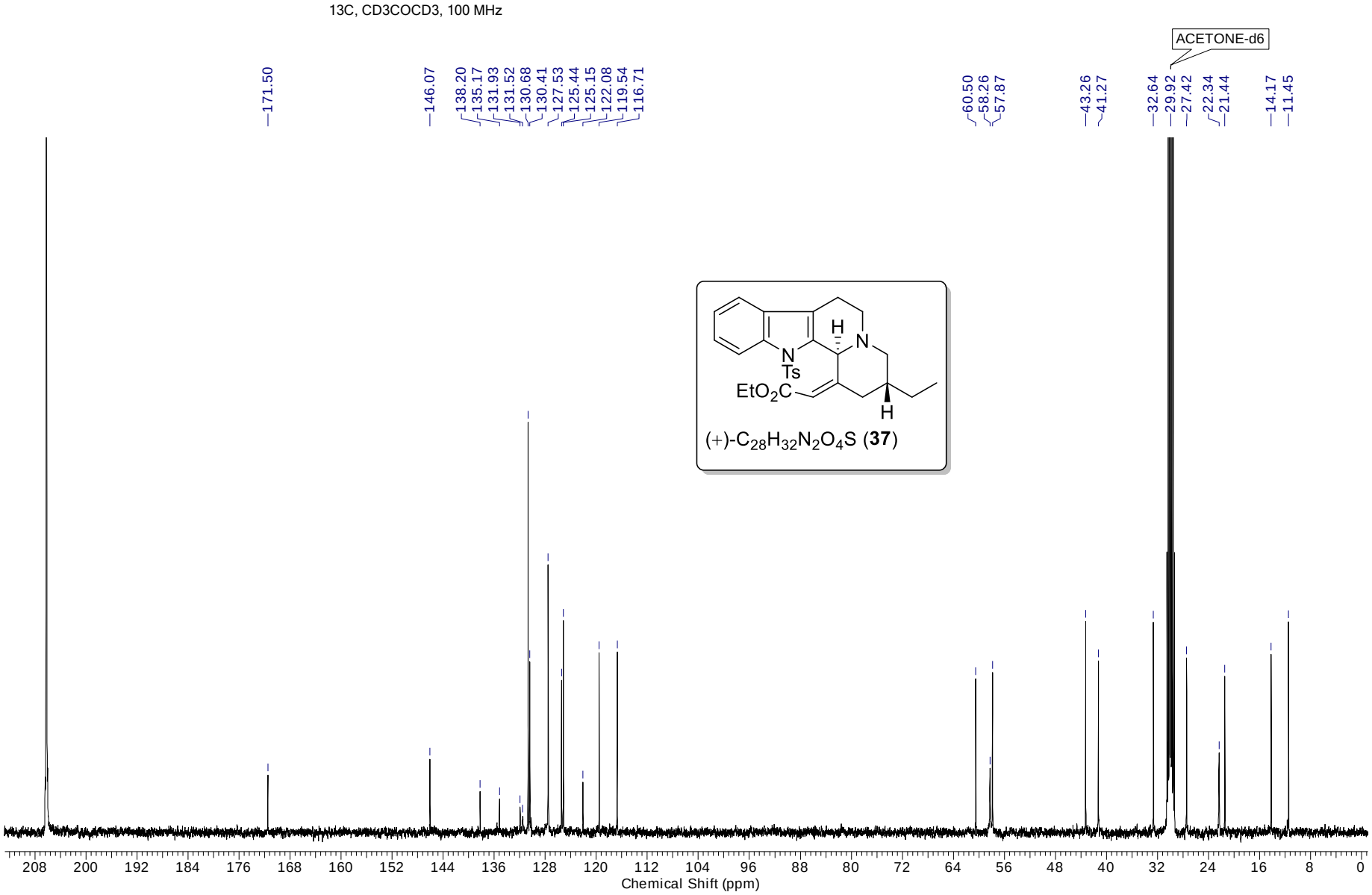

1H, CDCl3, 500 MHz
3.21 3.20 3.18 3.17 3.17 3.17
2.95 2.92 2.80 2.79 2.78 2.77 2.76 2.75 2.71 2.69 2.66 2.62 2.60 2.58 2.57
2.24 2.22 2.22 2.22 2.20 2.19 2.19 2.11 2.11 2.09 2.08
1.41 1.38 1.36 1.30 1.29 1.29 1.29 1.28 1.28 1.27 1.26
0.97 0.96 0.94
H
N
N
Ts
HO
H
CO2Et
(+)-C28H34N2O5S (36)
1.98 1.01 1.98 1.09 1.07 3.03 1.02 1.09 1.15 5.10 3.01
3.3 3.2 3.1 3.0 2.9 2.8 2.7 2.6 2.5 2.4 2.3 2.2 2.1 2.0 1.9 1.8 1.7 1.6 1.5 1.4 1.3 1.2 1.1 1.0 0.9 0.8
Chemical Shift (ppm)

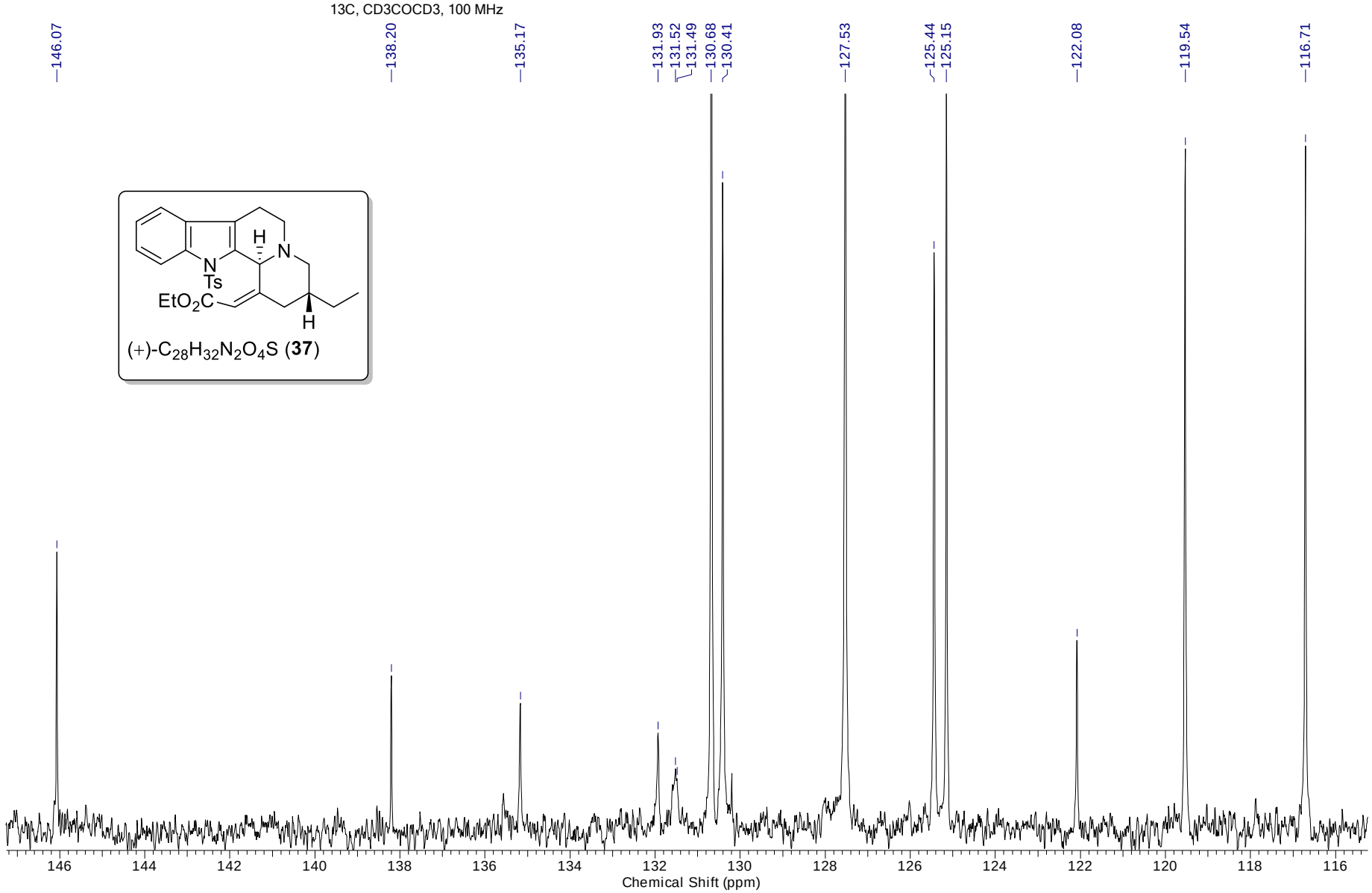

13C, CDCl3, 125 MHz
CHLOROFORM-d
172.93
144.41
138.57
133.41
132.25
131.87
129.01
126.81
126.30
124.80
124.64
118.50
117.21
77.00
73.11
63.84
60.92
60.55
47.05
43.60
43.44
30.01
29.62
26.66
21.85
21.46
14.13
11.27
(+)-C28H34N2O5S (36)
Chemical Shift (ppm)

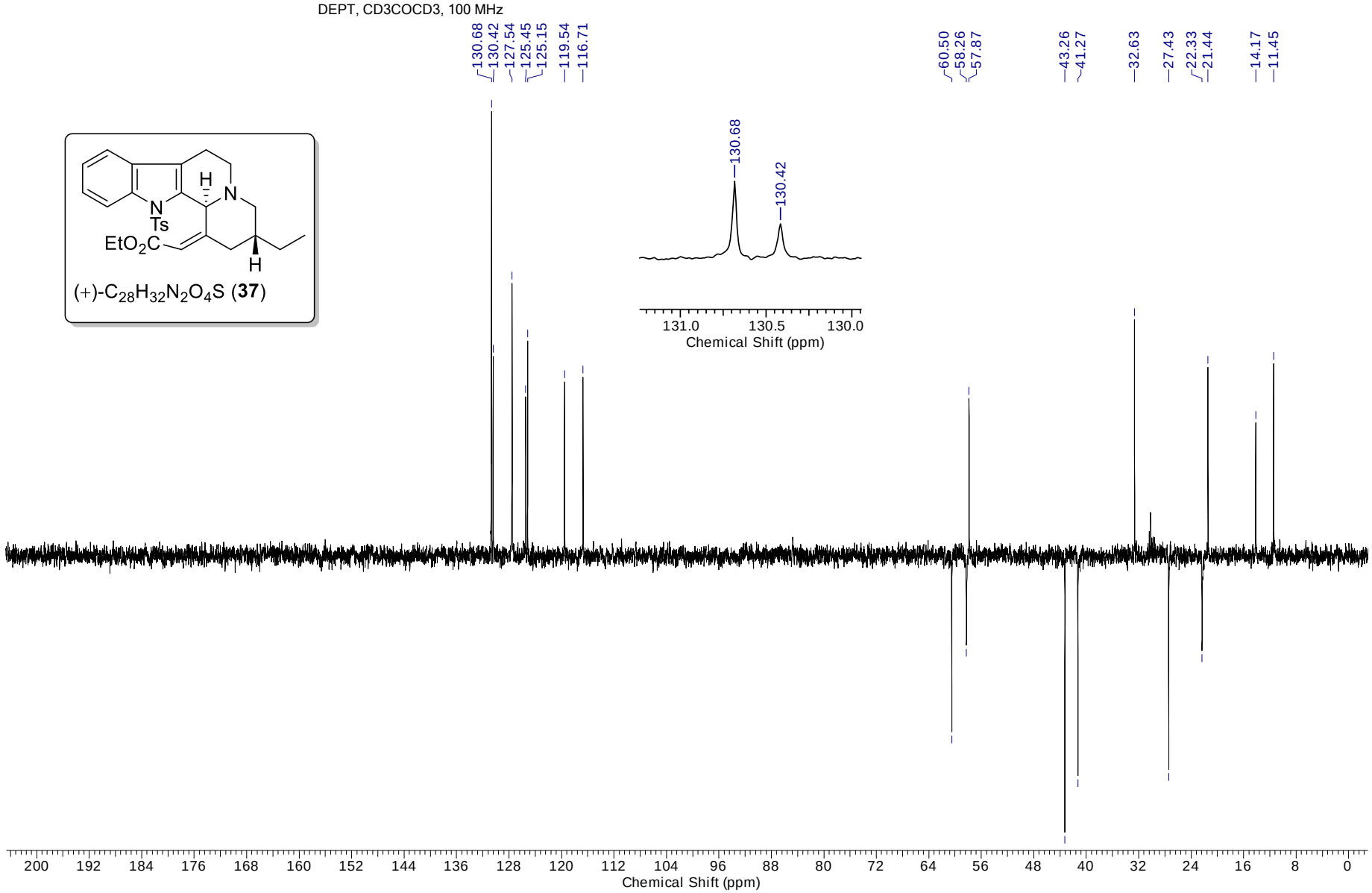

DEPT, CDCl3, 125 MHz
129.06
126.86
124.85
124.69
118.56
117.25
63.89
60.96
60.60
47.10
43.65
43.49
30.05
26.71
21.89
21.51
14.18
11.32
124.85
124.69
125.00 124.75 124.50
Chemical Shift (ppm)
43.65
43.49
44.00 43.75 43.50 43.25
Chemical Shift (ppm)
(+)-C28H34N2O5S (36)
208 200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

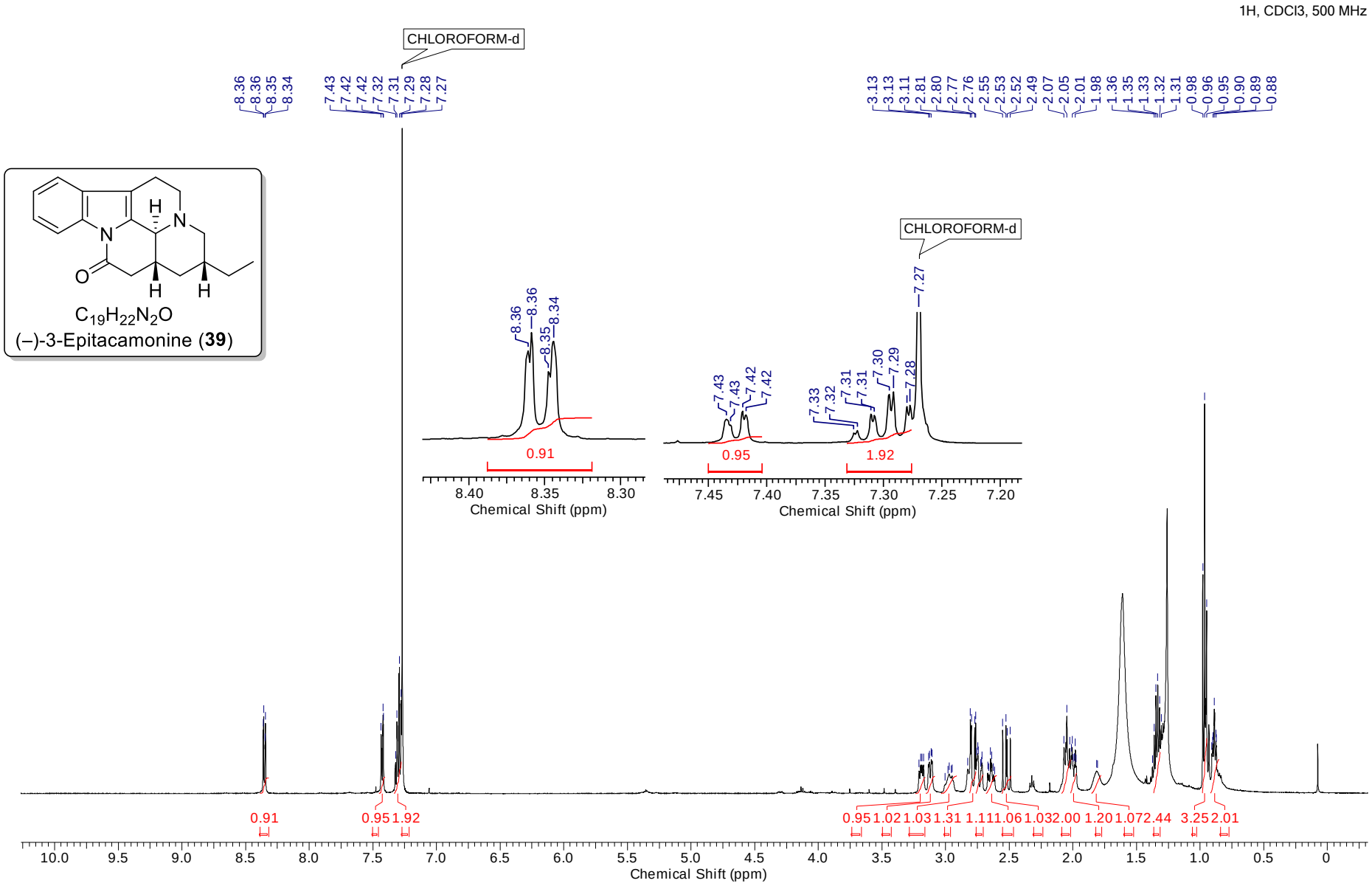

1H, CD3COCD3, 400 MHz
ACETONE-d6
(+)-C28H32N2O4S (37)
Chemical Shift (ppm)
Chemical Shift (ppm)
Chemical Shift (ppm)

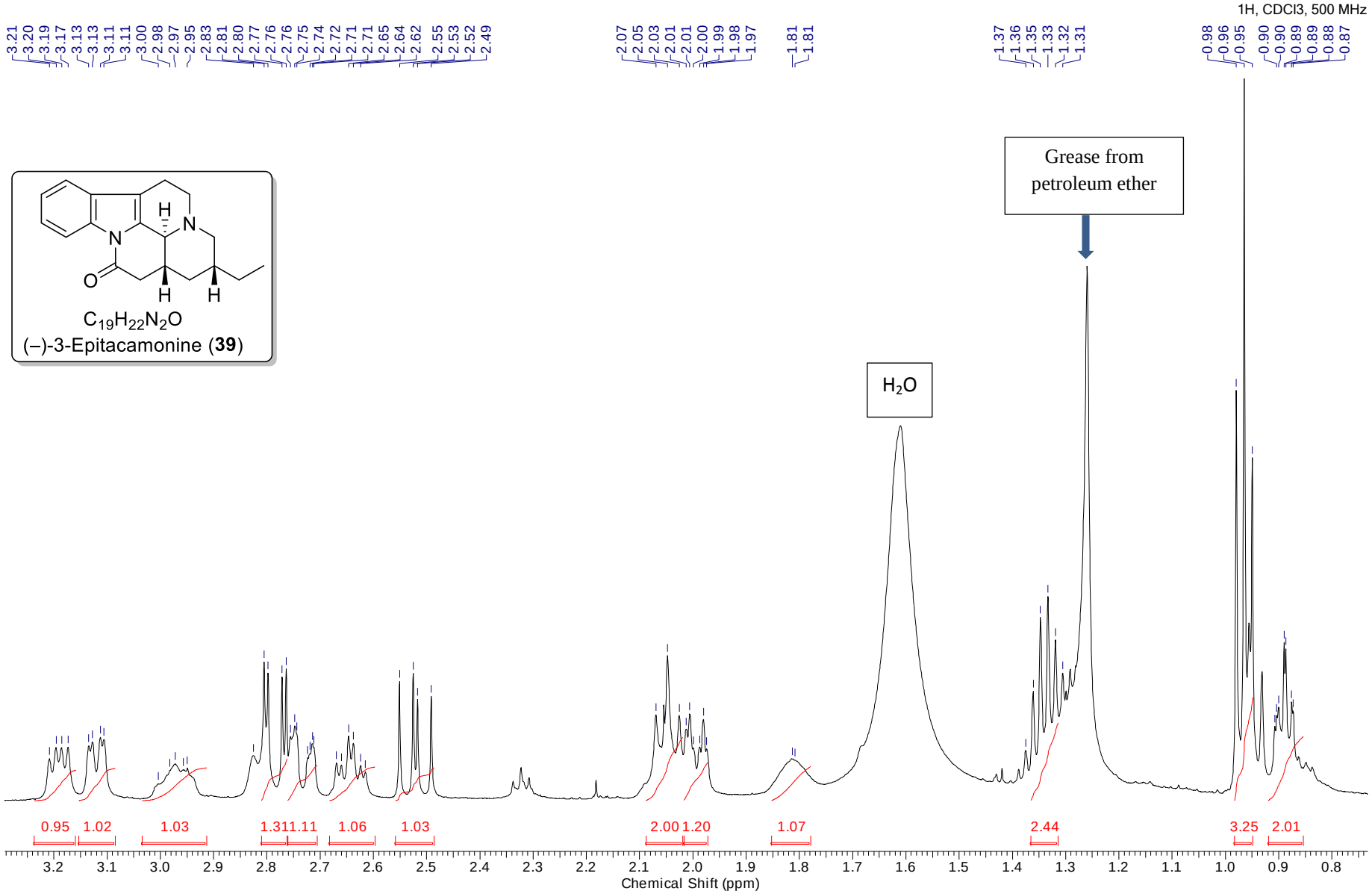

1H, CD3COCD3, 400 MHz
3.60 3.56 3.54 3.52 3.50 3.49 3.48 3.37 3.36 3.35 3.34 3.32 3.31 3.29 3.27 3.13 3.09 3.06 3.02 2.90 2.88 2.73 2.71 2.68 2.63 2.62 2.59 2.58 2.43 2.42 2.42 2.28 1.50 1.48 1.46 1.44 1.43 1.42 1.40 1.38 1.36 1.02 1.00 0.98 0.80 0.78
ACETONE-d6
(+)-C28H32N2O4S (37)
1.09 1.05 3.06 1.24 1.27 1.20 1.09 1.14 1.11 3.19 2.18 3.18 3.06
3.5 3.0 2.5 2.0 1.5 1.0
Chemical Shift (ppm)

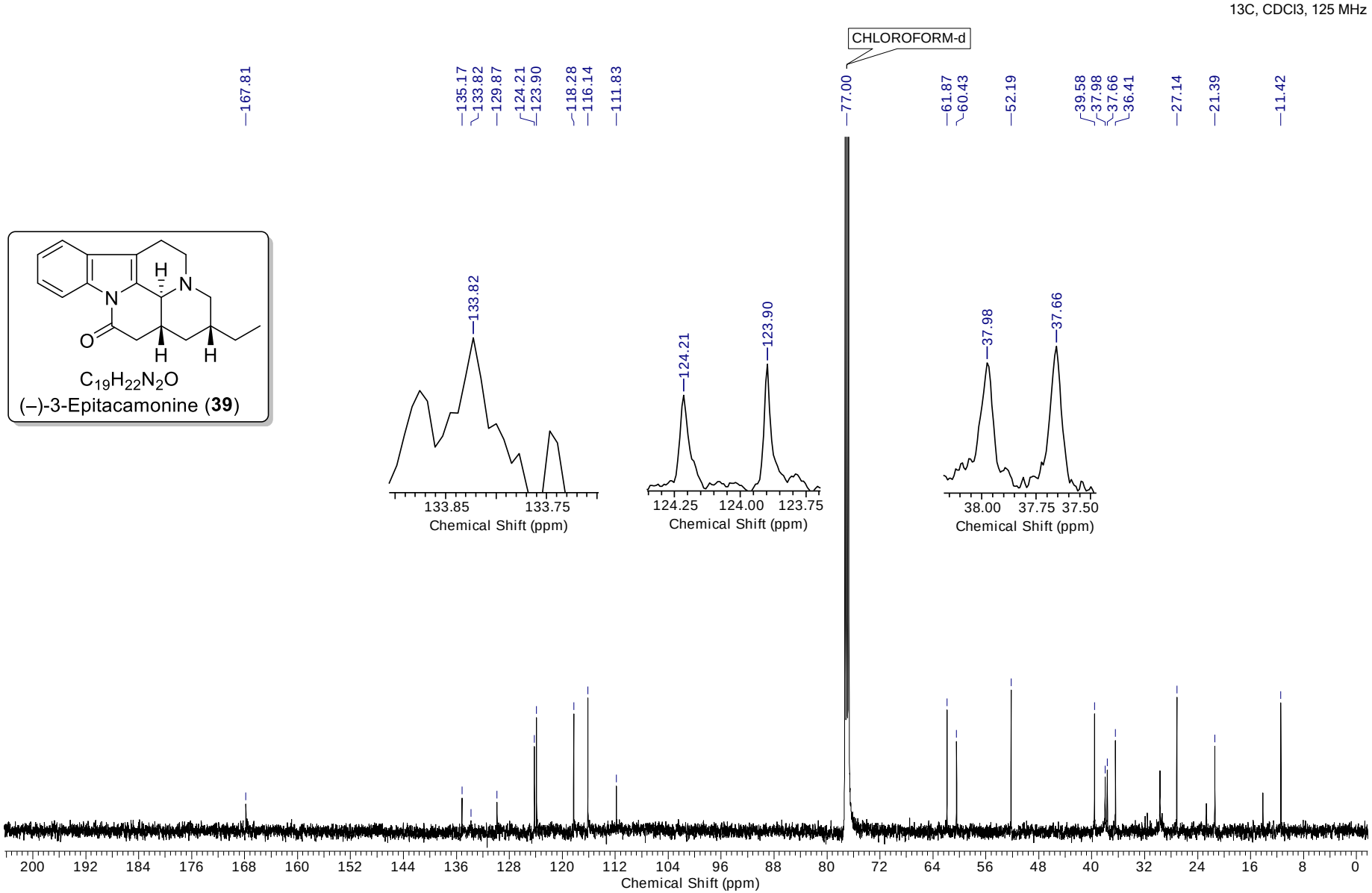

13C, CD3COCD3, 100 MHz
ACETONE-d6
171.50
146.07
138.20
135.17
131.93
131.52
130.68
130.41
127.53
125.44
125.15
122.08
119.54
116.71
60.50
58.26
57.87
43.26
41.27
32.64
29.92
27.42
22.34
21.44
14.17
11.45
H
N
N
Ts
EtO2C
H
(+)-C28H32N2O4S (37)
208 200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

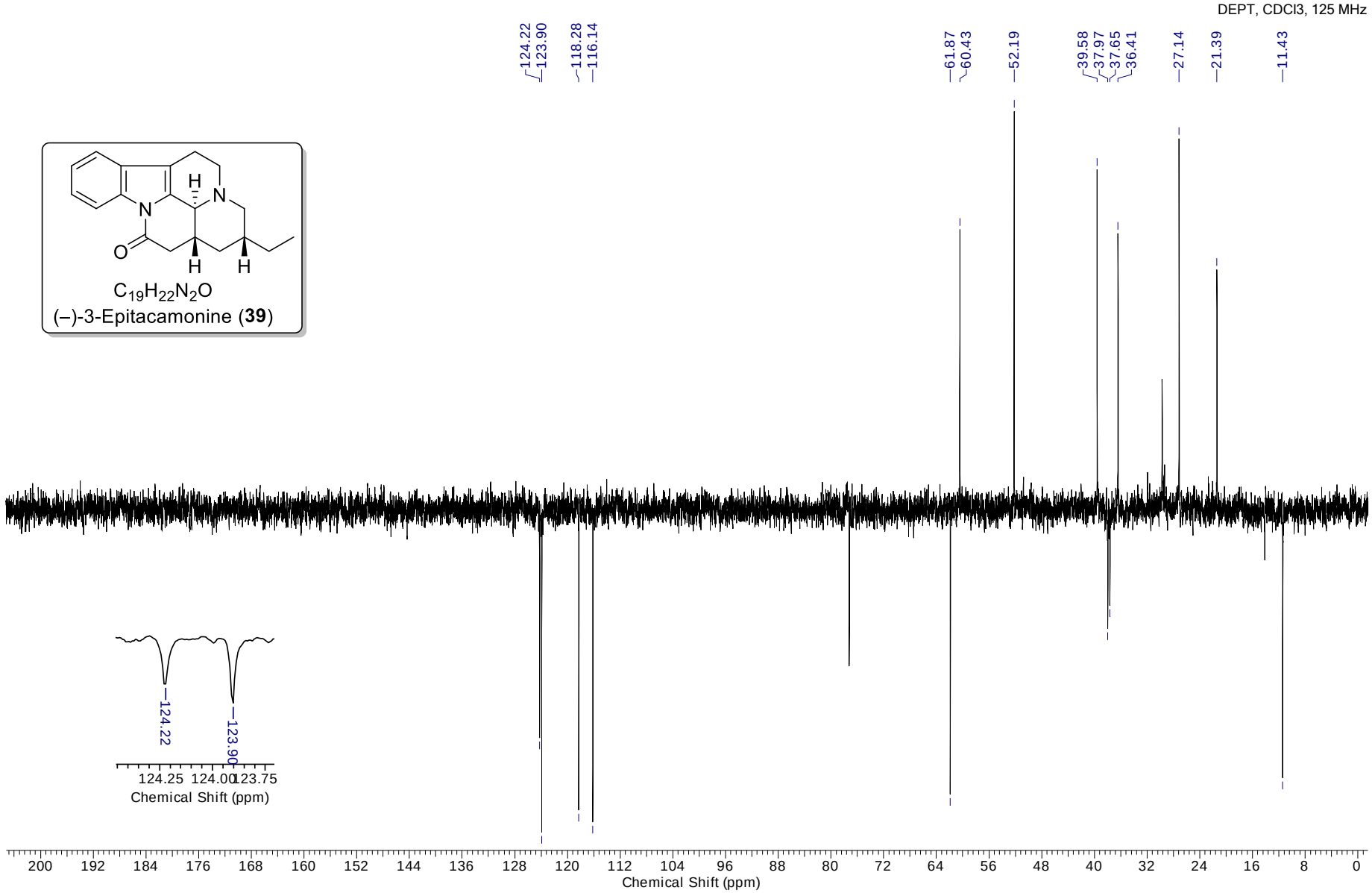

13C, CD3COCD3, 100 MHz
146.07
138.20
135.17
131.93
131.52
131.49
130.68
130.41
127.53
125.44
125.15
122.08
119.54
116.71
(+)-C28H32N2O4S (37)
146
144
142
140
138
136
134
132
130
128
126
124
122
120
118
116
Chemical Shift (ppm)


DEPT, CD3COCD3, 100 MHz
130.68
130.42
127.54
125.45
125.15
119.54
116.71
60.50
58.26
57.87
43.26
41.27
32.63
27.43
22.33
21.44
14.17
11.45
(+)-C28H32N2O4S (37)
Chemical Shift (ppm)
Chemical Shift (ppm)

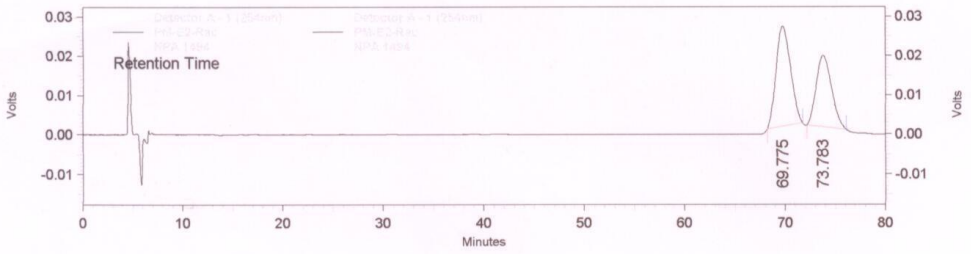

1H, CDCl3, 500 MHz
CHLOROFORM-d
8.36 8.36 8.35 8.34
7.43 7.42 7.42 7.32 7.31 7.29 7.28 7.27
3.13 3.13 3.11 2.81 2.80 2.77 2.76 2.55 2.53 2.52 2.49 2.07 2.05 2.01 1.98 1.36 1.35 1.33 1.32 1.31 0.98 0.96 0.95 0.90 0.89 0.88
C19H22N2O
(–)-3-Epitacamonine (39)
0.91
0.95 1.92
0.95 1.02 1.03 1.31 1.11 1.06 1.03 2.00 1.20 1.07 2.44 3.25 2.01
Chemical Shift (ppm)


1H, CDCl3, 500 MHz
3.21 3.20 3.19 3.17 3.13 3.13 3.11 3.11 3.00 2.98 2.97 2.95 2.83 2.81 2.80 2.77 2.76 2.76 2.75 2.74 2.72 2.71 2.71 2.65 2.64 2.62 2.55 2.53 2.52 2.49
2.07 2.05 2.03 2.01 2.01 2.00 1.99 1.98 1.97
1.81 1.81
1.37 1.36 1.35 1.33 1.32 1.31
0.98 0.96 0.95 0.90 0.90 0.89 0.89 0.88 0.87
Grease from petroleum ether
H2O
C19H22N2O
(–)-3-Epitacamonine (39)
0.95 1.02 1.03 1.31 1.11 1.06 1.03 2.00 1.20 1.07 2.44 3.25 2.01
Chemical Shift (ppm)


13C, CDCl3, 125 MHz
CHLOROFORM-d
167.81
135.17
133.82
129.87
124.21
123.90
118.28
116.14
111.83
77.00
61.87
60.43
52.19
39.58
37.98
37.66
36.41
27.14
21.39
11.42
C19H22N2O
(–)-3-Epitacamonine (39)
133.82
133.85
133.75
Chemical Shift (ppm)
124.21
123.90
124.25
124.00
123.75
Chemical Shift (ppm)
37.98
37.66
38.00
37.75
37.50
Chemical Shift (ppm)
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)


DEPT, CDCl3, 125 MHz
124.22
123.90
118.28
116.14
61.87
60.43
52.19
39.58
37.97
37.65
36.41
27.14
21.39
11.43
H
N
N
O
H
H
C19H22N2O
(–)-3-Epitacamonine (39)
124.22
123.90
124.25 124.00 123.75
Chemical Shift (ppm)
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

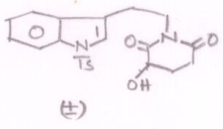
Shimadzu CLASS-VP V6.12 SP5
Method Name: C:\CLASS-VP\Data\Dr. CHAVAN S. P\PAPAL FH10 % IPAPE
Data Name: C:\CLASS-VP\Data\Dr. Argade\NPA 1494
User: System
Acquired: 3/19/14 6:14:11 PM
Printed: 3/19/14 7:36:07 PM
Sample Name PM-E2-Rac



Detector A - 1 (254nm)

Retention Time	C Area	Area %
69.775	2558321	56.764
73.783	1948621	43.236
Totals	4506942	100.000

Project Leader : Dr. N. P. Argade
Column : Kromasil 5-CelluCoat (250x4.6mm)
Mobile Phase : EtOH: n-Hexane: TFA (08:92:0.1)
Wavelength : 254 nm
Flow Rate : 0.7 ml/min
Conc. : 1.0 mg/ml
Inj vol- : 5ul

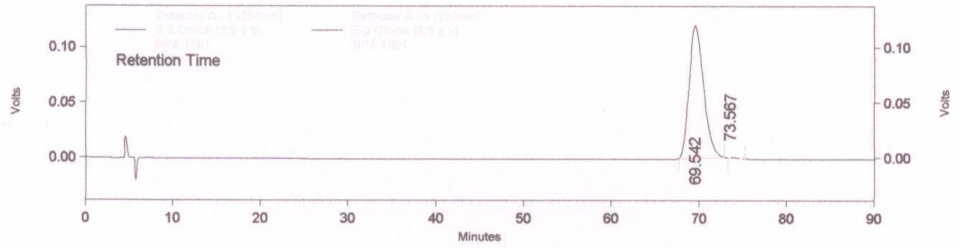


(±)

Data

HPLC profile of (–)-4

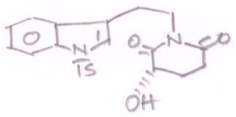
Shimadzu CLASS-VP V6.12 SP5
Method Name: C:\CLASS-VP\Data\Dr. CHAVAN S. P\PAPAL FH10 % IPAPE
Data Name: C:\CLASS-VP\Data\Dr. Argade\NPA 1501
User: System
Acquired: 3/26/14 5:18:54 PM
Printed: 3/26/14 7:10:20 PM
Sample Name E-2-Chiral (3.9 g s)



Detector A - 1 (254nm)

Retention Time	C Area	Area %
69.542	13780058	99.836
73.567	22598	0.164
Totals	13802656	100.000

Project Leader : Dr.N. P. Argade
Column :Kromasil 5-CelluCoat (250 X 4.6)
Mobile Phase :*EtOH: n-Hexane: TFA (08:92:0.1)*
Wavelength : 254 nm
Flow Rate : 0.7ml/min
Conc. : 1 mg/ml
Inj vol- : 5ul



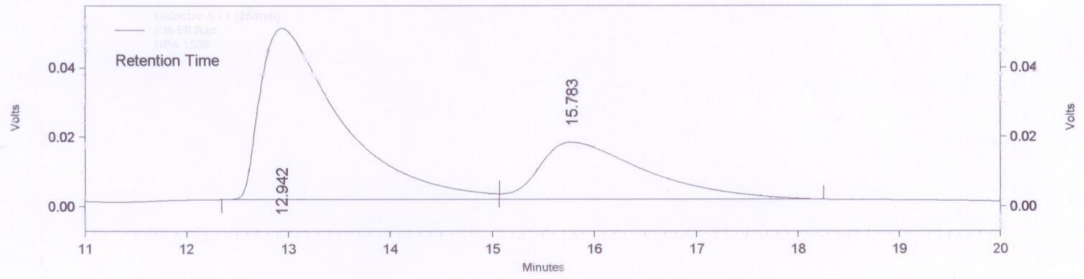
Data

HPLC profile of rac-12

Area % Report **Page 1 of 1**

PM-E8 Rac

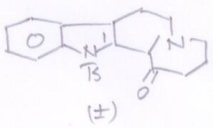
Method Name: C:\CLASS-VP\Data\Dr. Patil N. T\VSS
Data Name: C:\CLASS-VP\Data\Dr. Argade\NPA 1588
User: System
Acquired: 2/5/15 11:51:16 AM
Printed: 2/5/15 12:22:10 PM



Detector A - 1 (254nm)

Pk #	Retention Time	Area	Area %
1	12.942	2724322	69.607
2	15.783	1189532	30.393
Totals		3913854	100.000

Project Leader :Dr.N.P.Argade
Column Chiralcel OD-H (250mm x 4.6mm)
Mobile Phase :ETOH:Petether:TFA (20: 80:0.1)
Wavelength : 254 nm
Flow Rate :1.0ml/min
Conc. : 1mg/1ml
Inj vol- : 2ul



HPLC profile of (+)-12

Area % Report **Page 1 of 1**

PM-E8 Chiral

Method Name: C:\CLASS-VP\Data\Dr. Patil N. T\VSS
Data Name: C:\CLASS-VP\Data\Dr. Argade\NPA 1587
User: System
Acquired: 2/5/15 11:20:57 AM
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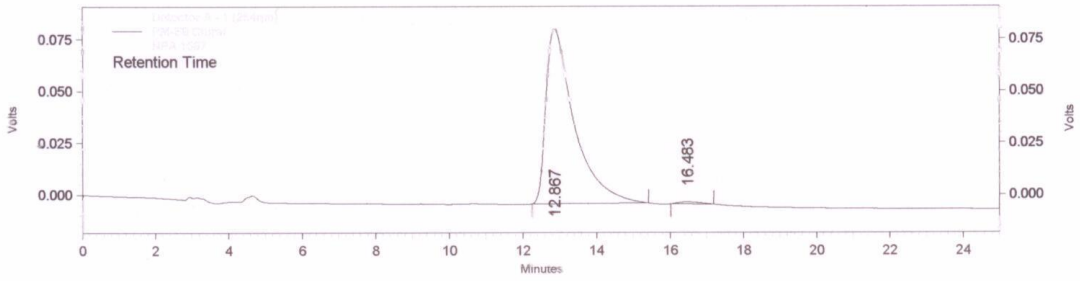
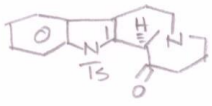


Detector A - 1 (254nm)

Pk #	Retention Time	Area	Area %
1	12.867	4541308	99.086
2	16.483	41881	0.914
Totals		4583189	100.000

Project Leader :Dr.N.P.Argade
Column Chiralcel OD-H (250mm x 4.6mm)
Mobile Phase :ETOH:Petether:TFA (20: 80:0.1)
Wavelength : 254 nm
Flow Rate :1.0ml/min
Conc. : 1mg/1ml
Inj vol- : 2ul



98/99.

X-ray Crystal Structure Analysis For (±)-13, (+)-14 and (+)-34

Experimental and Refinement Details for (±)-13,(+)-14 and (+)-34:

Colorless block like crystals of (±)-**13** was placed in 0.7 mm diameter nylon CryoLoops (Hampton Research) with Paraton-N (Hampton Research). The loop was mounted on a Super Nova Dual source X-ray Diffractometer system (Agilent Technologies) equipped with a CCD area detector and operated at 250 W power (50 kV, 0.8 mA) to generate Mo Kα radiation (λ = 0.71073 Å) and Cu Kα radiation (λ = 1.54178 Å) at 298 K. Initial scans of each specimen were performed to obtain preliminary unit cell parameters and to assess the mosaicity (breadth of spots between frames) of the crystal to select the required frame width for data collection. CrysAlis^{Pro} program software was used suite to carry out overlapping φ and ω scans at detector (2θ) settings (2θ = 28). Following data collection, reflections were sampled from all regions of the Ewald sphere to redetermine unit cell parameters for data integration. Data were integrated using CrysAlis^{Pro} software with a narrow frame algorithm. Data were subsequently corrected for absorption by the program SCALE3 ABSPACK[1] scaling algorithm. All non-hydrogen atoms were refined anisotropically with hydrogen atoms generated as spheres riding the coordinates of their parent atoms. The structure was refined using the SHELXTL 97[2] software suite.

Experimental and refinement details for (+)-**14** and (+)-**34** were done by using the above mentioned procedure.

References:

1. CrysAlisPro, Version 1.171.33.66; Oxford Diffraction Ltd.: Abingdon, U.K., 2010.

2. G. M. Sheldrick, (1997). SHELXS ‘97 and SHELXL ‘97. University of Göttingen, Germany.

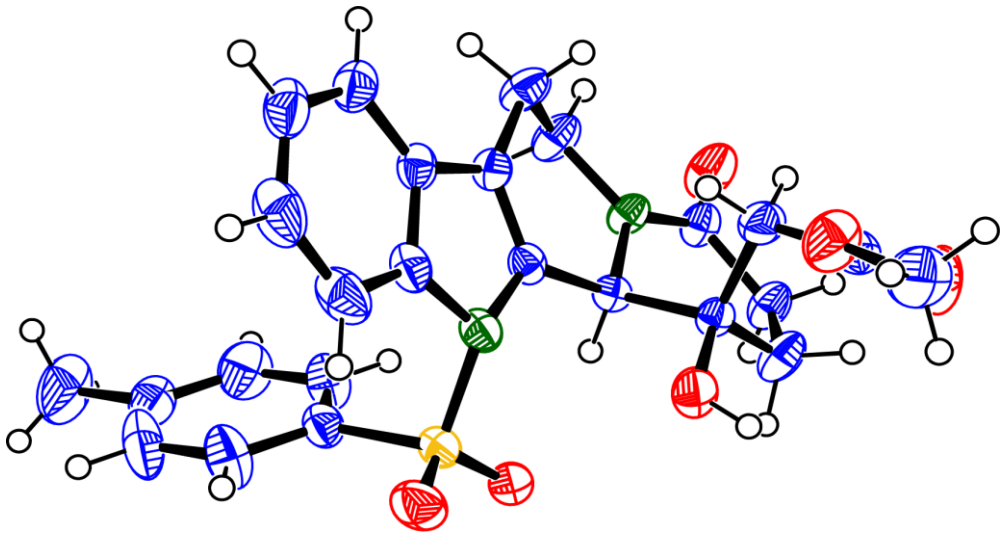


Figure S2: Ortep Drawing of Compound (±)-**13**. Thermal ellipsoids set to 50% probability level.

Table 1: Crystal data and structure refinement for Compound (±)-**13**

CCDC 1424170

Identification code	Compound (±)-**13**
Empirical formula	$C_{25}H_{26}N_2O_6S$
Formula weight	482.54
Temperature/K	298
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	7.4819(3)
b/Å	12.1332(5)
c/Å	25.2260(8)
α/°	90.00
β/°	90.00
γ/°	90.00
Volume/Å^3	2290.00(15)
Z	4
ρ_{calc}mg/mm^3	1.400
m/mm^{-1}	0.187
F(000)	1016.0
Crystal size/mm^3	0.31 × 0.19 × 0.15
2Θ range for data collection	5.9 to 58.16°
Index ranges	$-10 \leq h \leq 5$, $-16 \leq k \leq 16$, $-32 \leq l \leq 33$
Reflections collected	7533
Independent reflections	4489[R(int) = 0.0251]

Data/restraints/parameters	4489/0/310
Goodness-of-fit on F^2	1.074
Final R indexes [I>=2σ (I)]	R_1 = 0.0447, wR_2 = 0.1018
Final R indexes [all data]	R_1 = 0.0538, wR_2 = 0.1063
Largest diff. peak/hole / e $Å^{-3}$	0.26/-0.22
Flack parameter	0.00(10)

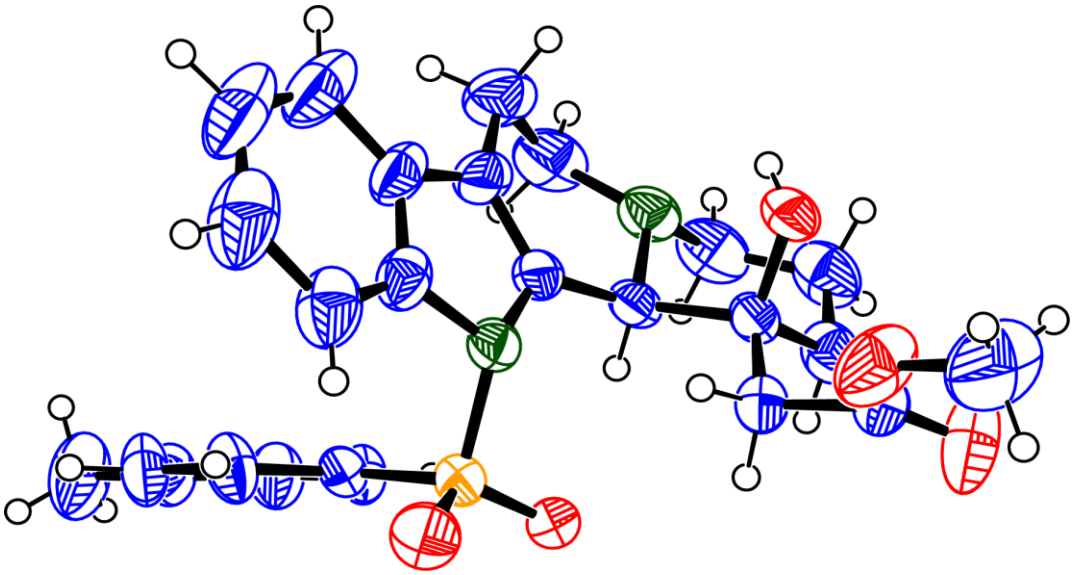


Figure S3: Ortep Drawing of Compound (+)-**14**. Thermal ellipsoids set to 50% probability level.

Table 2: Crystal data and structure refinement for Compound (+)-**14**

CCDC 1424171

Identification code	Compound (+)-**14**
Empirical formula	C_{25} H_{28} N_2 O_5 S
Formula weight	468.55
Temperature/K	298
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	9.0934(4)
b/Å	10.9932(4)
c/Å	25.0614(14)
α/°	90.00
β/°	90.00
γ/°	90.00

Volume/Å^3	2505.3(2)
Z	4
ρ_{calc}mg/mm^3	1.242
m/mm^{-1}	0.166
F(000)	992.0
Crystal size/mm^3	0.32 × 0.18 × 0.13
2Θ range for data collection	5.9 to 58.16°
Index ranges	$-12 \leq h \leq 7$, $-8 \leq k \leq 15$, $-18 \leq l \leq 34$
Reflections collected	8665
Independent reflections	5410 [R(int) = 0.0423]
Data/restraints/parameters	5410/0/ 301
Goodness-of-fit on F^2	1.056
Final R indexes [I>=2σ (I)]	R_1 = 0.0510, wR_2 = 0.1280
Final R indexes [all data]	R_1 = 0.0609, wR_2 = 0.1401
Largest diff. peak/hole / e Å^{-3}	0.17/-0.32
Flack parameter	0.00(10)

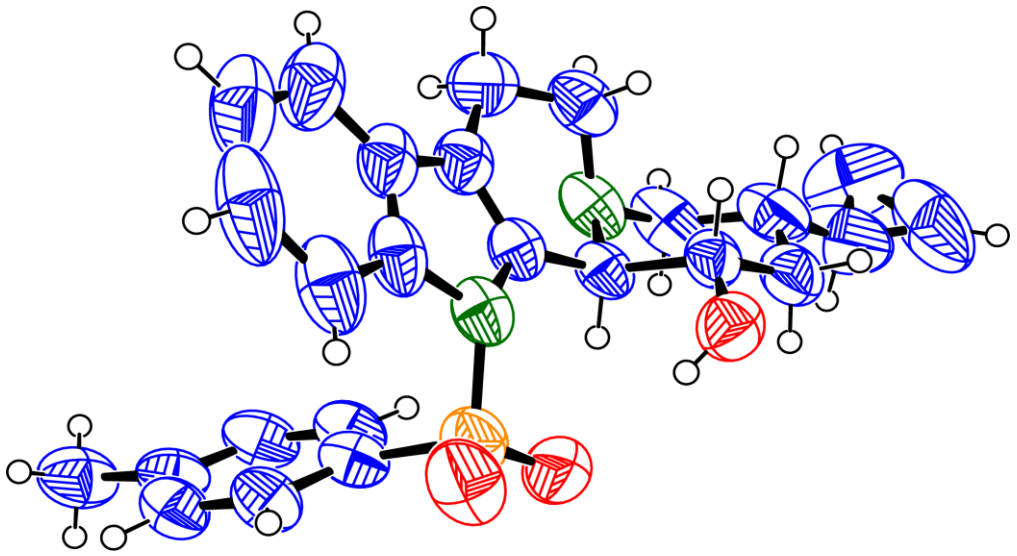


Figure S4: Ortep Drawing of Compound (+)-**34**. Thermal ellipsoids set to 50% probability level.

Table 3: Crystal data and structure refinement for Compound (+)-**34**

CCDC 1424172

Identification code	Compound (+)-**34**
Empirical formula	C_{48} H_{52} N_4 O_7 S_2

Formula weight	861.06
Temperature/K	298
Crystal system	orthorhombic
Space group	$C222_1$
a/Å	14.2702(11)
b/Å	14.2594(7)
c/Å	22.3948(15)
α/°	90.00
β/°	90.00
γ/°	90.00
Volume/$Å^3$	4557.0(5)
Z	4
$\rho_{calc}mg/mm^3$	1.255
μ/mm^{-1}	0.172
F(000)	1824
Crystal size/mm^3	0.32 × 0.16 × 0.14
2Θ range for data collection	5.9 to 58.16°
Index ranges	$-19 \leq h \leq 12$, $-19 \leq k \leq 15$, $-30 \leq l \leq 20$
Reflections collected	10530
Independent reflections	6150 [R(int) = 0.0386]
Data/restraints/parameters	6150/0/298
Goodness-of-fit on F^2	1.050
Final R indexes [I>=2σ (I)]	R_1 = 0.0626, wR_2 = 0.1576
Final R indexes [all data]	R_1 = 0.0908, wR_2 = 0.1861
Largest diff. peak/hole / e $Å^{-3}$	0.19/-0.22
Flack parameter	0.00(10)