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

Regioselective oxidation of indoles to 2-oxindoles

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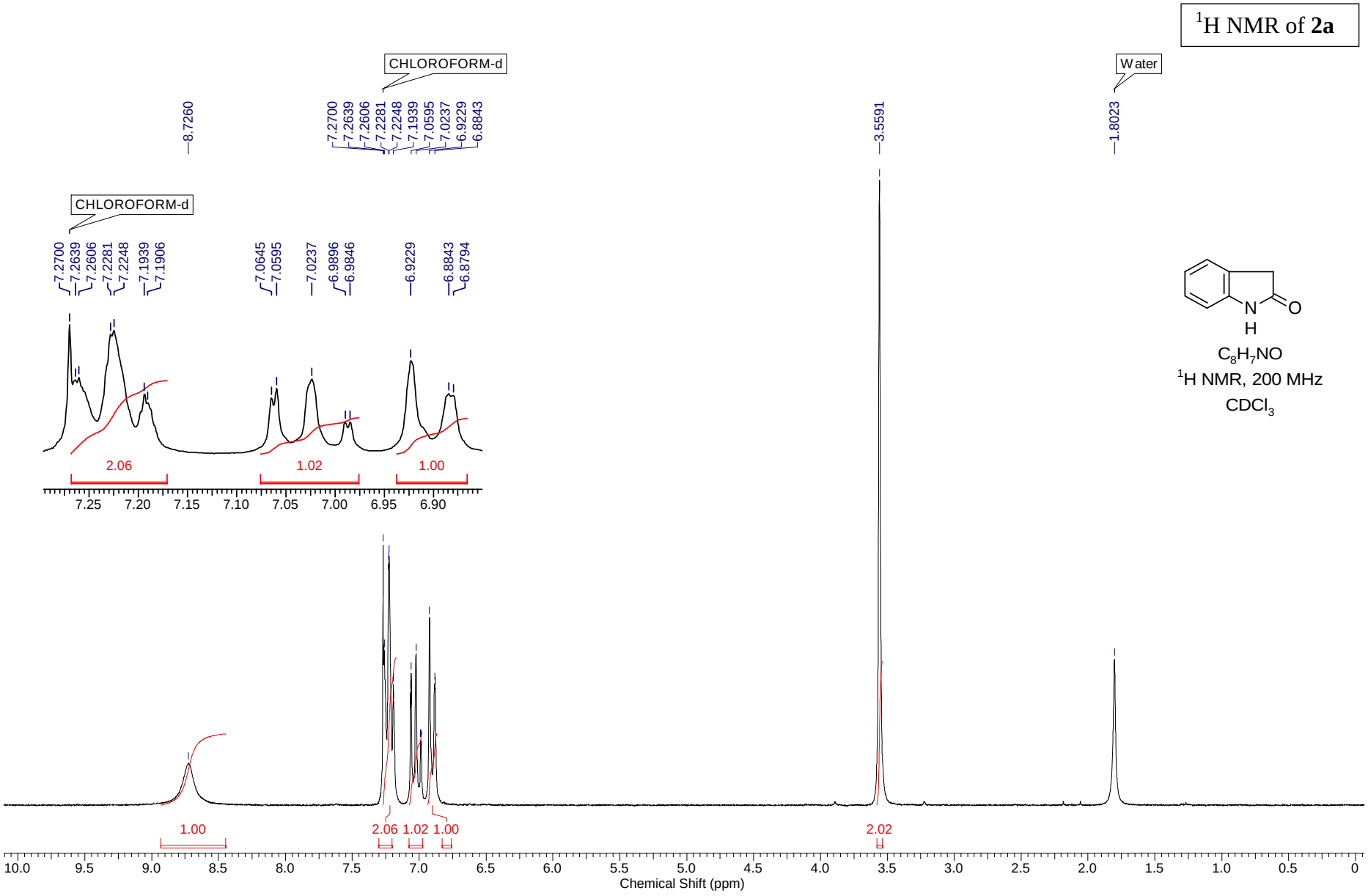
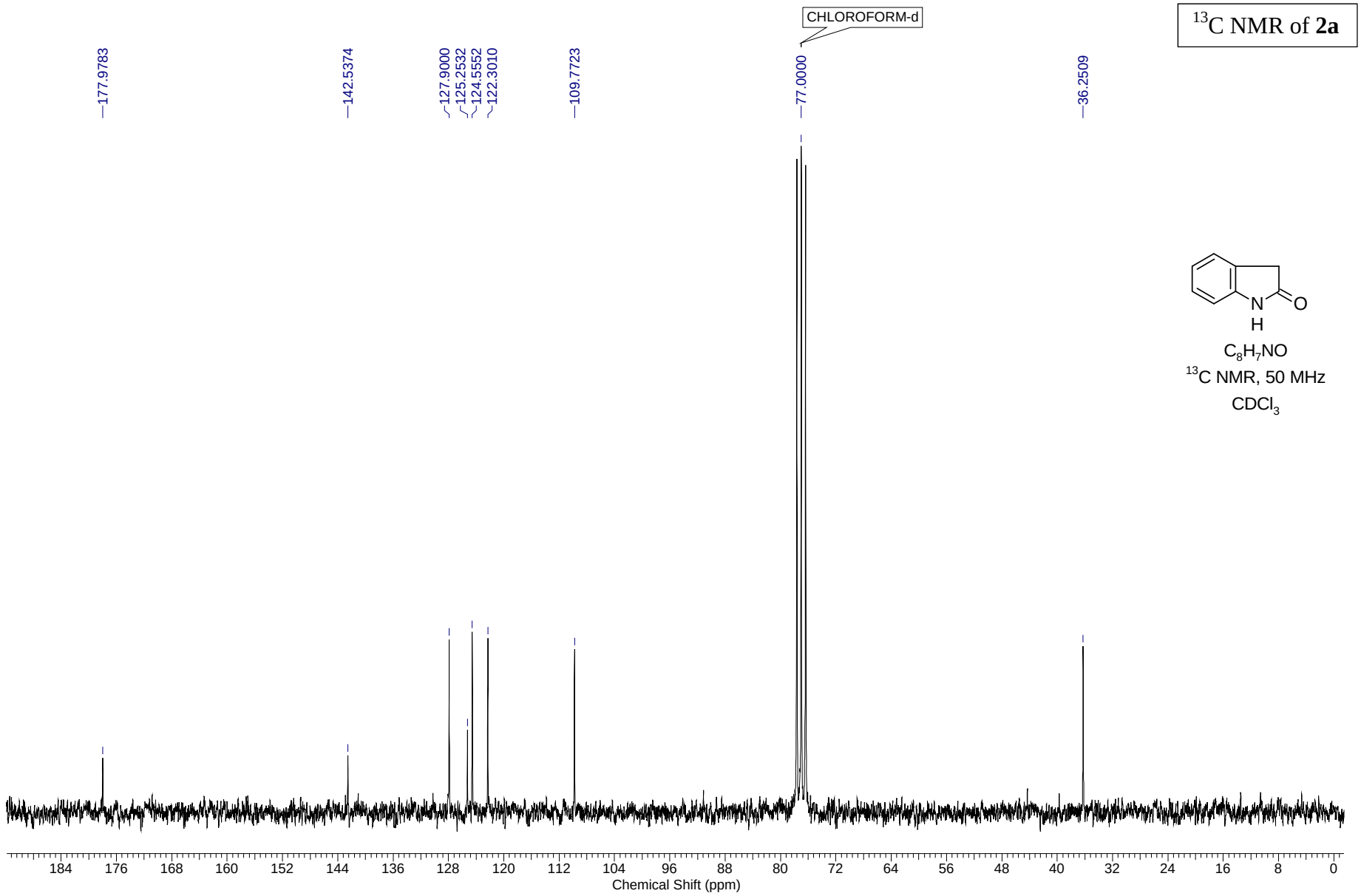
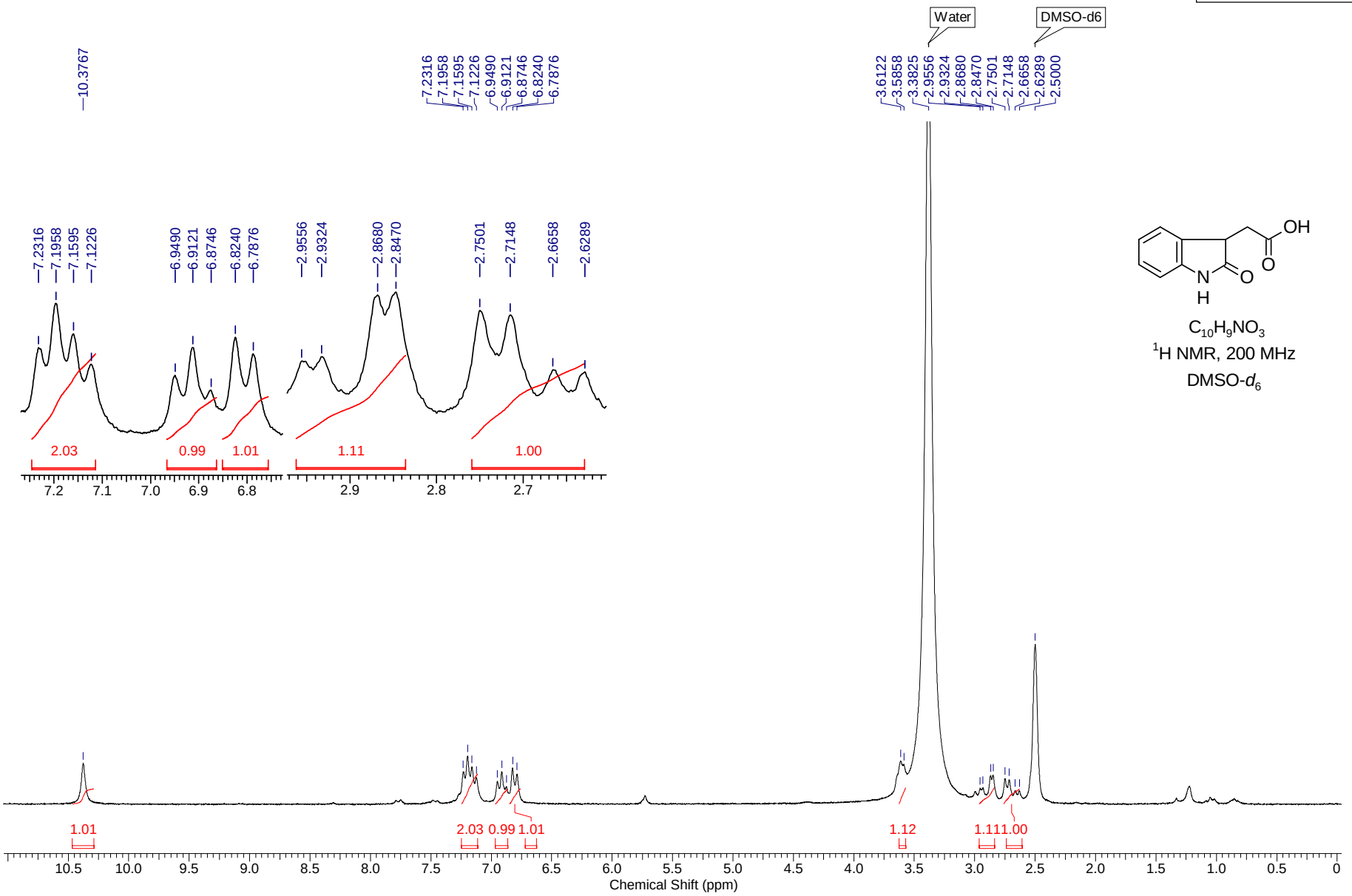

^{1}H NMR of **2a**
CHLOROFORM-d
Water
8.7260
7.2700
7.2639
7.2606
7.2281
7.2248
7.1939
7.0595
7.0237
6.9229
6.8843
3.5591
1.8023
CHLOROFORM-d
7.2700
7.2639
7.2606
7.2281
7.2248
7.1939
7.1906
7.0645
7.0595
7.0237
6.9896
6.9846
6.9229
6.8843
6.8794
2.06
1.02
1.00
7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90
N
H
O
C_8H_7NO
^{1}H NMR, 200 MHz
$CDCl_3$
1.00
2.06 1.02 1.00
2.02
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)


^{13}C NMR of **2a**
CHLOROFORM-d
177.9783
142.5374
127.9000
125.2532
124.5552
122.3010
109.7723
77.0000
36.2509
N
H
O
C_8H_7NO
^{13}C NMR, 50 MHz
$CDCl_3$
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)

^{1}H NMR of **2i**

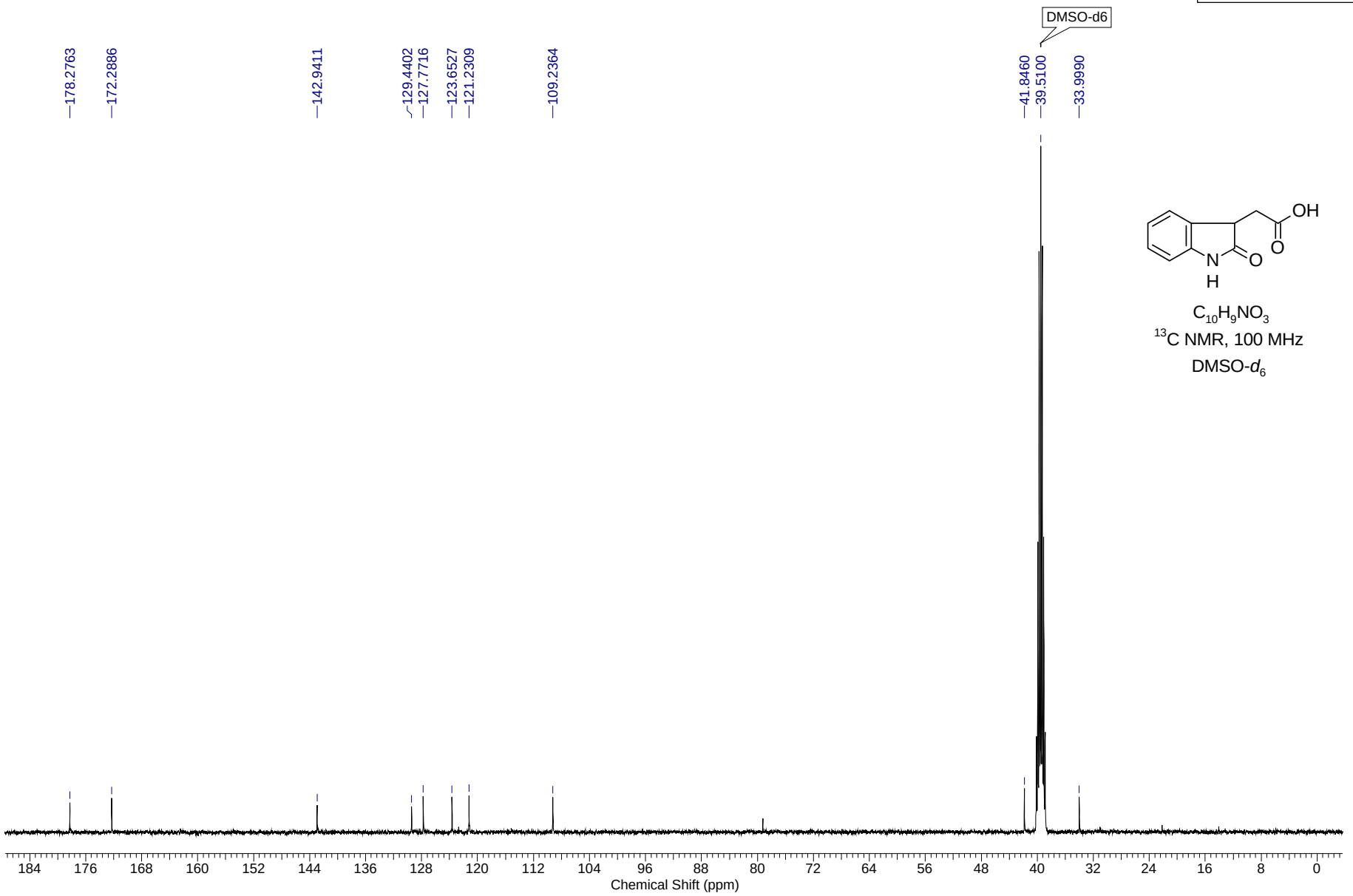


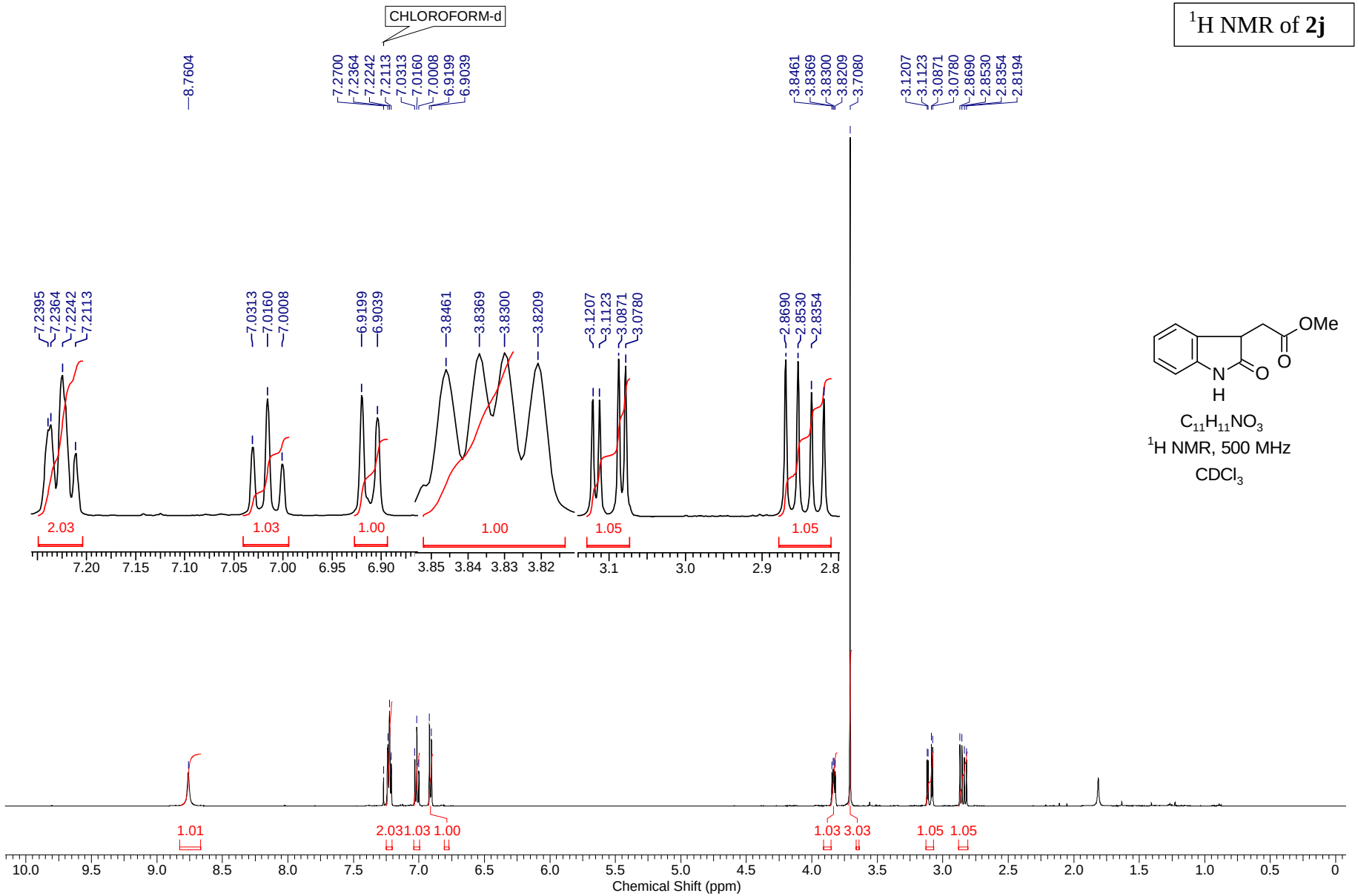
$C_{10}H_9NO_3$

^{1}H NMR, 200 MHz

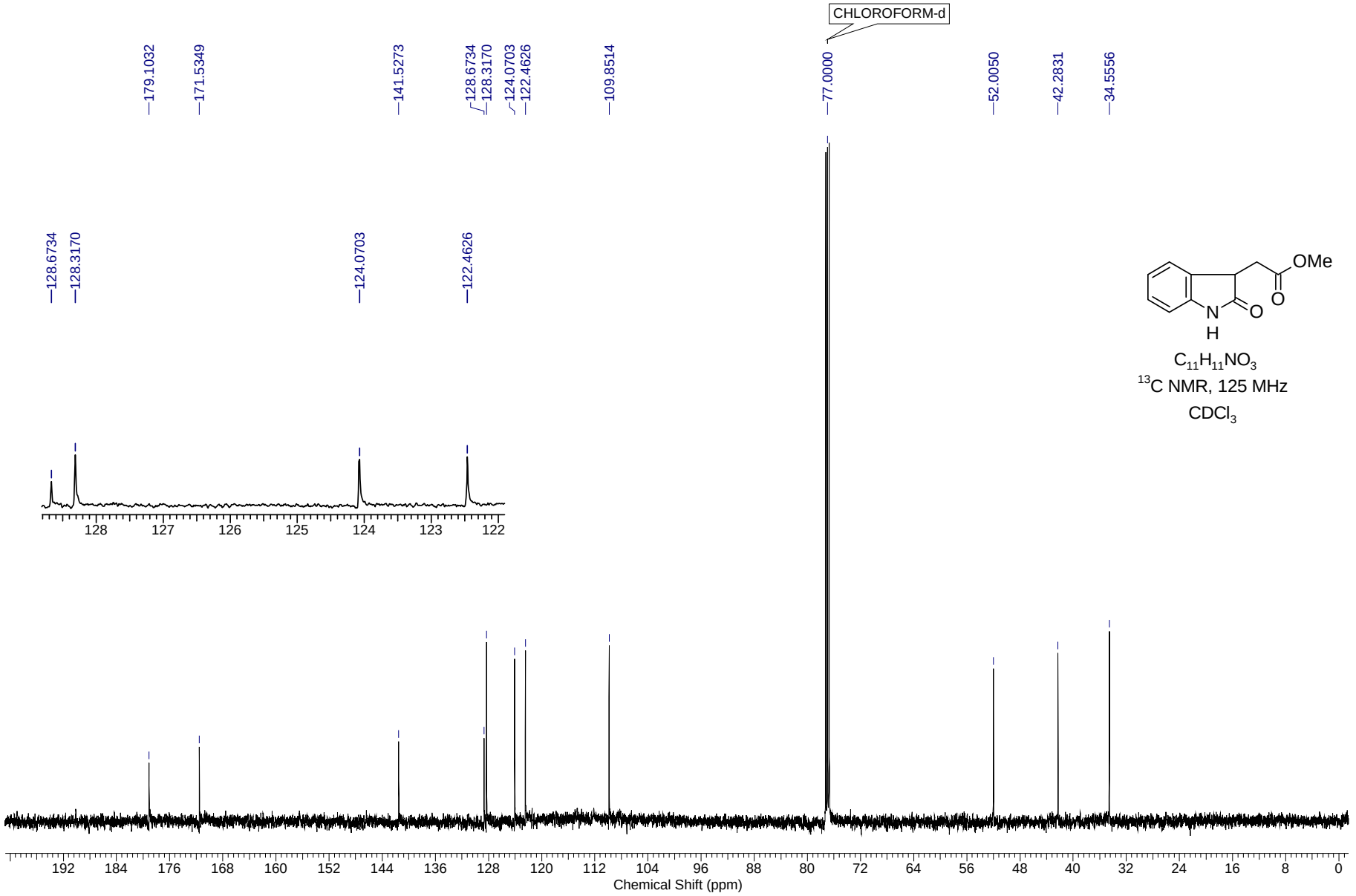
DMSO-d_6

^{13}C NMR of **2i**

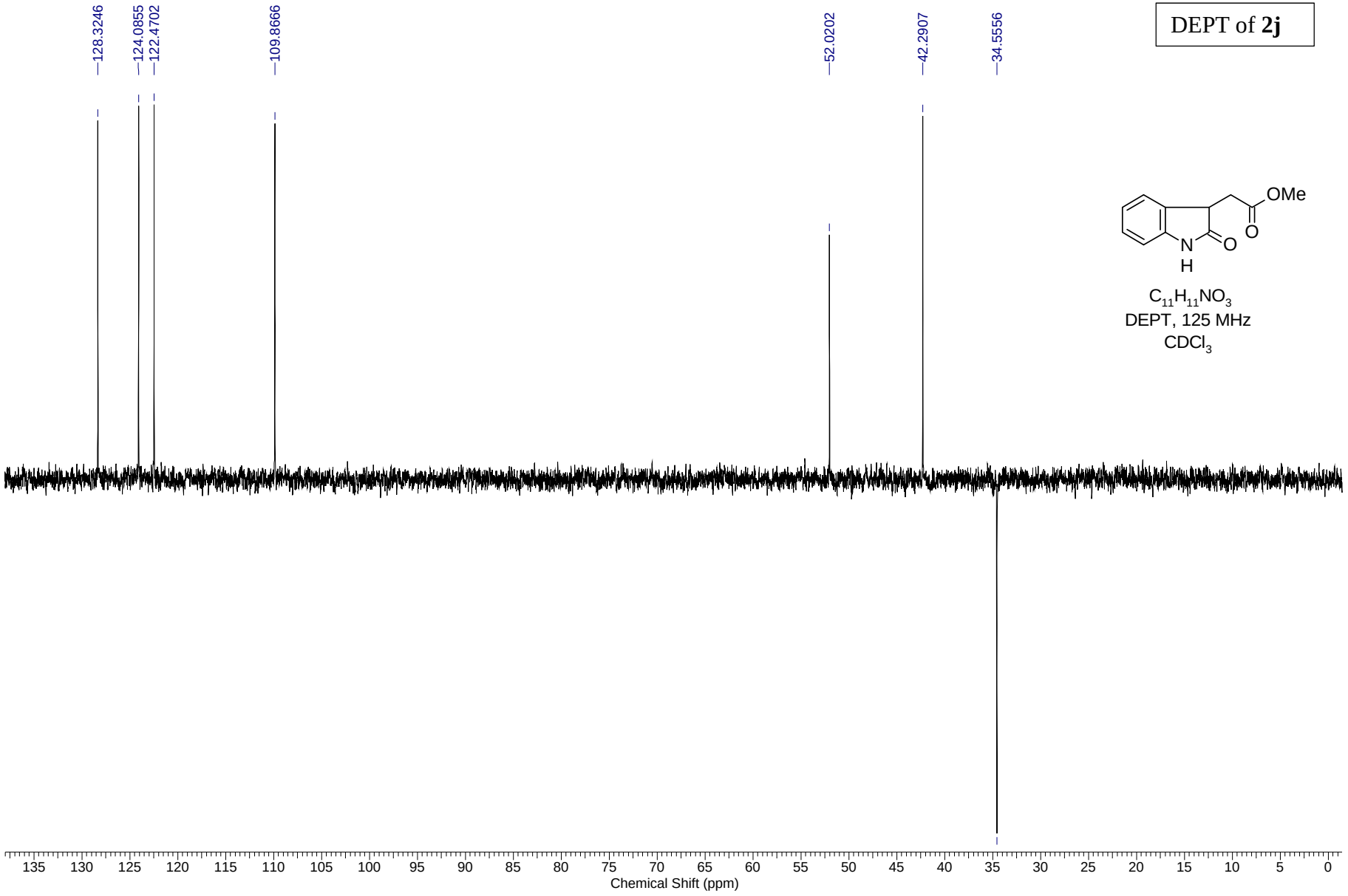

DMSO-d6
178.2763
172.2886
142.9411
129.4402
127.7716
123.6527
121.2309
109.2364
41.8460
39.5100
33.9990
OH
O
N
H
O
$C_{10}H_9NO_3$
^{13}C NMR, 100 MHz
DMSO-d_6
184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)


^{1}H NMR of **2j**
CHLOROFORM-d
8.7604
7.2700
7.2364
7.2242
7.2113
7.0313
7.0160
7.0008
6.9199
6.9039
3.8461
3.8369
3.8300
3.8209
3.7080
3.1207
3.1123
3.0871
3.0780
2.8690
2.8530
2.8354
2.8194
OMe
O
N
H
O
$C_{11}H_{11}NO_3$
^{1}H NMR, 500 MHz
$CDCl_3$
2.03
1.03
1.00
1.00
1.05
1.05
1.01
2.03
1.03
1.00
1.03
3.03
1.05
1.05
Chemical Shift (ppm)

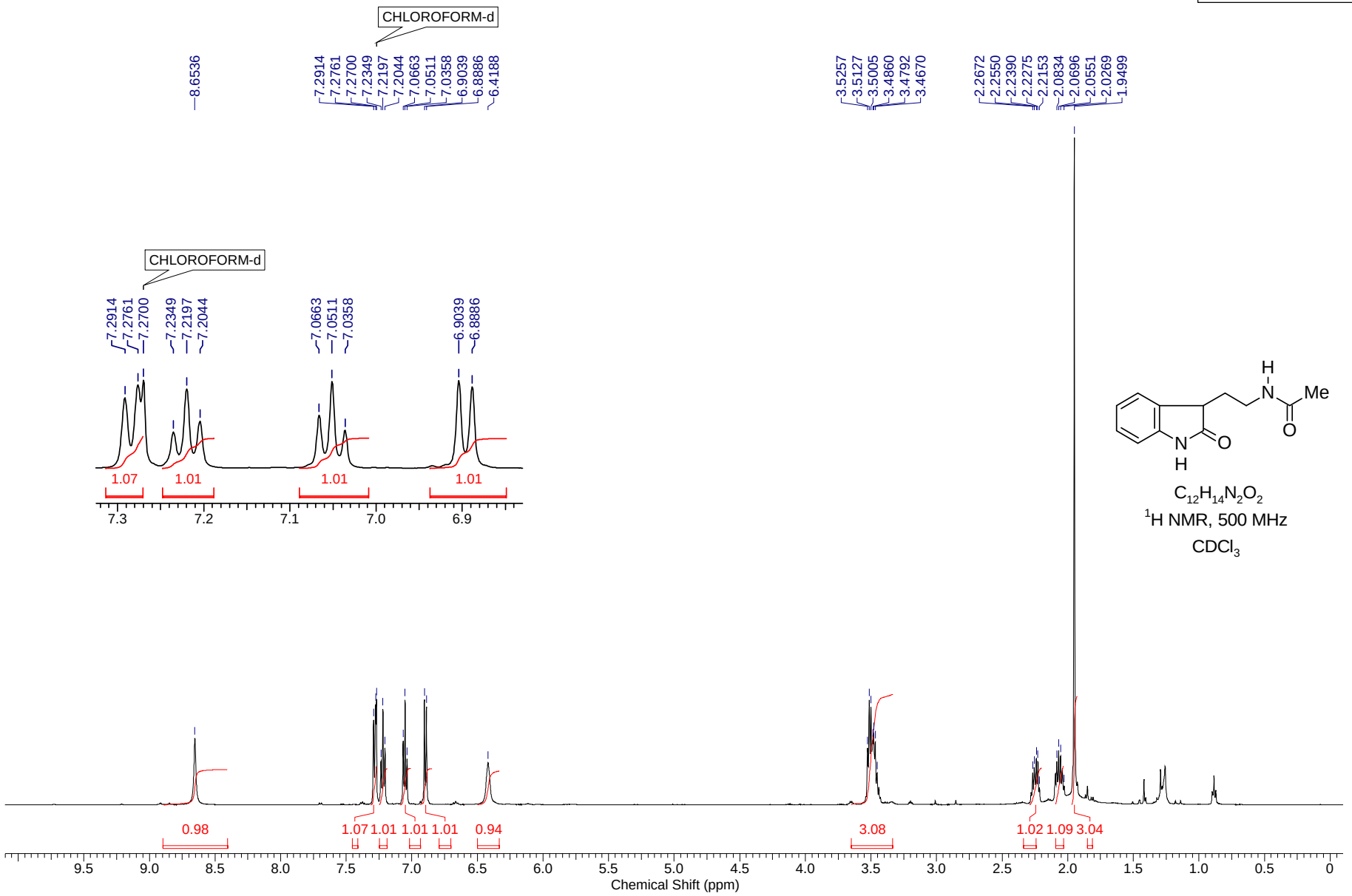
^{13}C NMR of **2j**


CHLOROFORM-d
179.1032
171.5349
141.5273
128.6734
128.3170
124.0703
122.4626
109.8514
77.0000
52.0050
42.2831
34.5556
OMe
O
N
H
O
$C_{11}H_{11}NO_3$
^{13}C NMR, 125 MHz
$CDCl_3$
Chemical Shift (ppm)

DEPT of **2j**


128.3246
124.0855
122.4702
109.8666
52.0202
42.2907
34.5556
OMe
O
N
H
O
$C_{11}H_{11}NO_3$
DEPT, 125 MHz
$CDCl_3$
135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
Chemical Shift (ppm)

^{1}H NMR of **2k**

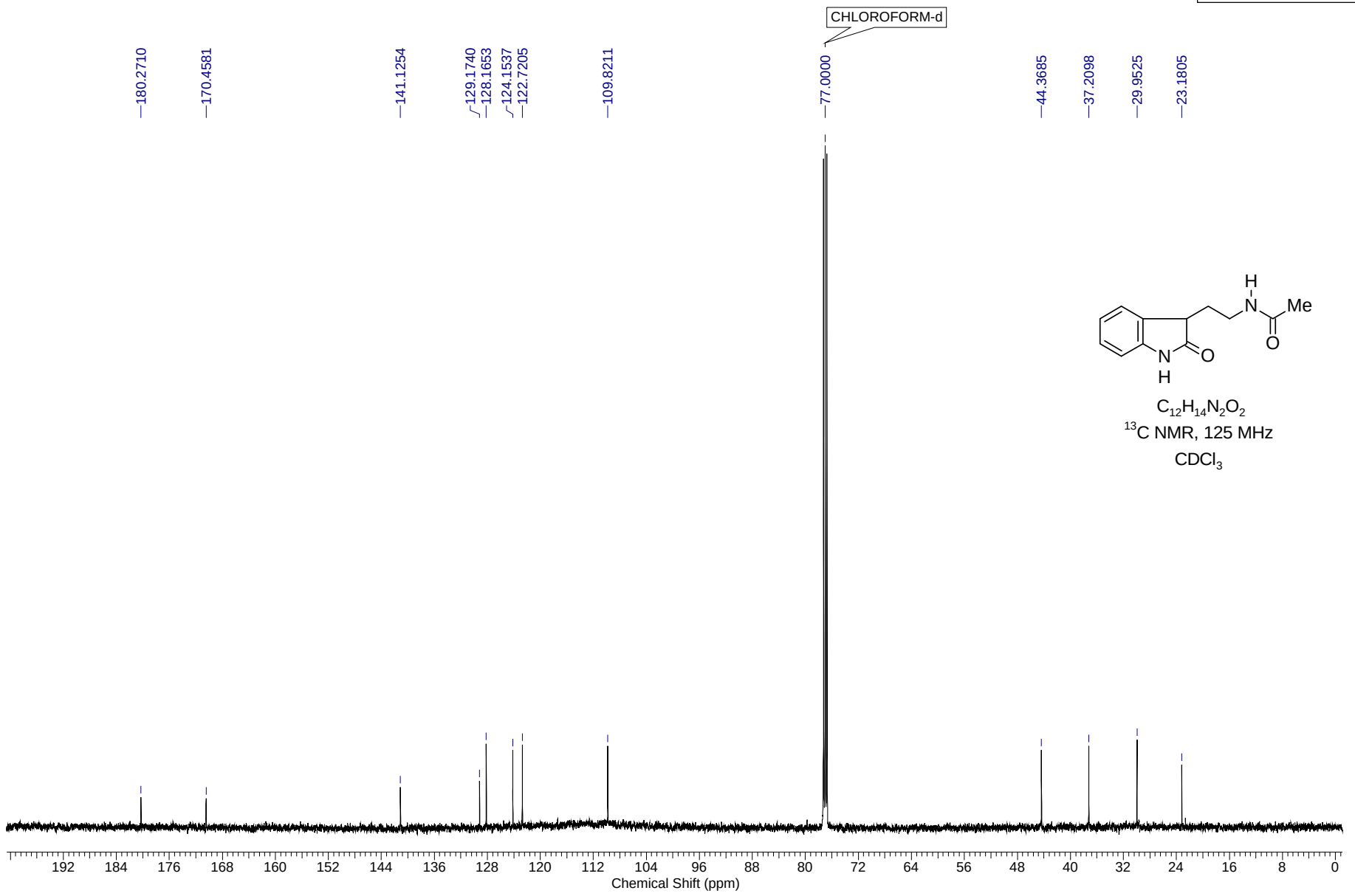


$C_{12}H_{14}N_2O_2$

^{1}H NMR, 500 MHz

$CDCl_3$

^{13}C NMR of **2k**

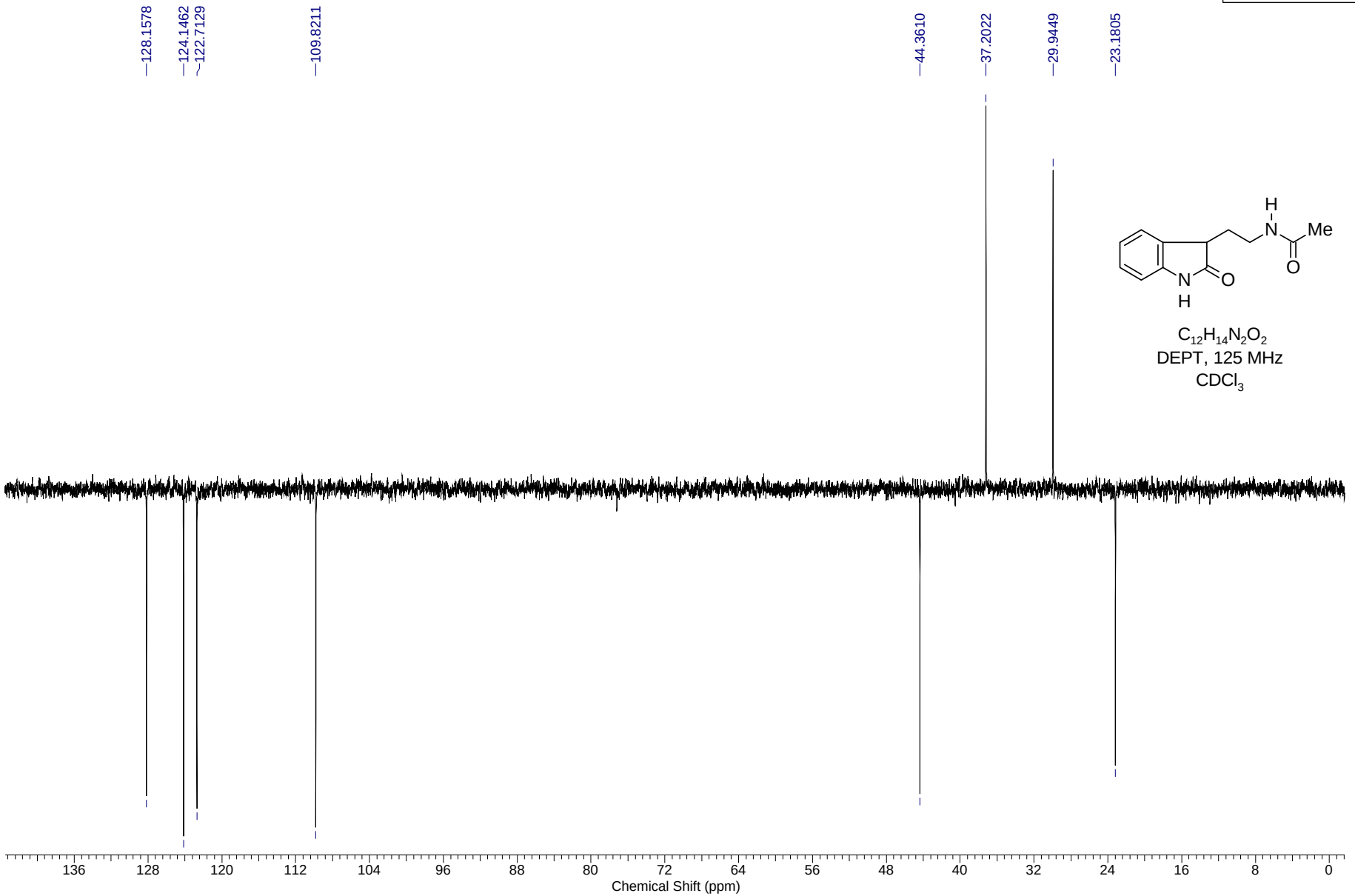


$C_{12}H_{14}N_2O_2$

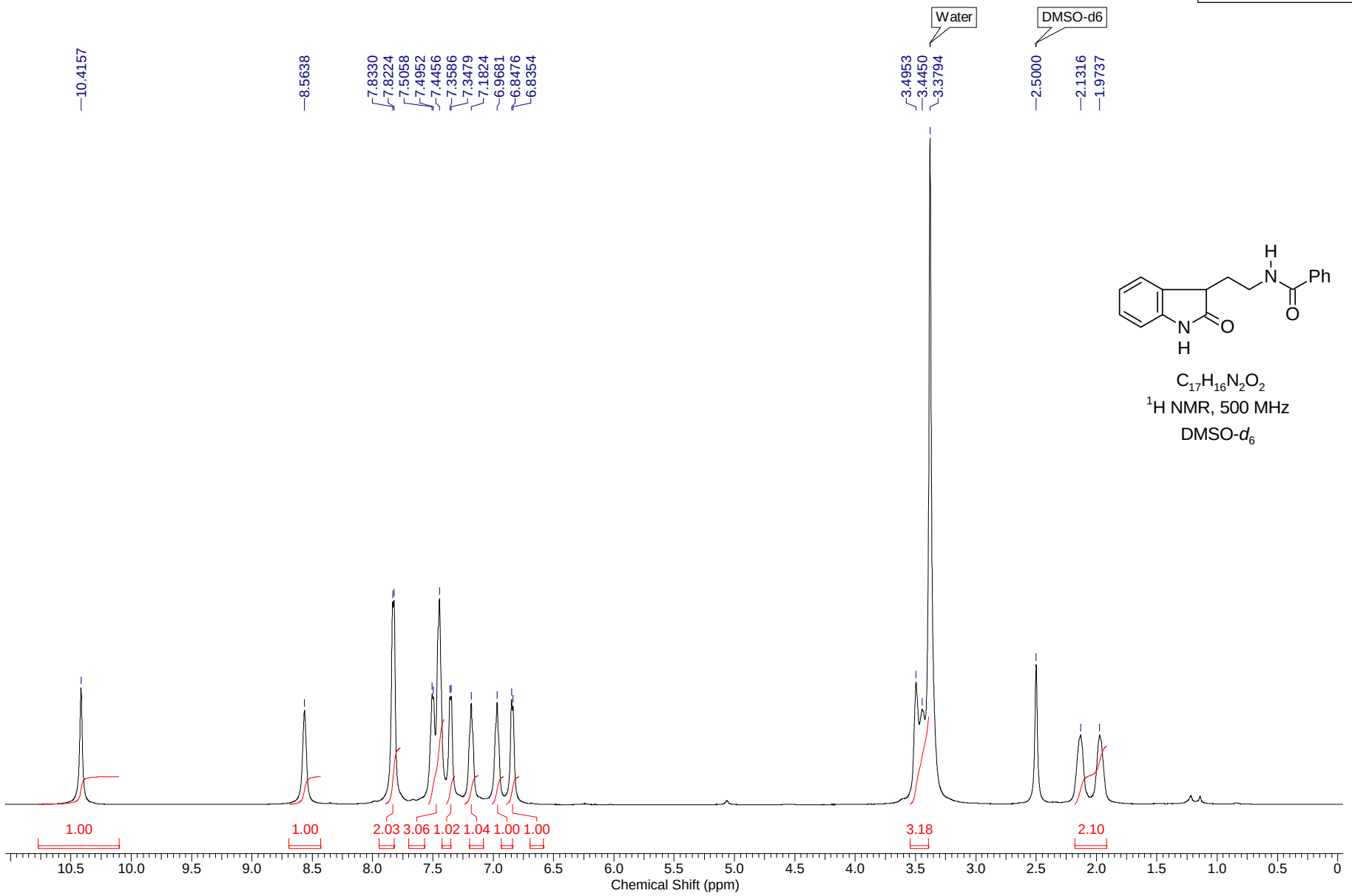
^{13}C NMR, 125 MHz

$CDCl_3$

DEPT of **2k**


128.1578
124.1462
122.7129
109.8211
44.3610
37.2022
29.9449
23.1805
H
N
Me
O
N
H
O
$C_{12}H_{14}N_2O_2$
DEPT, 125 MHz
$CDCl_3$
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

^{1}H NMR of **2l**

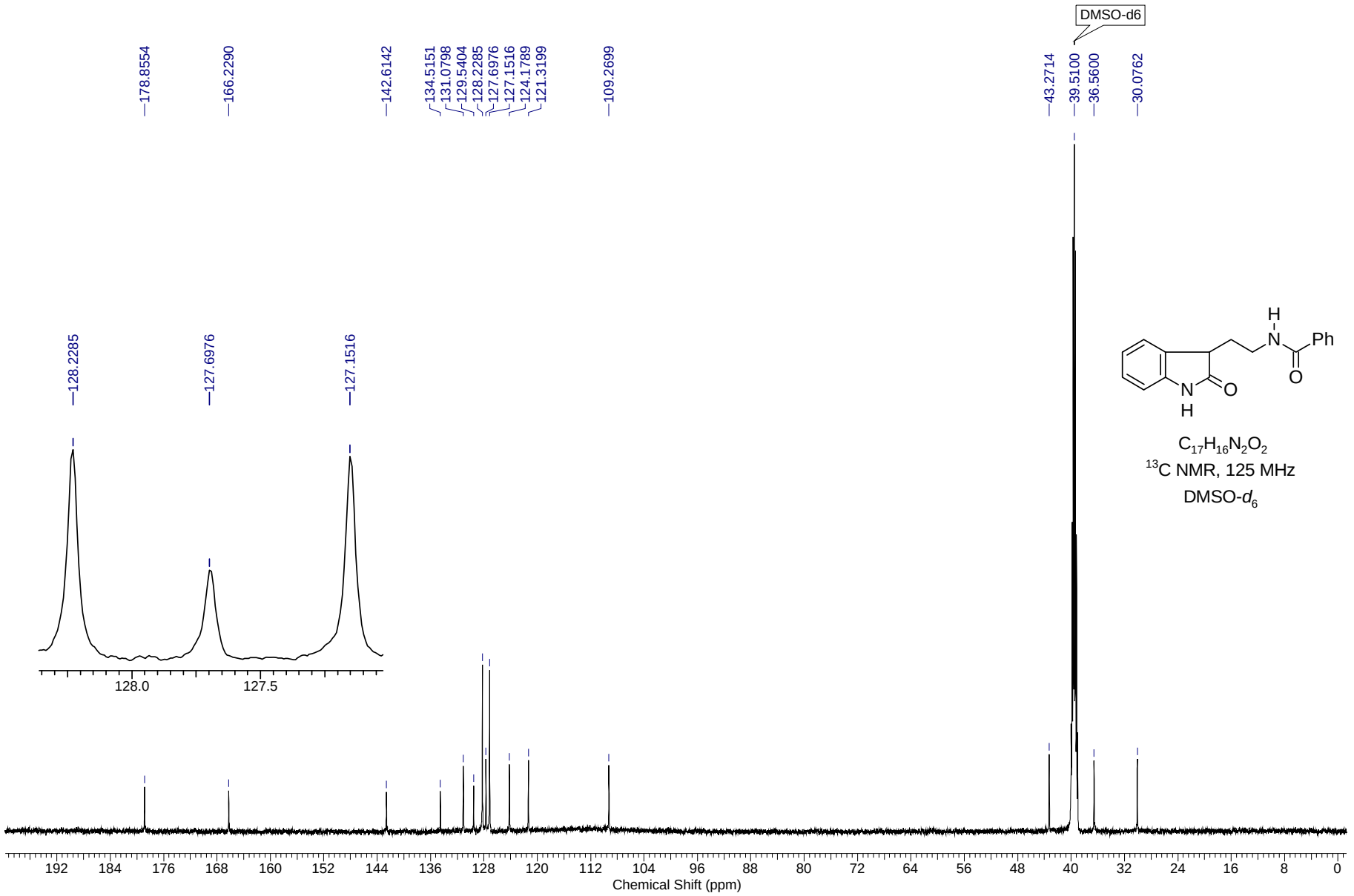


$C_{17}H_{16}N_2O_2$

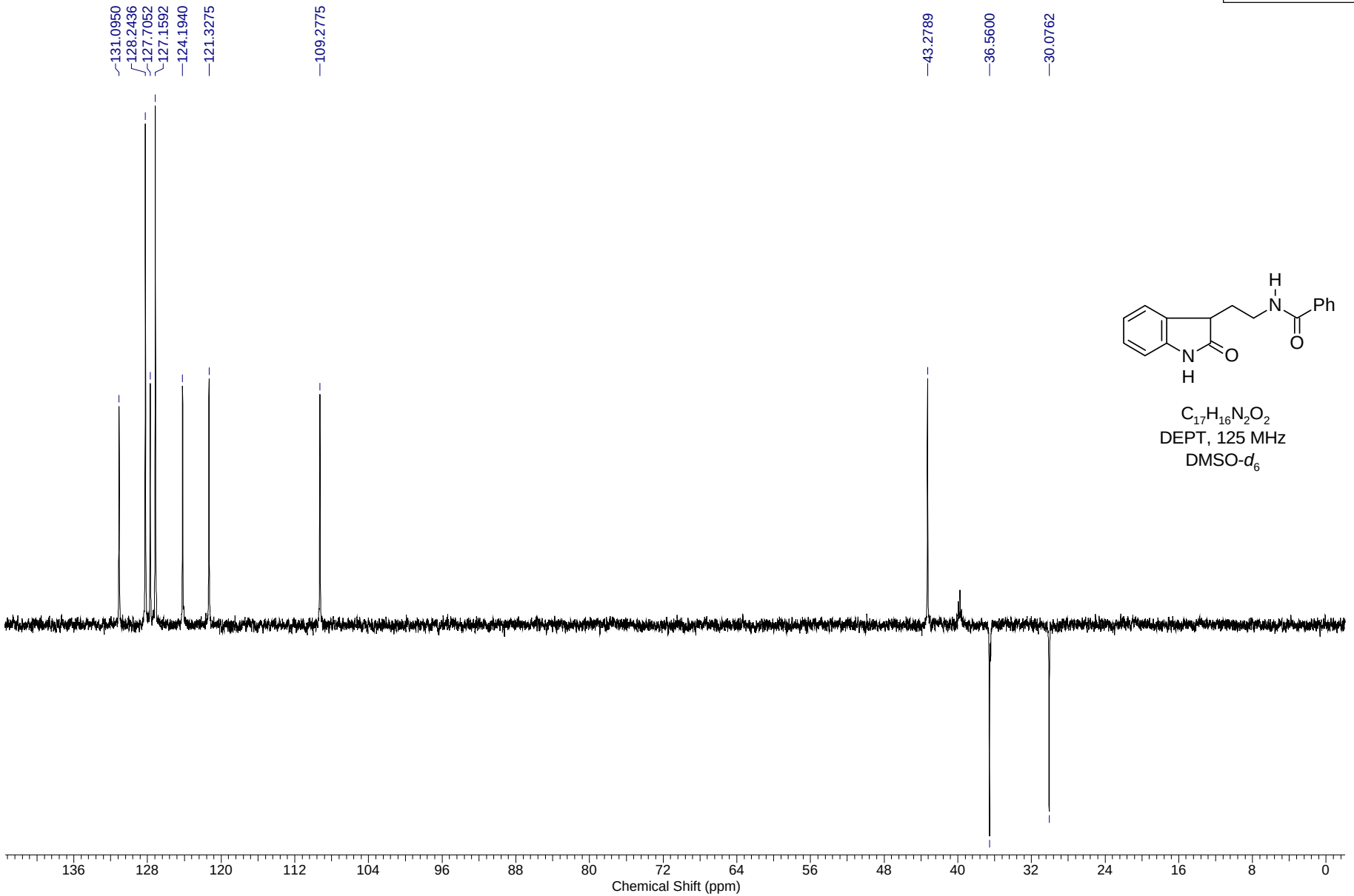
^{1}H NMR, 500 MHz

DMSO-d_6

^{13}C NMR of **2l**

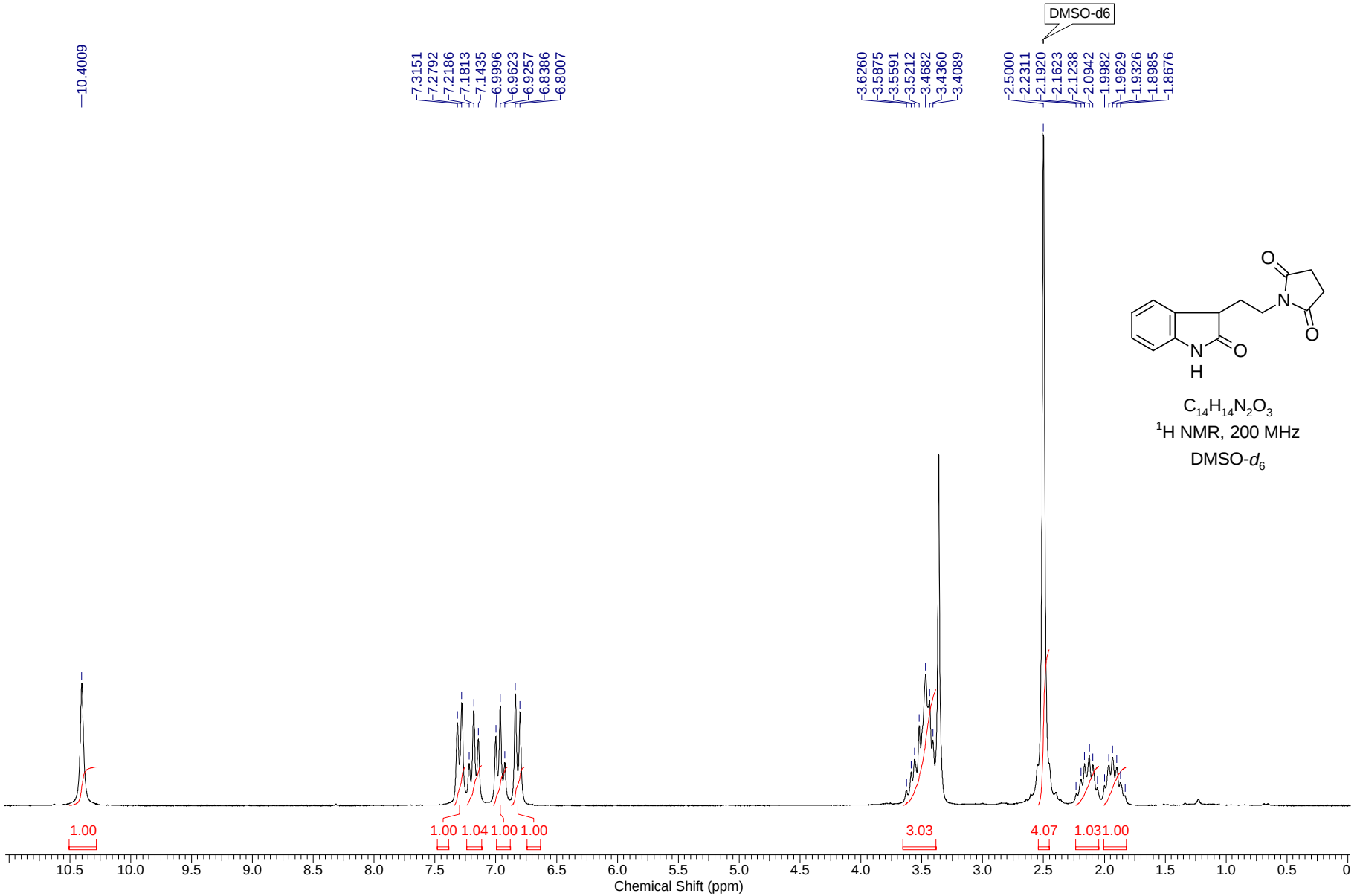

DMSO-d6
178.8554
166.2290
142.6142
134.5151
131.0798
129.5404
128.2285
127.6976
127.1516
124.1789
121.3199
109.2699
43.2714
39.5100
36.5600
30.0762
128.2285
127.6976
127.1516
128.0
127.5
H
N
Ph
O
N
H
O
C17H16N2O2
13C NMR, 125 MHz
DMSO-d6
192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

DEPT of **2l**



$C_{17}H_{16}N_2O_2$
DEPT, 125 MHz
DMSO-d_6

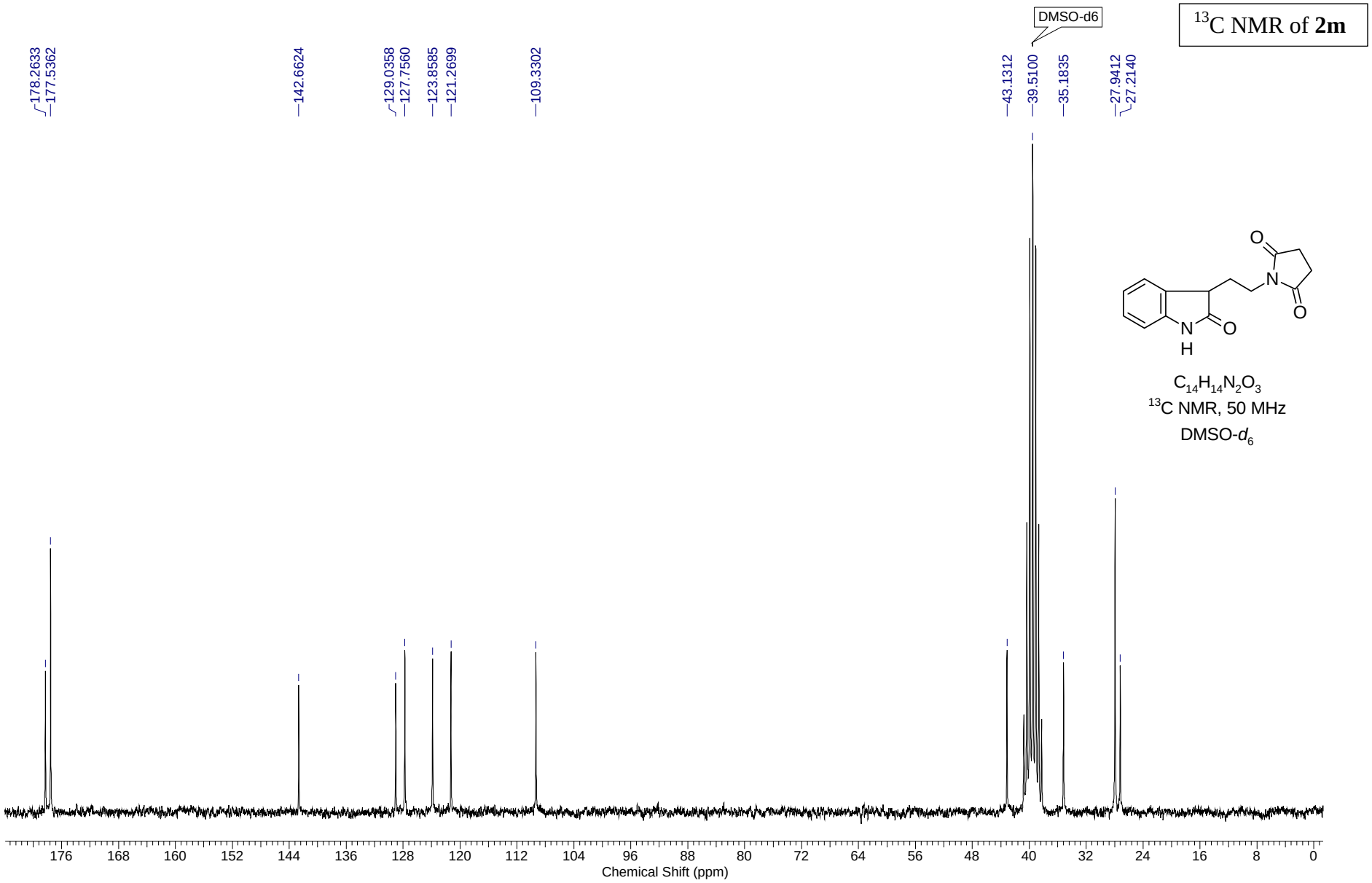
^{1}H NMR of **2m**



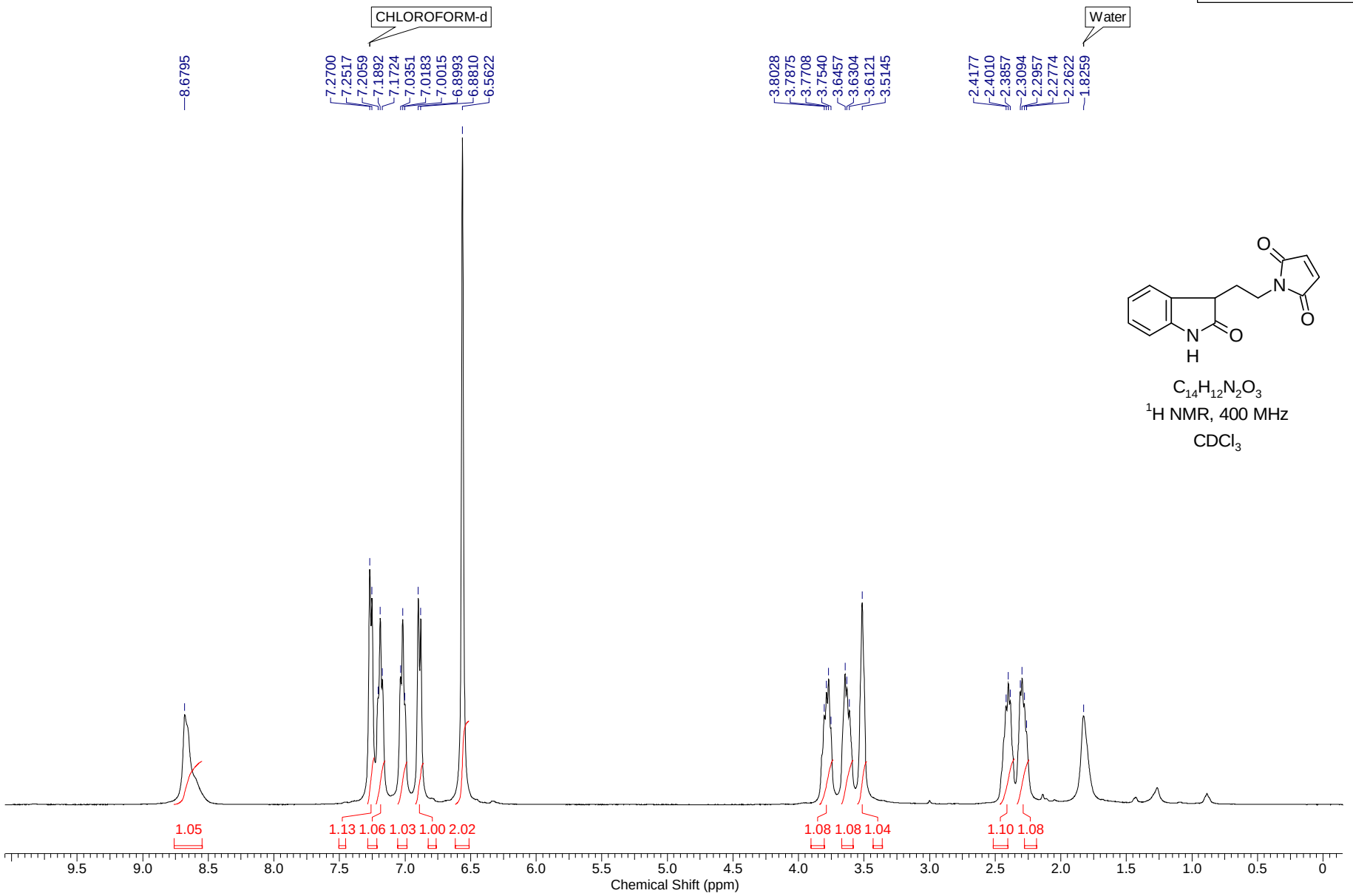
$C_{14}H_{14}N_2O_3$

^{1}H NMR, 200 MHz

DMSO-d_6


^{13}C NMR of **2m**
DMSO-d6
178.2633
177.5362
142.6624
129.0358
127.7560
123.8585
121.2699
109.3302
43.1312
39.5100
35.1835
27.9412
27.2140
O
N
O
N
H
O
$C_{14}H_{14}N_2O_3$
^{13}C NMR, 50 MHz
DMSO-d_6
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)

^{1}H NMR of **2n**

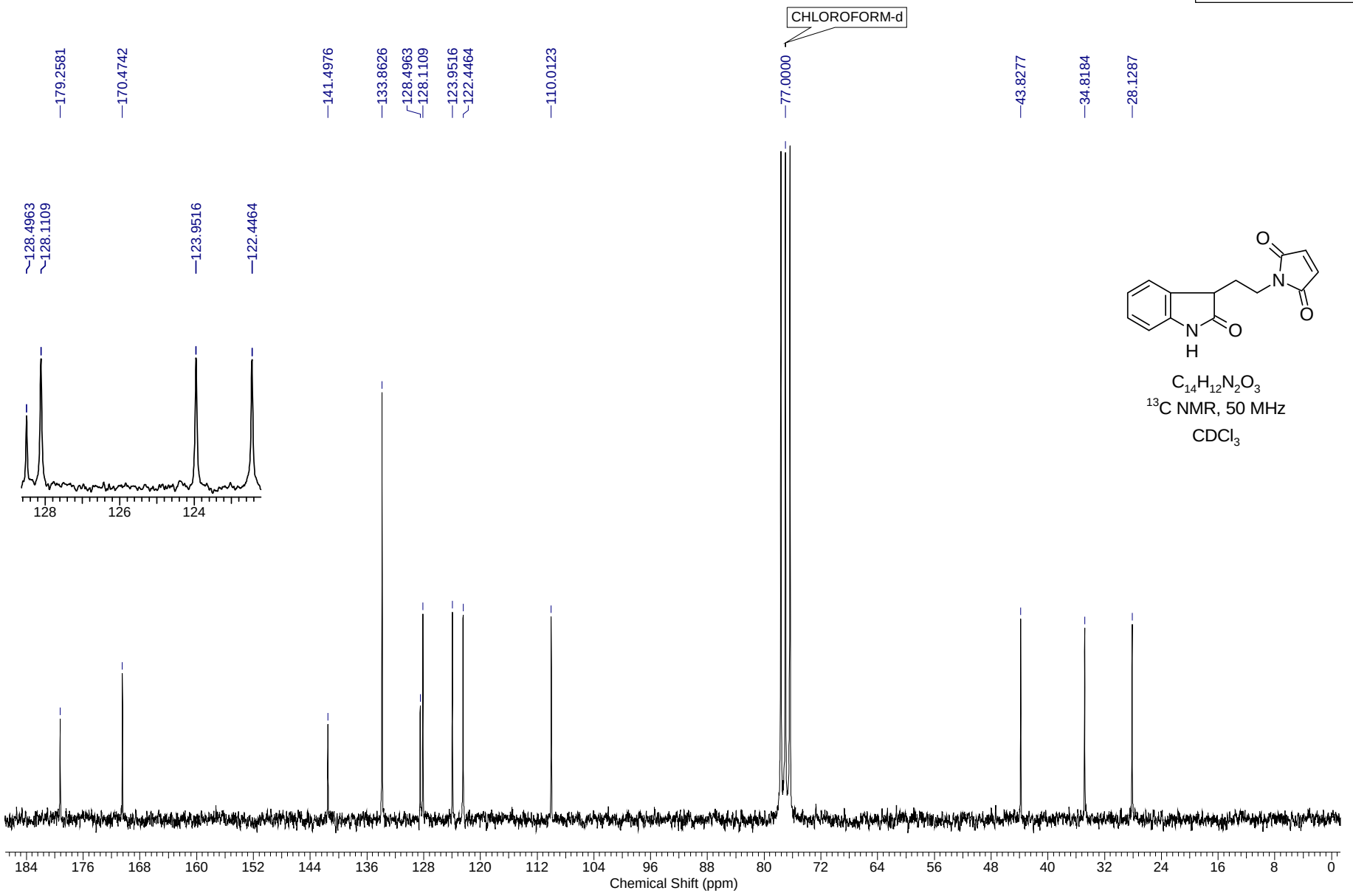


$C_{14}H_{12}N_2O_3$

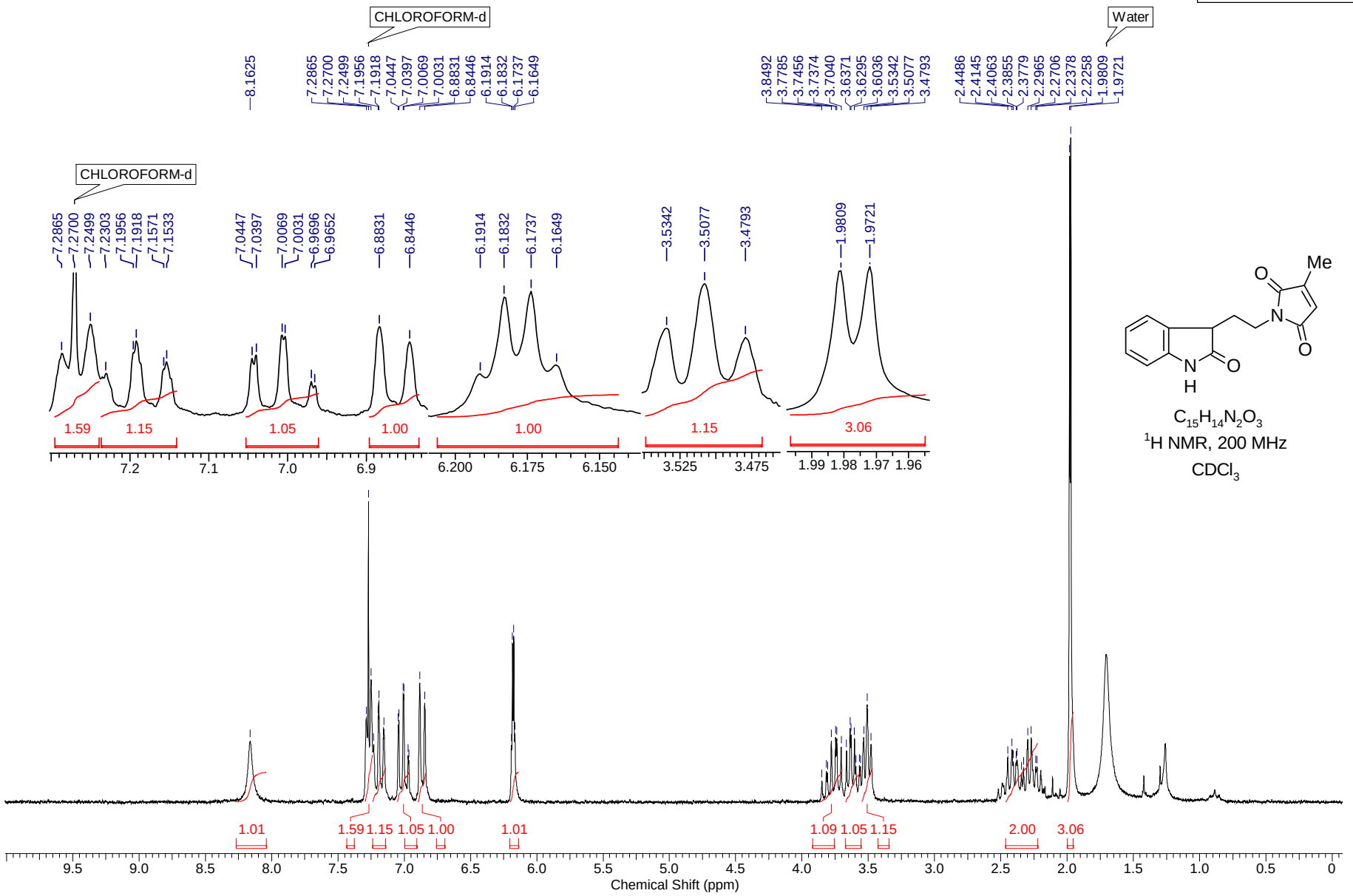
^{1}H NMR, 400 MHz

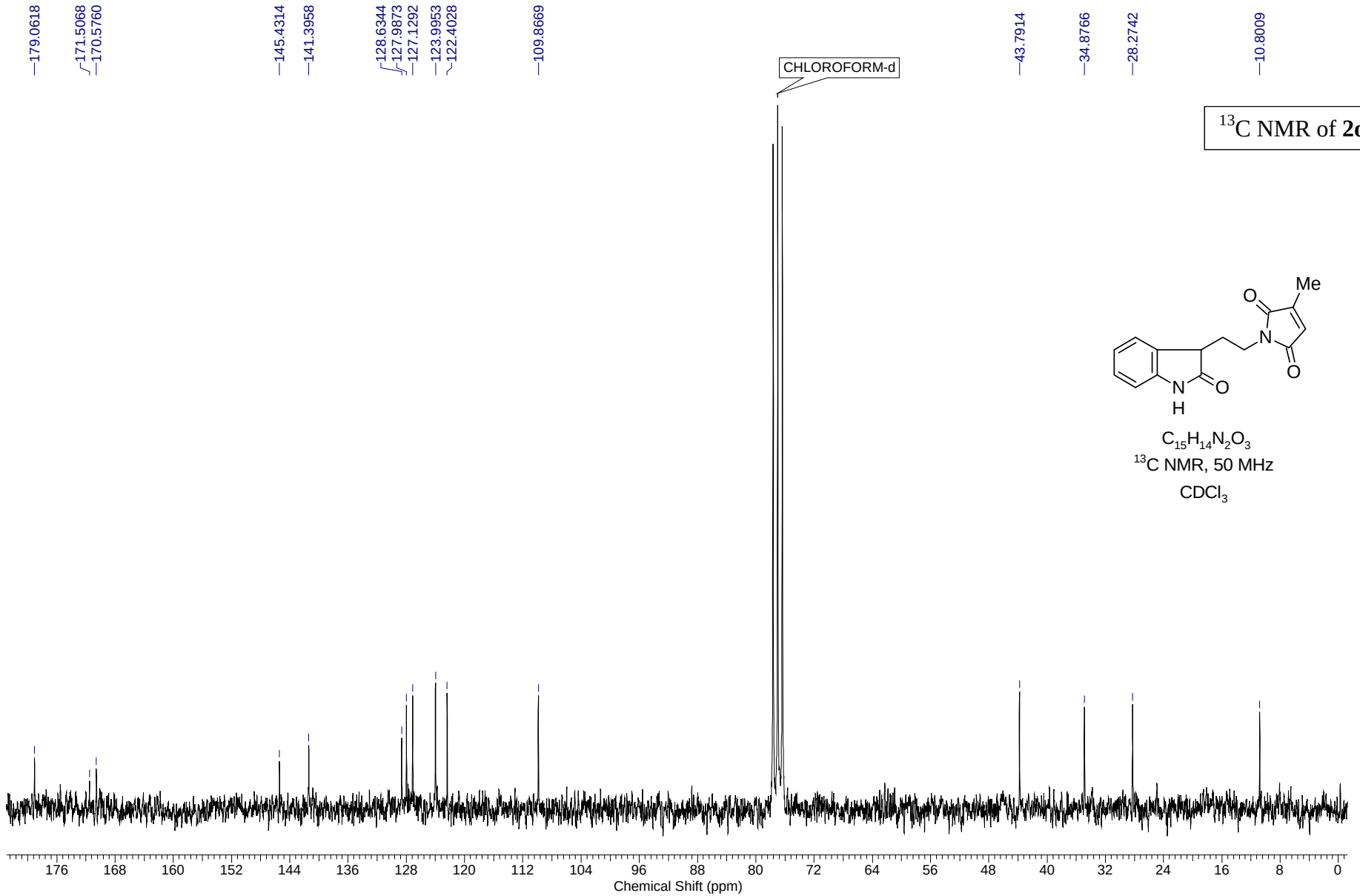
$CDCl_3$

^{13}C NMR of **2n**


CHLOROFORM-d
179.2581
170.4742
141.4976
133.8626
128.4963
128.1109
123.9516
122.4464
110.0123
77.0000
43.8277
34.8184
28.1287
128.4963
128.1109
123.9516
122.4464
128
126
124
O
N
O
O
N
H
$C_{14}H_{12}N_2O_3$
^{13}C NMR, 50 MHz
$CDCl_3$
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)

^{1}H NMR of **2o**


CHLOROFORM-d
Water
CHLOROFORM-d
8.1625
7.2865
7.2700
7.2499
7.1956
7.1918
7.0447
7.0397
7.0069
7.0031
6.8831
6.8446
6.1914
6.1832
6.1737
6.1649
3.8492
3.7785
3.7456
3.7374
3.7040
3.6371
3.6295
3.6036
3.5342
3.5077
3.4793
2.4486
2.4145
2.4063
2.3855
2.3779
2.2965
2.2706
2.2378
2.2258
1.9809
1.9721
7.2865
7.2700
7.2499
7.2303
7.1956
7.1918
7.1571
7.1533
7.0447
7.0397
7.0069
7.0031
6.9696
6.9652
6.8831
6.8446
6.1914
6.1832
6.1737
6.1649
3.5342
3.5077
3.4793
1.9809
1.9721
1.59
1.15
1.05
1.00
1.00
1.15
3.06
1.01
1.59
1.15
1.05
1.00
1.01
1.09
1.05
1.15
2.00
3.06
Chemical Shift (ppm)
Me
O
N
O
N
H
O
$C_{15}H_{14}N_2O_3$
^{1}H NMR, 200 MHz
$CDCl_3$

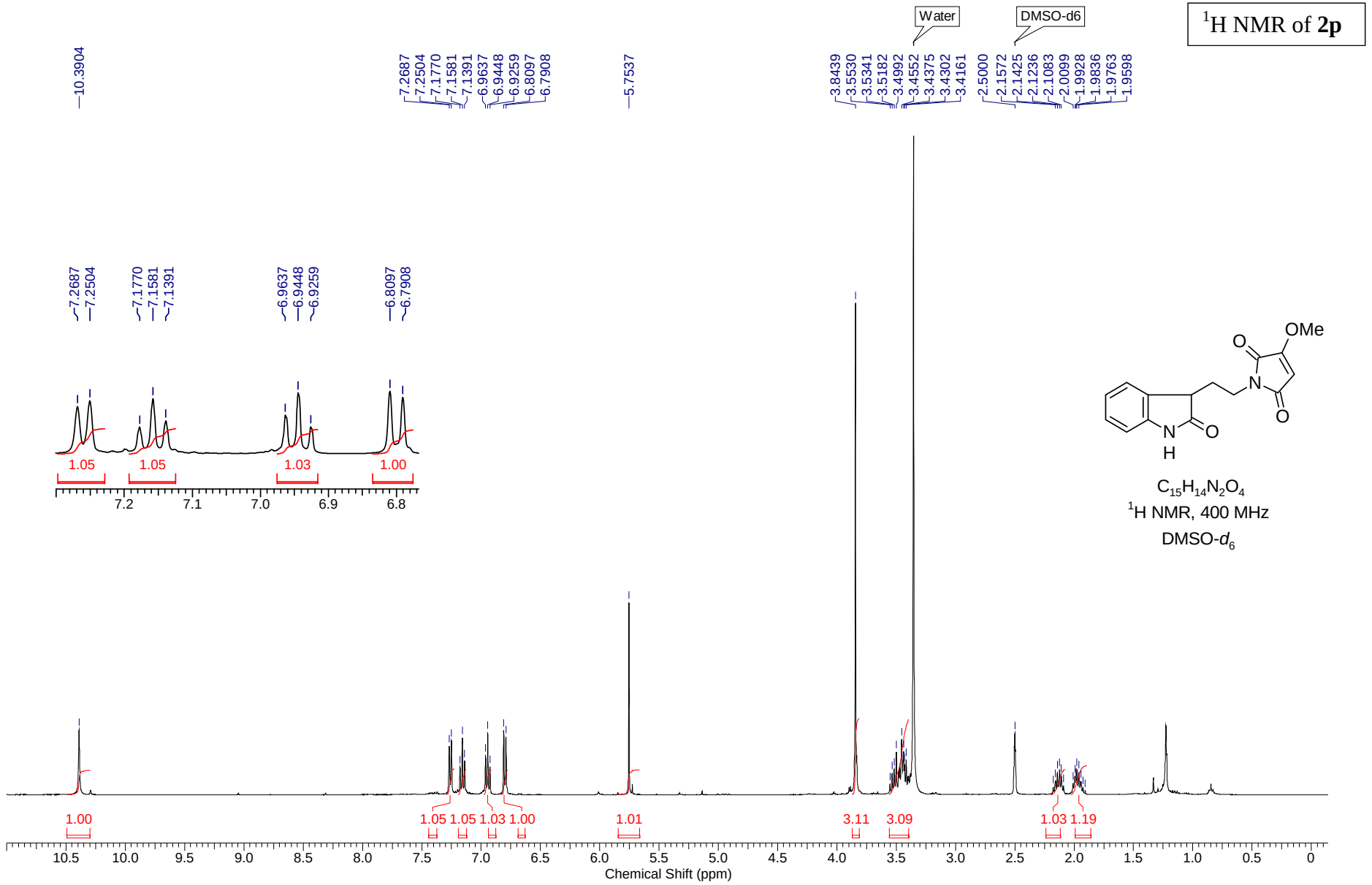


^{13}C NMR of **2o**

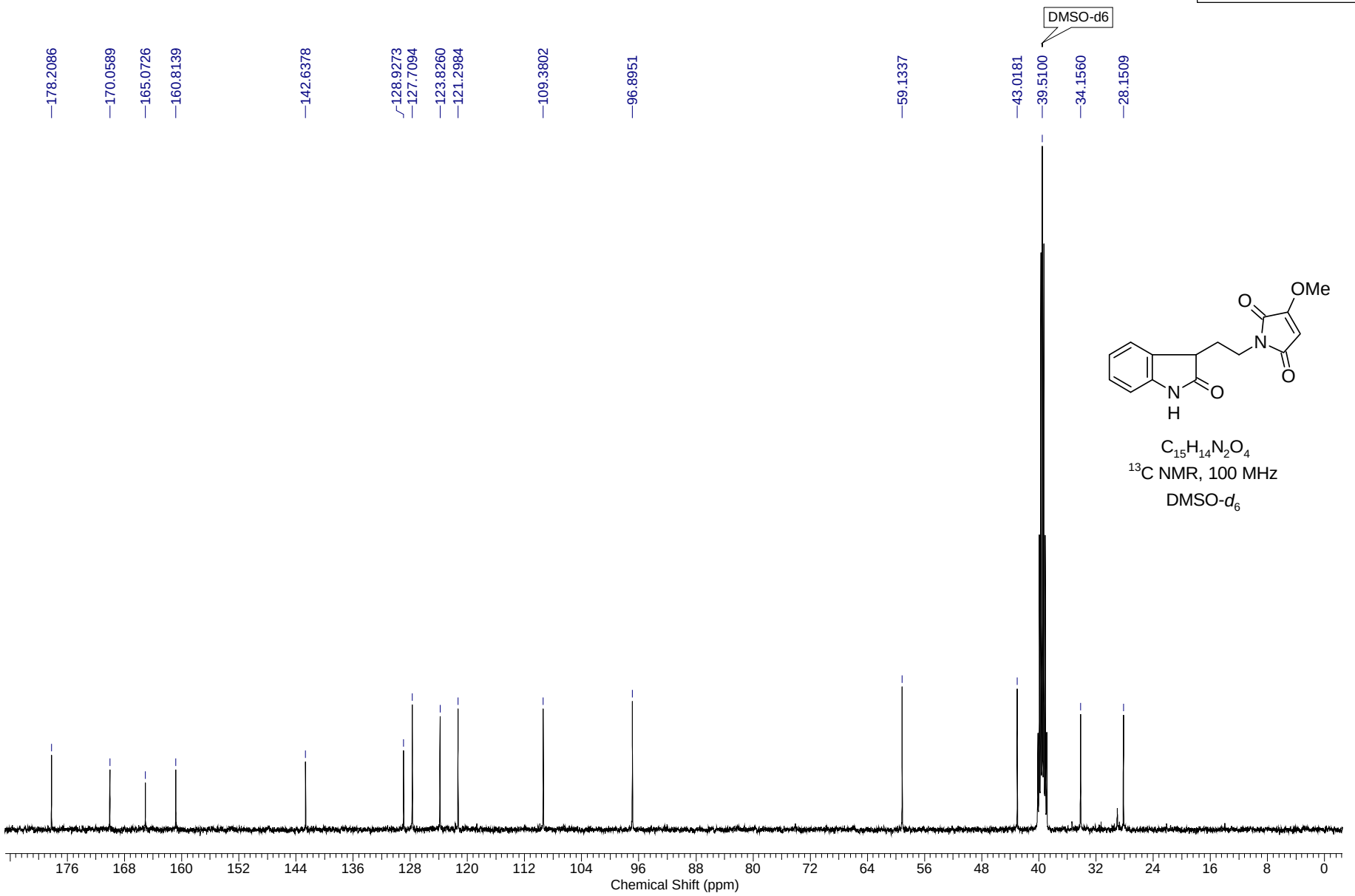
$C_{15}H_{14}N_2O_3$

^{13}C NMR, 50 MHz

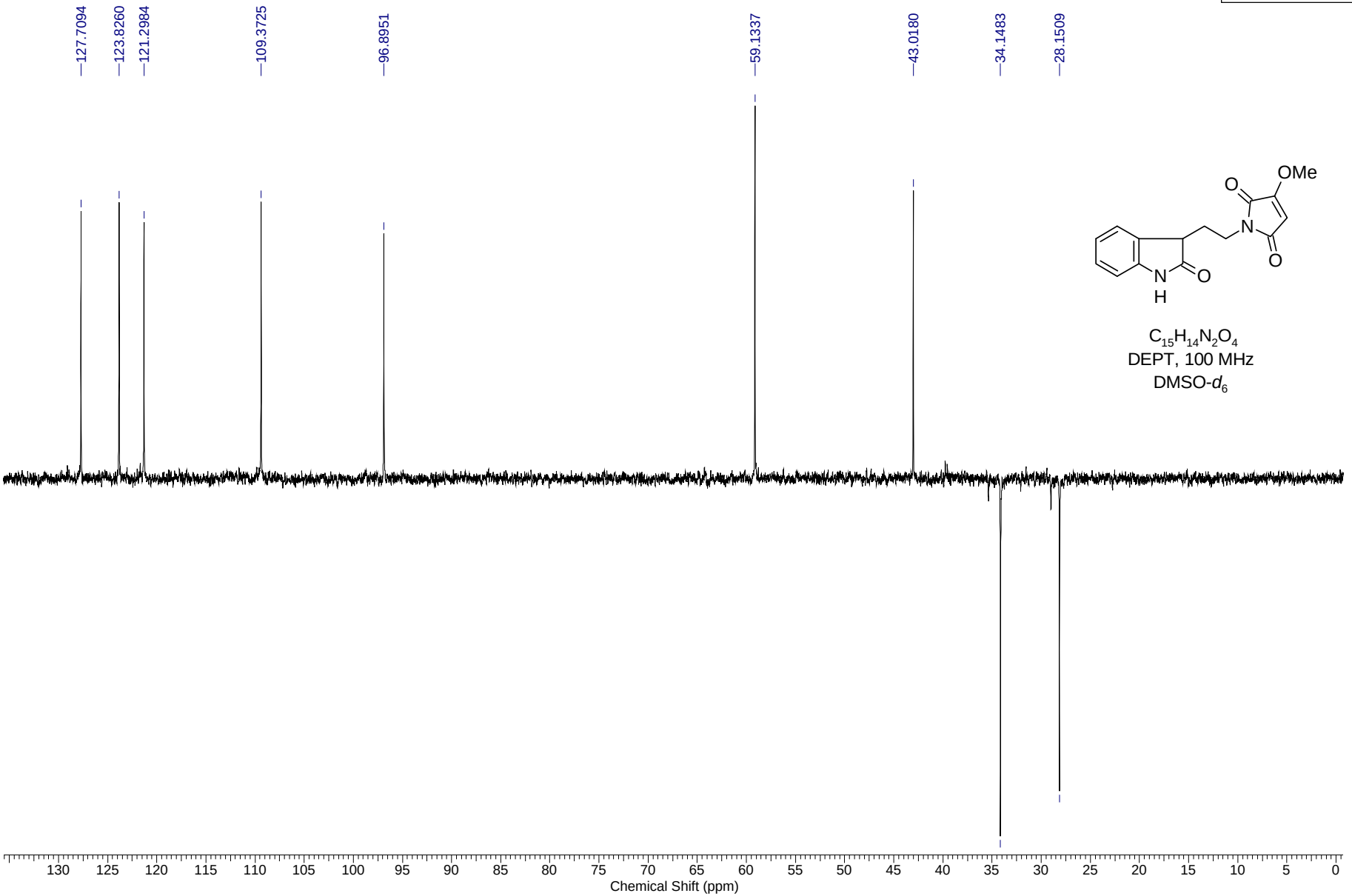
$CDCl_3$


^{1}H NMR of **2p**
Water
DMSO-d6
10.3904
7.2687
7.2504
7.1770
7.1581
7.1391
6.9637
6.9448
6.9259
6.8097
6.7908
5.7537
3.8439
3.5530
3.5341
3.5182
3.4992
3.4552
3.4375
3.4302
3.4161
2.5000
2.1572
2.1425
2.1236
2.1083
2.0099
1.9928
1.9836
1.9763
1.9598
1.05
1.05
1.03
1.00
1.00
1.05
1.05
1.03
1.00
1.01
3.11
3.09
1.03
1.19
Chemical Shift (ppm)
OMe
$C_{15}H_{14}N_2O_4$
^{1}H NMR, 400 MHz
DMSO-d_6

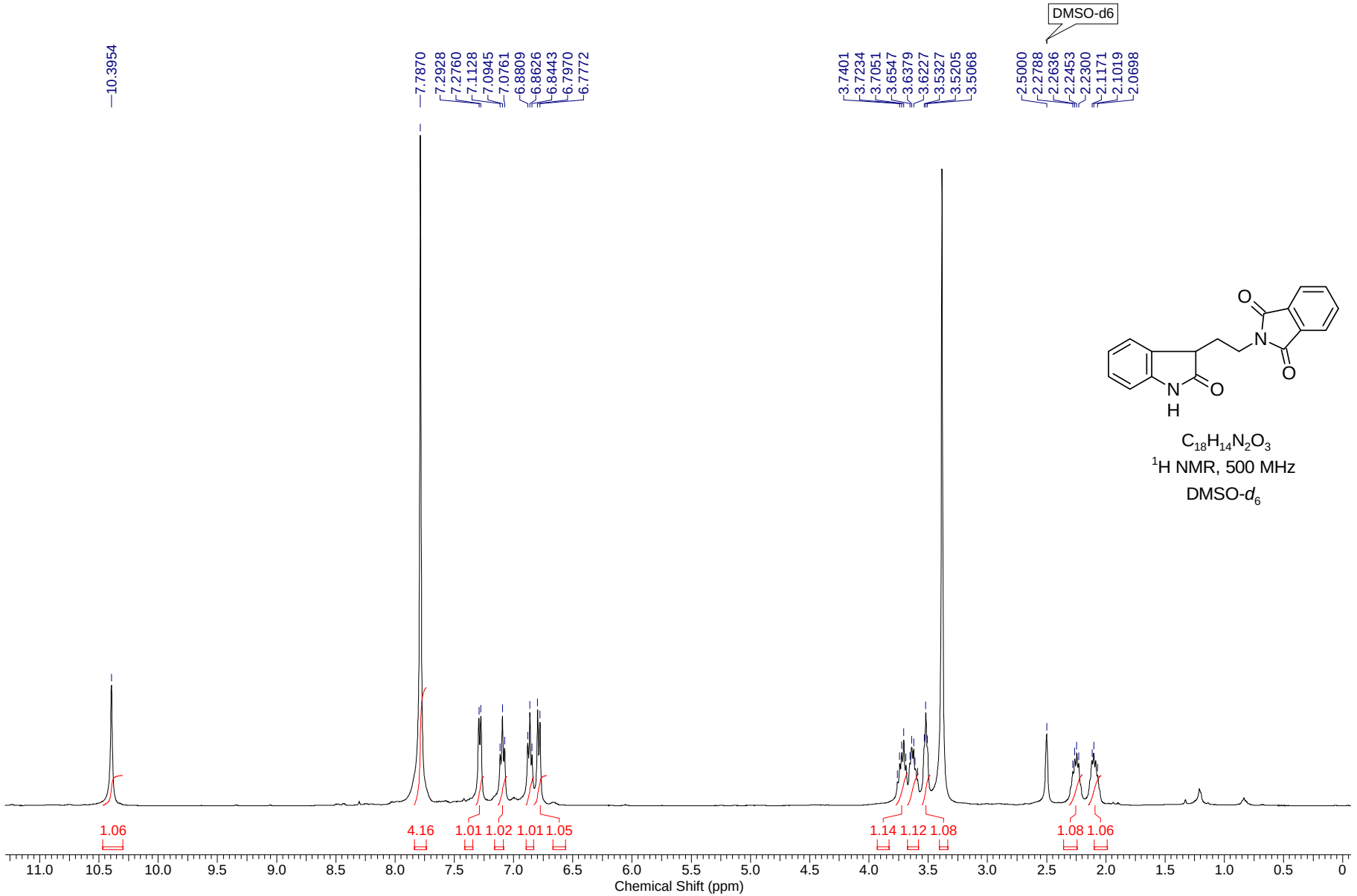
^{13}C NMR of **2p**


DMSO-d6
178.2086
170.0589
165.0726
160.8139
142.6378
128.9273
127.7094
123.8260
121.2984
109.3802
96.8951
59.1337
43.0181
39.5100
34.1560
28.1509
OMe
O
N
O
O
N
H
O
$C_{15}H_{14}N_2O_4$
^{13}C NMR, 100 MHz
DMSO-d_6
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 of **2p**


127.7094
123.8260
121.2984
109.3725
96.8951
59.1337
43.0180
34.1483
28.1509
OMe
O
N
O
N
H
O
$C_{15}H_{14}N_2O_4$
DEPT, 100 MHz
DMSO-d_6
130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
Chemical Shift (ppm)

^{1}H NMR of **2q**

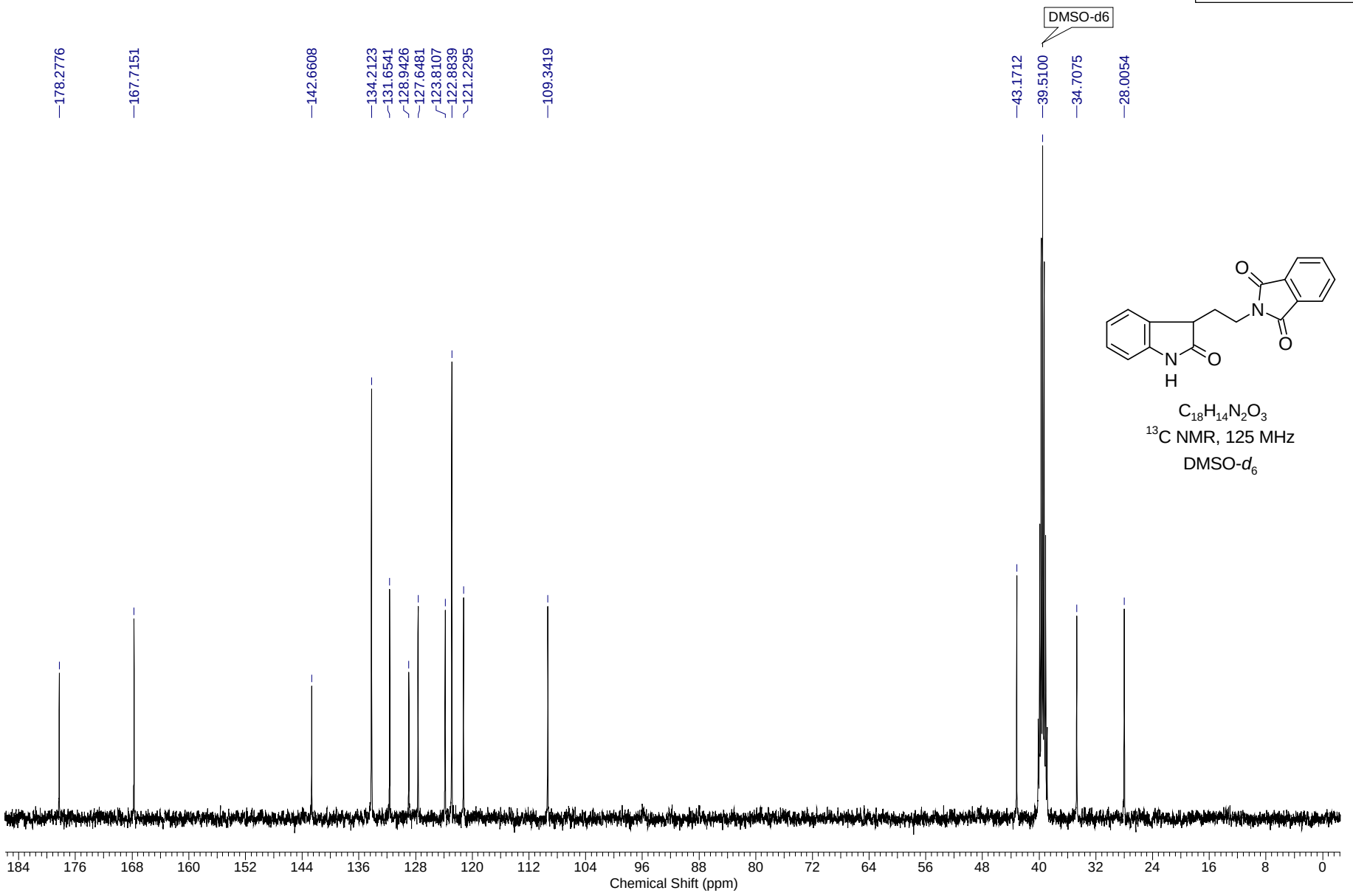


$C_{18}H_{14}N_2O_3$

^{1}H NMR, 500 MHz

DMSO-d_6

^{13}C NMR of **2q**

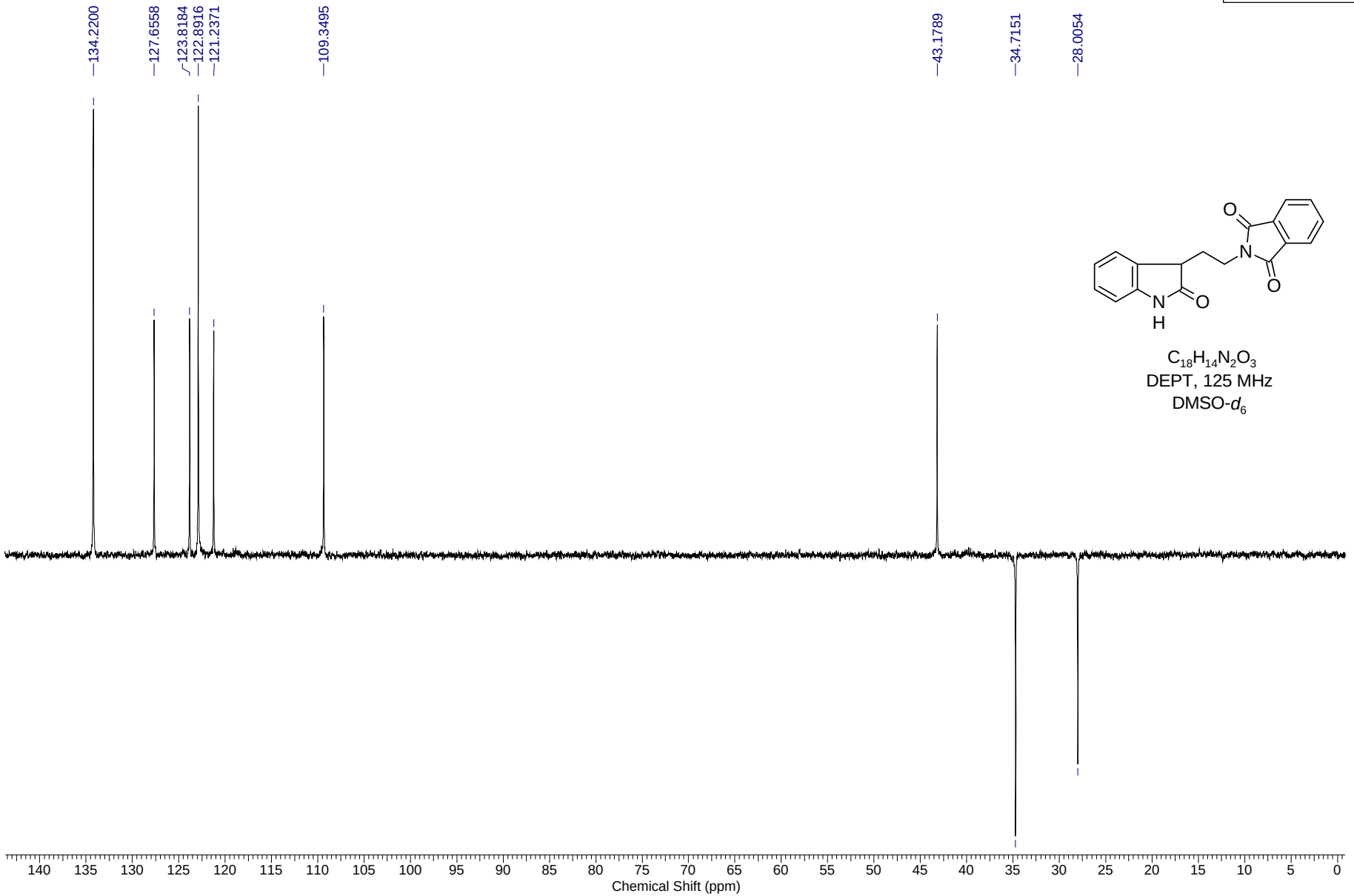


$C_{18}H_{14}N_2O_3$

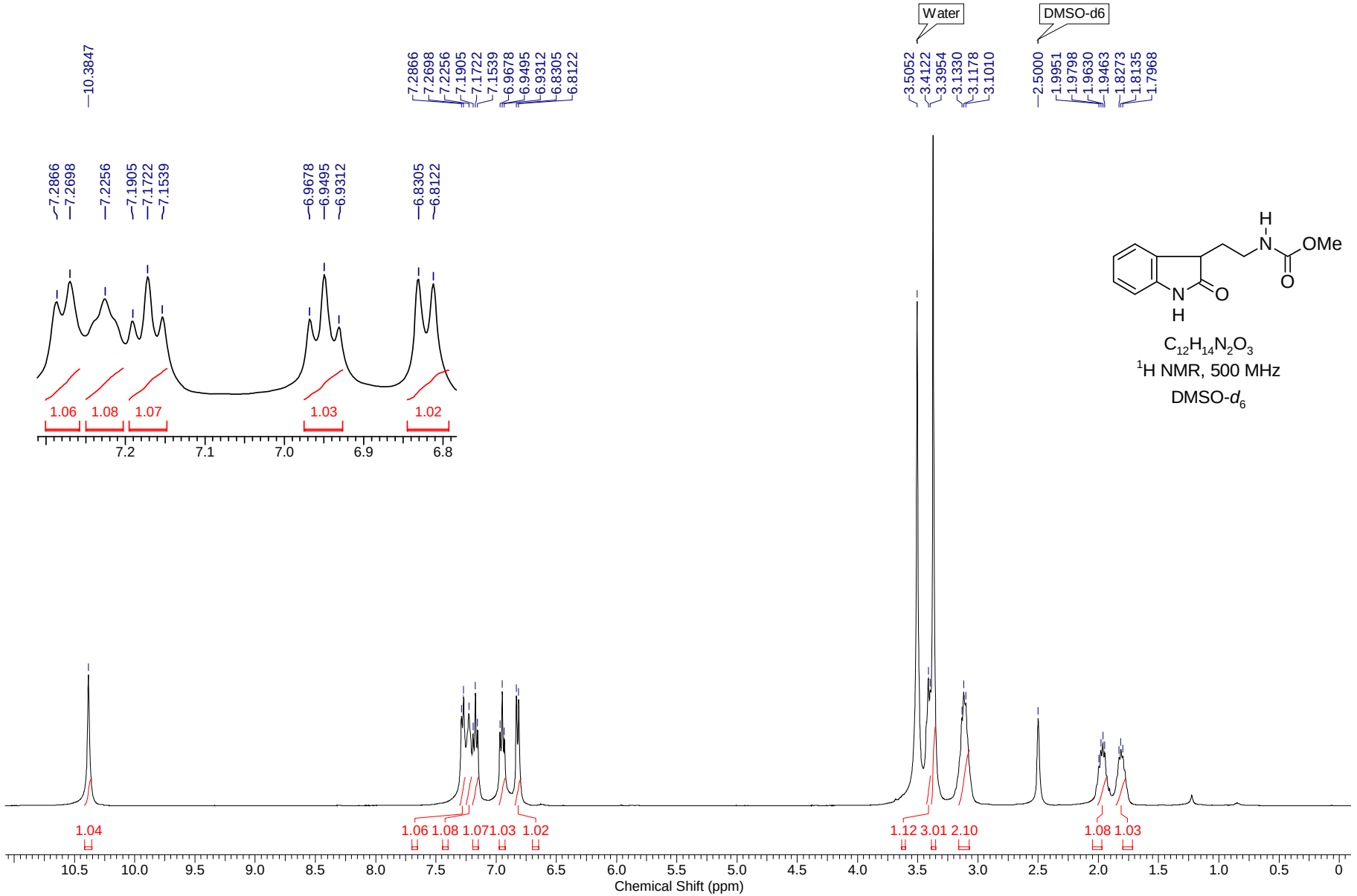
^{13}C NMR, 125 MHz

DMSO-d_6

DEPT of **2q**


134.2200
127.6558
123.8184
122.8916
121.2371
109.3495
43.1789
34.7151
28.0054
O
N
O
O
N
H
$C_{18}H_{14}N_2O_3$
DEPT, 125 MHz
DMSO-d_6
140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
Chemical Shift (ppm)

^{1}H NMR of **2t**

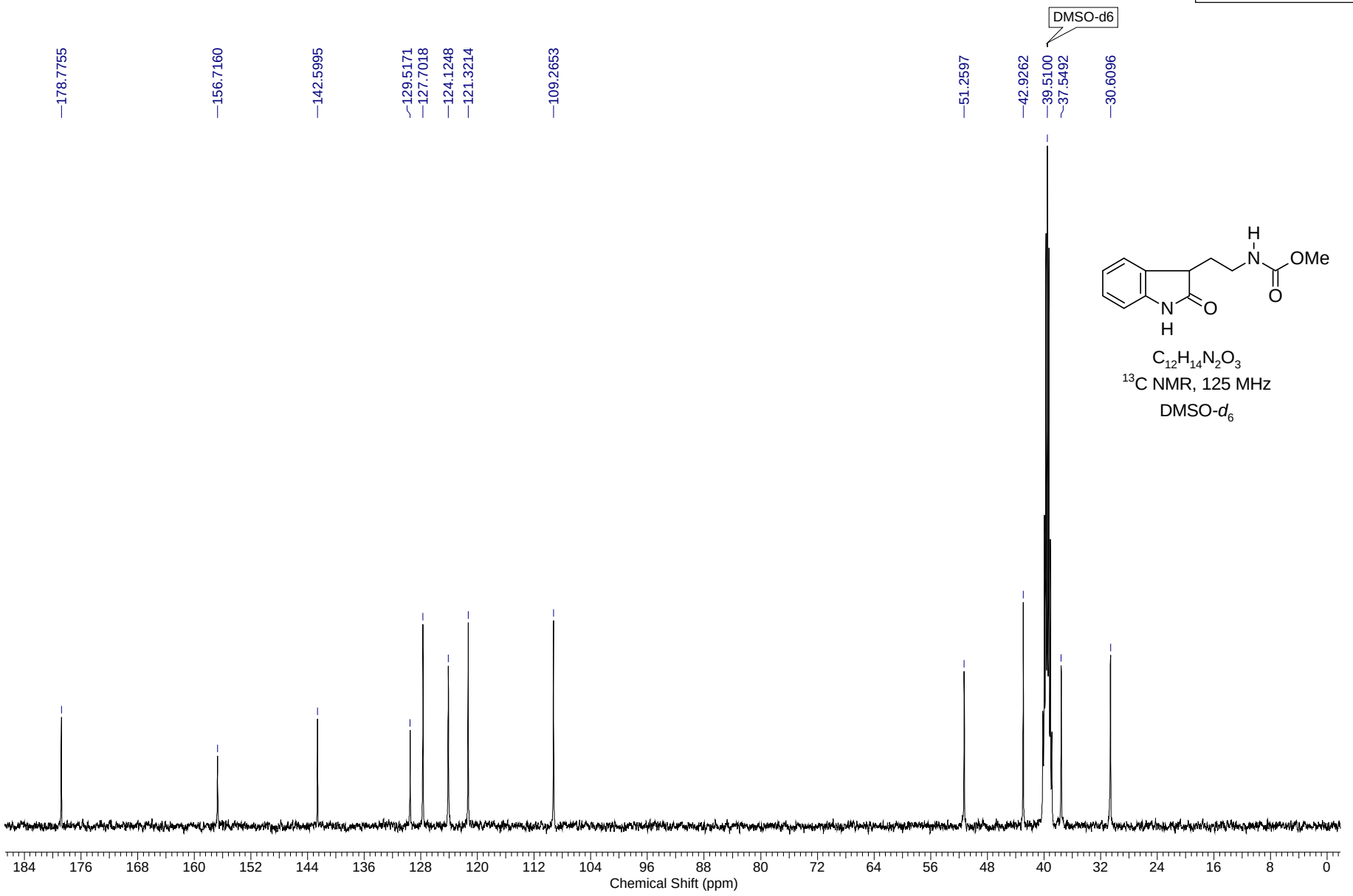


$C_{12}H_{14}N_2O_3$

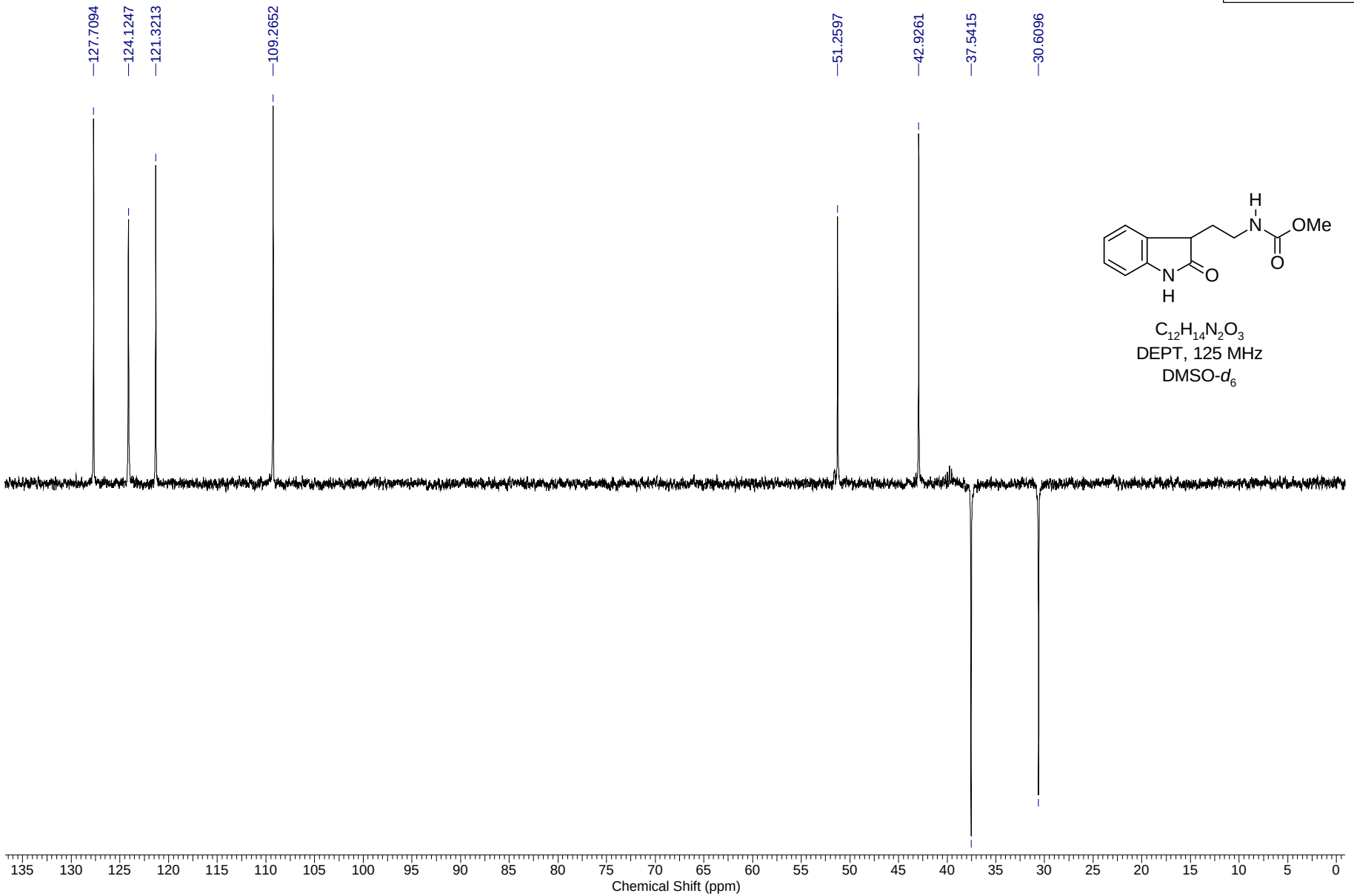
^{1}H NMR, 500 MHz

DMSO-d_6

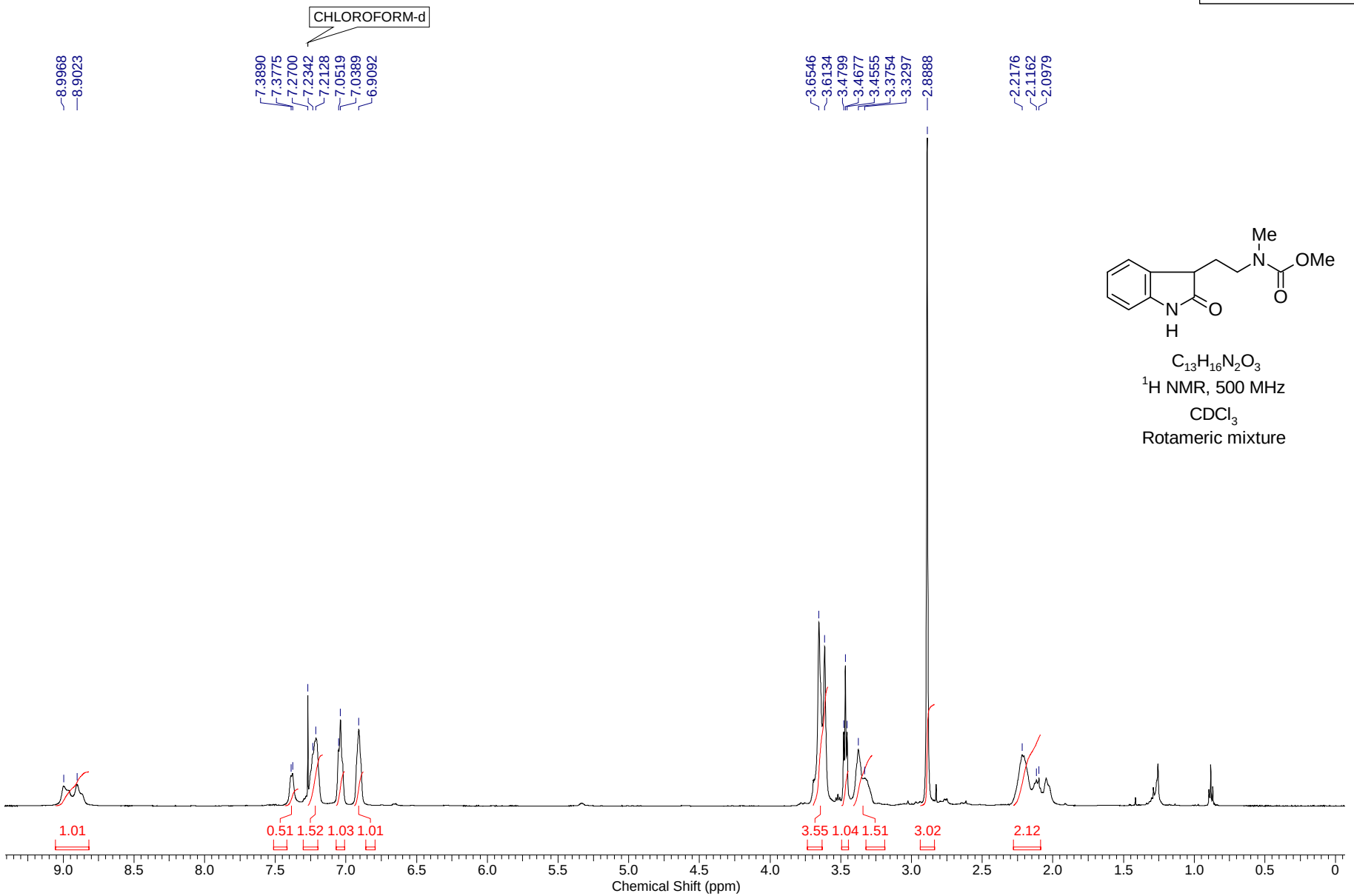
^{13}C NMR of **2t**


DMSO-d6
178.7755
156.7160
142.5995
129.5171
127.7018
124.1248
121.3214
109.2653
51.2597
42.9262
39.5100
37.5492
30.6096
H
N
OMe
O
N
H
O
C12H14N2O3
13C NMR, 125 MHz
DMSO-d6
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 of **2t**


127.7094
124.1247
121.3213
109.2652
51.2597
42.9261
37.5415
30.6096
H
N
OMe
O
N
H
O
C12H14N2O3
DEPT, 125 MHz
DMSO-d6
135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
Chemical Shift (ppm)

^{1}H NMR of **2u**



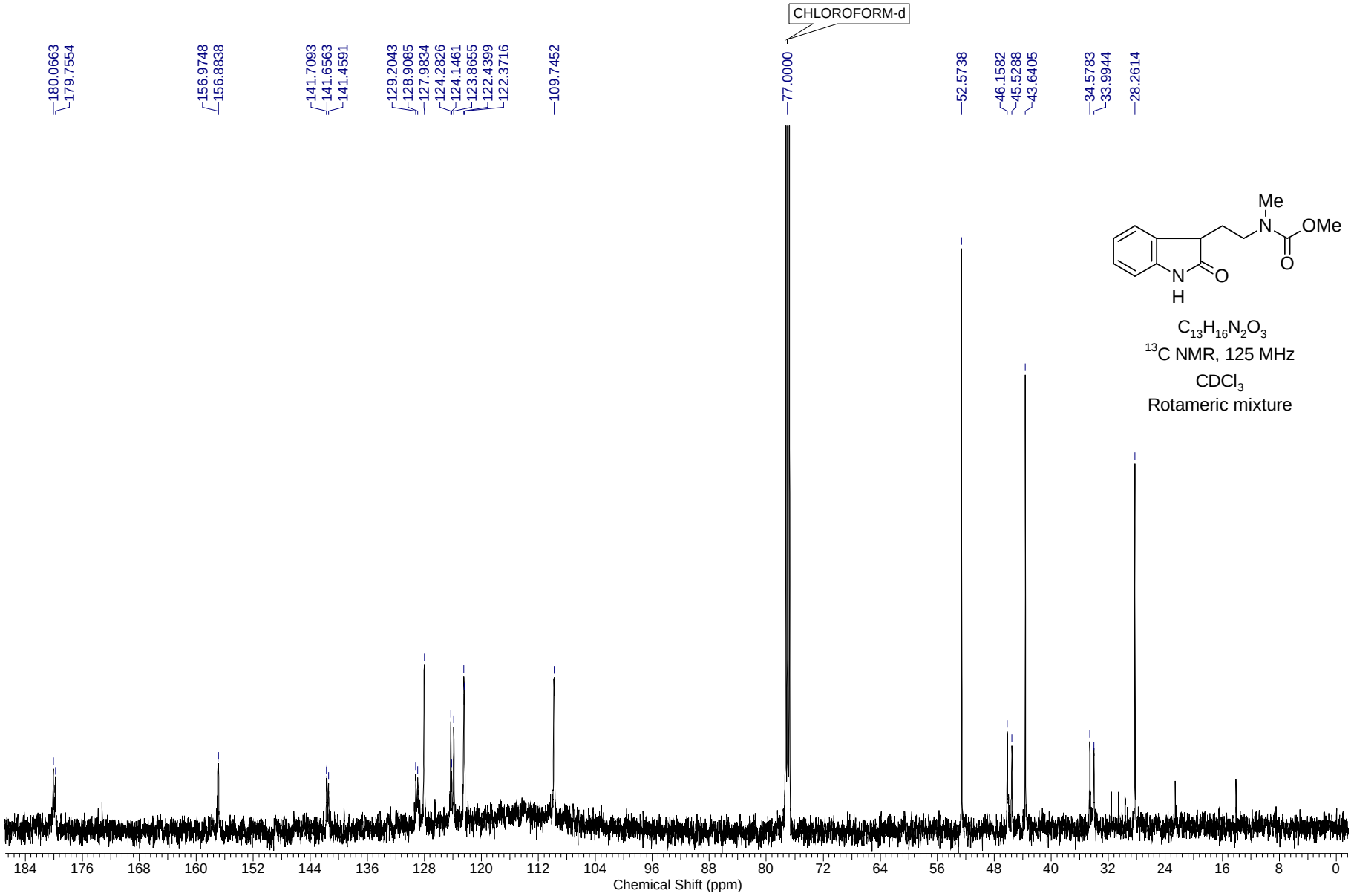
$C_{13}H_{16}N_2O_3$

^{1}H NMR, 500 MHz

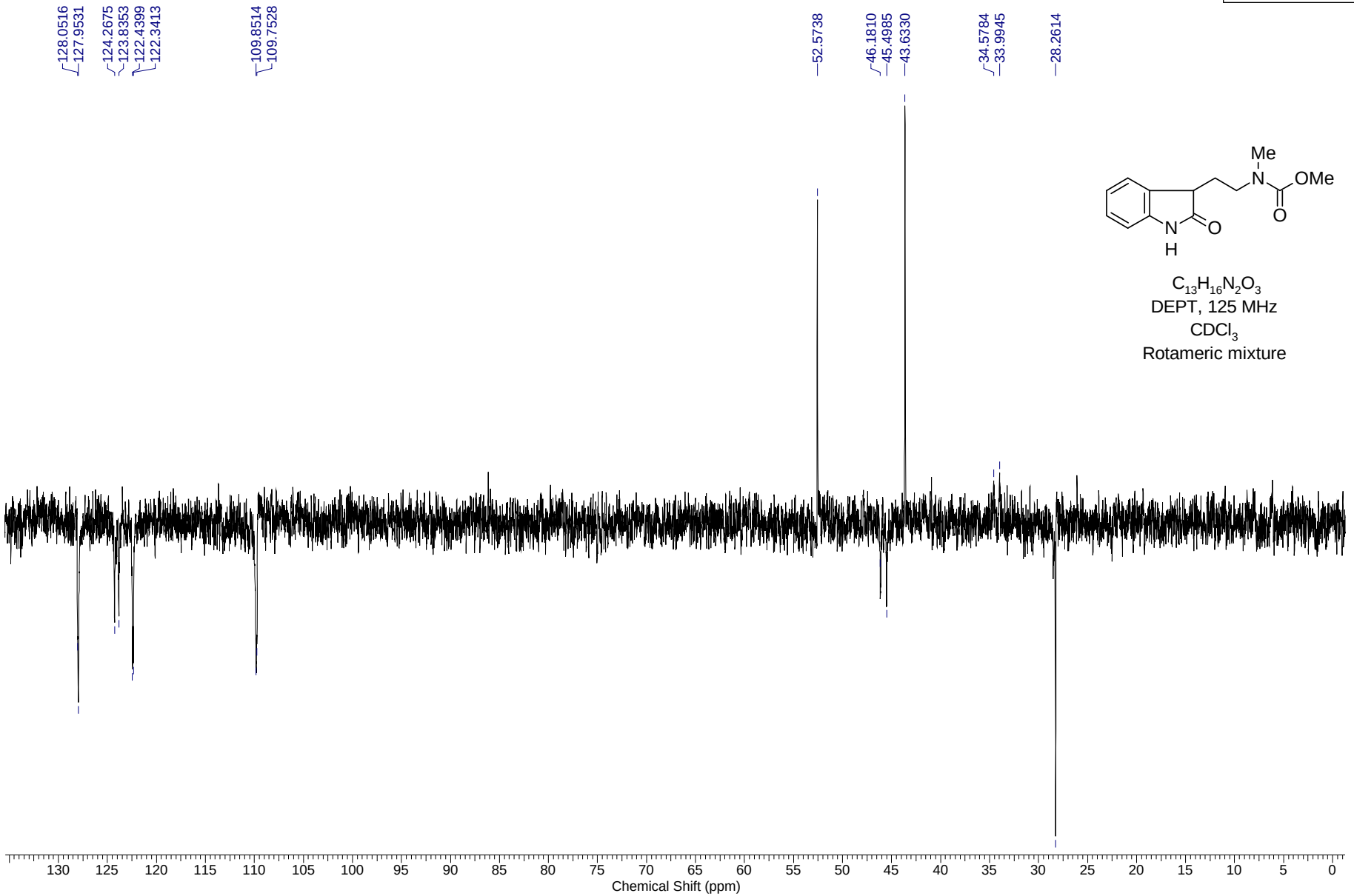
$CDCl_3$

Rotameric mixture

^{13}C NMR of **2u**

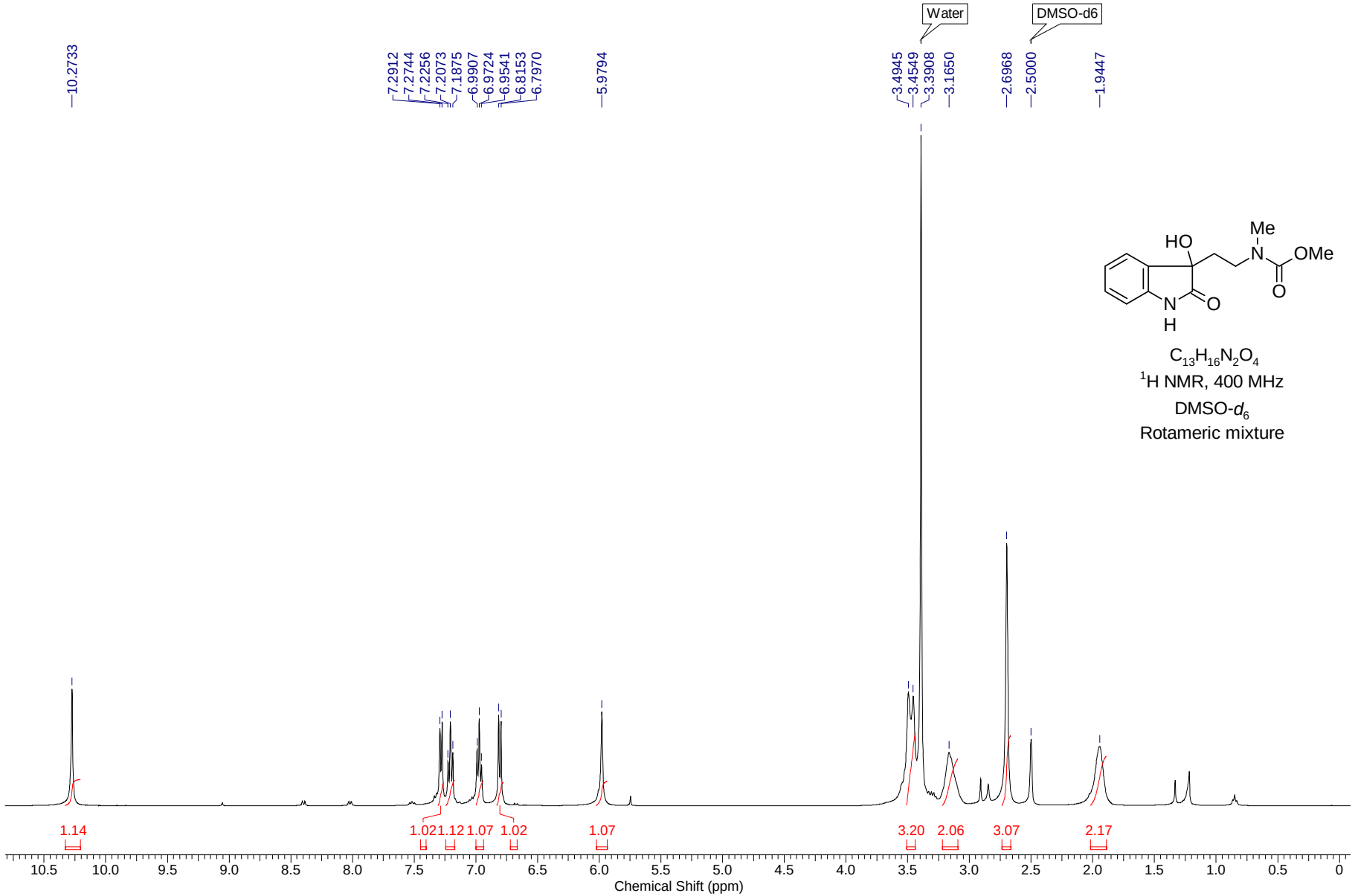

CHLOROFORM-d
180.0663
179.7554
156.9748
156.8838
141.7093
141.6563
141.4591
129.2043
128.9085
127.9834
124.2826
124.1461
123.8655
122.4399
122.3716
109.7452
77.0000
52.5738
46.1582
45.5288
43.6405
34.5783
33.9944
28.2614
Me
N
OMe
O
N
H
O
$C_{13}H_{16}N_2O_3$
^{13}C NMR, 125 MHz
$CDCl_3$
Rotameric mixture
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 of **2u**



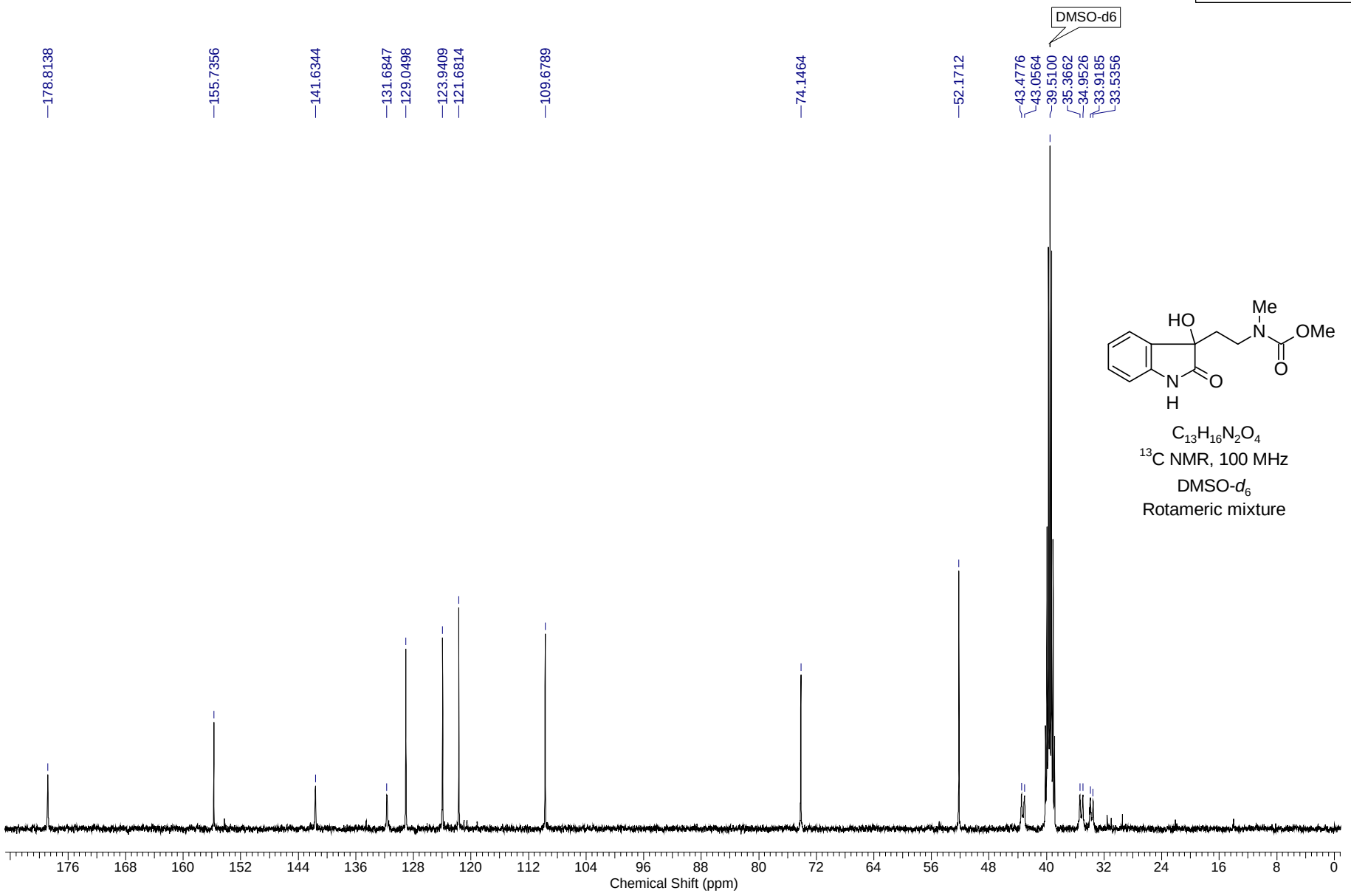
$C_{13}H_{16}N_2O_3$
DEPT, 125 MHz
$CDCl_3$
Rotameric mixture

^{1}H NMR of **3**



$C_{13}H_{16}N_2O_4$
^{1}H NMR, 400 MHz
DMSO-d_6
Rotameric mixture

^{13}C NMR of **3**



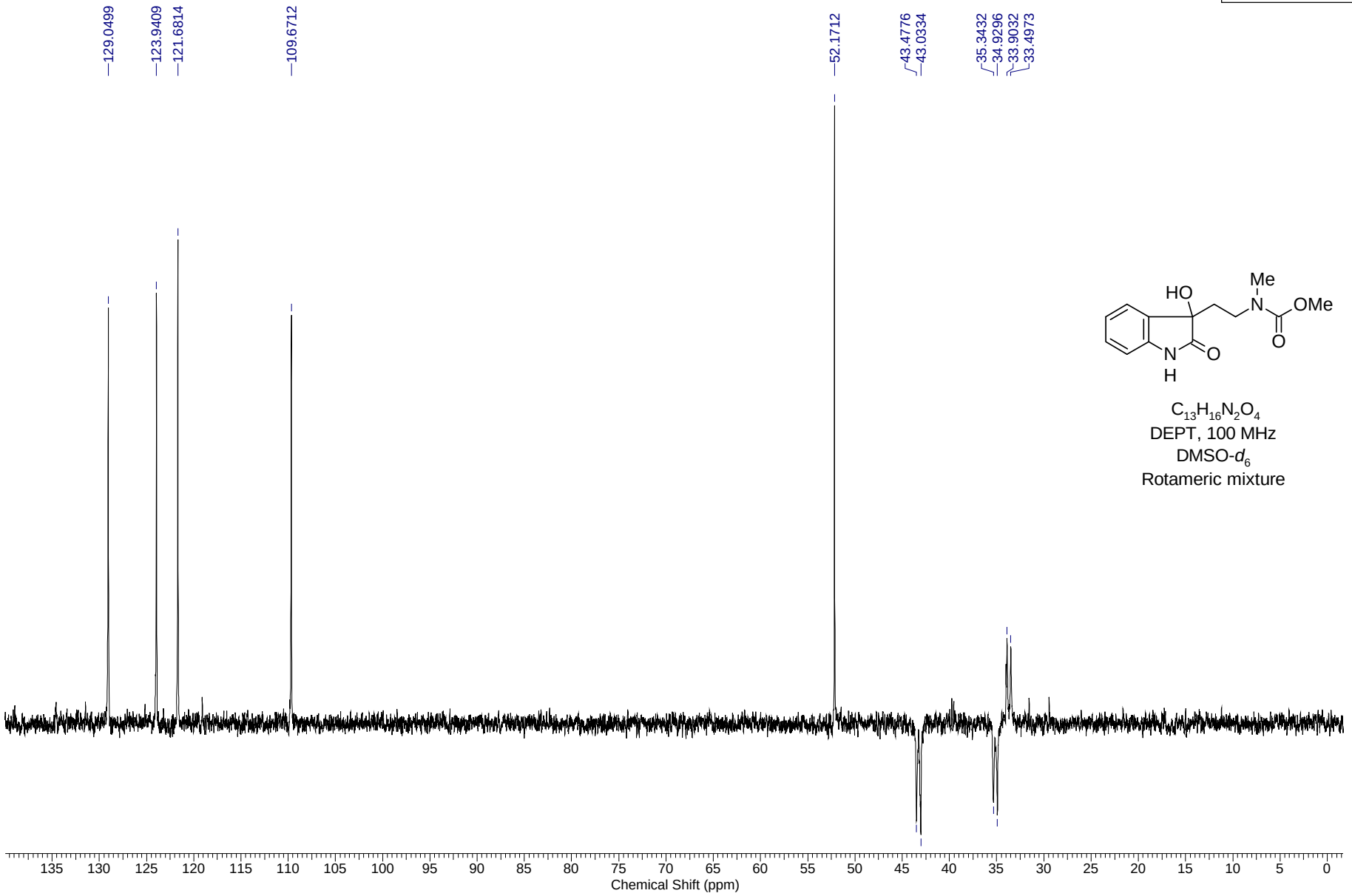
$C_{13}H_{16}N_2O_4$

^{13}C NMR, 100 MHz

DMSO-d_6

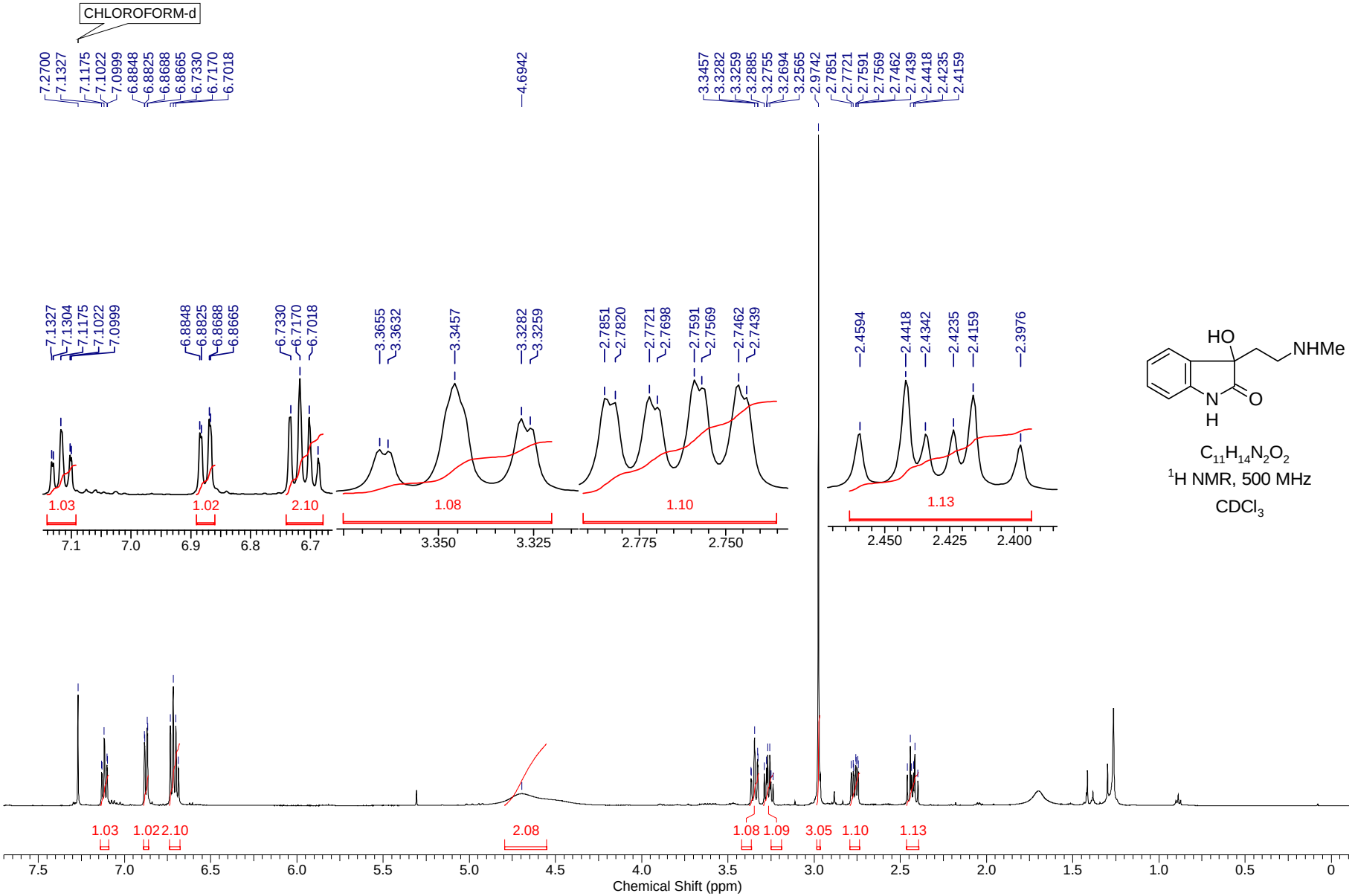
Rotameric mixture

DEPT of **3**



$C_{13}H_{16}N_2O_4$
DEPT, 100 MHz
DMSO-d_6
Rotameric mixture

^{1}H NMR of **4**

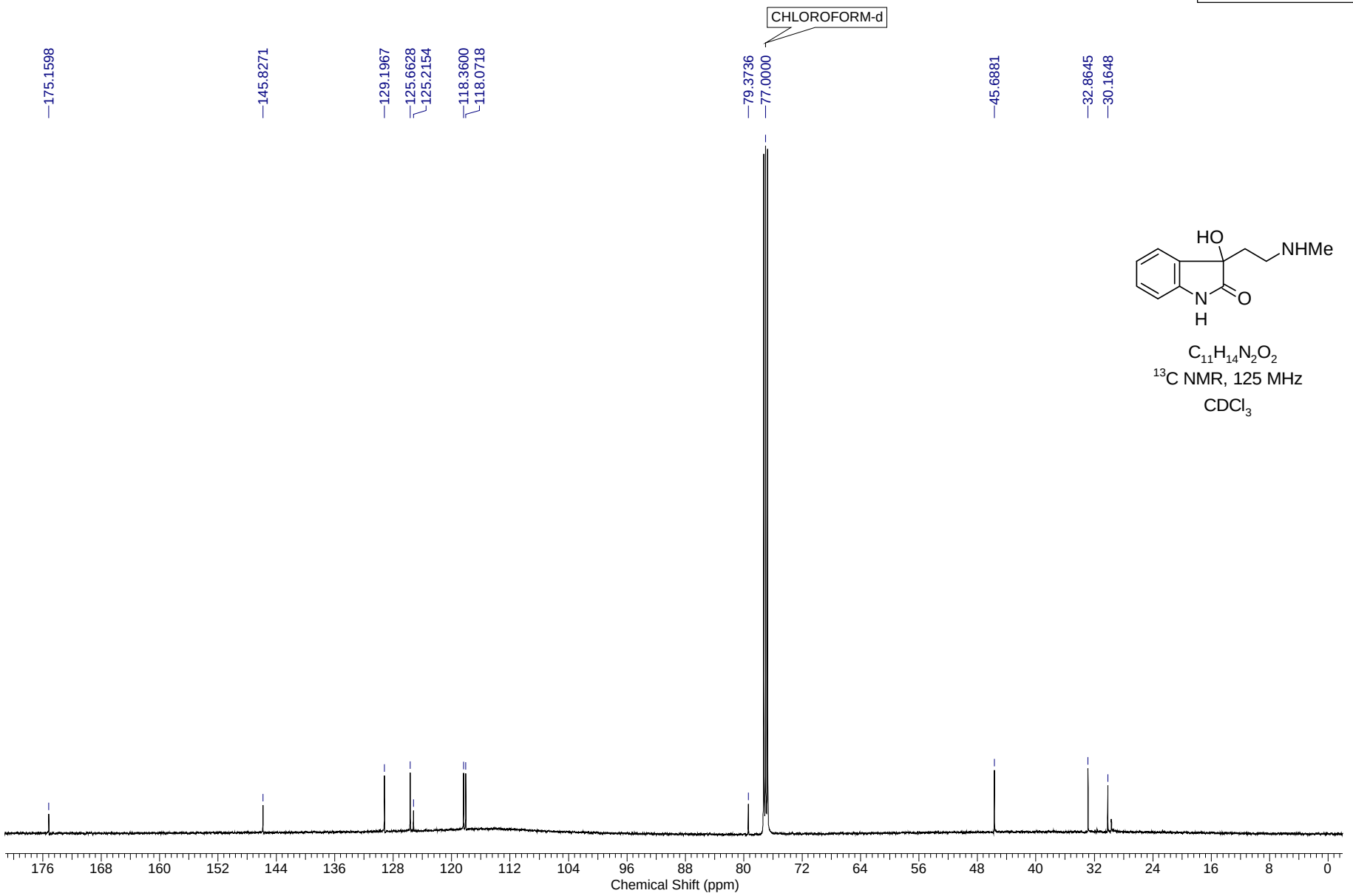


$C_{11}H_{14}N_2O_2$

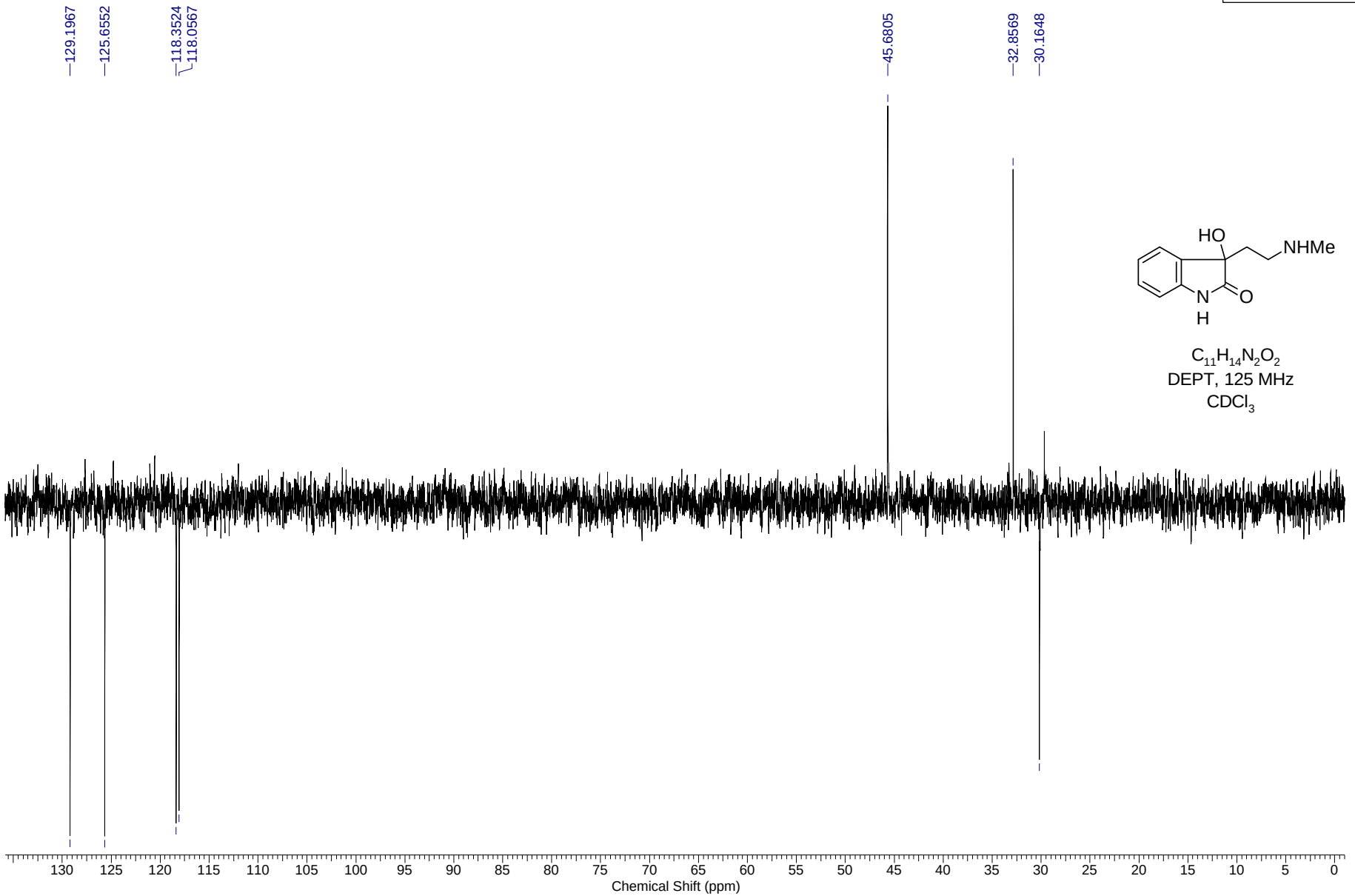
^{1}H NMR, 500 MHz

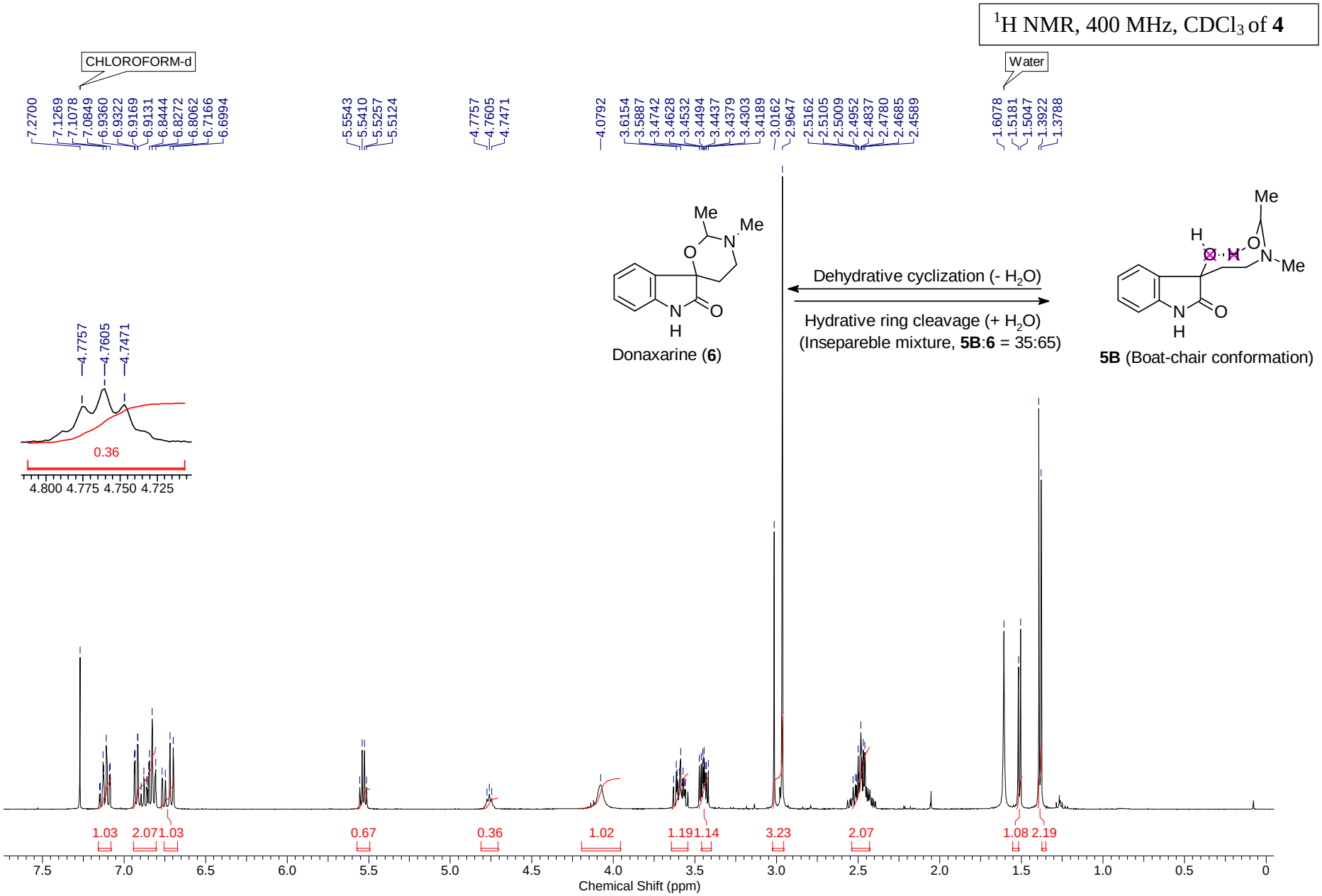
$CDCl_3$

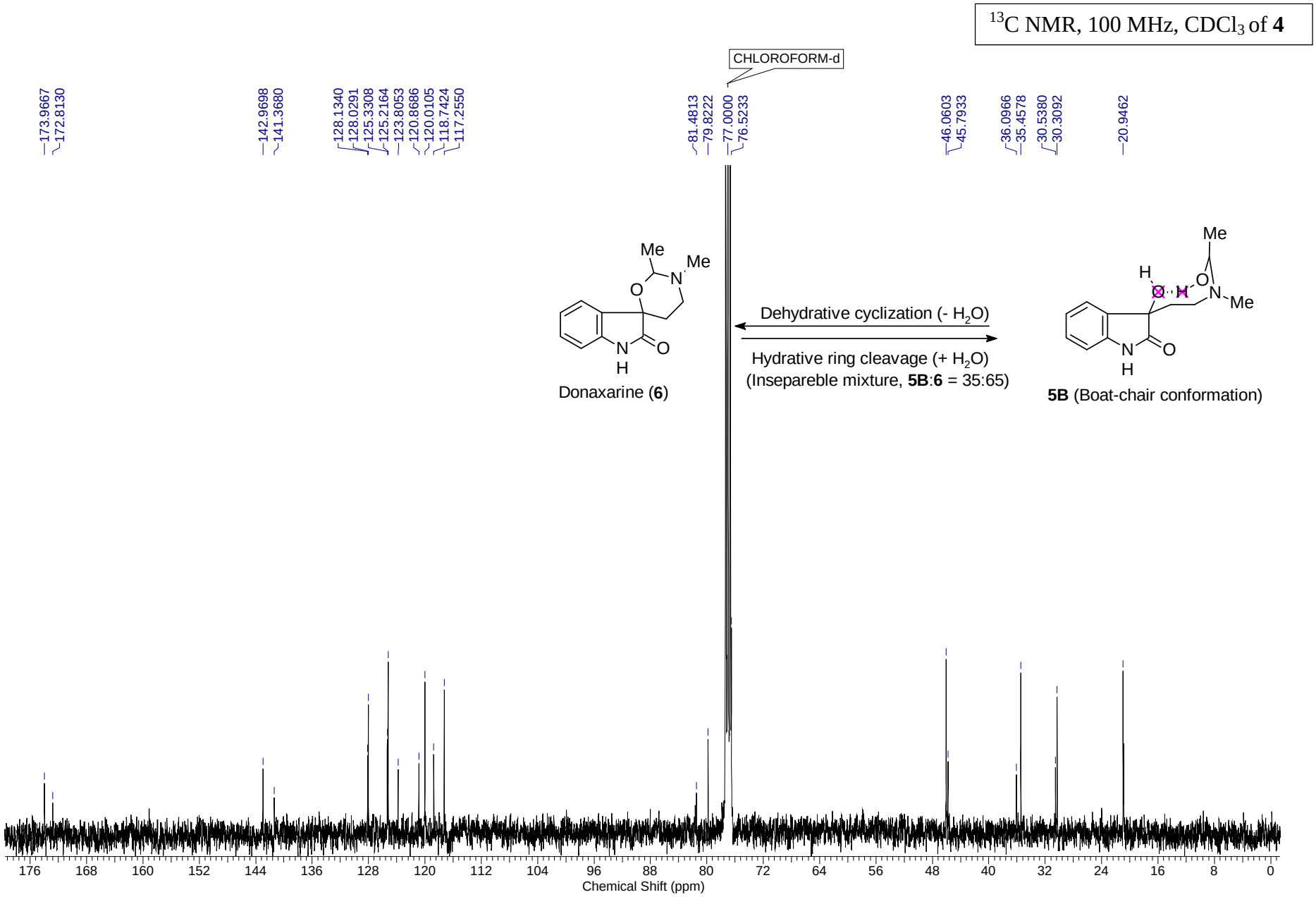
^{13}C NMR of **4**


CHLOROFORM-d
175.1598
145.8271
129.1967
125.6628
125.2154
118.3600
118.0718
79.3736
77.0000
45.6881
32.8645
30.1648
HO
NHMe
N
H
O
$C_{11}H_{14}N_2O_2$
^{13}C NMR, 125 MHz
$CDCl_3$
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)

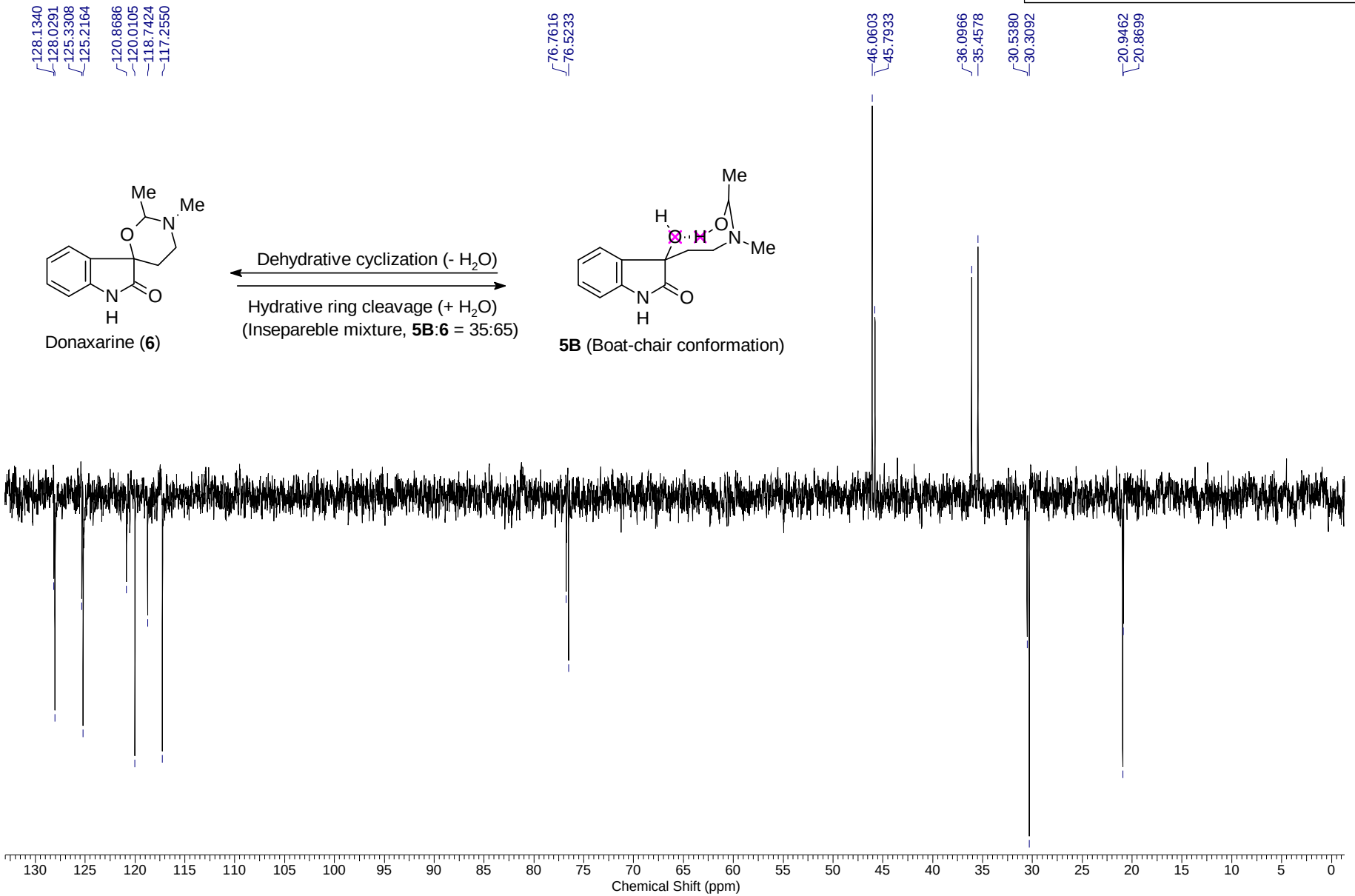
DEPT of **4**


129.1967
125.6552
118.3524
118.0567
45.6805
32.8569
30.1648
HO
NHMe
N
H
O
$C_{11}H_{14}N_2O_2$
DEPT, 125 MHz
$CDCl_3$
130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
Chemical Shift (ppm)

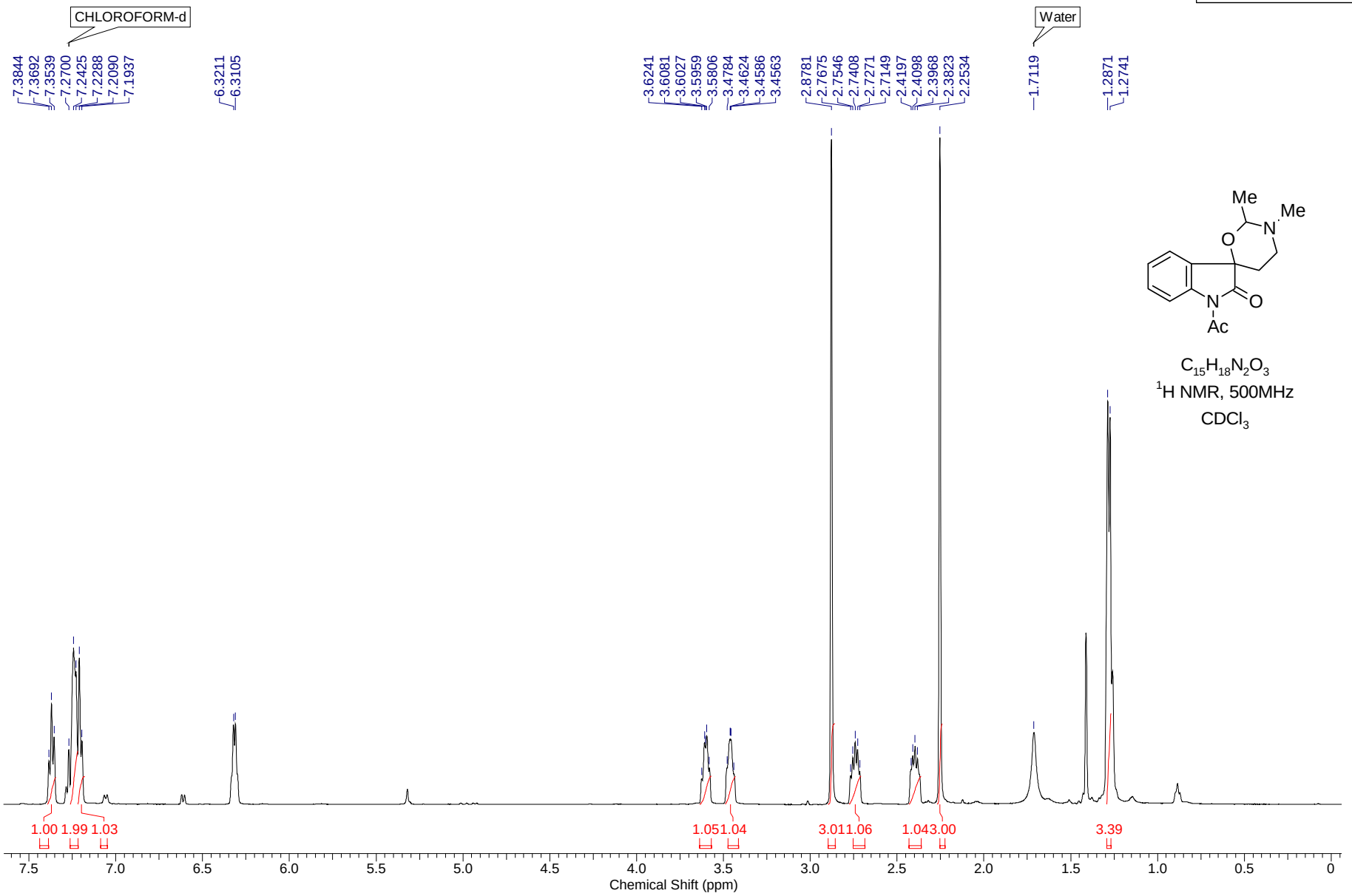

^{1}H NMR, 400 MHz, $CDCl_3$ of **4**
CHLOROFORM-d
Water
7.2700 7.1269 7.1078 7.0849 6.9360 6.9322 6.9169 6.9131 6.8444 6.8272 6.8062 6.7166 6.6994
5.5543 5.5410 5.5257 5.5124
4.7757 4.7605 4.7471
4.0792
3.6154 3.5887 3.4742 3.4628 3.4532 3.4494 3.4437 3.4379 3.4303 3.4189
3.0162
2.9647
2.5162 2.5105 2.5009 2.4952 2.4837 2.4780 2.4685 2.4589
1.6078 1.5181 1.5047 1.3922 1.3788
Donaxarine (**6**)
Dehydrative cyclization (- H_2O)
Hydrative ring cleavage (+ H_2O)
(Inseparable mixture, **5B**:**6** = 35:65)
5B (Boat-chair conformation)
0.36
4.800 4.775 4.750 4.725
1.03 2.07 1.03 0.67 0.36 1.02 1.19 1.14 3.23 2.07 1.08 2.19
Chemical Shift (ppm)


^{13}C NMR, 100 MHz, $CDCl_3$ of **4**
CHLOROFORM-d
173.9667
172.8130
142.9698
141.3680
128.1340
128.0291
125.3308
125.2164
123.8053
120.8686
120.0105
118.7424
117.2550
81.4813
79.8222
77.0000
76.5233
46.0603
45.7933
36.0966
35.4578
30.5380
30.3092
20.9462
Me
Me
N
O
N
H
O
Donaxarine (**6**)
Dehydrative cyclization (- H_2O)
Hydrative ring cleavage (+ H_2O)
(Inseparable mixture, **5B**:**6** = 35:65)
Me
H
O
H
O
N
Me
N
H
O
5B (Boat-chair conformation)
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)

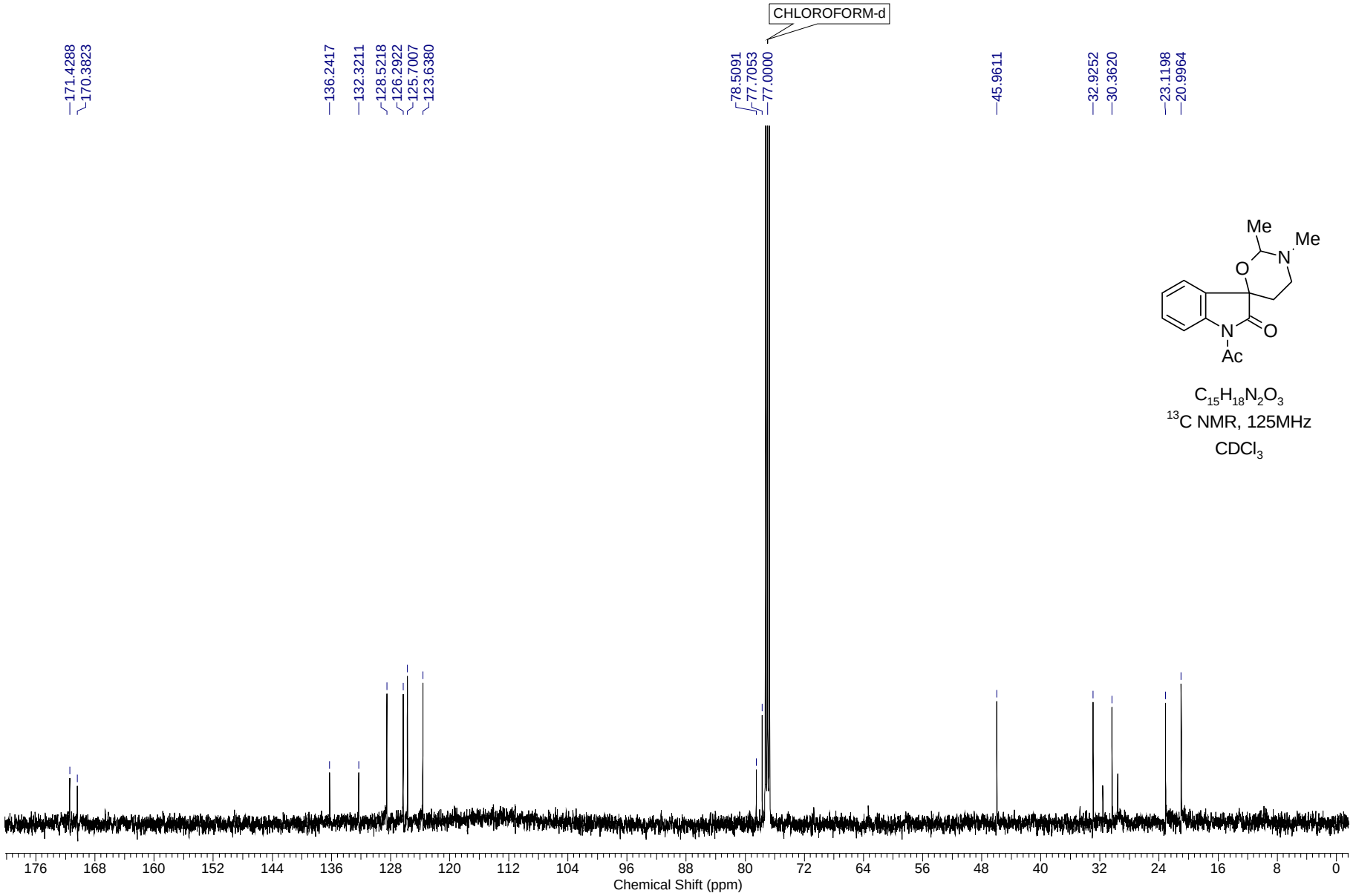
DEPT, 100 MHz, $CDCl_3$ of **4**



^{1}H NMR of **7**


CHLOROFORM-d
Water
7.3844
7.3692
7.3539
7.2700
7.2425
7.2288
7.2090
7.1937
6.3211
6.3105
3.6241
3.6081
3.6027
3.5959
3.5806
3.4784
3.4624
3.4586
3.4563
2.8781
2.7675
2.7546
2.7408
2.7271
2.7149
2.4197
2.4098
2.3968
2.3823
2.2534
1.7119
1.2871
1.2741
Me
Me
N
O
N
O
Ac
$C_{15}H_{18}N_2O_3$
^{1}H NMR, 500MHz
$CDCl_3$
1.00 1.99 1.03
1.05 1.04
3.01 1.06
1.04 3.00
3.39
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)

^{13}C NMR of **7**

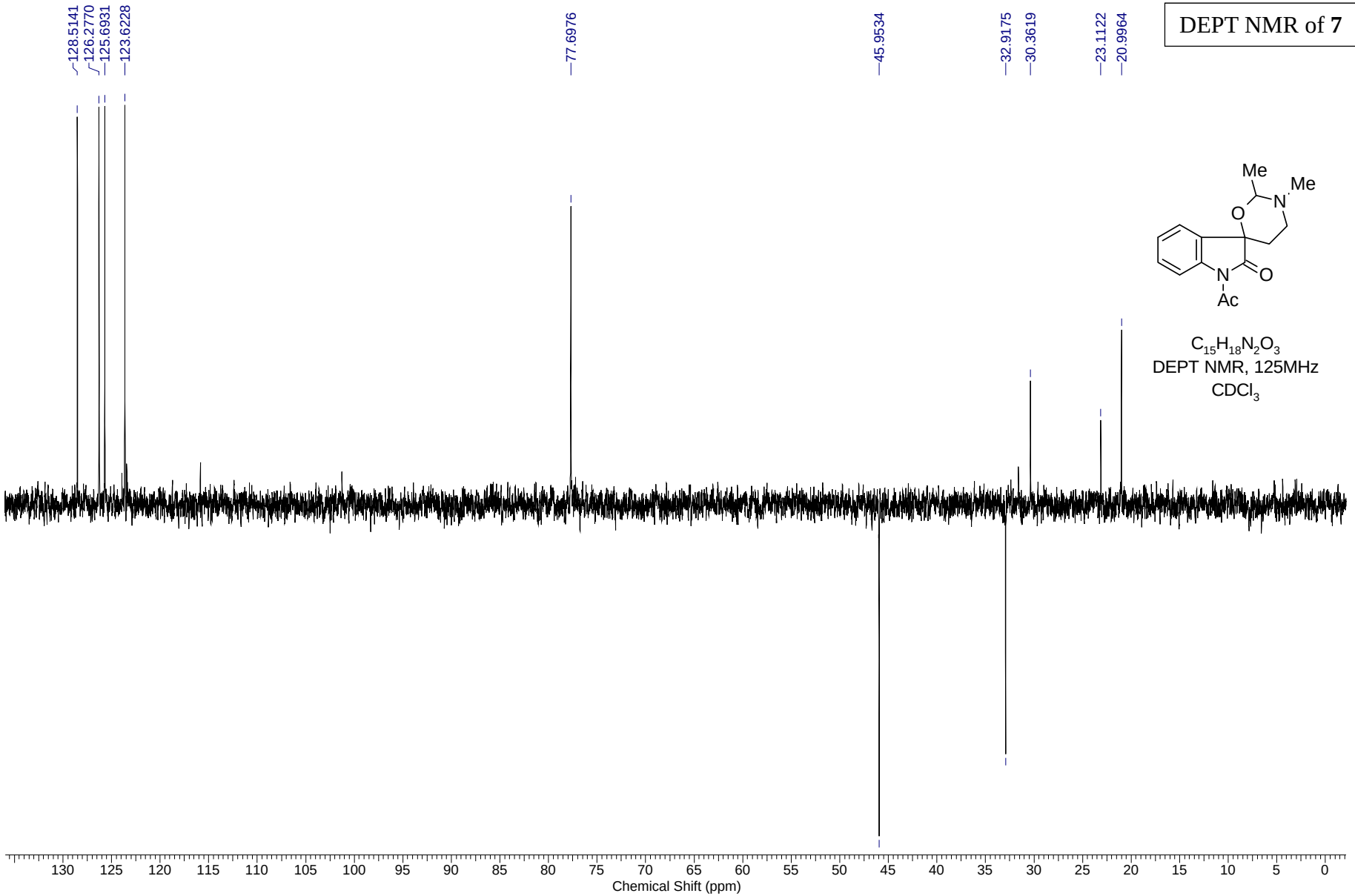


$C_{15}H_{18}N_2O_3$

^{13}C NMR, 125MHz

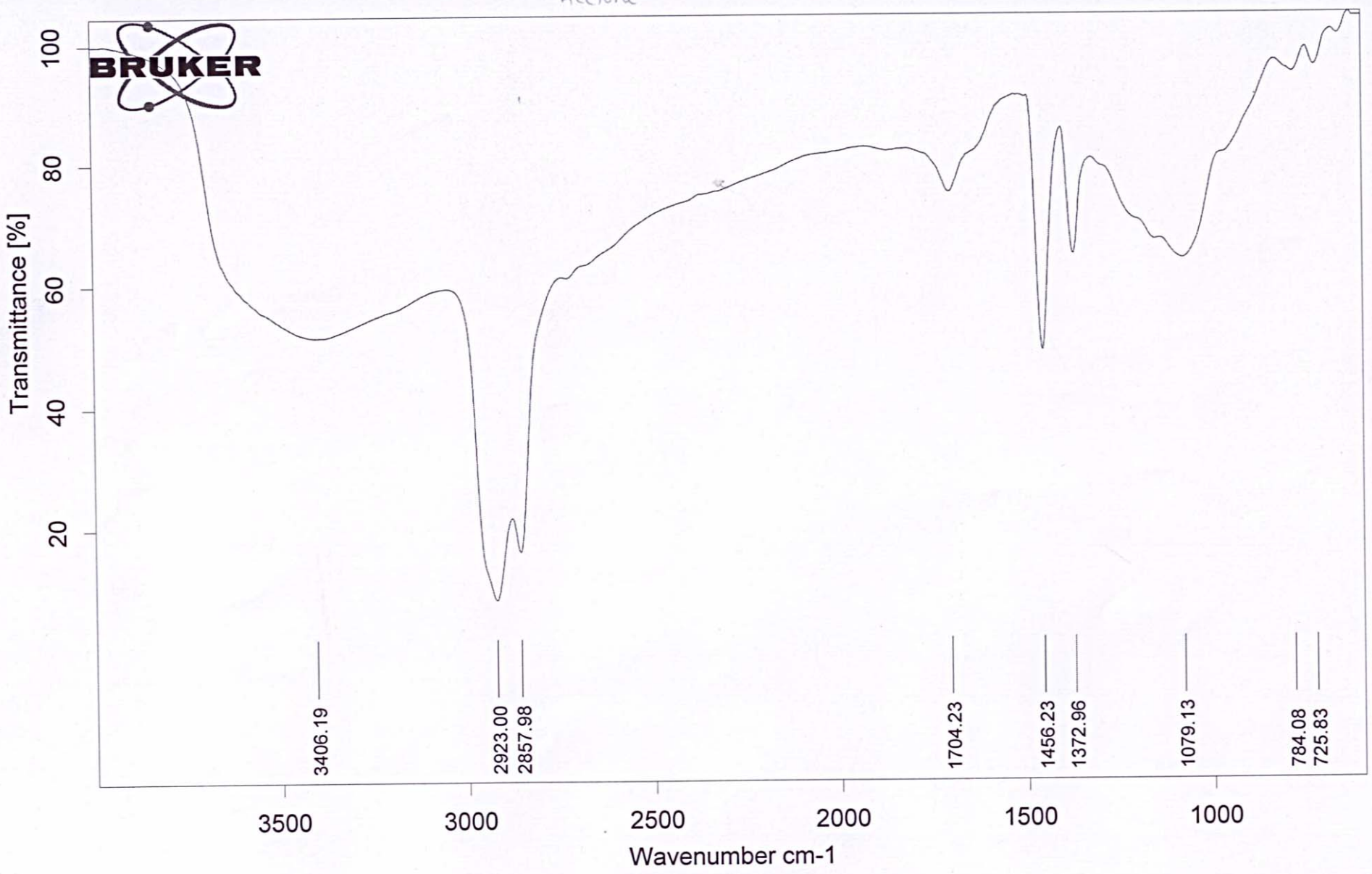
$CDCl_3$

DEPT NMR of **7**



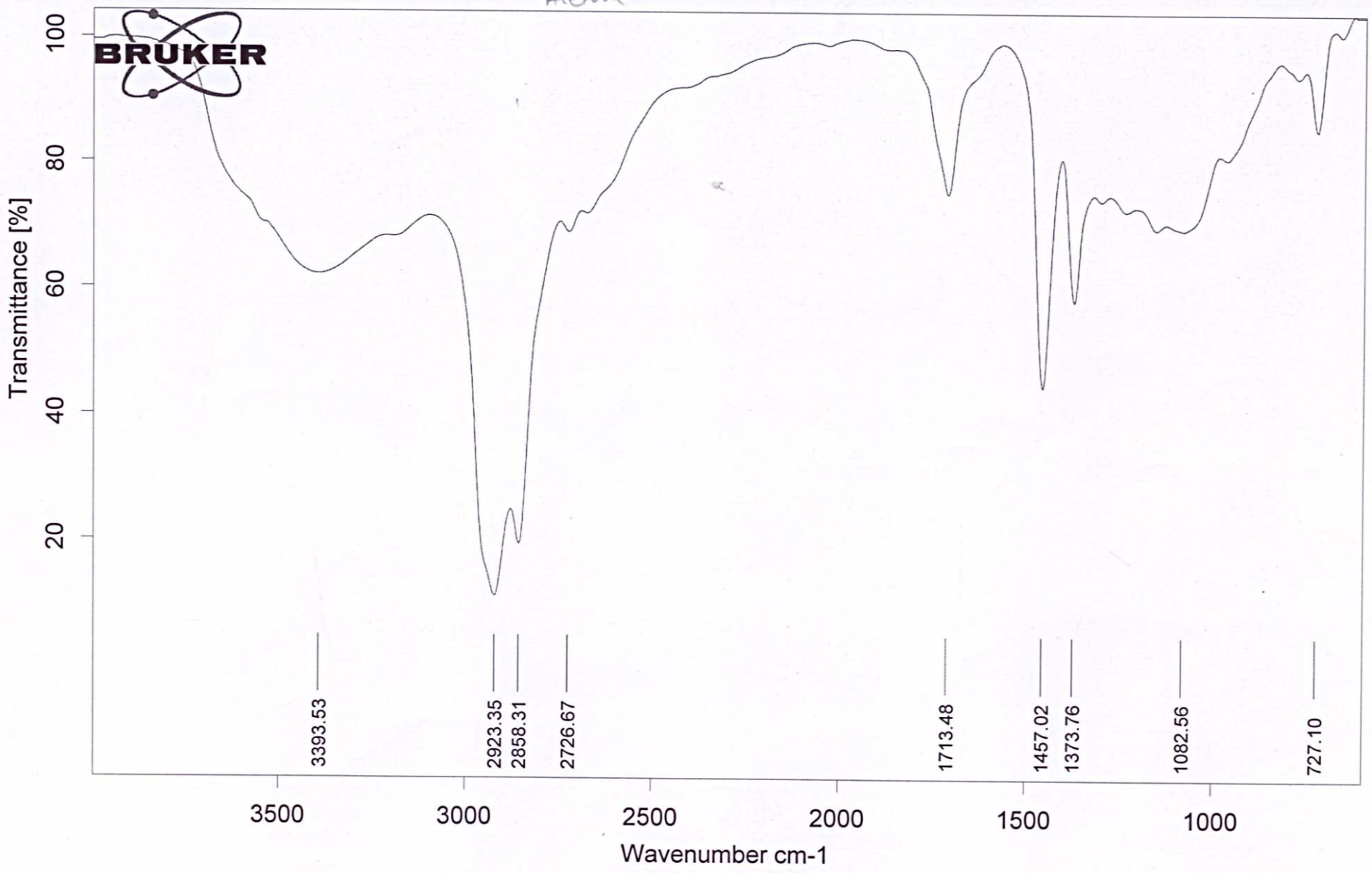
$C_{15}H_{18}N_2O_3$
DEPT NMR, 125MHz
$CDCl_3$

SiO_2 + H_2SO_4
Acetone



D:\IR SPECTRA\SV-1.4	SV-1	Instrument type and / or accessory	4/29/2019

SiO_2 + H_2SO_4 Recycle
Acetone



D:\IR SPECTRA\SV-1.3	SV-1	Instrument type and / or accessory	4/29/2019