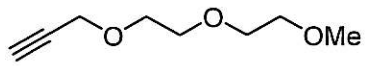
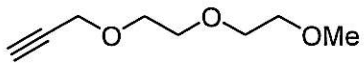
Towards Multivalent CD1d Ligands: Synthesis and Biological Activity of Homodimeric α-Galactosyl Ceramide Analogues

Peter J. Jervis *et al.*

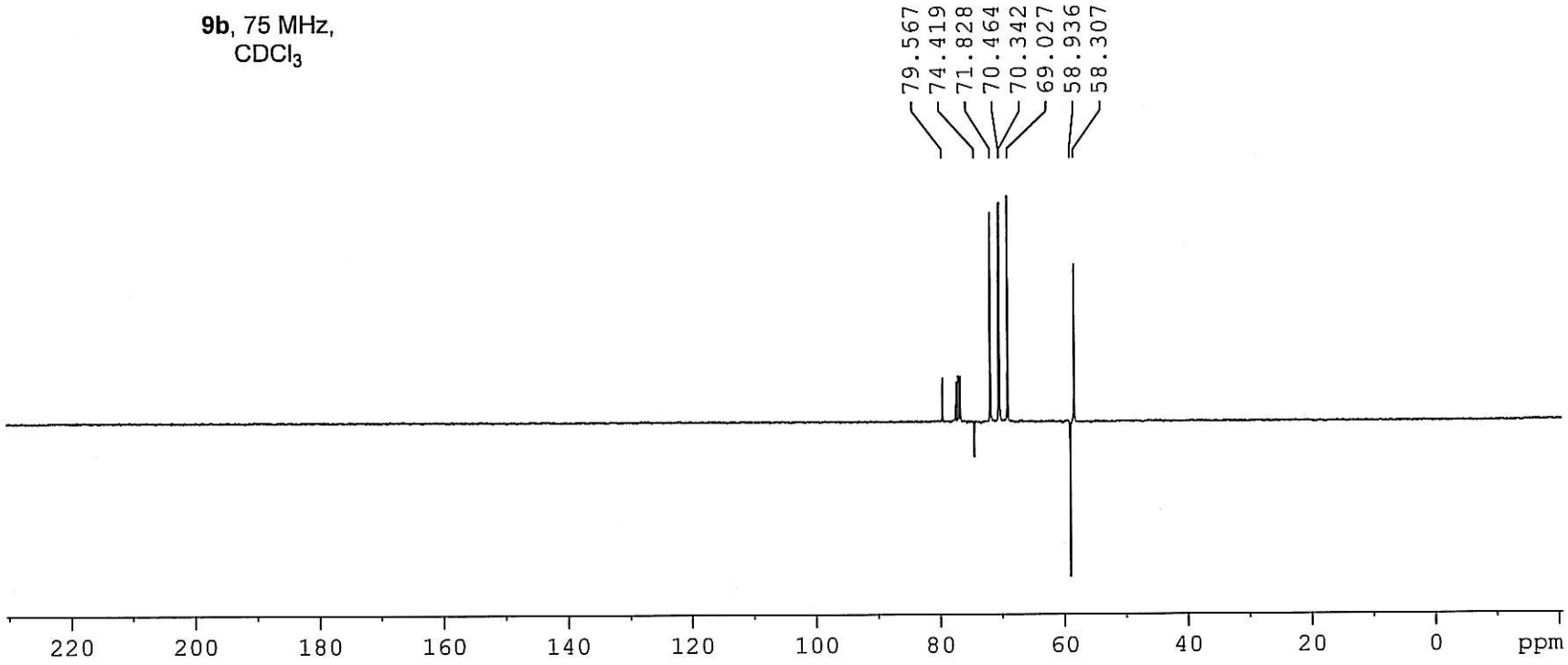
Scanned ^{1}H-NMR and ^{13}C-NMR Spectra

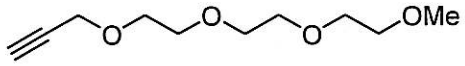


9b, 300 MHz,
$CDCl_3$

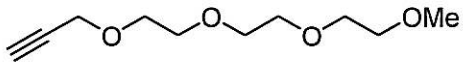


9b, 75 MHz,
$CDCl_3$

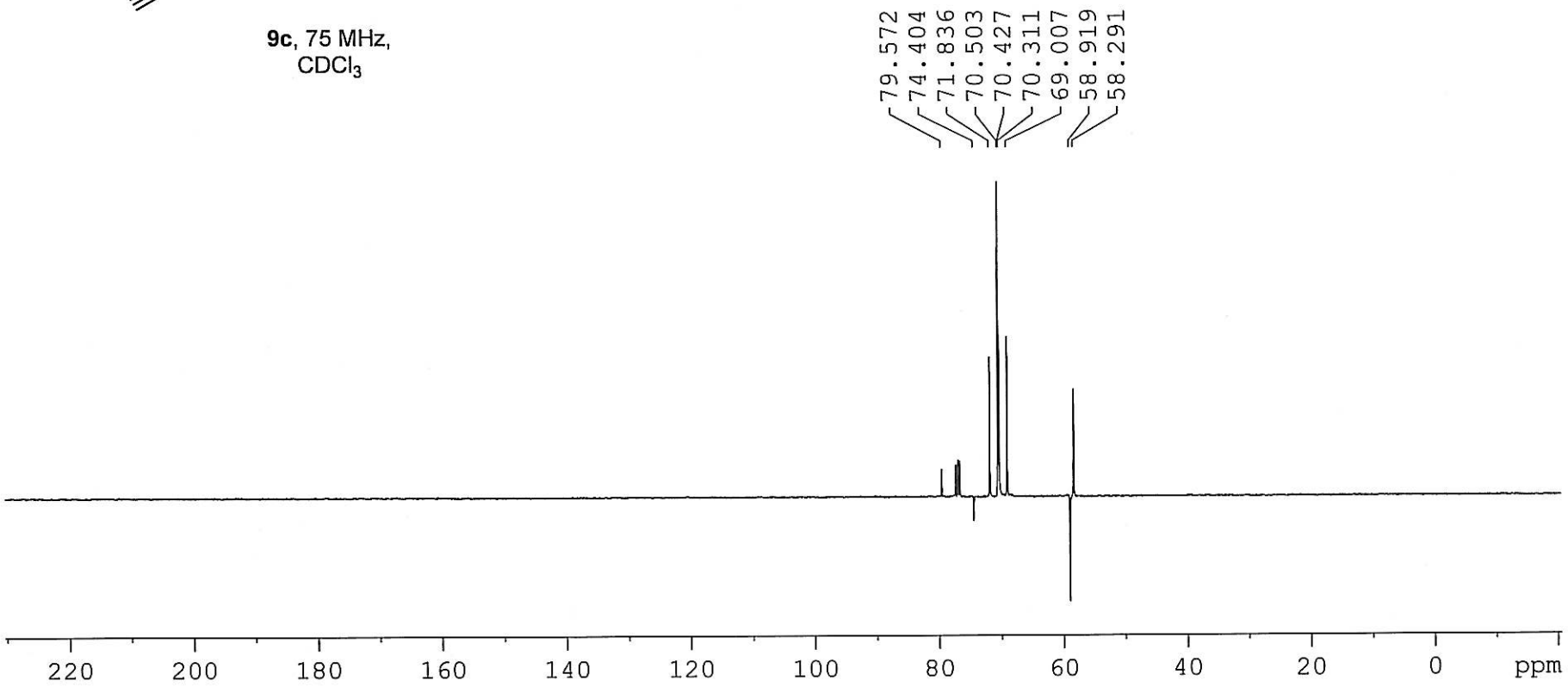


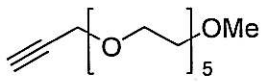


9c, 300 MHz,
$CDCl_3$

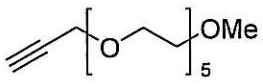


9c, 75 MHz,
$CDCl_3$

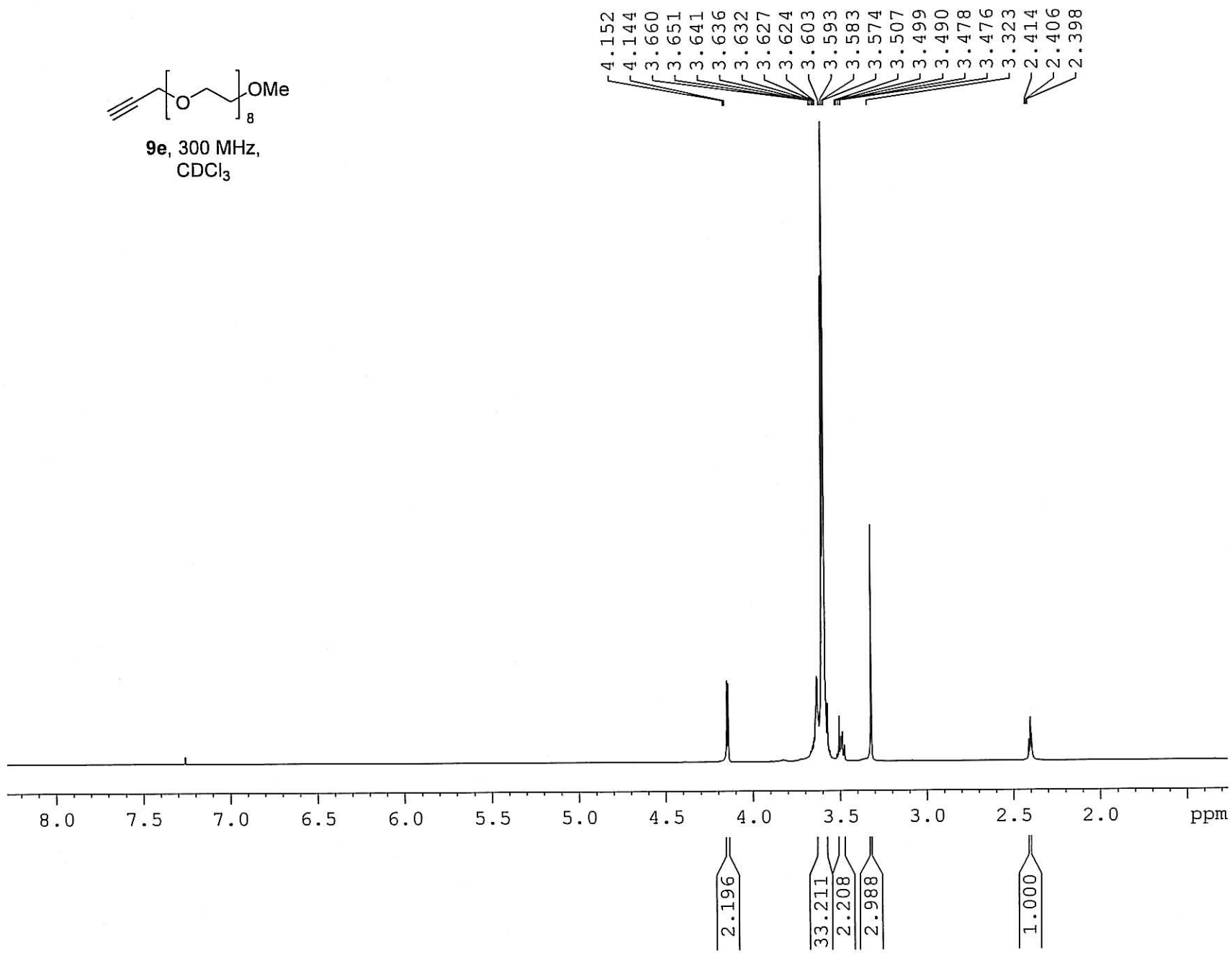


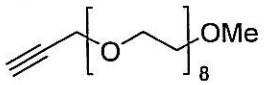


9d, 300 MHz,
$CDCl_3$

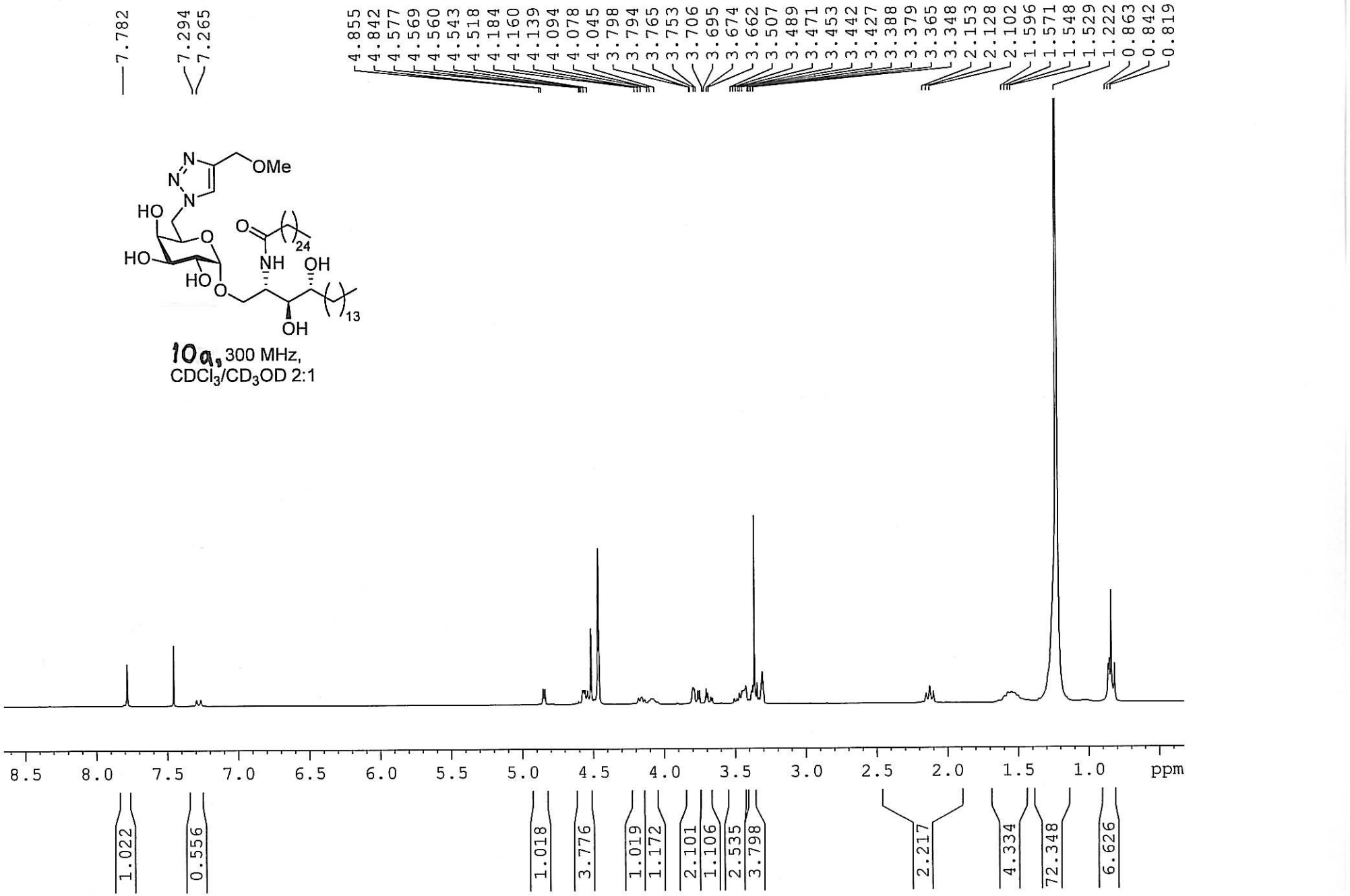


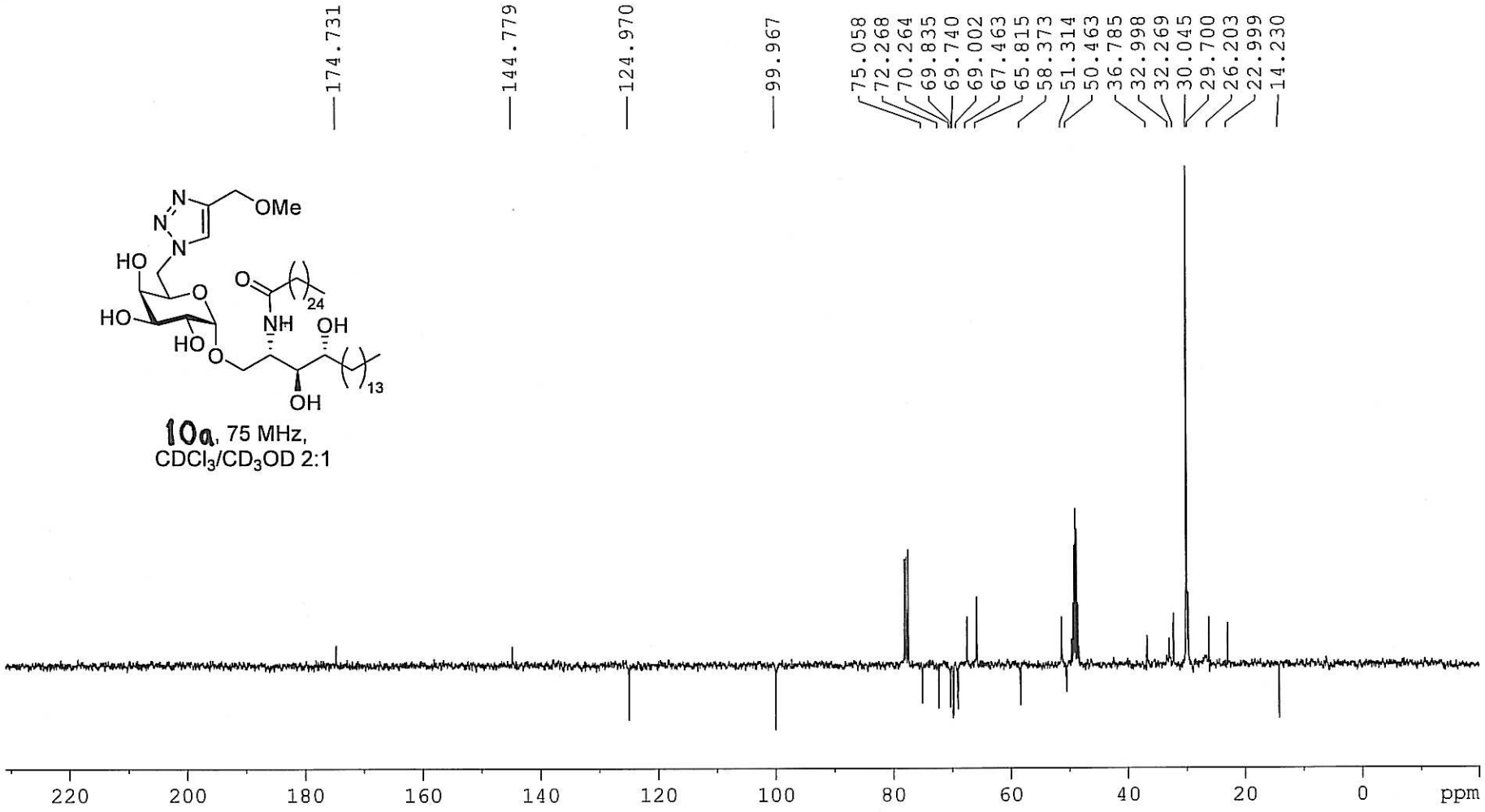
9d, 75 MHz,
$CDCl_3$


OMe
8
9e, 300 MHz,
CDCl3
4.152
4.144
3.660
3.651
3.641
3.636
3.632
3.627
3.624
3.603
3.593
3.583
3.574
3.507
3.499
3.490
3.478
3.476
3.323
2.414
2.406
2.398
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
ppm
2.196
33.211
2.208
2.988
1.000

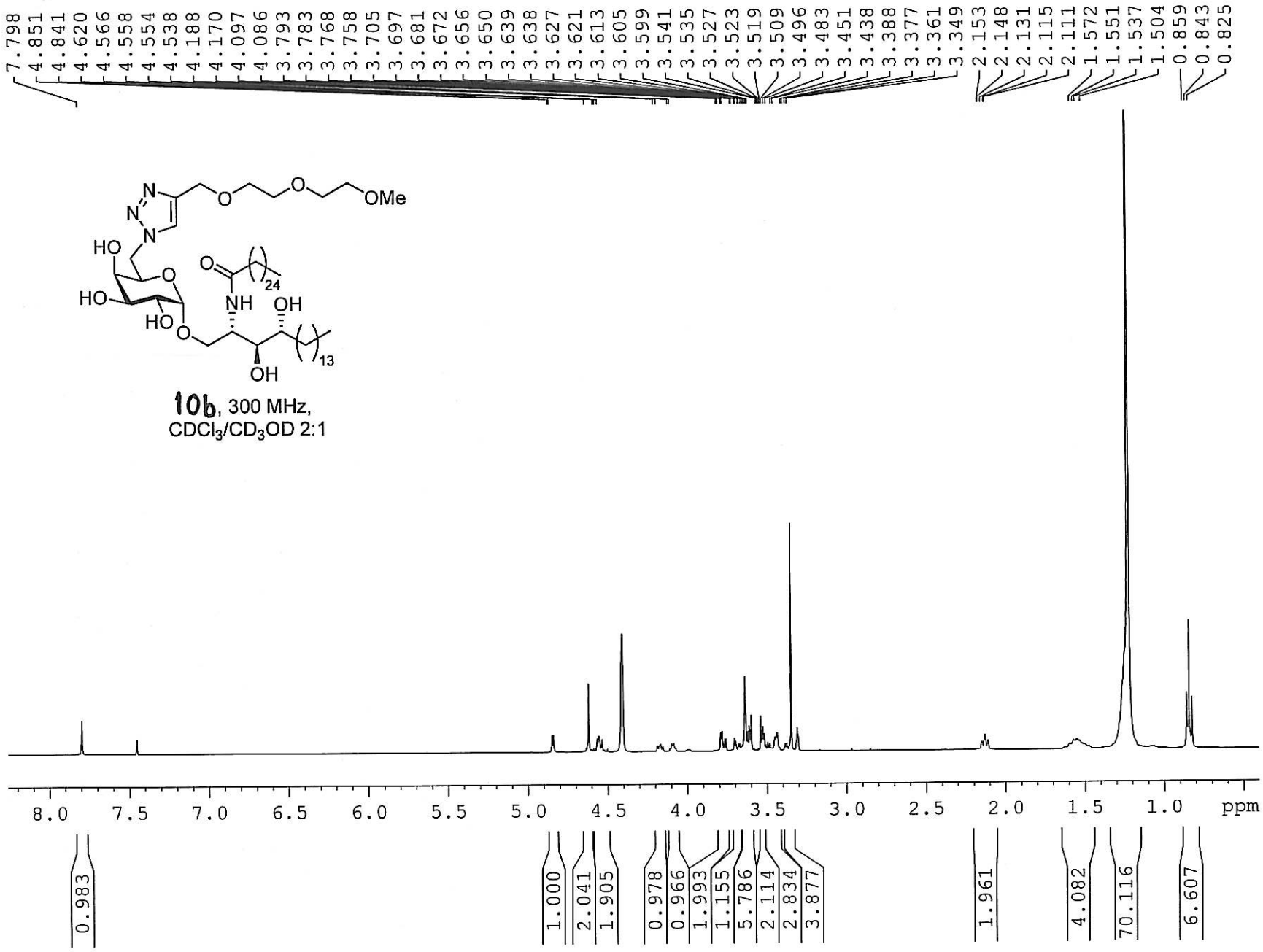


9e, 75 MHz,
$CDCl_3$

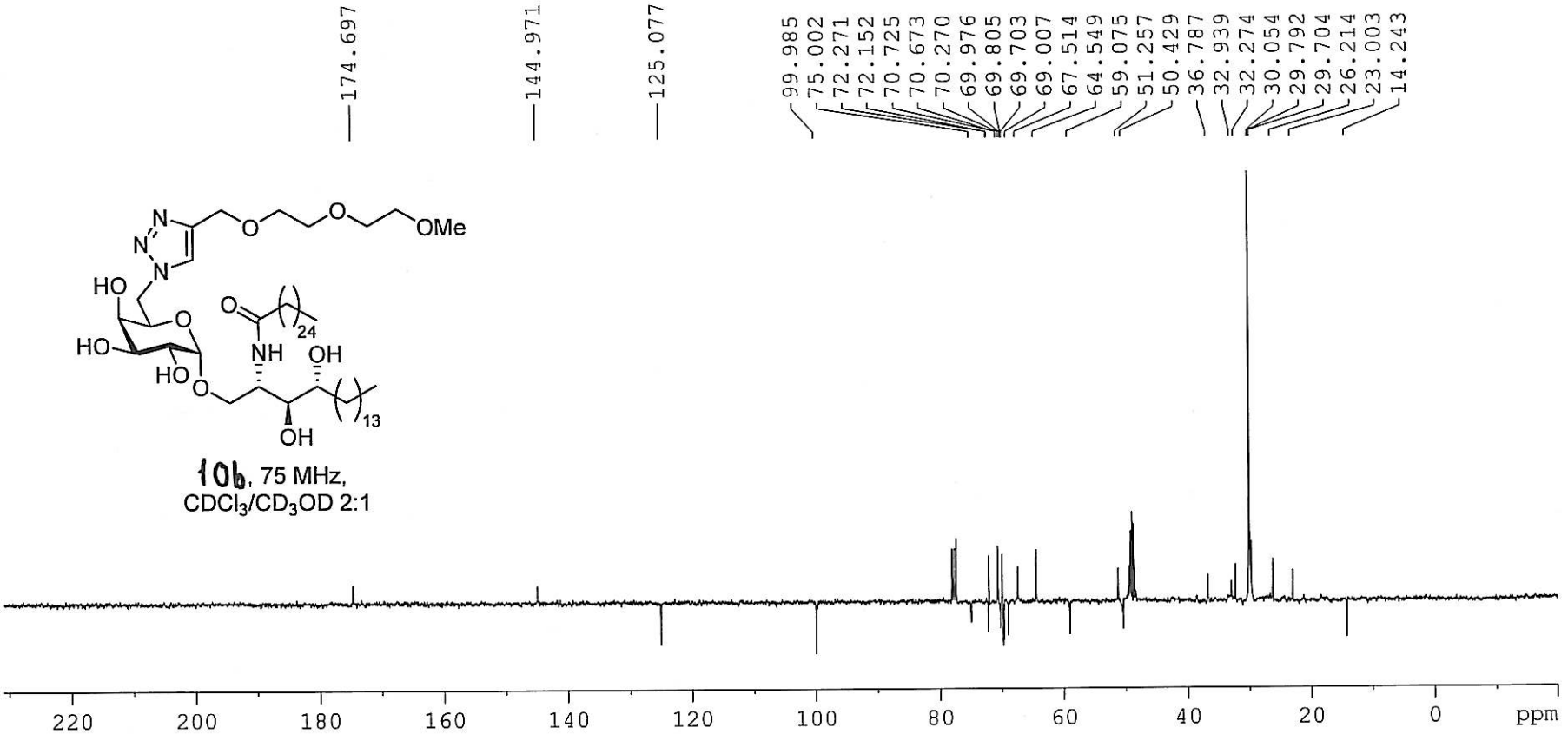

7.782
7.294
7.265
4.855
4.842
4.577
4.569
4.560
4.543
4.518
4.184
4.160
4.139
4.094
4.078
4.045
3.798
3.794
3.765
3.753
3.706
3.695
3.674
3.662
3.507
3.489
3.471
3.453
3.442
3.427
3.388
3.379
3.365
3.348
2.153
2.128
2.102
1.596
1.571
1.548
1.529
1.222
0.863
0.842
0.819
OMe
HO
HO
HO
O
NH
OH
OH
24
13
10a, 300 MHz,
CDCl3/CD3OD 2:1
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
ppm
1.022
0.556
1.018
3.776
1.019
1.172
2.101
1.106
2.535
3.798
2.217
4.334
72.348
6.626



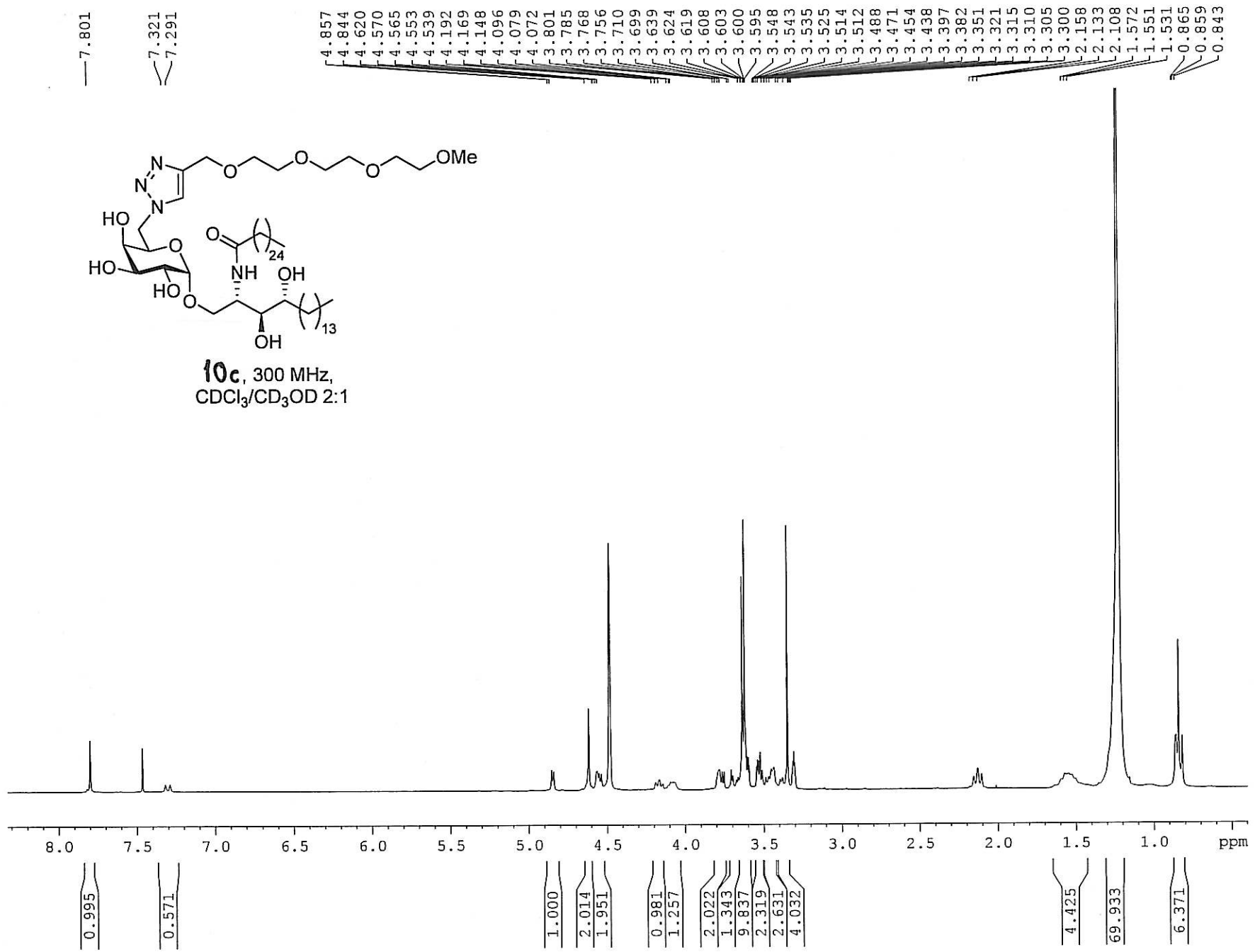
10a, 75 MHz,
$CDCl_3/CD_3OD$ 2:1



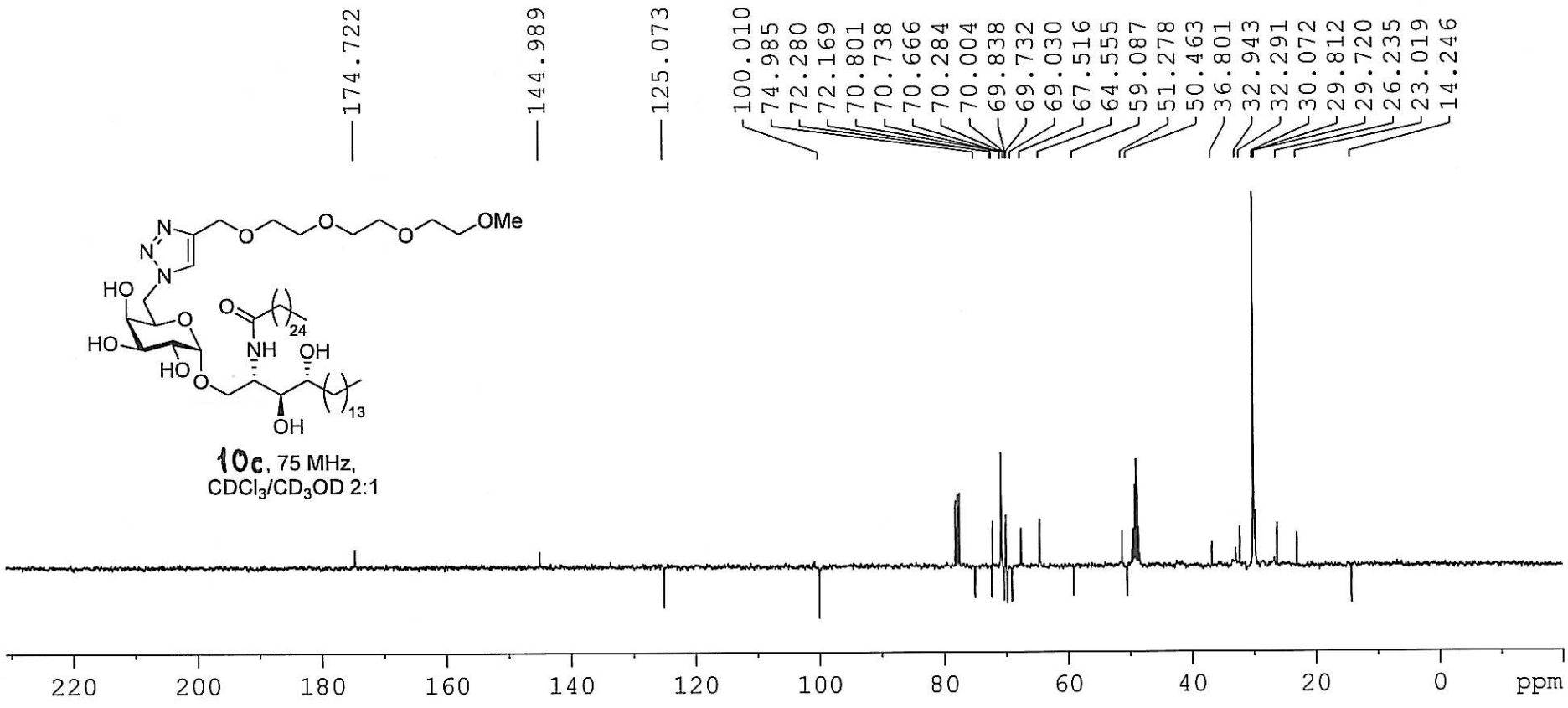
10b, 300 MHz,
$CDCl_3/CD_3OD$ 2:1

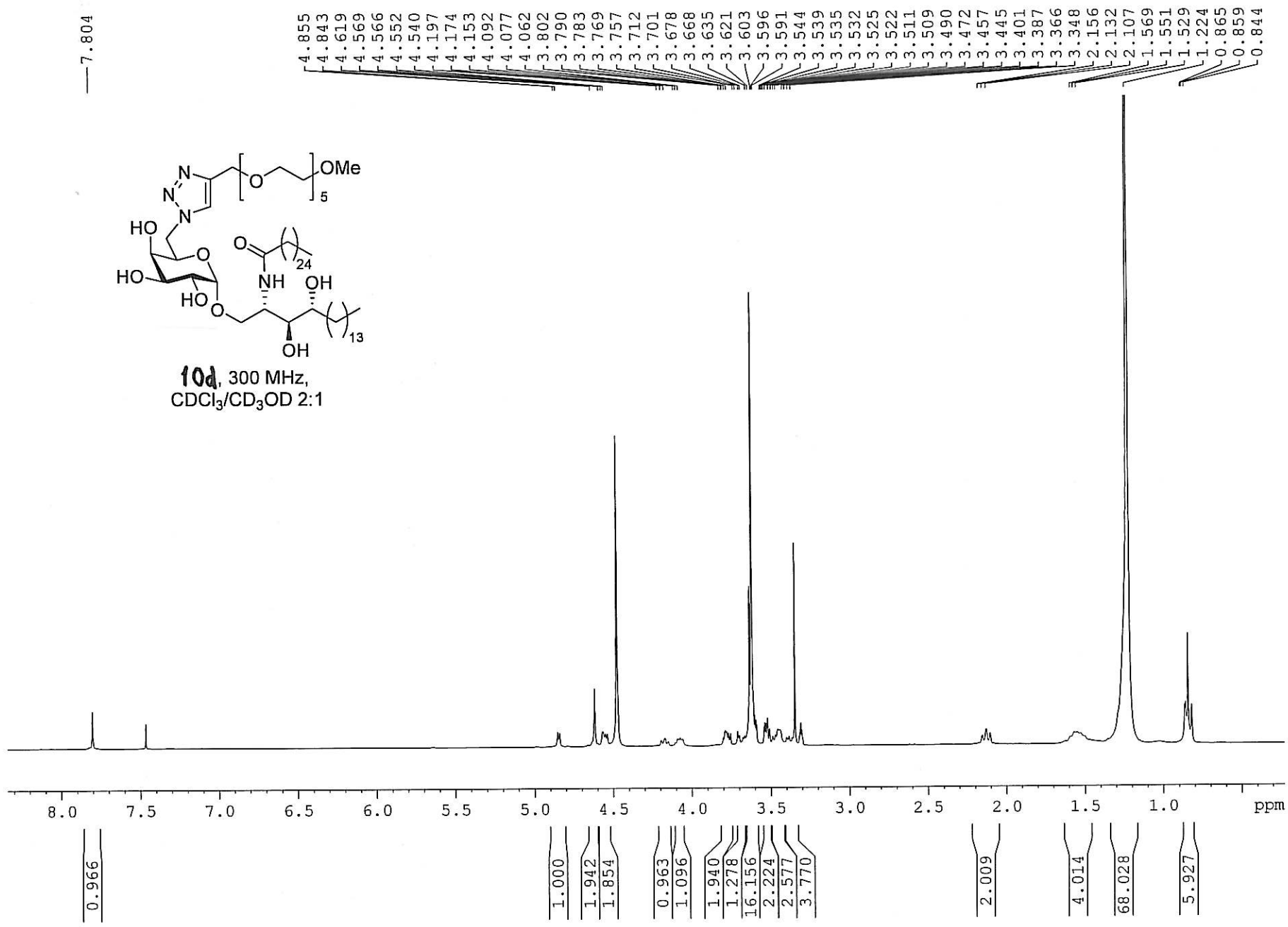


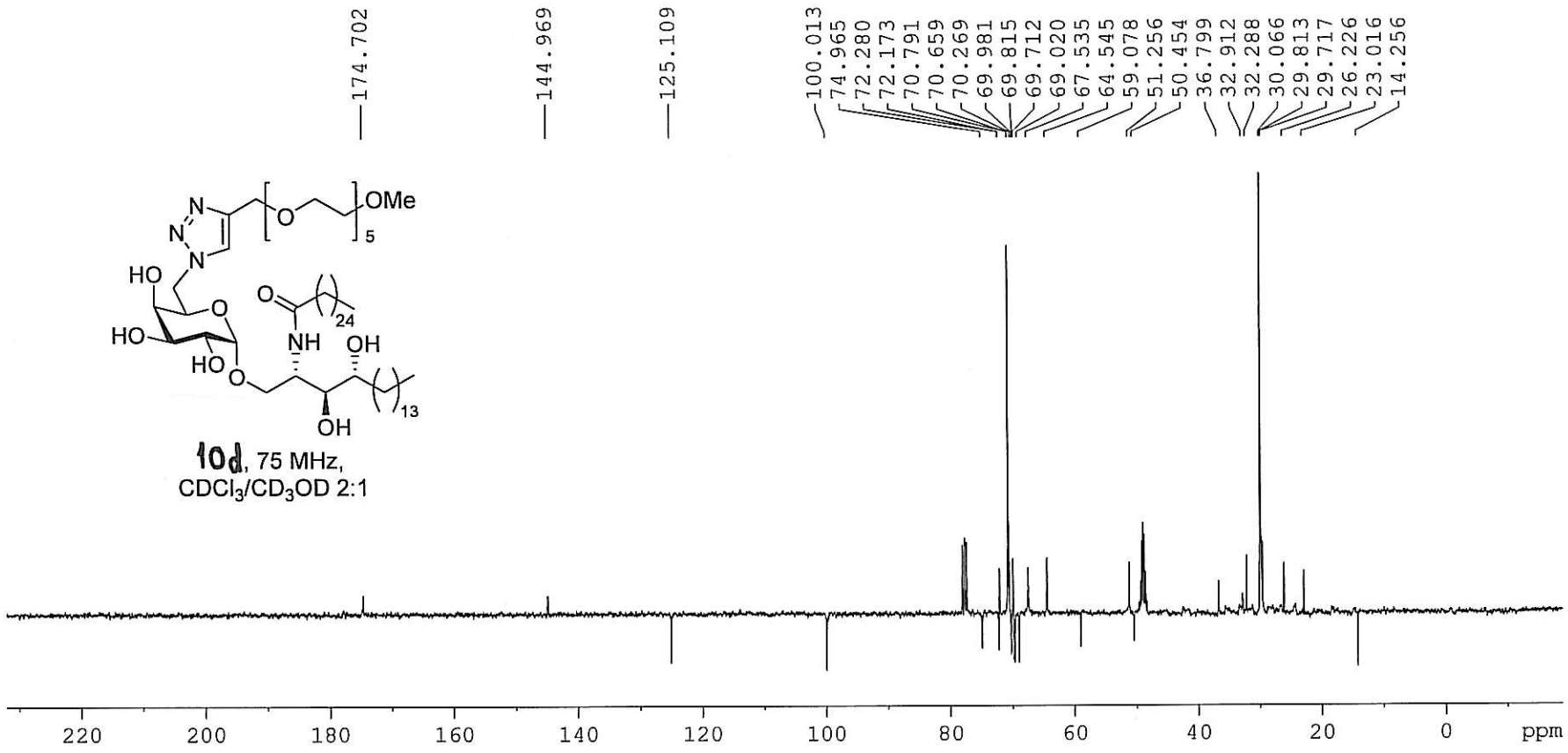
10b, 75 MHz,
$CDCl_3/CD_3OD$ 2:1



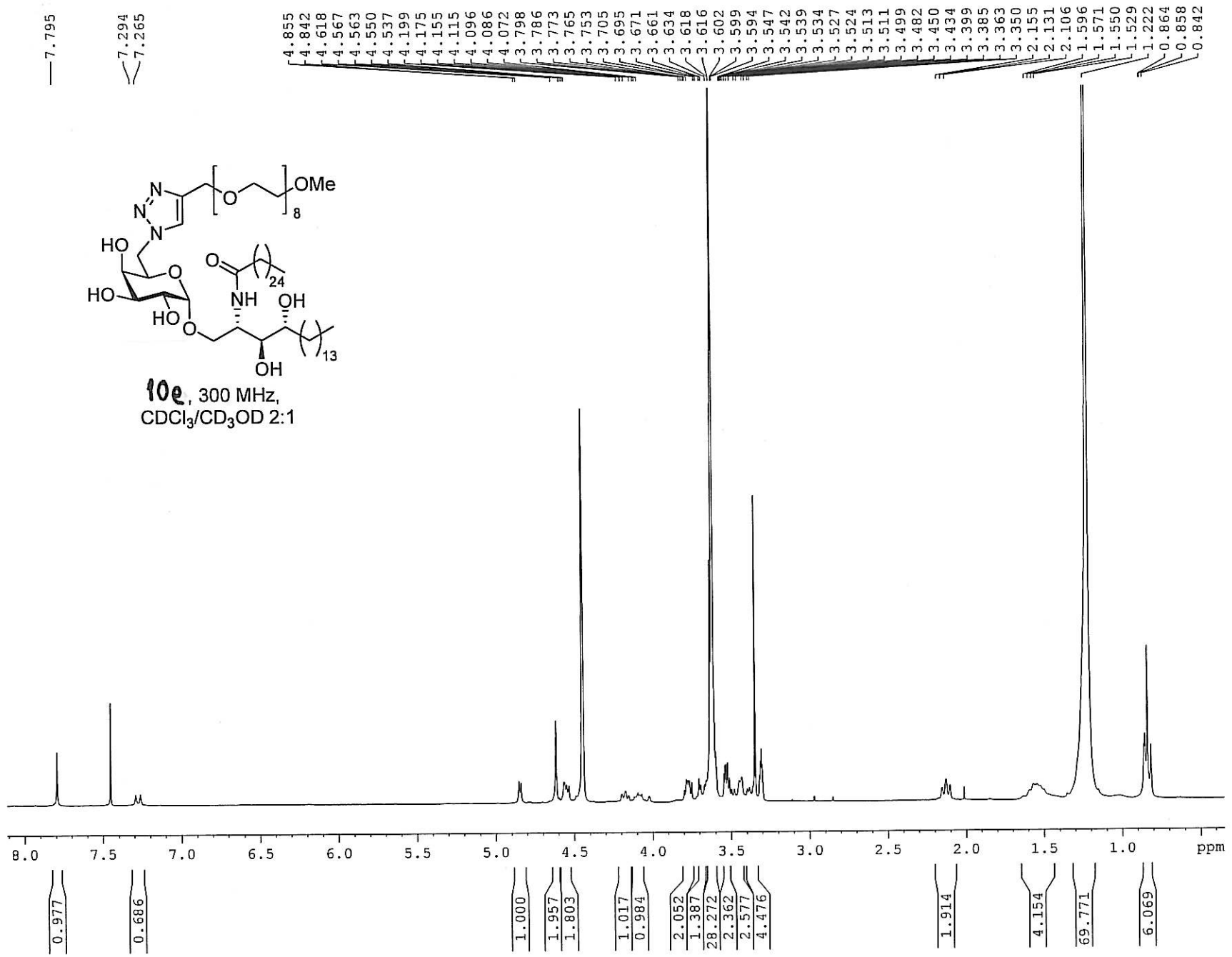
10c, 300 MHz,
$CDCl_3/CD_3OD$ 2:1

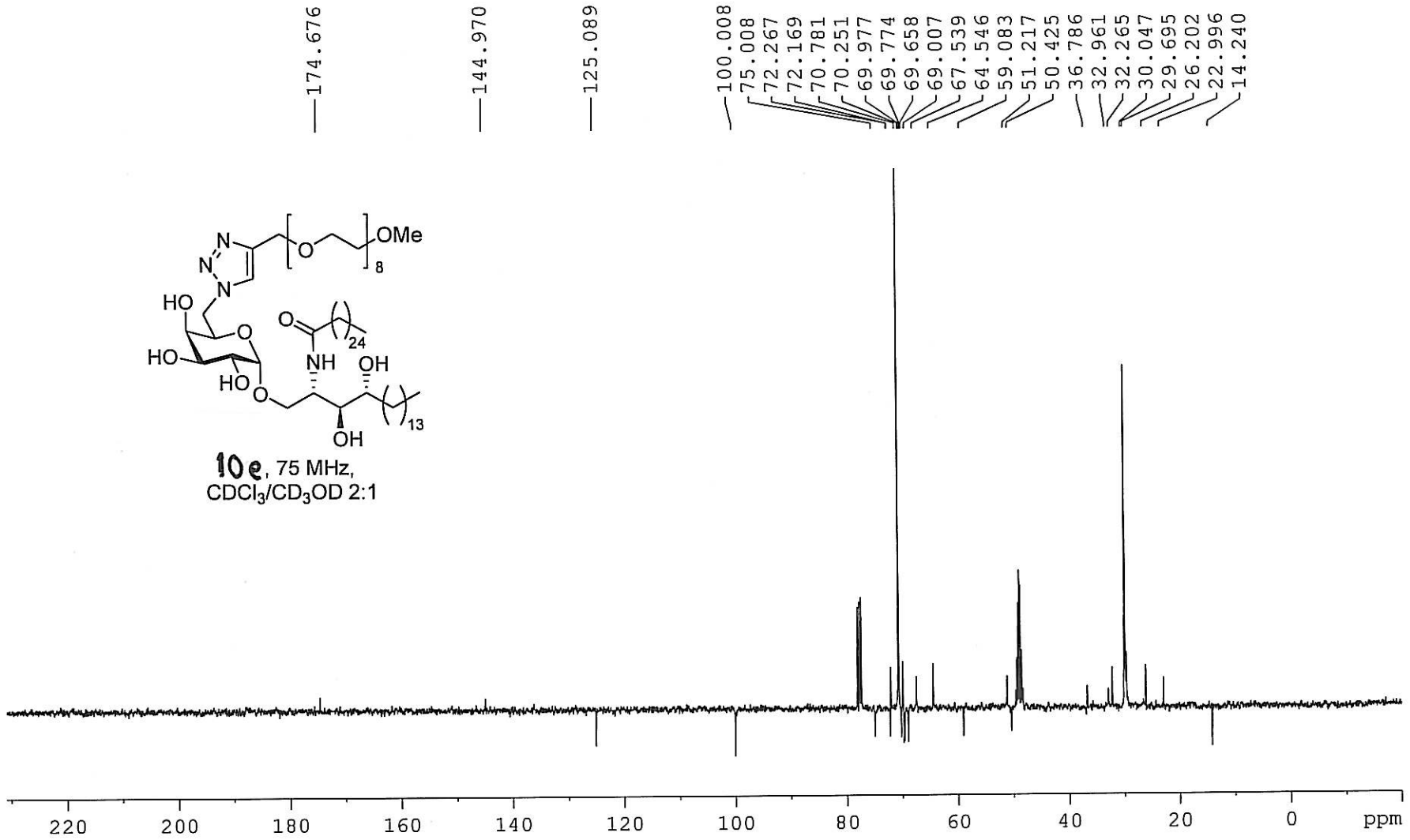

174.722
144.989
125.073
100.010
74.985
72.280
72.169
70.801
70.738
70.666
70.284
70.004
69.838
69.732
69.030
67.516
64.555
59.087
51.278
50.463
36.801
32.943
32.291
30.072
29.812
29.720
26.235
23.019
14.246
OMe
HO
HO
HO
NH
OH
OH
24
13
10c, 75 MHz,
CDCl3/CD3OD 2:1
220
200
180
160
140
120
100
80
60
40
20
0
ppm


7.804
4.855
4.843
4.619
4.569
4.566
4.552
4.540
4.197
4.174
4.153
4.092
4.077
4.062
3.802
3.790
3.783
3.769
3.757
3.712
3.701
3.678
3.668
3.635
3.621
3.603
3.596
3.591
3.544
3.539
3.535
3.532
3.525
3.522
3.511
3.509
3.490
3.472
3.457
3.445
3.401
3.387
3.366
3.348
2.156
2.132
2.107
1.569
1.551
1.529
1.224
0.865
0.859
0.844
OMe
5
HO
HO
HO
O
NH
OH
OH
24
13
10d, 300 MHz,
CDCl3/CD3OD 2:1
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
ppm
0.966
1.000
1.942
1.854
0.963
1.096
1.940
1.278
16.156
2.224
2.577
3.770
2.009
4.014
68.028
5.927

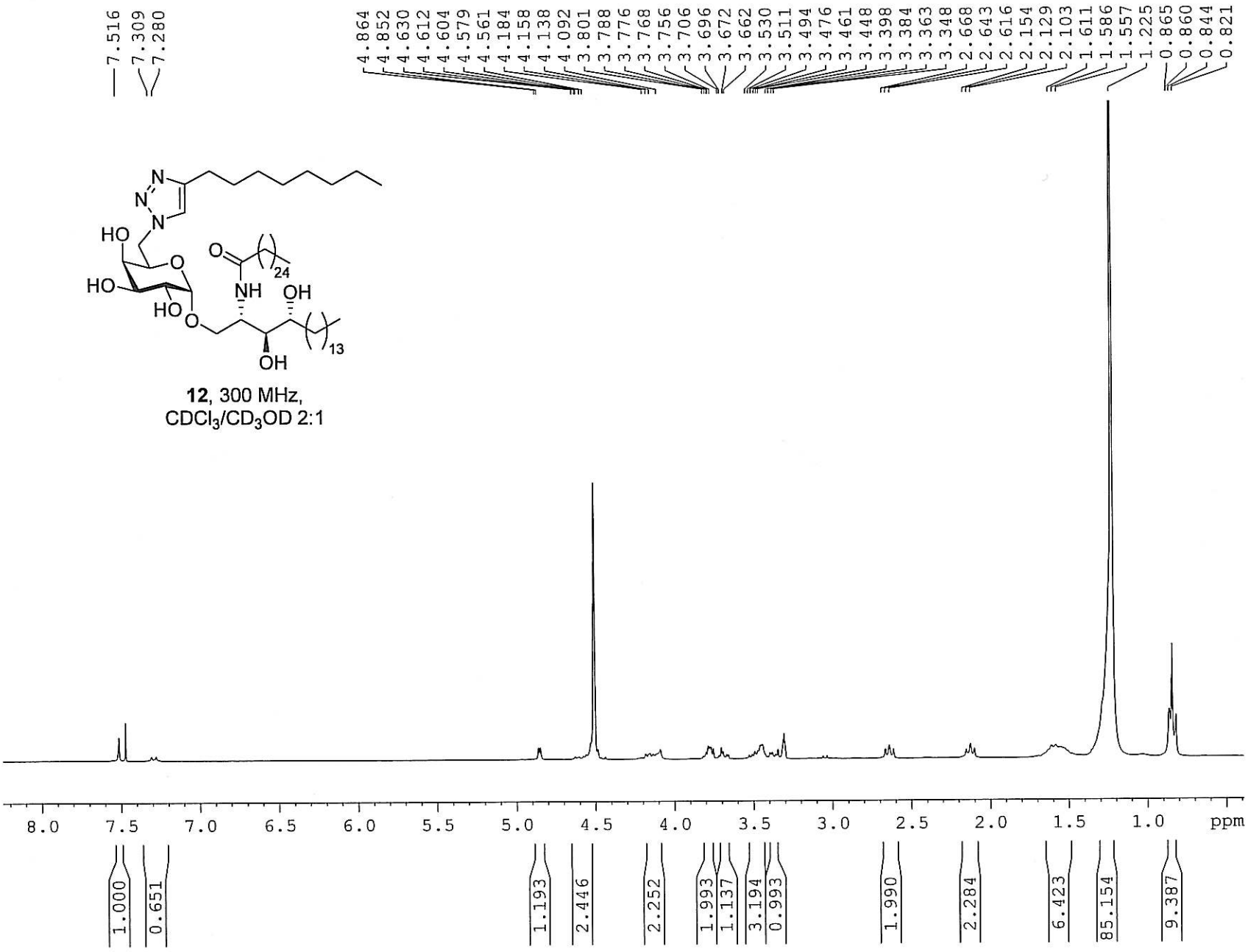


10d, 75 MHz,
$CDCl_3/CD_3OD$ 2:1

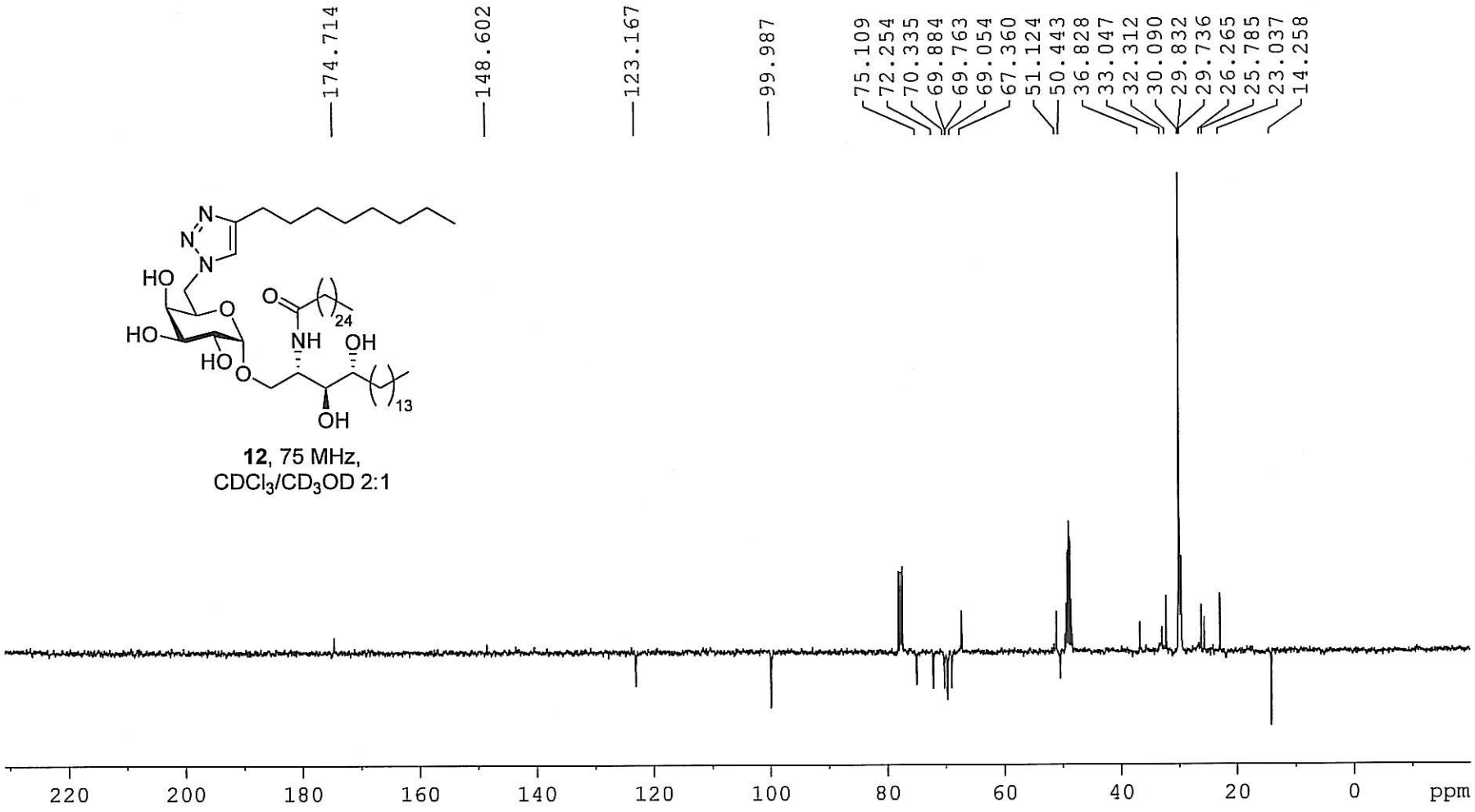

7.795
7.294
7.265
4.855
4.842
4.618
4.567
4.563
4.550
4.537
4.199
4.175
4.155
4.115
4.096
4.086
4.072
3.798
3.786
3.773
3.765
3.753
3.705
3.695
3.671
3.661
3.634
3.618
3.616
3.602
3.599
3.594
3.547
3.542
3.539
3.534
3.527
3.524
3.513
3.511
3.499
3.482
3.450
3.434
3.399
3.385
3.363
3.350
2.155
2.131
2.106
1.596
1.571
1.550
1.529
1.222
0.864
0.858
0.842
OMe
8
HO
HO
HO
O
NH
OH
OH
24
13
10e, 300 MHz,
CDCl3/CD3OD 2:1
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
ppm
0.977
0.686
1.000
1.957
1.803
1.017
0.984
2.052
1.387
28.272
2.362
2.577
4.476
1.914
4.154
69.771
6.069



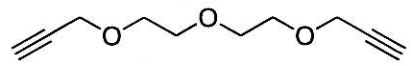
10e, 75 MHz,
$CDCl_3/CD_3OD$ 2:1



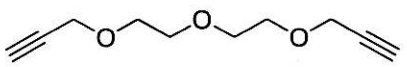
12, 300 MHz,
$CDCl_3/CD_3OD$ 2:1



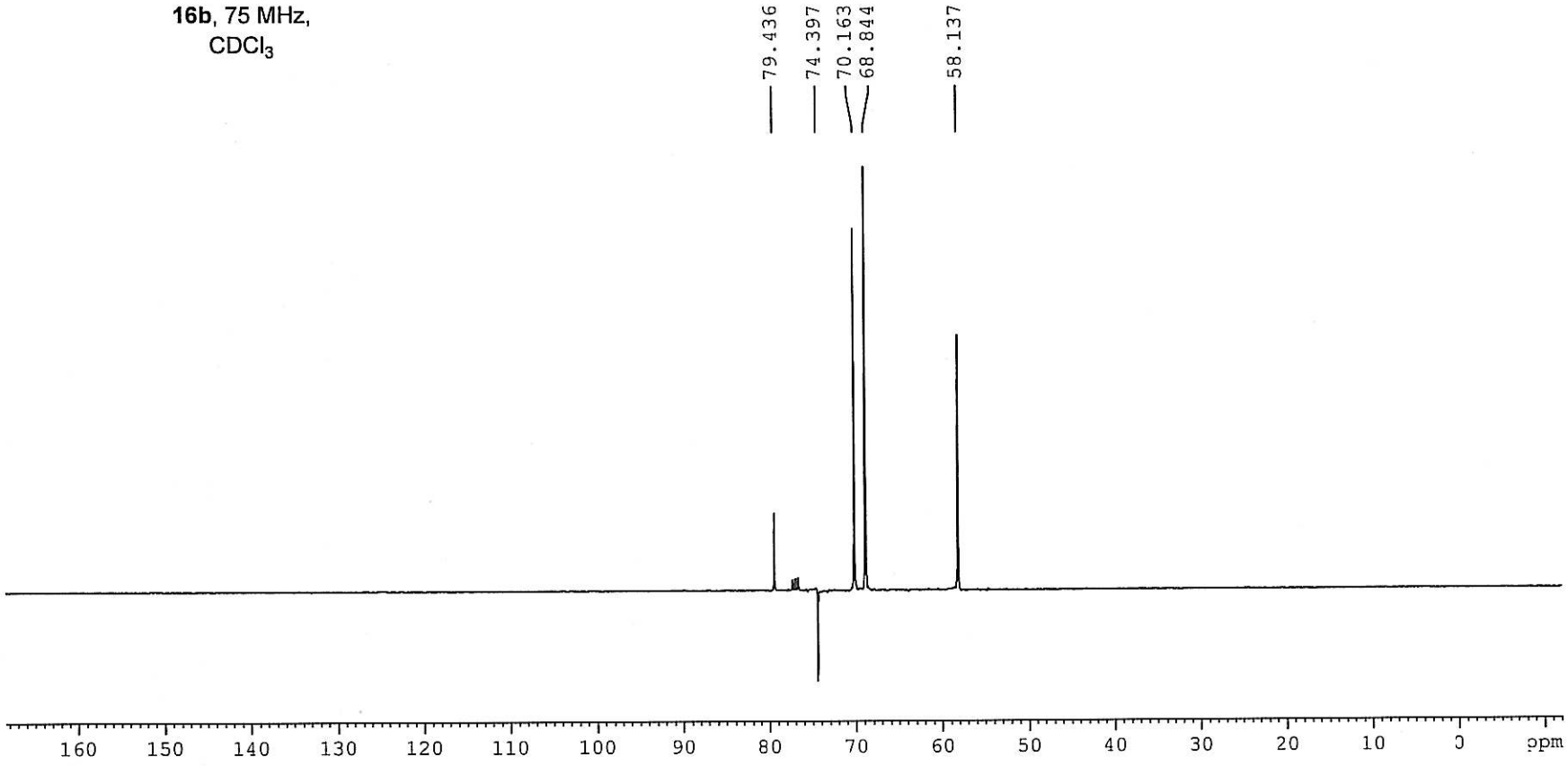
12, 75 MHz,
$CDCl_3/CD_3OD$ 2:1

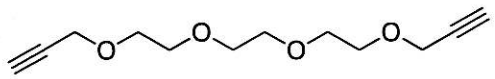


16b, 300 MHz,
$CDCl_3$

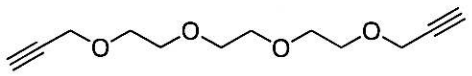


16b, 75 MHz,
$CDCl_3$

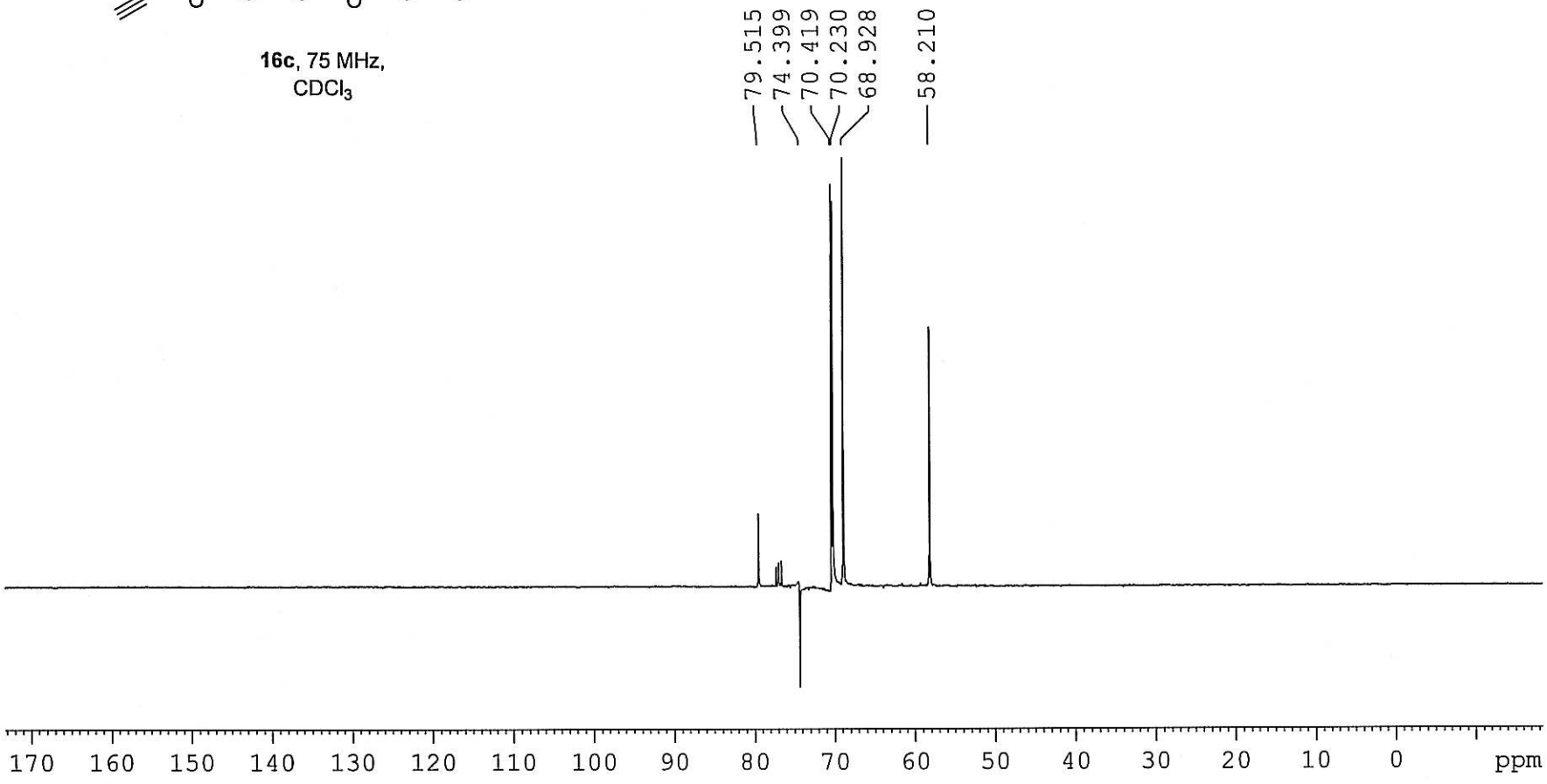


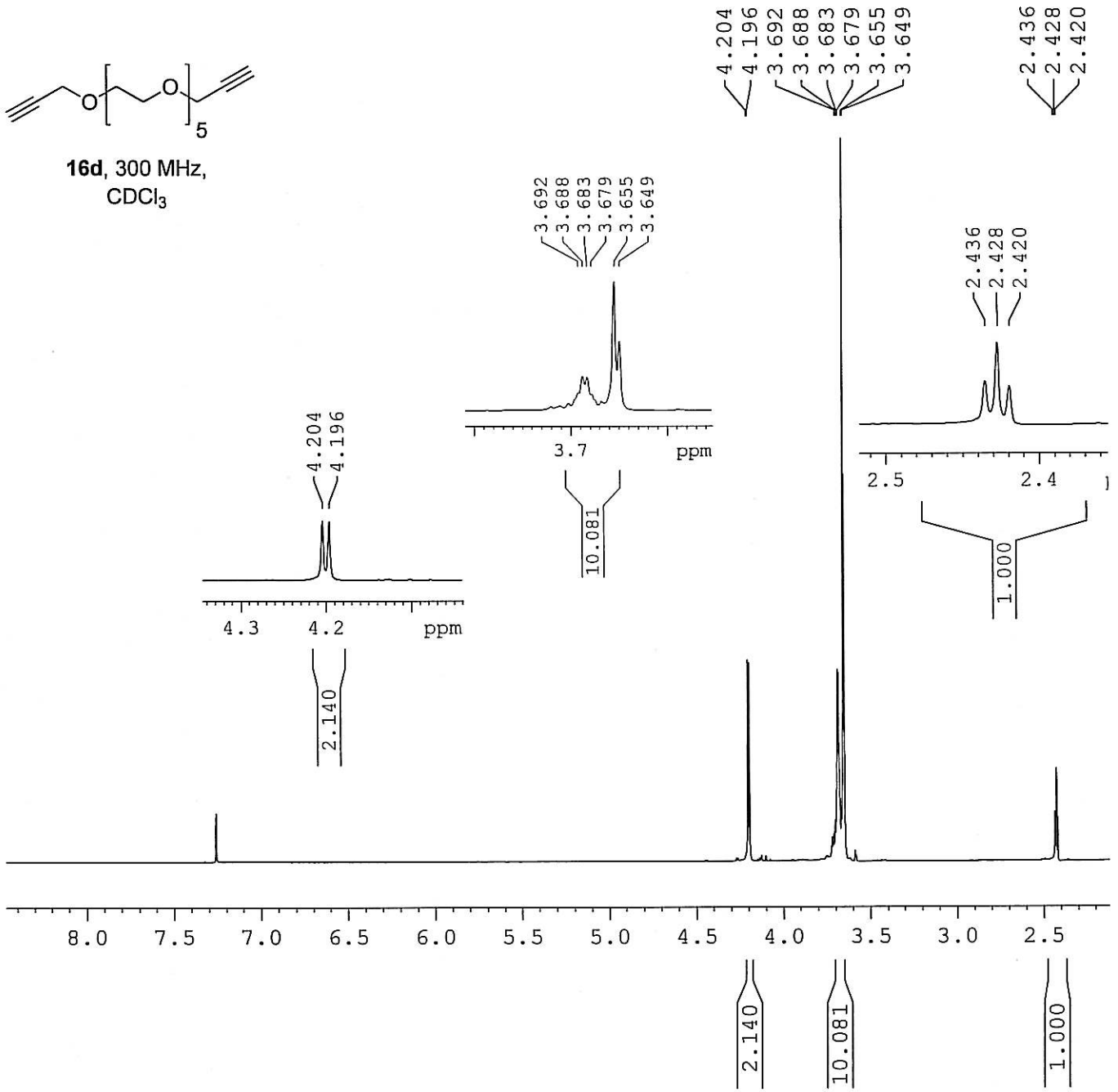


16c, 300 MHz,
$CDCl_3$

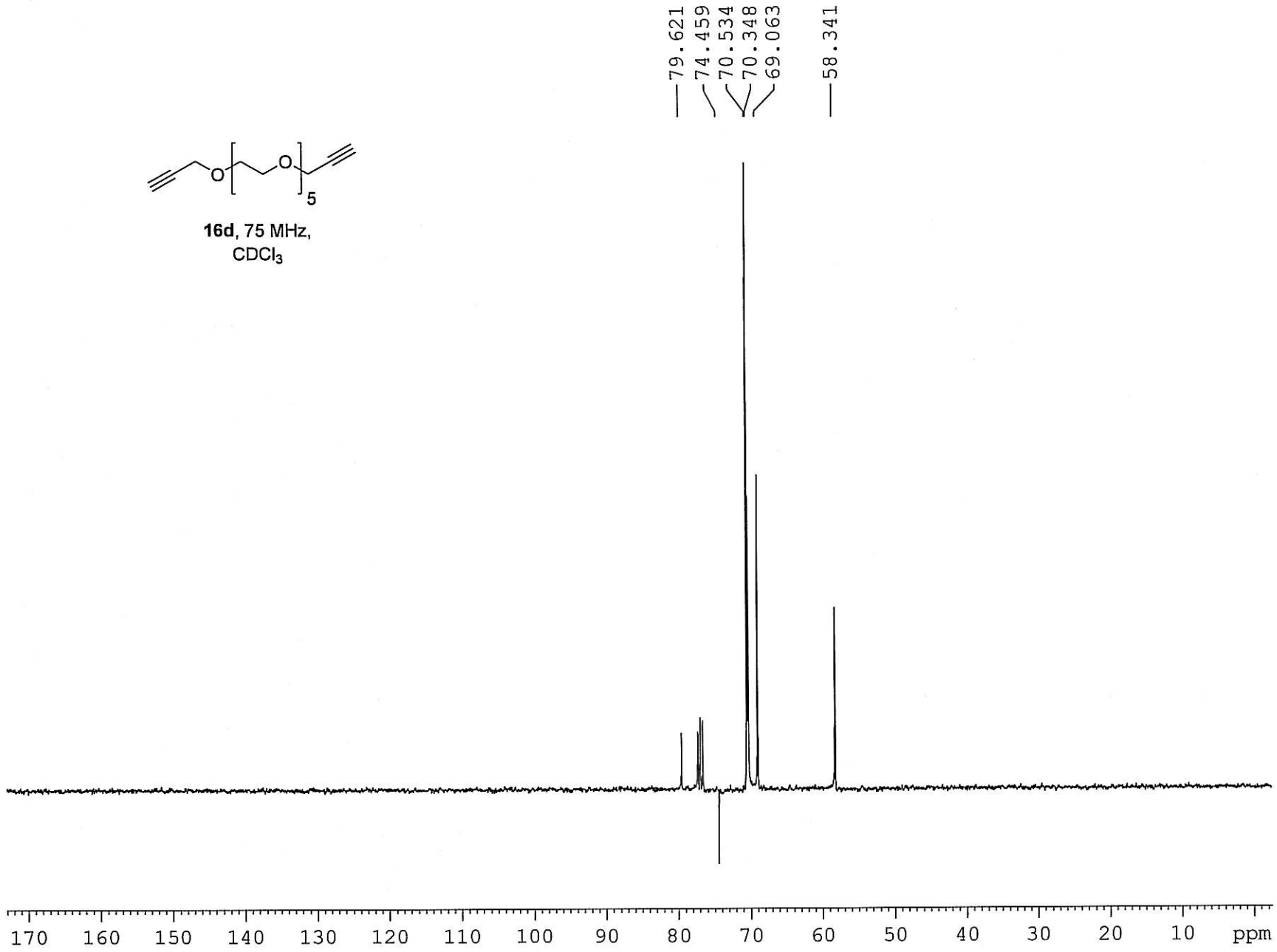


16c, 75 MHz,
$CDCl_3$

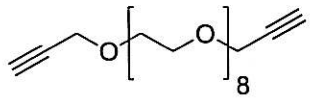




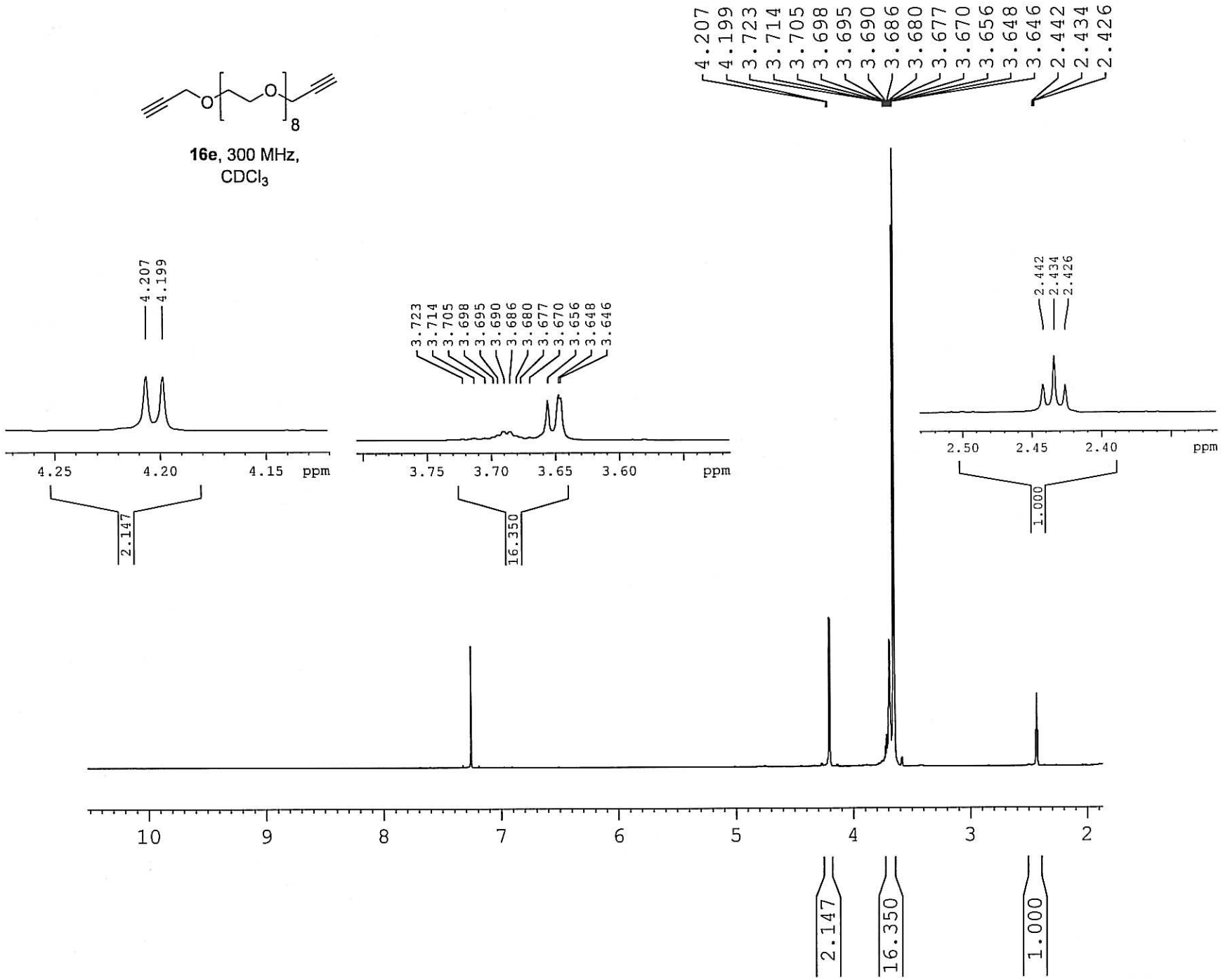
16d, 300 MHz,
$CDCl_3$

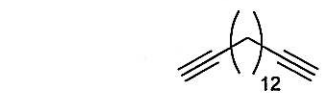


16d, 75 MHz,
$CDCl_3$

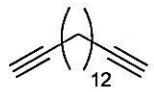


16e, 75 MHz,
$CDCl_3$

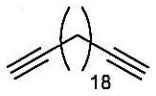

4.207
4.199
3.723
3.714
3.705
3.698
3.695
3.690
3.686
3.680
3.677
3.670
3.656
3.648
3.646
2.442
2.434
2.426
8
16e, 300 MHz,
CDCl3
4.25
4.20
4.15
ppm
2.147
3.75
3.70
3.65
3.60
ppm
16.350
2.50
2.45
2.40
ppm
1.000
10
9
8
7
6
5
4
3
2
2.147
16.350
1.000



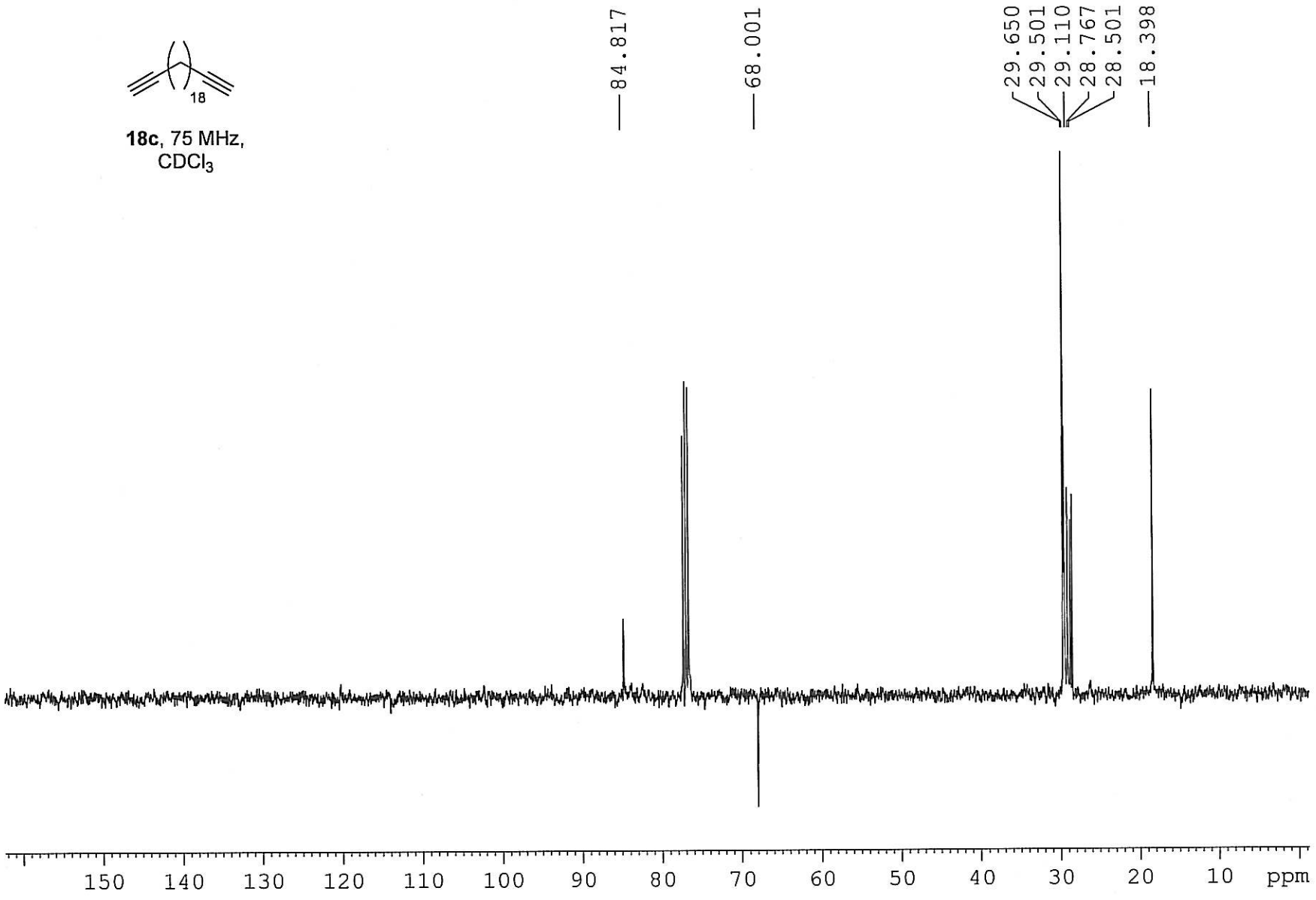
18b, 300 MHz,
$CDCl_3$



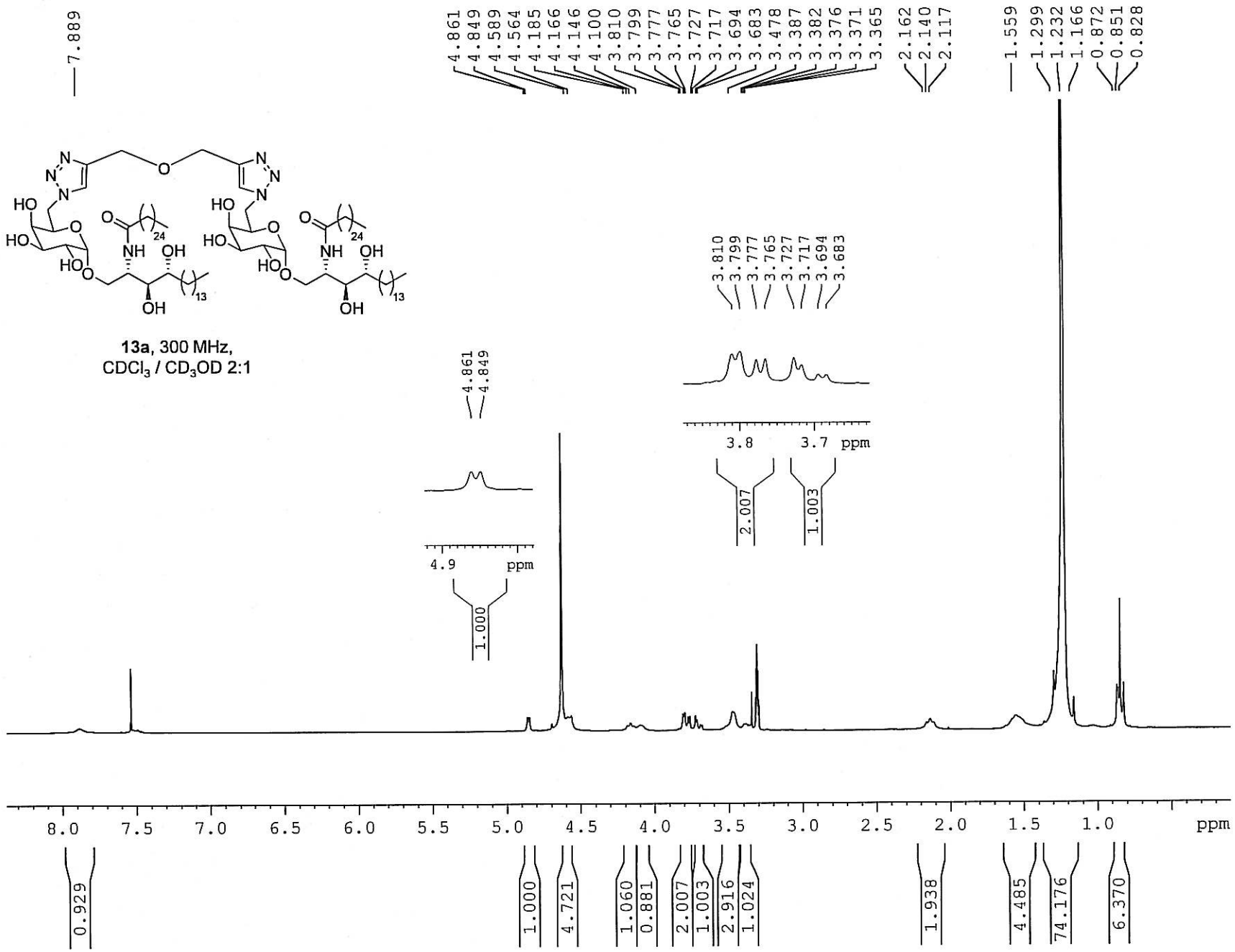
18b, 75 MHz,
$CDCl_3$

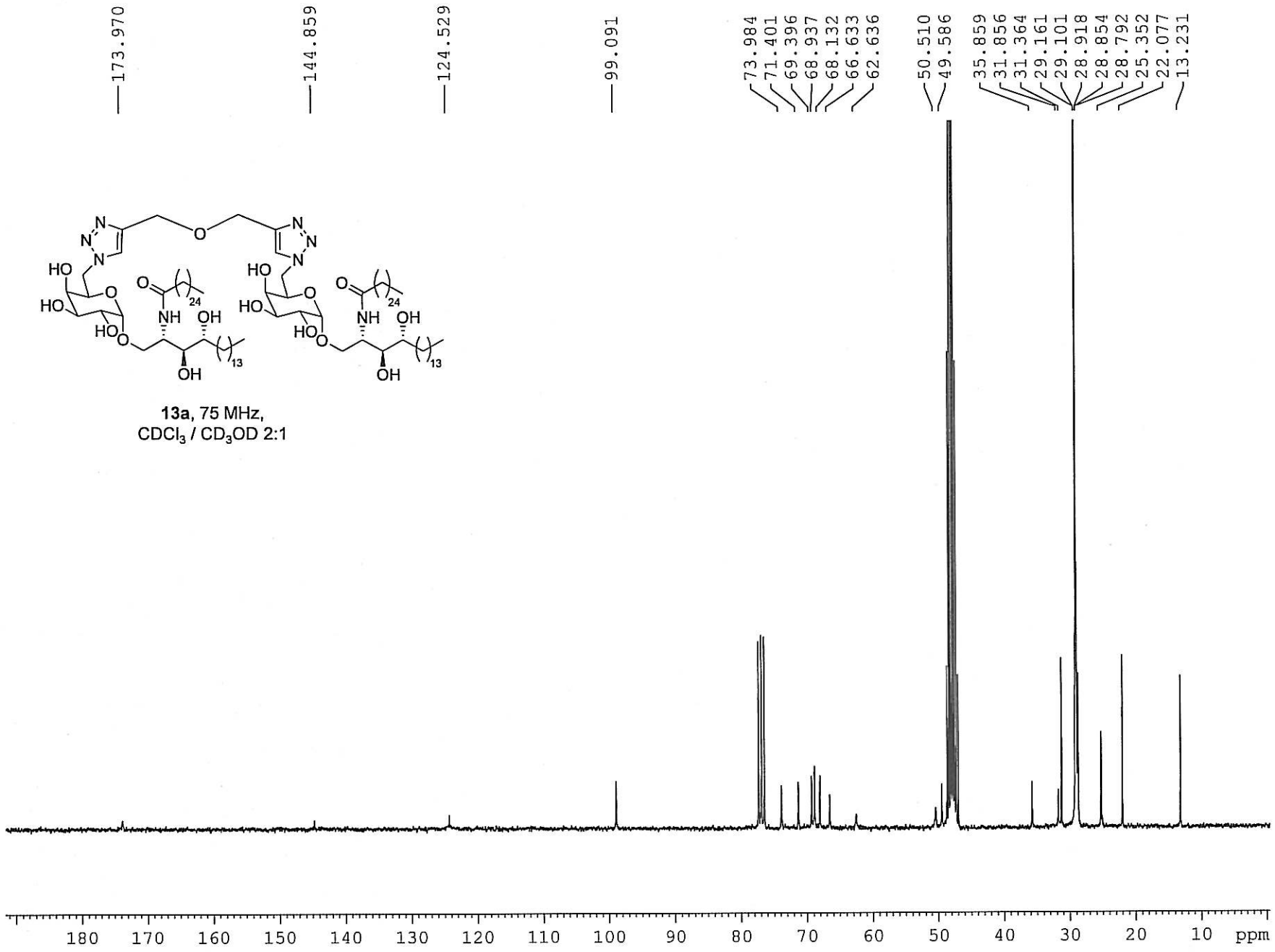


18c, 300 MHz,
$CDCl_3$

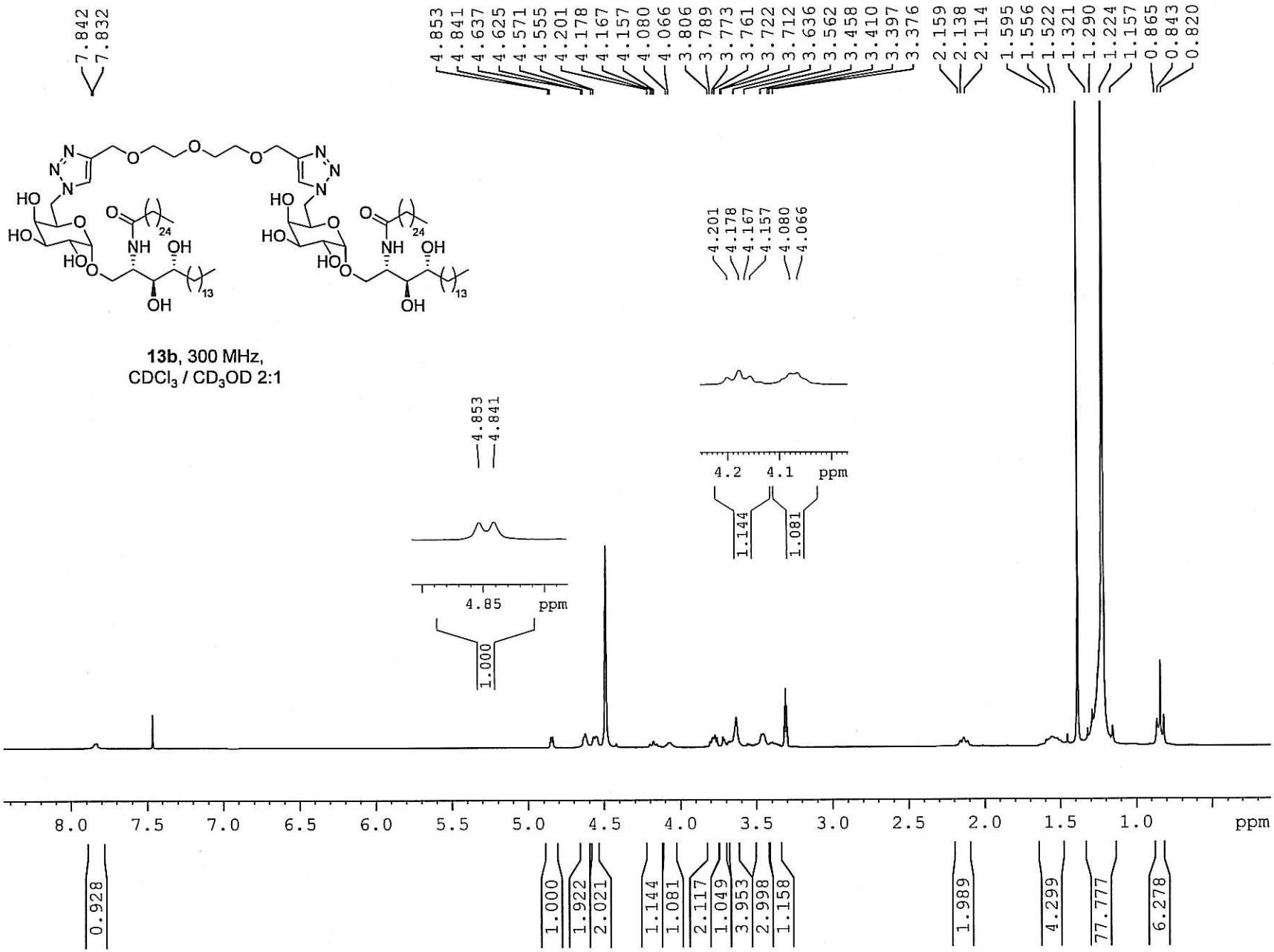


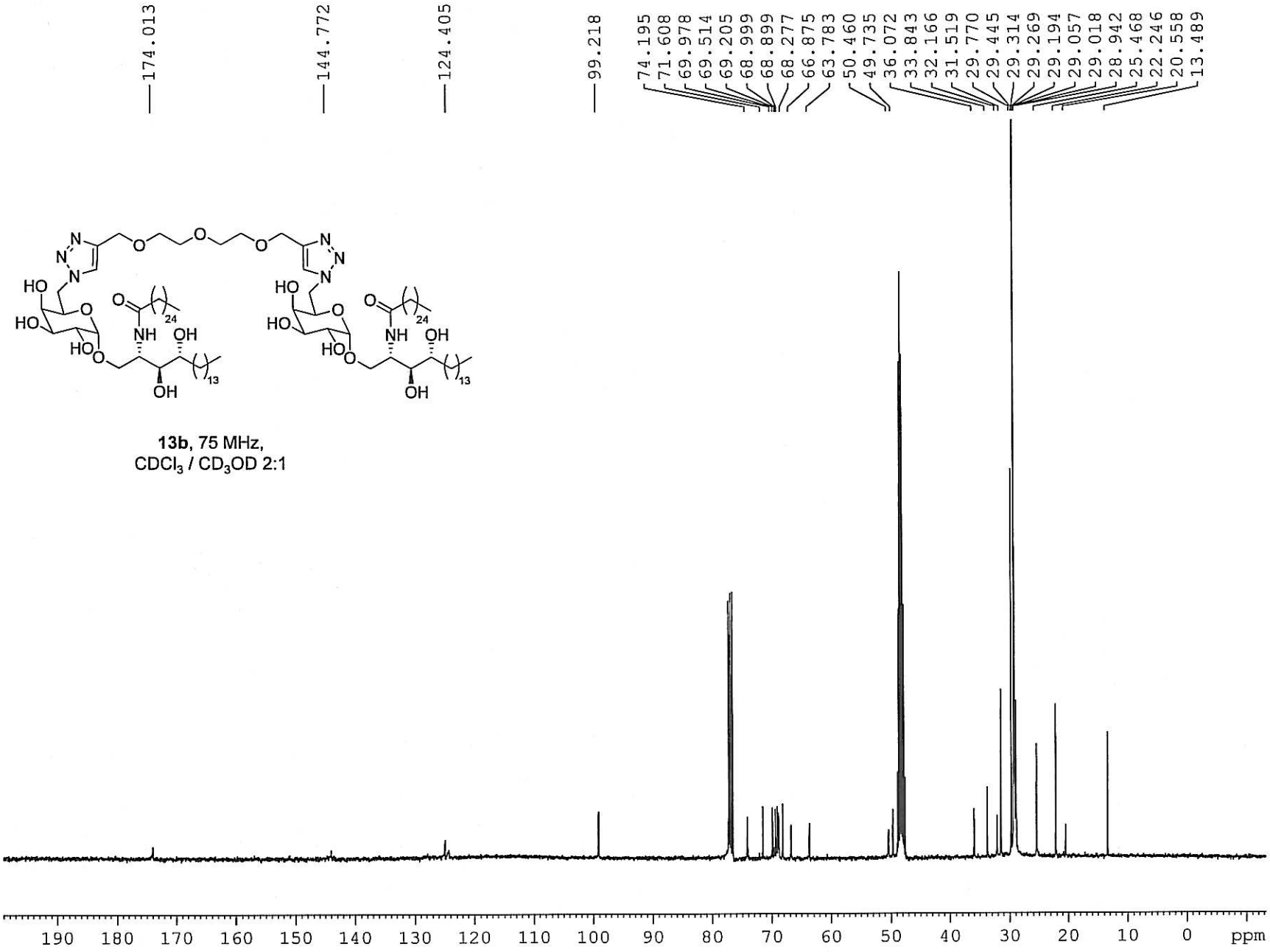
18c, 75 MHz,
$CDCl_3$


7.889
4.861
4.849
4.589
4.564
4.185
4.166
4.146
4.100
3.810
3.799
3.777
3.765
3.727
3.717
3.694
3.683
3.478
3.387
3.382
3.376
3.371
3.365
2.162
2.140
2.117
1.559
1.299
1.232
1.166
0.872
0.851
0.828
13a, 300 MHz,
CDCl3 / CD3OD 2:1
4.9 ppm
3.8 3.7 ppm
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 ppm
0.929
1.000
4.721
1.060
0.881
2.007
1.003
2.916
1.024
1.938
4.485
74.176
6.370

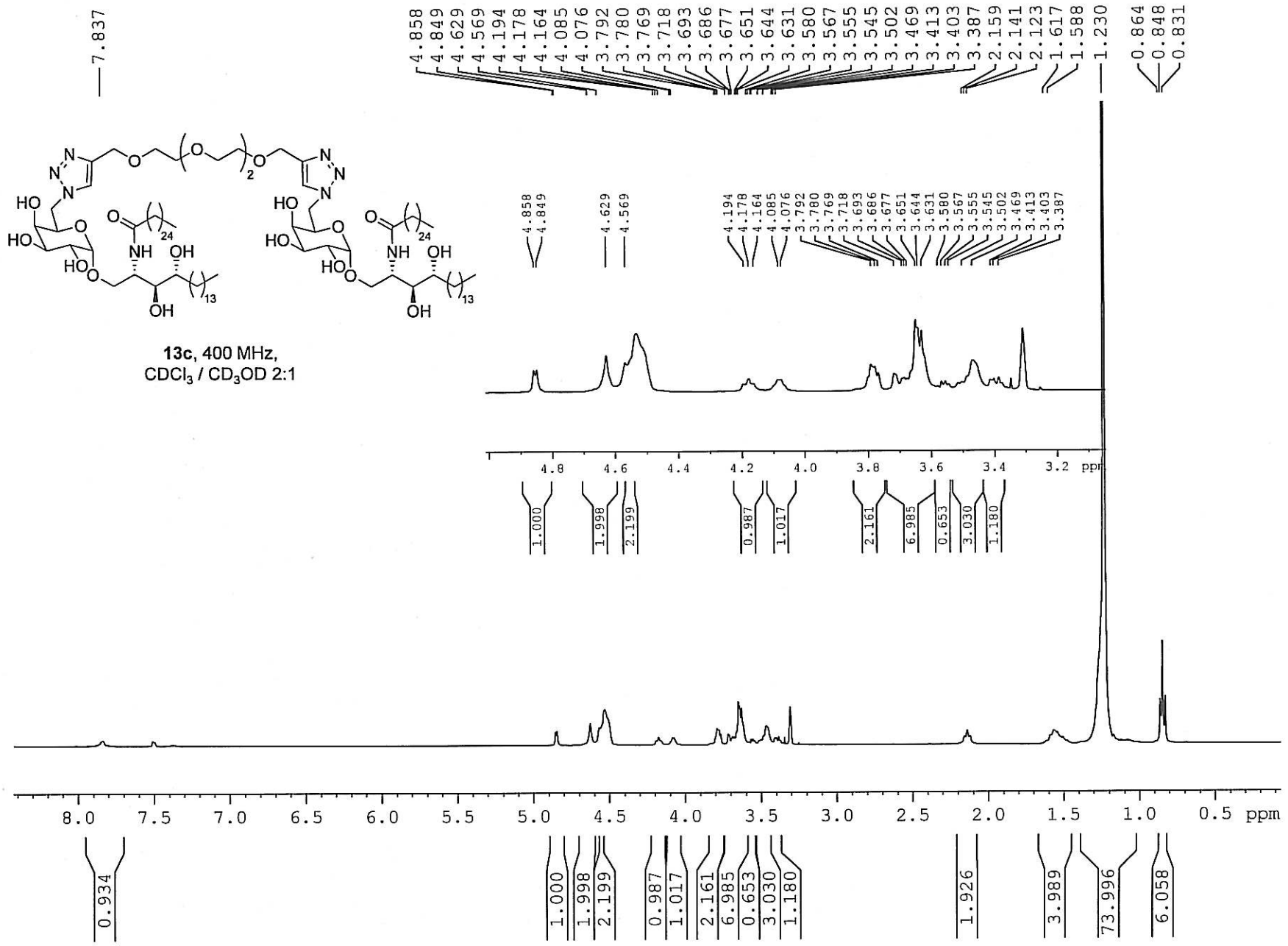


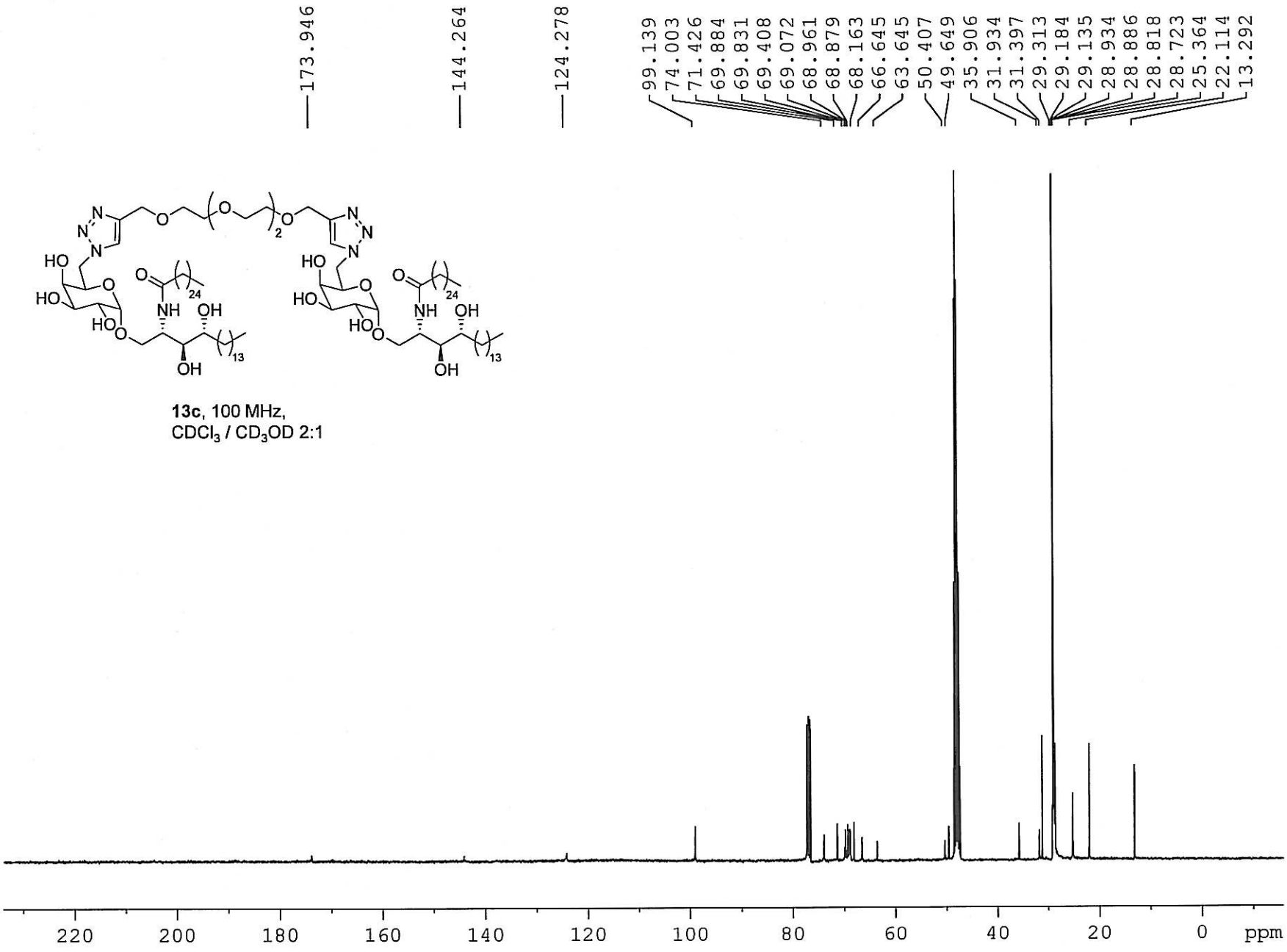
13a, 75 MHz,
$CDCl_3$ / CD_3OD 2:1


7.842
7.832
4.853
4.841
4.637
4.625
4.571
4.555
4.201
4.178
4.167
4.157
4.080
4.066
3.806
3.789
3.773
3.761
3.722
3.712
3.636
3.562
3.458
3.410
3.397
3.376
2.159
2.138
2.114
1.595
1.556
1.522
1.321
1.290
1.224
1.157
0.865
0.843
0.820
13b, 300 MHz,
CDCl3 / CD3OD 2:1
4.85
ppm
1.000
4.2
4.1
ppm
1.144
1.081
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
ppm
0.928
1.000
1.922
2.021
1.144
1.081
2.117
1.049
3.953
2.998
1.158
1.989
4.299
77.777
6.278

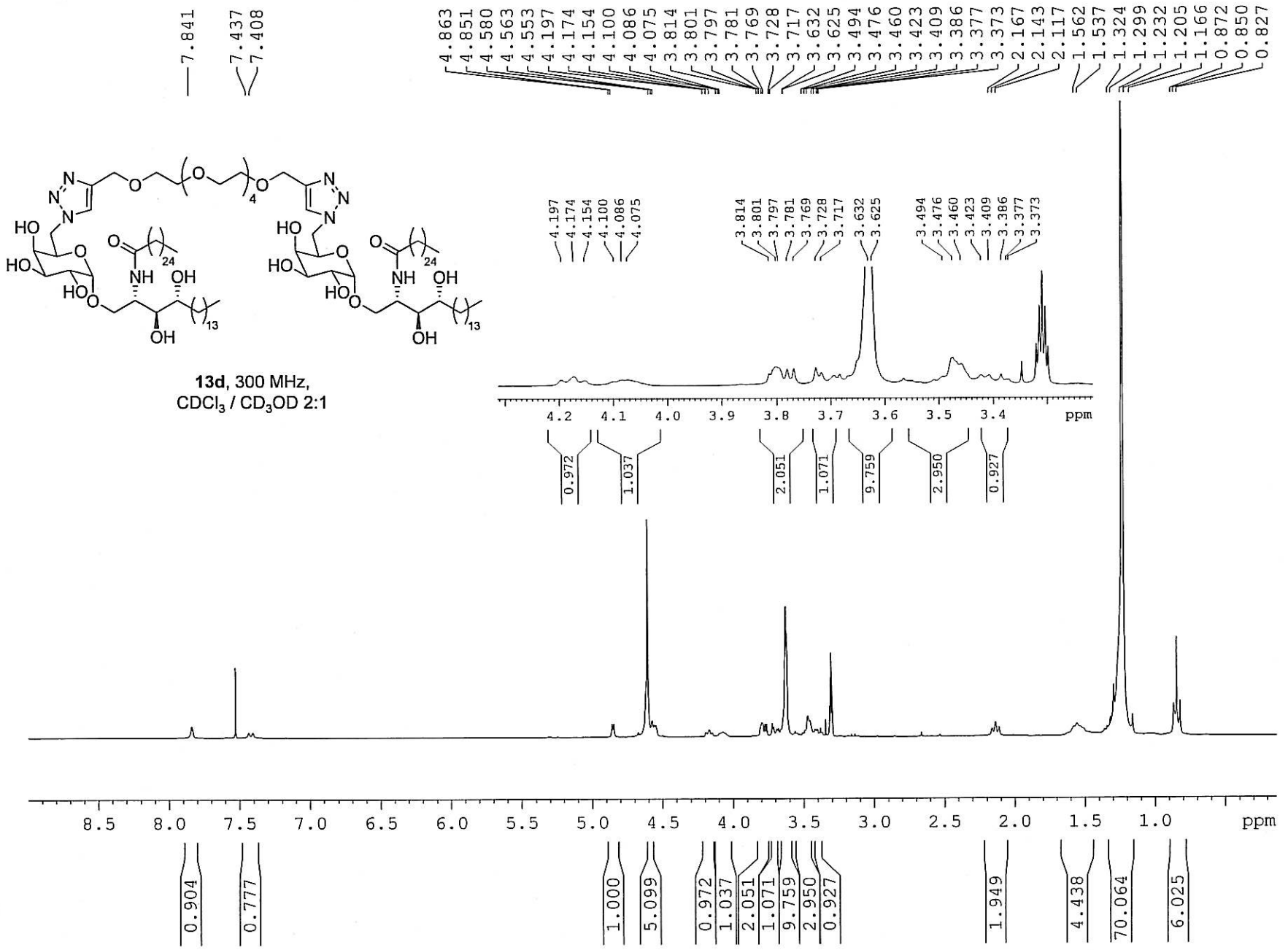


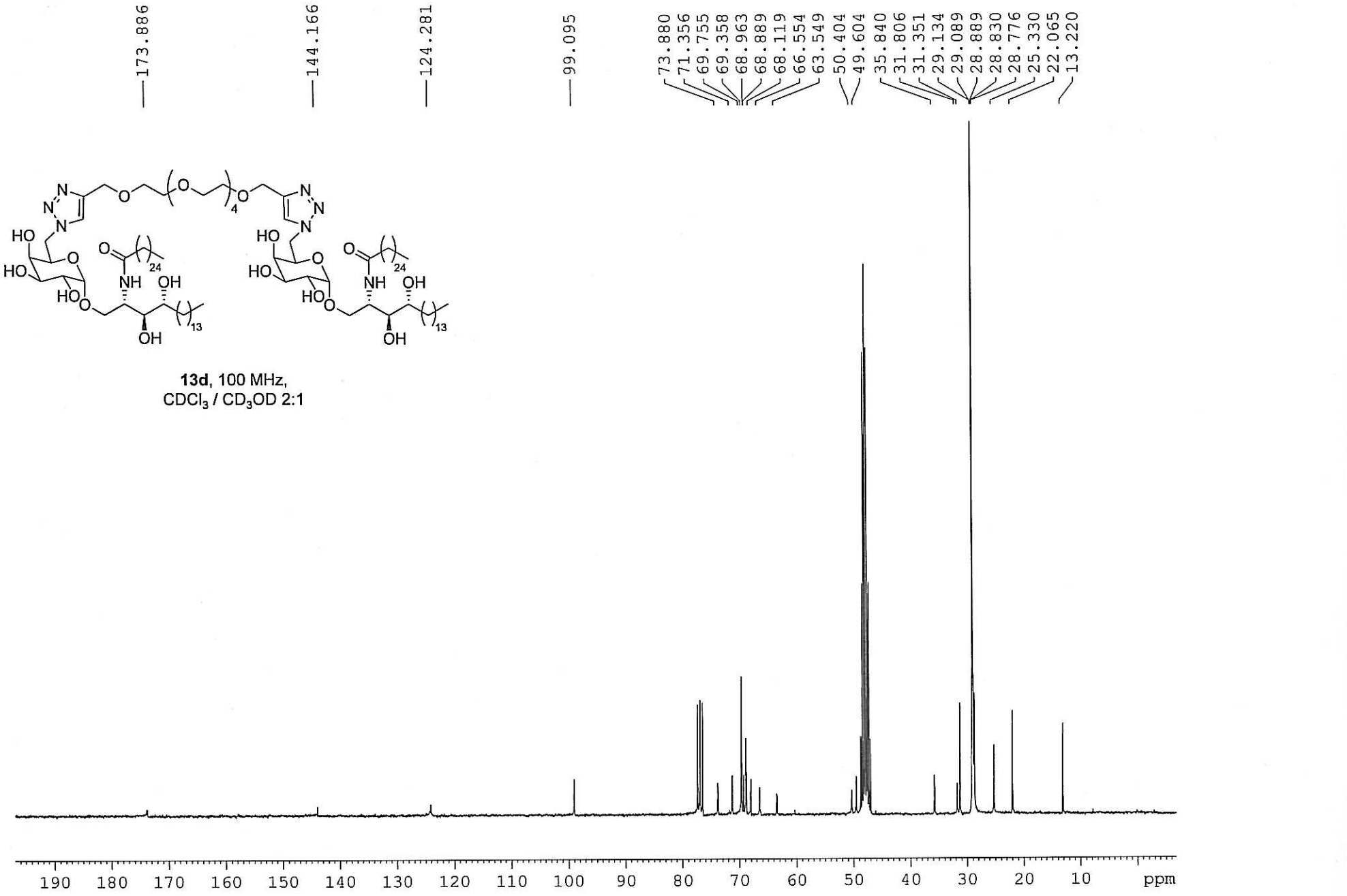
13b, 75 MHz,
$CDCl_3$ / CD_3OD 2:1


7.837
4.858
4.849
4.629
4.569
4.194
4.178
4.164
4.085
4.076
3.792
3.780
3.769
3.718
3.693
3.686
3.677
3.651
3.644
3.631
3.580
3.567
3.555
3.545
3.502
3.469
3.413
3.403
3.387
2.159
2.141
2.123
1.617
1.588
1.230
0.864
0.848
0.831
13c, 400 MHz,
CDCl3 / CD3OD 2:1
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 ppm
0.934
1.000
1.998
2.199
0.987
1.017
2.161
6.985
0.653
3.030
1.180
1.926
3.989
73.996
6.058

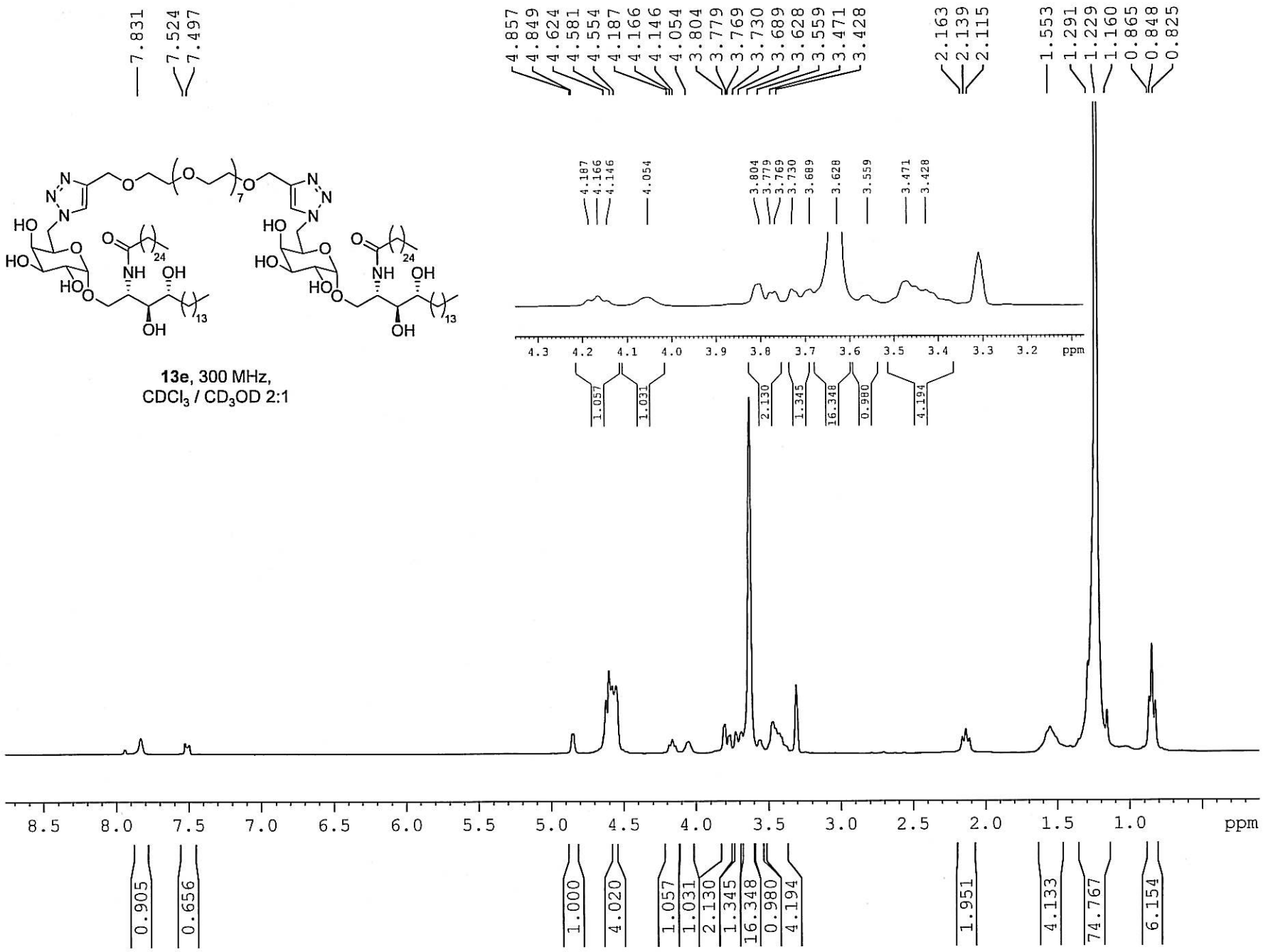


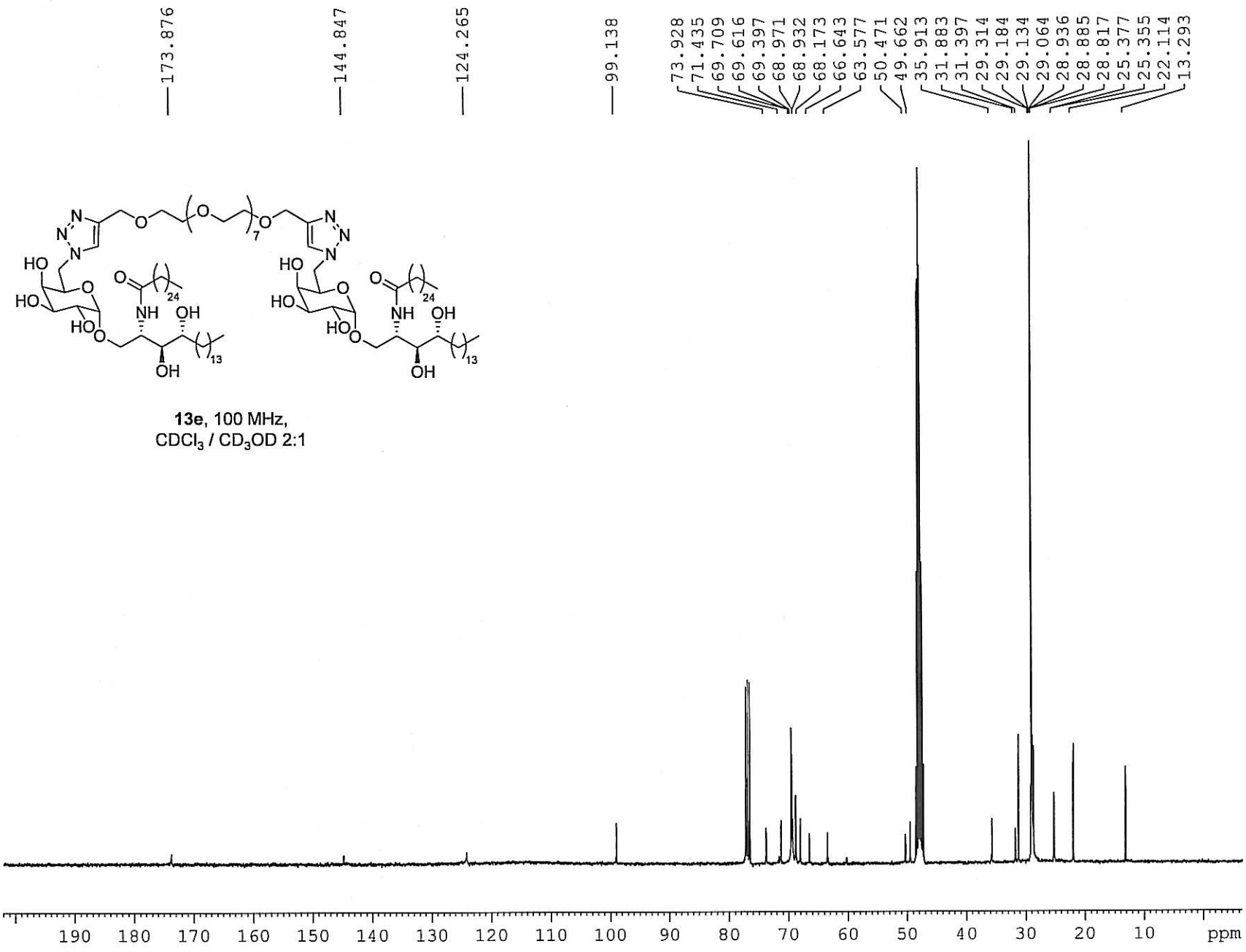
13c, 100 MHz,
$CDCl_3$ / CD_3OD 2:1


7.841
7.437
7.408
4.863
4.851
4.580
4.563
4.553
4.197
4.174
4.154
4.100
4.086
4.075
3.814
3.801
3.797
3.781
3.769
3.728
3.717
3.632
3.625
3.494
3.476
3.460
3.423
3.409
3.386
3.377
3.373
2.167
2.143
2.117
1.562
1.537
1.324
1.299
1.232
1.205
1.166
0.872
0.850
0.827
HO
HO
HO
HO
O
N
N
N
O
4
O
O
O
24
NH
OH
13
OH
HO
HO
HO
HO
O
N
N
N
O
O
O
24
NH
OH
13
OH
13d, 300 MHz,
CDCl3 / CD3OD 2:1
4.2
4.1
4.0
3.9
3.8
3.7
3.6
3.5
3.4
ppm
0.972
1.037
2.051
1.071
9.759
2.950
0.927
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
ppm
0.904
0.777
1.000
5.099
0.972
1.037
2.051
1.071
9.759
2.950
0.927
1.949
4.438
70.064
6.025

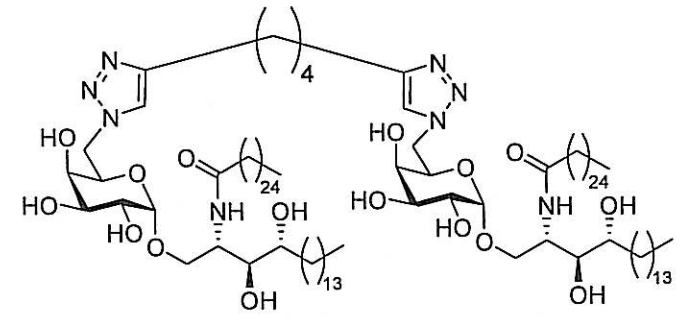


13d, 100 MHz,
$CDCl_3$ / CD_3OD 2:1

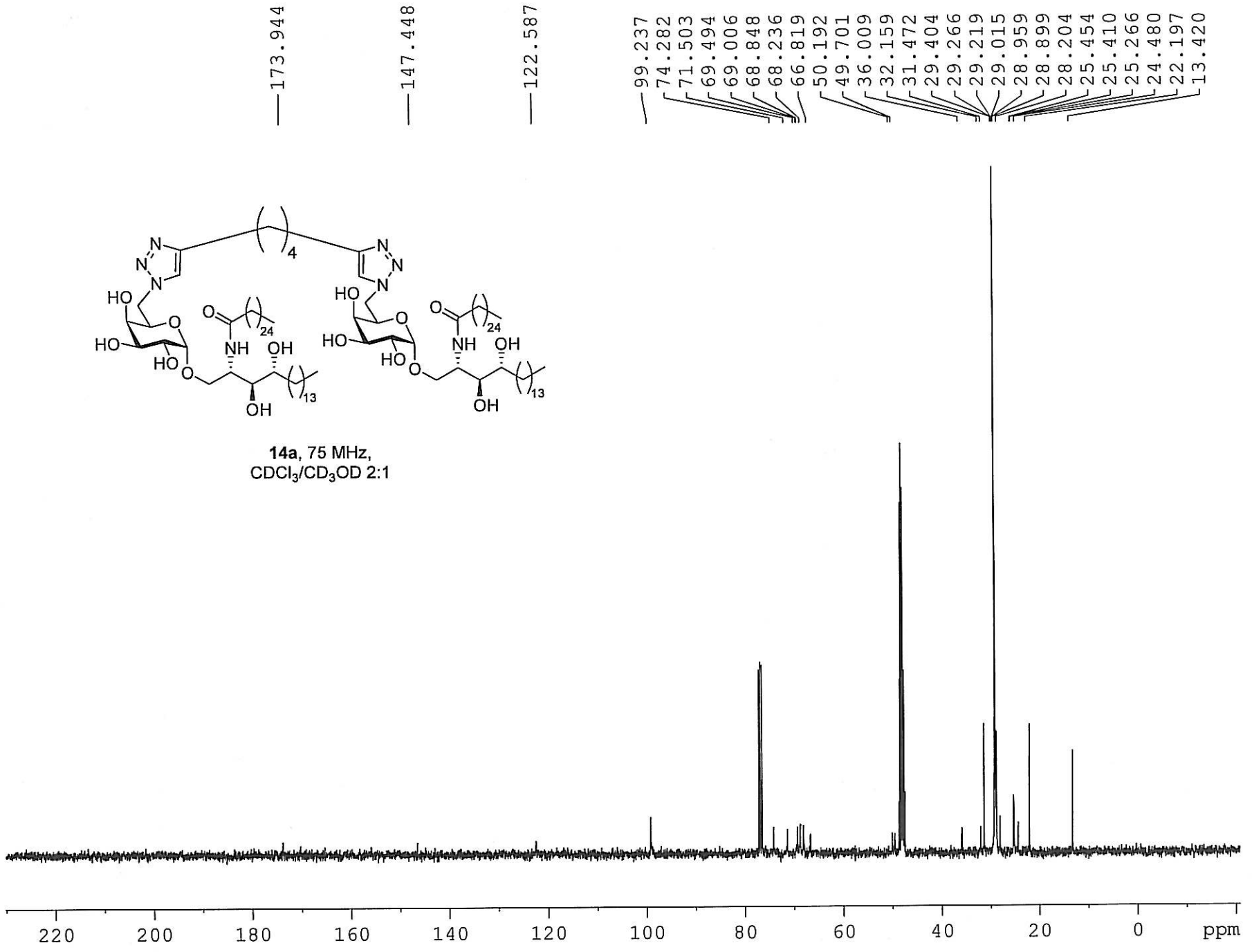

7.831
7.524
7.497
4.857
4.849
4.624
4.581
4.554
4.187
4.166
4.146
4.054
3.804
3.779
3.769
3.730
3.689
3.628
3.559
3.471
3.428
2.163
2.139
2.115
1.553
1.291
1.229
1.160
0.865
0.848
0.825
HO
HO
HO
O
N
N
N
O
7
O
NH
OH
24
13
OH
13e, 300 MHz,
CDCl3 / CD3OD 2:1
4.3
4.2
4.1
4.0
3.9
3.8
3.7
3.6
3.5
3.4
3.3
3.2
ppm
1.057
1.031
2.130
1.345
16.348
0.980
4.194
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
ppm
0.905
0.656
1.000
4.020
1.057
1.031
2.130
1.345
16.348
0.980
4.194
1.951
4.133
74.767
6.154



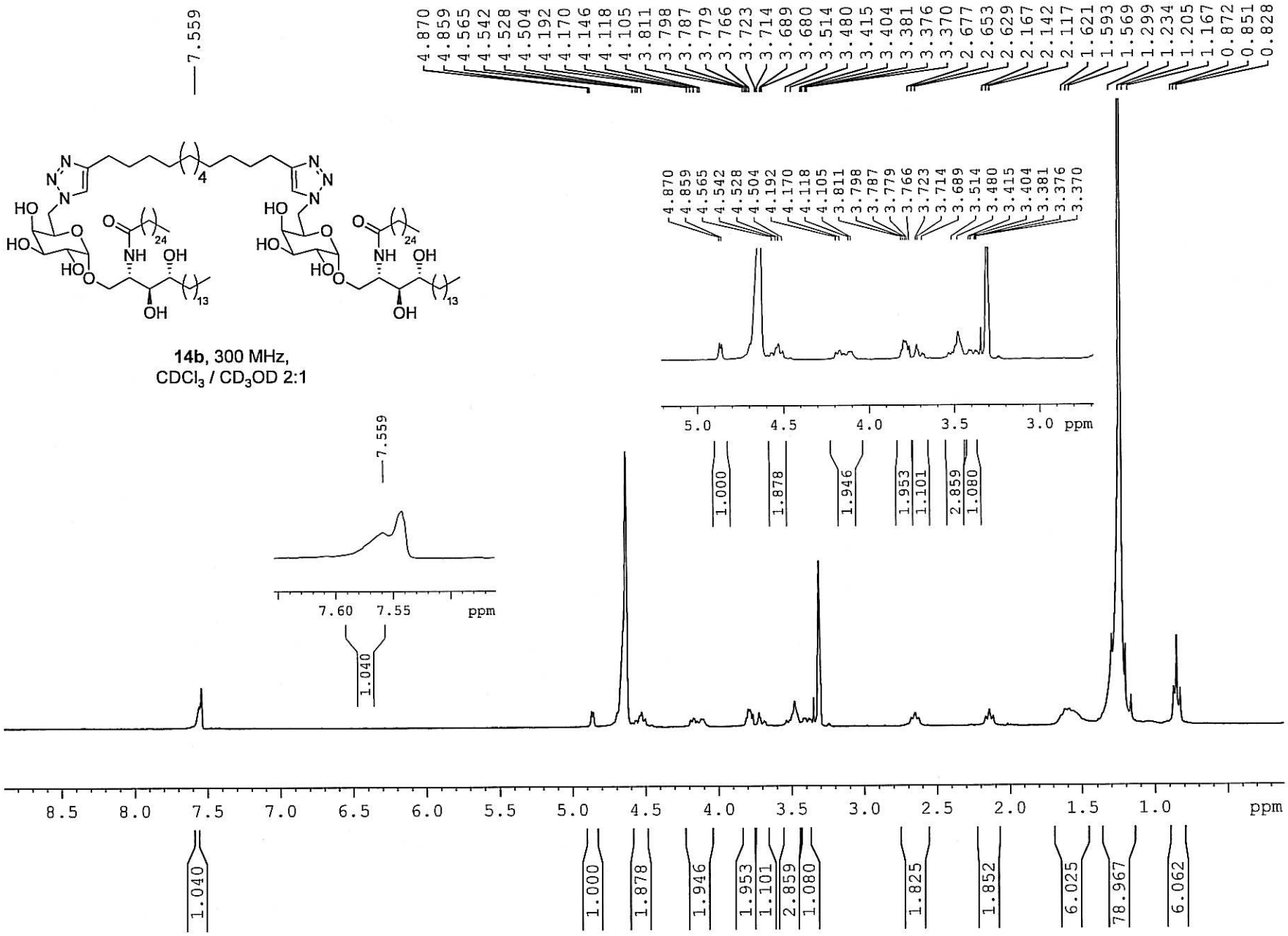
13e, 100 MHz,
$CDCl_3$ / CD_3OD 2:1

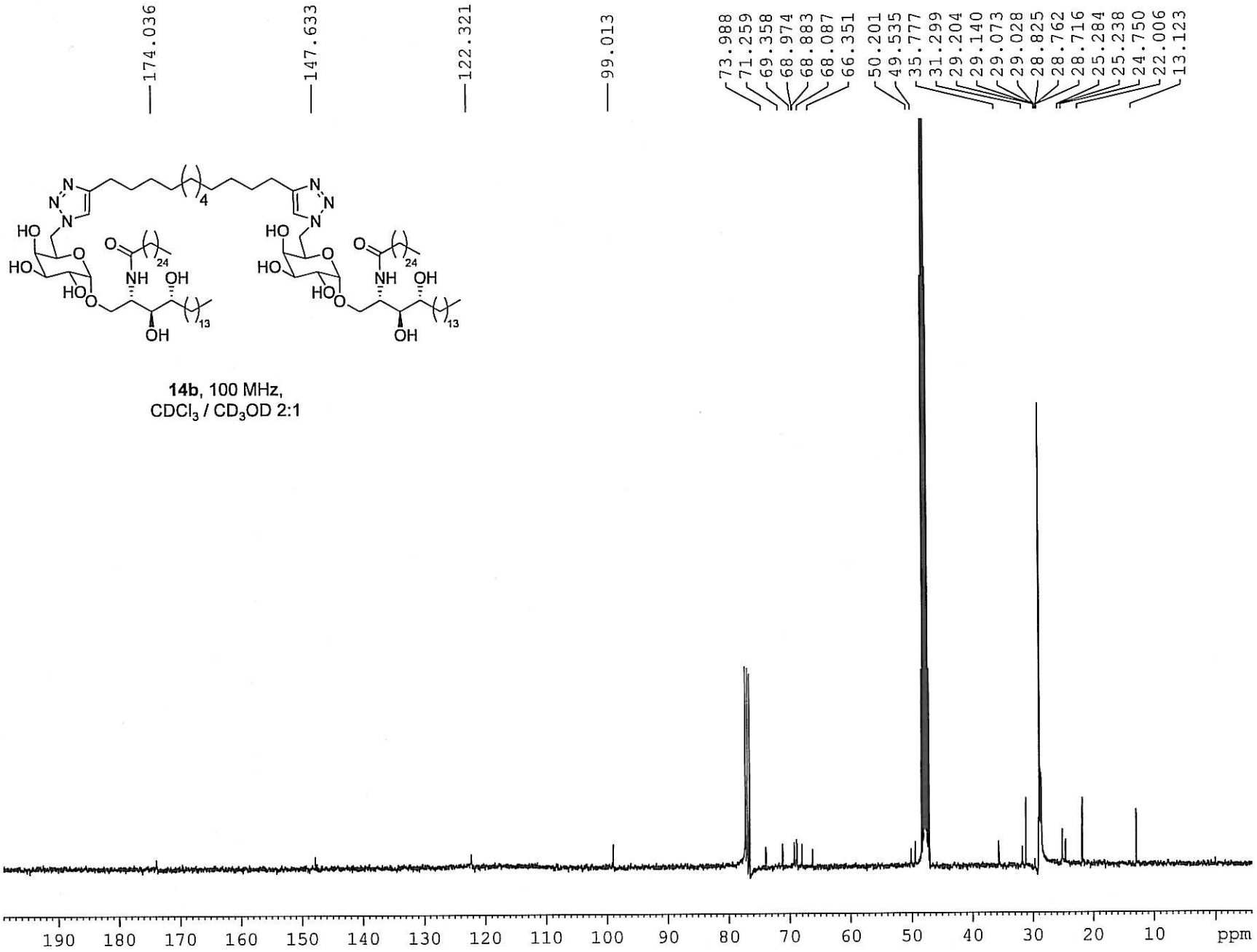


14a, 300 MHz,
$CDCl_3/CD_3OD$ 2:1

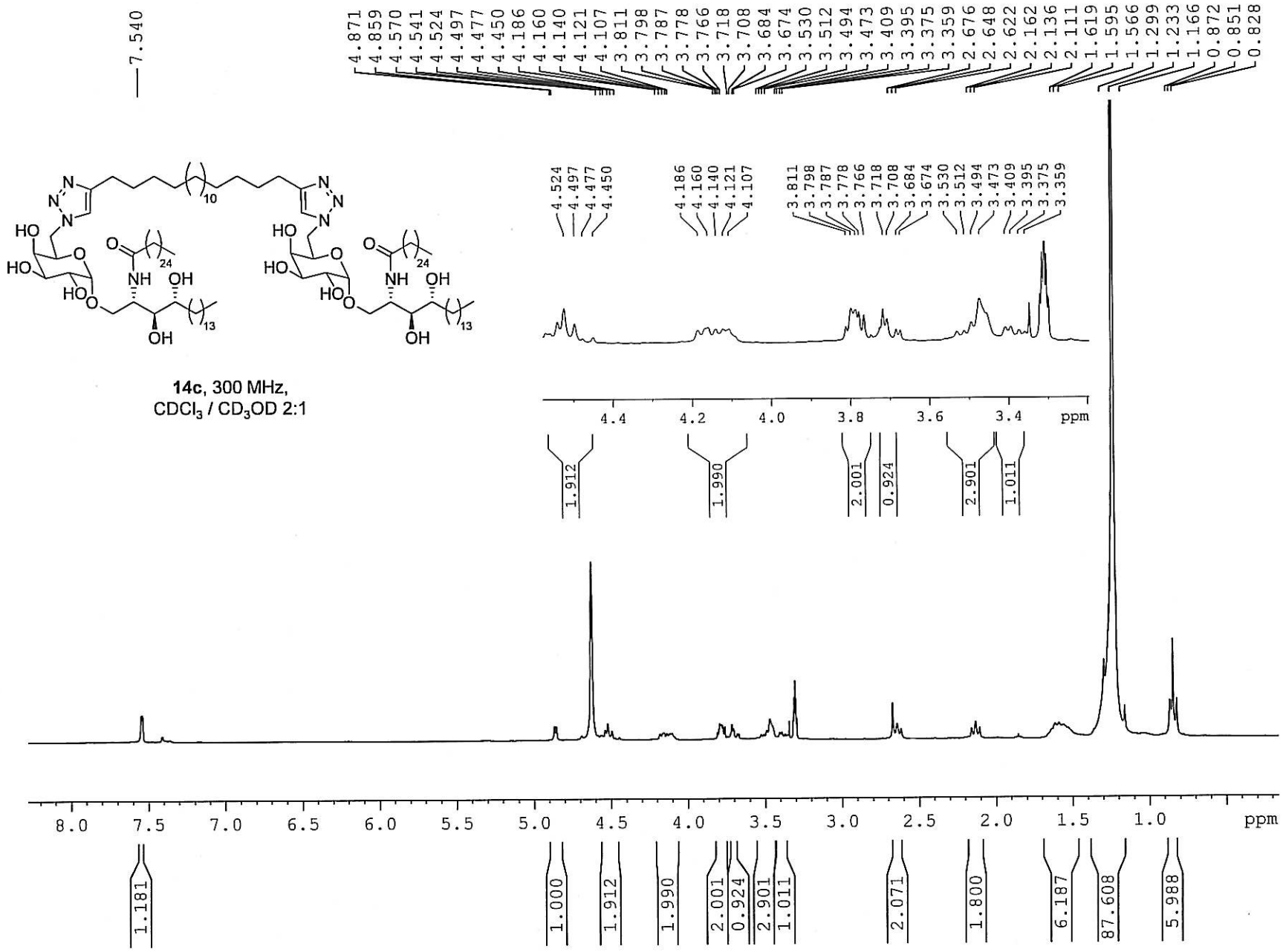


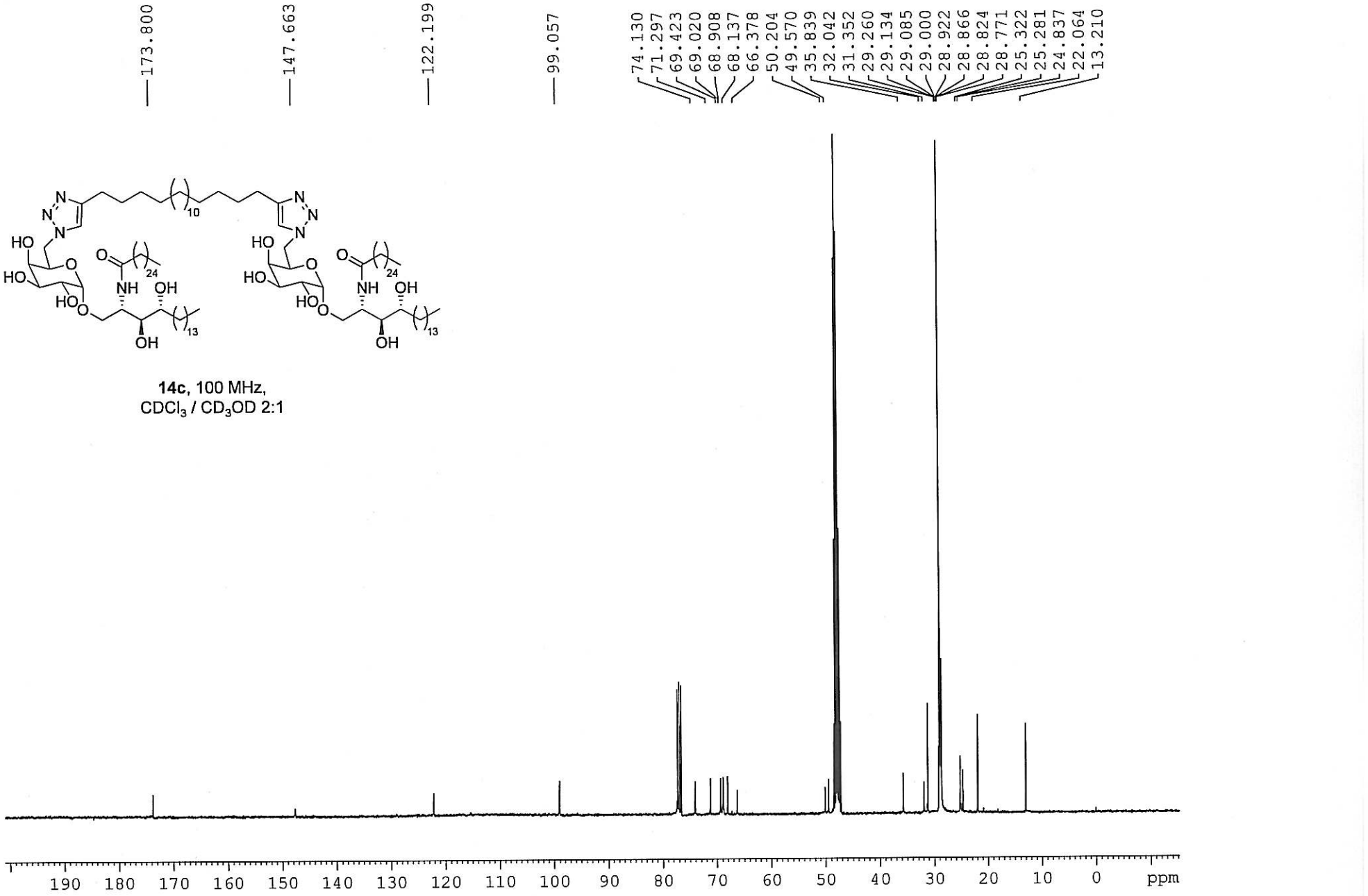
14a, 75 MHz,
$CDCl_3/CD_3OD$ 2:1


7.559
4.870
4.859
4.565
4.542
4.528
4.504
4.192
4.170
4.146
4.118
4.105
3.811
3.798
3.787
3.779
3.766
3.723
3.714
3.689
3.680
3.514
3.480
3.415
3.404
3.381
3.376
3.370
2.677
2.653
2.629
2.167
2.142
2.117
1.621
1.593
1.569
1.299
1.234
1.205
1.167
0.872
0.851
0.828
HO
HO
HO
O
N
N
N
4
24
NH
OH
13
14b, 300 MHz,
CDCl3 / CD3OD 2:1
5.0
4.5
4.0
3.5
3.0 ppm
1.000
1.878
1.946
1.953
1.101
2.859
1.080
7.60
7.55
ppm
1.040
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
ppm
1.040
1.000
1.878
1.946
1.953
1.101
2.859
1.080
1.825
1.852
6.025
78.967
6.062



14b, 100 MHz,
$CDCl_3$ / CD_3OD 2:1


7.540
4.871
4.859
4.570
4.541
4.524
4.497
4.477
4.450
4.186
4.160
4.140
4.121
4.107
3.811
3.798
3.787
3.778
3.766
3.718
3.708
3.684
3.674
3.530
3.512
3.494
3.473
3.409
3.395
3.375
3.359
2.676
2.648
2.622
2.162
2.136
2.111
1.619
1.595
1.566
1.299
1.233
1.166
0.872
0.851
0.828
14c, 300 MHz,
CDCl3 / CD3OD 2:1
ppm
1.912
1.990
2.001
0.924
2.901
1.011
1.181
1.000
1.912
1.990
2.001
0.924
2.901
1.011
2.071
1.800
6.187
87.608
5.988



14c, 100 MHz,
$CDCl_3$ / CD_3OD 2:1