SUPPORTING INFORMATION

Synthesis and intramolecular nitrile oxide cycloaddition of 3,5'-ether-linked pseudooligosaccharide derivatives: an approach to chiral macrooxacycles

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[b] National Chemical Laboratory, Pune 411008, India

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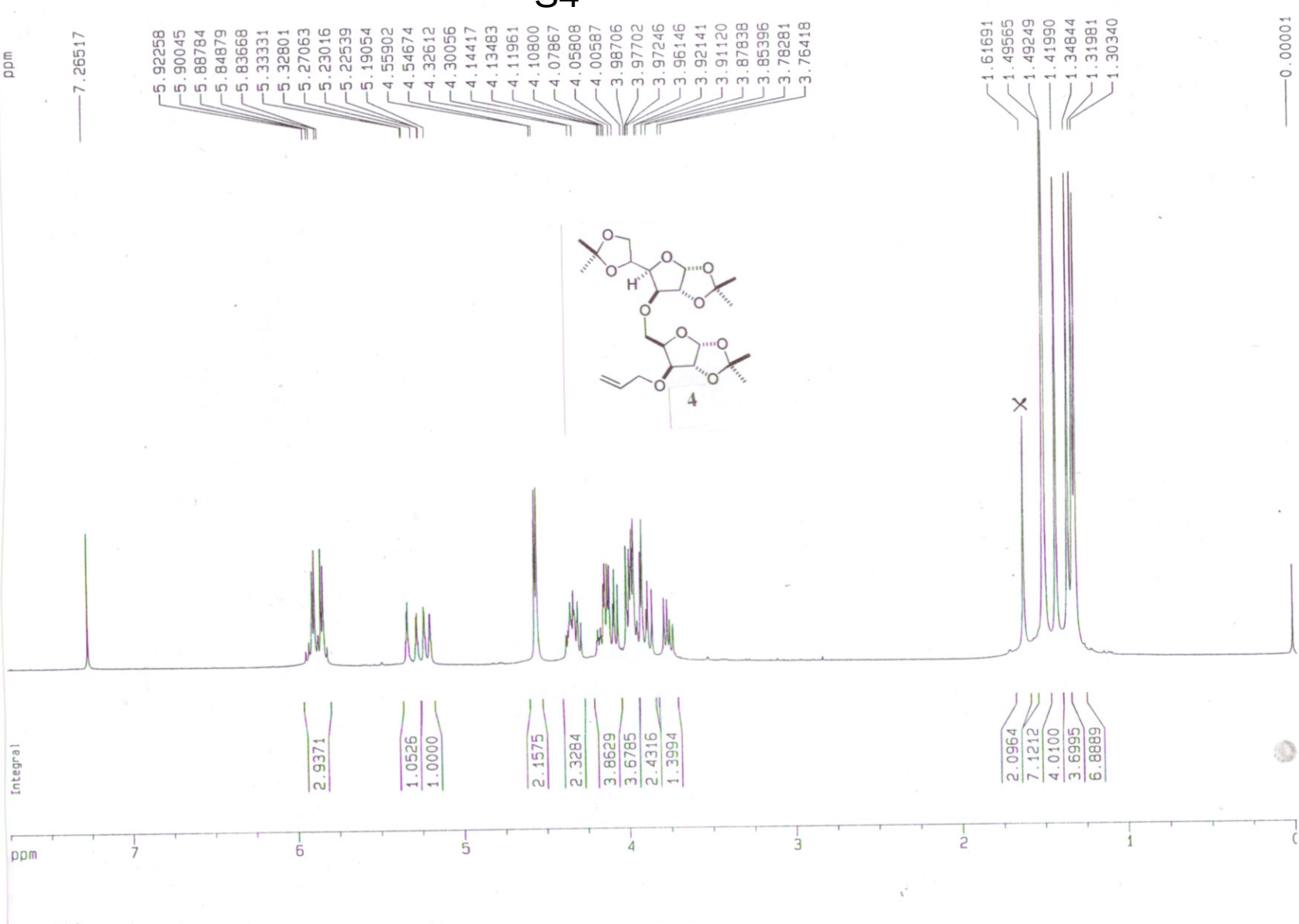
^{1}H and ^{13}C NMR spectra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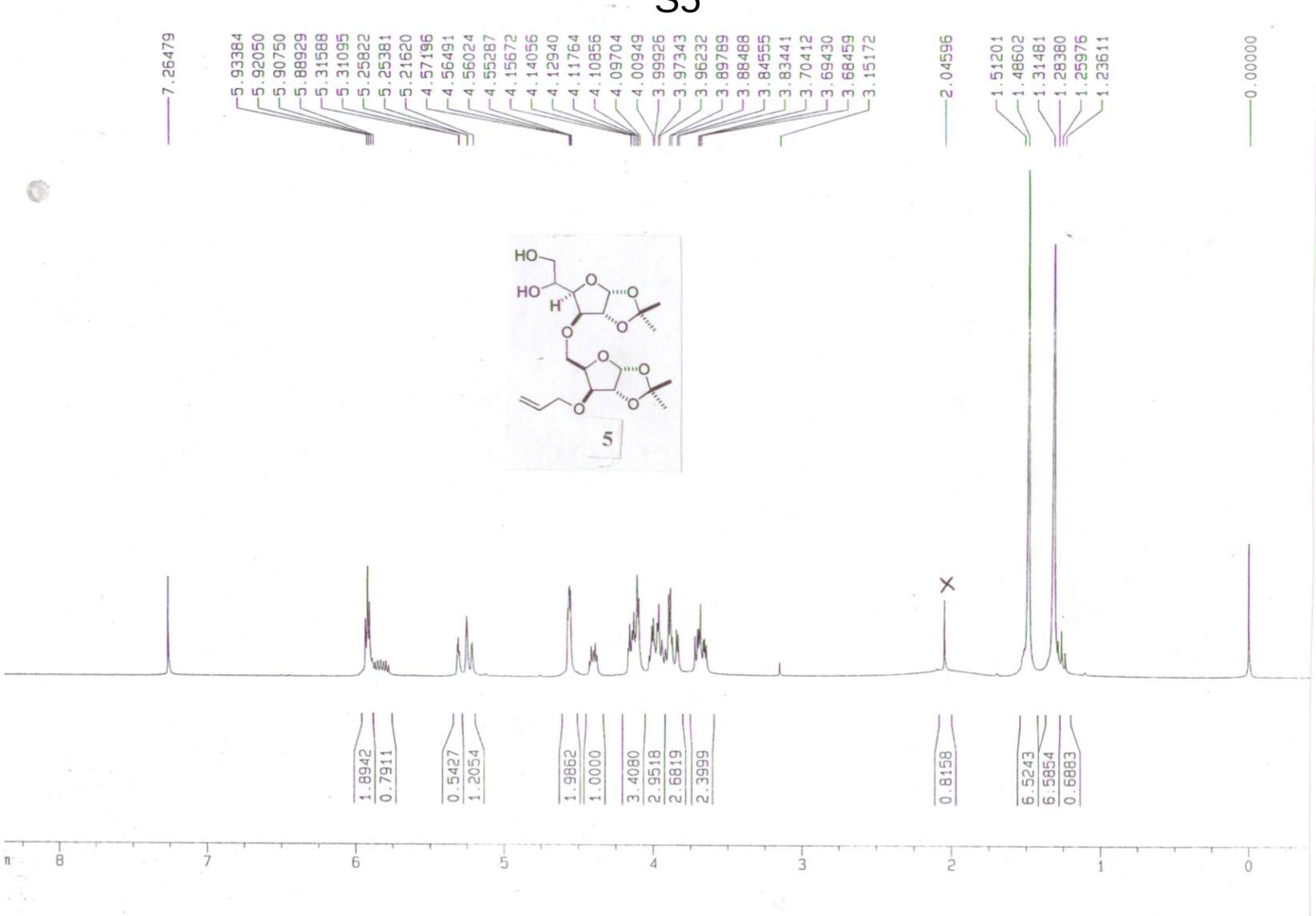
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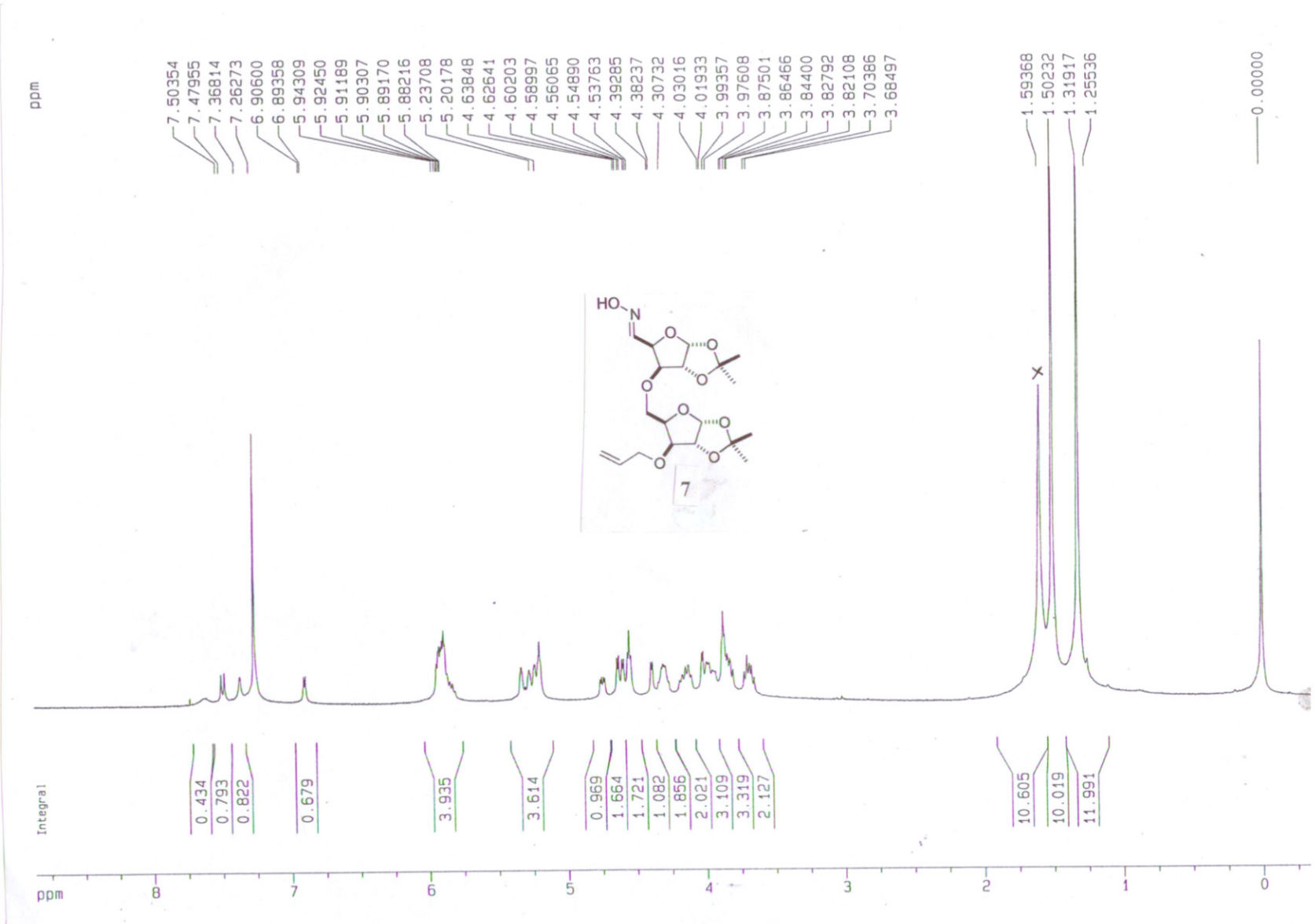
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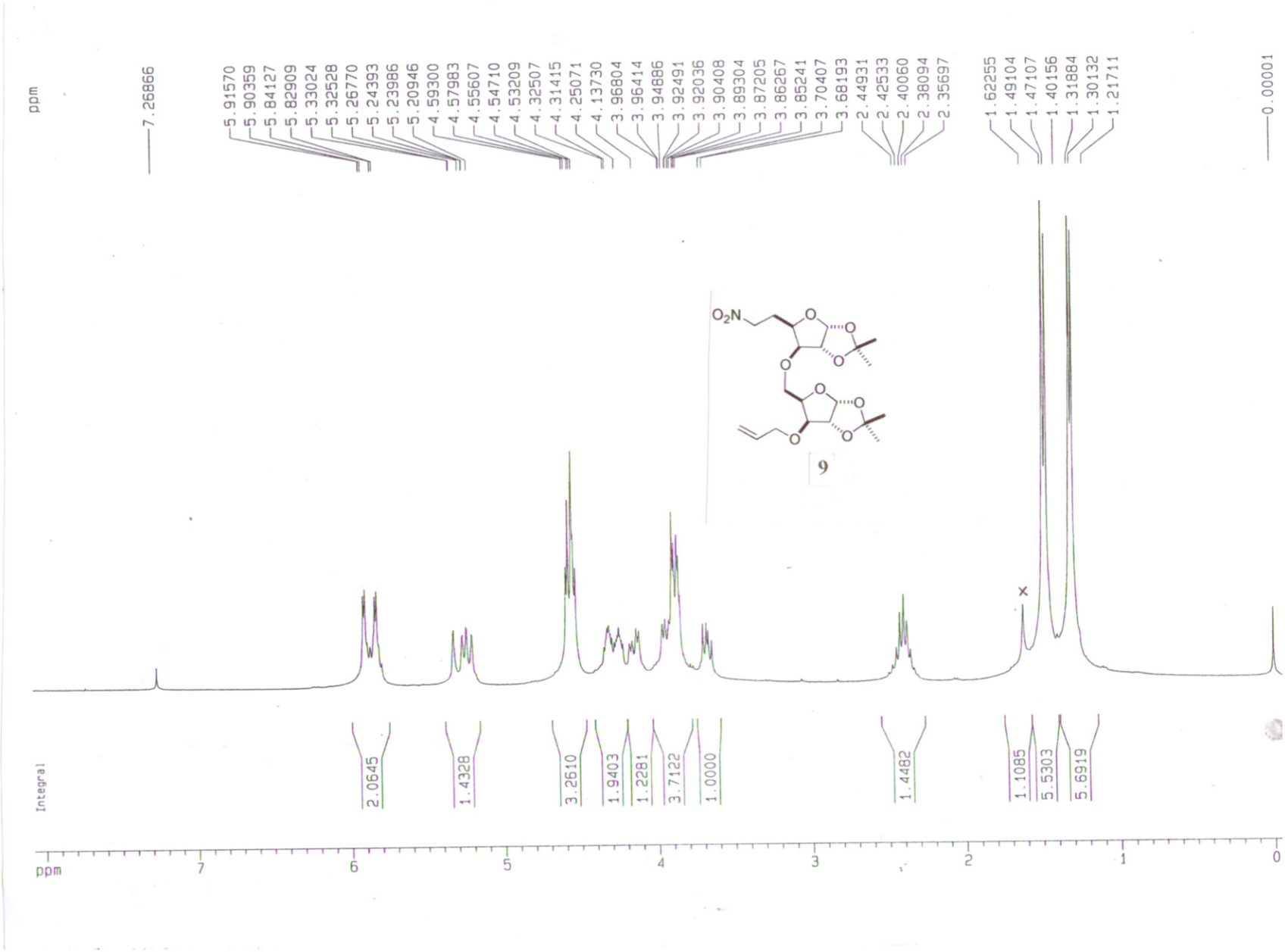
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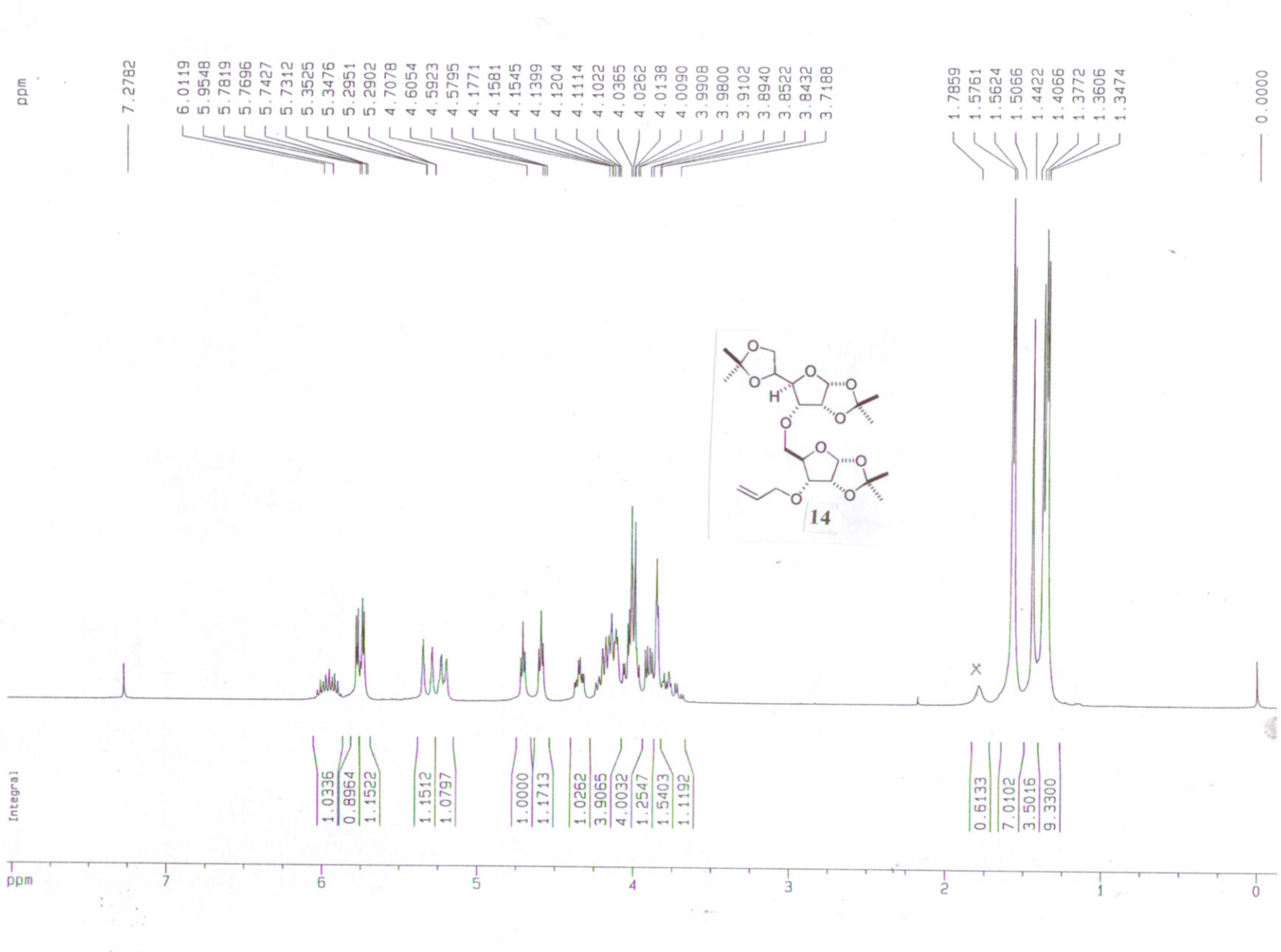
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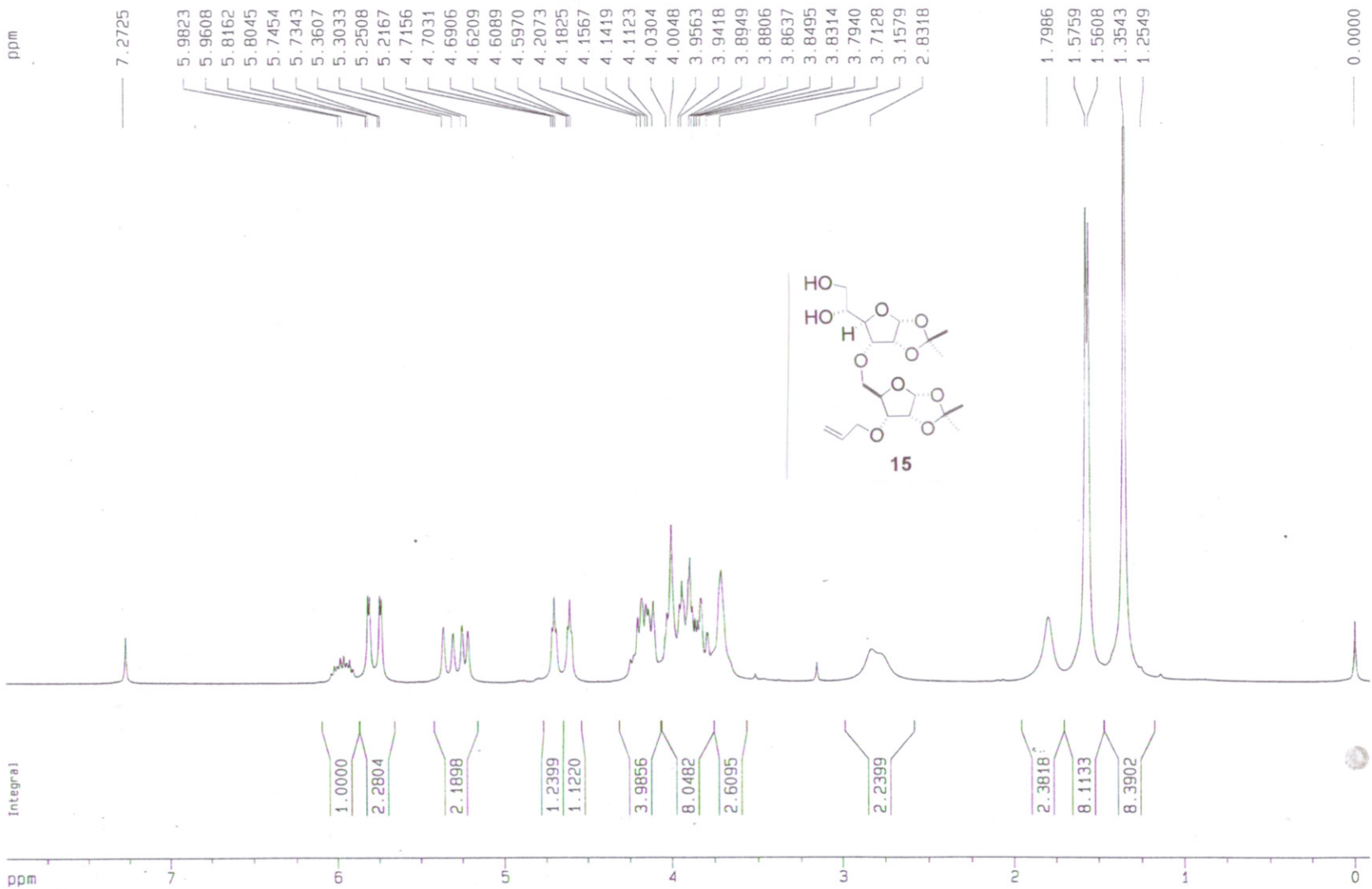
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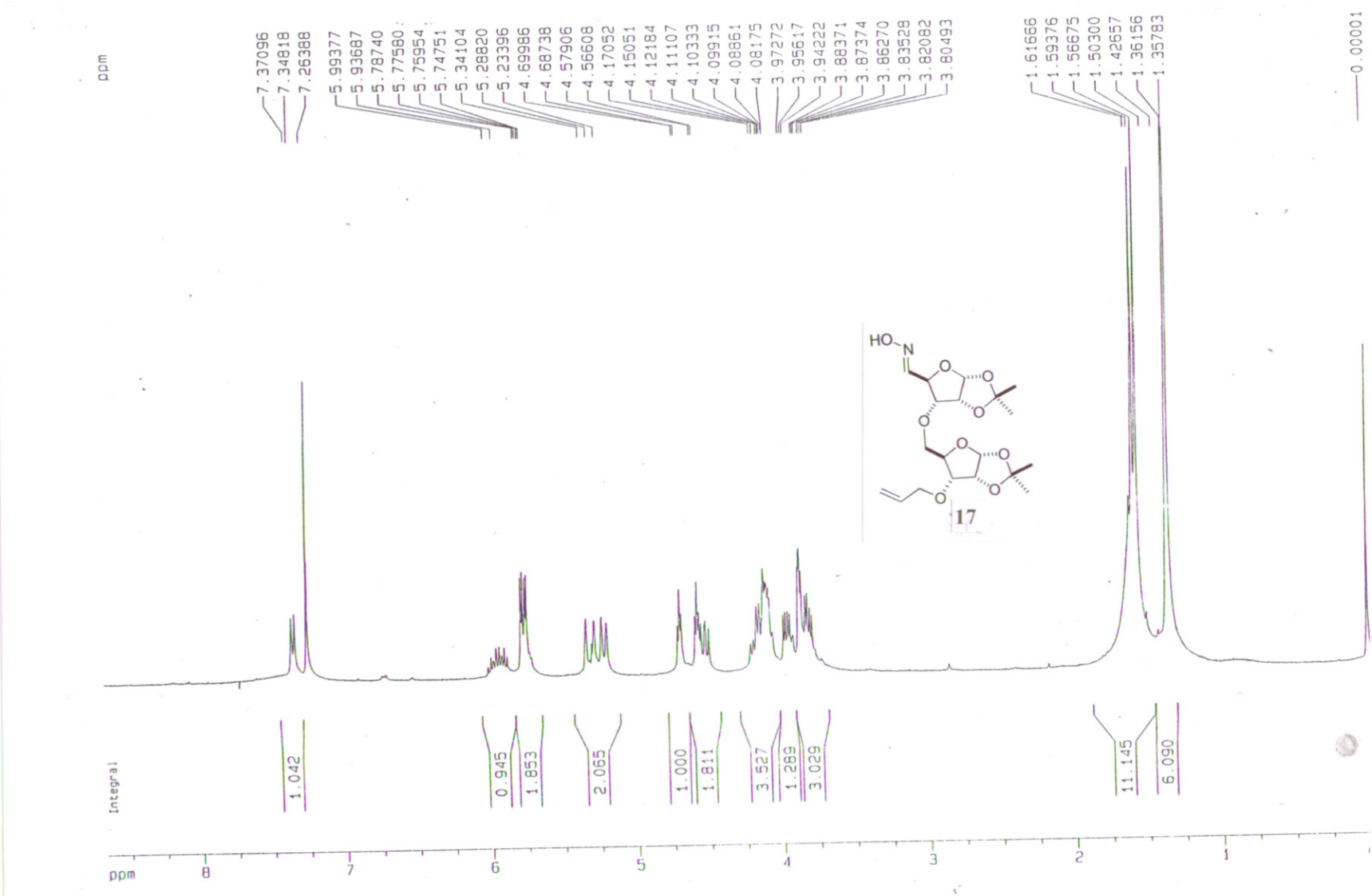
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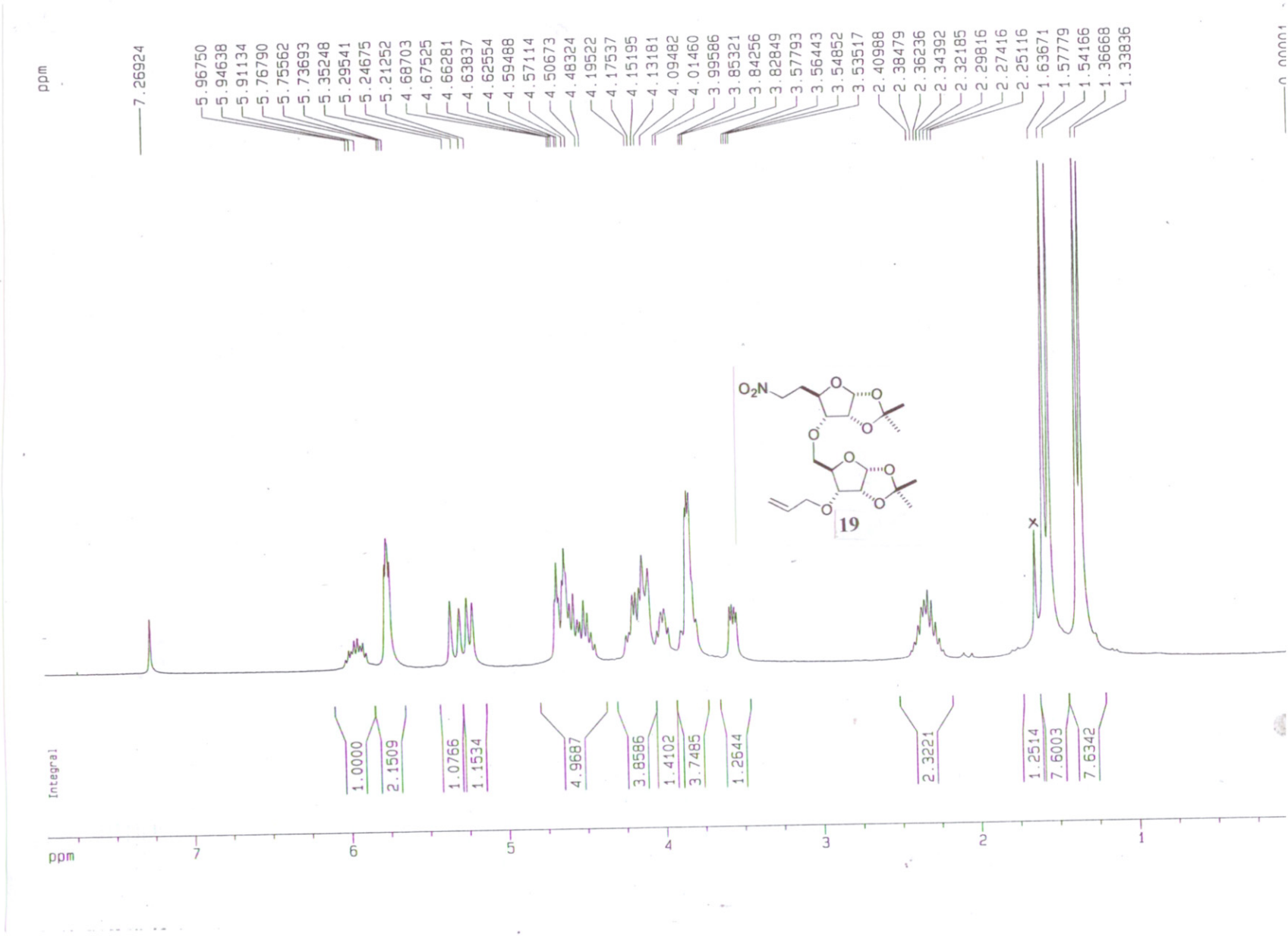
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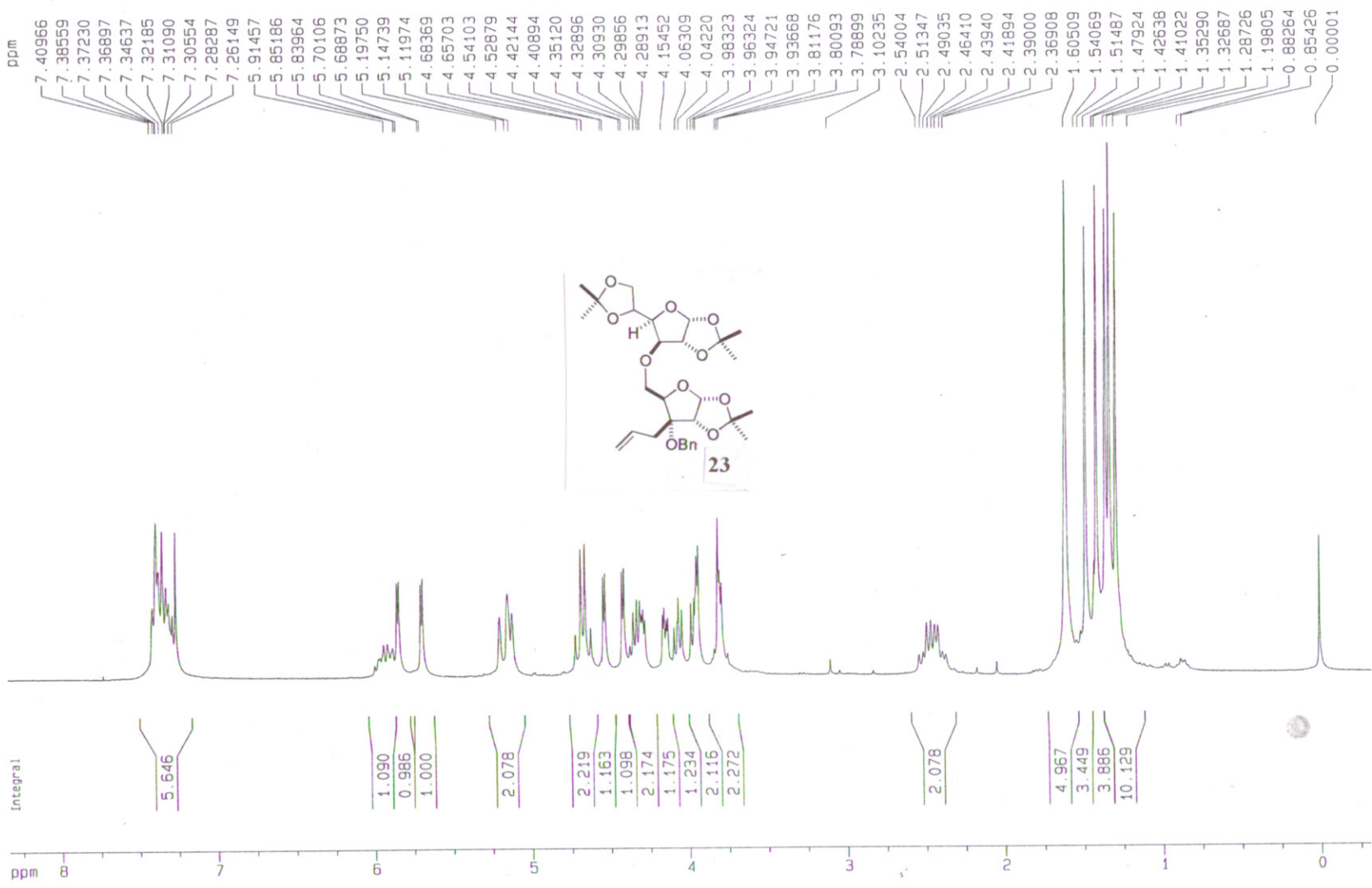
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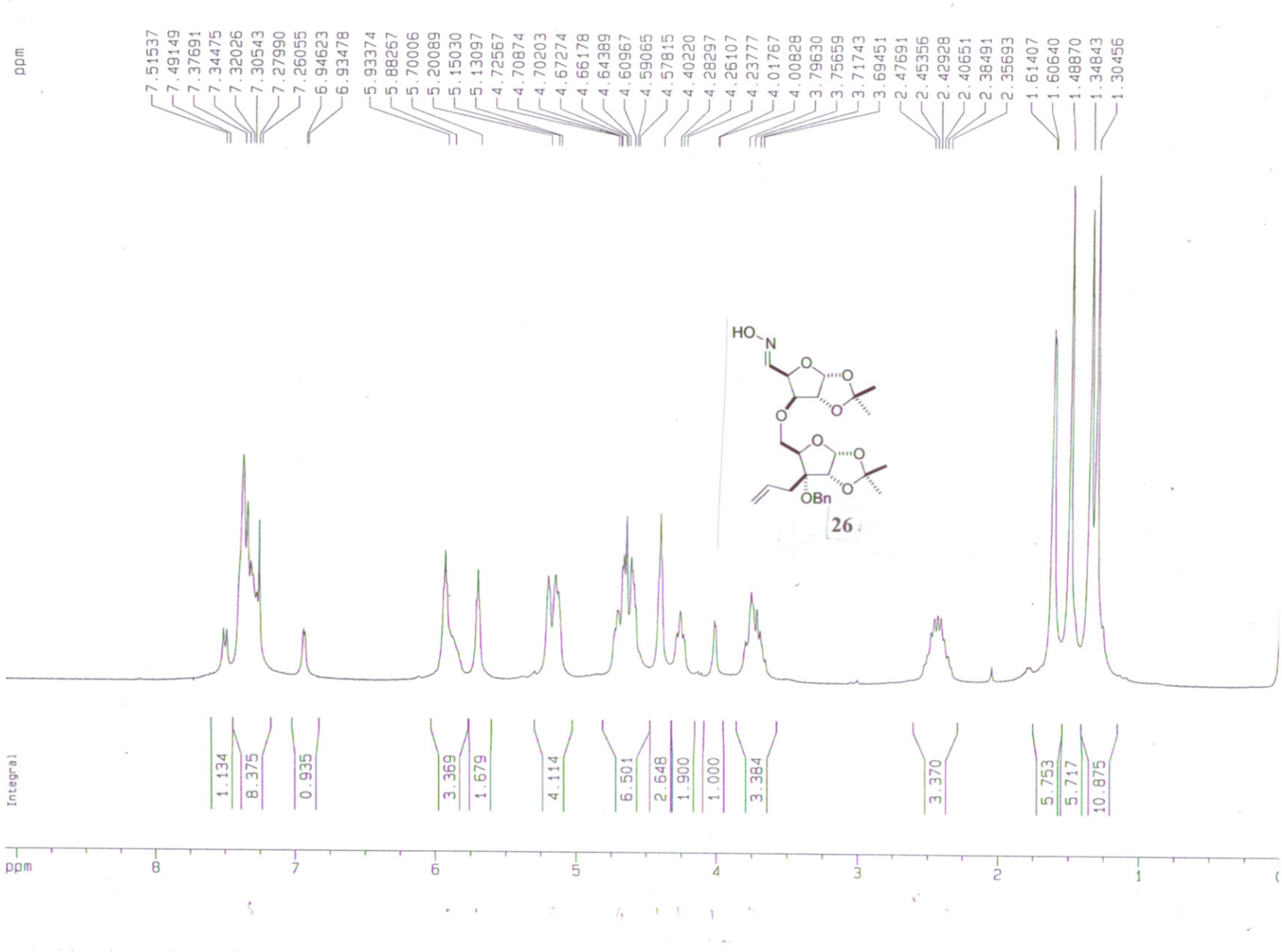
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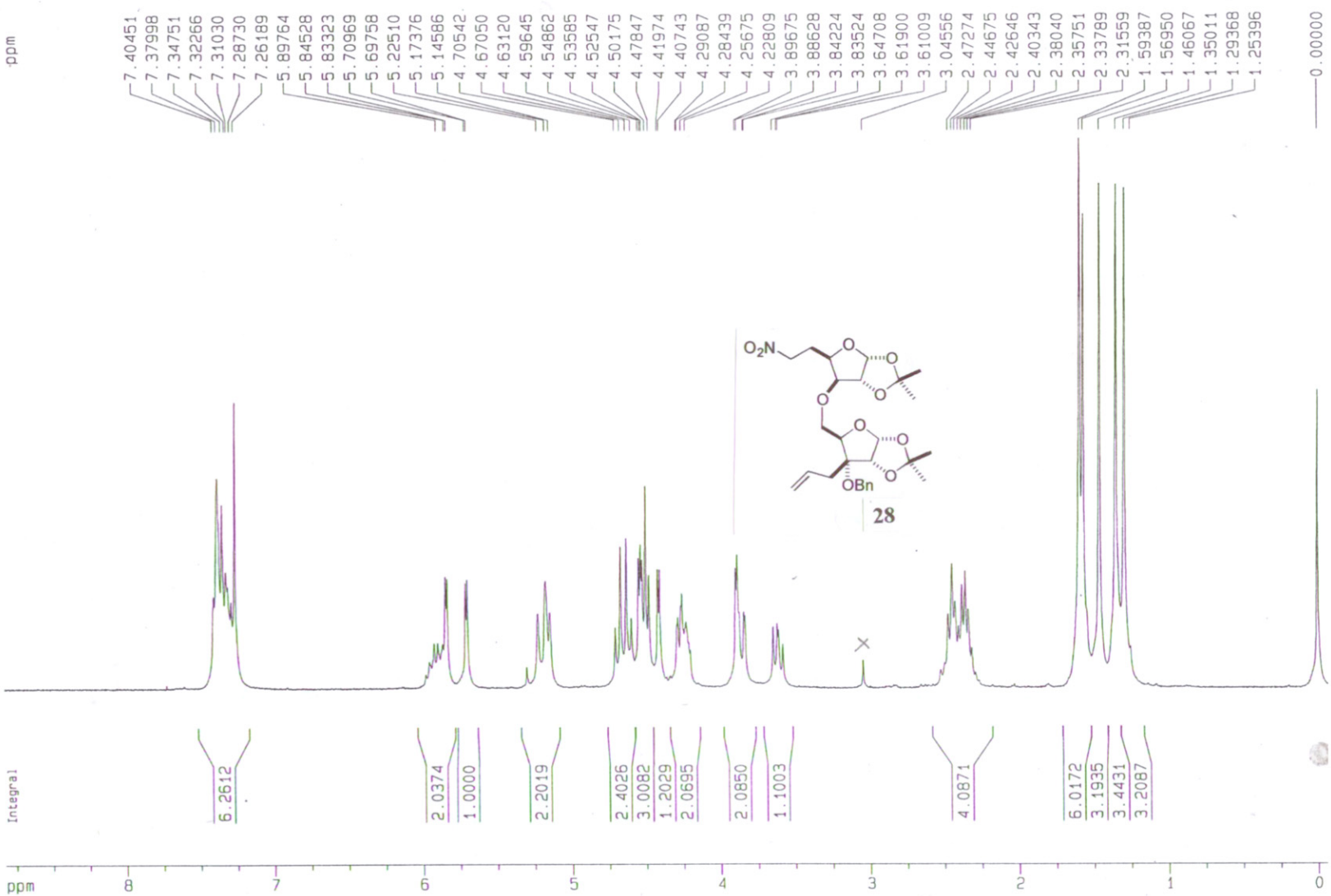
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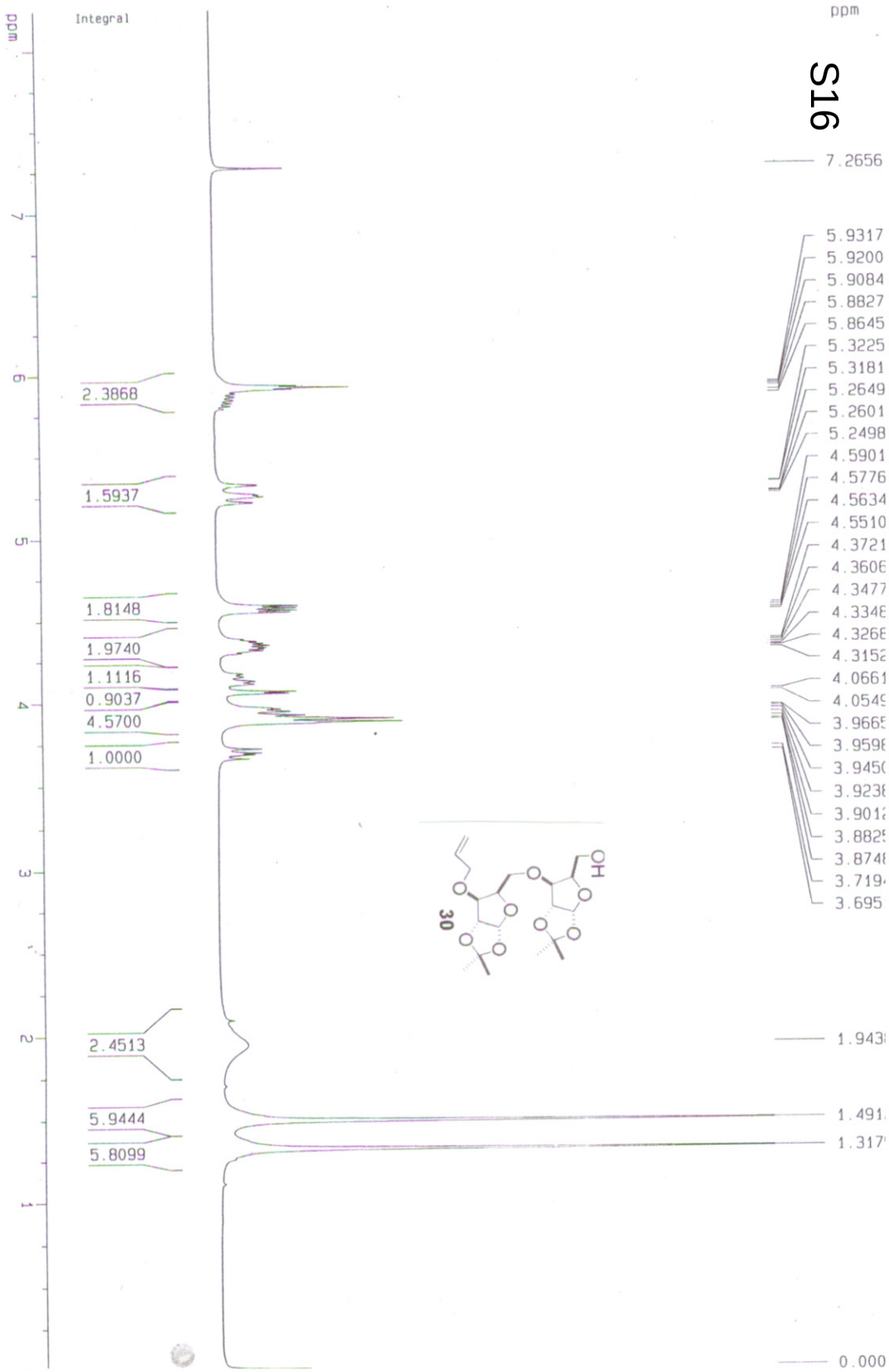
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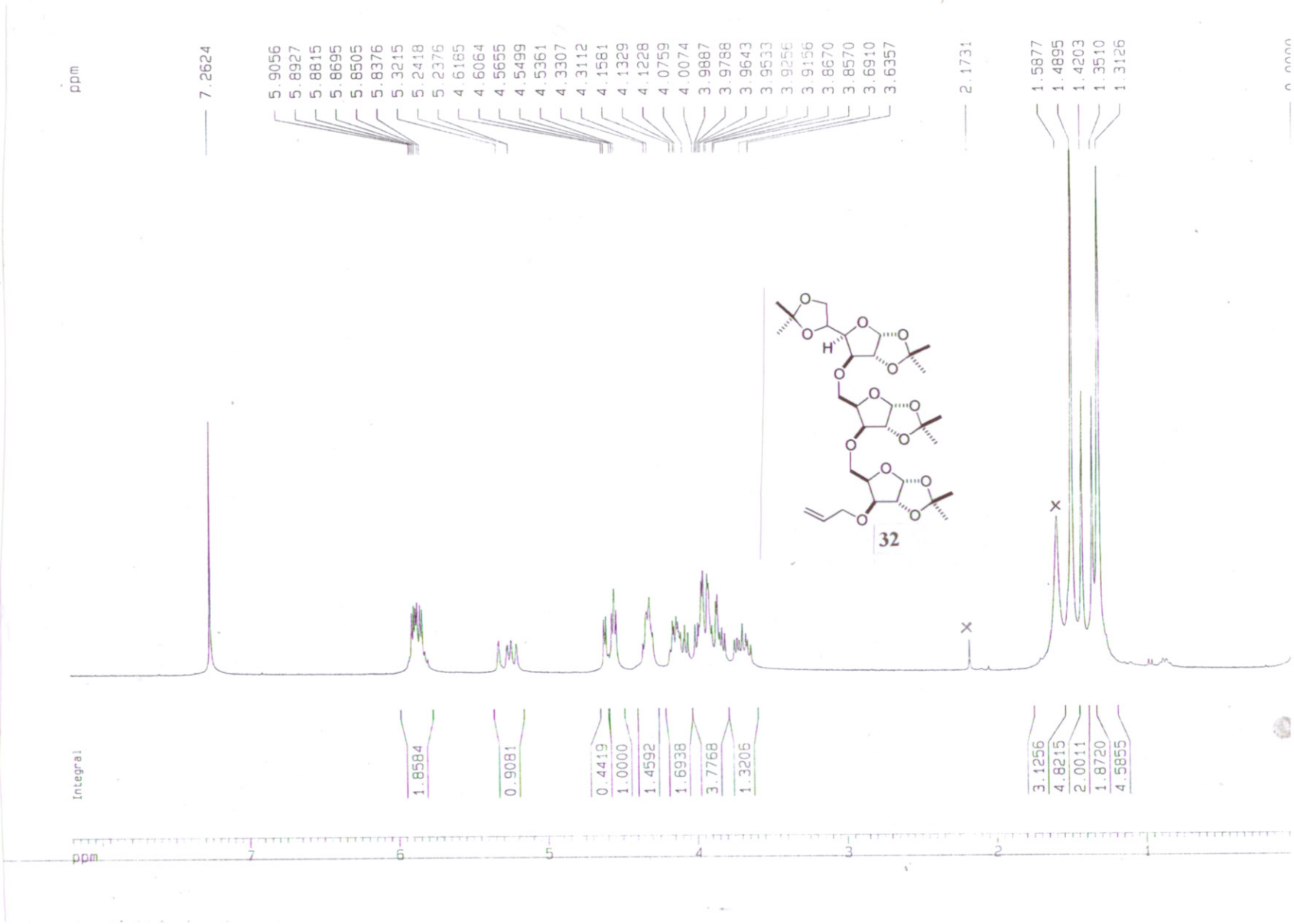
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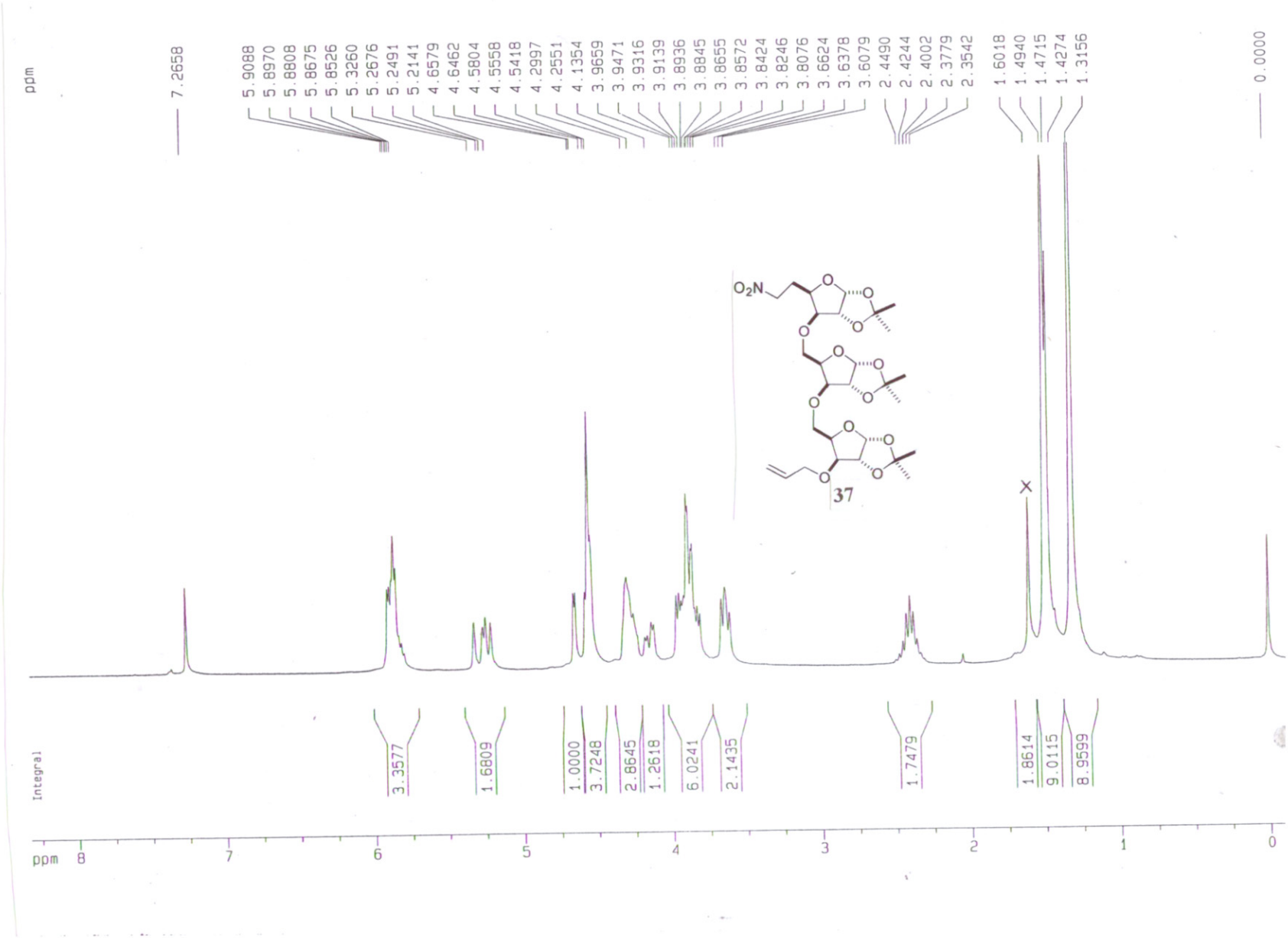
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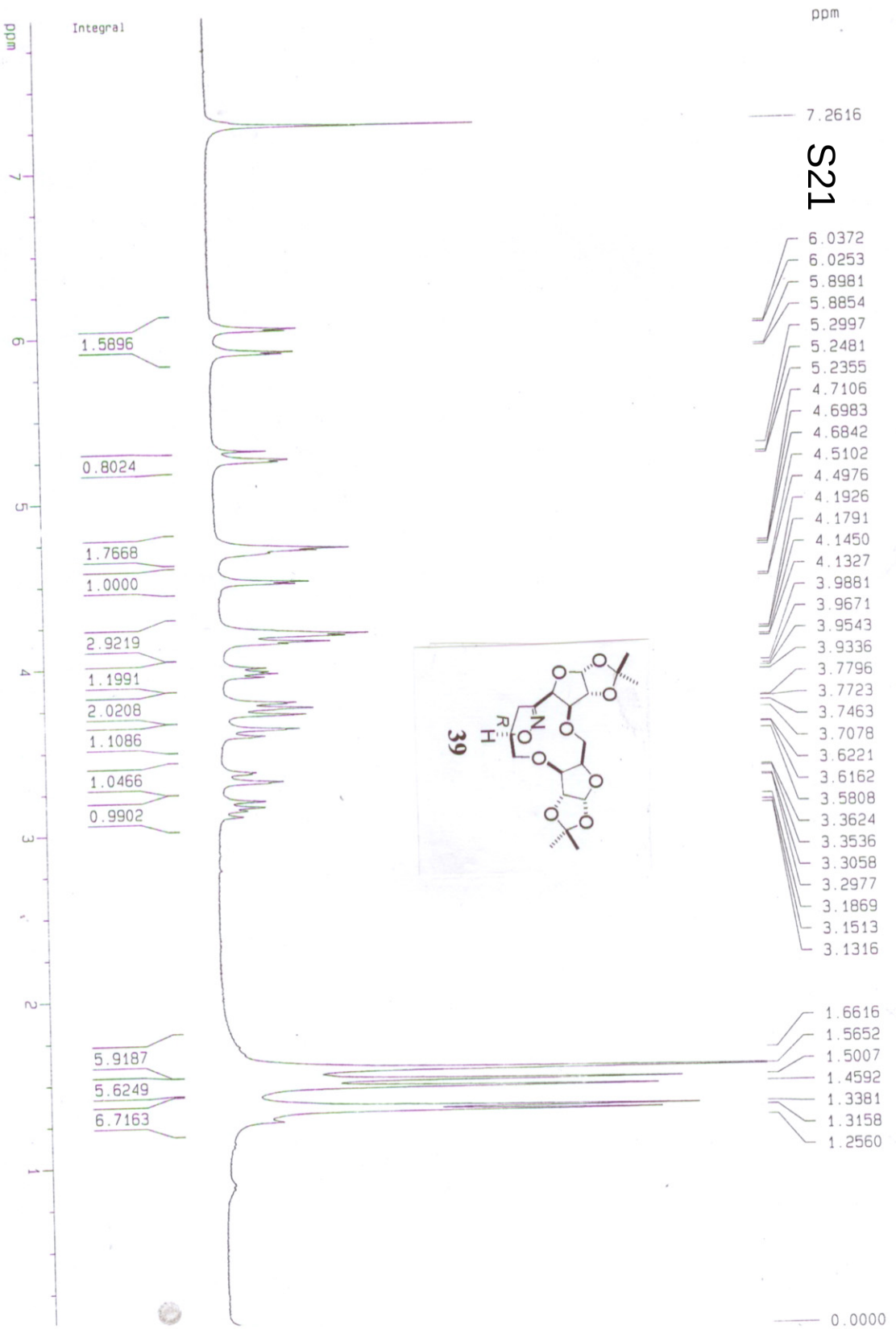
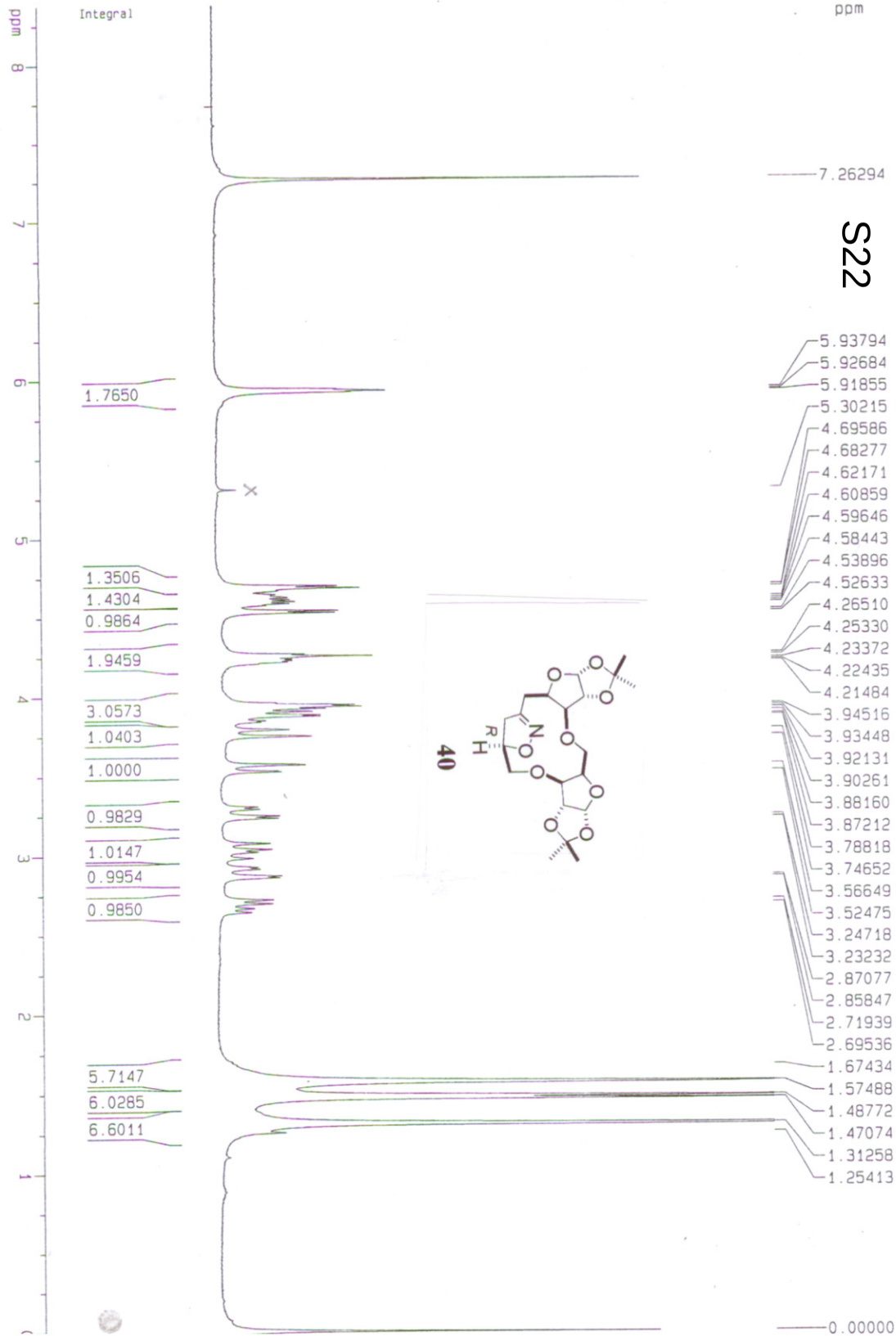
5.8970
5.8808
5.8675
5.8526
5.3260
5.2676
5.2491
5.2141
4.6579
4.6462
4.5804
4.5558
4.5418
4.2997
4.2551
4.1354
3.9659
3.9471
3.9316
3.9139
3.8936
3.8845
3.8655
3.8572
3.8424
3.8246
3.8076
3.6624
3.6378
3.6079
2.4490
2.4244
2.4002
2.3779
2.3542
1.6018
1.4940
1.4715
1.4274
1.3156
0.0000
O2N
37
X
Integral
3.3577
1.6809
1.0000
3.7248
2.8645
1.2618
6.0241
2.1435
1.7479
1.8614
9.0115
8.9599
ppm 8
7
6
5
4
3
2
1
0

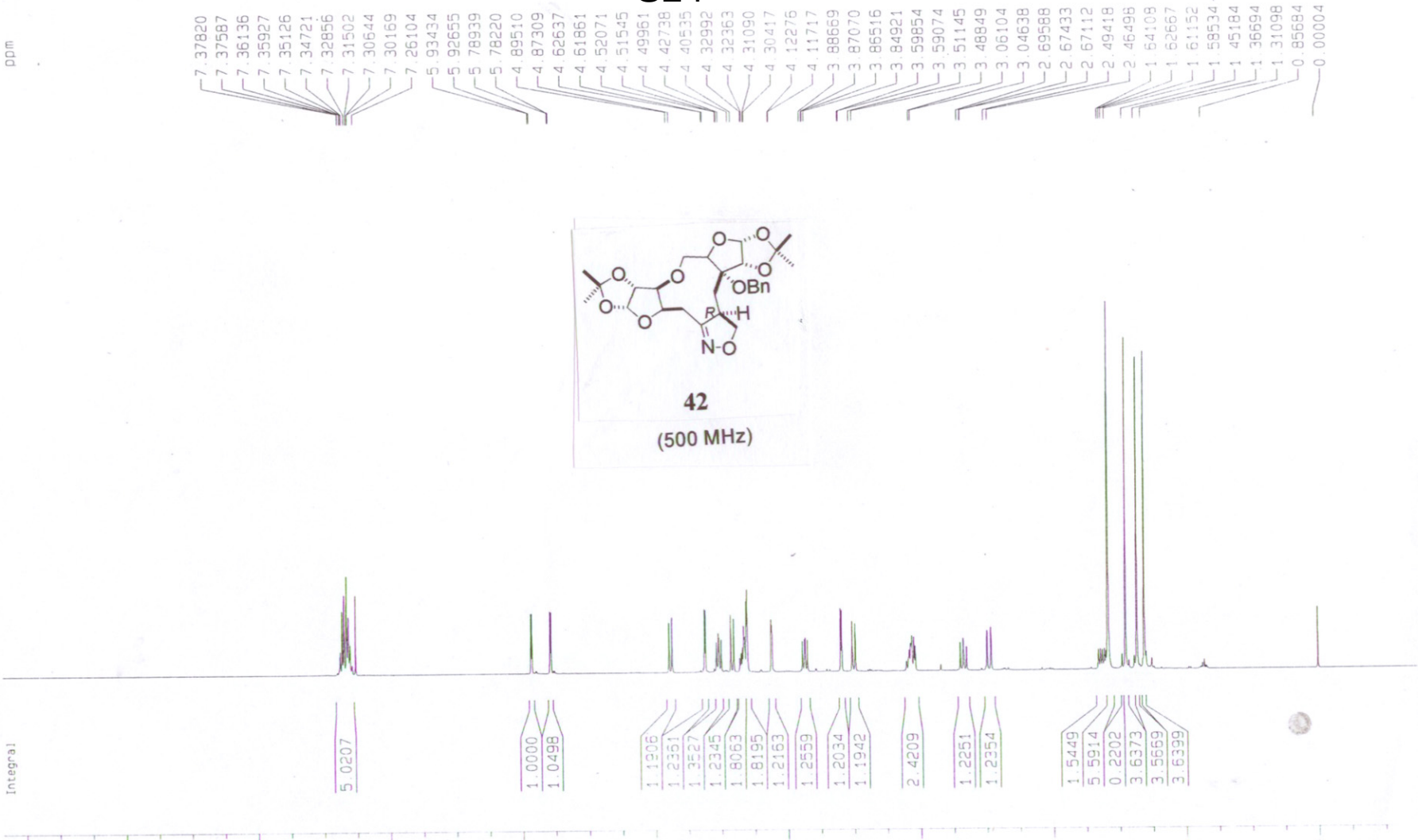

Integral
ppm
1.5896
0.8024
1.7668
1.0000
2.9219
1.1991
2.0208
1.1086
1.0466
0.9902
5.9187
5.6249
6.7163
39
ppm
7.2616
6.0372
6.0253
5.8981
5.8854
5.2997
5.2481
5.2355
4.7106
4.6983
4.6842
4.5102
4.4976
4.1926
4.1791
4.1450
4.1327
3.9881
3.9671
3.9543
3.9336
3.7796
3.7723
3.7463
3.7078
3.6221
3.6162
3.5808
3.3624
3.3536
3.3058
3.2977
3.1869
3.1513
3.1316
1.6616
1.5652
1.5007
1.4592
1.3381
1.3158
1.2560
0.0000

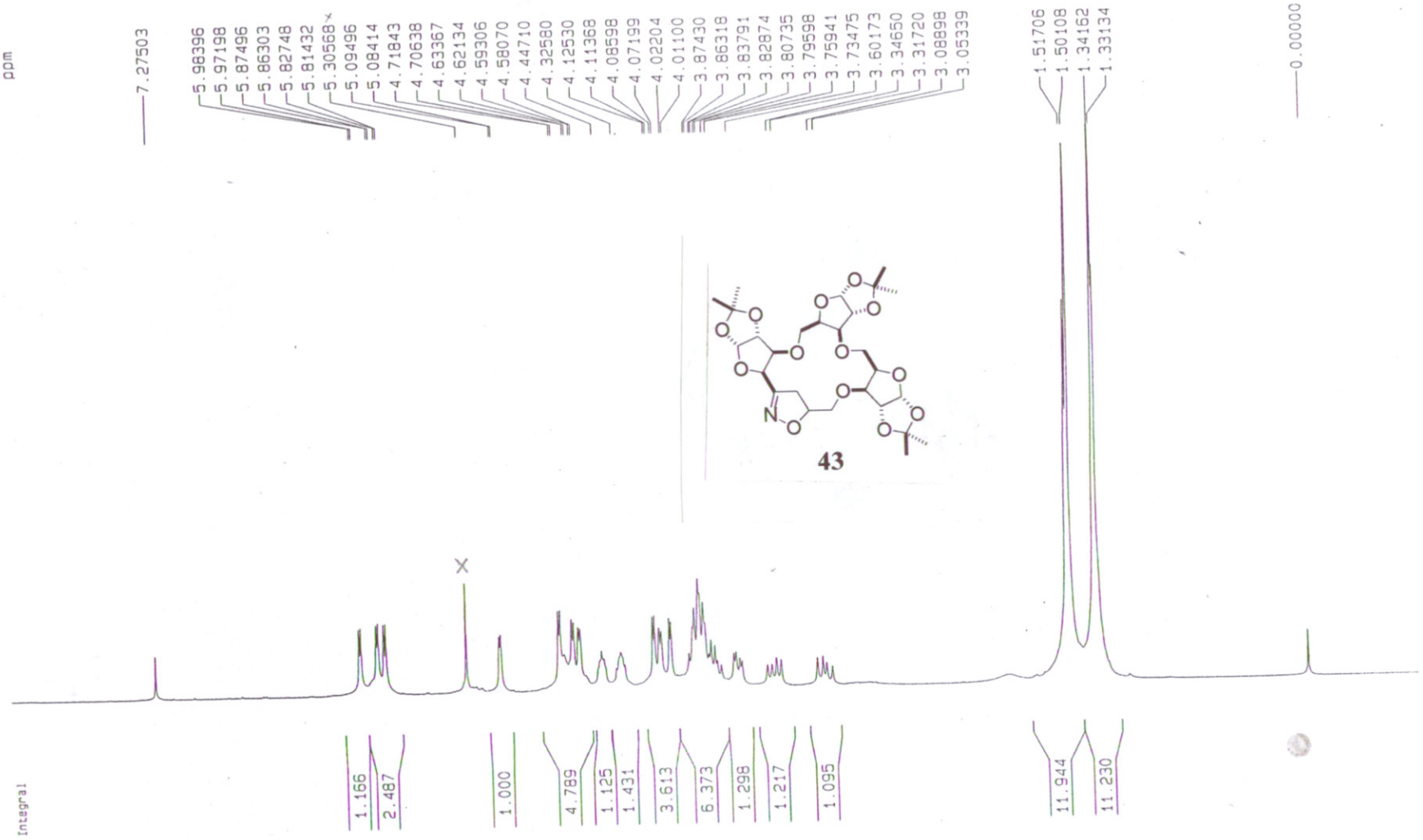

Integral
ppm
1.7650
1.3506
1.4304
0.9864
1.9459
3.0573
1.0403
1.0000
0.9829
1.0147
0.9954
0.9850
5.7147
6.0285
6.6011
X
40
ppm
7.26294
5.93794
5.92684
5.91855
5.30215
4.69586
4.68277
4.62171
4.60859
4.59646
4.58443
4.53896
4.52633
4.26510
4.25330
4.23372
4.22435
4.21484
3.94516
3.93448
3.92131
3.90261
3.88160
3.87212
3.78818
3.74652
3.56649
3.52475
3.24718
3.23232
2.87077
2.85847
2.71939
2.69536
1.67434
1.57488
1.48772
1.47074
1.31258
1.25413
0.00000

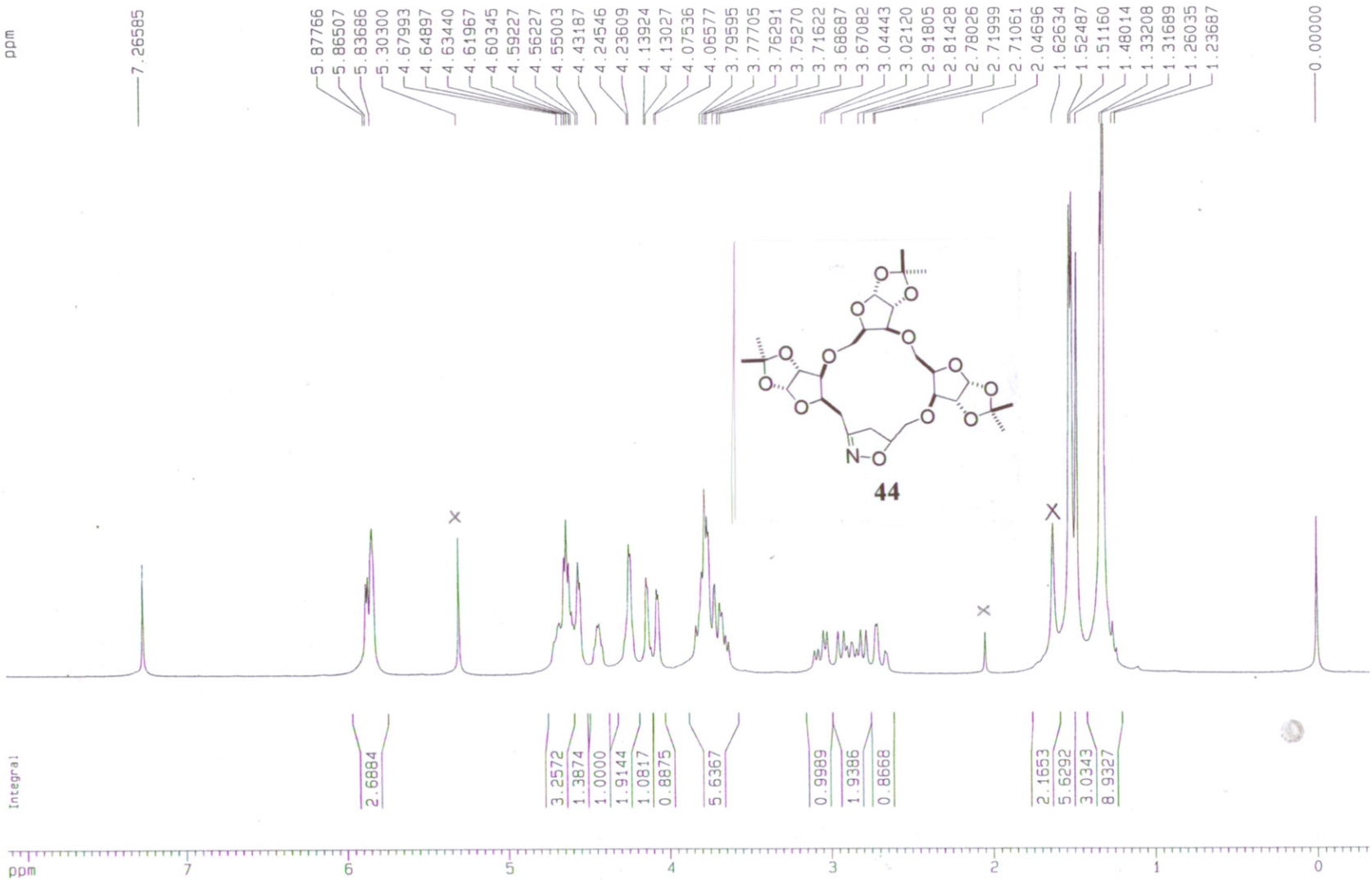
ppm
7.2654
7.2459
5.7982
5.7876
5.7110
5.7010
5.3032
5.2837
4.7924
4.7574
4.7195
4.6517
4.1719
4.1635
4.0316
4.0144
3.9726
3.9259
3.7801
3.7337
3.5370
3.5090
3.4817
3.4536
3.4188
3.4030
3.3905
3.3776
3.2400
2.8859
2.8429
2.8345
2.8100
2.7949
2.6674
2.6285
2.5873
1.6560
1.5857
1.5589
1.3681
1.3486
1.2588
X
X
Integral
1.7029
2.8410
2.7854
2.0659
1.0879
1.8586
1.0000
1.8461
0.9885
1.6187
6.0158
6.2388
ppm
7
6
5
4
3
2
1

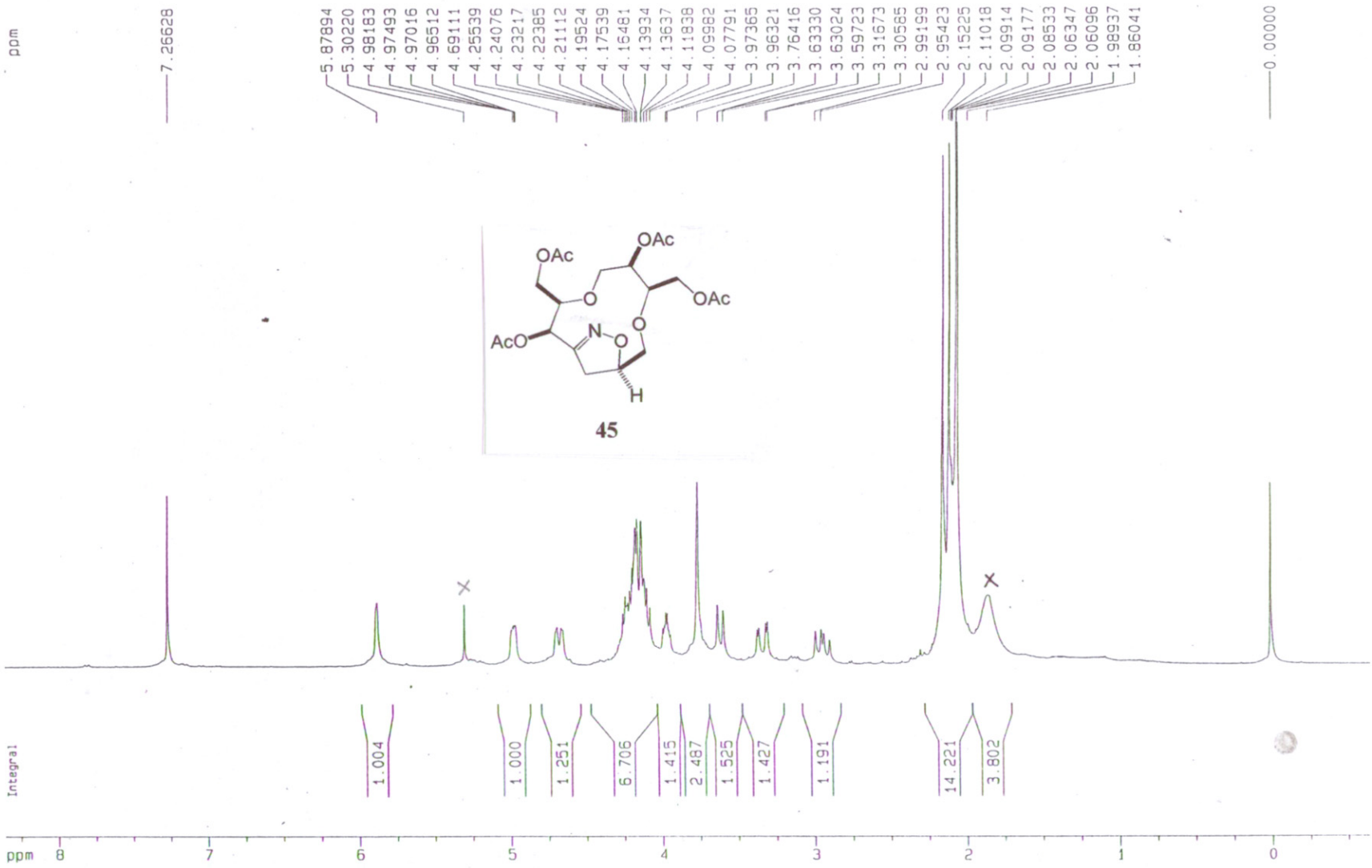
O
O
O
O
O
O
O
O
O
S
N
O
H

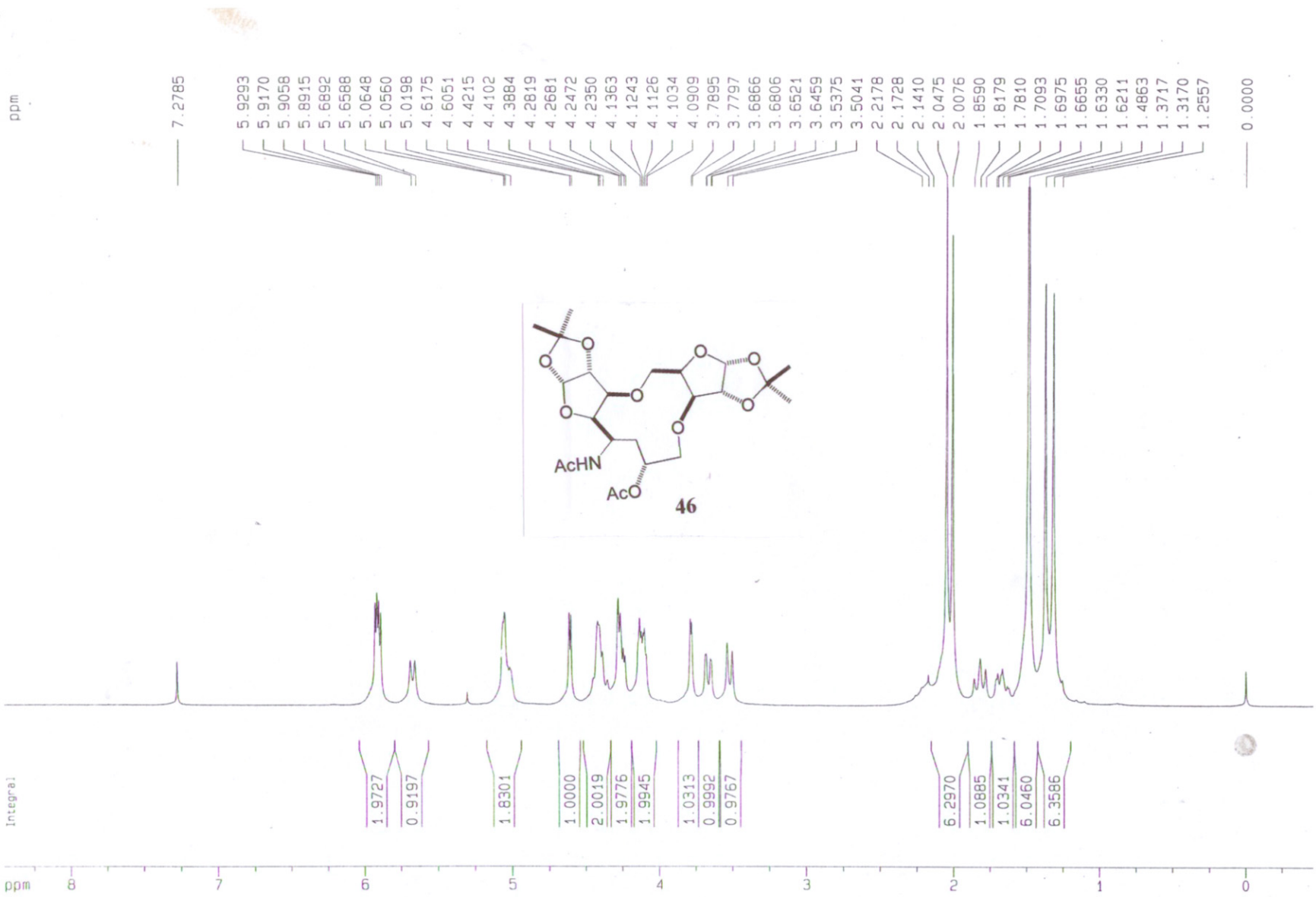
41

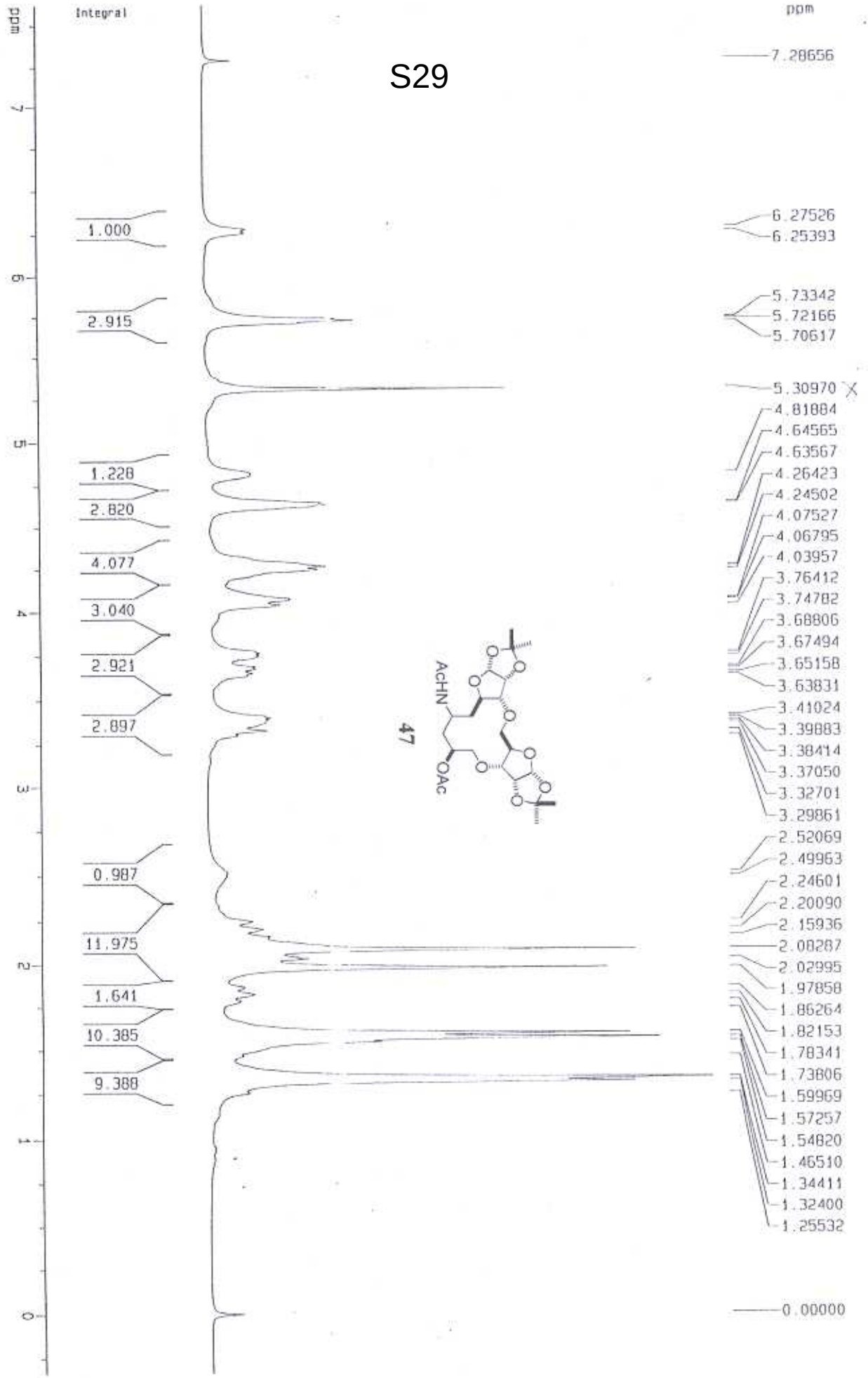

ppm
7.37820
7.37587
7.36136
7.35927
7.35126
7.34721
7.32856
7.31502
7.30644
7.30169
7.26104
5.93434
5.92655
5.78939
5.78220
4.89510
4.87309
4.62637
4.61861
4.52071
4.51545
4.49961
4.42738
4.40535
4.32992
4.32363
4.31090
4.30417
4.12276
4.11717
3.88669
3.87070
3.86516
3.84921
3.59854
3.59074
3.51145
3.48849
3.06104
3.04638
2.69588
2.67433
2.67112
2.49418
2.46498
1.64109
1.62667
1.61152
1.58534
1.45184
1.36694
1.31098
0.85684
0.00004
OBn
R
H
N-O
42
(500 MHz)
Integral
5.0207
1.0000
1.0498
1.1906
1.2361
1.3527
1.2345
1.8063
1.8195
1.2163
1.2559
1.2034
1.1942
2.4209
1.2251
1.2354
1.5449
5.5914
0.2202
3.6373
3.5669
3.6399

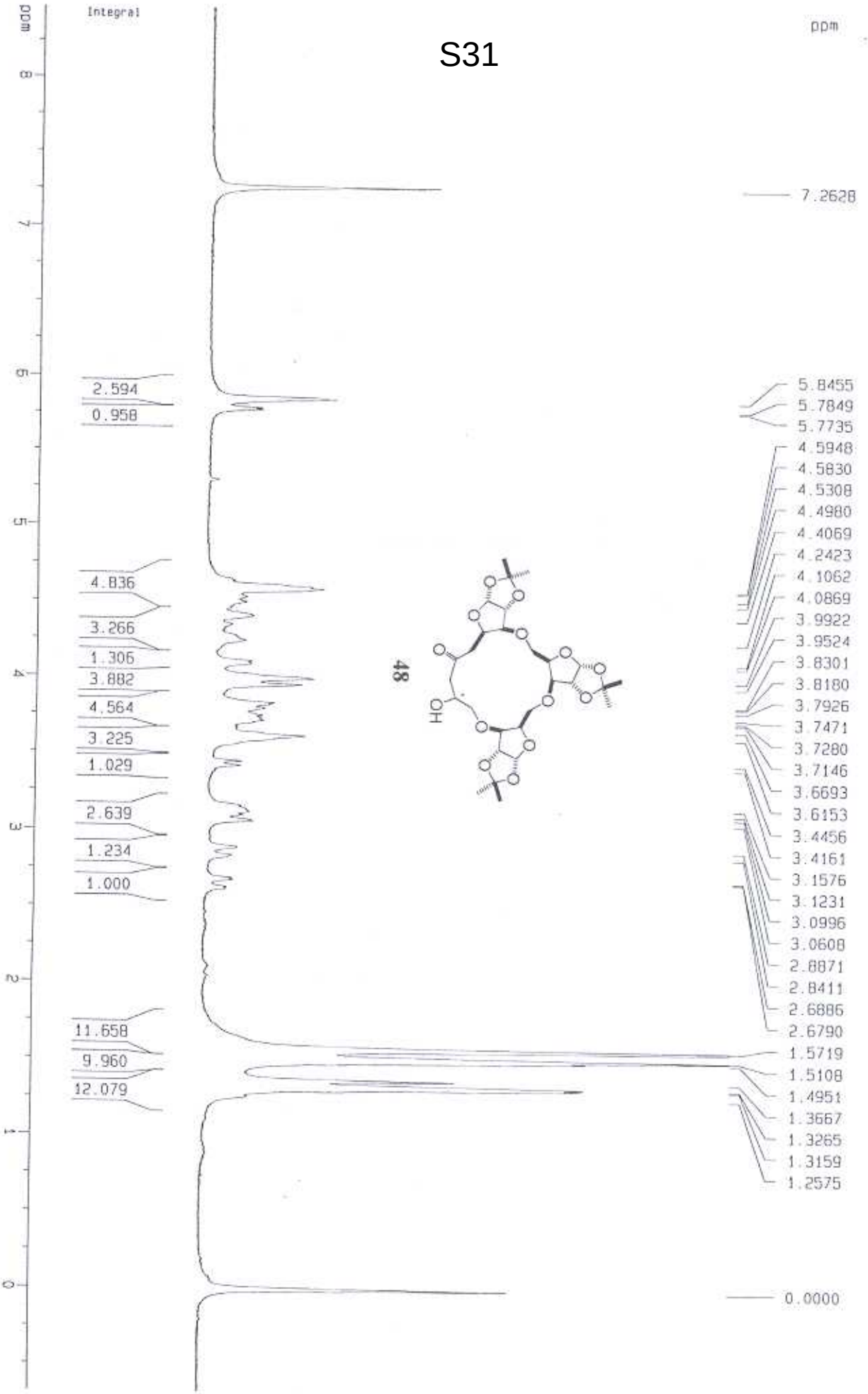

ppm
7.27503
5.98396
5.97198
5.87496
5.86303
5.82748
5.81432
5.30568
5.09496
5.08414
4.71843
4.70638
4.63367
4.62134
4.59306
4.58070
4.44710
4.32580
4.12530
4.11368
4.08598
4.07199
4.02204
4.01100
3.87430
3.86318
3.83791
3.82874
3.80735
3.79598
3.75941
3.73475
3.60173
3.34650
3.31720
3.08898
3.05339
1.51706
1.50108
1.34162
1.33134
0.00000
X
43
Integral
1.166
2.487
1.000
4.789
1.125
1.431
3.613
6.373
1.298
1.217
1.095
11.944
11.230
ppm
7
6
5
4
3
2
1
0

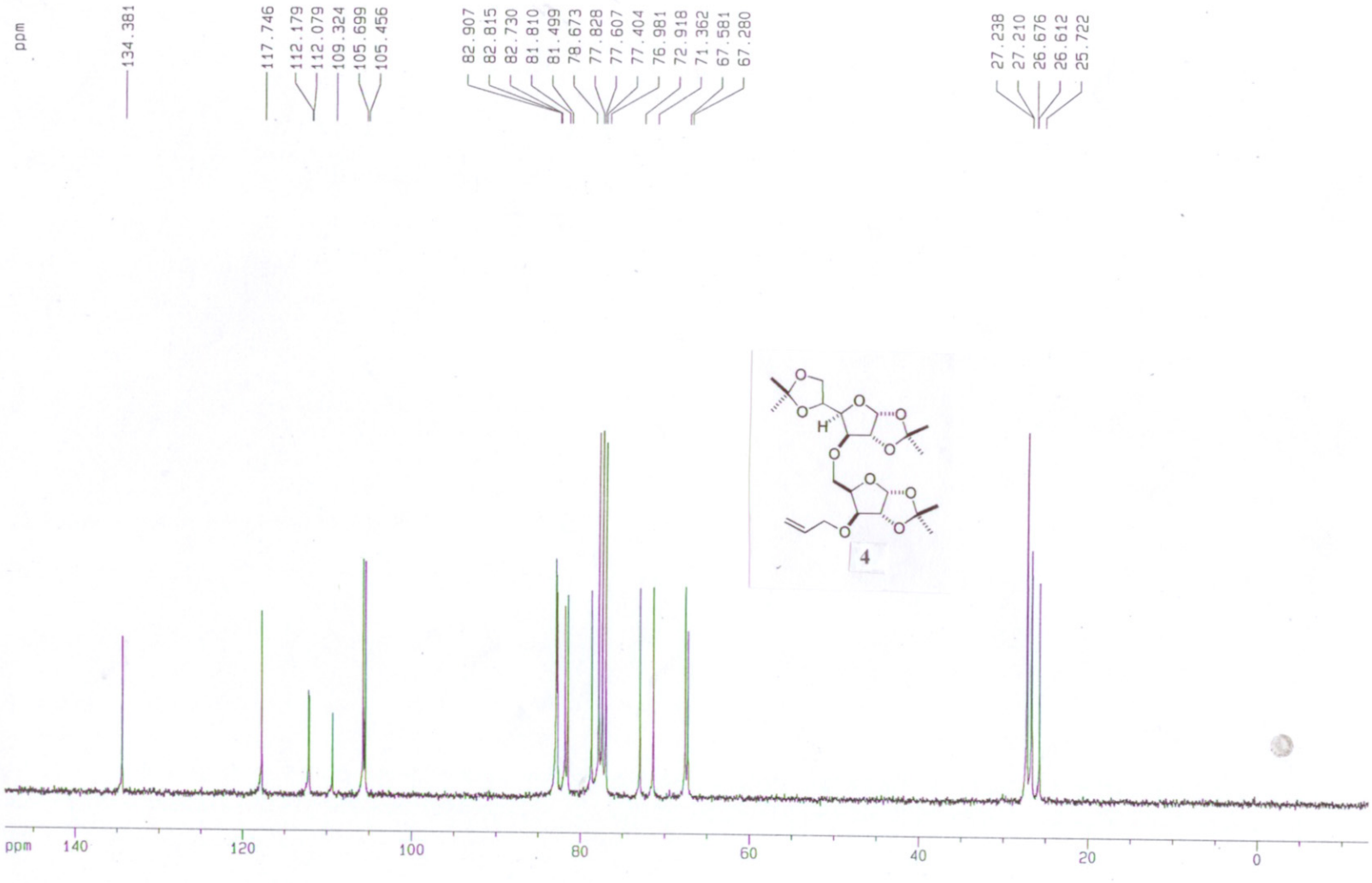

ppm
7.26585
5.87766
5.86507
5.83686
5.30300
4.67993
4.64897
4.63440
4.61967
4.60345
4.59227
4.56227
4.55003
4.43187
4.24546
4.23609
4.13924
4.13027
4.07536
4.06577
3.79595
3.77705
3.76291
3.75270
3.71622
3.68687
3.67082
3.04443
3.02120
2.91805
2.81428
2.78026
2.71999
2.71061
2.04696
1.62634
1.52487
1.51160
1.48014
1.33208
1.31689
1.26035
1.23687
0.00000
44
Integral
2.6884
3.2572
1.3874
1.0000
1.9144
1.0817
0.8875
5.6367
0.9989
1.9386
0.8668
2.1653
5.6292
3.0343
8.9327
ppm
7
6
5
4
3
2
1
0

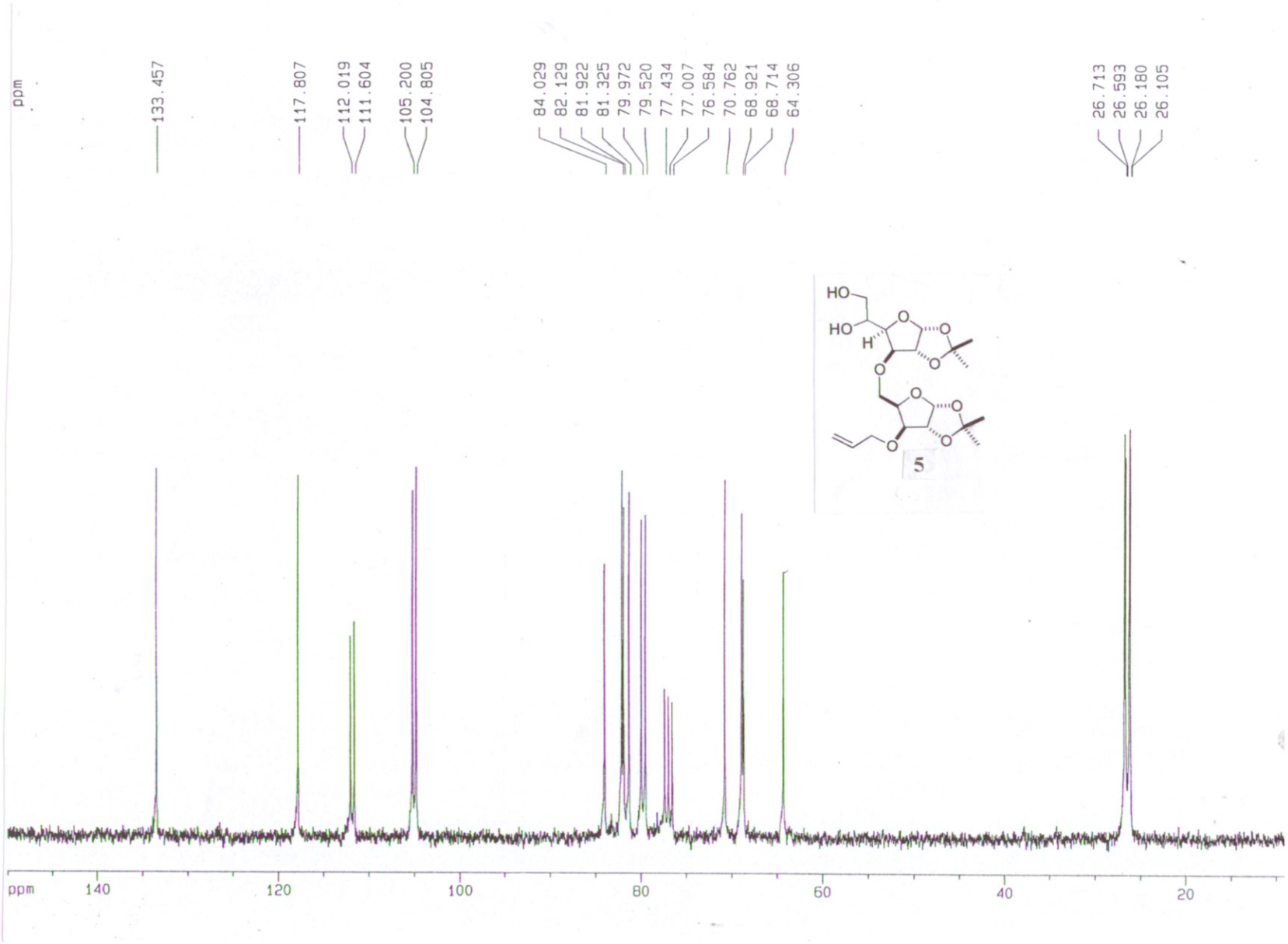

ppm
7.26628
5.87894
5.30220
4.98183
4.97493
4.97016
4.96512
4.69111
4.25539
4.24076
4.23217
4.22385
4.21112
4.19524
4.17539
4.16481
4.13934
4.13637
4.11838
4.09982
4.07791
3.97365
3.96321
3.76416
3.63330
3.63024
3.59723
3.31673
3.30565
2.99199
2.95423
2.15225
2.11018
2.09914
2.09177
2.08533
2.06347
2.06096
1.98937
1.86041
0.00000
OAc
OAc
OAc
AcO
O
N
O
O
H
45
Integral
1.004
1.000
1.251
6.706
1.415
2.487
1.525
1.427
1.191
14.221
3.802
ppm
8
7
6
5
4
3
2
1
0

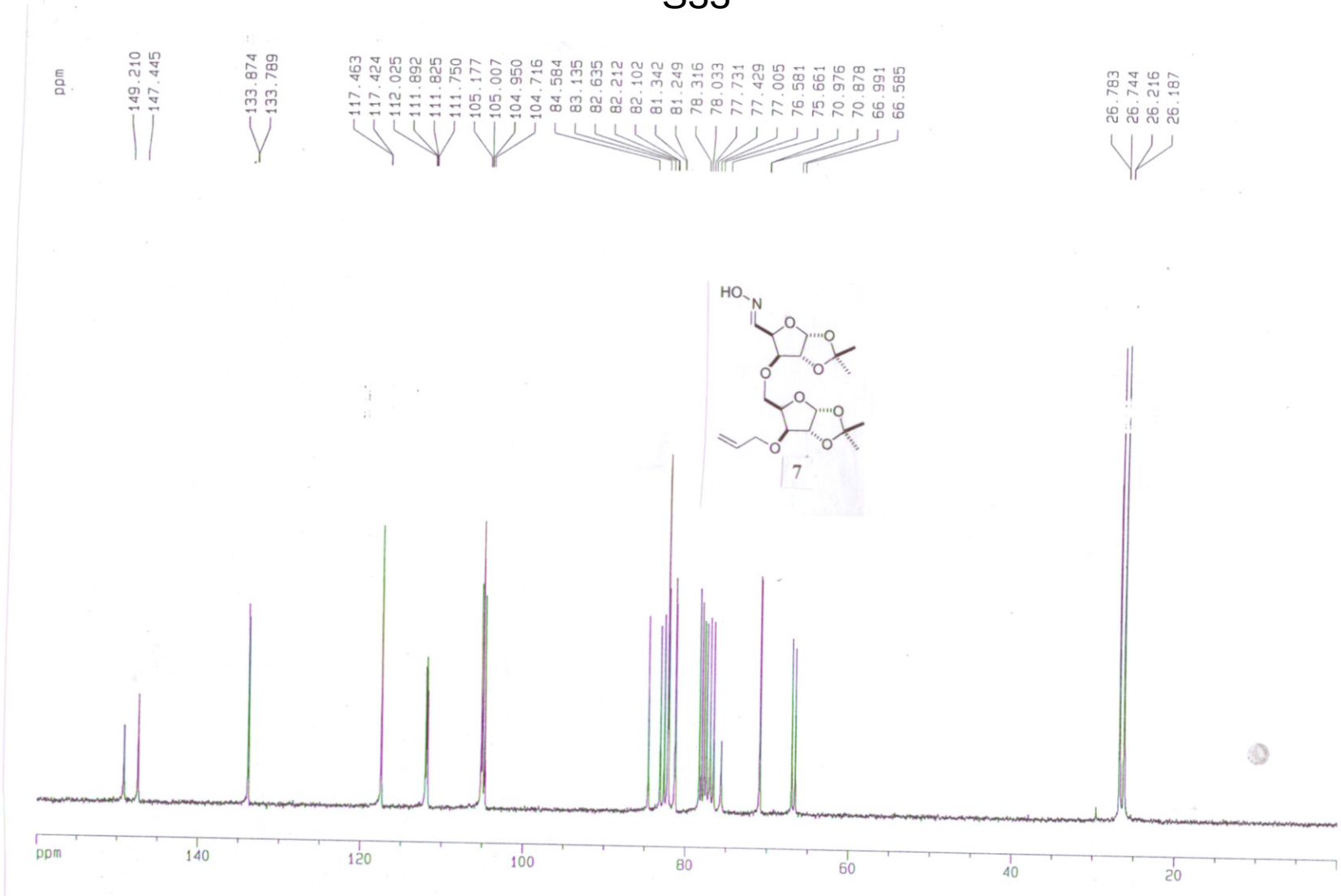

ppm
7.2785
5.9293
5.9170
5.9058
5.8915
5.6892
5.6588
5.0648
5.0560
5.0198
4.6175
4.6051
4.4215
4.4102
4.3884
4.2819
4.2681
4.2472
4.2350
4.1363
4.1243
4.1126
4.1034
4.0909
3.7895
3.7797
3.6866
3.6806
3.6521
3.6459
3.5375
3.5041
2.2178
2.1728
2.1410
2.0475
2.0076
1.8590
1.8179
1.7810
1.7093
1.6975
1.6655
1.6330
1.6211
1.4863
1.3717
1.3170
1.2557
0.0000
AcHN
AcO
46
Integral
1.9727
0.9197
1.8301
1.0000
2.0019
1.9776
1.9945
1.0313
0.9992
0.9767
6.2970
1.0885
1.0341
6.0460
6.3586
ppm
8
7
6
5
4
3
2
1
0

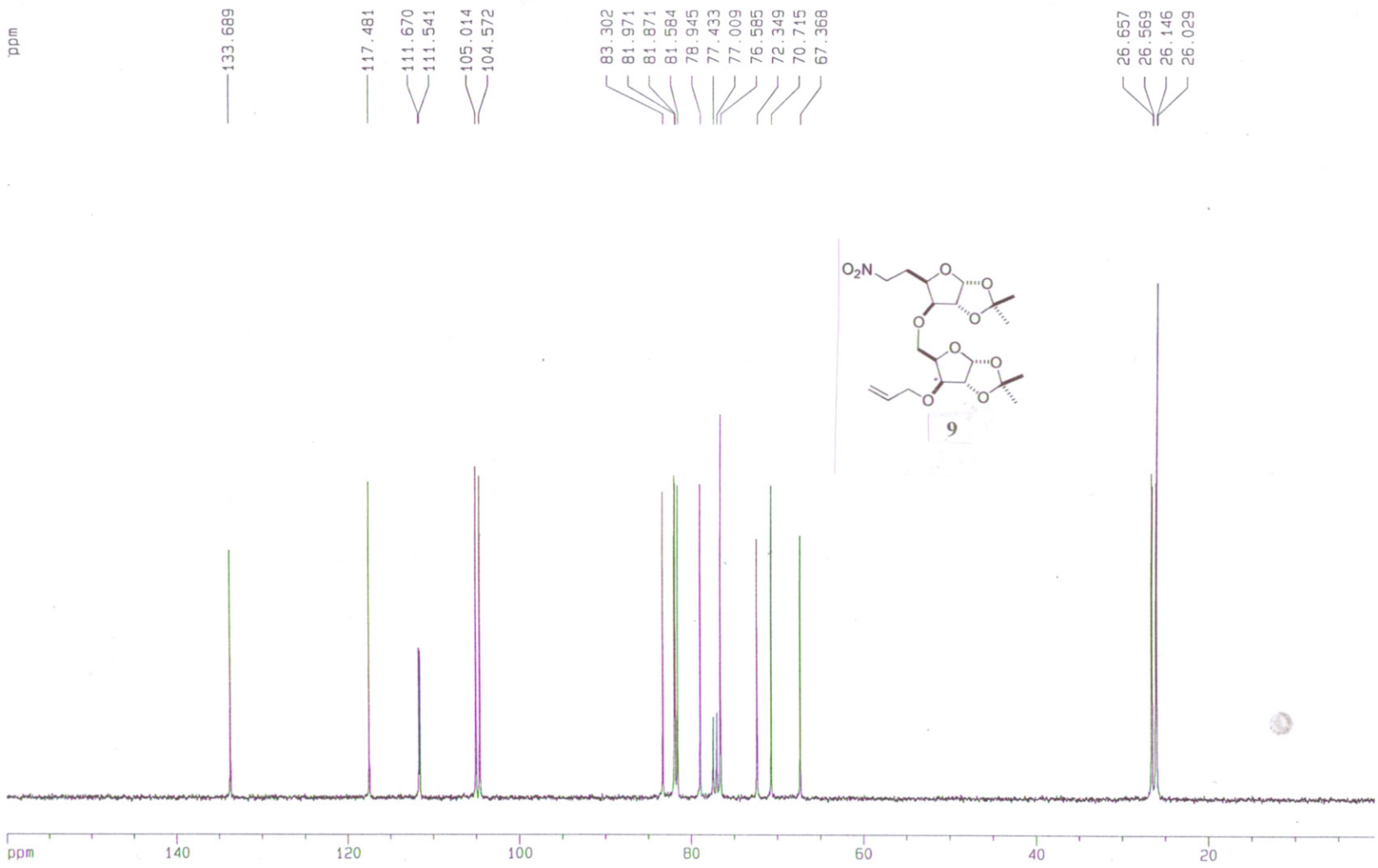

ppm
Integral
7.28656
6.27526
6.25393
5.73342
5.72166
5.70617
5.30970
4.81884
4.64565
4.63567
4.26423
4.24502
4.07527
4.06795
4.03957
3.76412
3.74782
3.68806
3.67494
3.65158
3.63831
3.41024
3.39883
3.38414
3.37050
3.32701
3.29861
2.52069
2.49963
2.24601
2.20090
2.15936
2.08287
2.02995
1.97858
1.86264
1.82153
1.78341
1.73806
1.59969
1.57257
1.54820
1.46510
1.34411
1.32400
1.25532
0.00000
1.000
2.915
1.228
2.820
4.077
3.040
2.921
2.897
0.987
11.975
1.641
10.385
9.388
AcHN
OAc
47

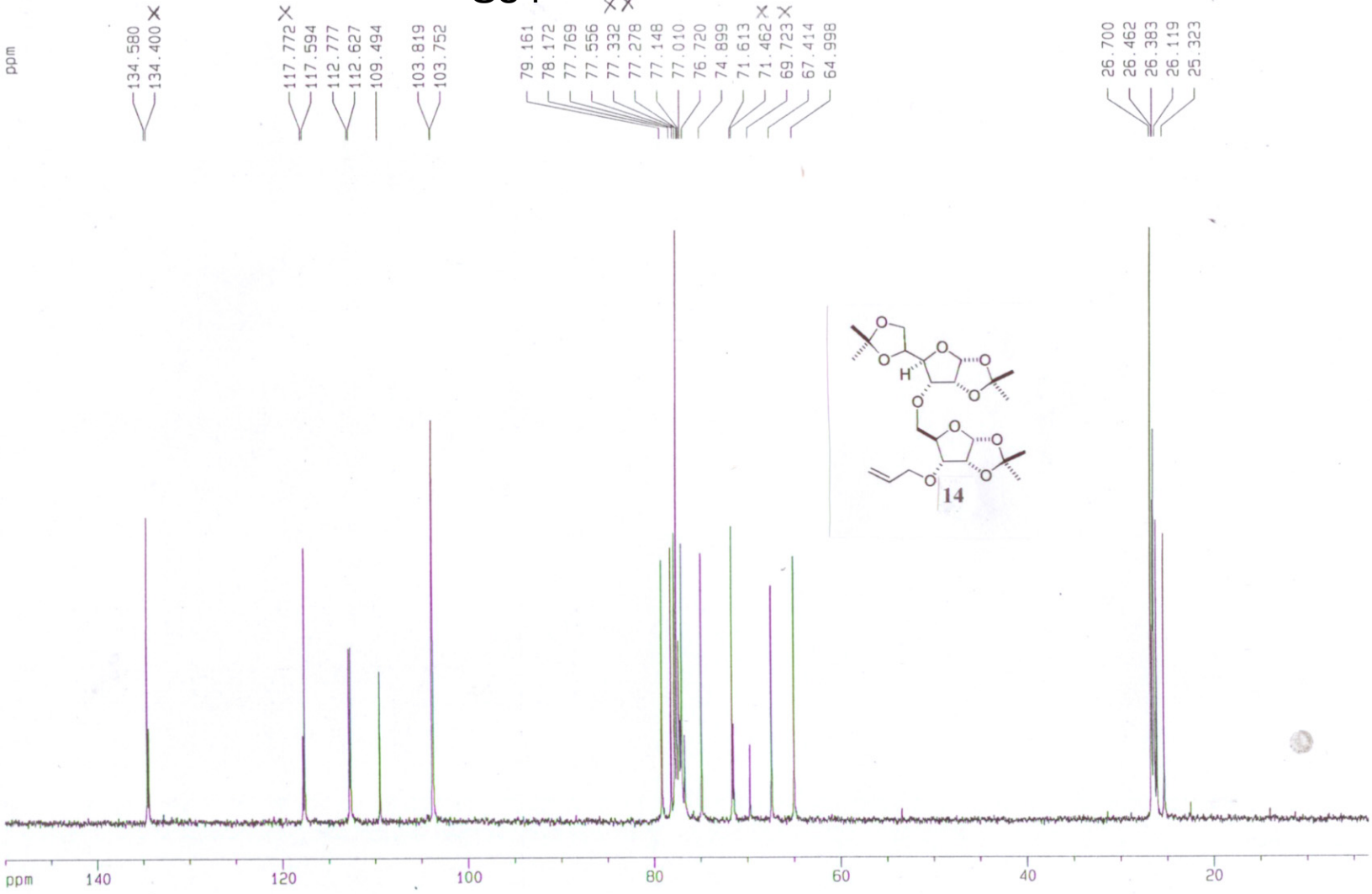

Integral
ppm
7.2628
5.8455
5.7849
5.7735
4.5948
4.5830
4.5308
4.4980
4.4069
4.2423
4.1062
4.0869
3.9922
3.9524
3.8301
3.8180
3.7926
3.7471
3.7280
3.7146
3.6693
3.6153
3.4456
3.4161
3.1576
3.1231
3.0996
3.0608
2.8871
2.8411
2.6886
2.6790
1.5719
1.5108
1.4951
1.3667
1.3265
1.3159
1.2575
0.0000
2.594
0.958
4.836
3.266
1.306
3.882
4.564
3.225
1.029
2.639
1.234
1.000
11.658
9.960
12.079
48
OH

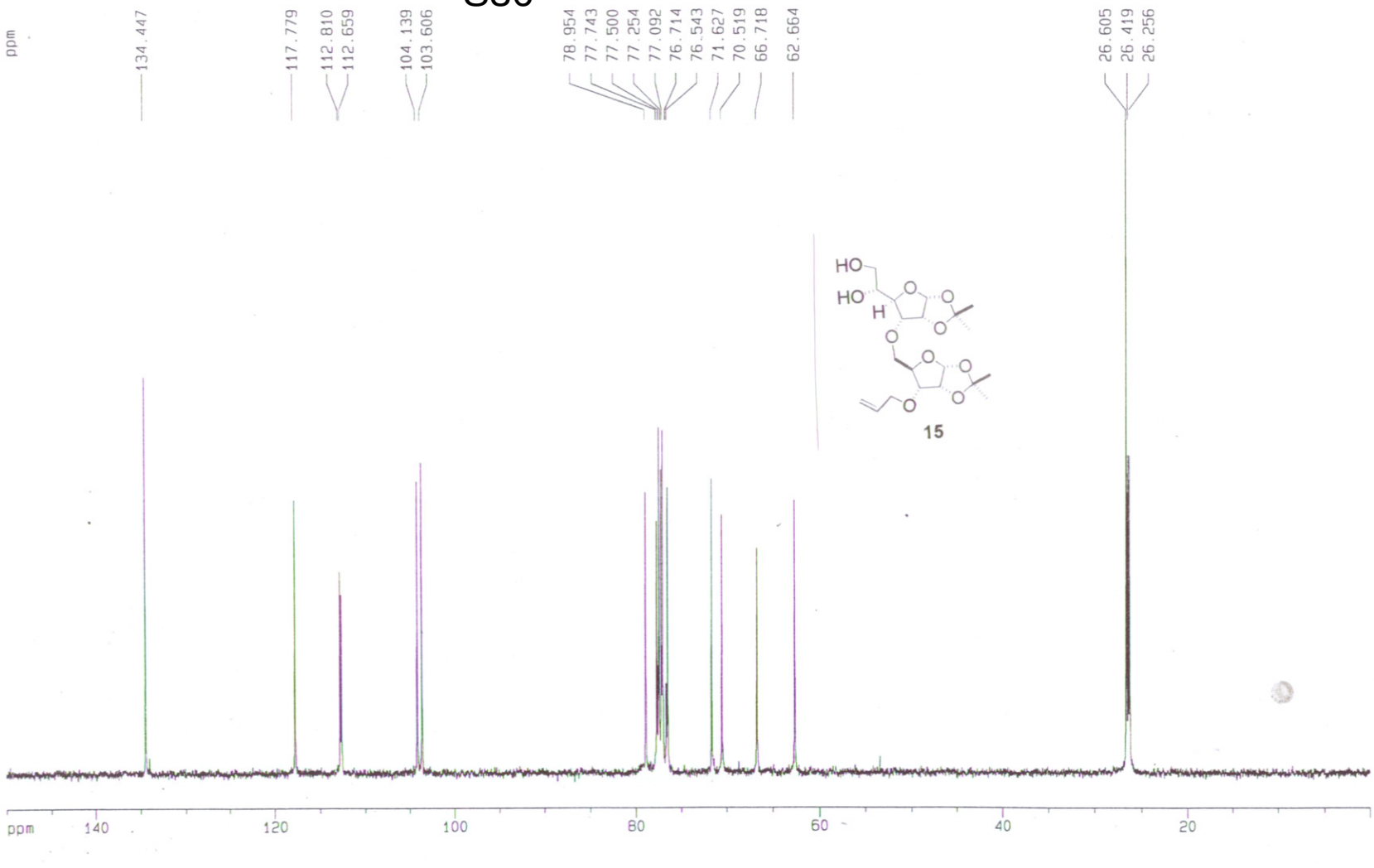

ppm
134.381
117.746
112.179
112.079
109.324
105.699
105.456
82.907
82.815
82.730
81.810
81.499
78.673
77.828
77.607
77.404
76.981
72.918
71.362
67.581
67.280
27.238
27.210
26.676
26.612
25.722
H
4
ppm
140
120
100
80
60
40
20
0

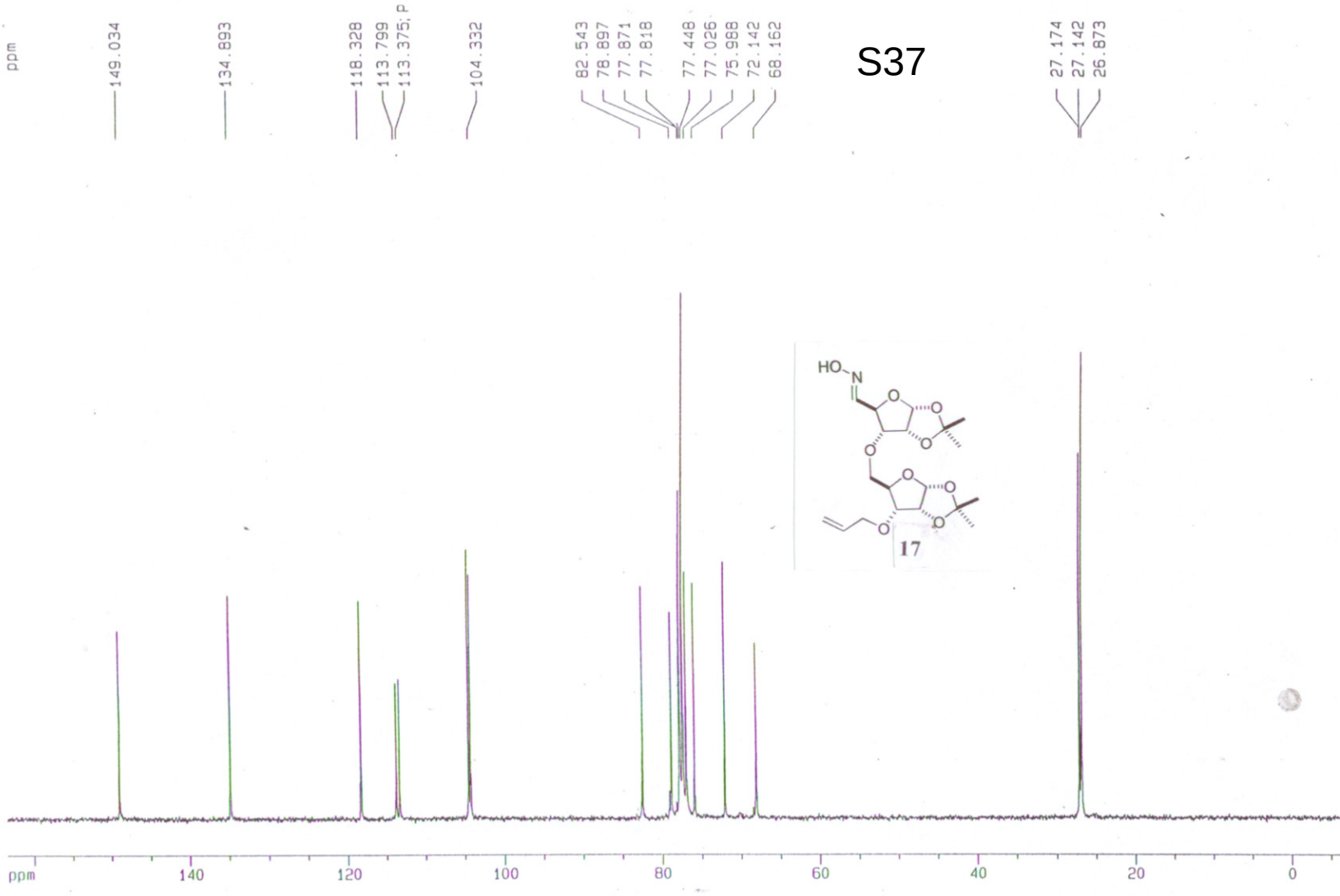

ppm
133.457
117.807
112.019
111.604
105.200
104.805
84.029
82.129
81.922
81.325
79.972
79.520
77.434
77.007
76.584
70.762
68.921
68.714
64.306
26.713
26.593
26.180
26.105
HO
HO
H
O
O
O
O
O
O
O
5
ppm
140
120
100
80
60
40
20

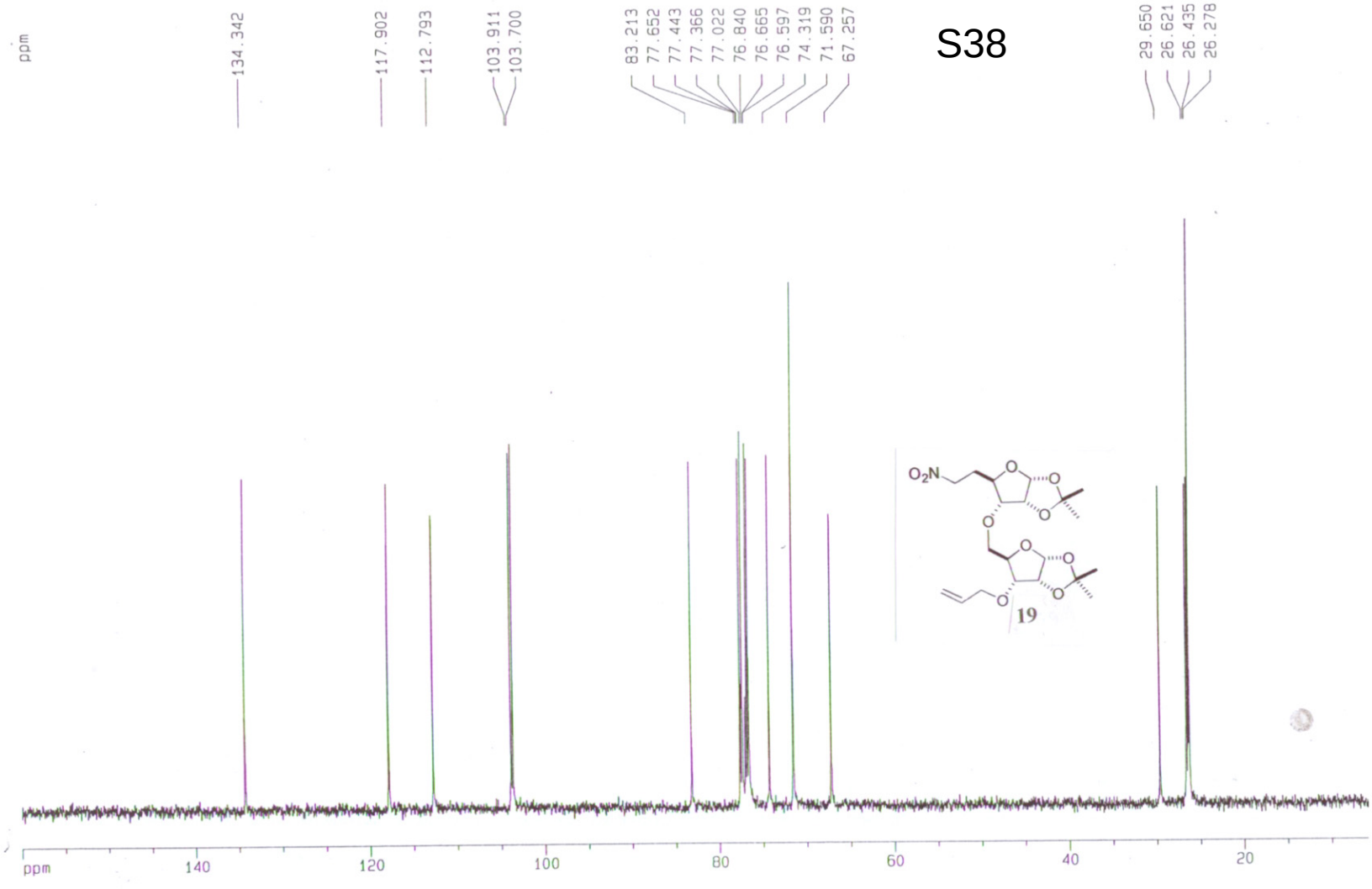

ppm
149.210
147.445
133.874
133.789
117.463
117.424
112.025
111.892
111.825
111.750
105.177
105.007
104.950
104.716
84.584
83.135
82.635
82.212
82.102
81.342
81.249
78.316
78.033
77.731
77.429
77.005
76.581
75.661
70.976
70.878
66.991
66.585
26.783
26.744
26.216
26.187
HO
N
O
7
ppm
140
120
100
80
60
40
20

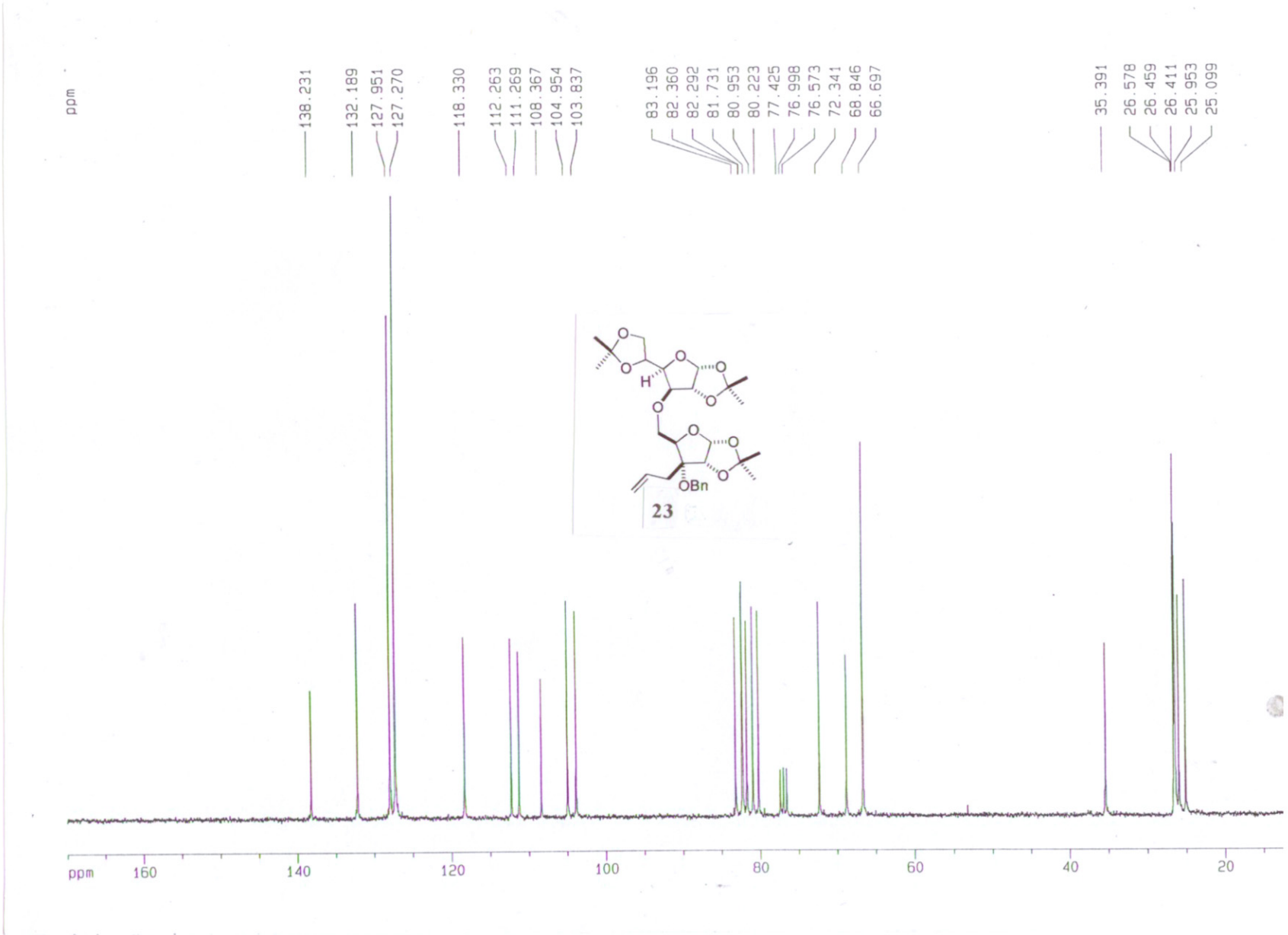

ppm
133.689
117.481
111.670
111.541
105.014
104.572
83.302
81.971
81.871
81.584
78.945
77.433
77.009
76.585
72.349
70.715
67.368
26.657
26.569
26.146
26.029
O2N
9
ppm
140
120
100
80
60
40
20

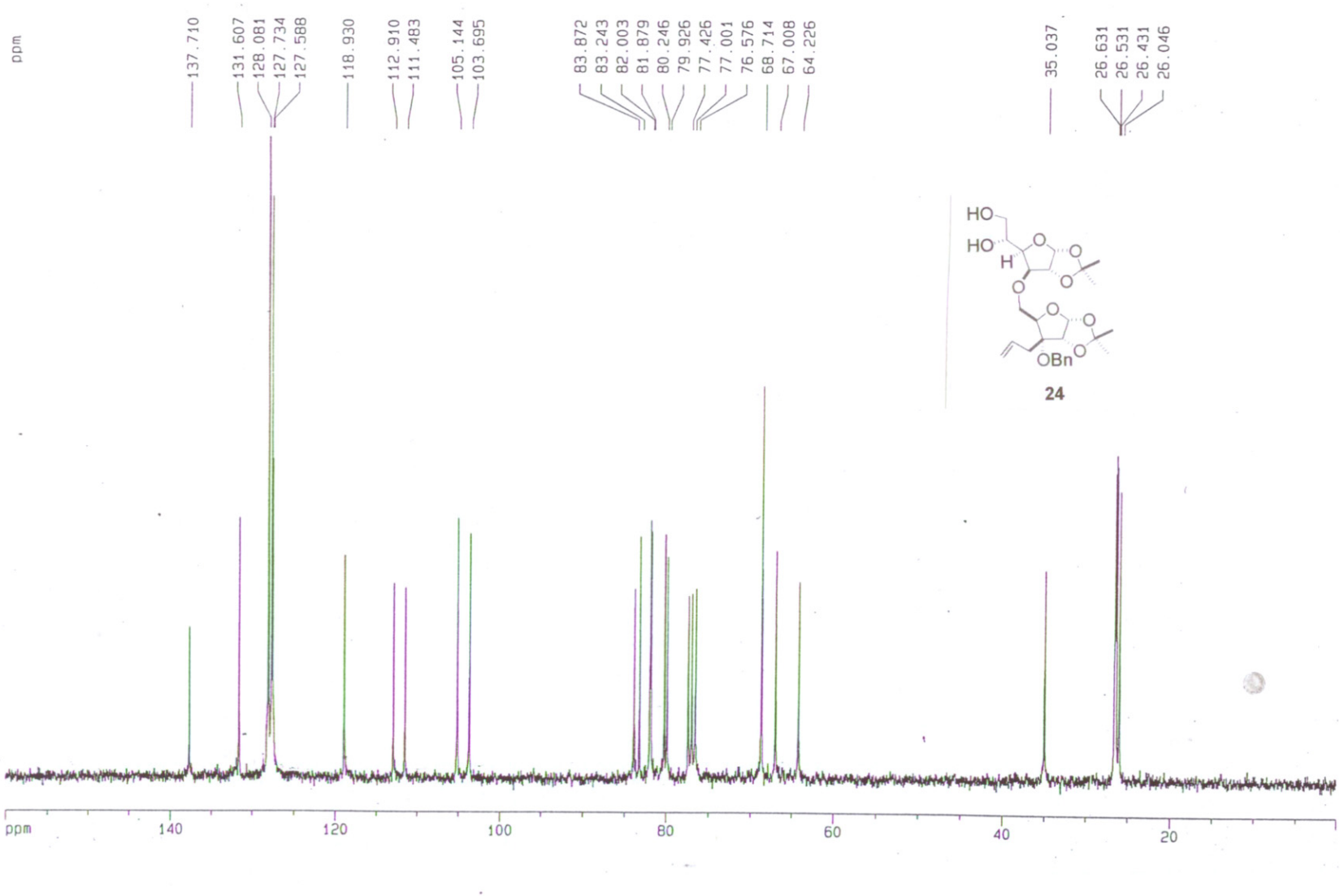

ppm
134.580
134.400
117.772
117.594
112.777
112.627
109.494
103.819
103.752
79.161
78.172
77.769
77.556
77.332
77.278
77.148
77.010
76.720
74.899
71.613
71.462
69.723
67.414
64.998
26.700
26.462
26.383
26.119
25.323
14
ppm
140
120
100
80
60
40
20

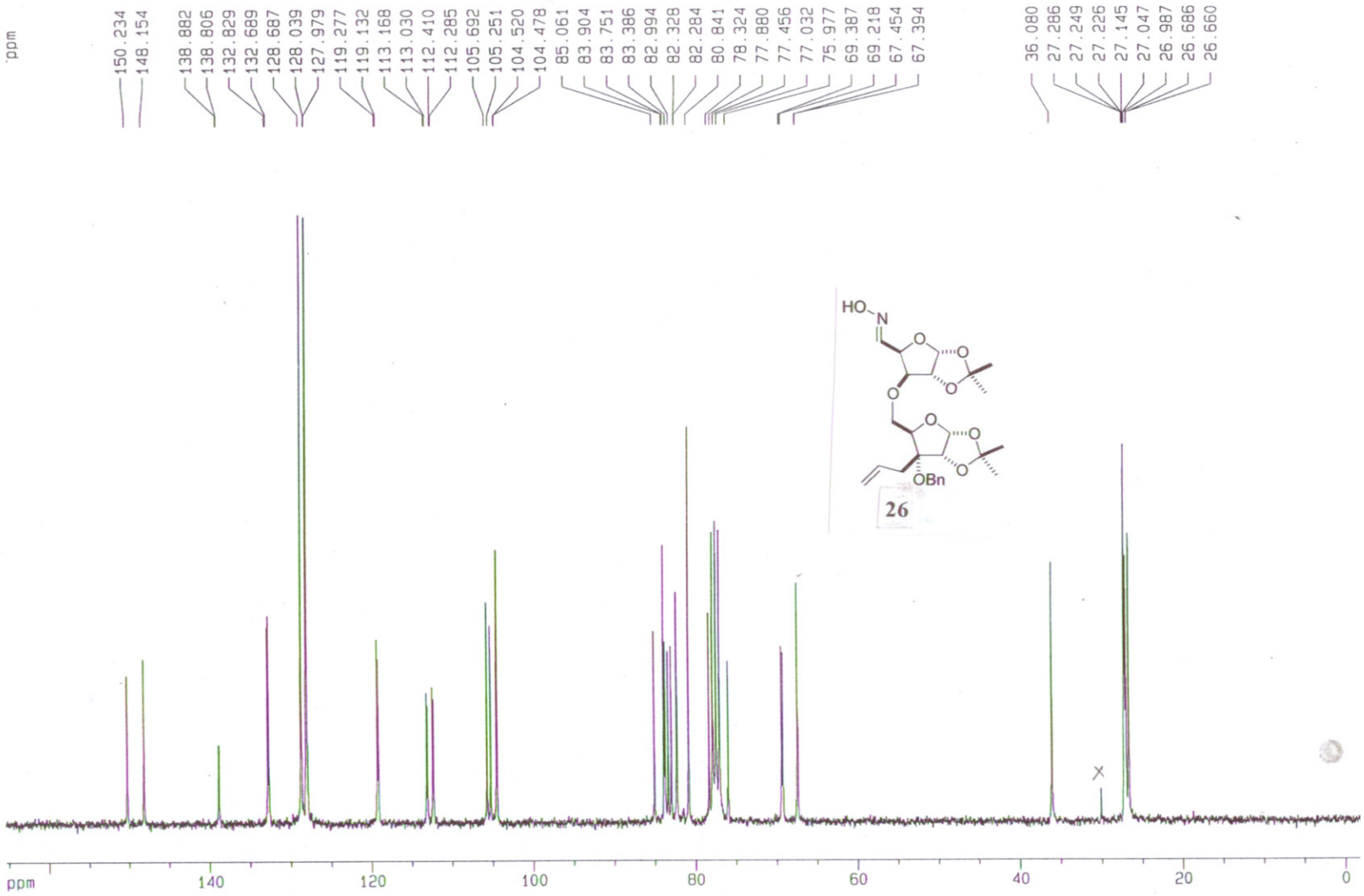

ppm
134.447
117.779
112.810
112.659
104.139
103.606
78.954
77.743
77.500
77.254
77.092
76.714
76.543
71.627
70.519
66.718
62.664
26.605
26.419
26.256
HO
HO
H
O
O
O
O
O
O
O
15
ppm
140
120
100
80
60
40
20

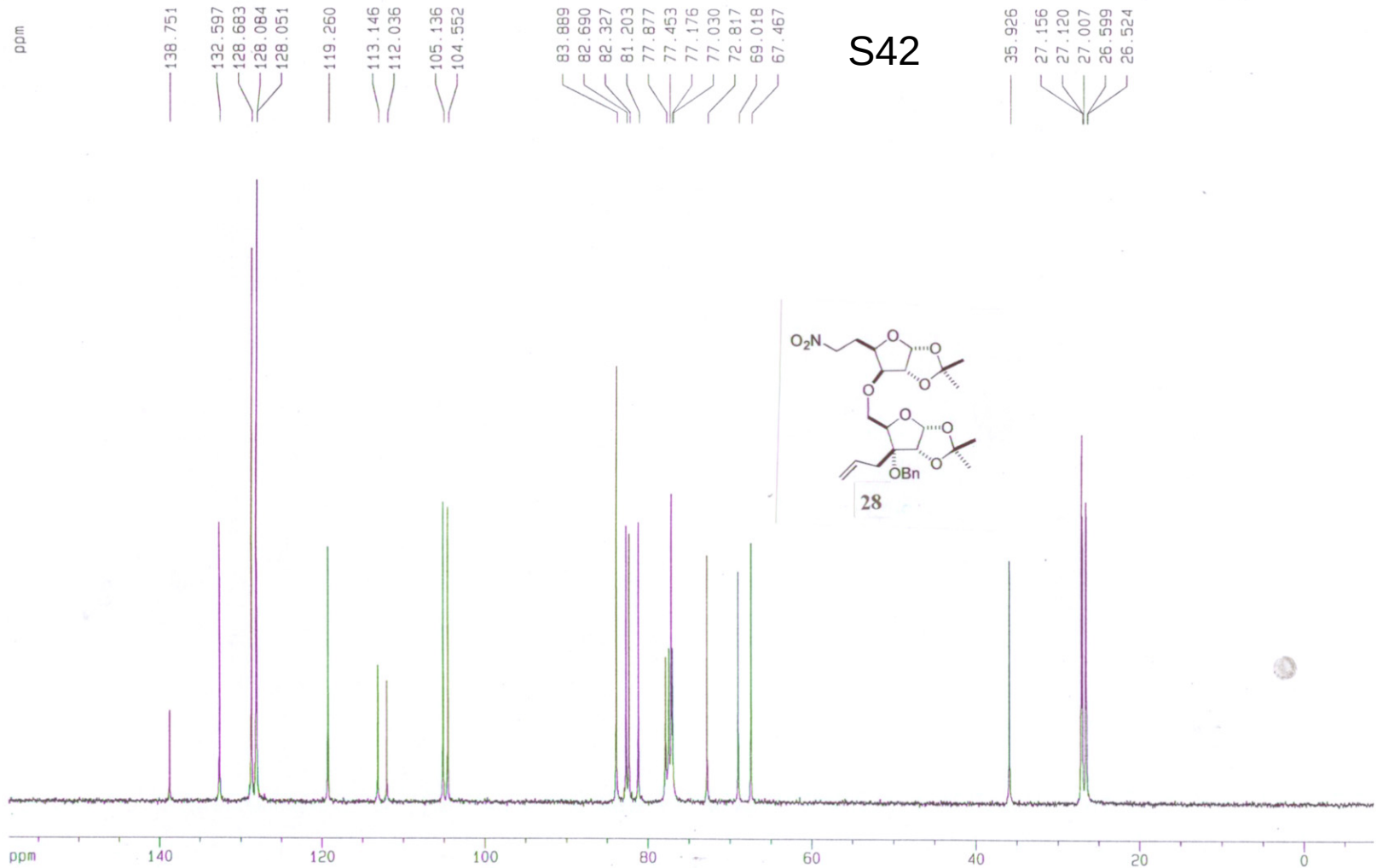

ppm
149.034
134.893
118.328
113.799
113.375
104.332
82.543
78.897
77.871
77.818
77.448
77.026
75.988
72.142
68.162
27.174
27.142
26.873
HO
N
O
17
ppm
140
120
100
80
60
40
20
0

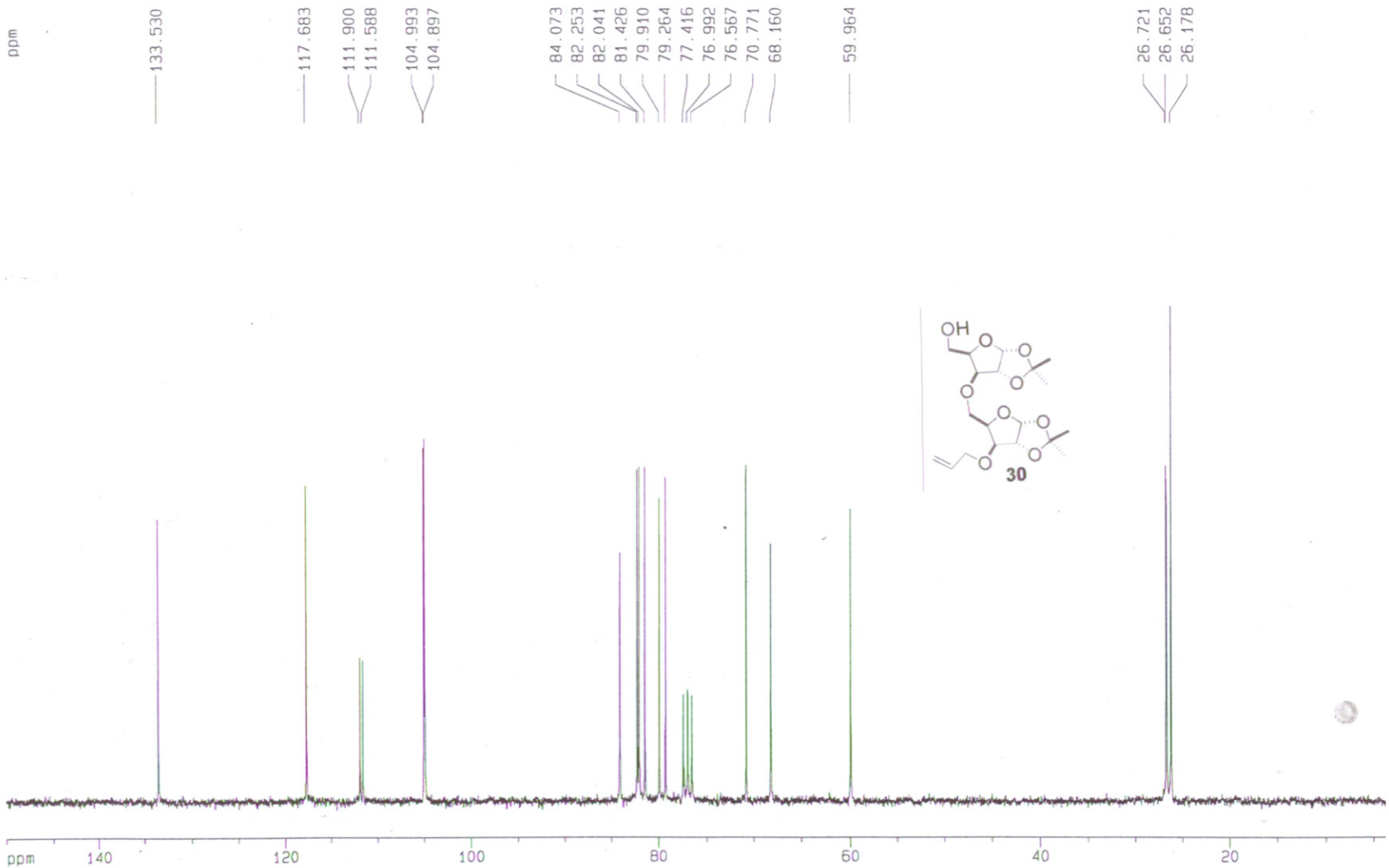

ppm
134.342
117.902
112.793
103.911
103.700
83.213
77.652
77.443
77.366
77.022
76.840
76.665
76.597
74.319
71.590
67.257
29.650
26.621
26.435
26.278
O2N
19
ppm
140
120
100
80
60
40
20

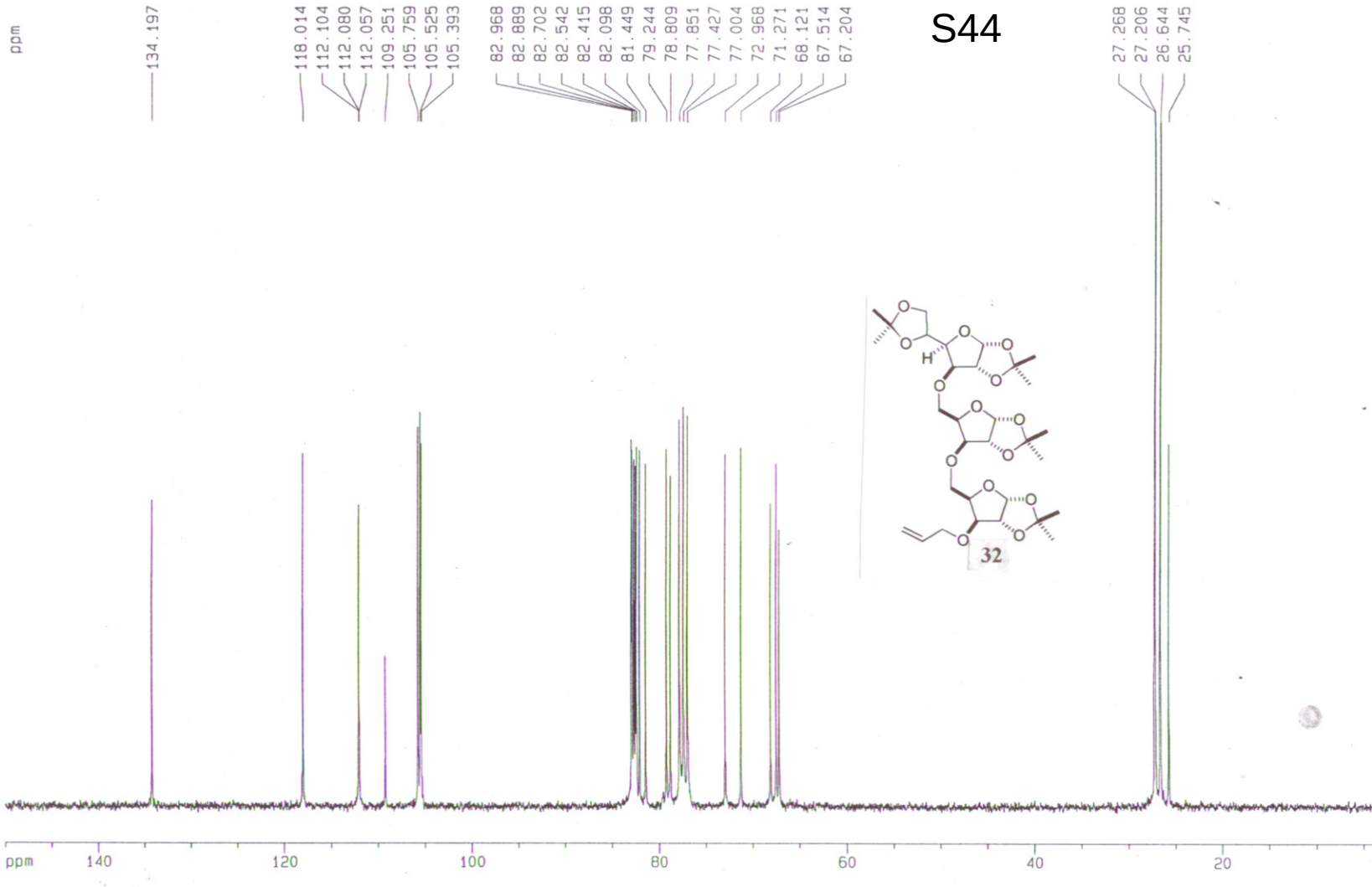

ppm
138.231
132.189
127.951
127.270
118.330
112.263
111.269
108.367
104.954
103.837
83.196
82.360
82.292
81.731
80.953
80.223
77.425
76.998
76.573
72.341
68.846
66.697
35.391
26.578
26.459
26.411
25.953
25.099
H
OBn
23
ppm
160
140
120
100
80
60
40
20

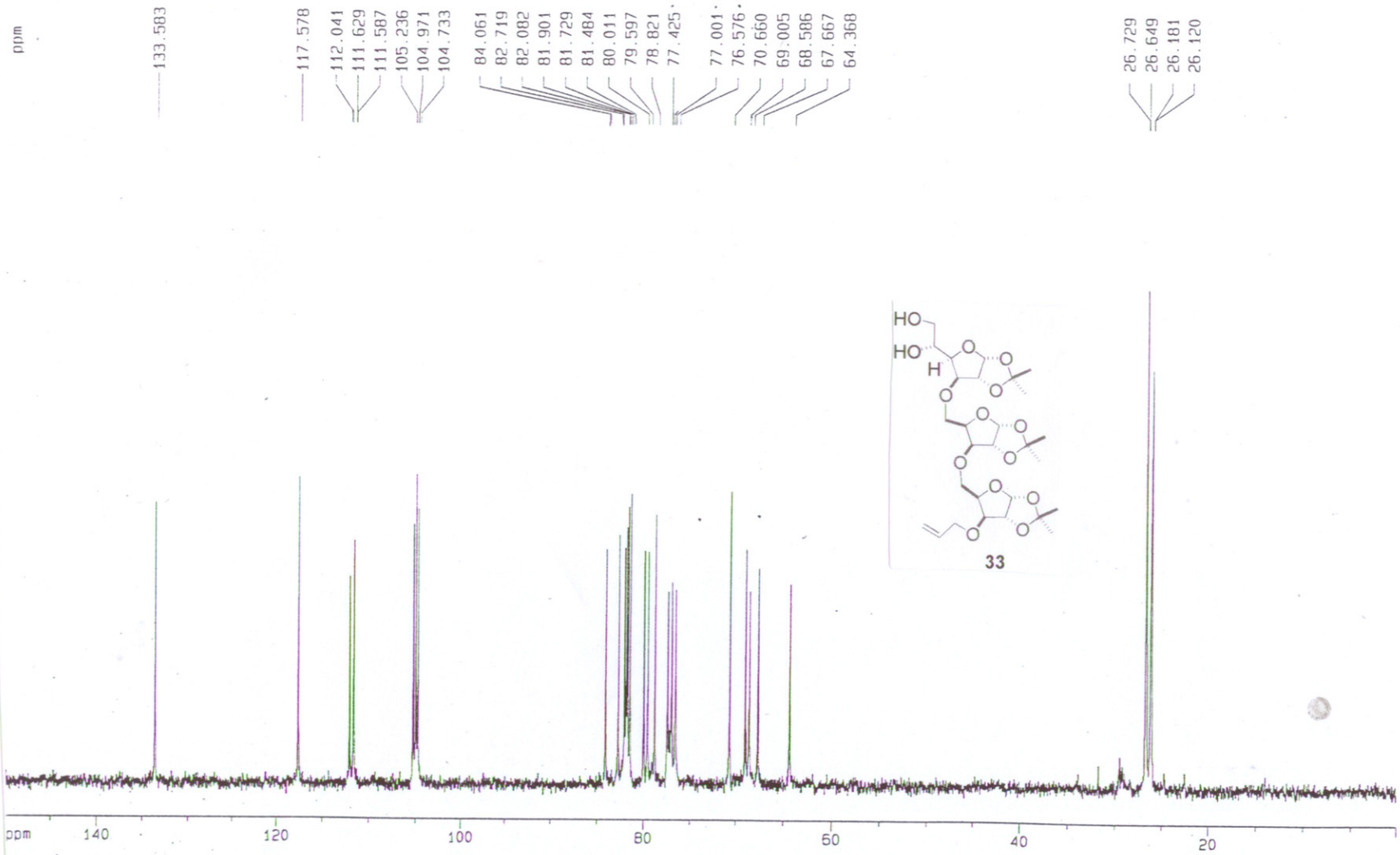

ppm
137.710
131.607
128.081
127.734
127.588
118.930
112.910
111.483
105.144
103.695
83.872
83.243
82.003
81.879
80.246
79.926
77.426
77.001
76.576
68.714
67.008
64.226
35.037
26.631
26.531
26.431
26.046
HO
HO
H
OBn
24
ppm
140
120
100
80
60
40
20

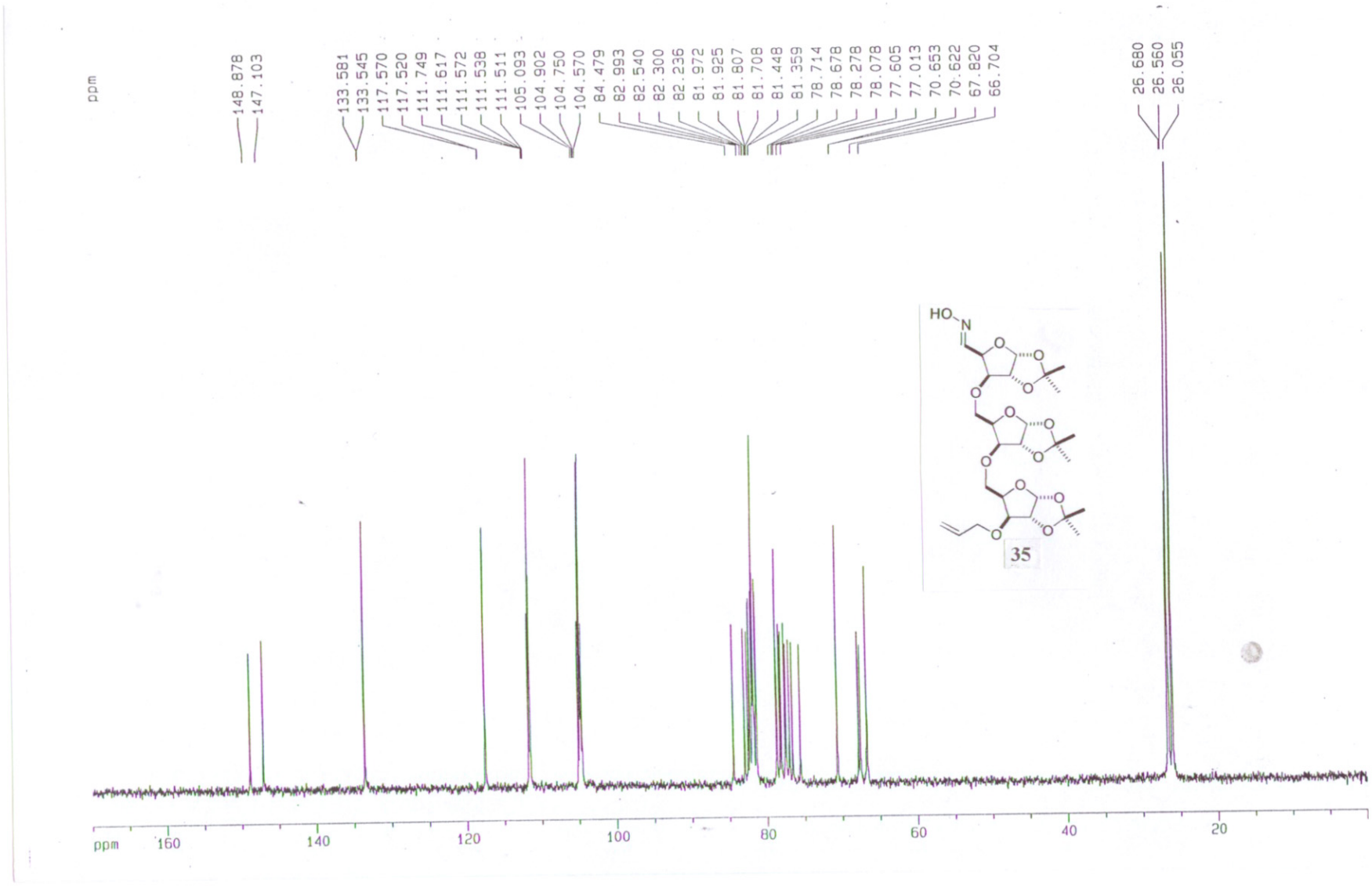

ppm
150.234
148.154
138.882
138.806
132.829
132.689
128.687
128.039
127.979
119.277
119.132
113.168
113.030
112.410
112.285
105.692
105.251
104.520
104.478
85.061
83.904
83.751
83.386
82.994
82.328
82.284
80.841
78.324
77.880
77.456
77.032
75.977
69.387
69.218
67.454
67.394
36.080
27.286
27.249
27.226
27.145
27.047
26.987
26.686
26.660
HO
N
O
OBn
26
X
ppm
140
120
100
80
60
40
20
0

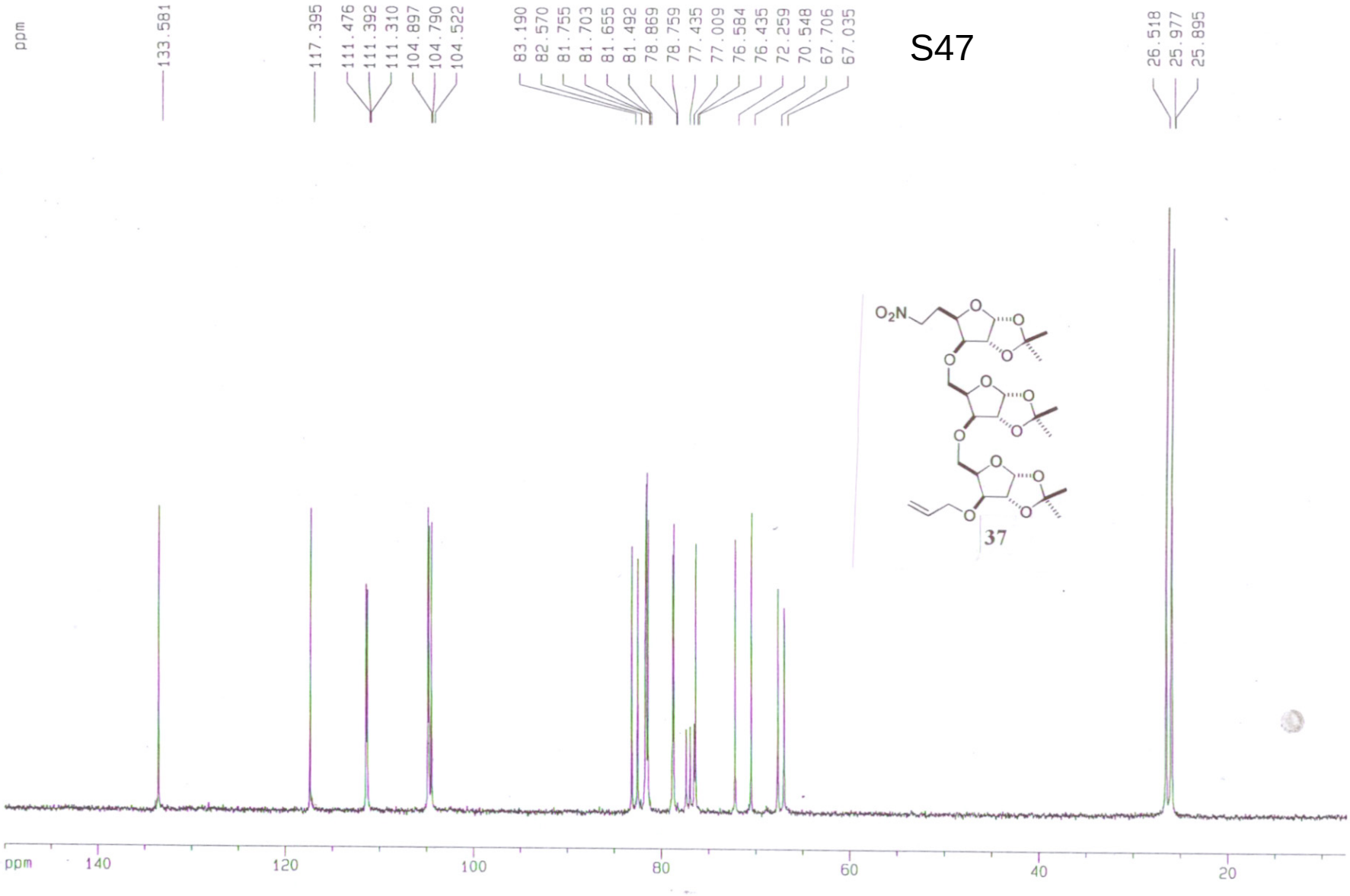

ppm
138.751
132.597
128.683
128.084
128.051
119.260
113.146
112.036
105.136
104.552
83.889
82.690
82.327
81.203
77.877
77.453
77.176
77.030
72.817
69.018
67.467
35.926
27.156
27.120
27.007
26.599
26.524
O2N
OBn
28
ppm
140
120
100
80
60
40
20
0

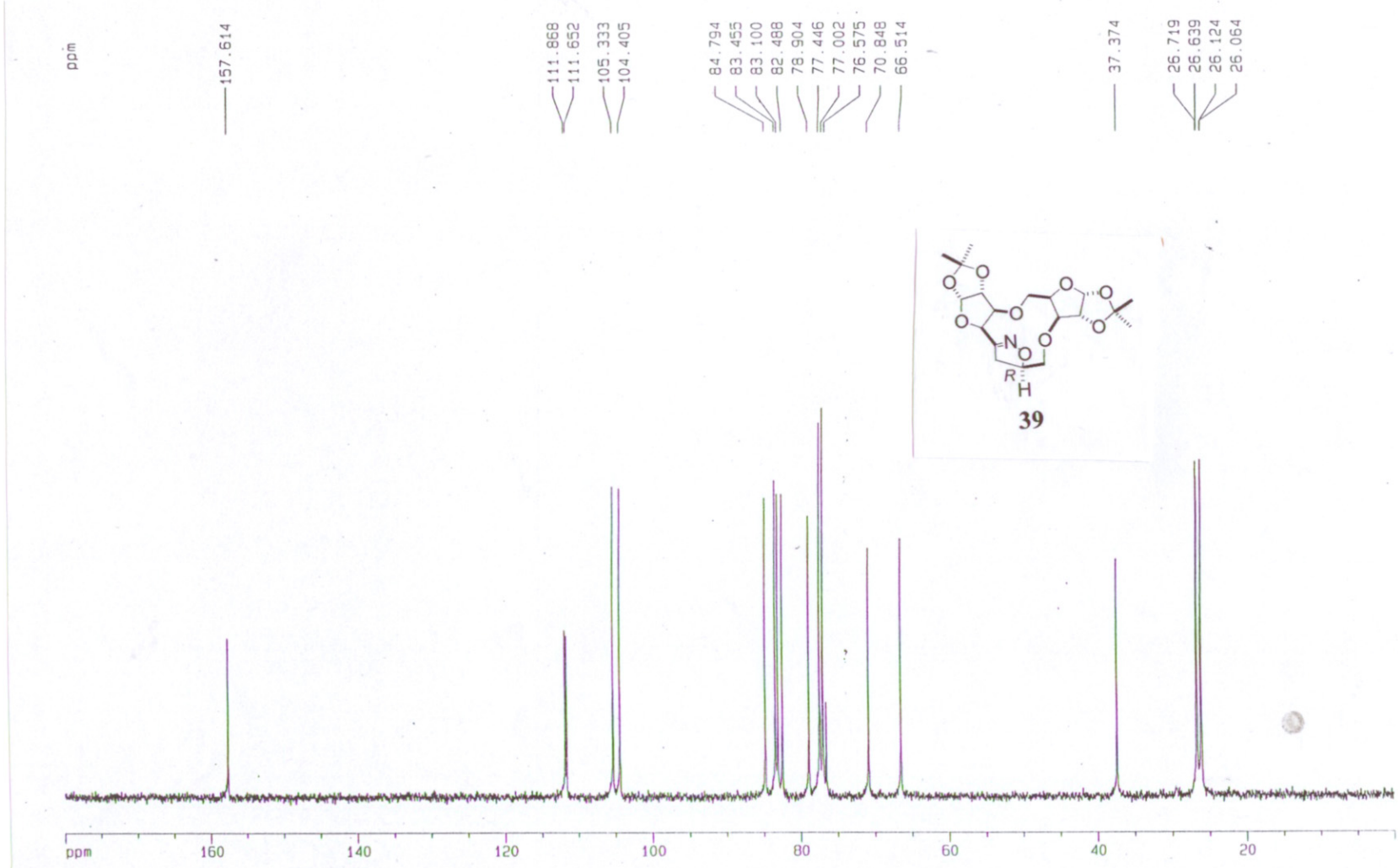

ppm
133.530
117.683
111.900
111.588
104.993
104.897
84.073
82.253
82.041
81.426
79.910
79.264
77.416
76.992
76.567
70.771
68.160
59.964
26.721
26.652
26.178
OH
O
30
ppm
140
120
100
80
60
40
20

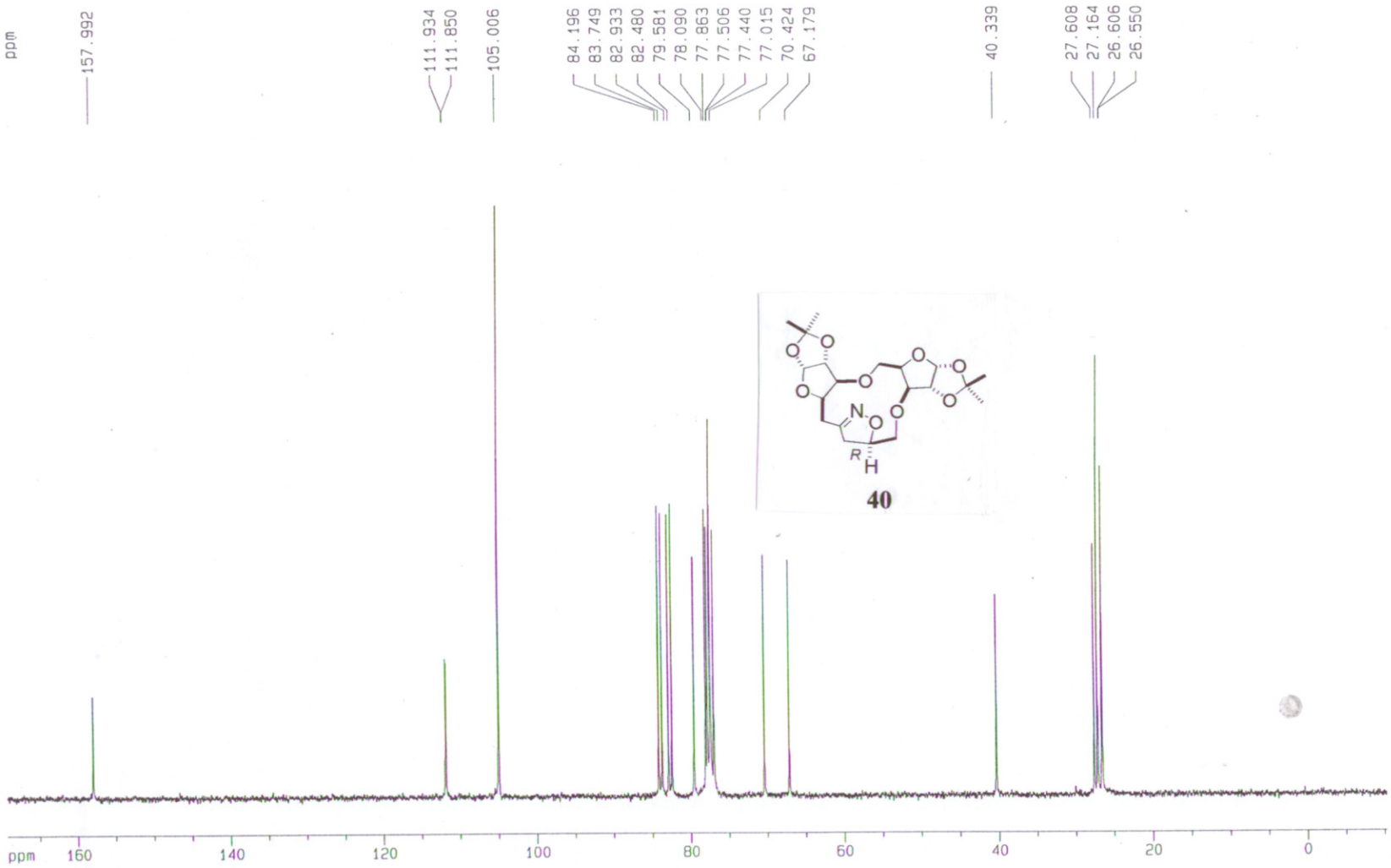

ppm
134.197
118.014
112.104
112.080
112.057
109.251
105.759
105.525
105.393
82.968
82.889
82.702
82.542
82.415
82.098
81.449
79.244
78.809
77.851
77.427
77.004
72.968
71.271
68.121
67.514
67.204
27.268
27.206
26.644
25.745
32
ppm
140
120
100
80
60
40
20


ppm
133.583
117.578
112.041
111.629
111.587
105.236
104.971
104.733
84.061
82.719
82.082
81.901
81.729
81.484
80.011
79.597
78.821
77.425
77.001
76.576
70.660
69.005
68.586
67.667
64.368
26.729
26.649
26.181
26.120
HO
HO
H
33
ppm
140
120
100
80
60
40
20

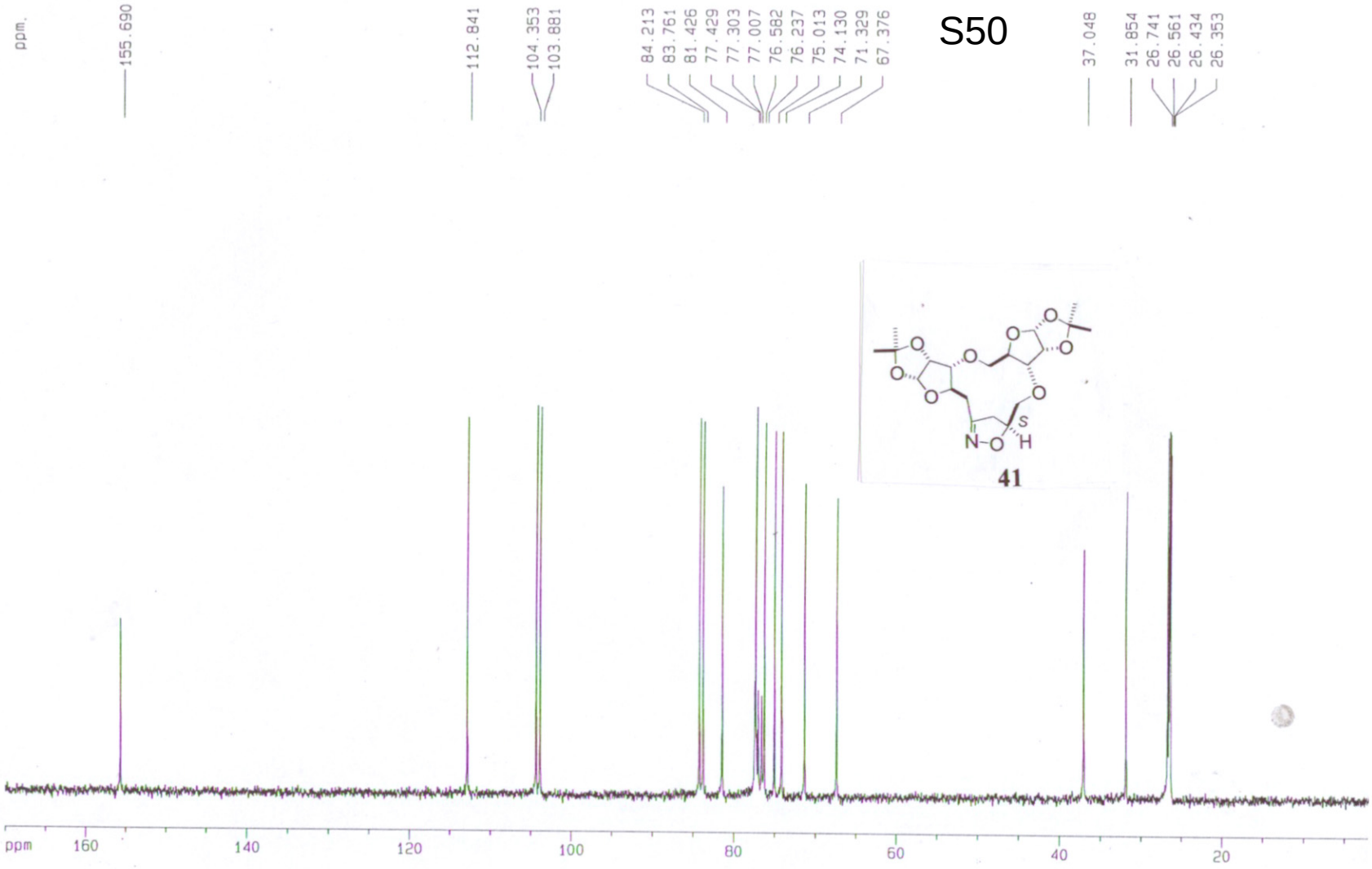

ppm
148.878
147.103
133.581
133.545
117.570
117.520
111.749
111.617
111.572
111.538
111.511
105.093
104.902
104.750
104.570
84.479
82.993
82.540
82.300
82.236
81.972
81.925
81.807
81.708
81.448
81.359
78.714
78.678
78.278
78.078
77.605
77.013
70.653
70.622
67.820
66.704
26.680
26.560
26.055
HO
N
35
ppm
160
140
120
100
80
60
40
20

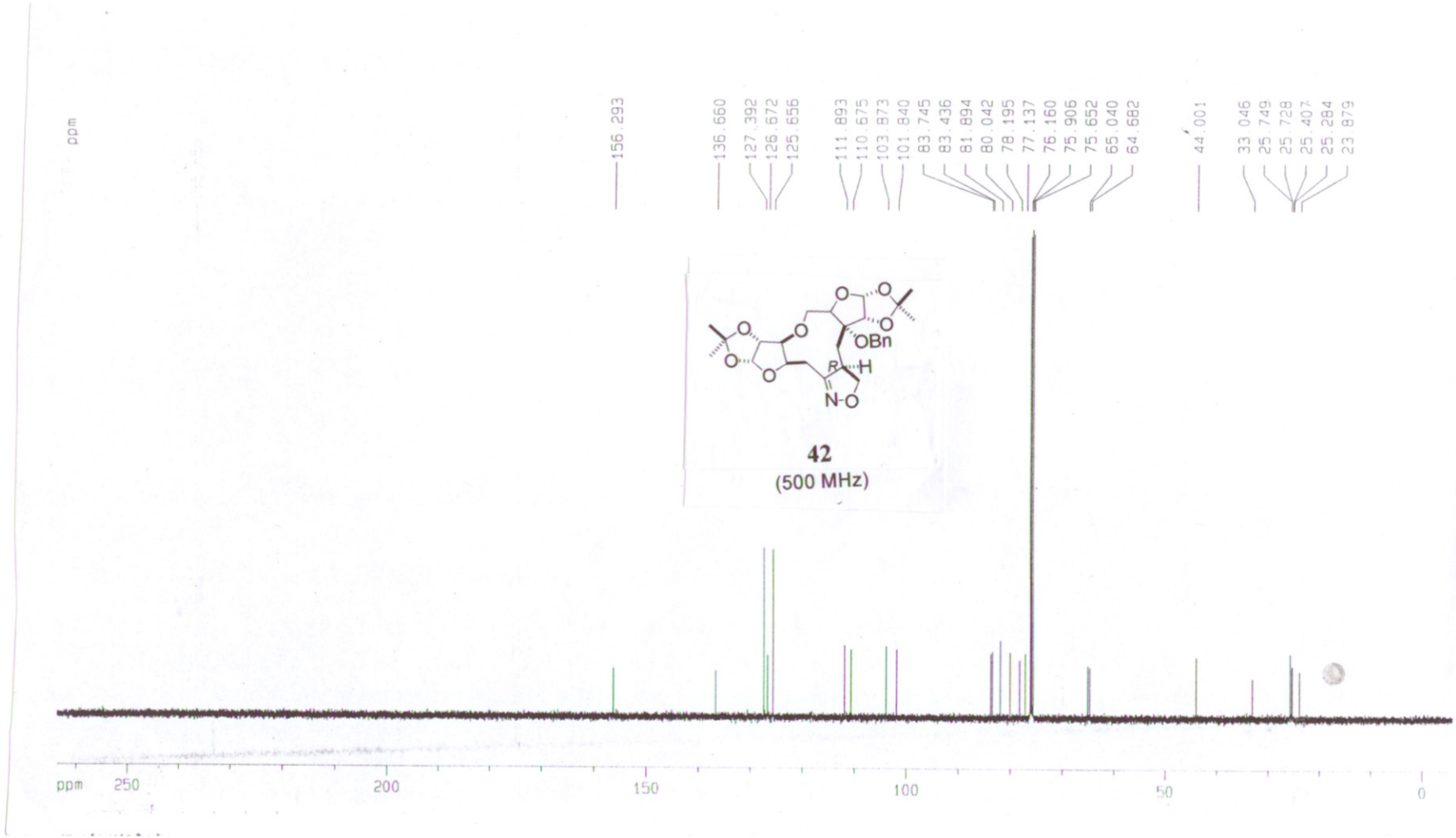

ppm
133.581
117.395
111.476
111.392
111.310
104.897
104.790
104.522
83.190
82.570
81.755
81.703
81.655
81.492
78.869
78.759
77.435
77.009
76.584
76.435
72.259
70.548
67.706
67.035
26.518
25.977
25.895
O2N
37
ppm
140
120
100
80
60
40
20

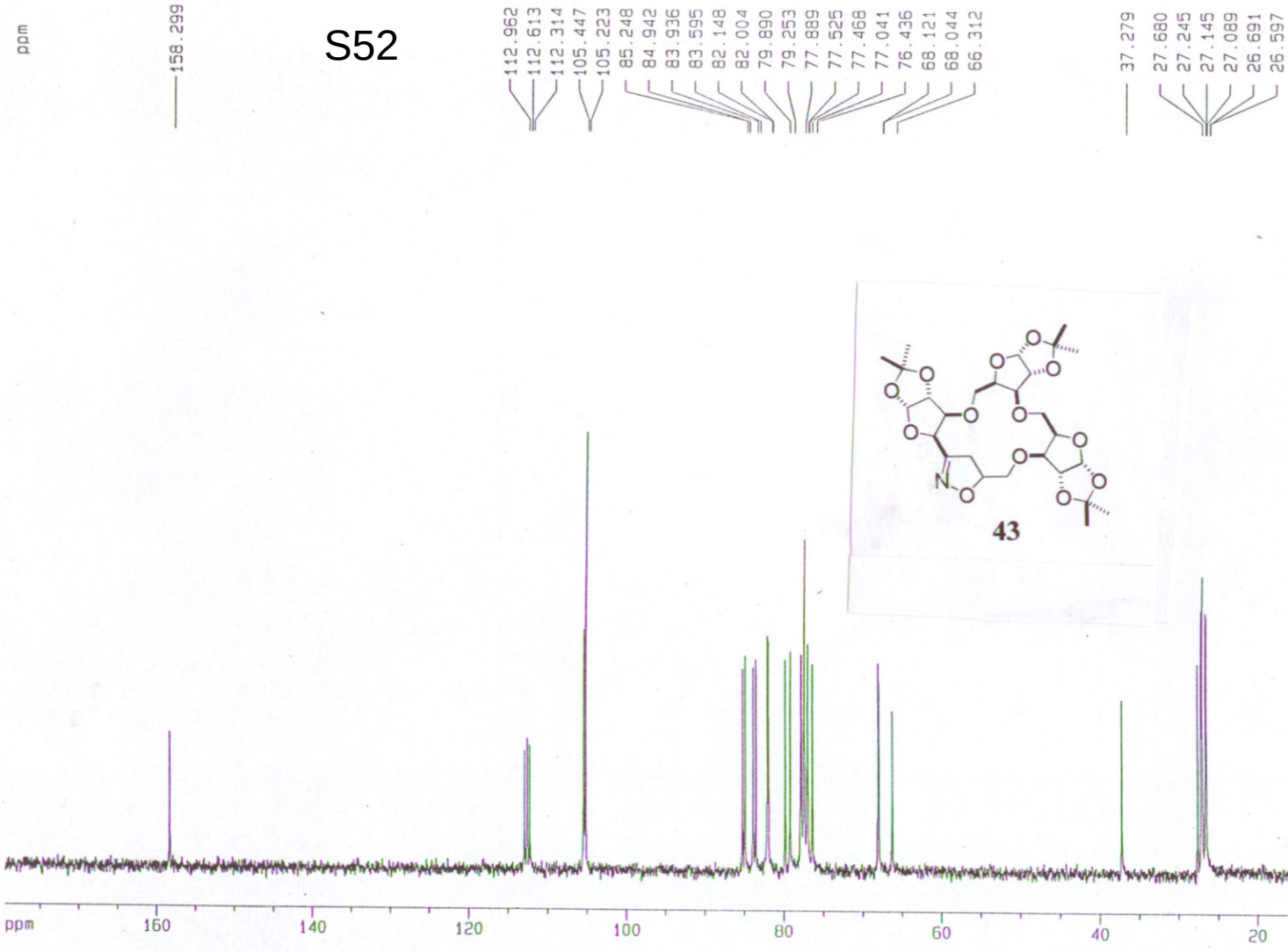

ppm
157.614
111.868
111.652
105.333
104.405
84.794
83.455
83.100
82.488
78.904
77.446
77.002
76.575
70.848
66.514
37.374
26.719
26.639
26.124
26.064
39
ppm
160
140
120
100
80
60
40
20

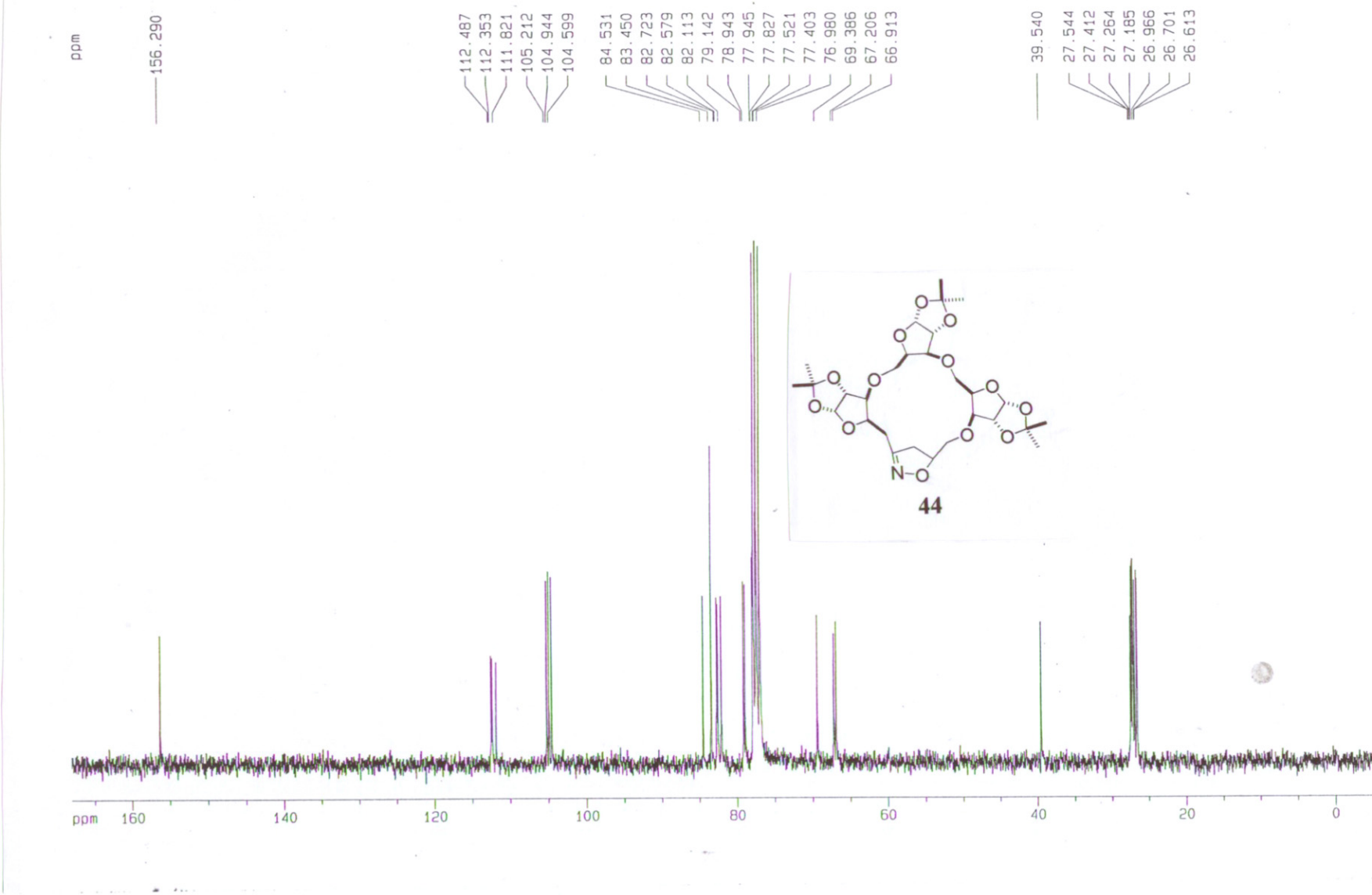

ppm
157.992
111.934
111.850
105.006
84.196
83.749
82.933
82.480
79.581
78.090
77.863
77.506
77.440
77.015
70.424
67.179
40.339
27.608
27.164
26.606
26.550
ppm
160
140
120
100
80
60
40
20
0

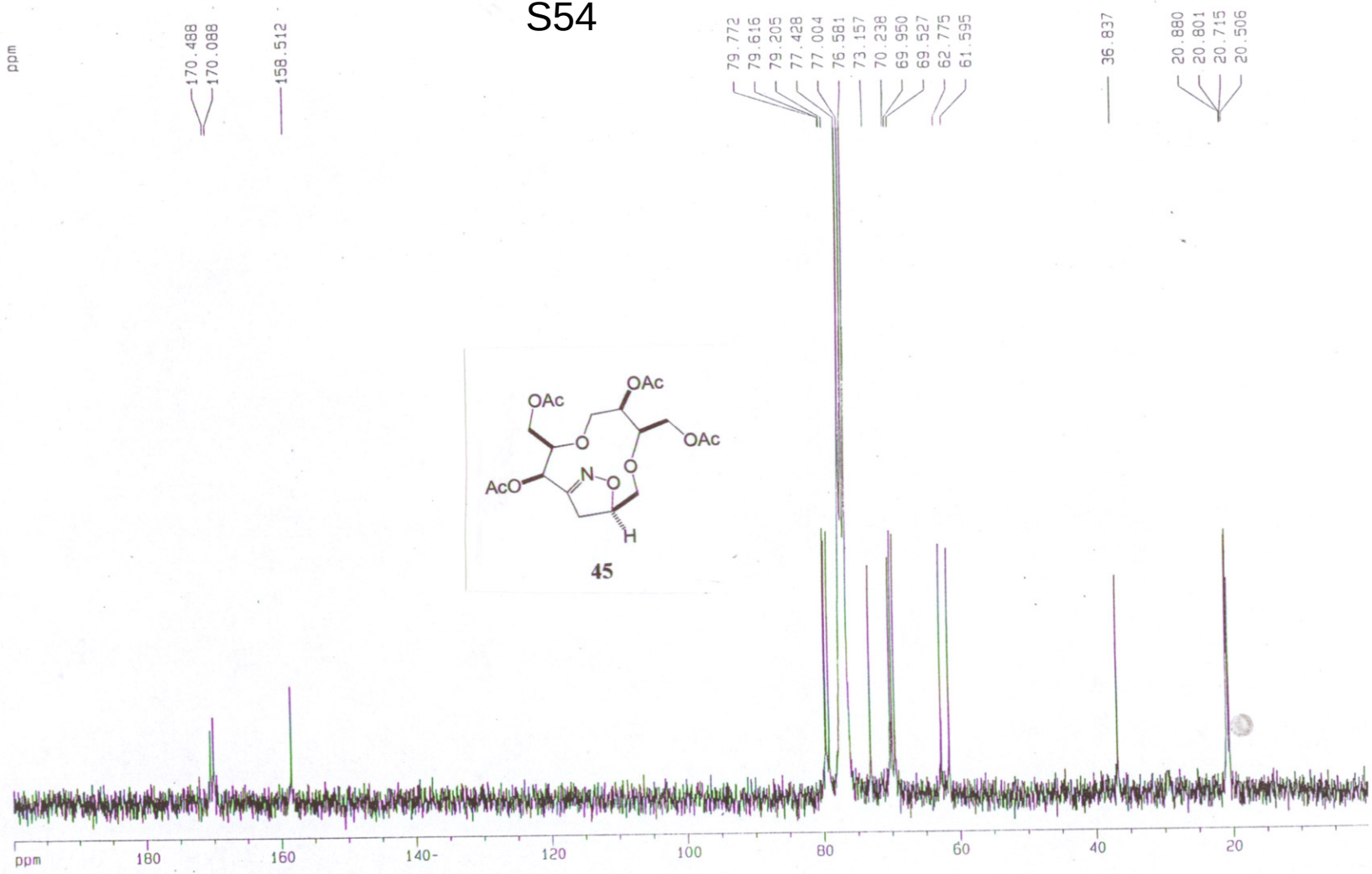
40

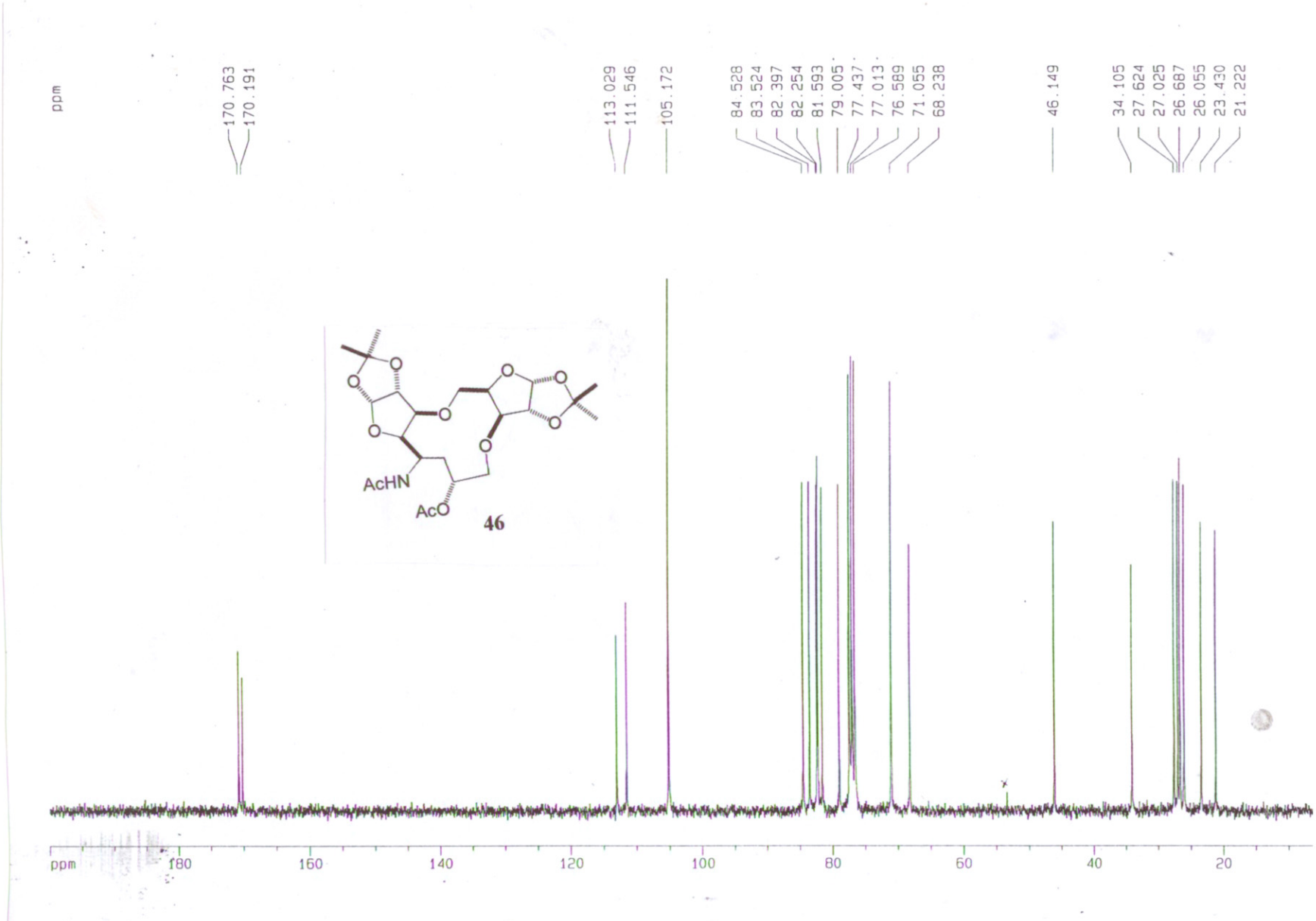

ppm
155.690
112.841
104.353
103.881
84.213
83.761
81.426
77.429
77.303
77.007
76.582
76.237
75.013
74.130
71.329
67.376
37.048
31.854
26.741
26.561
26.434
26.353
41
ppm
160
140
120
100
80
60
40
20

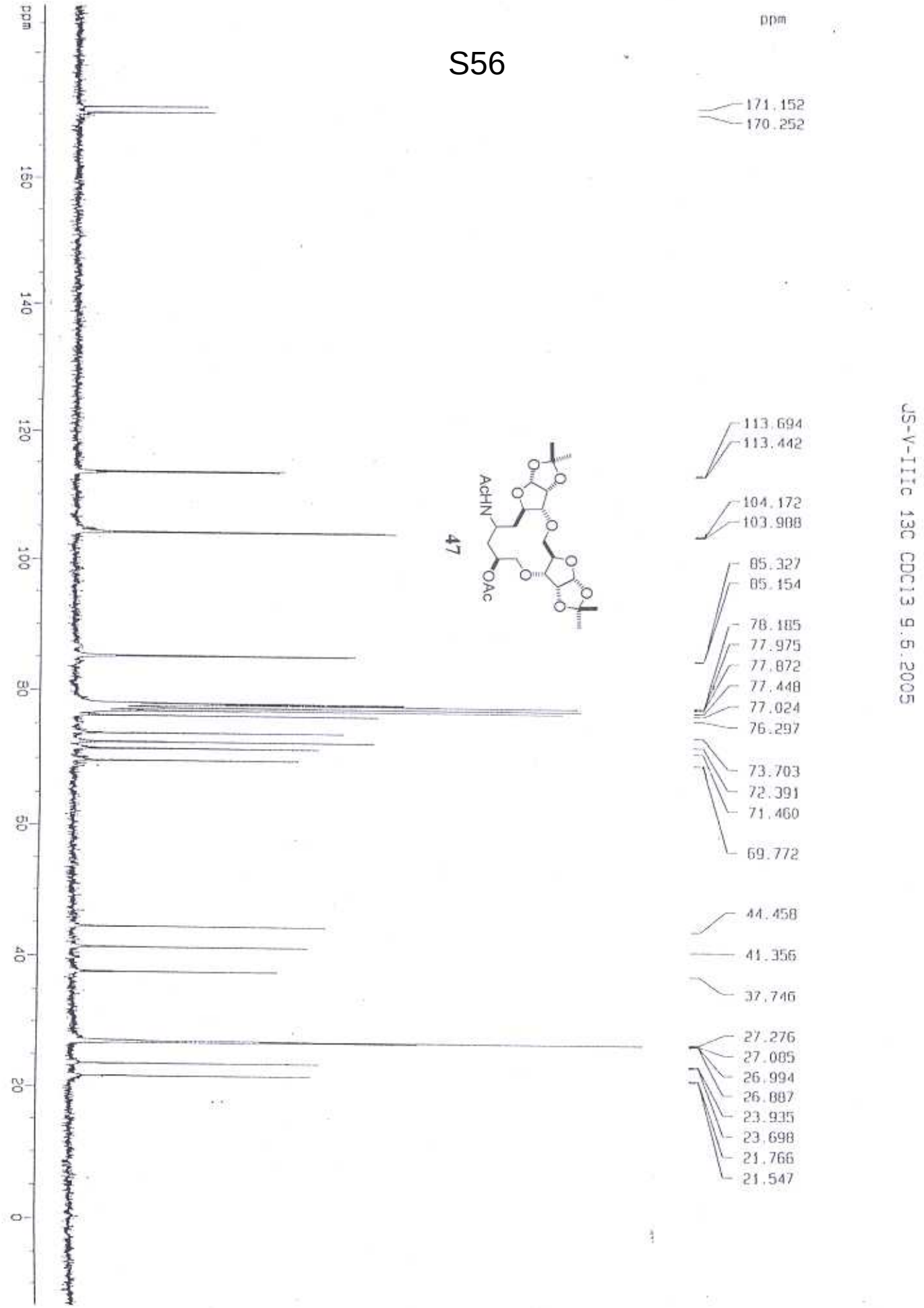

ppm
156.293
136.660
127.392
126.672
125.656
111.893
110.675
103.873
101.840
83.745
83.436
81.894
80.042
78.195
77.137
76.160
75.906
75.652
65.040
64.682
44.001
33.046
25.749
25.728
25.407
25.284
23.879
OBn
R
H
N-O
42
(500 MHz)
ppm
250
200
150
100
50
0

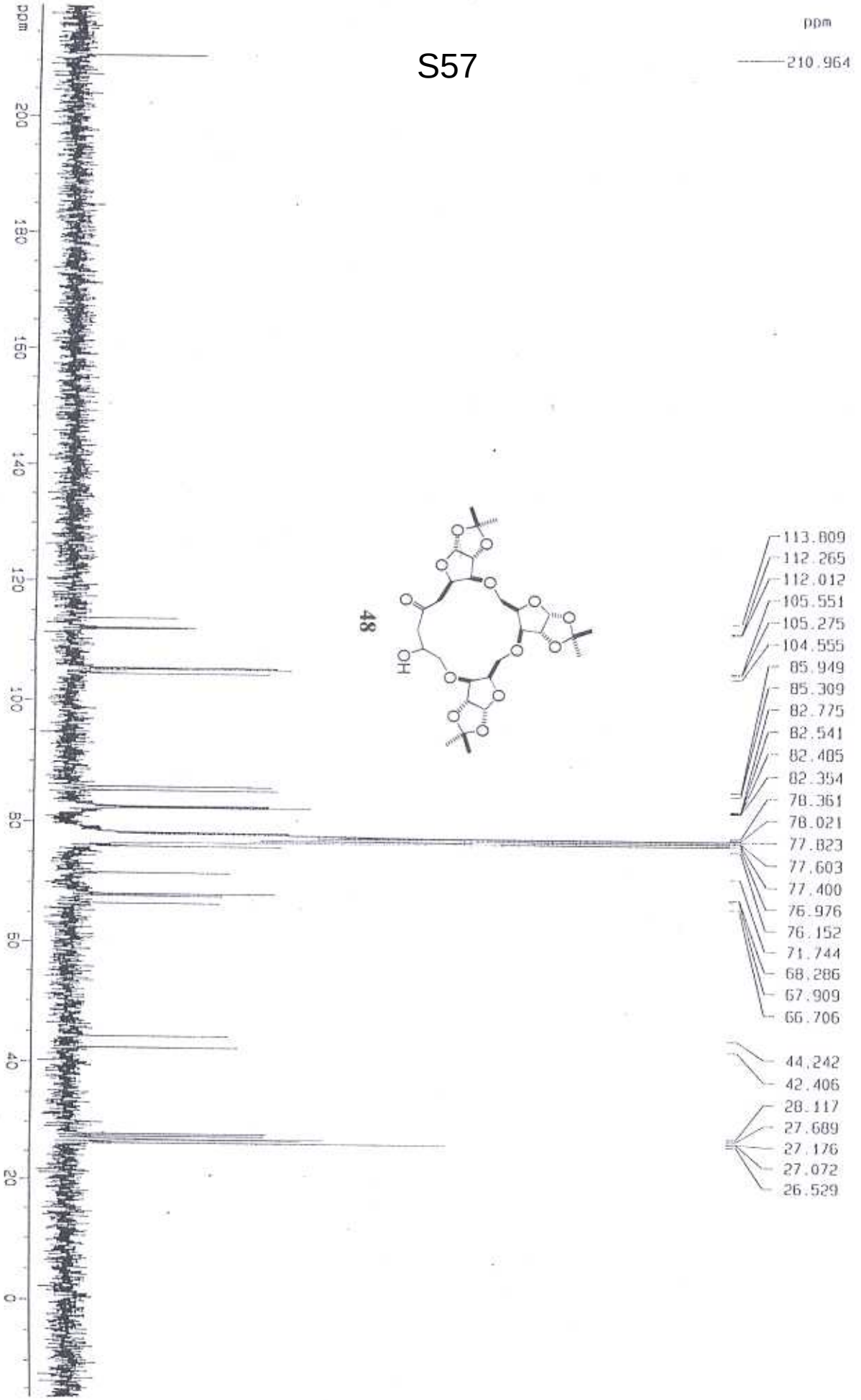

ppm
158.299
112.962
112.613
112.314
105.447
105.223
85.248
84.942
83.936
83.595
82.148
82.004
79.890
79.253
77.889
77.525
77.468
77.041
76.436
68.121
68.044
66.312
37.279
27.680
27.245
27.145
27.089
26.691
26.597
43
ppm
160
140
120
100
80
60
40
20
0


ppm
156.290
112.487
112.353
111.821
105.212
104.944
104.599
84.531
83.450
82.723
82.579
82.113
79.142
78.943
77.945
77.827
77.521
77.403
76.980
69.386
67.206
66.913
39.540
27.544
27.412
27.264
27.185
26.966
26.701
26.613
44
ppm
160
140
120
100
80
60
40
20
0


ppm
170.488
170.088
158.512
79.772
79.616
79.205
77.428
77.004
76.581
73.157
70.238
69.950
69.527
62.775
61.595
36.837
20.880
20.801
20.715
20.506
OAc
OAc
OAc
AcO
N
O
O
O
H
45
ppm
180
160
140
120
100
80
60
40
20


ppm
170.763
170.191
113.029
111.546
105.172
84.528
83.524
82.397
82.254
81.593
79.005
77.437
77.013
76.589
71.055
68.238
46.149
34.105
27.624
27.025
26.687
26.055
23.430
21.222
AcHN
AcO
46
ppm
180
160
140
120
100
80
60
40
20


ppm
171.152
170.252
113.694
113.442
104.172
103.908
85.327
85.154
78.185
77.975
77.872
77.448
77.024
76.297
73.703
72.391
71.460
69.772
44.458
41.356
37.746
27.276
27.085
26.994
26.887
23.935
23.698
21.766
21.547
AcHN
OAc
47
JS-V-IIIc 13C CDCl3 9.5.2005


ppm
210.964
113.809
112.265
112.012
105.551
105.275
104.555
85.949
85.309
82.775
82.541
82.485
82.354
78.361
78.021
77.823
77.603
77.400
76.976
76.152
71.744
68.286
67.909
66.706
44.242
42.406
28.117
27.689
27.176
27.072
26.529
48
OH