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Electronic Supplementary Information

Efficient one-pot per-*O*-acetylation–thioglycosidation of native sugars, 4,6-*O*-arylidenation and one-pot 4,6-*O*-benzylidenation–acetylation of *S*-/*O*-glycosides catalyzed by $Mg(OTf)_2$

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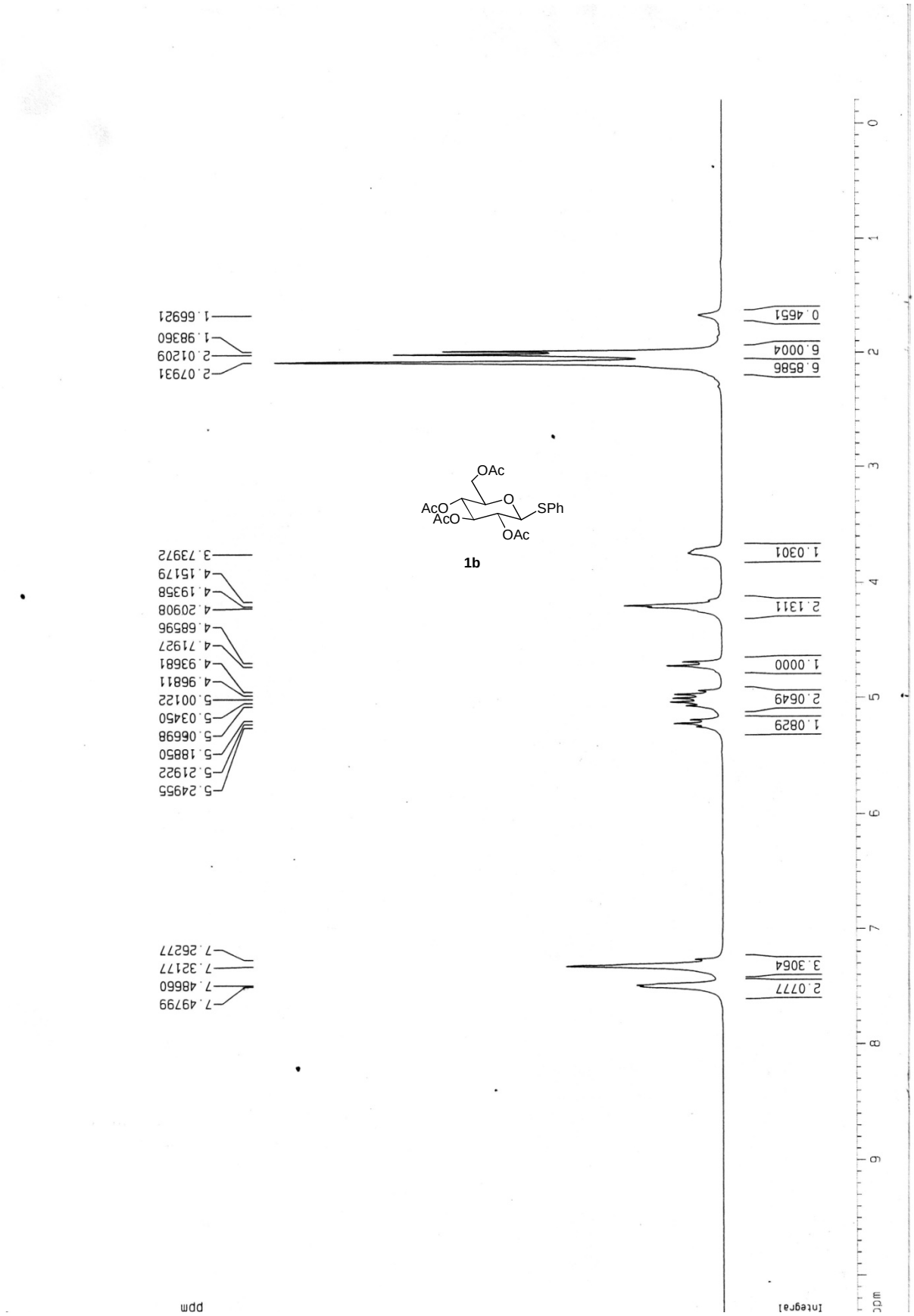
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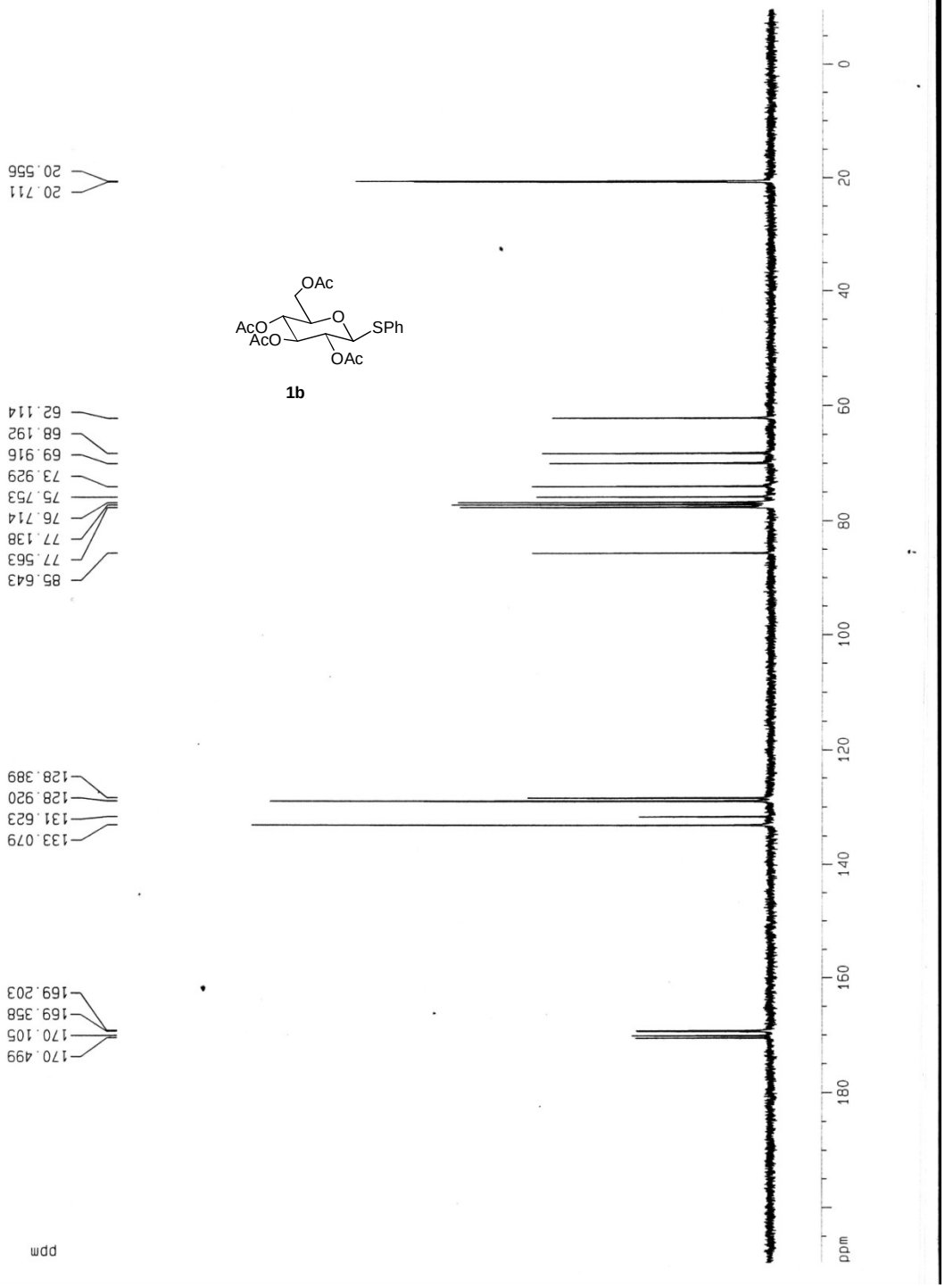
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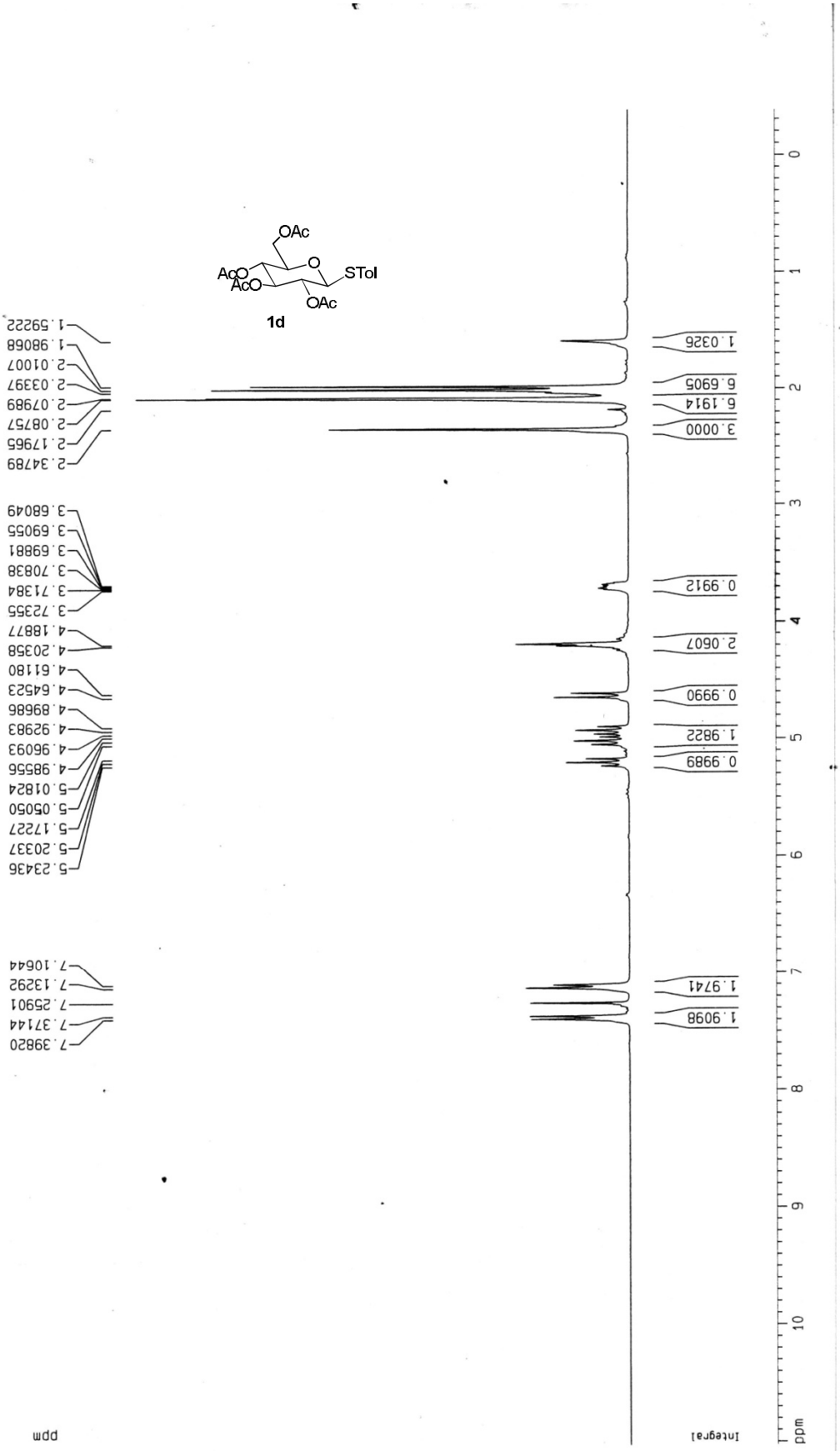
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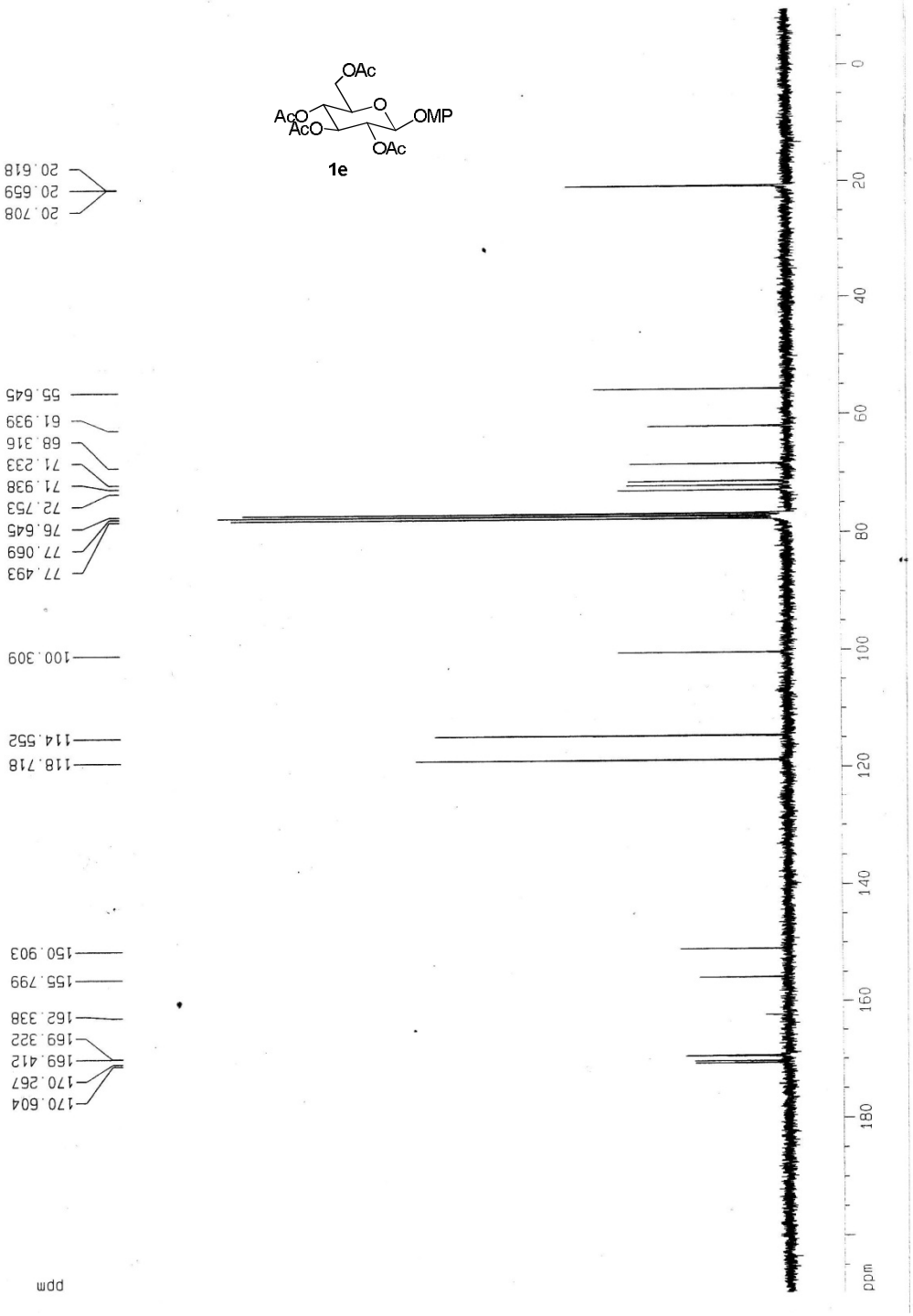
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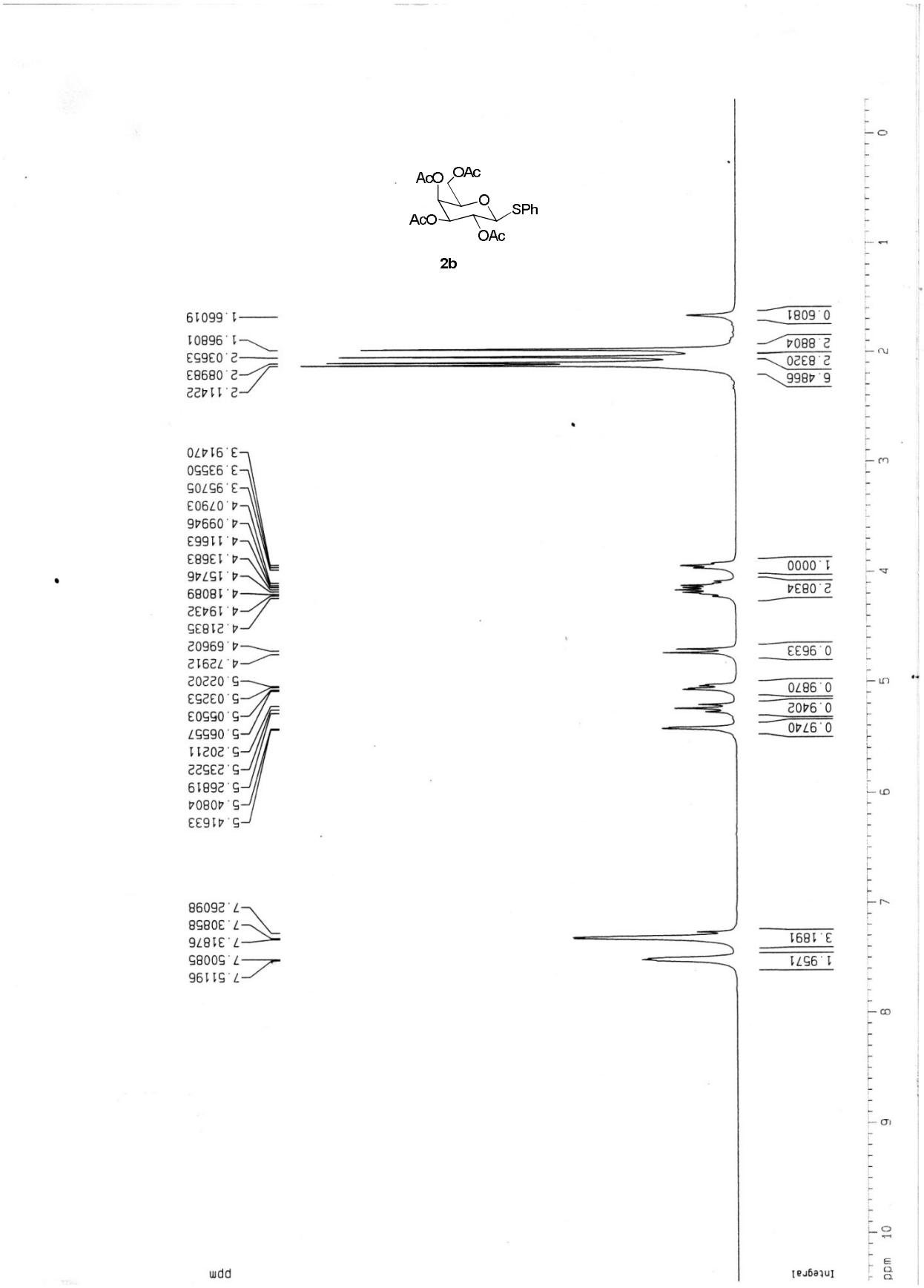
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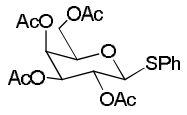
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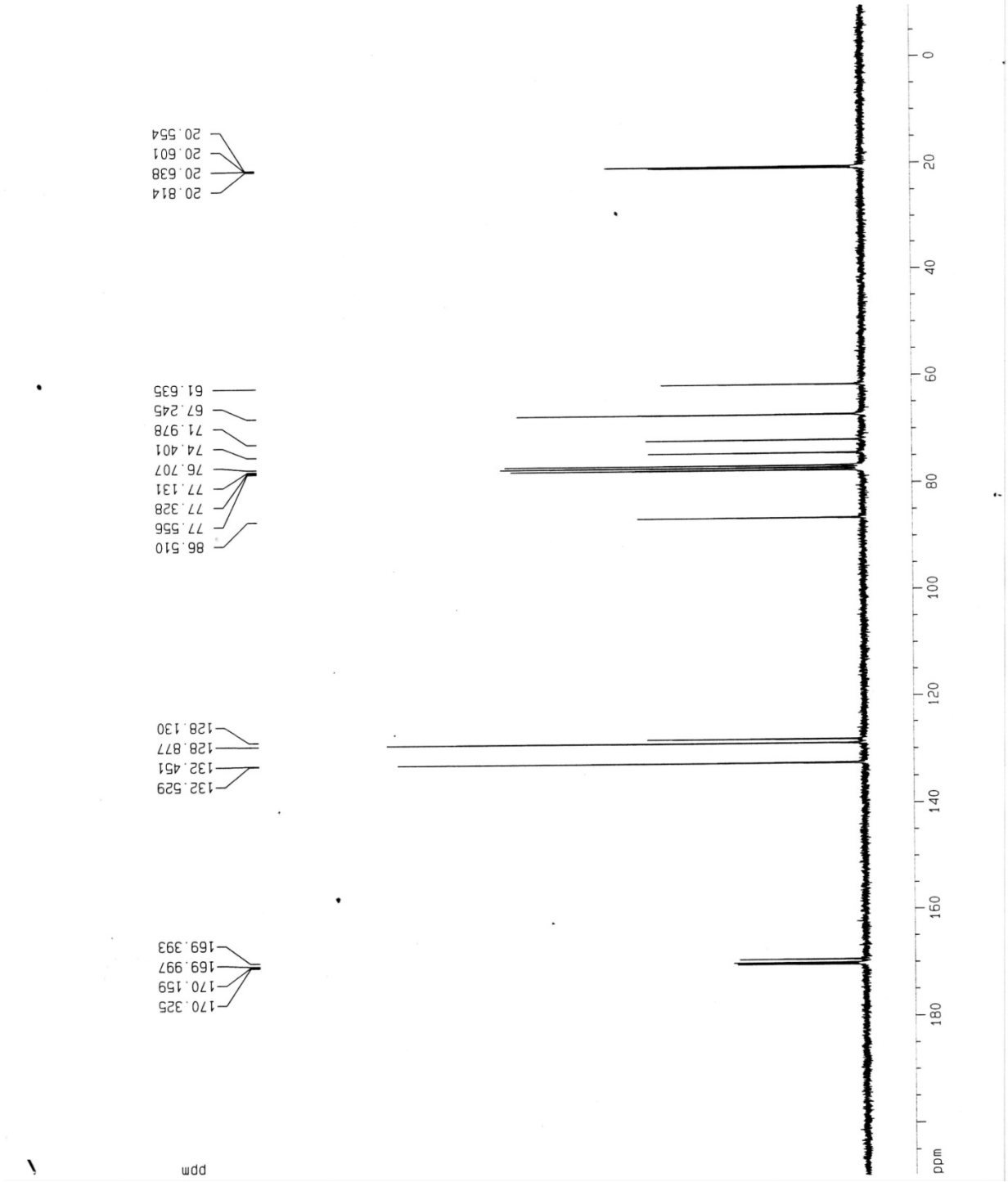
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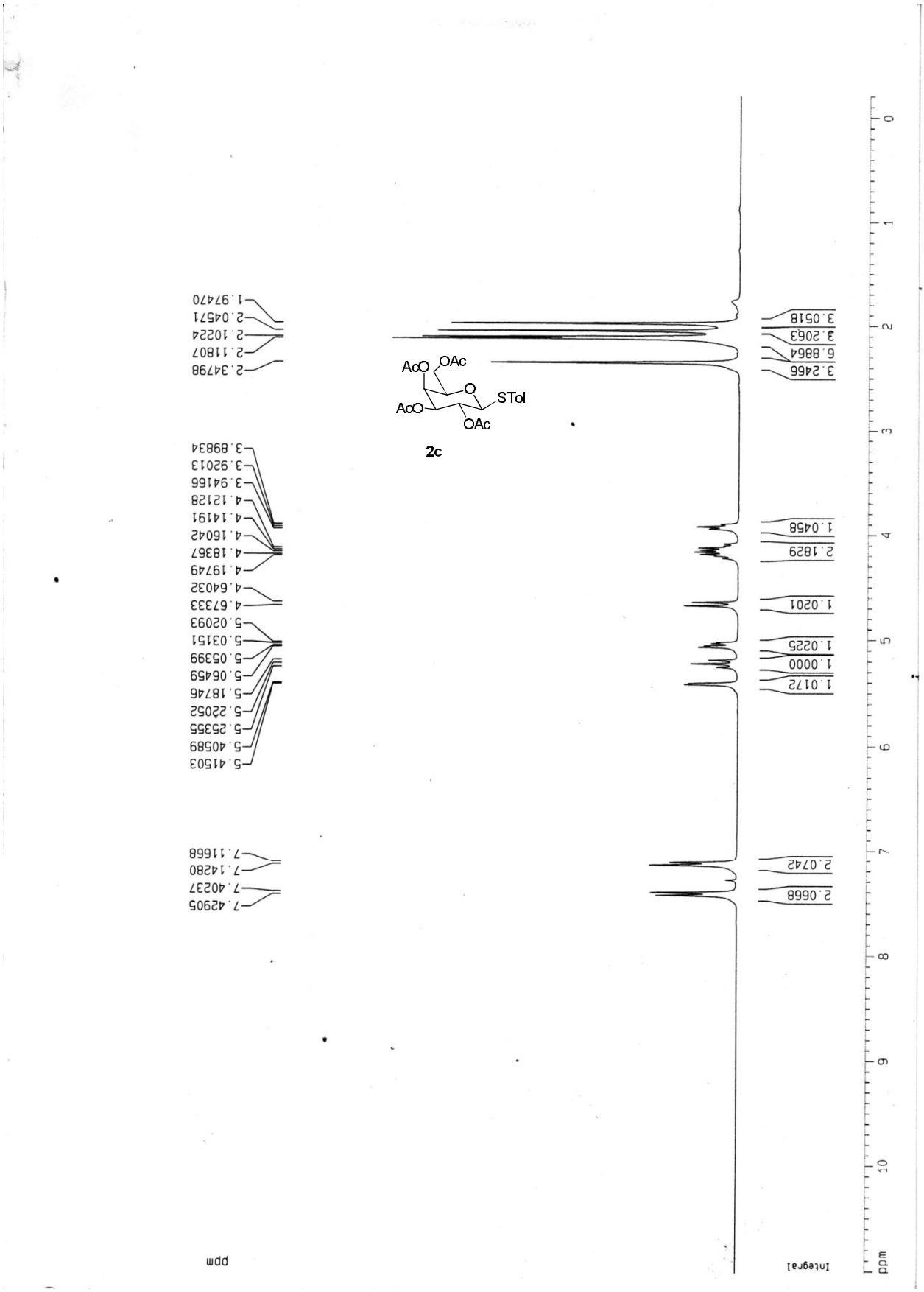
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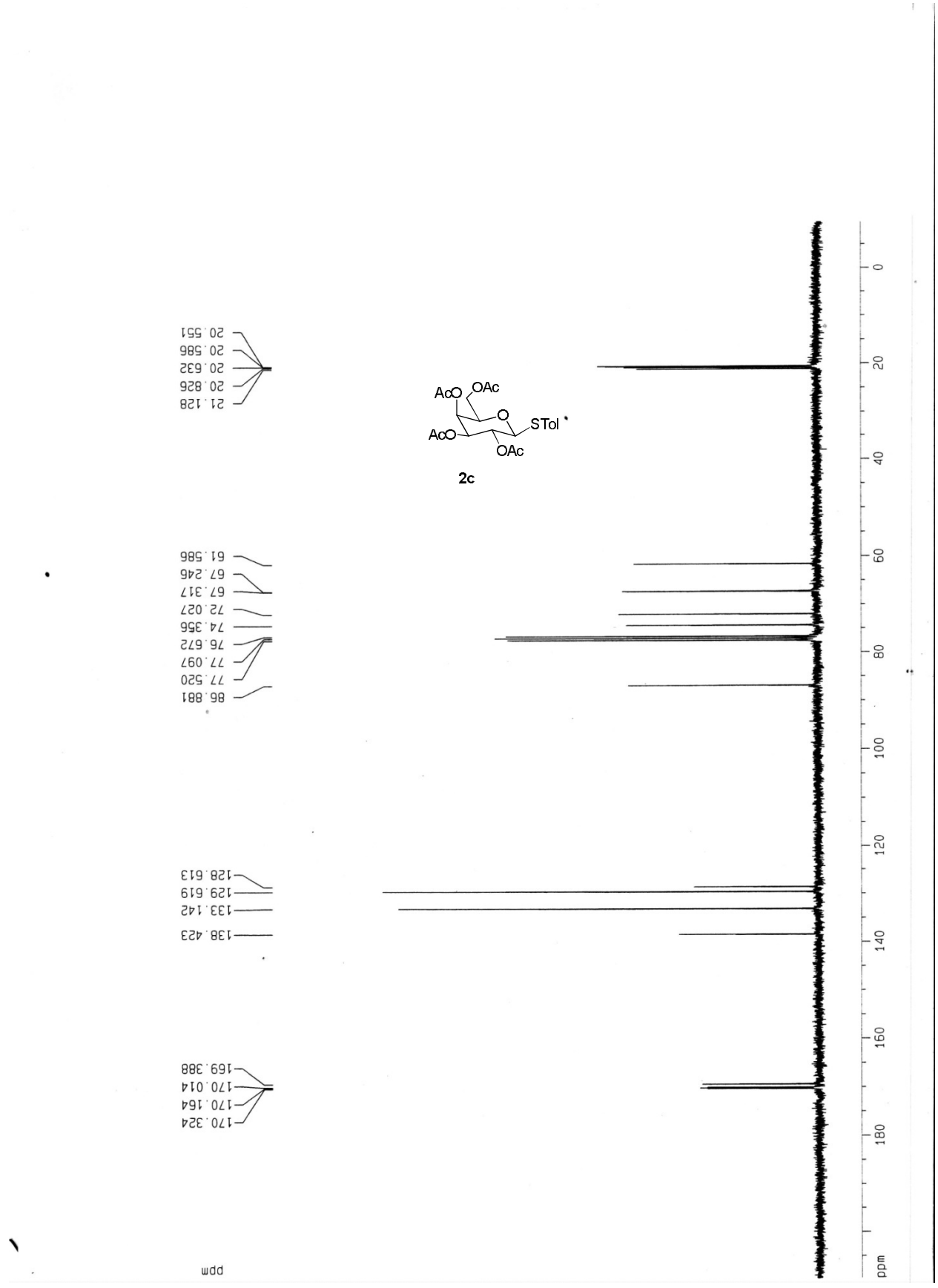
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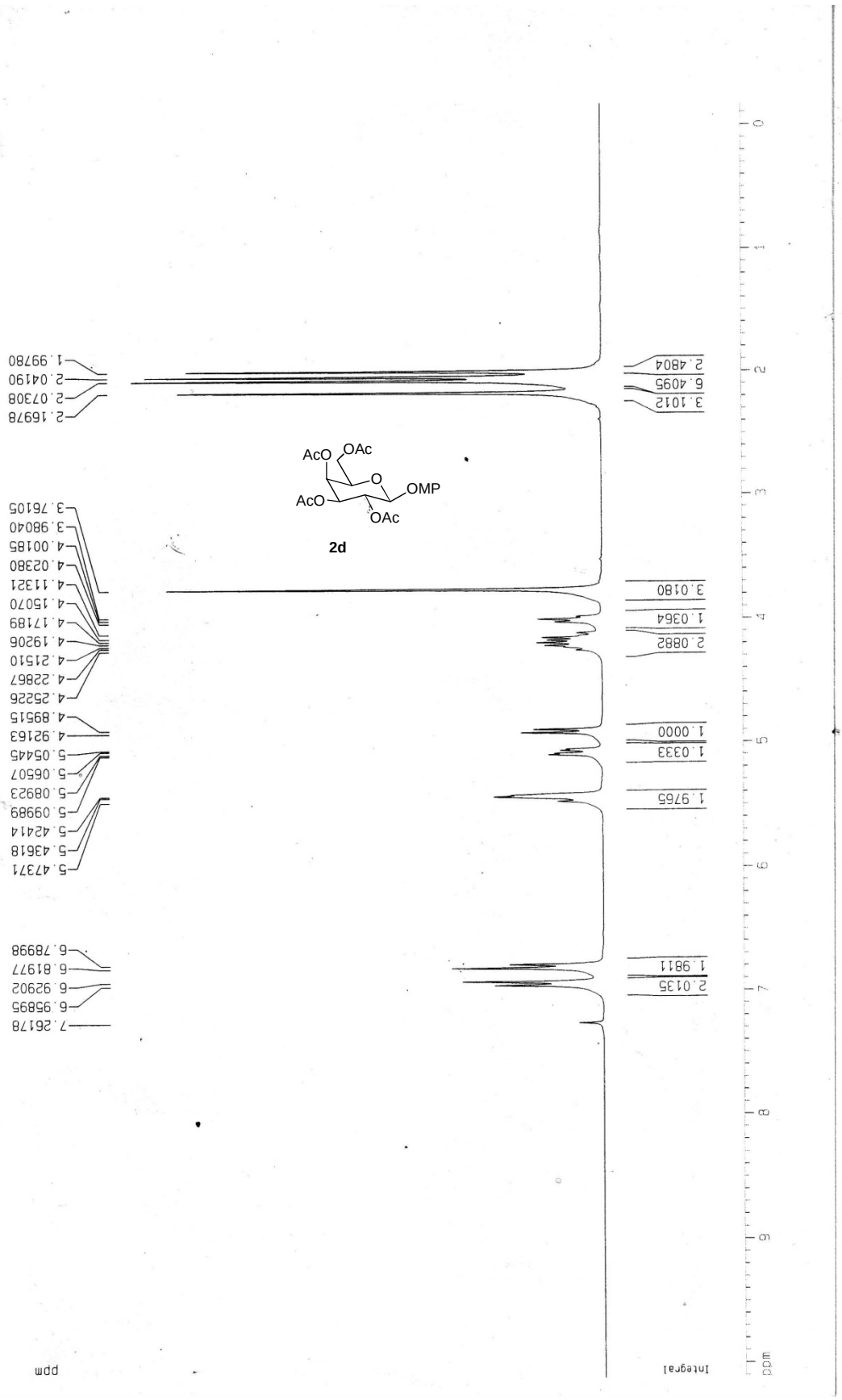
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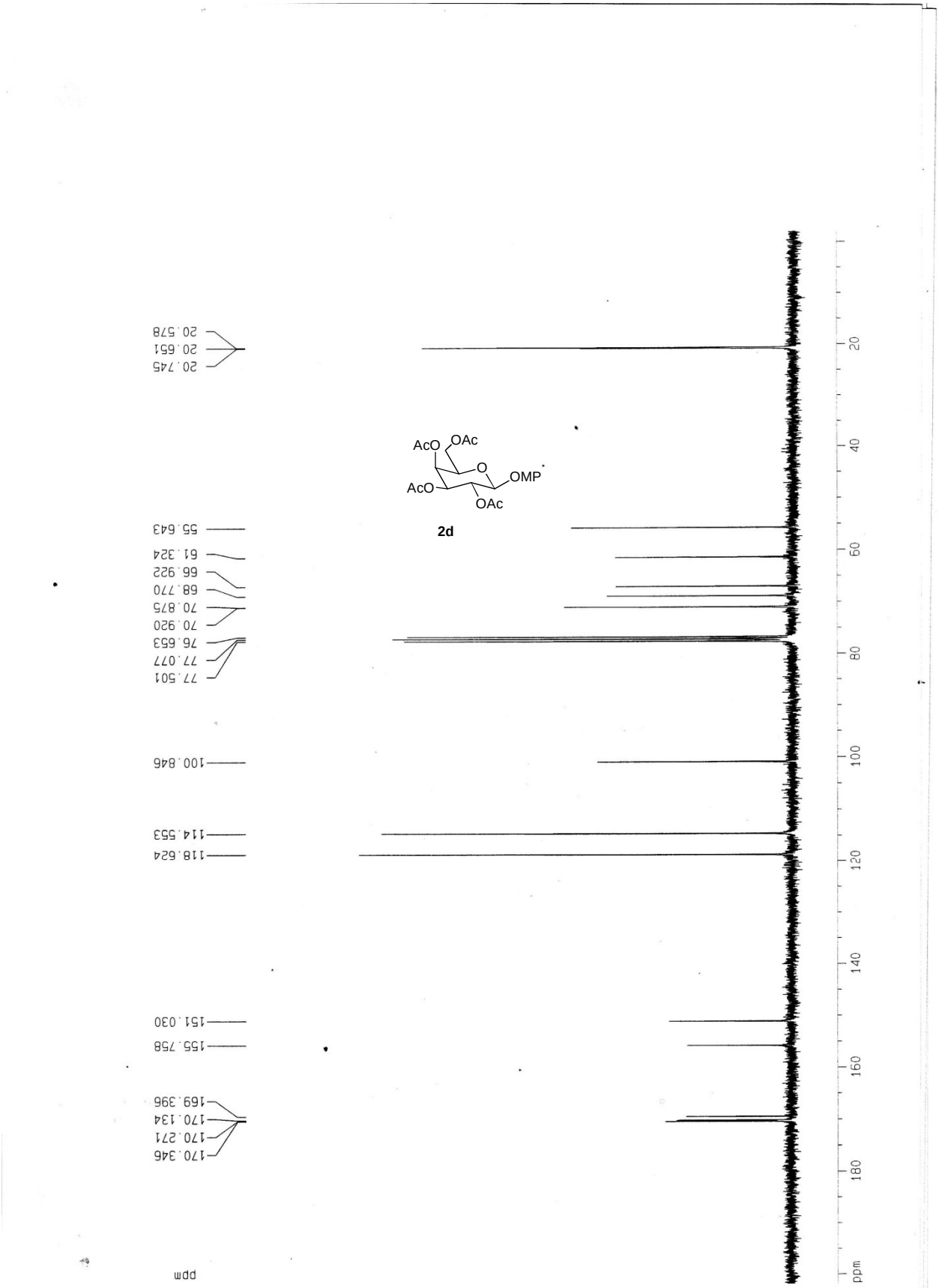
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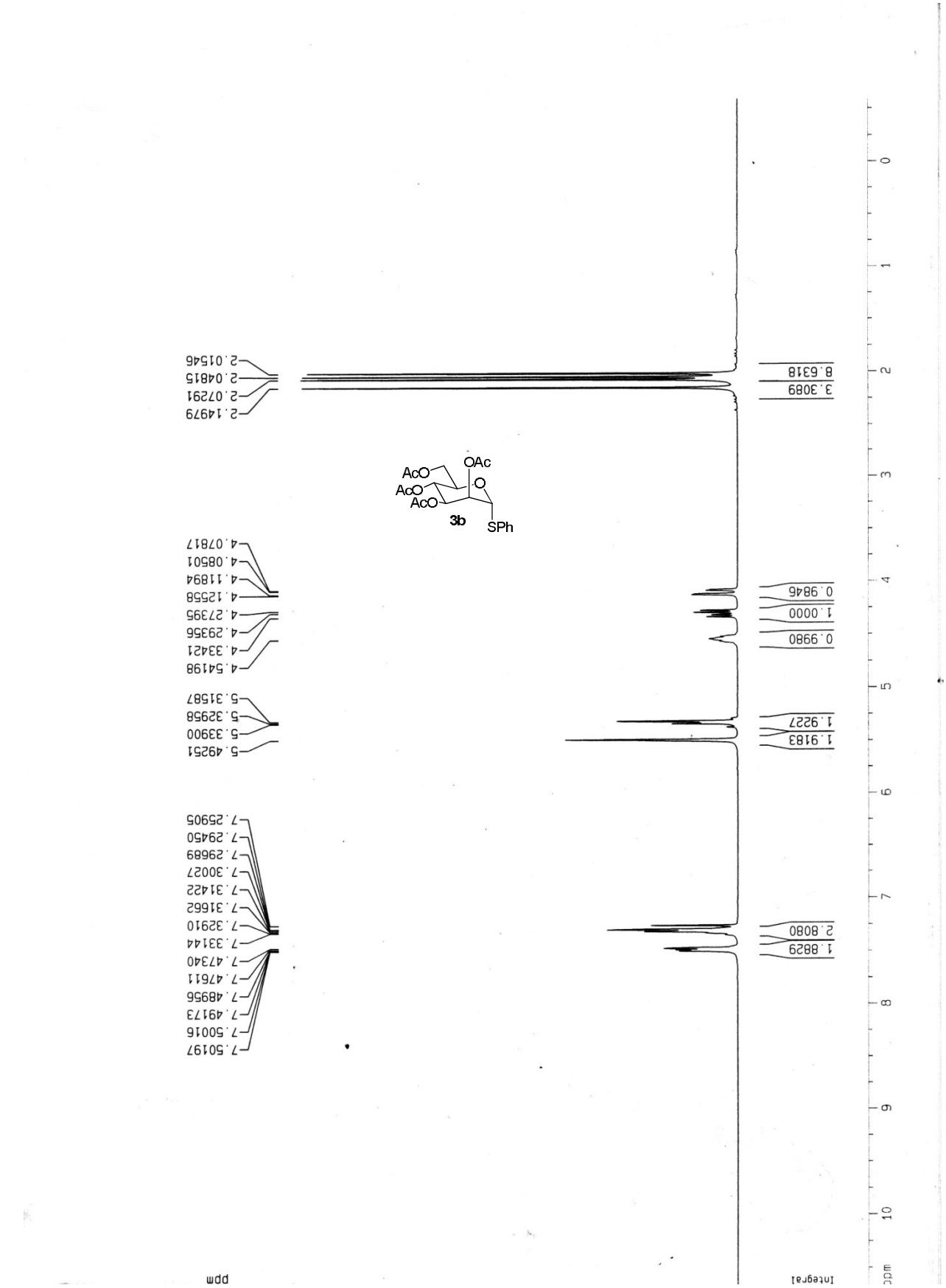
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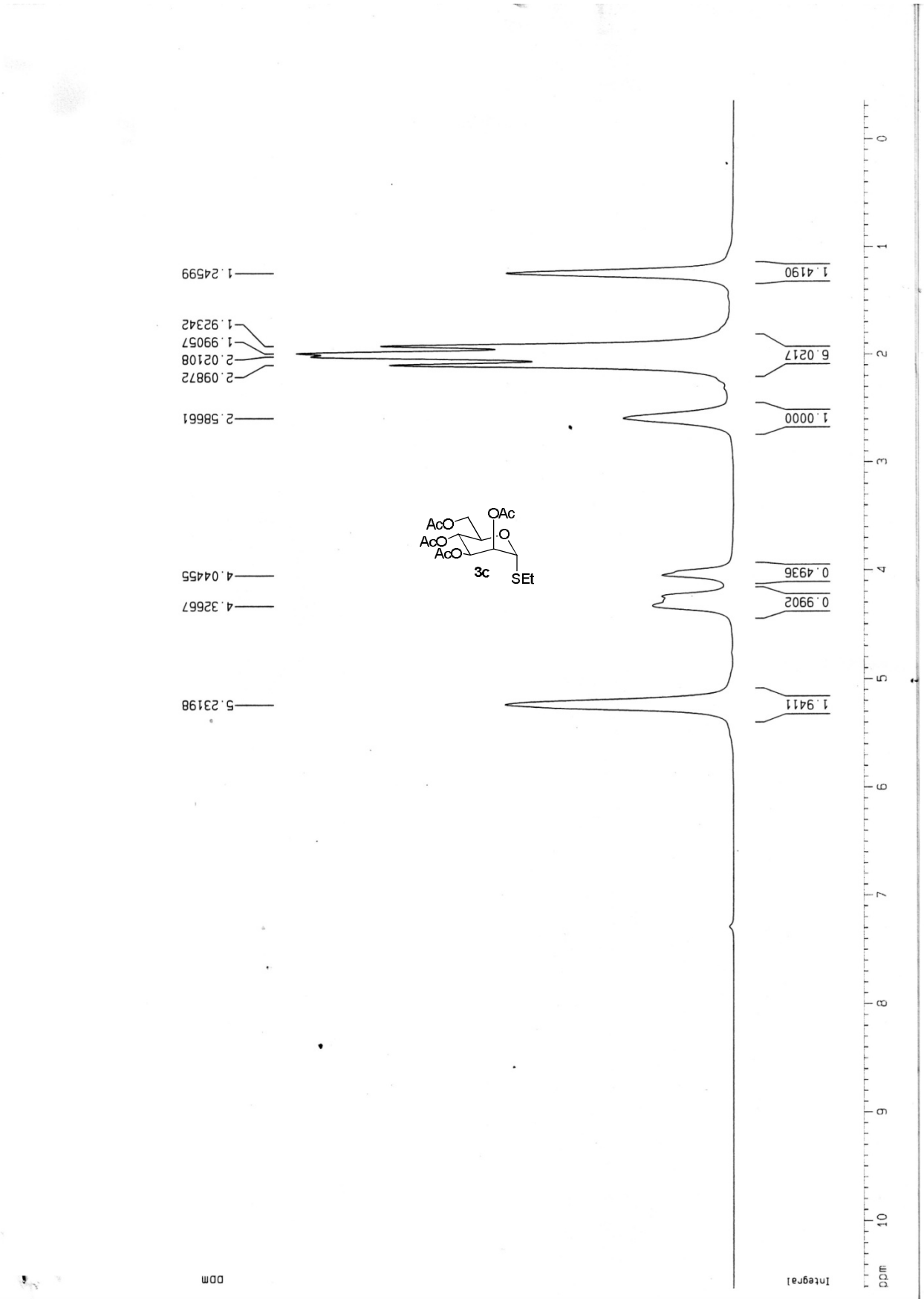
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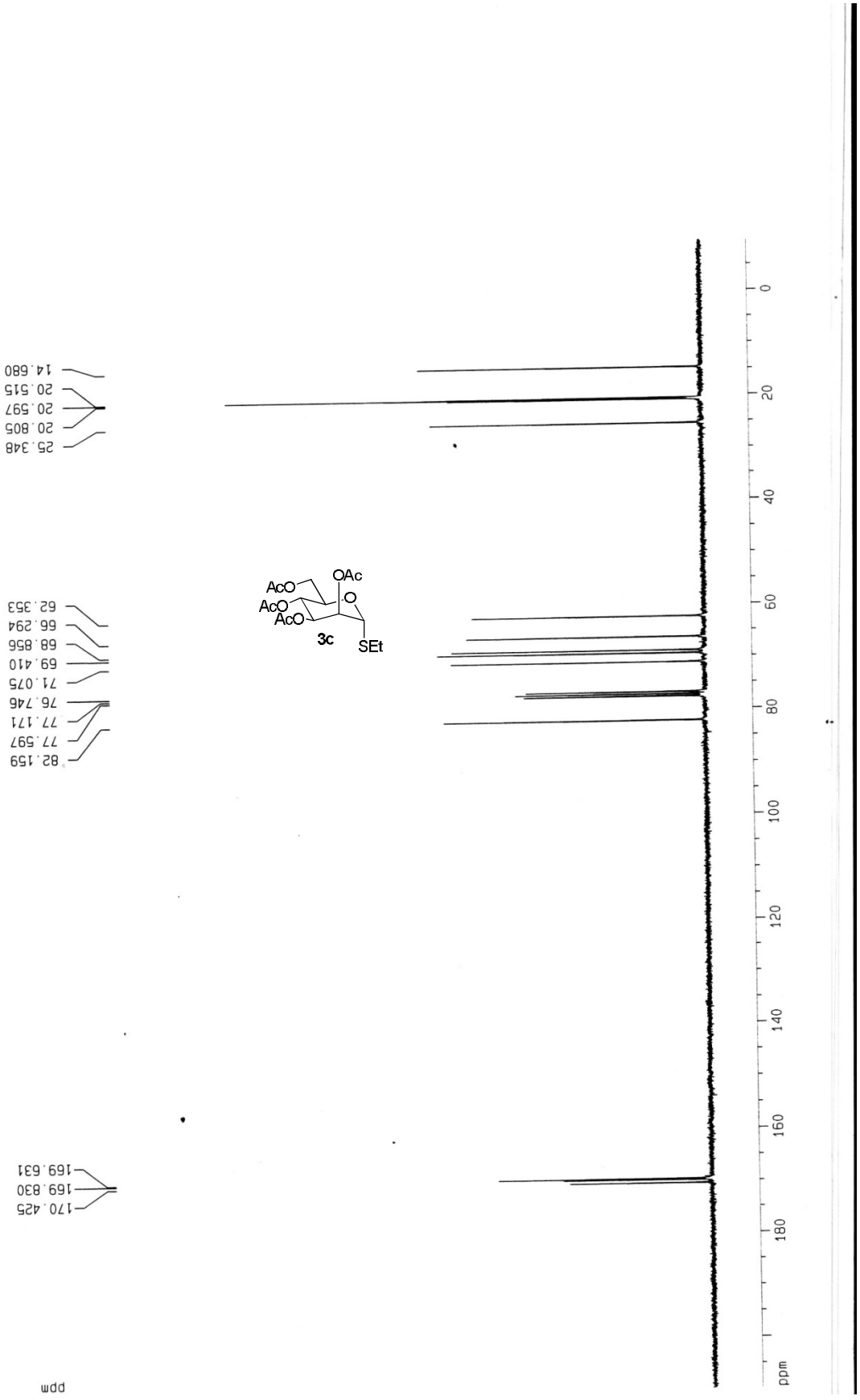
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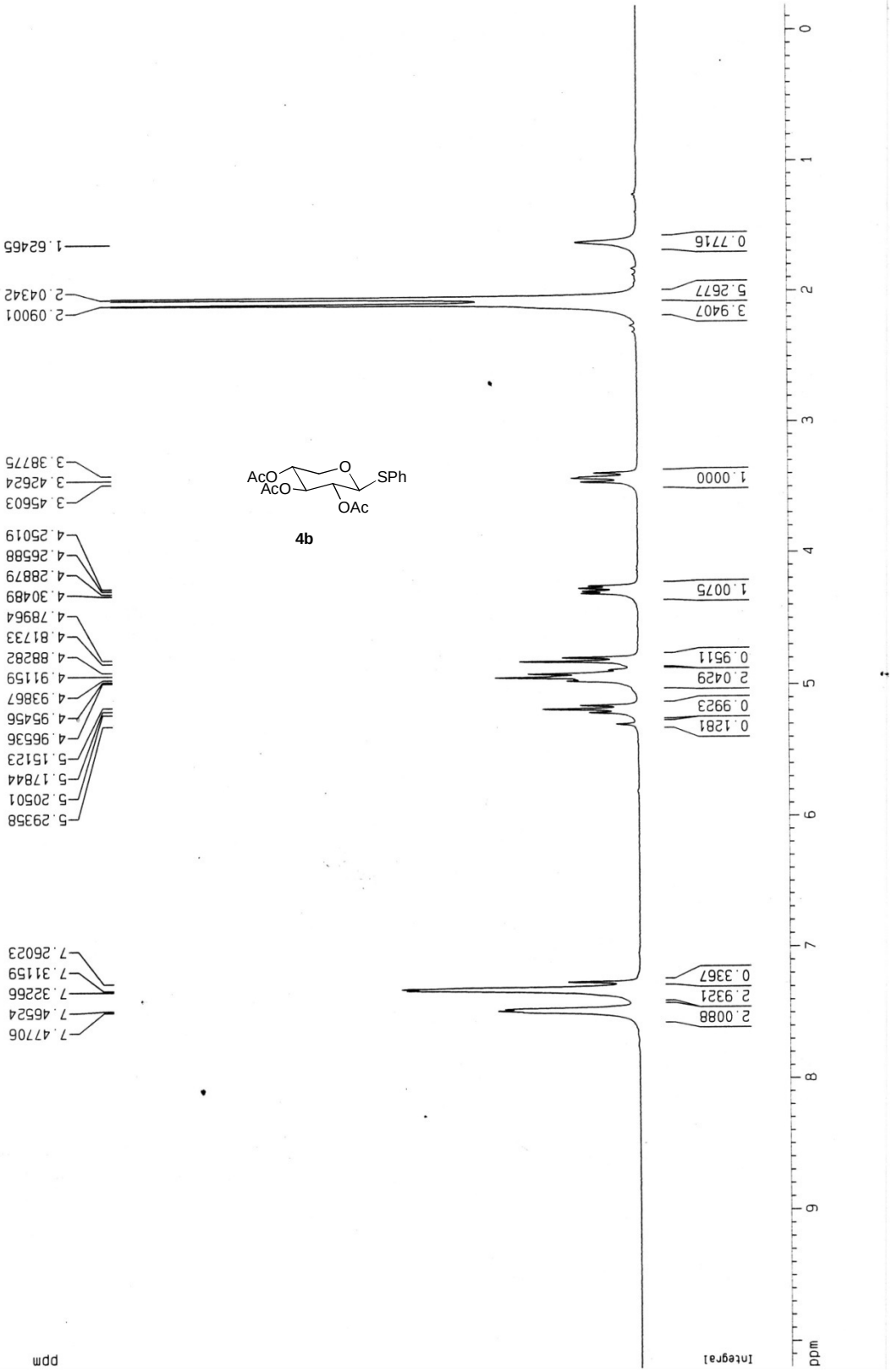
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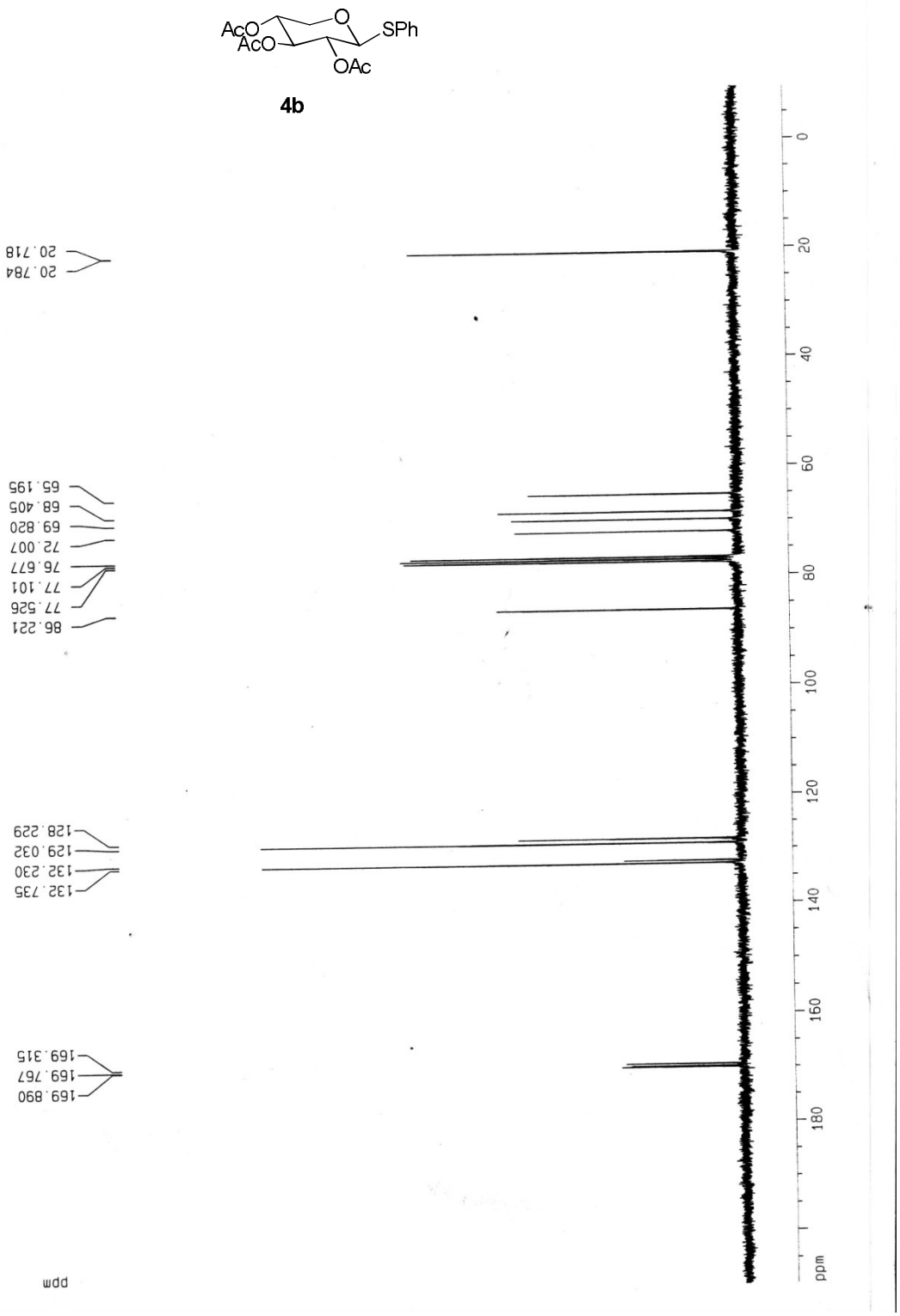
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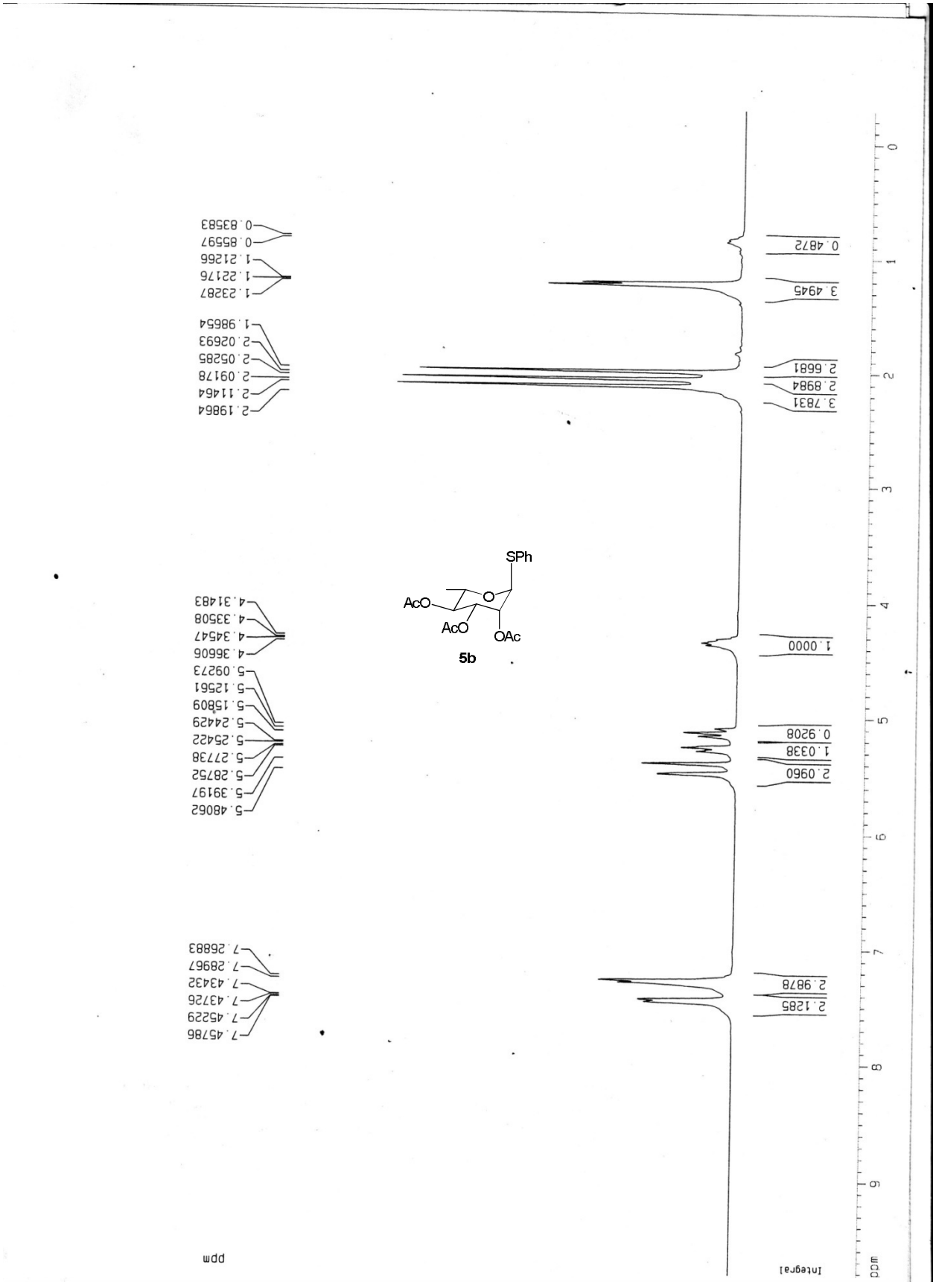
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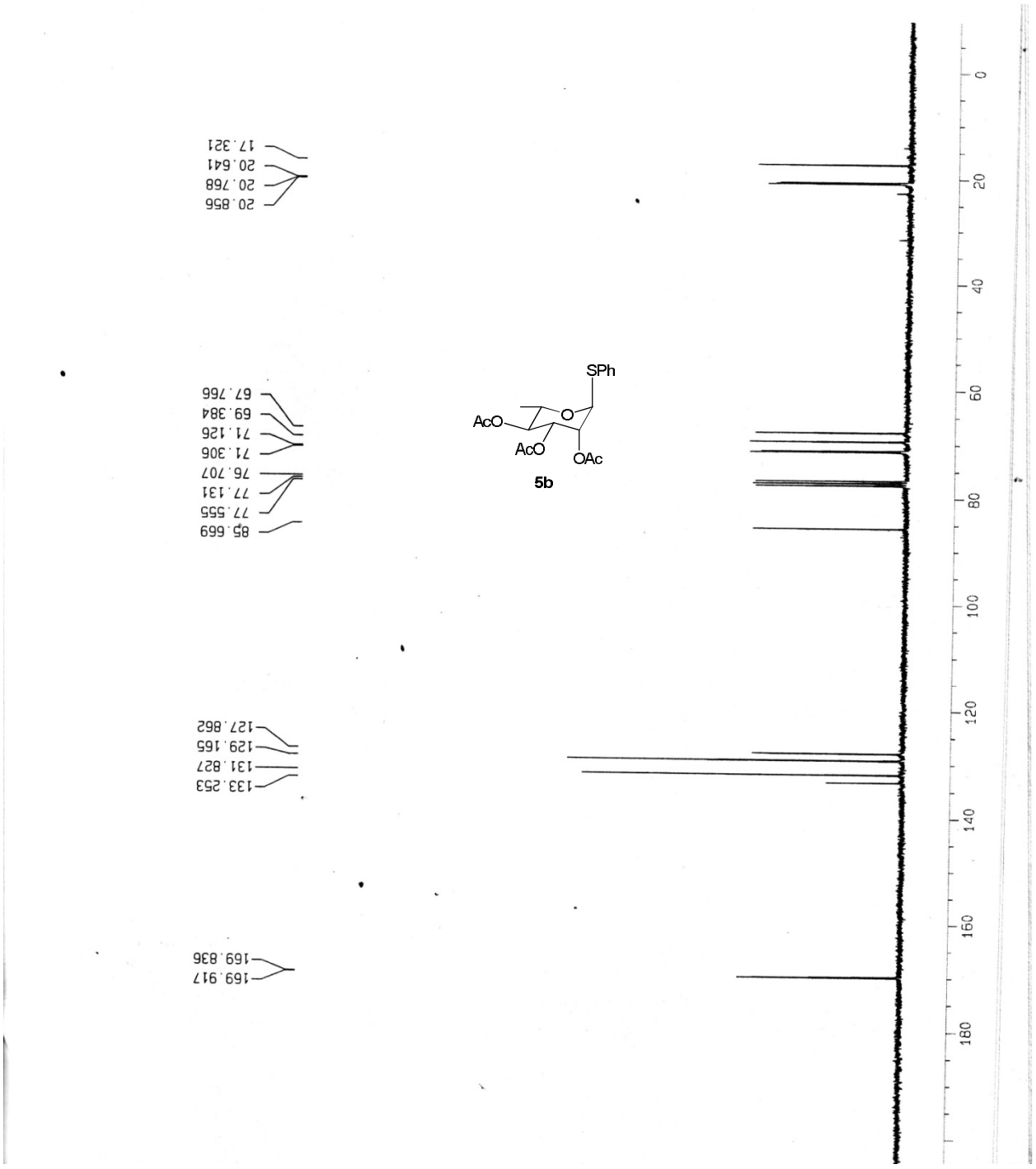
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SPh
AcO
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OMP
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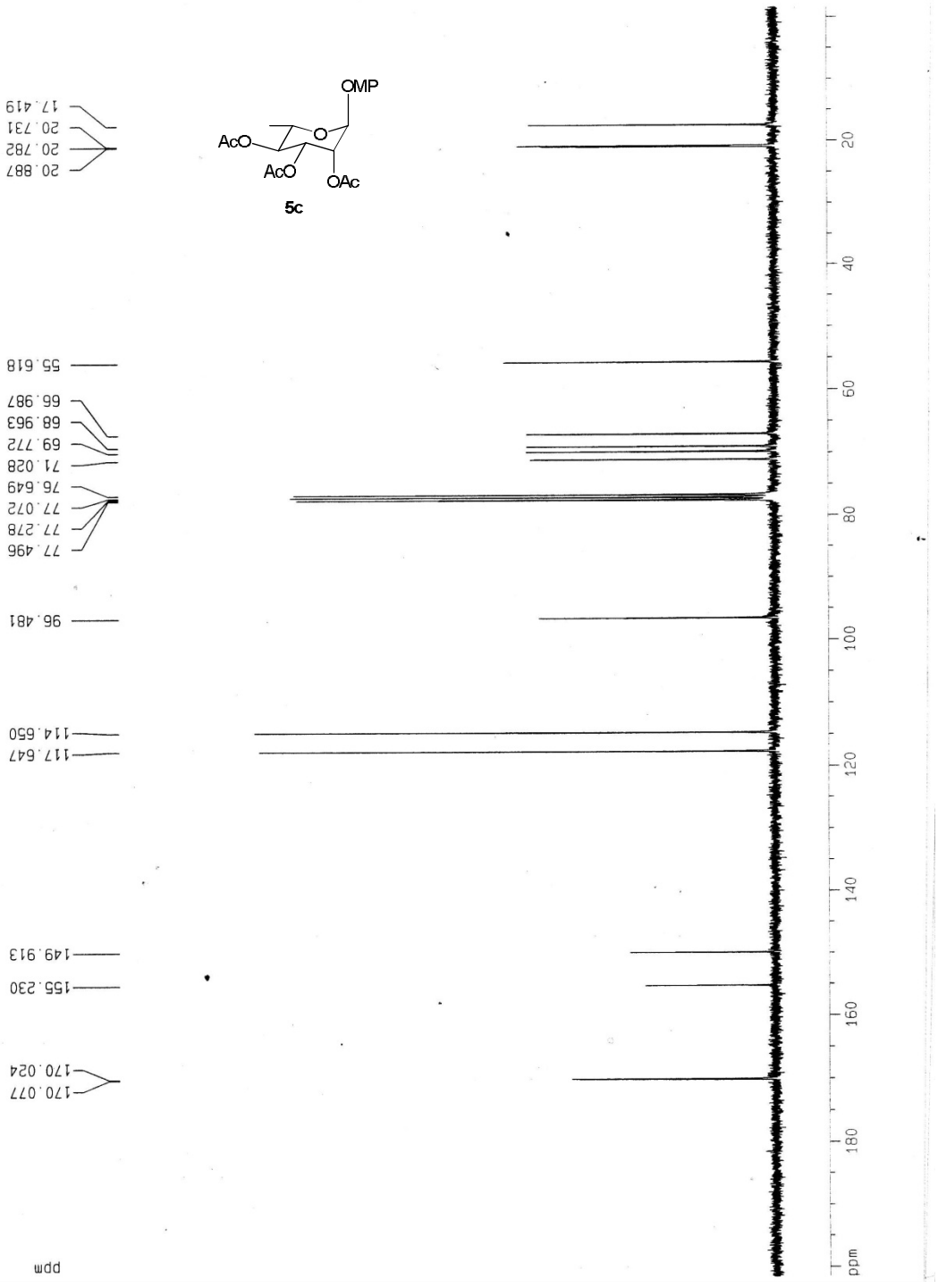
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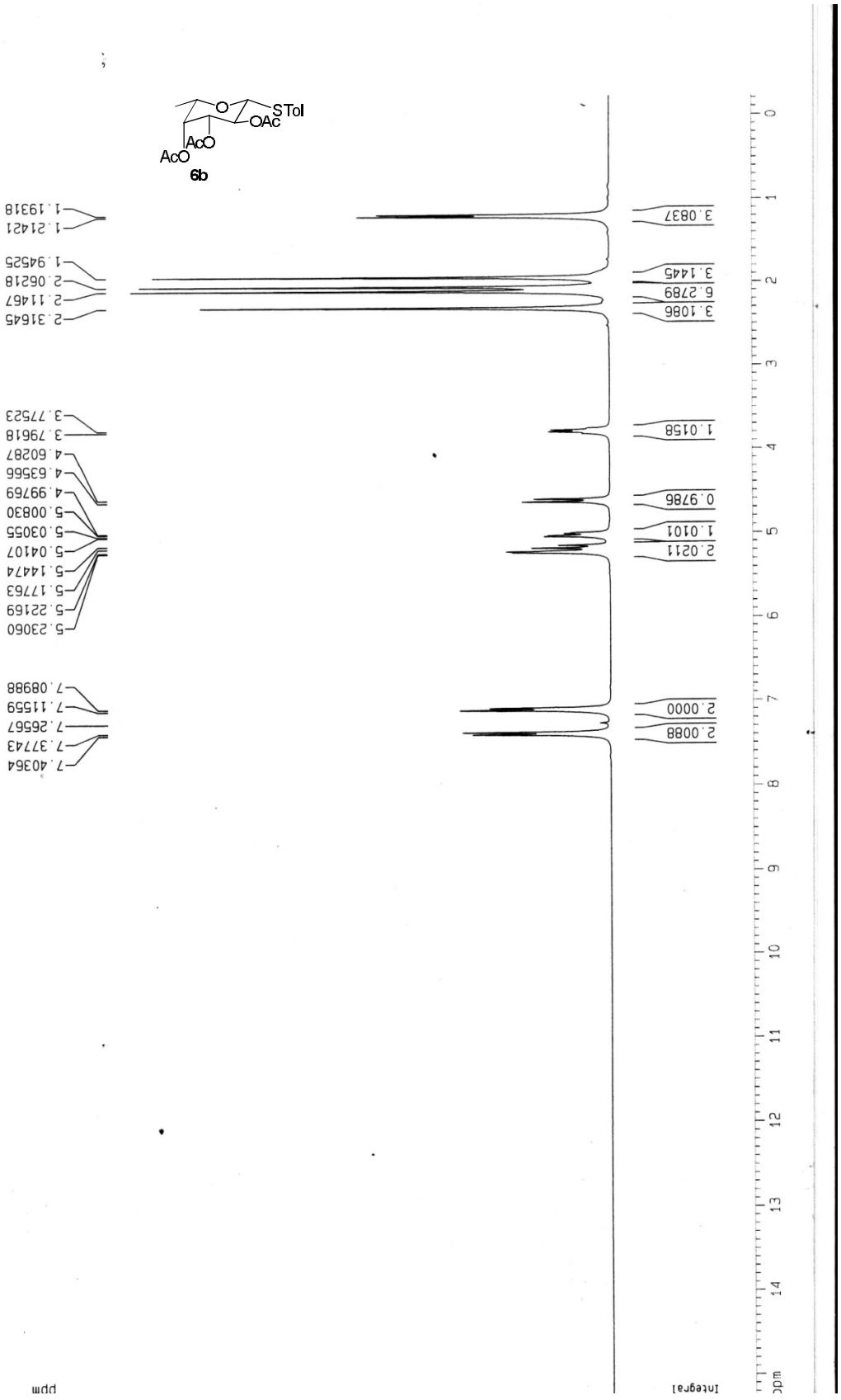
STol
OAc
AcO
AcO
6b
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2.11467
2.31645
3.77523
3.79618
4.60287
4.63566
4.99769
5.00830
5.03055
5.04107
5.14474
5.17763
5.22169
5.23060
7.08988
7.11559
7.26567
7.37743
7.40364
3.0837
3.1445
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3.1086
1.0158
0.9786
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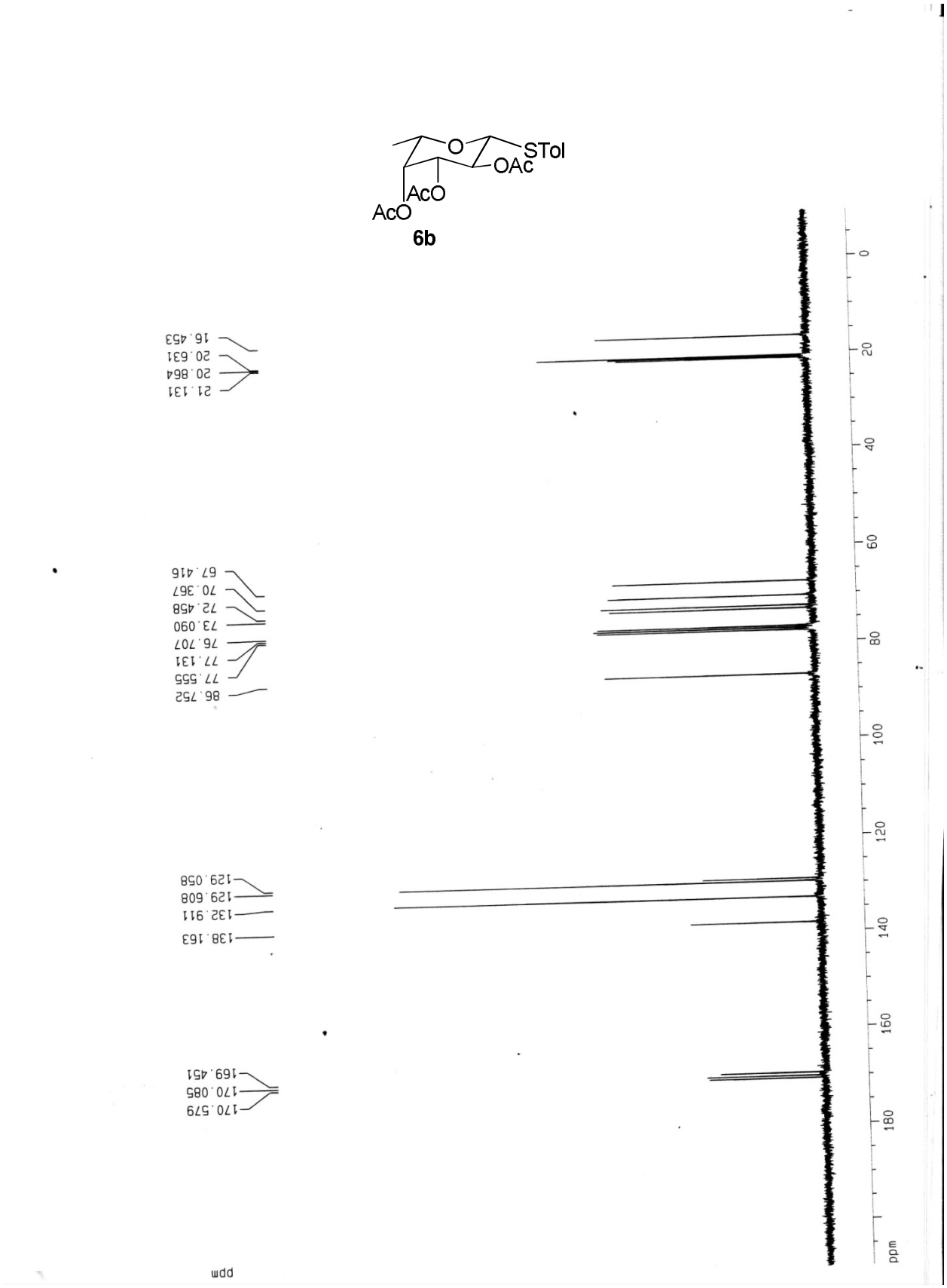
2.0211
2.0000
2.0088
ppm
Integral


O
STol
OAc
AcO
AcO
6b
16.453
20.631
20.864
21.131
67.416
70.367
72.458
73.090
76.707
77.131
77.555
86.752
129.058
129.608
132.911
138.163
169.451
170.085
170.579
ppm
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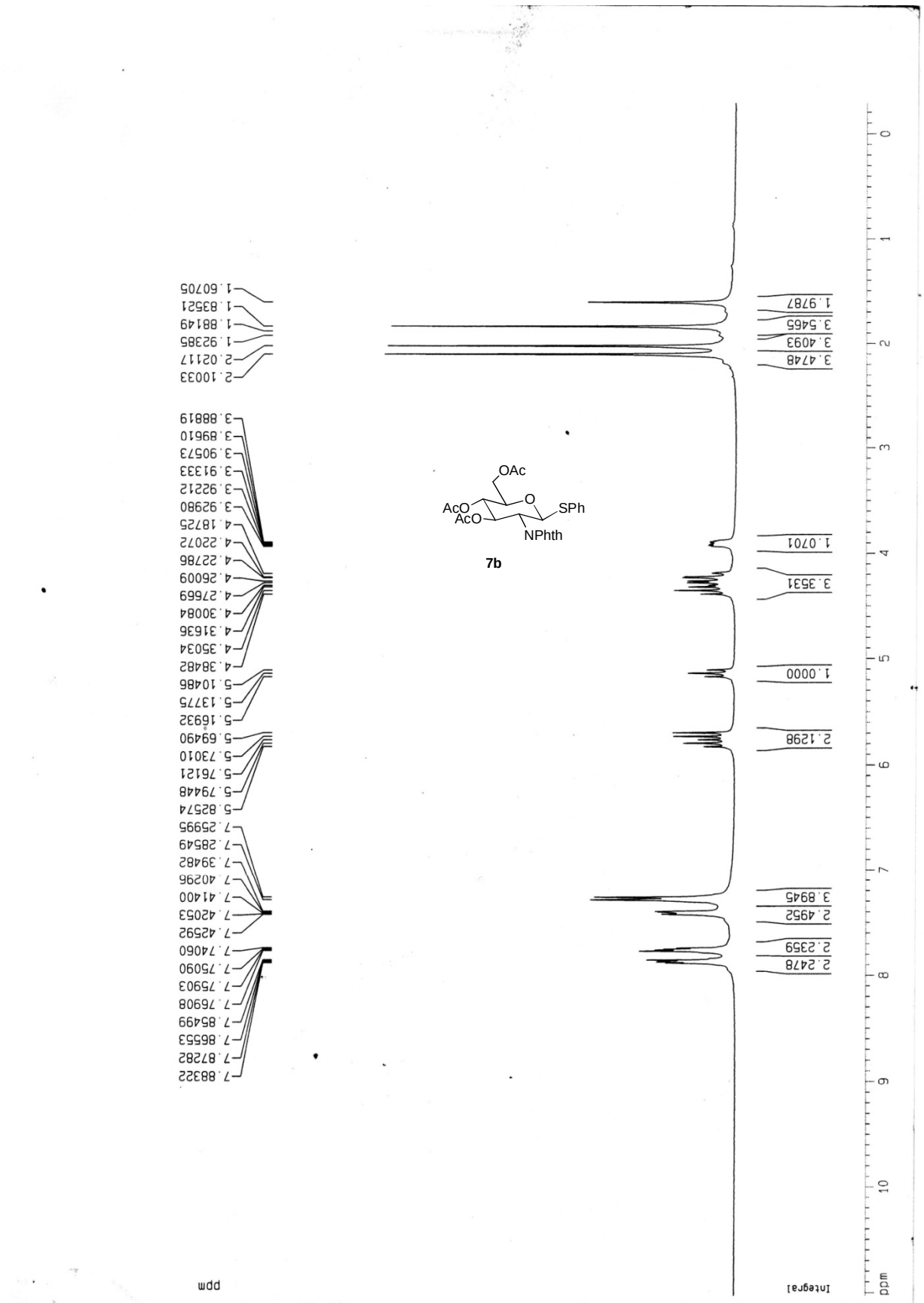
40
60
80
100
120
140
160
180
ppm


OAc
O
AcO
AcO
SPh
NPhth
7b
1.60705
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1.88149
1.92385
2.02117
2.10033
3.88819
3.89610
3.90573
3.91333
3.92212
3.92980
4.18725
4.22072
4.22786
4.26009
4.27669
4.30084
4.31636
4.35034
4.38482
5.10486
5.13775
5.16932
5.69490
5.73010
5.76121
5.79448
5.82574
7.25995
7.28549
7.39482
7.40296
7.41400
7.42053
7.42592
7.74060
7.75090
7.75903
7.76908
7.85499
7.86553
7.87282
7.88322
1.9787
3.5465
3.4093
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3.8945
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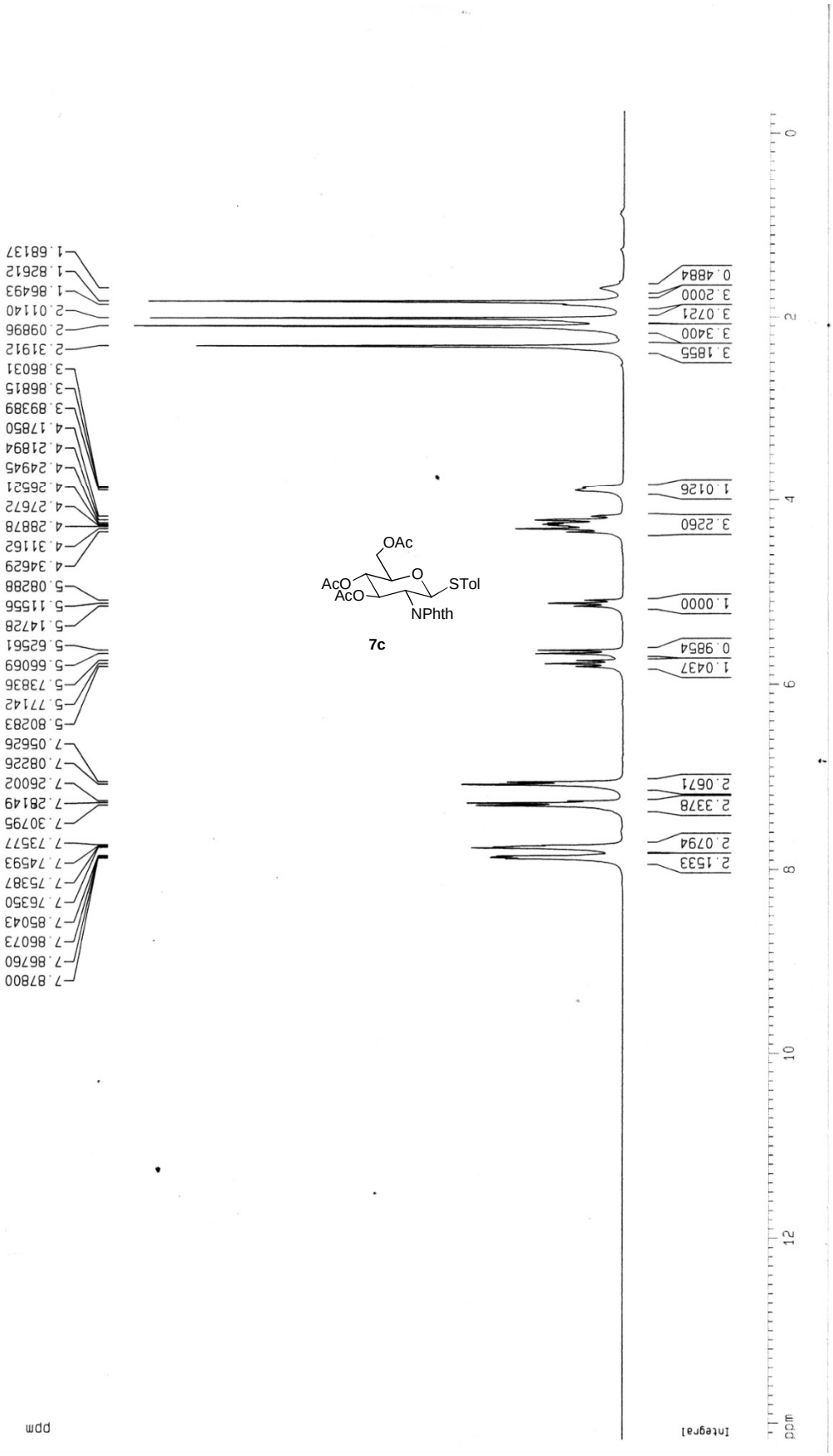
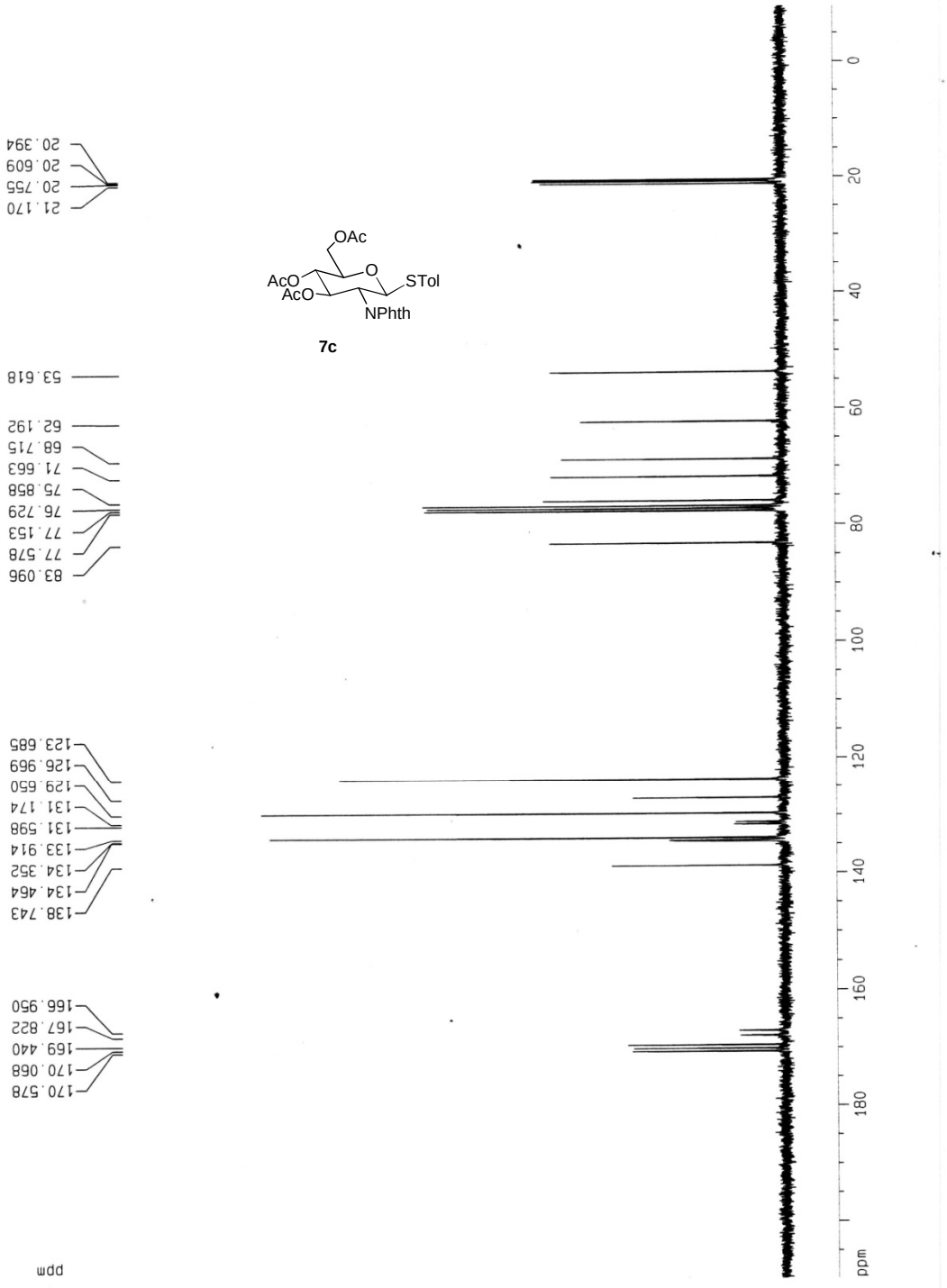
2.2478
ppm
Integral


OAc
AcO
AcO
O
STol
NPhth
7c
ppm
Integral


OAc
AcO
AcO
O
STol
NPhth
7c
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20.609
20.755
21.170
53.618
62.192
68.715
71.663
75.858
76.729
77.153
77.578
83.096
123.685
126.969
129.650
131.174
131.598
133.914
134.352
134.464
138.743
166.950
167.822
169.440
170.068
170.578
ppm
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40
60
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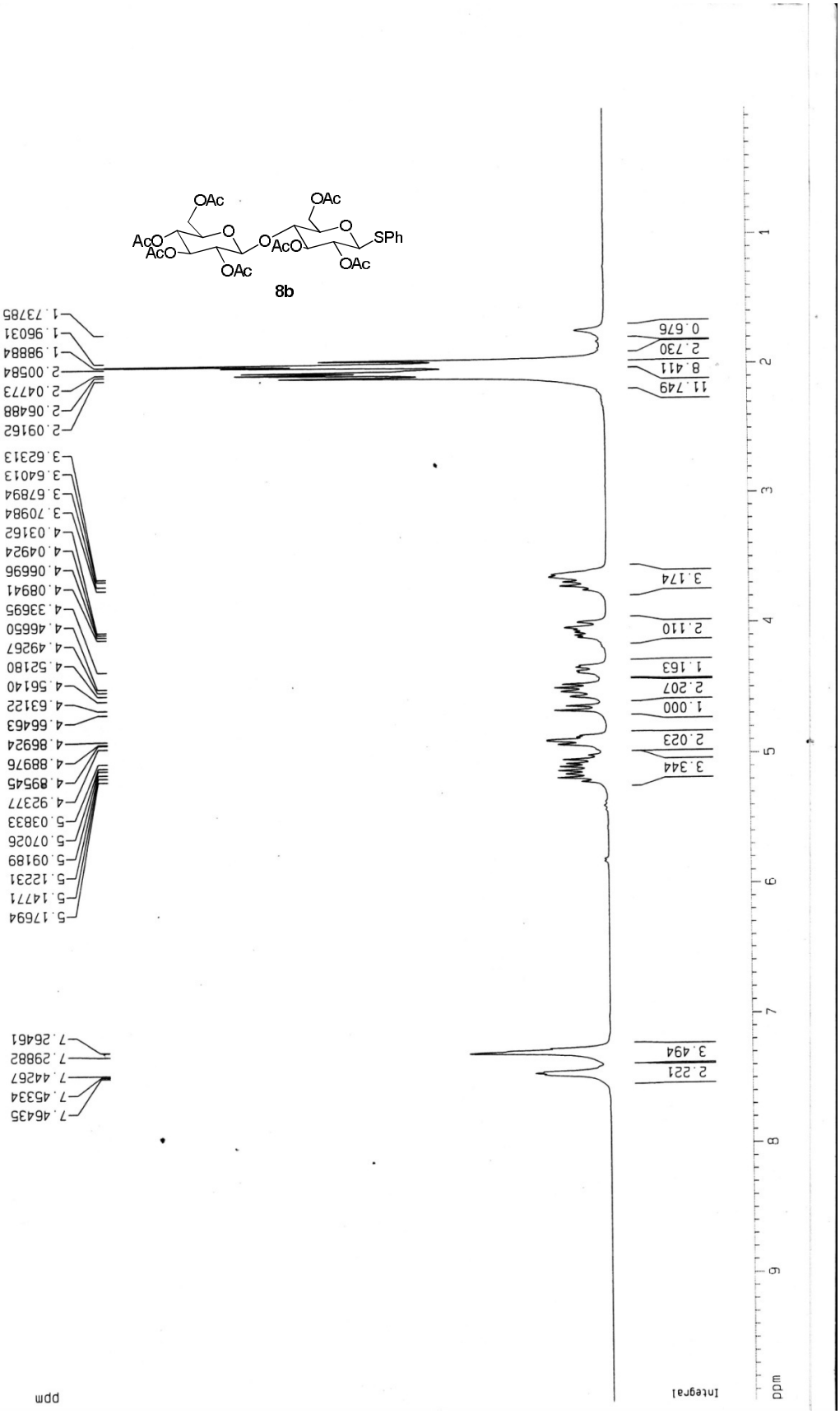
120
140
160
180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OAc
AcO
O
O
AcO
O
SPh
AcO
OAc
OAc
8b
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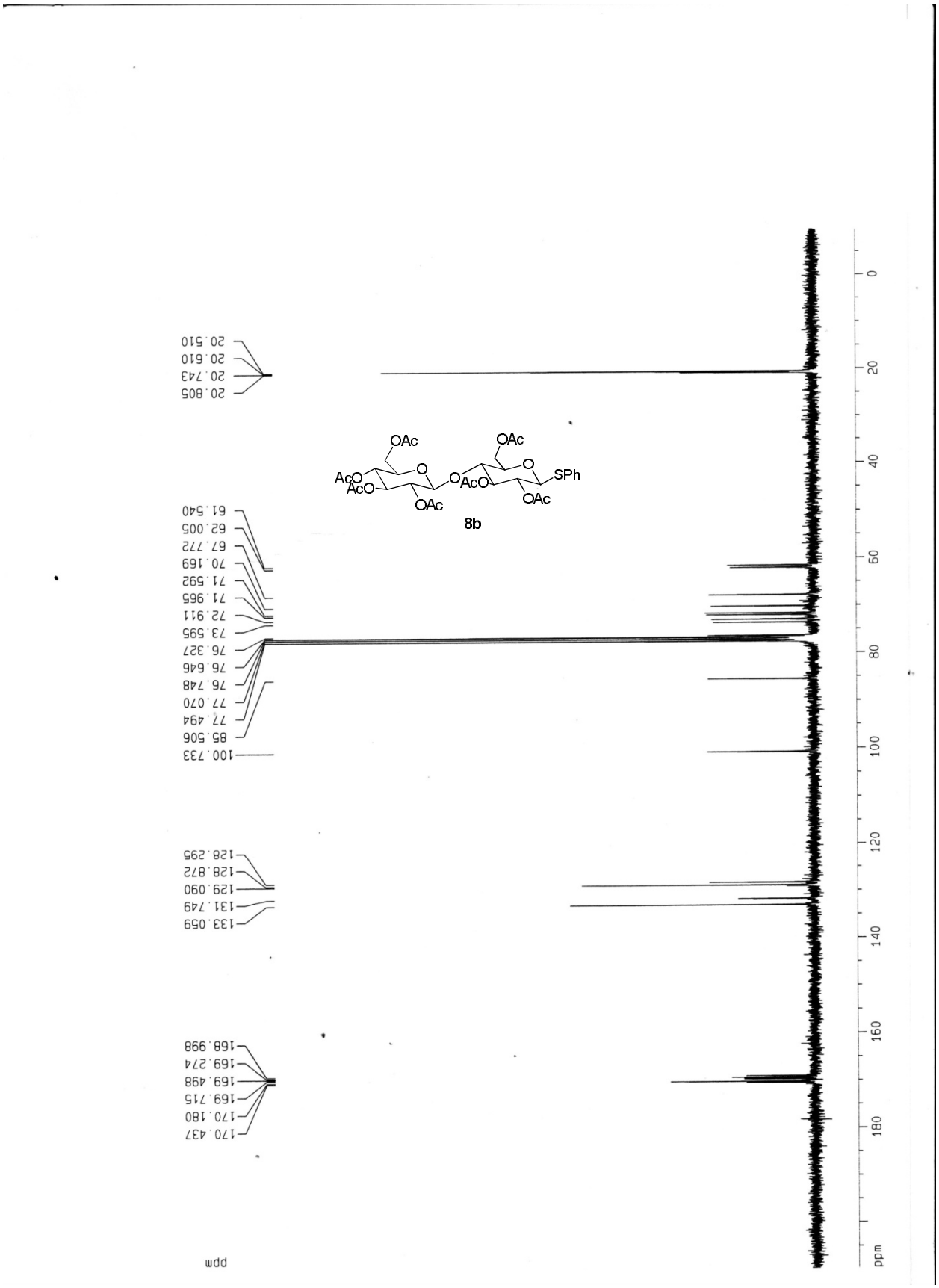
1.96031
1.98884
2.00584
2.04773
2.06488
2.09162
3.62313
3.64013
3.67894
3.70984
4.03162
4.04924
4.06696
4.08941
4.33695
4.46650
4.49267
4.52180
4.56140
4.63122
4.66463
4.86924
4.88976
4.89545
4.92377
5.03833
5.07026
5.09189
5.12231
5.14771
5.17694
7.26461
7.29882
7.44267
7.45334
7.46435
0.676
2.730
8.411
11.749
3.174
2.110
1.163
2.207
1.000
2.023
3.344
3.494
2.221
ppm
Integral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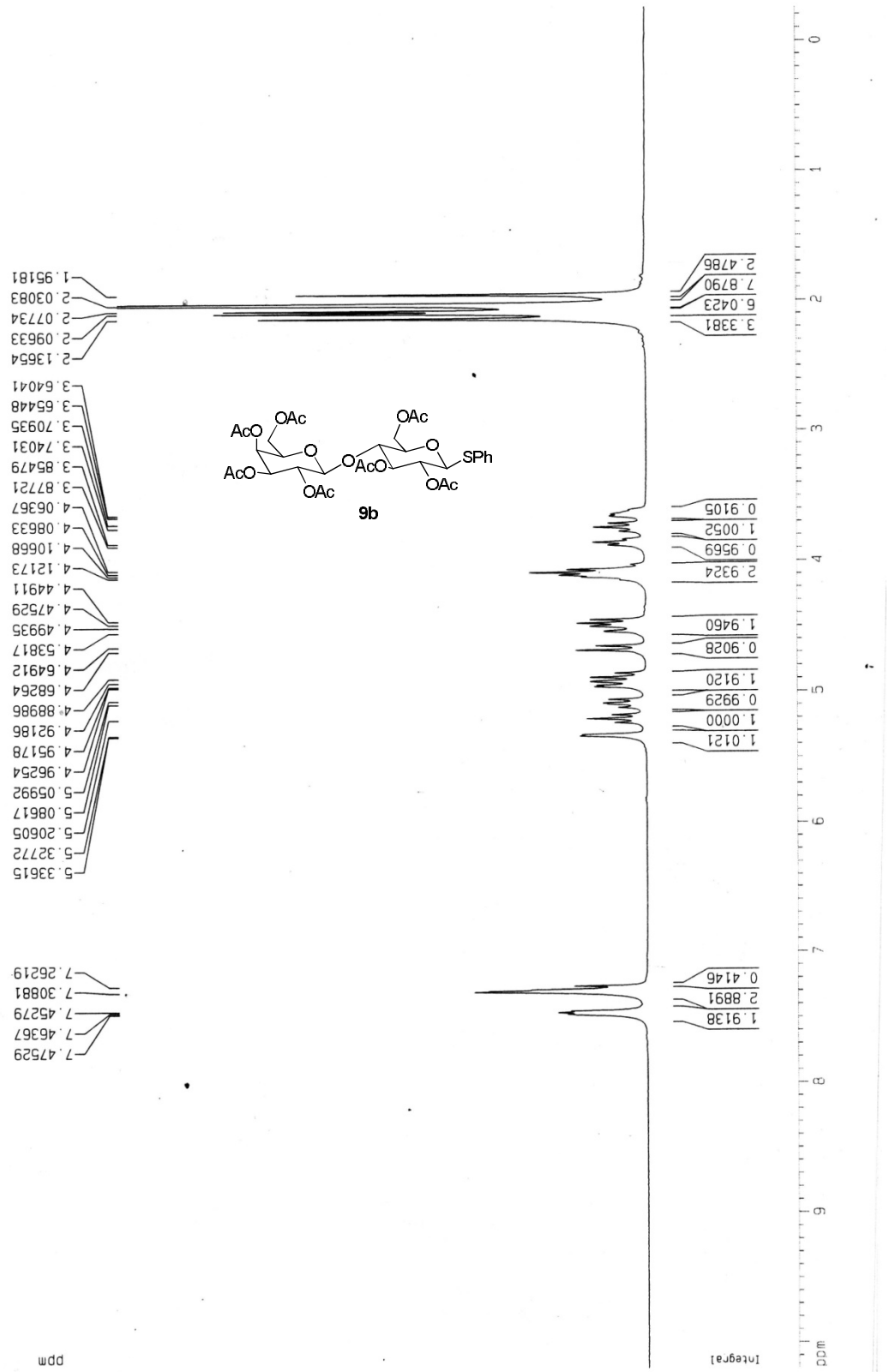
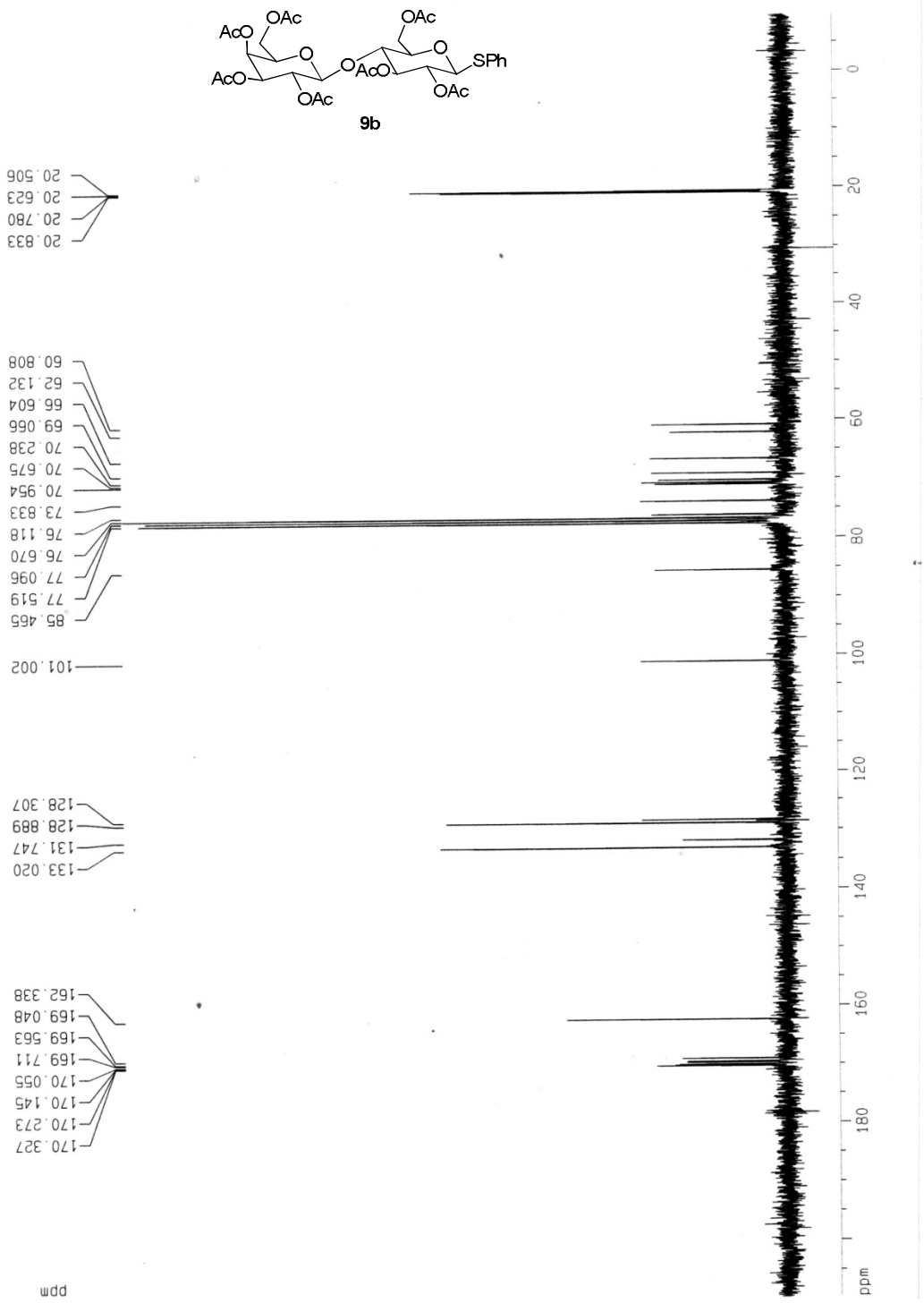
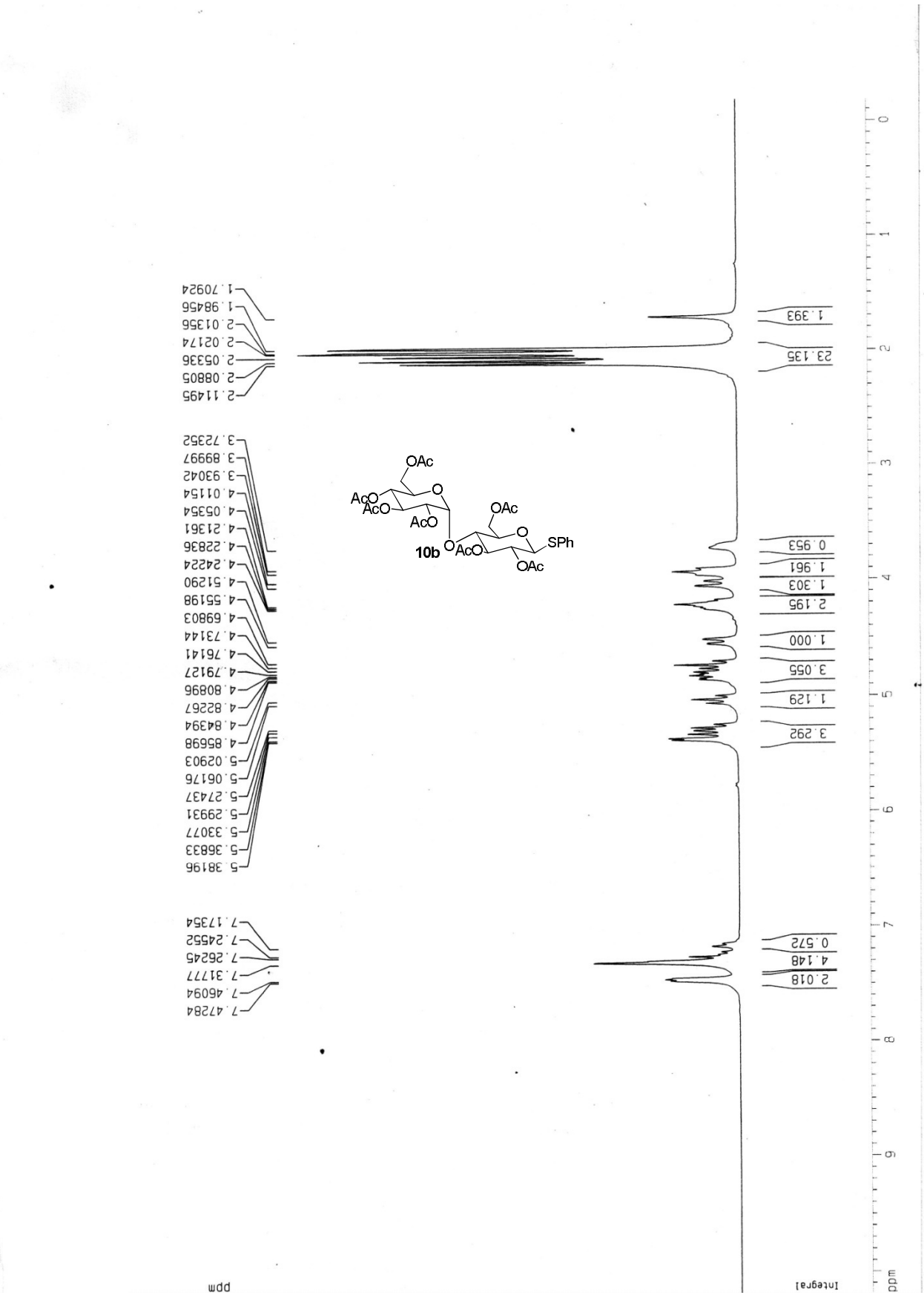
20.610
20.743
20.805
61.540
62.005
67.772
70.169
71.592
71.965
72.911
73.595
76.327
76.646
76.748
77.070
77.494
85.506
100.733
128.295
128.872
129.090
131.749
133.059
168.998
169.274
169.498
169.715
170.180
170.437
OAc
OAc
AcO
AcO
AcO
SPh
OAc
OAc
8b
ppm
0
20
40
60
80
100
120
140
160
180
ppm



AcO
OAc
O
AcO
OAc
O
AcO
OAc
O
SPh
OAc
9b
1.95181
2.03083
2.07734
2.09633
2.13654
3.64041
3.65448
3.70935
3.74031
3.85479
3.87721
4.06367
4.08633
4.10668
4.12173
4.44911
4.47529
4.49935
4.53817
4.64912
4.68264
4.88986
4.92186
4.95178
4.96254
5.05992
5.08617
5.20605
5.32772
5.33615
7.26219
7.30881
7.45279
7.46367
7.47529
2.4786
7.8790
6.0423
3.3381
0.9105
1.0052
0.9569
2.9324
1.9460
0.9028
1.9120
0.9929
1.0000
1.0121
0.4146
2.8891
1.9138
ppm
Integral


AcO
OAc
O
AcO
OAc
O
AcO
OAc
O
SPh
OAc
9b
20.506
20.623
20.780
20.833
60.808
62.132
66.604
69.066
70.238
70.675
70.954
73.833
76.118
76.670
77.096
77.519
85.465
101.002
128.307
128.889
131.747
133.020
162.338
169.048
169.563
169.711
170.055
170.145
170.273
170.327
ppm



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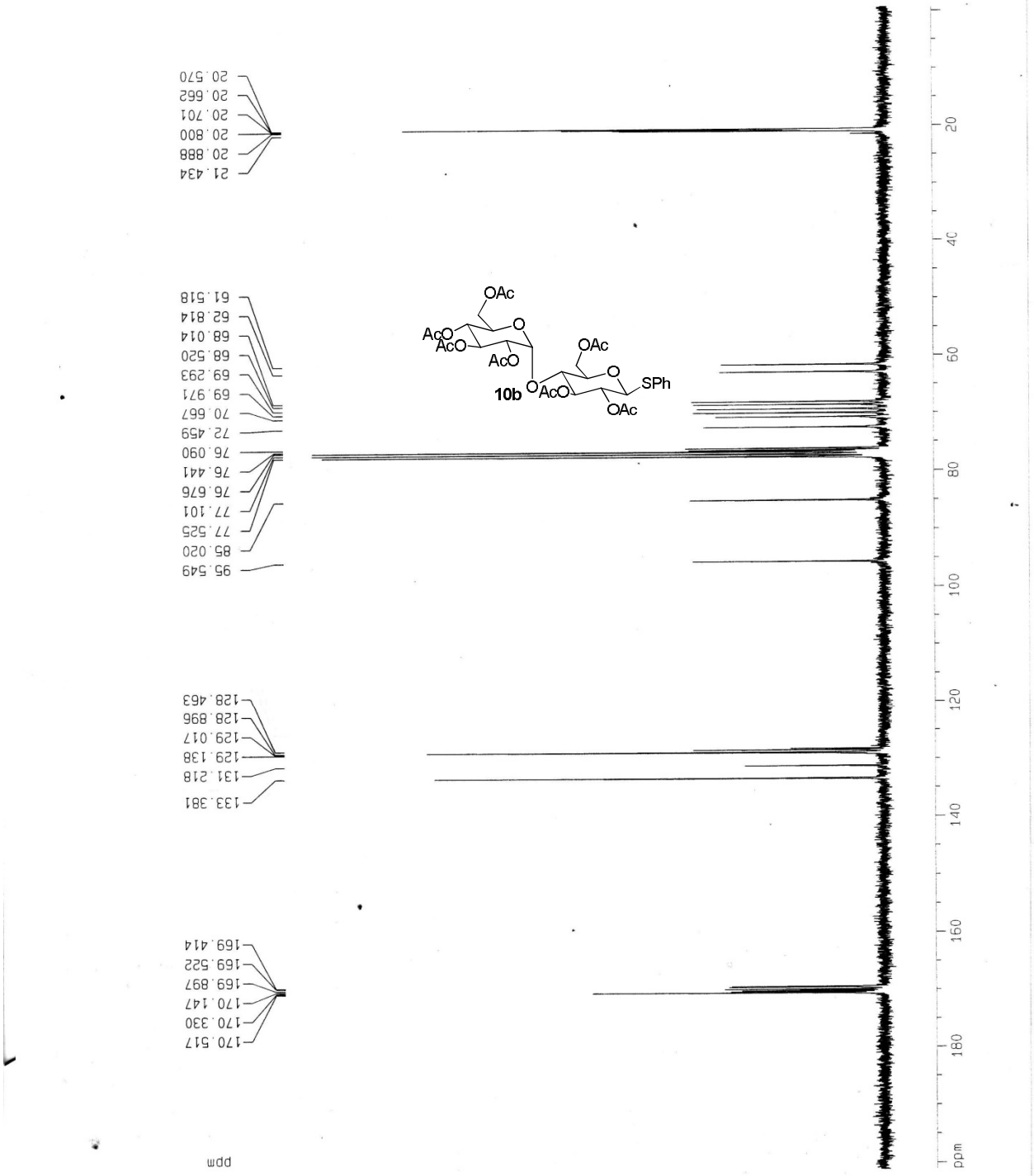
1.98456
2.01356
2.02174
2.05336
2.08805
2.11495
3.72352
3.89997
3.93042
4.01154
4.05354
4.21361
4.22836
4.24224
4.51290
4.55198
4.69803
4.73144
4.76141
4.79127
4.80896
4.82267
4.84394
4.85698
5.02903
5.06176
5.27437
5.29931
5.33077
5.36833
5.38196
7.17354
7.24552
7.26245
7.31777
7.46094
7.47284
OAc
AcO
AcO
AcO
O
OAc
O
SPh
10b
O
AcO
OAc
1.393
23.135
0.953
1.961
1.303
2.195
1.000
3.055
1.129
3.292
0.572
4.148
2.018
ppm
Integral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20.701
20.800
20.888
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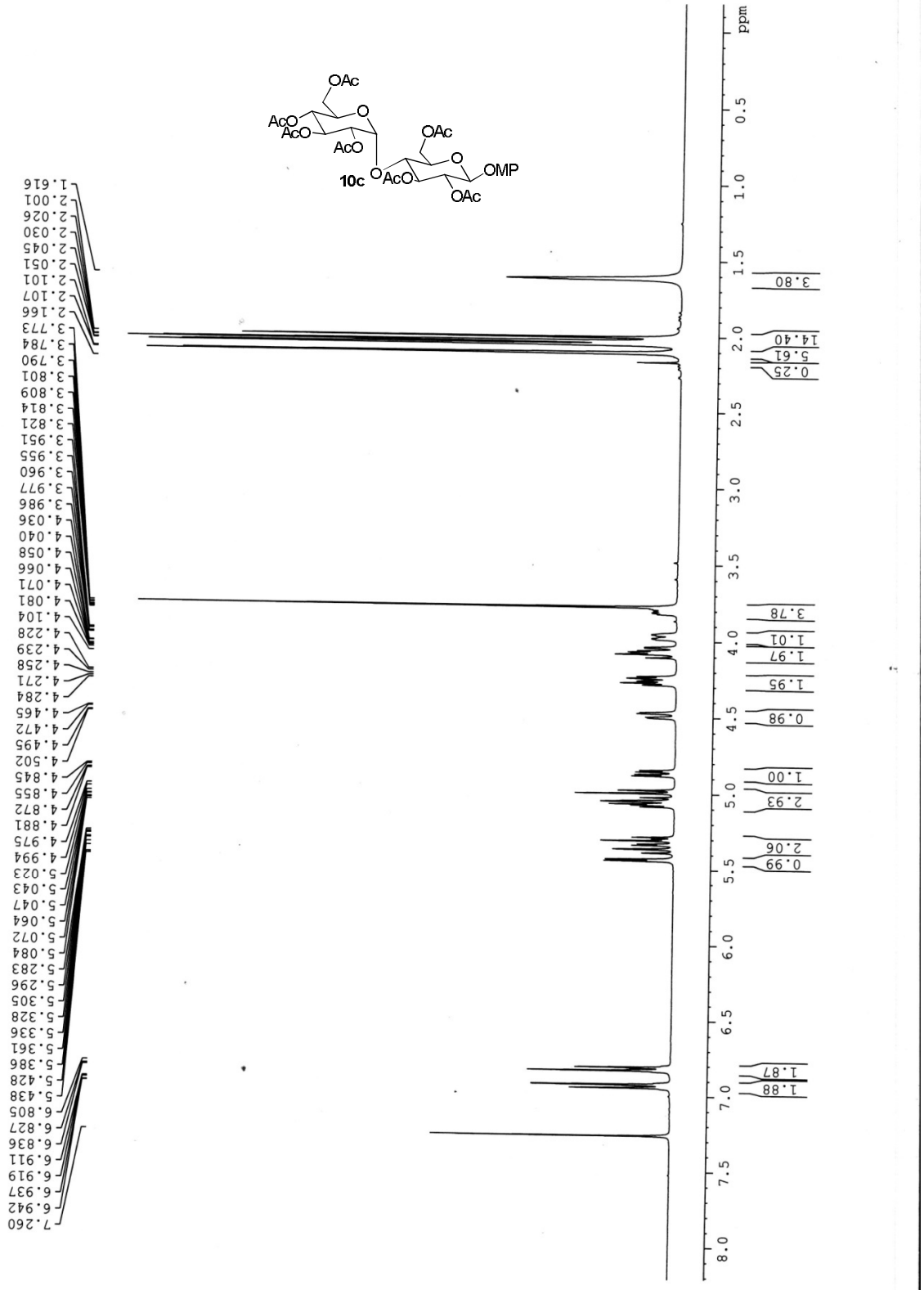
62.814
68.014
68.520
69.293
69.971
70.667
72.459
76.090
76.441
76.676
77.101
77.525
85.020
95.549
128.463
128.896
129.017
129.138
131.218
133.381
169.414
169.522
169.897
170.147
170.330
170.517
OAc
AcO
AcO
AcO
O
OAc
O
SPh
10b
AcO
OAc
ppm
20
40
60
80
100
120
140
160
180


OAc
AcO
AcO
AcO
O
OAc
O
OMP
10c
AcO
OAc
ppm

Ph
O
O
HO
O
SPh
OH
11b
1.7255
0.4115
1.2199
1.1568
0.5343
0.5212
0.5318
0.5880
0.5451
1.2115
0.6986
1.0791
1.0000
2.1723
3.4418
3.4732
3.5033
3.5162
3.5323
3.5384
3.7533
3.7698
3.7872
3.8132
3.8223
3.8543
3.8818
4.3644
4.3785
4.3995
4.4055
4.4130
4.6263
4.6586
5.5364
7.2630
7.3362
7.3474
7.3569
7.3634
7.3715
7.3753
7.3786
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7.4849
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7.5433
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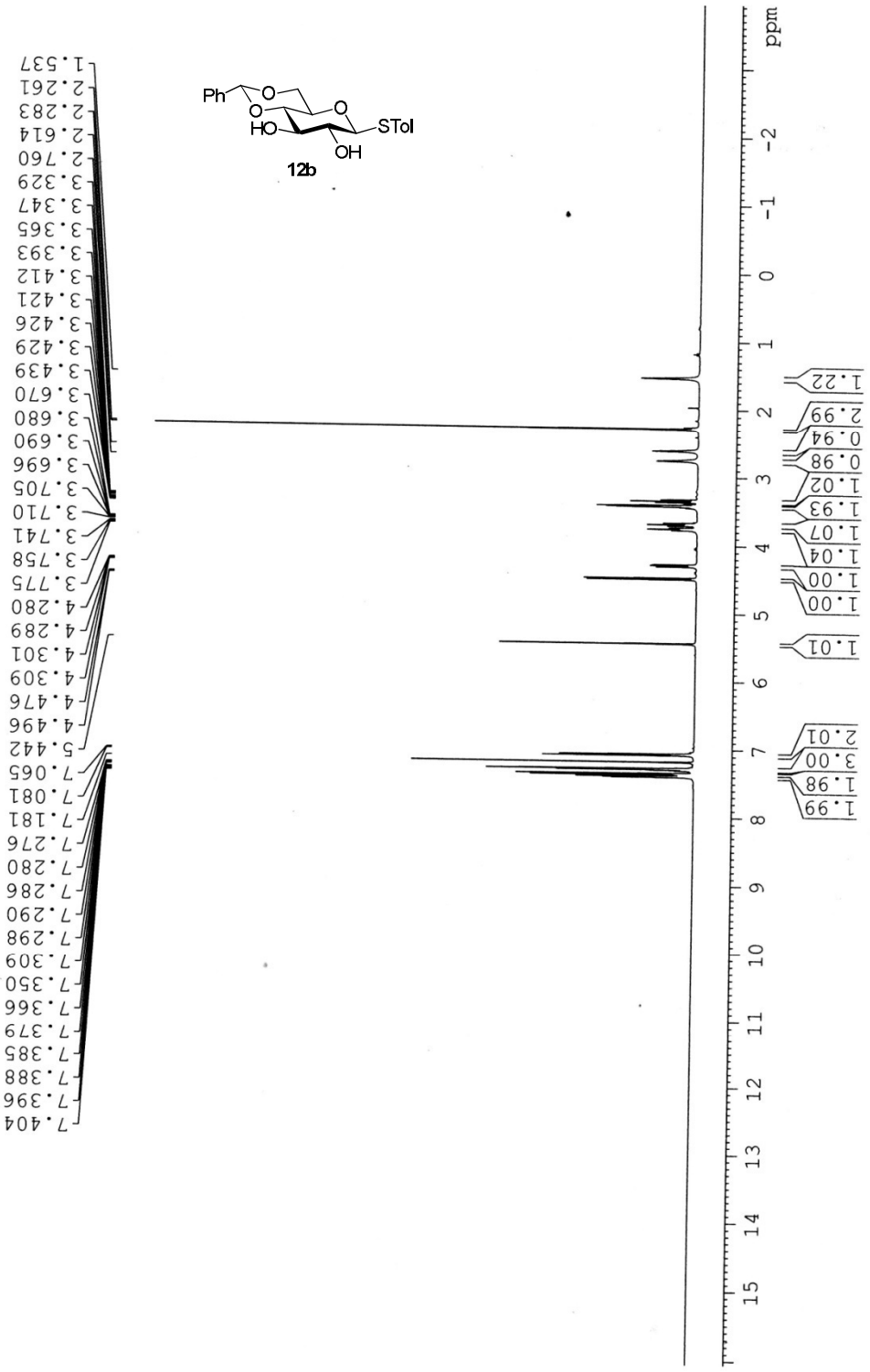
7.5550
ppm
Integral


Ph
O
O
O
HO
STol
OH
12b
ppm
1.537
2.261
2.283
2.614
2.760
3.329
3.347
3.365
3.393
3.412
3.421
3.426
3.429
3.439
3.670
3.680
3.690
3.696
3.705
3.710
3.741
3.758
3.775
4.280
4.289
4.301
4.309
4.476
4.496
5.442
7.065
7.081
7.181
7.276
7.280
7.286
7.290
7.298
7.309
7.350
7.366
7.379
7.385
7.388
7.396
7.404
1.99
1.98
3.00
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1.00
1.00
1.04
1.07
1.93
1.02
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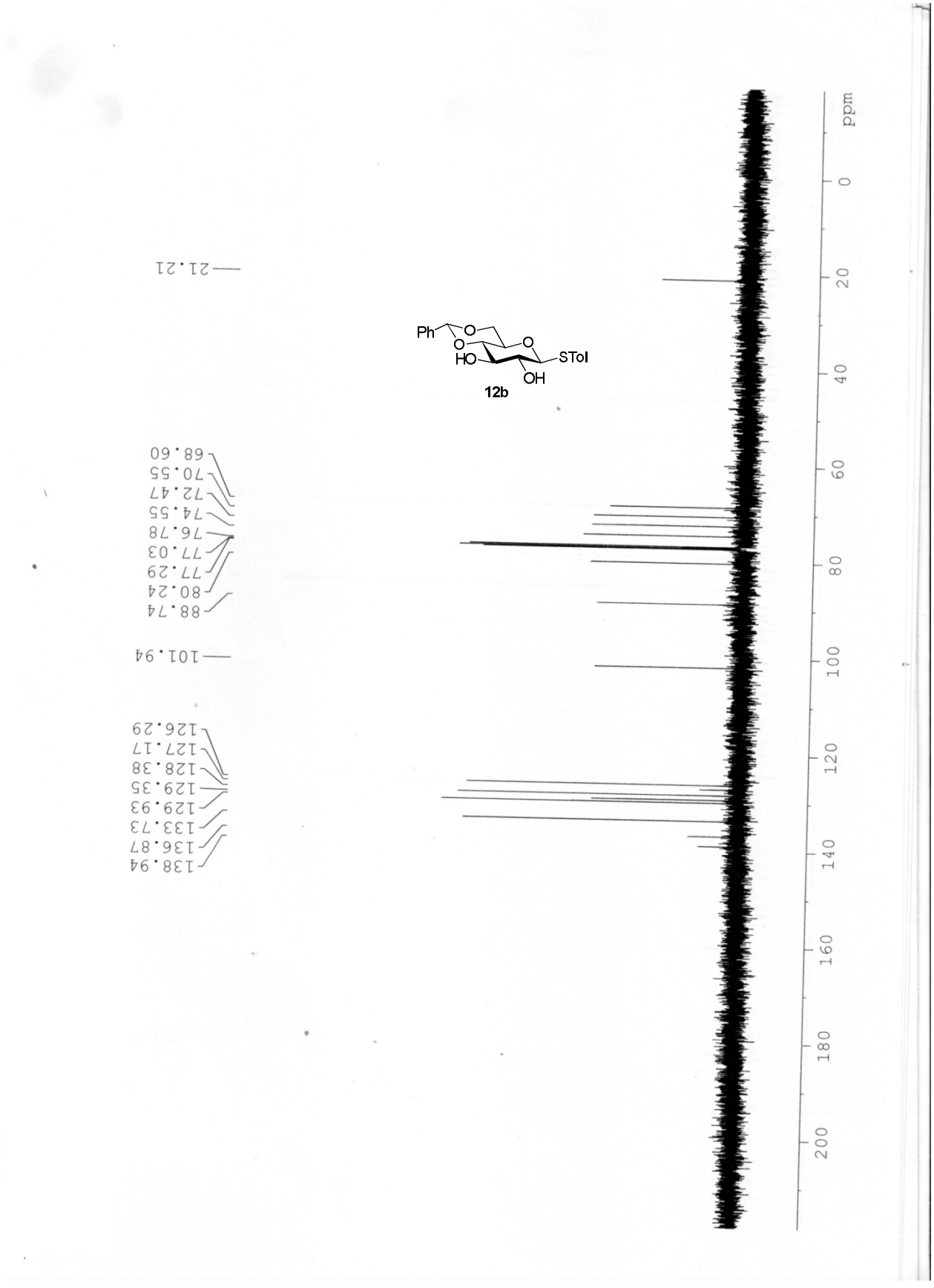
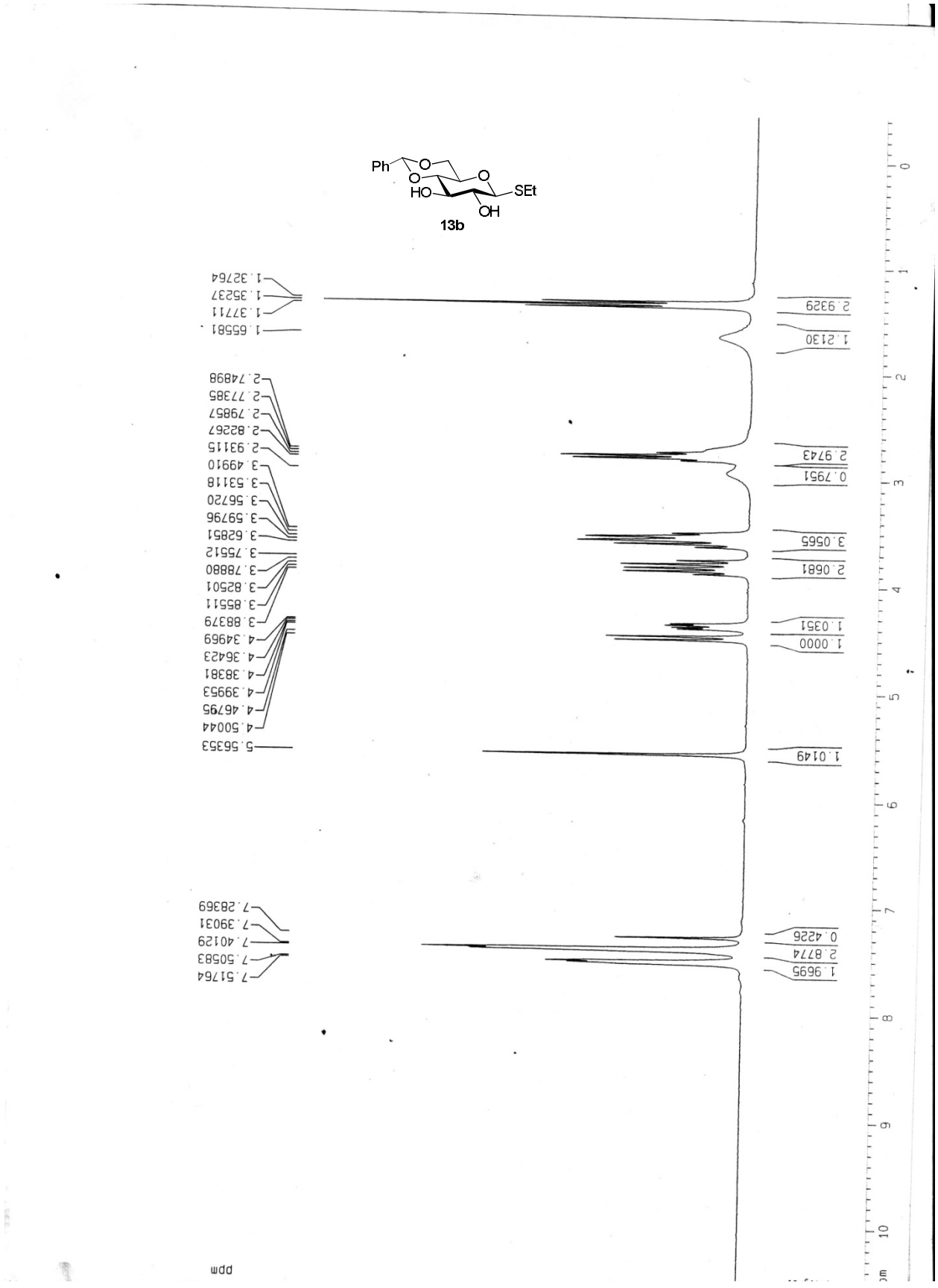
2.99
1.22


21.21
68.60
70.55
72.47
74.55
76.78
77.03
77.29
80.24
88.74
101.94
126.29
127.17
128.38
129.35
129.93
133.73
136.87
138.94
Ph
O
O
O
HO
OH
STol
12b
ppm
0
20
40
60
80
100
120
140
160
180
200


Ph
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O
O
HO
SEt
OH
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1.65581
2.74898
2.77385
2.79857
2.82267
2.93115
3.49910
3.53118
3.56720
3.59796
3.62851
3.75512
3.78880
3.82501
3.85511
3.88379
4.34969
4.36423
4.38381
4.39953
4.46795
4.50044
5.56353
7.51764
7.50583
7.40129
7.39031
7.28369
2.9329
1.2130
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2.0681
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1.0000
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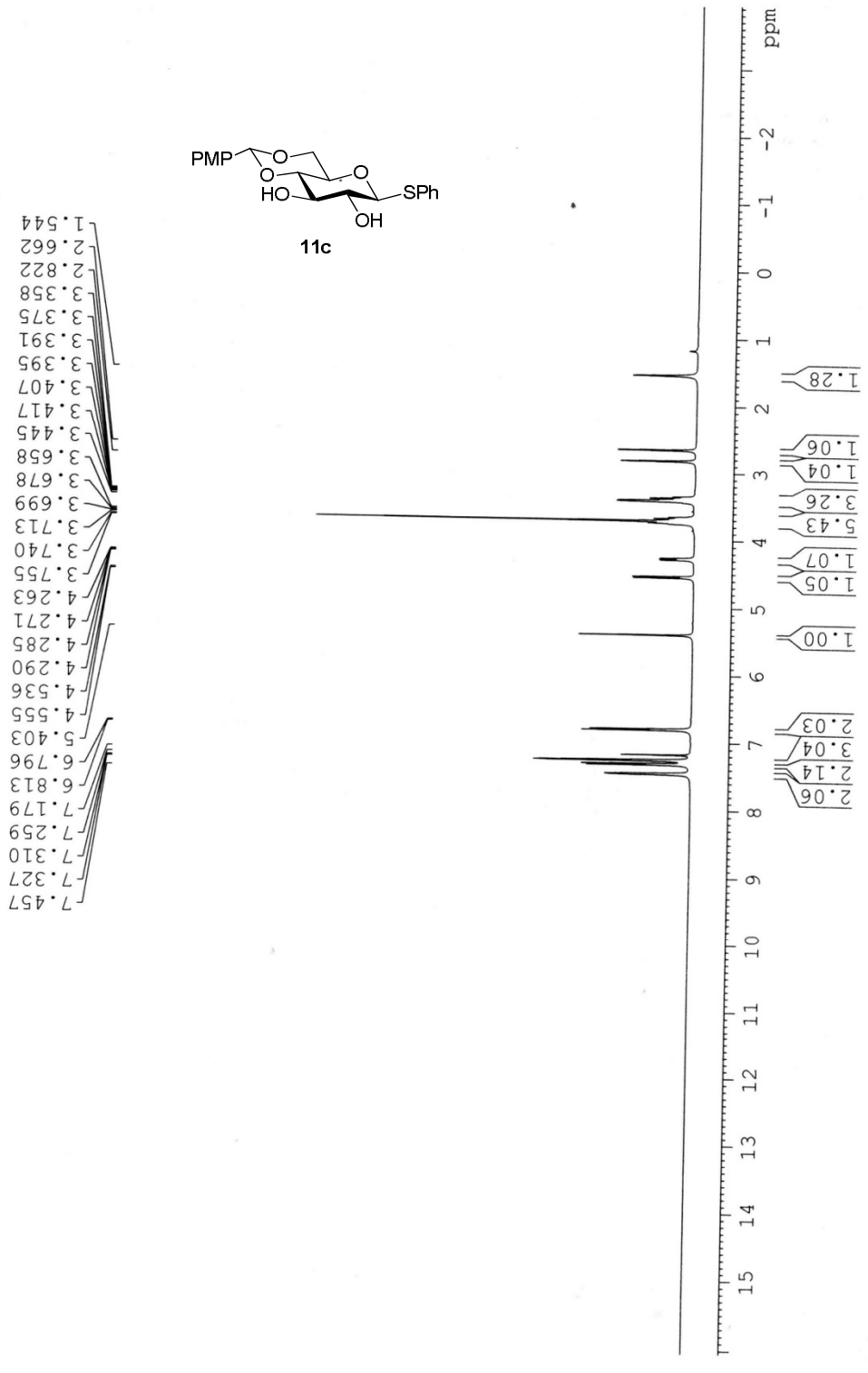
1.9695
ppm


PMP
O
O
O
HO
SPh
OH
11c
ppm
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3.358
3.375
3.391
3.395
3.407
3.417
3.445
3.658
3.678
3.699
3.713
3.740
3.755
4.263
4.271
4.285
4.290
4.536
4.555
5.403
6.796
6.813
7.179
7.259
7.310
7.327
7.457
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1.06
1.04
3.26
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1.07
1.05
1.00
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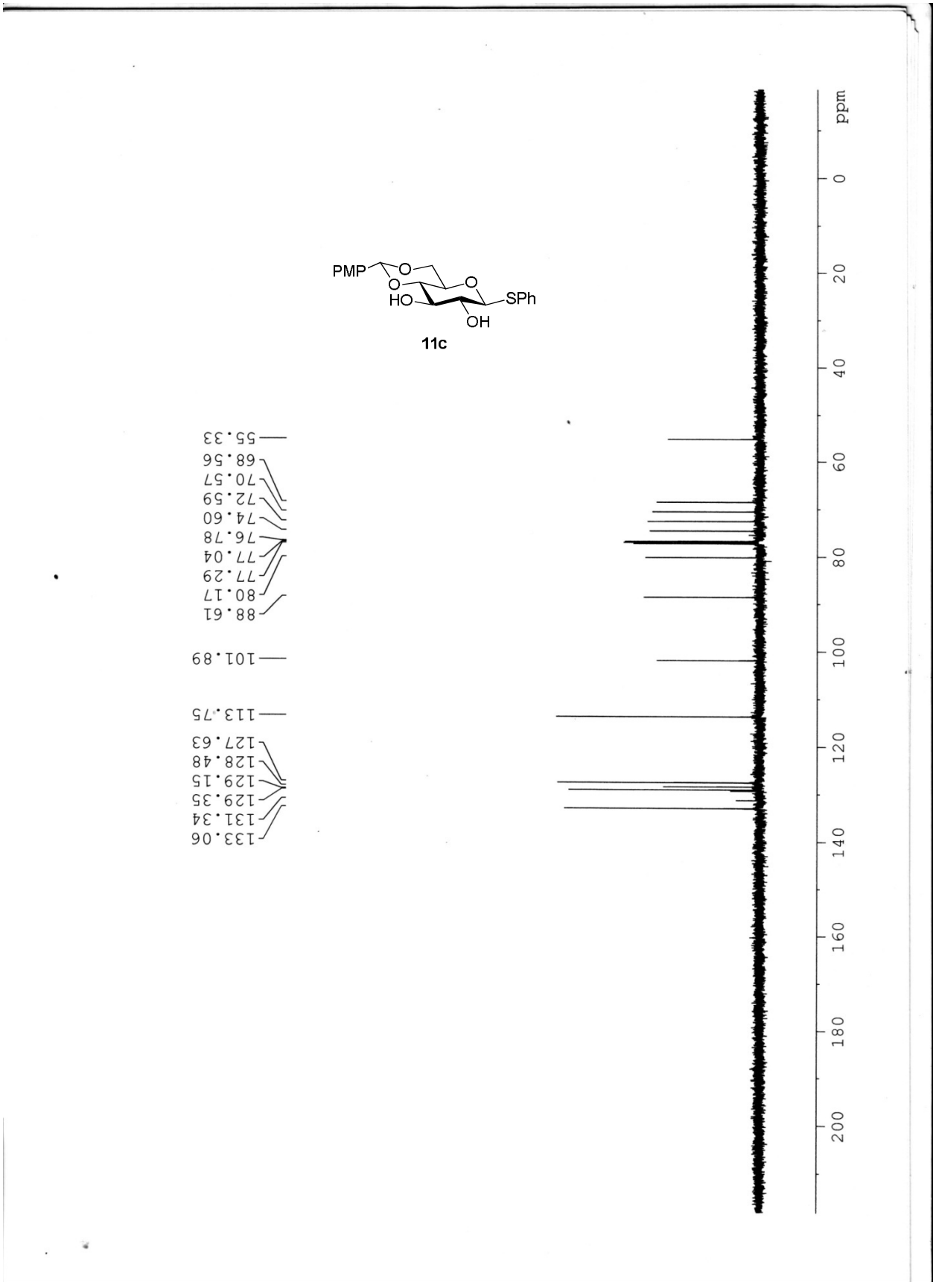
3.04
2.14
2.06


PMP
O
O
O
HO
SPh
OH
11c
55.33
68.56
70.57
72.59
74.60
76.78
77.04
77.29
80.17
88.61
101.89
113.75
127.63
128.48
129.15
129.35
131.34
133.06
ppm
0
20
40
60
80
100
120
140
160
180
200

Ph
O
O
O
HO
OMP
OH
14b
2.49597
3.36620
3.46330
3.49297
3.56974
3.70449
4.19591
4.21331
4.96301
4.98737
5.44075
5.45326
5.59789
6.85004
6.87834
6.98881
7.01692
7.37497
7.44947
0.2433
1.6981
0.5970
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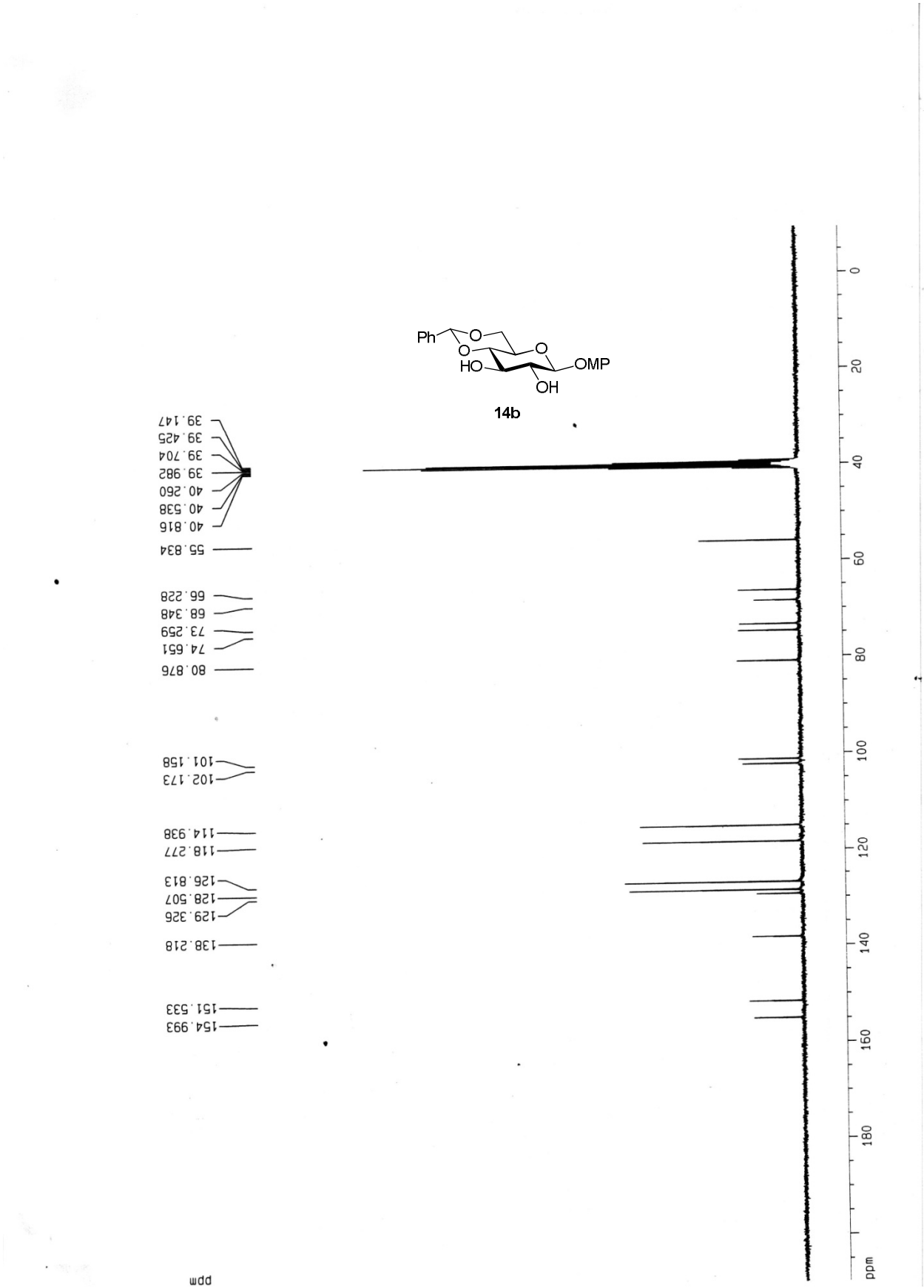
1.0000
2.5016
ppm
Integral


Ph
O
O
O
HO
OMP
OH
14b
39.147
39.425
39.704
39.982
40.260
40.538
40.816
55.834
66.228
68.348
73.259
74.651
80.876
101.158
102.173
114.938
118.277
126.813
128.507
129.326
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151.533
154.993
ppm
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20
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60
80
100
120
140
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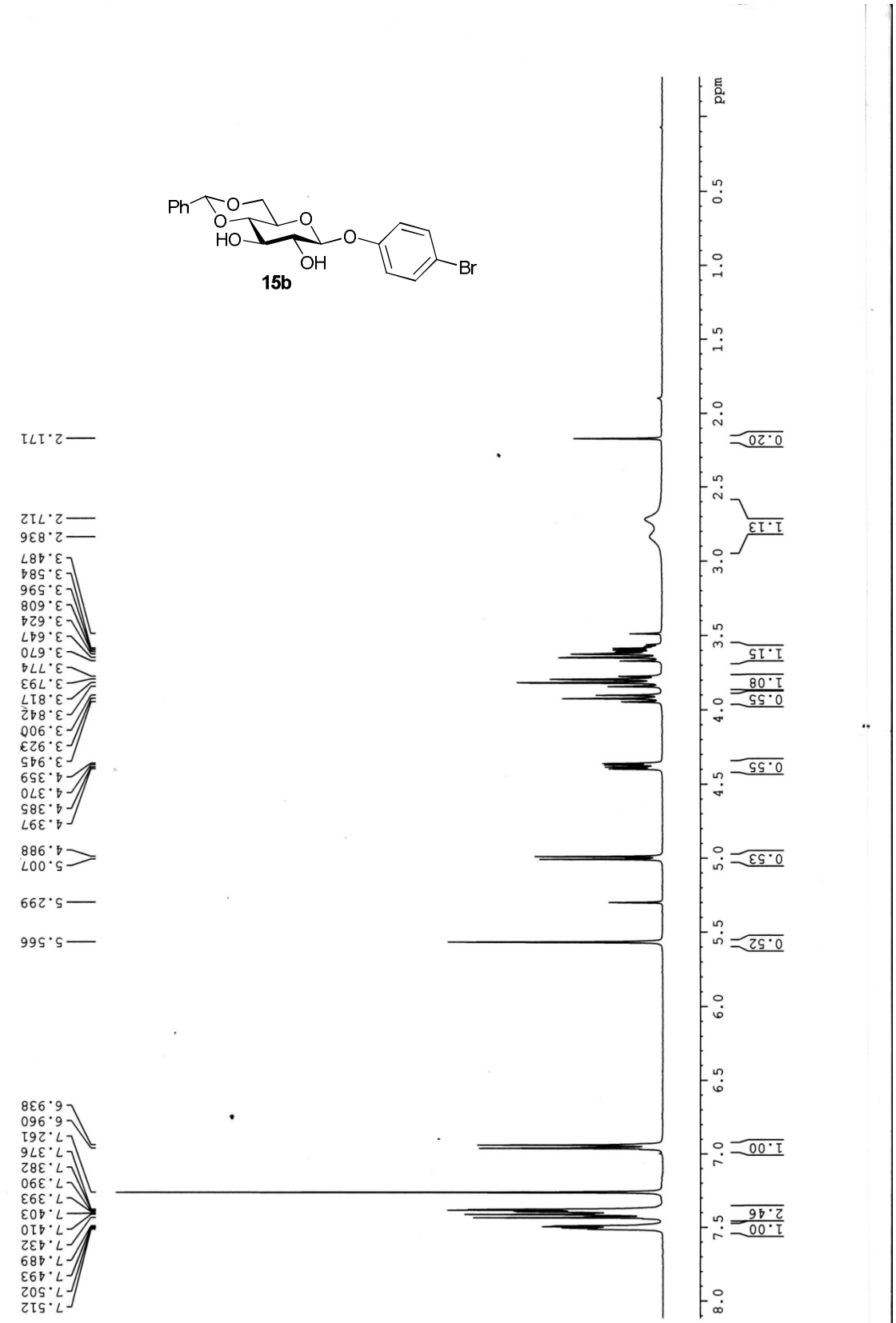
180
ppm


Ph
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O
O
HO
OH
O
Br
15b
ppm
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2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
0.20
1.13
1.15
1.08
0.55
0.55
0.53
0.52
1.00
1.00
2.46
2.171
2.712
2.836
3.487
3.584
3.596
3.608
3.624
3.647
3.670
3.774
3.793
3.817
3.842
3.905
3.923
3.945
4.359
4.370
4.385
4.397
4.988
5.007
5.299
5.566
6.938
6.960
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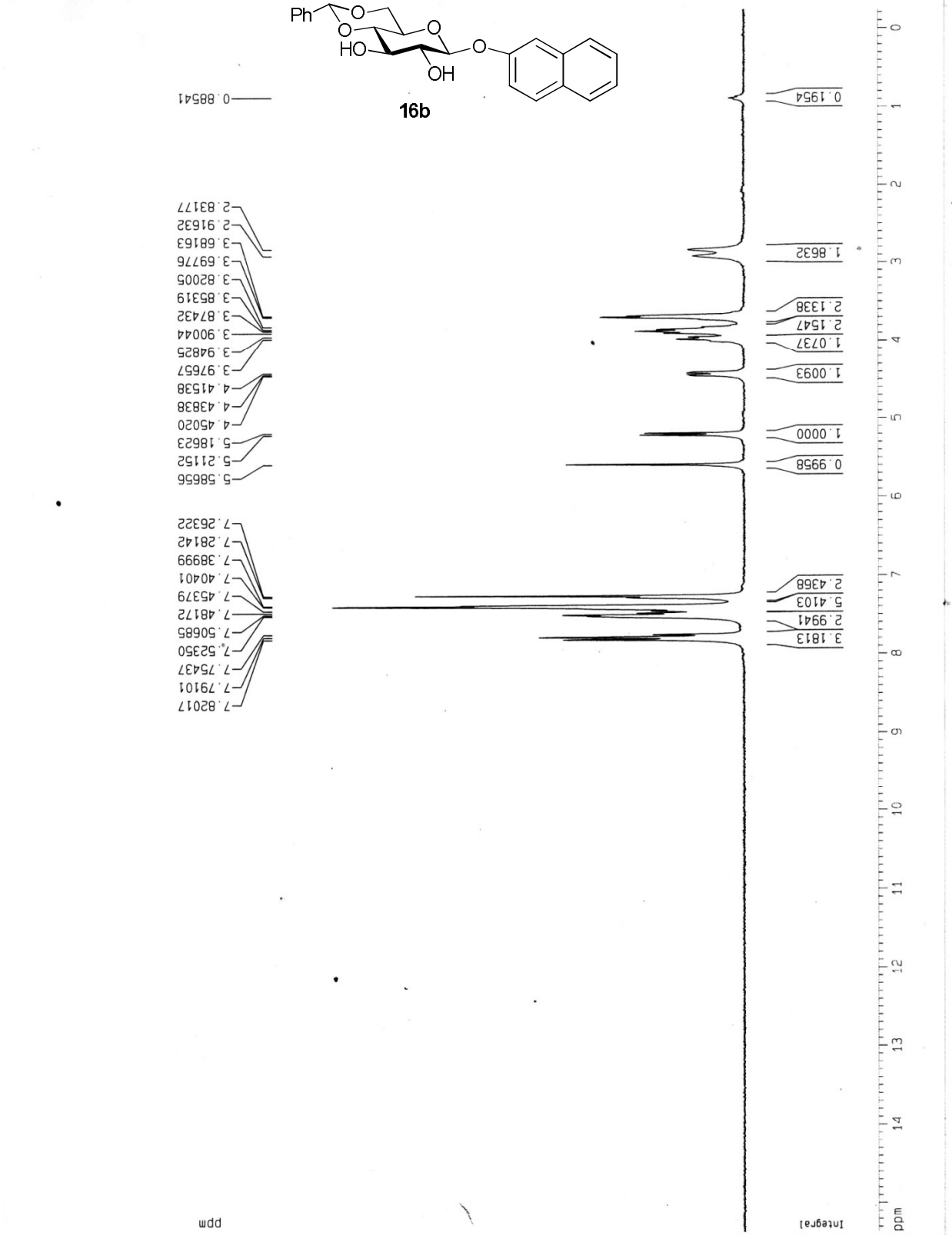
7.376
7.382
7.390
7.393
7.403
7.410
7.432
7.489
7.493
7.502
7.512


Ph
O
O
O
HO
OH
O
16b
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3.69776
3.82005
3.85319
3.87432
3.90044
3.94825
3.97657
4.41538
4.43838
4.45020
5.18623
5.21152
5.58656
7.26322
7.28142
7.38999
7.40401
7.45379
7.48172
7.50685
7.52350
7.75437
7.79101
7.82017
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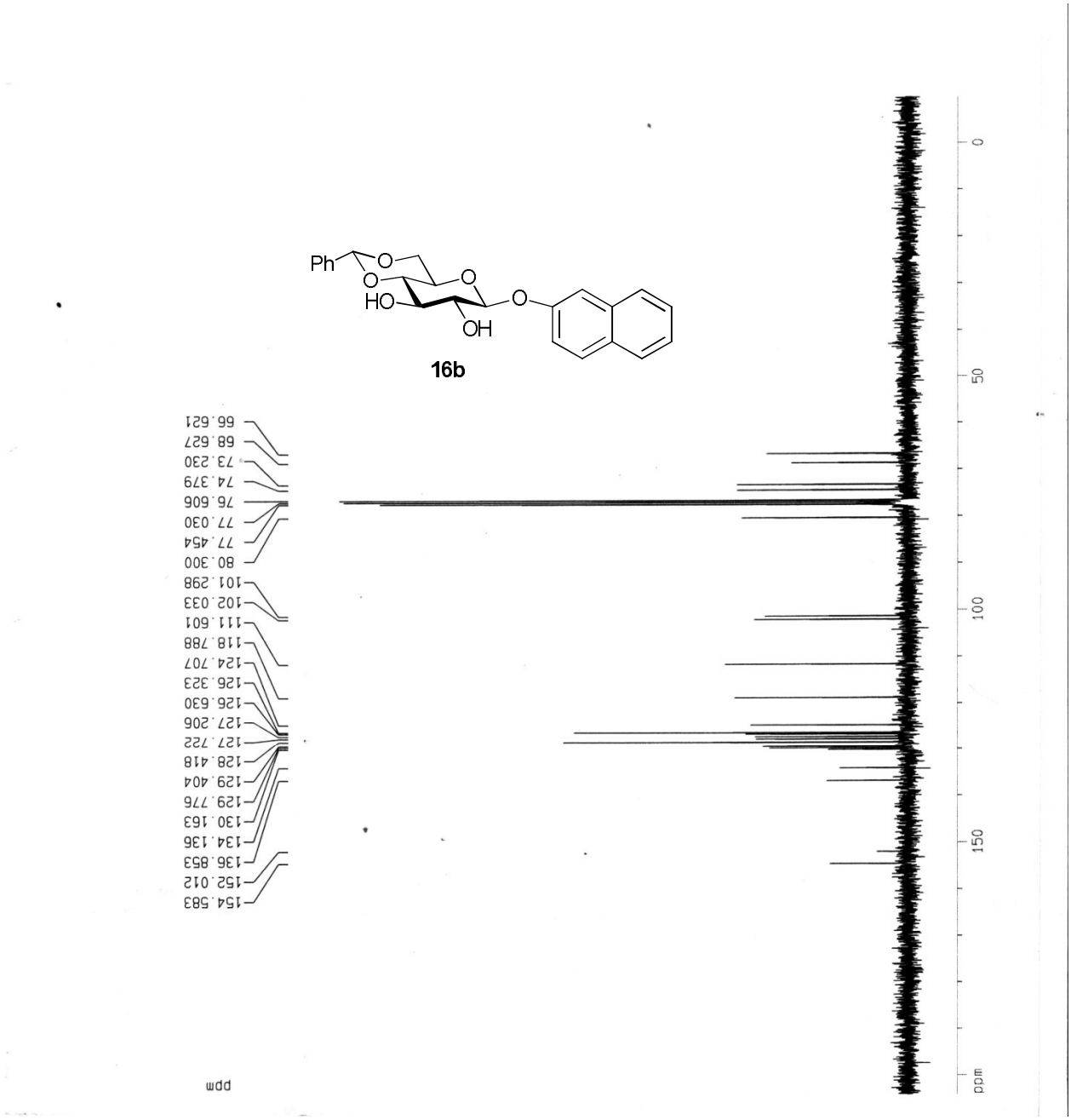
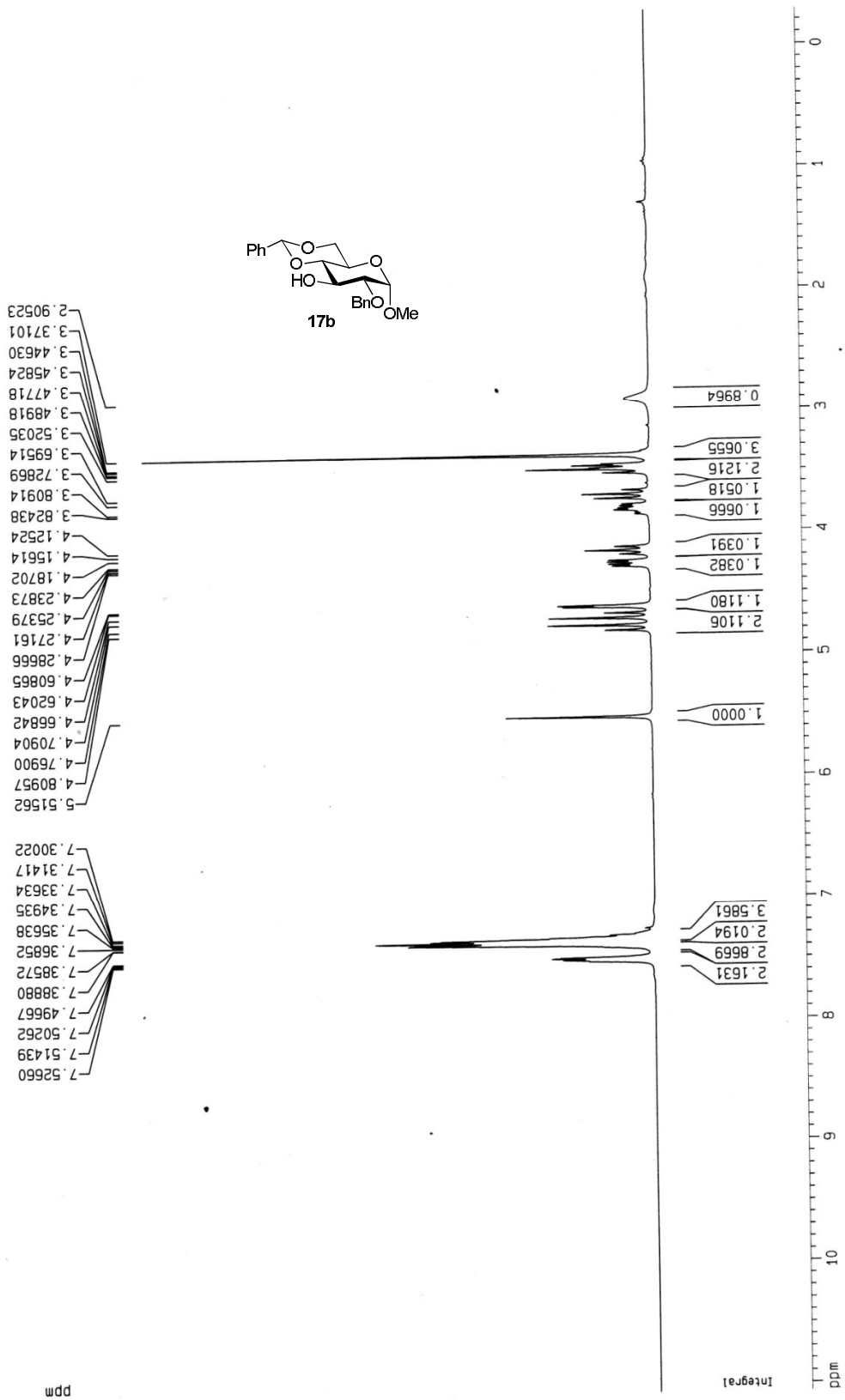
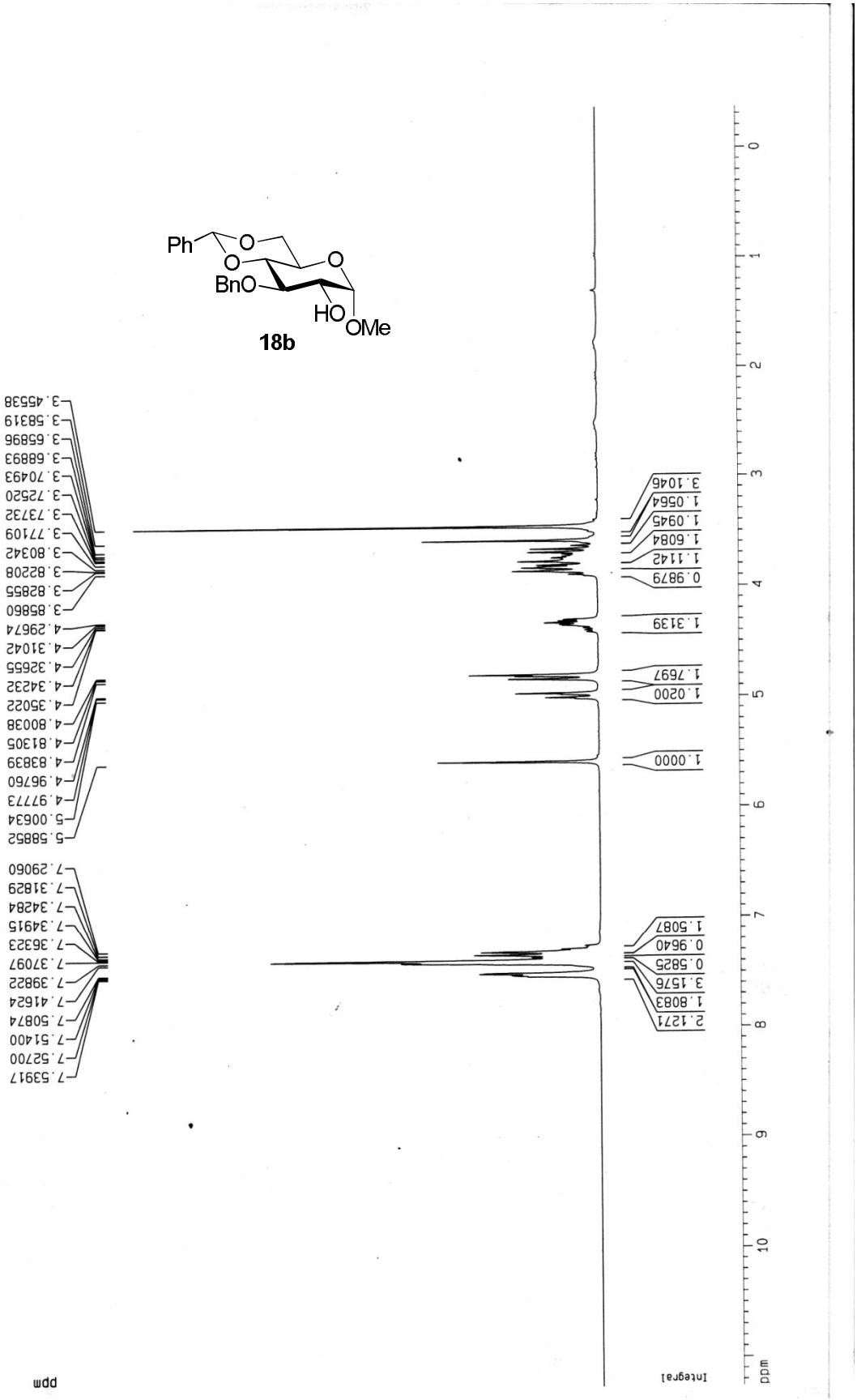
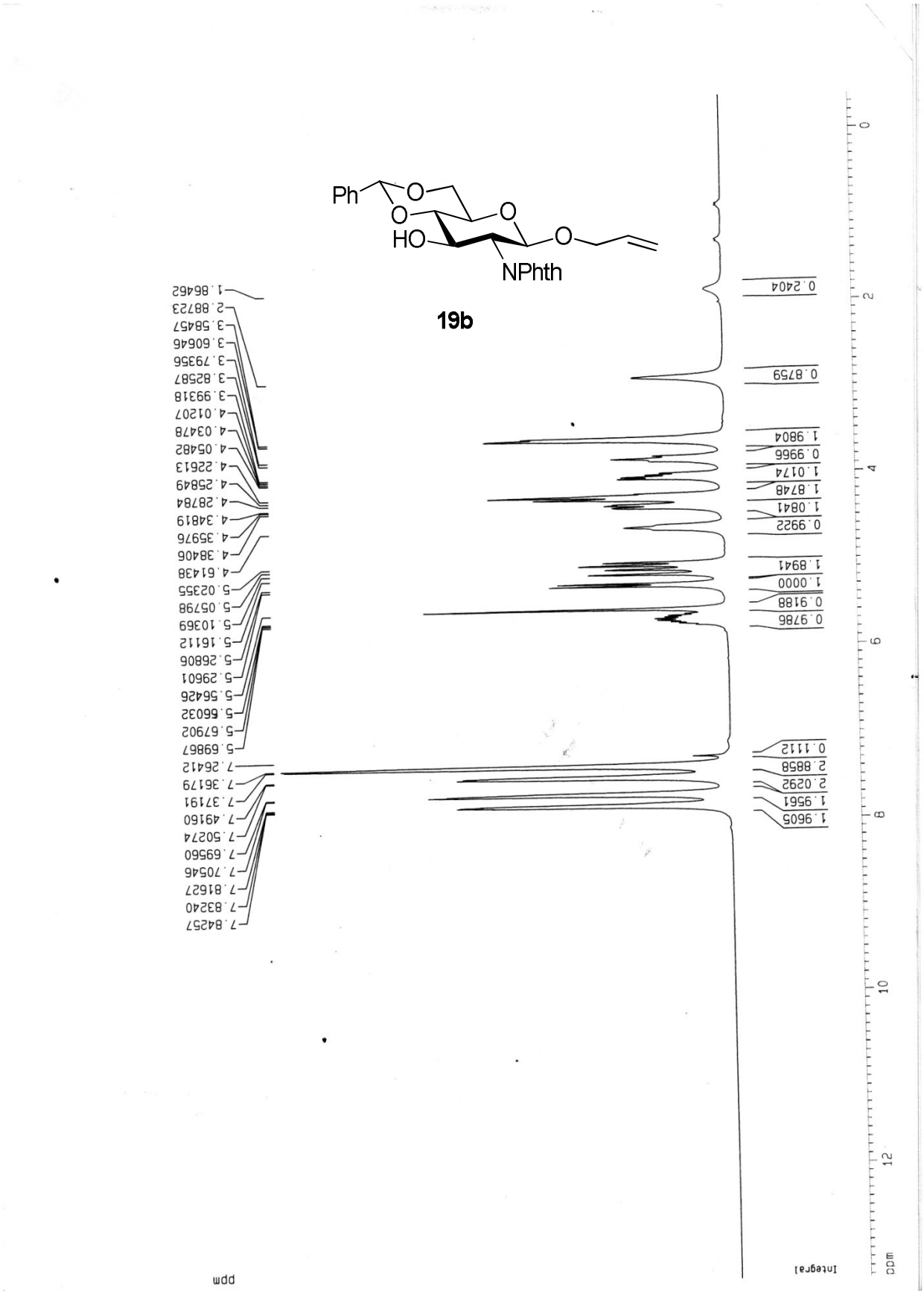
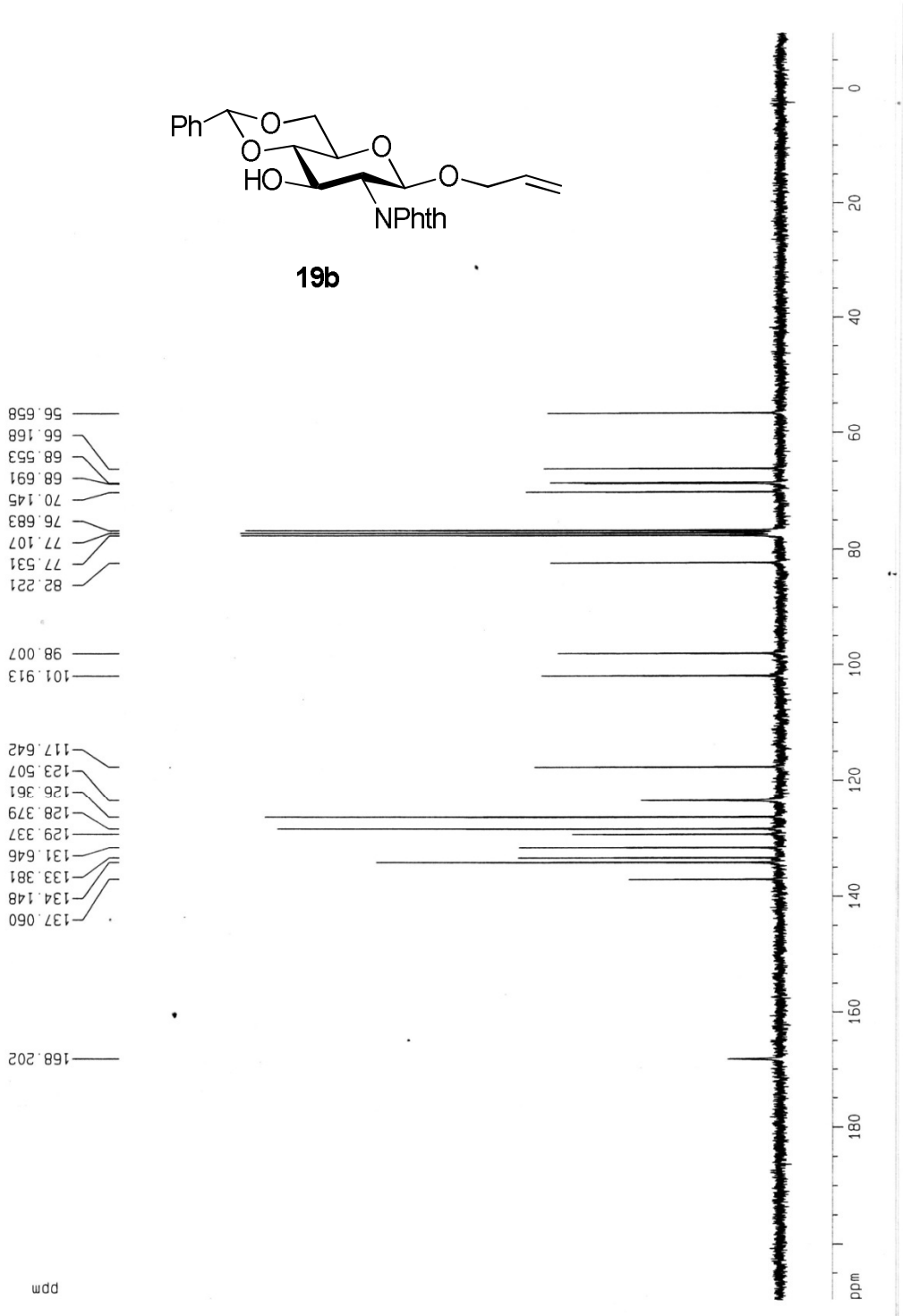
2.1338
2.1547
1.0737
1.0093
1.0000
0.9958
2.4368
5.4103
2.9941
3.1813
ppm
Integral

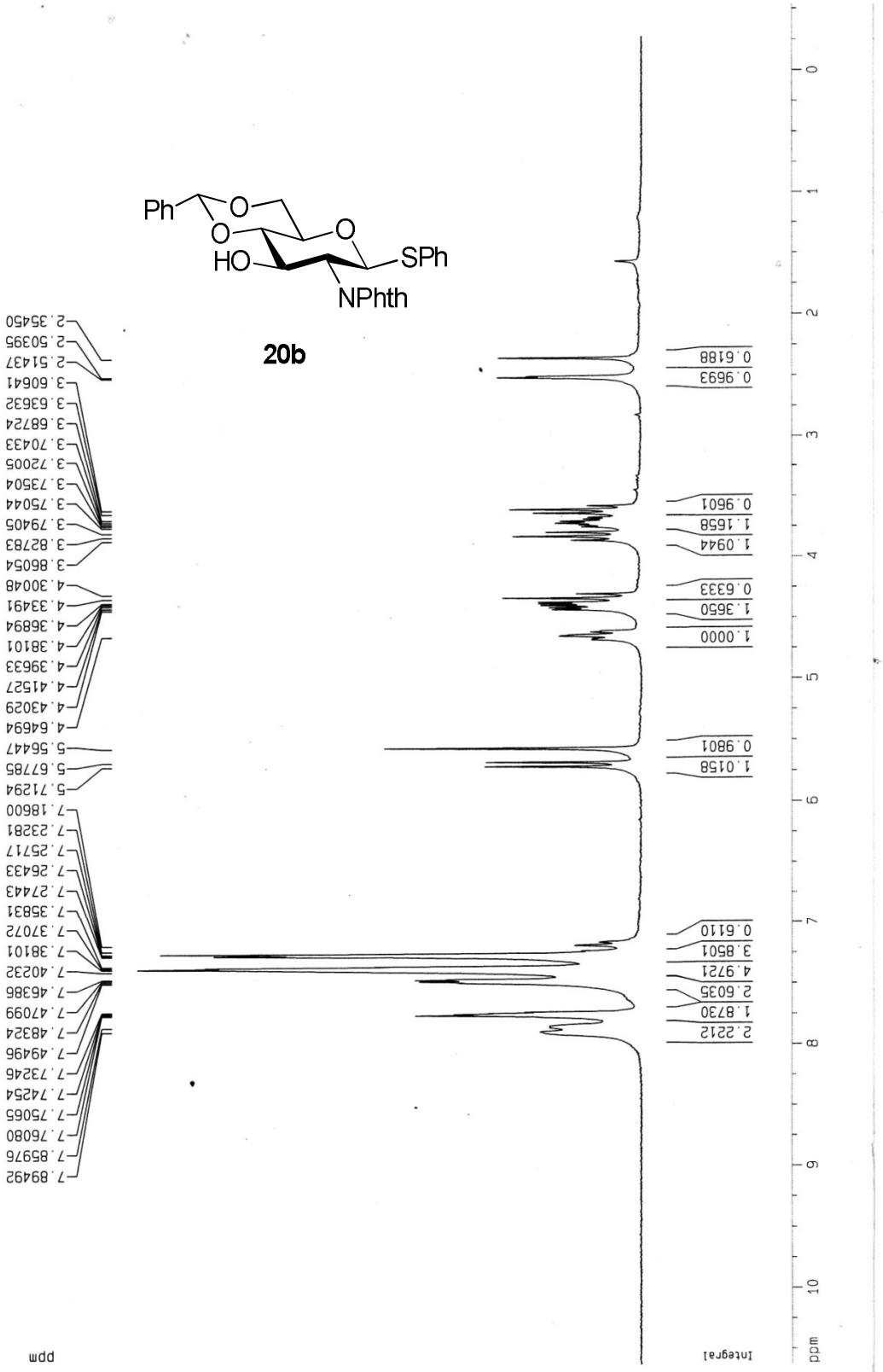

Ph
O
O
O
HO
OH
O
16b
66.621
68.627
73.230
74.379
76.606
77.030
77.454
80.300
101.298
102.033
111.601
118.788
124.707
126.323
126.630
127.206
127.722
128.418
129.404
129.776
130.163
134.136
136.853
152.012
154.583
ppm
0
50
100
150
ppm


Ph
O
O
O
HO
BnO
OMe
17b
2.90523
3.37101
3.44630
3.45824
3.47718
3.48918
3.52035
3.69514
3.72869
3.80914
3.82438
4.12524
4.15614
4.18702
4.23873
4.25379
4.27161
4.28666
4.60865
4.62043
4.66842
4.70904
4.76900
4.80957
5.51562
7.30022
7.31417
7.33634
7.34935
7.35638
7.36852
7.38572
7.38880
7.49667
7.50262
7.51439
7.52660
0.8964
3.0655
2.1216
1.0518
1.0666
1.0391
1.0382
1.1180
2.1106
1.0000
3.5861
2.0194
2.8669
2.1631
Integral
ppm

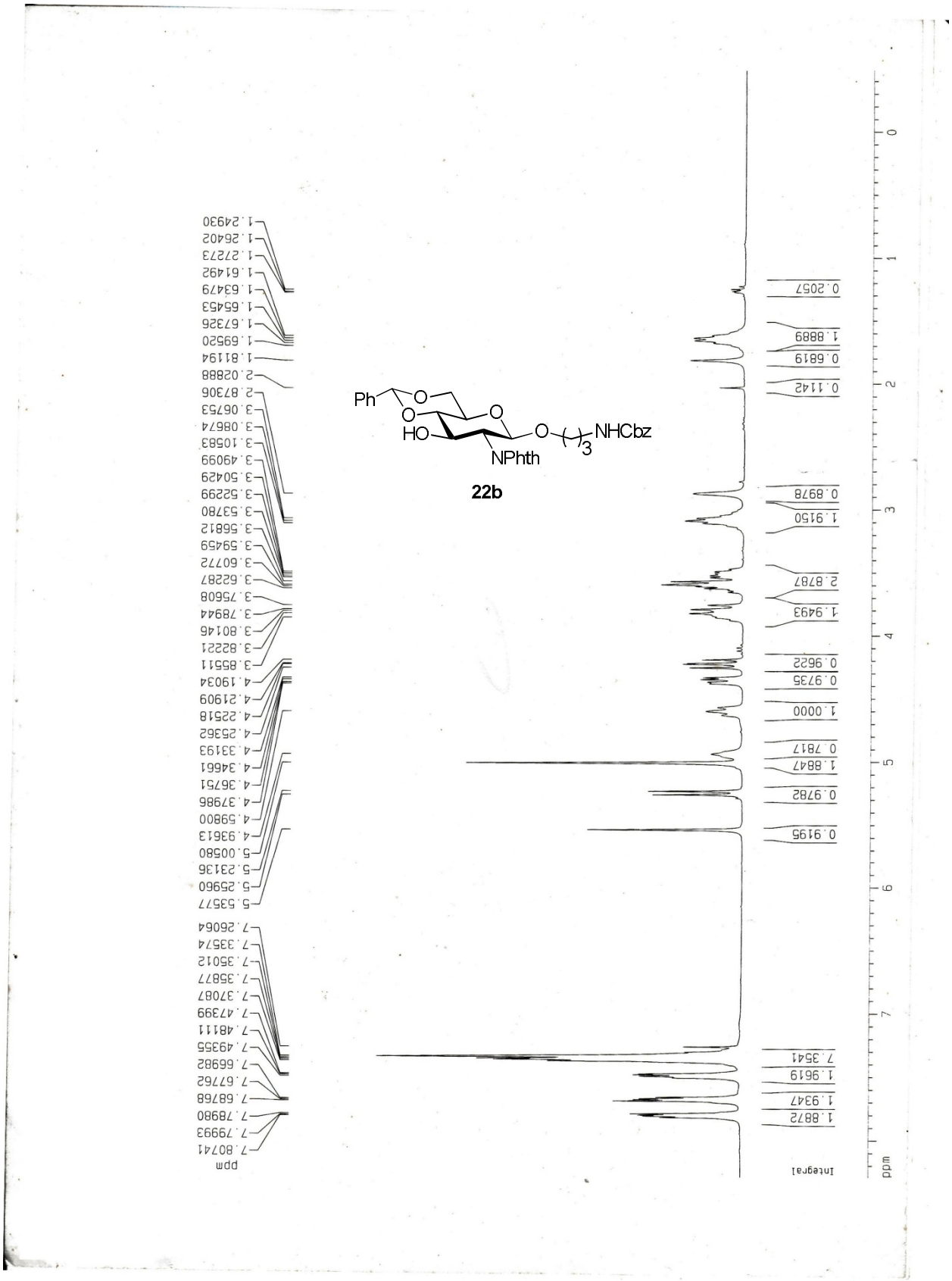

Ph
O
O
O
BnO
HO
OMe
18b
3.45538
3.58319
3.65896
3.68893
3.70493
3.72520
3.73732
3.77109
3.80342
3.82208
3.82855
3.85860
4.29674
4.31042
4.32655
4.34232
4.35022
4.80038
4.81305
4.83839
4.96760
4.97773
5.00634
5.88852
7.29060
7.31829
7.34284
7.34915
7.36323
7.37097
7.39822
7.41624
7.50874
7.51400
7.52700
7.53917
3.1046
1.0564
1.0945
1.6084
1.1142
0.9879
1.3139
1.7697
1.0200
1.0000
1.5087
0.9640
0.5825
3.1576
1.8083
2.1271
Integral
ppm

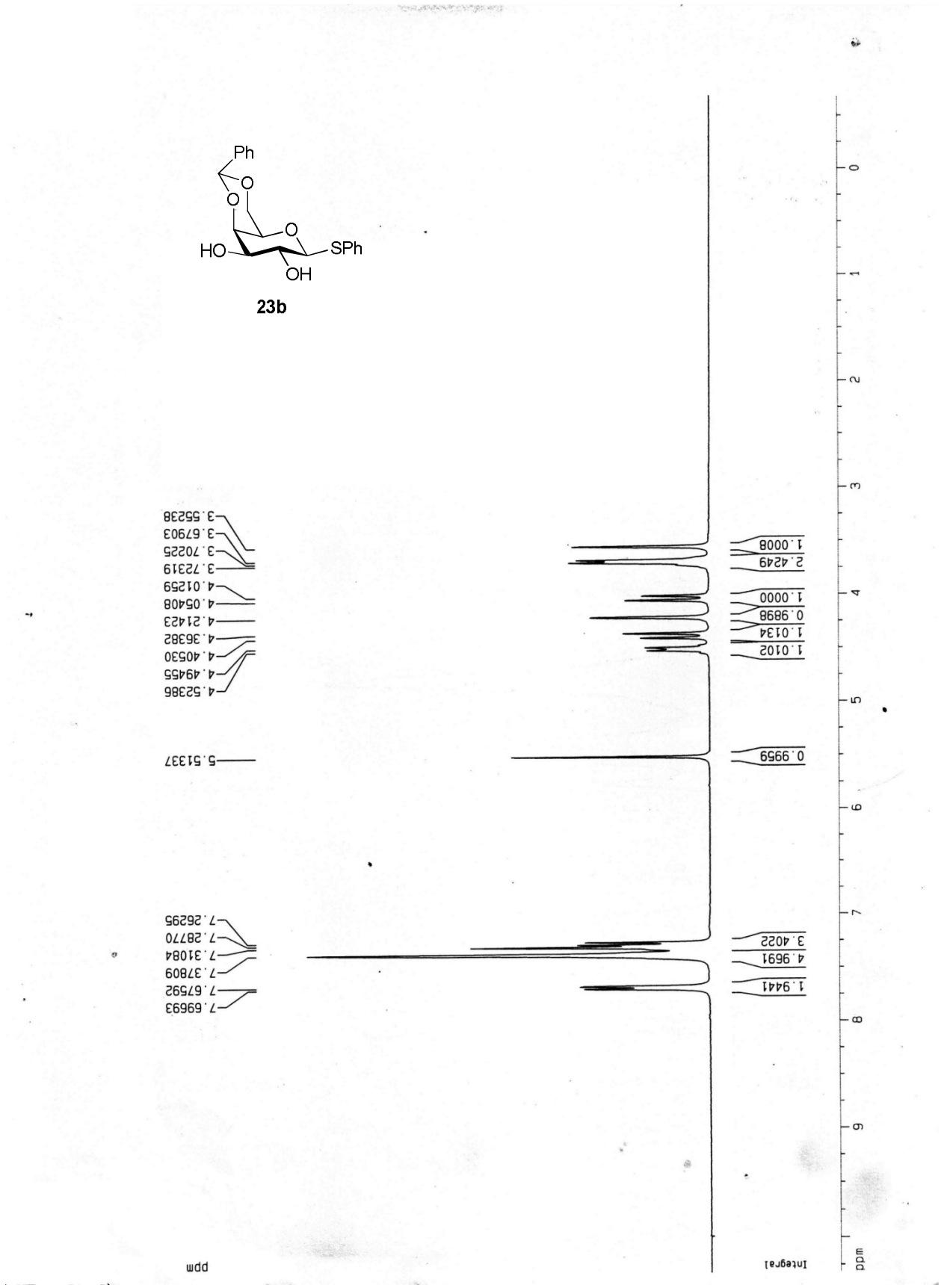

Ph
O
O
O
HO
O
NPhth
19b
1.86462
2.88723
3.58457
3.60646
3.79356
3.82587
3.99318
4.01207
4.03478
4.05482
4.22613
4.25849
4.28784
4.34819
4.35976
4.38406
4.61438
5.02355
5.05798
5.10369
5.16112
5.26806
5.29601
5.56426
5.66032
5.67902
5.69867
7.26412
7.36179
7.37191
7.49160
7.50274
7.69560
7.70546
7.81627
7.83240
7.84257
0.2404
0.8759
1.9804
0.9966
1.0174
1.8748
1.0841
0.9922
1.8941
1.0000
0.9188
0.9786
0.1112
2.8858
2.0292
1.9561
1.9605
ppm
Integral

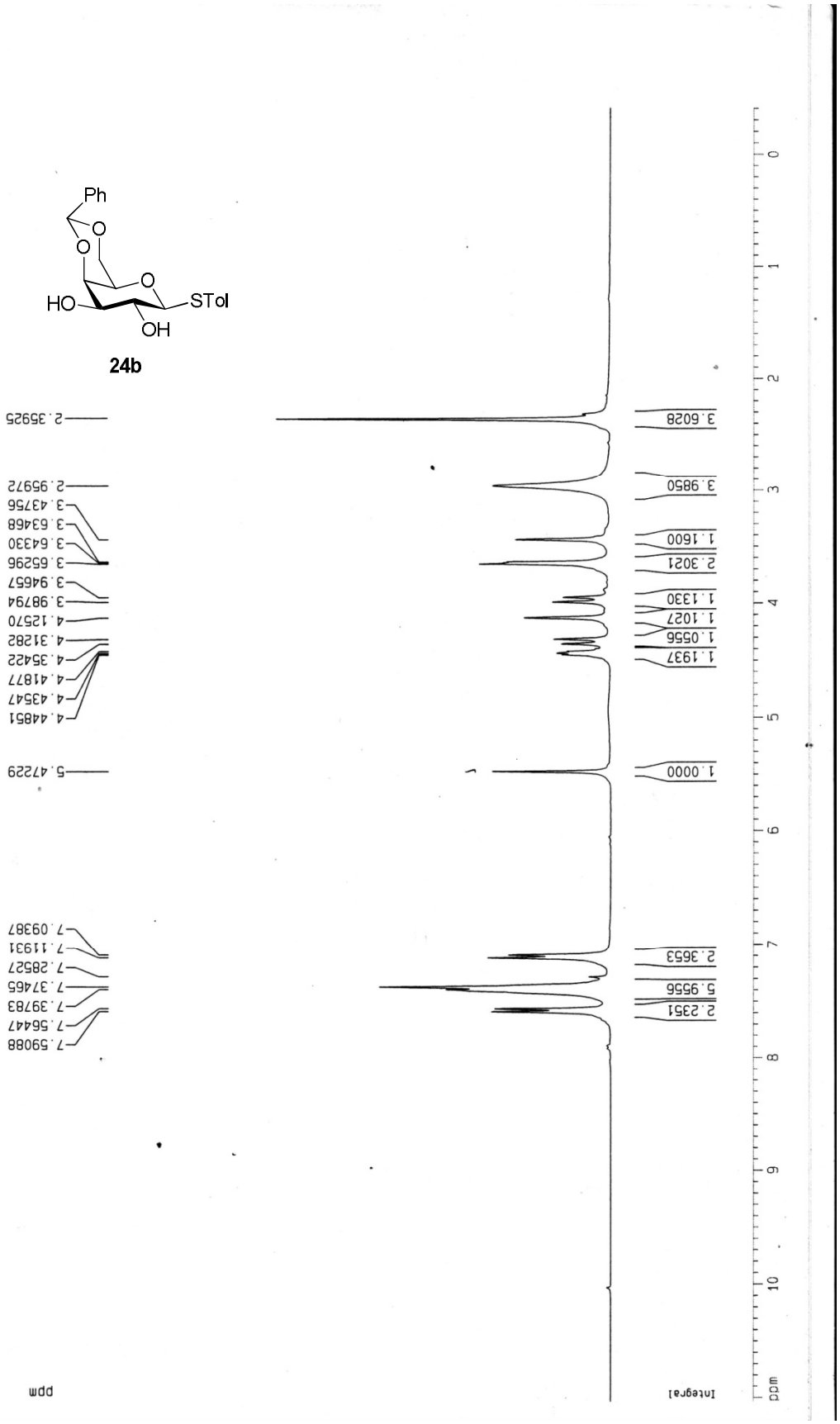

Ph
O
O
O
HO
O
NPhth
19b
56.658
66.168
68.553
68.691
70.145
76.683
77.107
77.531
82.221
98.007
101.913
117.642
123.507
126.361
128.379
129.337
131.646
133.381
134.148
137.060
168.202
ppm
0
20
40
60
80
100
120
140
160
180
ppm

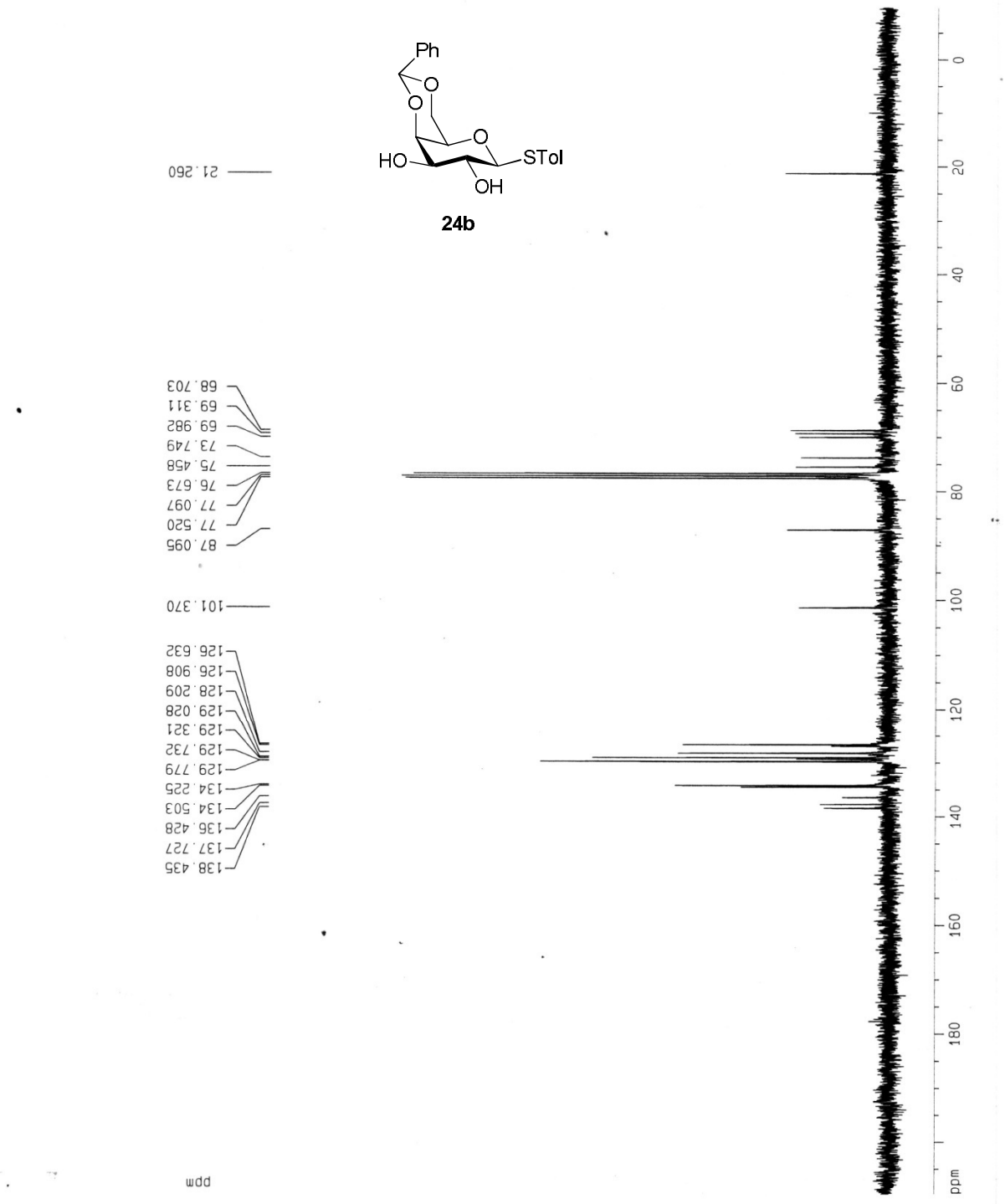

Ph
O
O
O
HO
SPh
NPhth
20b
2.35450
2.50395
2.51437
3.60641
3.63632
3.68724
3.70433
3.72005
3.73504
3.75044
3.79405
3.82783
3.86054
4.30048
4.33491
4.36894
4.38101
4.39633
4.41527
4.43029
4.64694
5.56447
5.67785
5.71294
7.18600
7.23281
7.25717
7.26433
7.27443
7.35831
7.37072
7.38101
7.40232
7.46386
7.47099
7.48324
7.49496
7.73246
7.74254
7.75065
7.76080
7.85976
7.89492
ppm
0.6188
0.9693
0.9601
1.1658
1.0944
0.6333
1.3650
1.0000
0.9801
1.0158
0.6110
3.8501
4.9721
2.6035
1.8730
2.2212
Integral
0
1
2
3
4
5
6
7
8
9
10
ppm

Ph
O
O
O
HO
OMP
NPhth
21b
1.25967
1.89002
1.95712
2.03935
2.09809
2.13513
3.66705
3.69743
3.72012
3.77009
3.83698
3.86962
3.90196
4.37958
4.39226
4.42516
4.46847
4.50329
4.53164
4.67443
4.70129
4.73582
5.29600
5.59396
5.78794
5.81600
6.72482
6.75494
6.83392
6.86424
7.26124
7.36822
7.37716
7.38959
7.48923
7.49590
7.50807
7.51959
7.70982
7.71375
7.72010
7.72753
7.73785
7.84966
7.85883
0.3106
1.4917
2.5168
0.5637
0.5215
0.5104
0.4870
0.0564
0.4805
0.4834
1.0165
0.9835
1.4147
1.0082
1.0530
1.0000
ppm
Integral

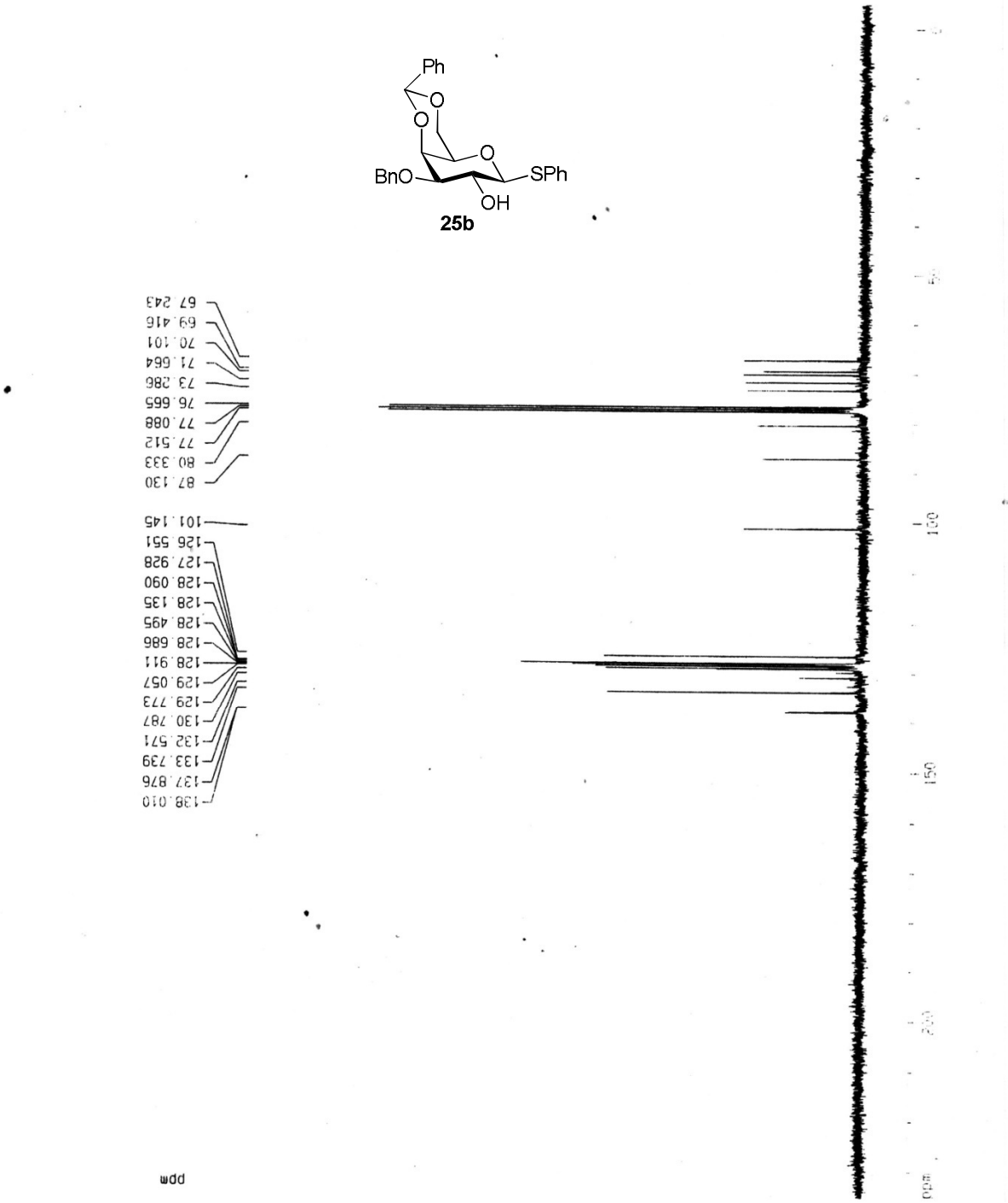

Ph
O
O
O
HO
O
NHCbz
3
NPhth
22b
ppm
Integral


Ph
O
O
O
HO
SPh
OH
23b
3.55238
3.67903
3.70225
3.72319
4.01259
4.05408
4.21423
4.36382
4.40530
4.49455
4.52386
5.51337
7.26295
7.28770
7.31084
7.37809
7.67592
7.69693
1.0008
2.4249
1.0000
0.9898
1.0134
1.0102
0.9959
3.4022
4.9691
1.9441
Integral
ppm
0
1
2
3
4
5
6
7
8
9

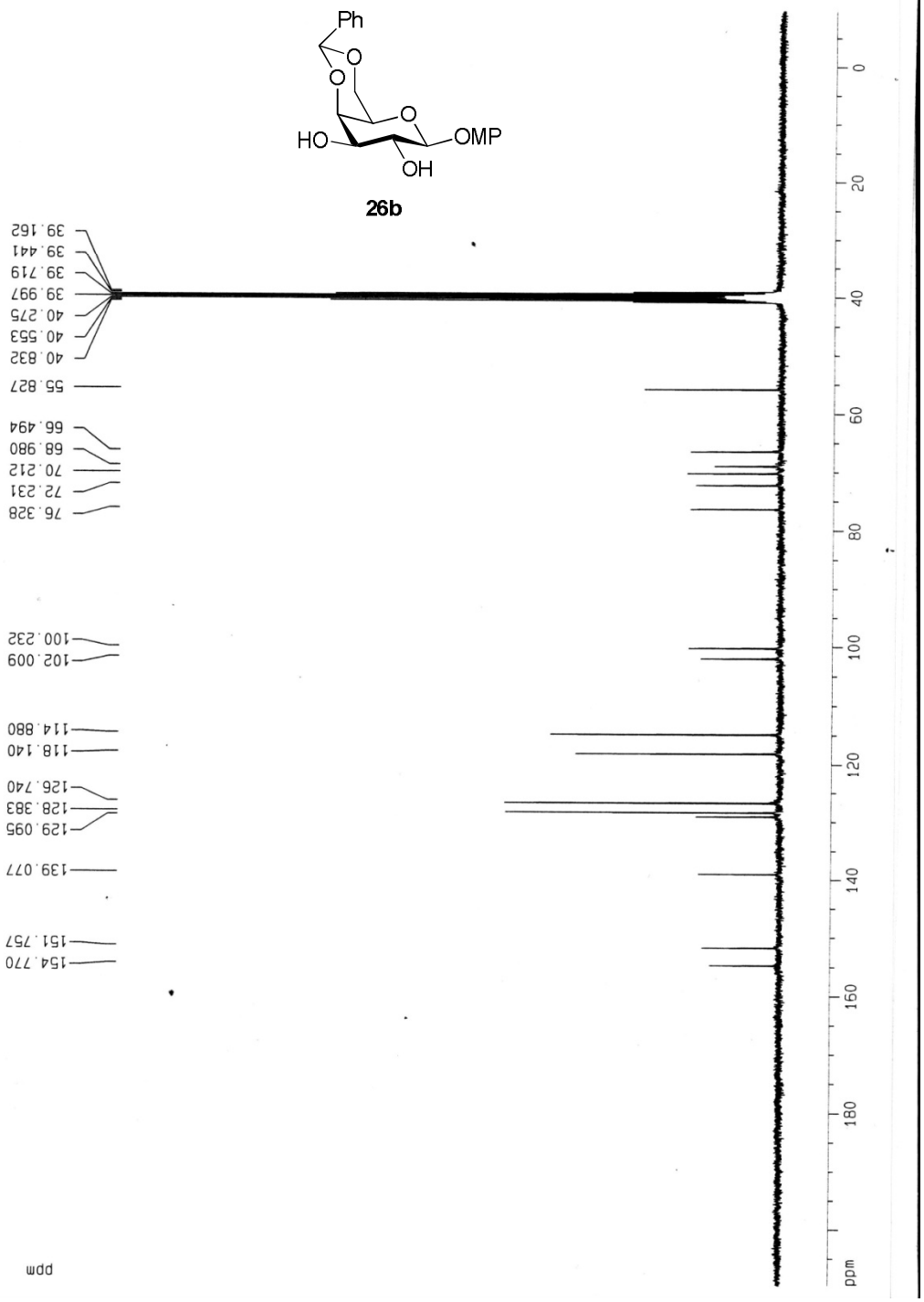

Ph
O
O
O
HO
STol
OH
24b
2.35925
2.95972
3.43756
3.53468
3.64330
3.65296
3.94657
3.98794
4.12570
4.31282
4.35422
4.41877
4.43547
4.44851
5.47229
7.09387
7.11931
7.28527
7.37465
7.39783
7.56447
7.59088
3.6028
3.9850
1.1600
2.3021
1.1330
1.1027
1.0556
1.1937
1.0000
2.3653
5.9556
2.2351
ppm
Integral

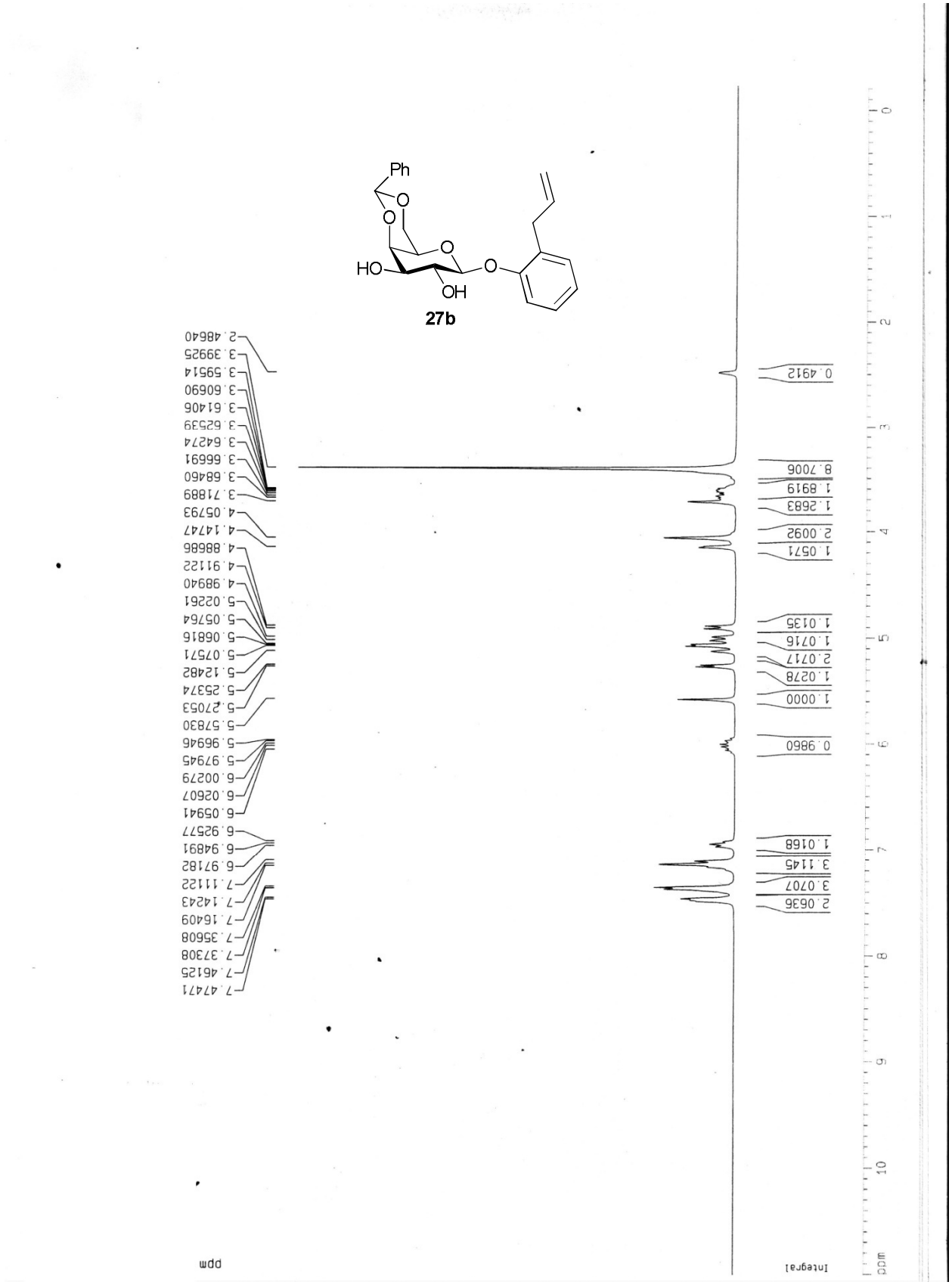

Ph
O
O
O
HO
STol
OH
24b
21.260
68.703
69.311
69.982
73.749
75.458
76.673
77.097
77.520
87.095
101.370
126.632
126.908
128.209
129.028
129.321
129.732
129.779
134.225
134.503
136.428
137.727
138.435
ppm
0
20
40
60
80
100
120
140
160
180
ppm

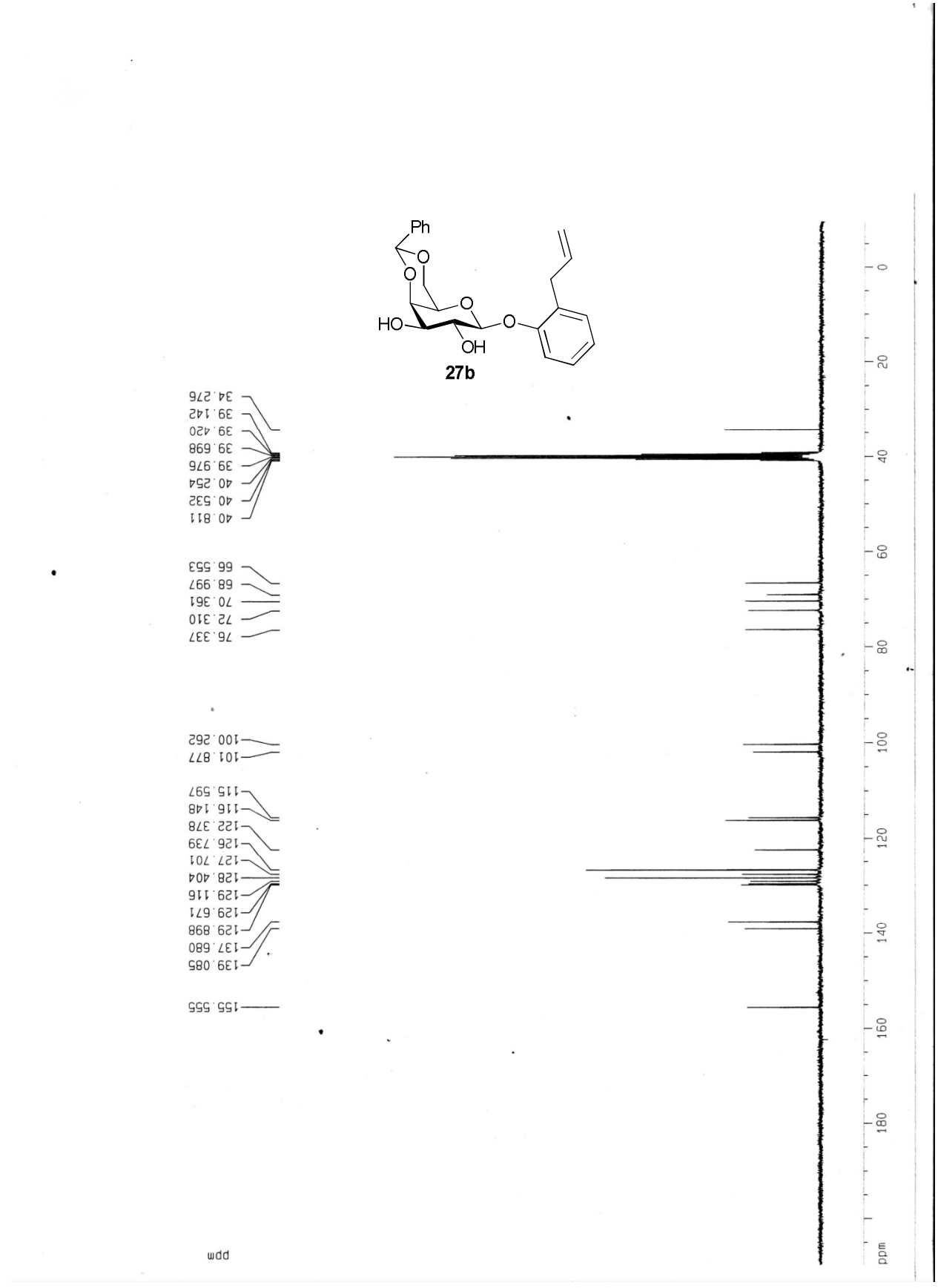
Ph
O
O
O
BnO
SPh
OH
25b
RG (0) -AGRGA3-1H
7.69492
7.69065
7.66888
7.66343
7.41037
7.39801
7.36215
7.35250
7.34622
7.34051
7.31778
7.30847
7.29264
7.27419
7.26923
7.25894
7.23322
5.43044
4.72682
4.72146
4.53711
4.50556
4.37828
4.37370
4.37709
4.33245
4.15517
4.14459
4.00275
3.99758
3.97477
3.96141
3.95609
3.94382
3.91256
3.53381
3.52286
3.50288
3.49191
3.45374
3.09183
3.08371
2.50792
1.0000
2.1817
1.0396
1.0435
1.0509
2.1461
1.1256
1.0160
0.5740
0.8732
1.1014
2.1808
2.9798
5.6503
2.2854
1.9987
ppm
Integral

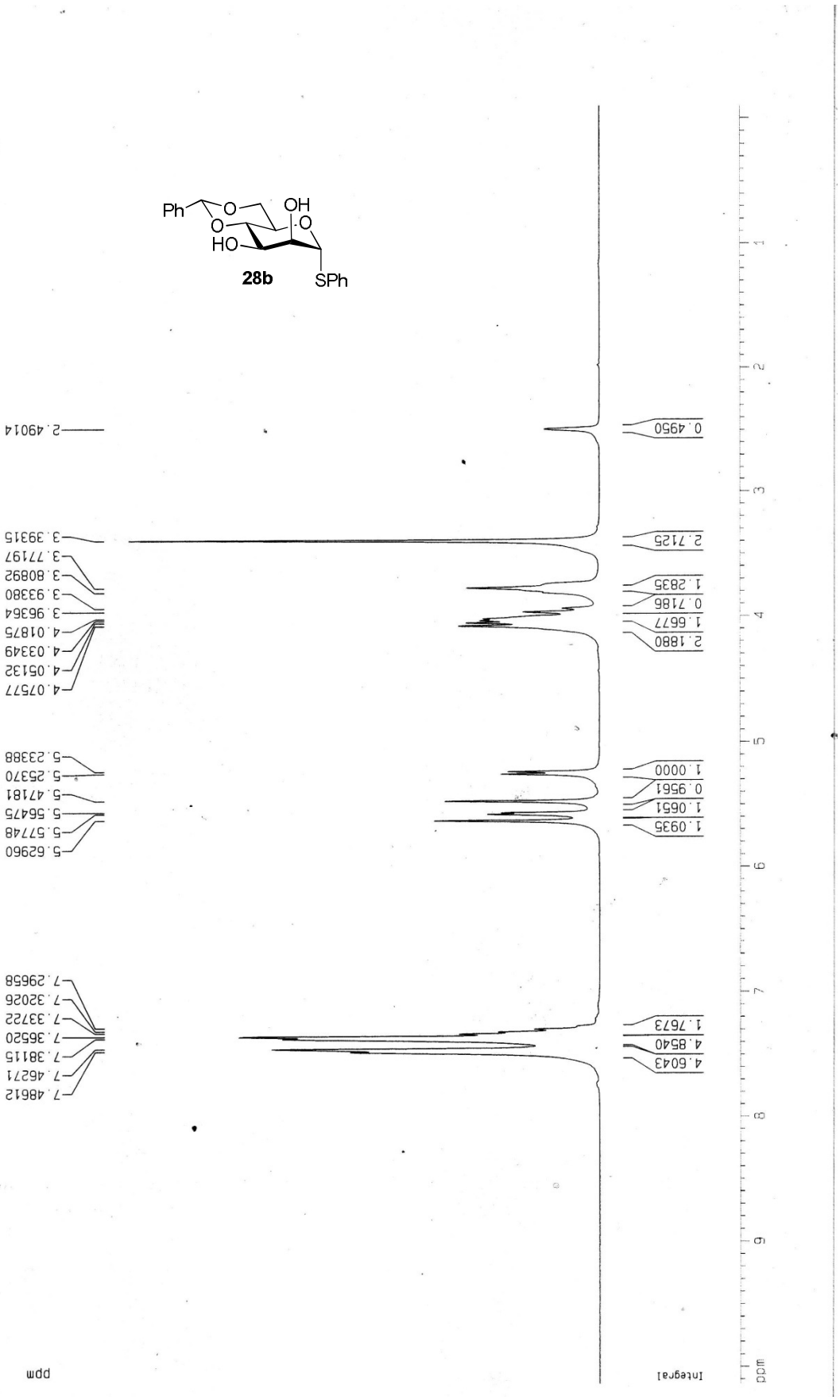

Ph
O
O
O
BnO
SPh
OH
25b
67.243
69.416
70.101
71.664
73.286
76.665
77.088
77.512
80.333
87.130
101.145
126.551
127.928
128.090
128.135
128.495
128.686
128.911
129.057
129.773
130.787
132.571
133.739
137.876
138.010
ppm

Ph
O
O
O
HO
OMP
OH
26b
0.0864
0.1356
0.2156
0.7782
0.0327
3.9590
2.0341
3.8626
1.8955
1.0726
0.1686
1.0000
0.9724
0.8850
0.9762
1.8709
1.8384
5.1065
1.90369
2.00613
2.49071
3.35337
3.58593
3.64077
3.69793
4.04608
4.12372
4.82905
4.84902
5.04810
5.26951
5.57311
6.84059
6.87018
6.99212
7.02169
7.35921
7.37010
7.45257
7.46519
Integral
ppm


Ph
O
O
O
HO
OMP
OH
26b
39.162
39.441
39.719
39.997
40.275
40.553
40.832
55.827
66.494
68.980
70.212
72.231
76.328
100.232
102.009
114.880
118.140
126.740
128.383
129.095
139.077
151.757
154.770
ppm
0
20
40
60
80
100
120
140
160
180
ppm


Ph
O
O
O
HO
O
OH
27b
2.48640
3.39925
3.59514
3.60690
3.61406
3.62539
3.64274
3.66691
3.68460
3.71889
4.05793
4.14747
4.88686
4.91122
4.98940
5.02261
5.05764
5.06816
5.07571
5.12482
5.25374
5.27053
5.57830
5.96946
5.97945
6.00279
6.02607
6.05941
6.92577
6.94891
6.97182
7.11122
7.14243
7.16409
7.35608
7.37308
7.46125
7.47471
0.4912
8.7006
1.8919
1.2683
2.0092
1.0571
1.0135
1.0716
2.0717
1.0278
1.0000
0.9860
1.0168
3.1145
3.0707
2.0636
ppm
Integral


Ph
O
O
O
HO
O
OH
27b
34.276
39.142
39.420
39.698
39.976
40.254
40.532
40.811
66.553
68.997
70.361
72.310
76.337
100.262
101.877
115.597
116.148
122.378
126.739
127.701
128.404
129.116
129.671
129.898
137.680
139.085
155.555
ppm
0
20
40
60
80
100
120
140
160
180
ppm


Ph
O
O
OH
O
HO
28b
SPh
2.49014
3.39315
3.77197
3.80892
3.93380
3.96364
4.01875
4.03349
4.05132
4.07577
5.23388
5.25370
5.47181
5.56475
5.57748
5.62960
7.29658
7.32026
7.33722
7.36520
7.38115
7.46271
7.48612
0.4950
2.7125
1.2835
0.7186
1.6677
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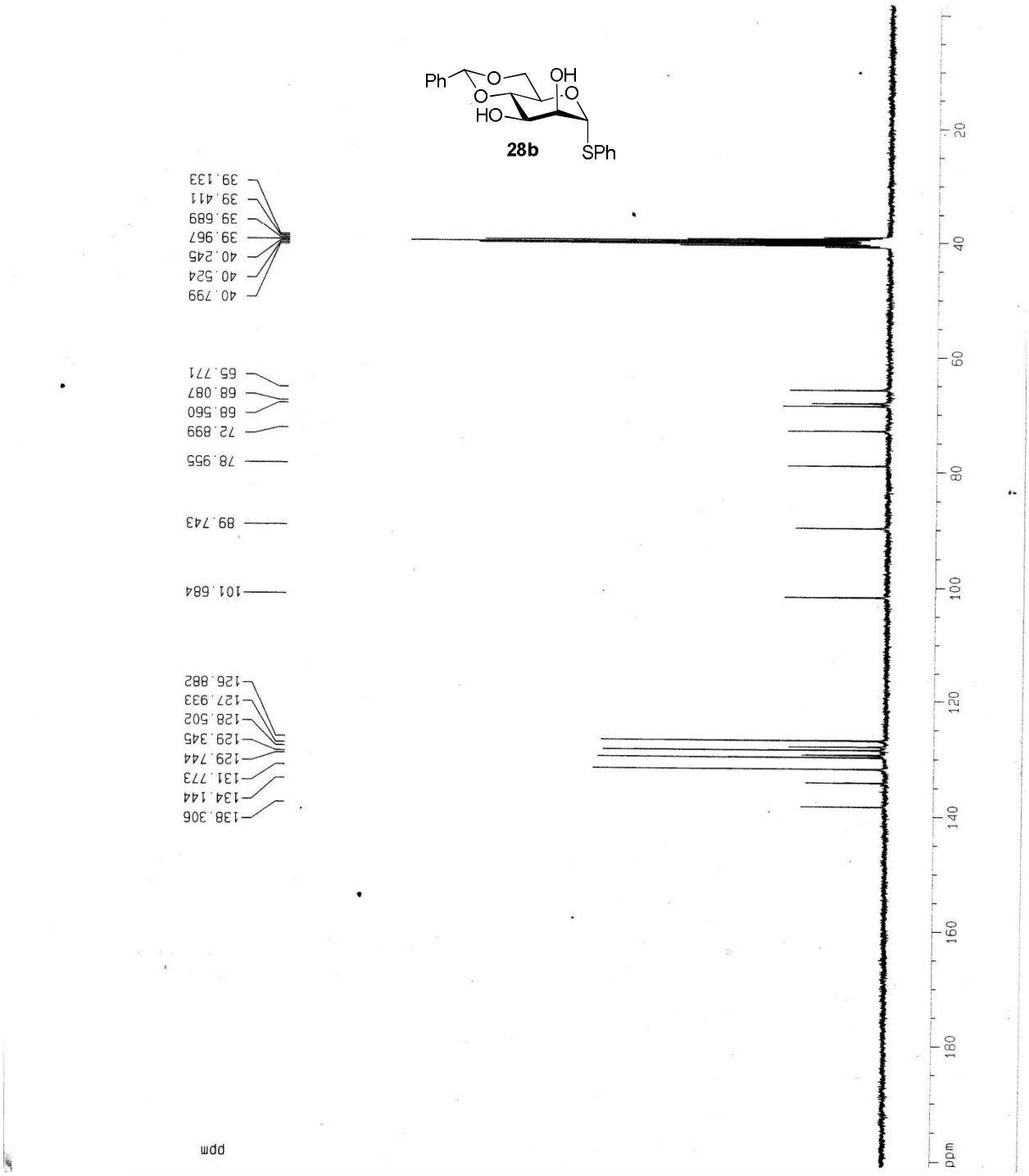
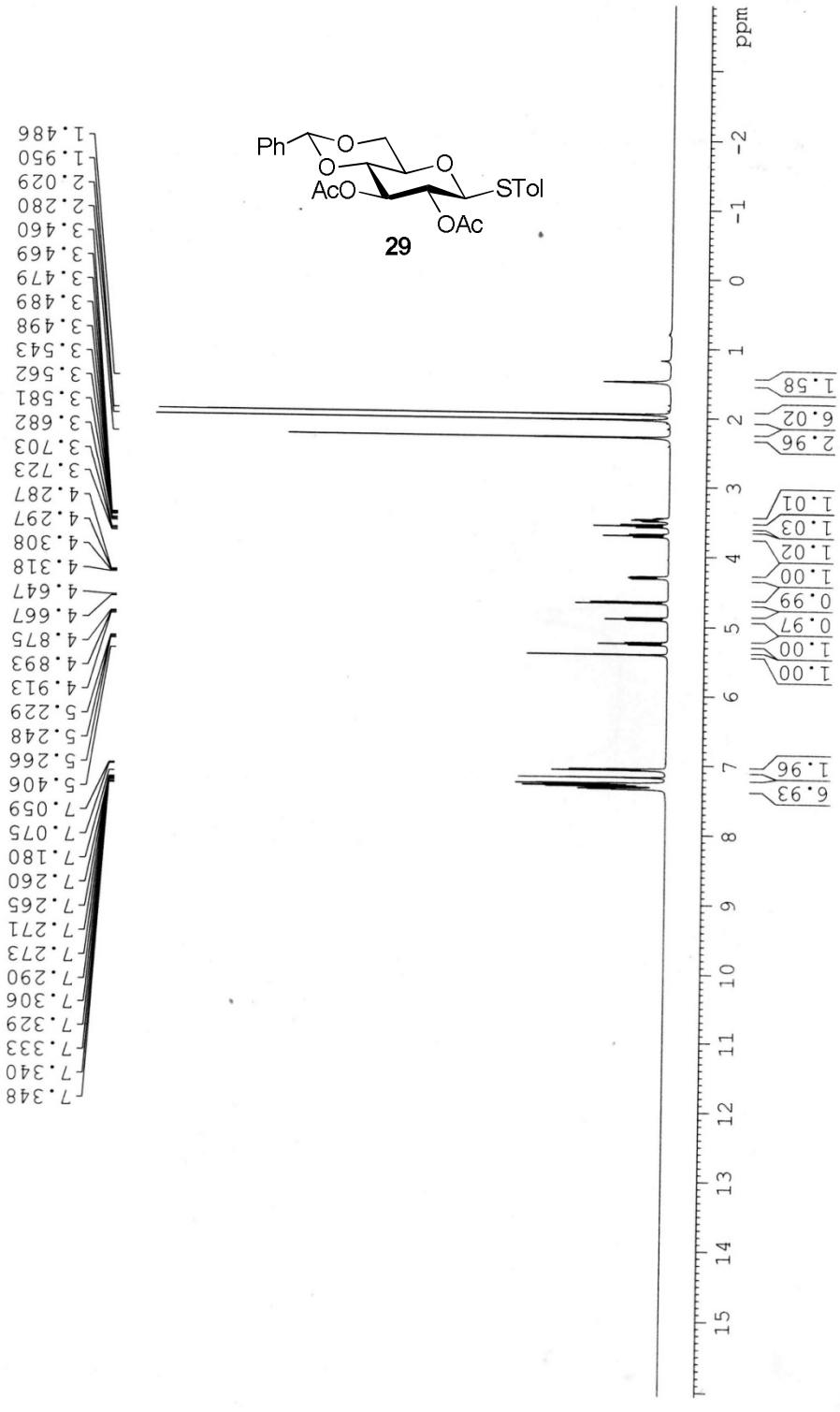
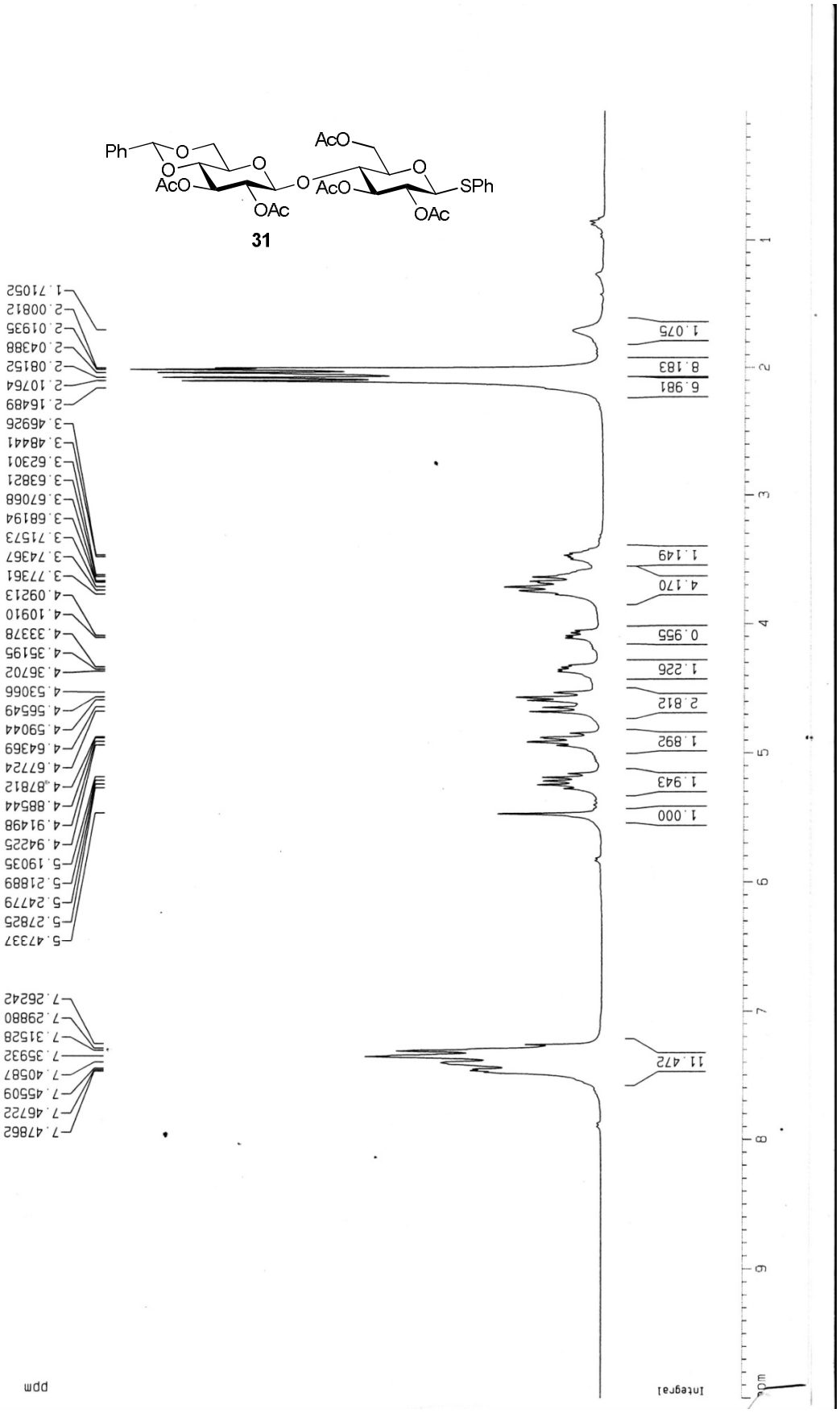
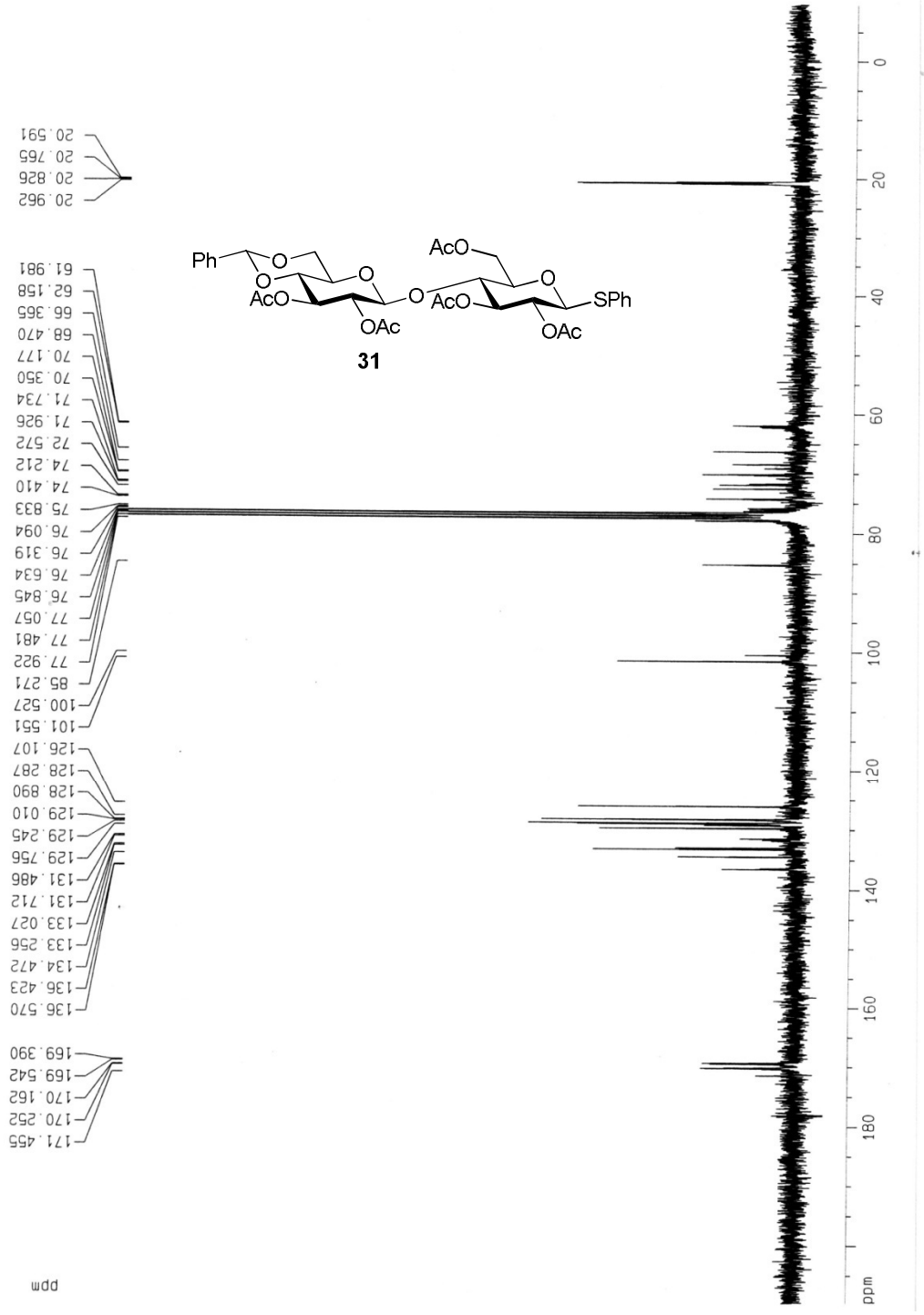
1.0000
0.9561
1.0651
1.0935
1.7673
4.8540
4.6043
ppm
Integral


Ph
O
O
OH
O
HO
28b
SPh
39.133
39.411
39.689
39.967
40.245
40.524
40.799
65.771
68.087
68.560
72.899
78.955
89.743
101.684
126.882
127.933
128.502
129.345
129.744
131.773
134.144
138.306
ppm
20
40
60
80
100
120
140
160
180
ppm


Ph
O
O
O
AcO
STol
OAc
29
ppm
1.58
6.02
2.96
1.01
1.03
1.02
1.00
0.99
0.97
1.00
1.00
6.93
1.96
1.486
1.950
2.029
2.280
3.460
3.469
3.479
3.489
3.498
3.543
3.562
3.581
3.682
3.703
3.723
4.287
4.297
4.308
4.318
4.647
4.667
4.875
4.893
4.913
5.229
5.248
5.266
5.406
7.059
7.075
7.180
7.260
7.265
7.271
7.273
7.290
7.306
7.329
7.333
7.340
7.348

Ph
O
O
O
AcO
STol
OAc
29
21.21
20.84
20.79
68.48
70.65
70.81
72.99
76.77
77.03
77.28
78.13
86.77
101.51
126.16
127.72
128.26
129.17
129.82
133.71
136.77
138.83
169.53
170.12
ppm
0
20
40
60
80
100
120
140
160
180
200


Ph
O
O
O
AcO
OAc
O
AcO
AcO
O
SPh
OAc
31
1.000
1.943
1.892
2.812
1.226
0.955
4.170
1.149
6.981
8.183
1.075
11.472
ppm
Integral


Ph
O
O
AcO
O
OAc
O
AcO
AcO
O
SPh
OAc
31
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129.010
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129.756
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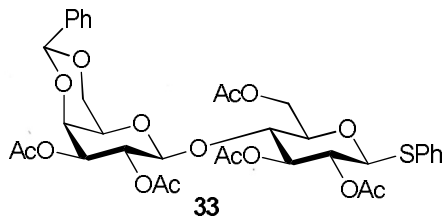
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OAc

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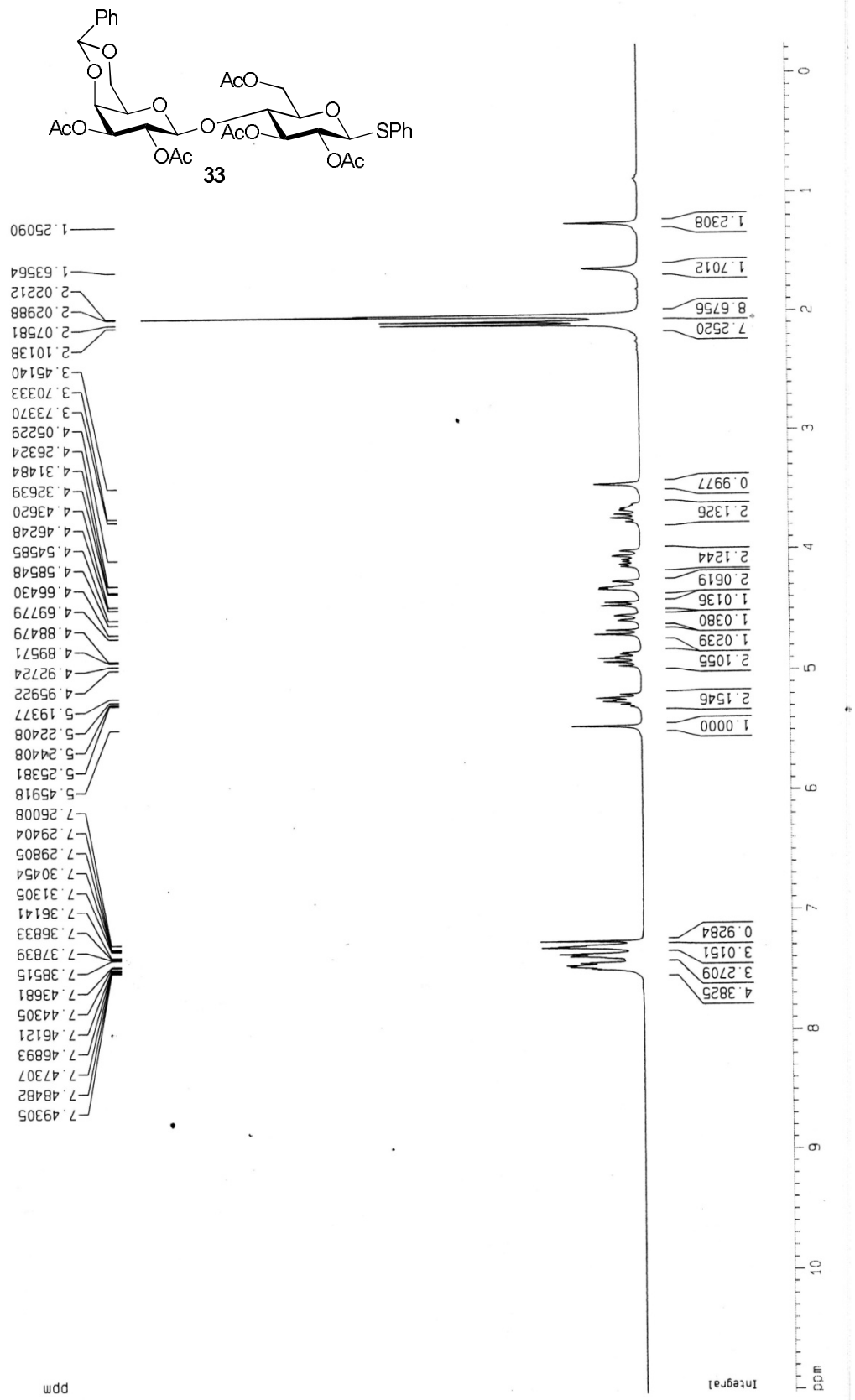
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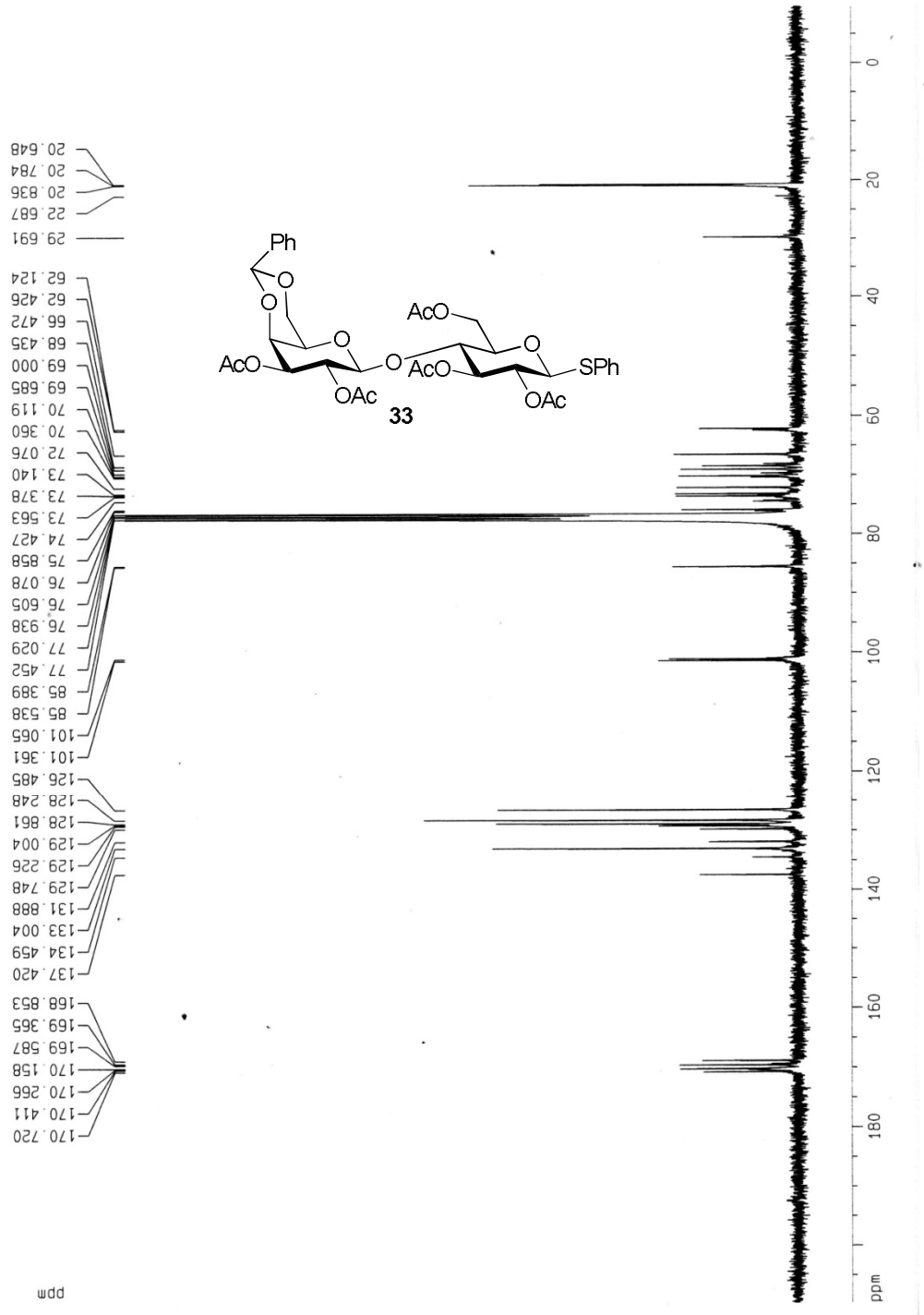
ppm
Integral
ppm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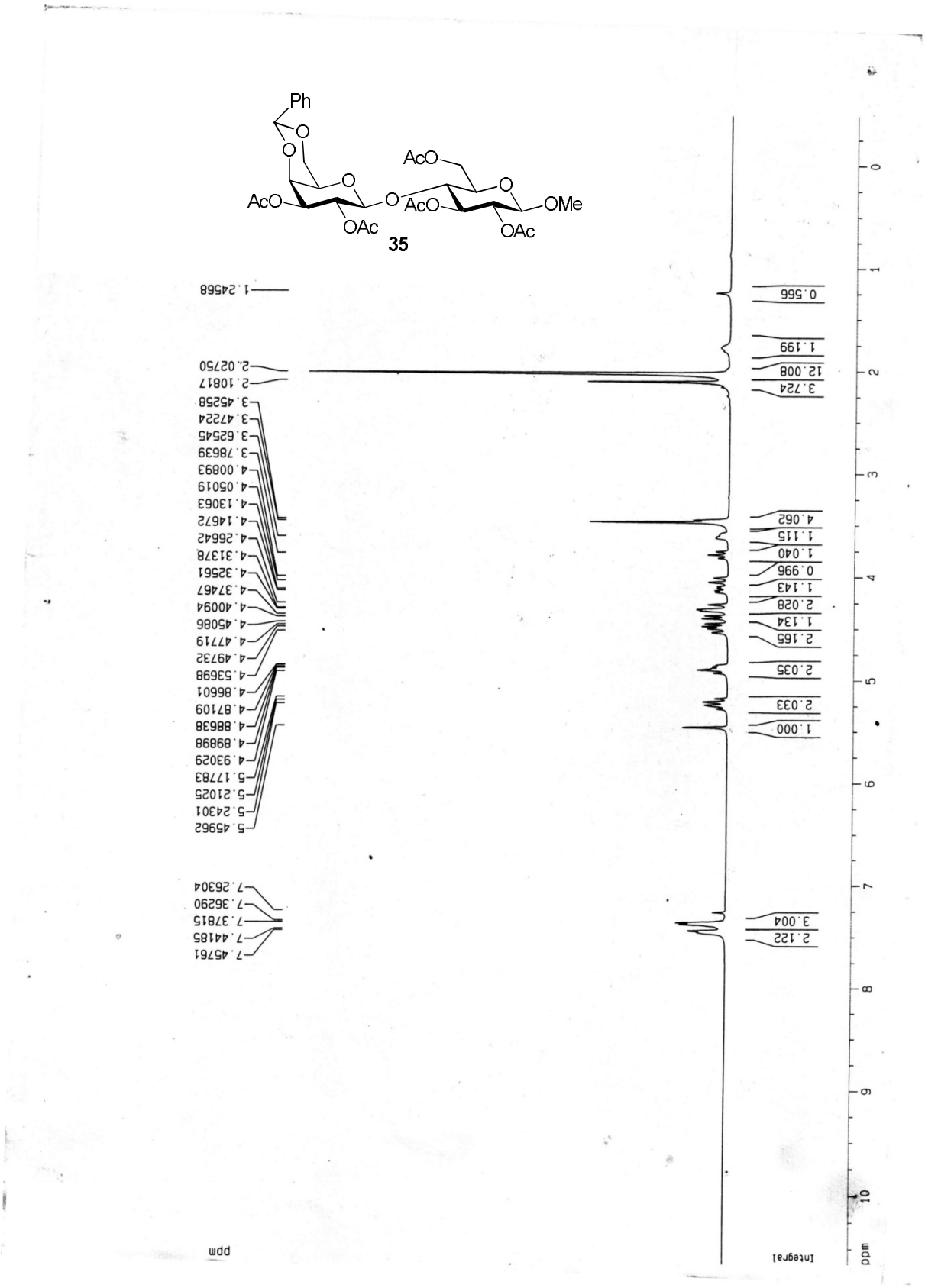
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OMe
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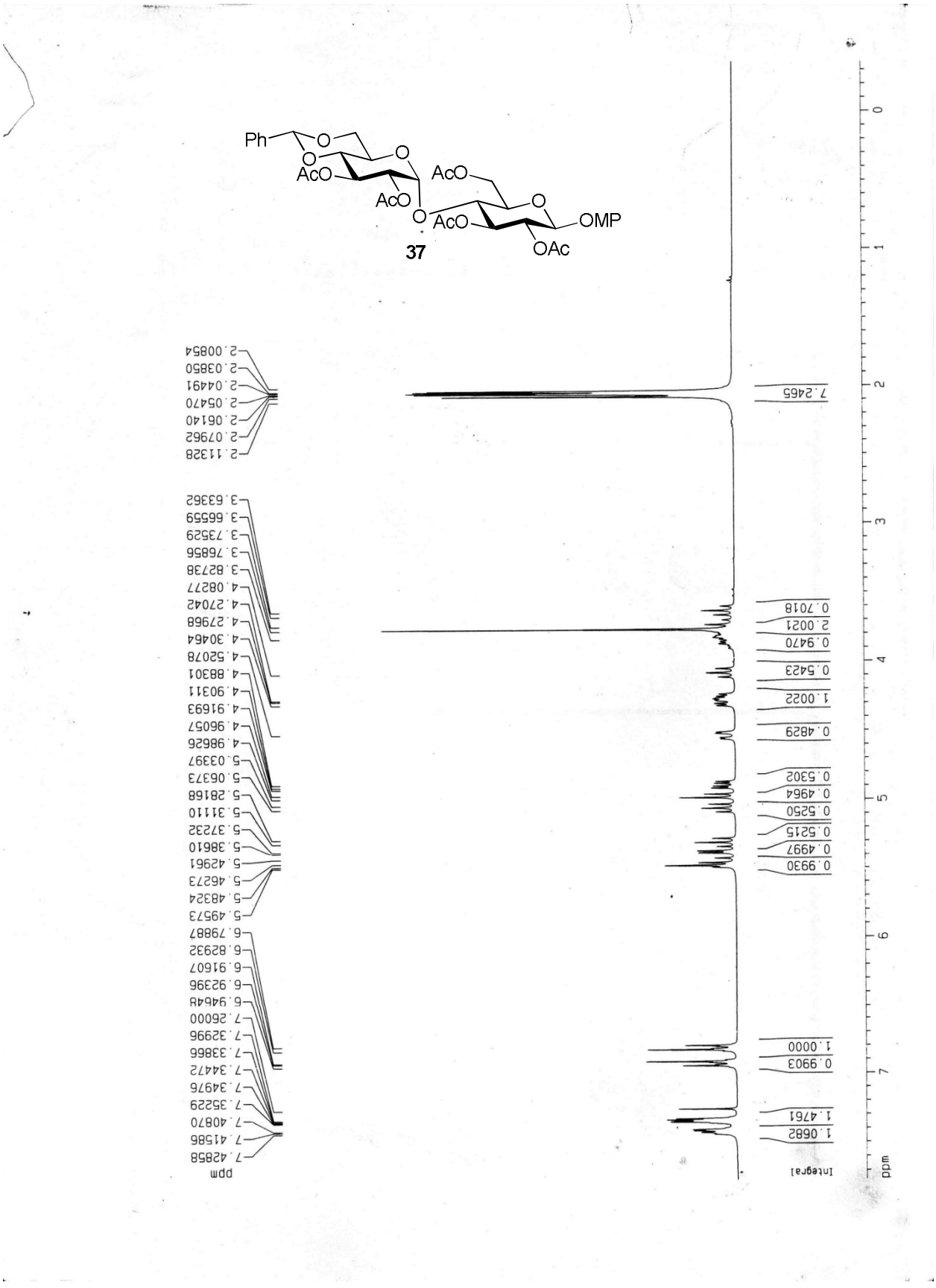
Integral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37
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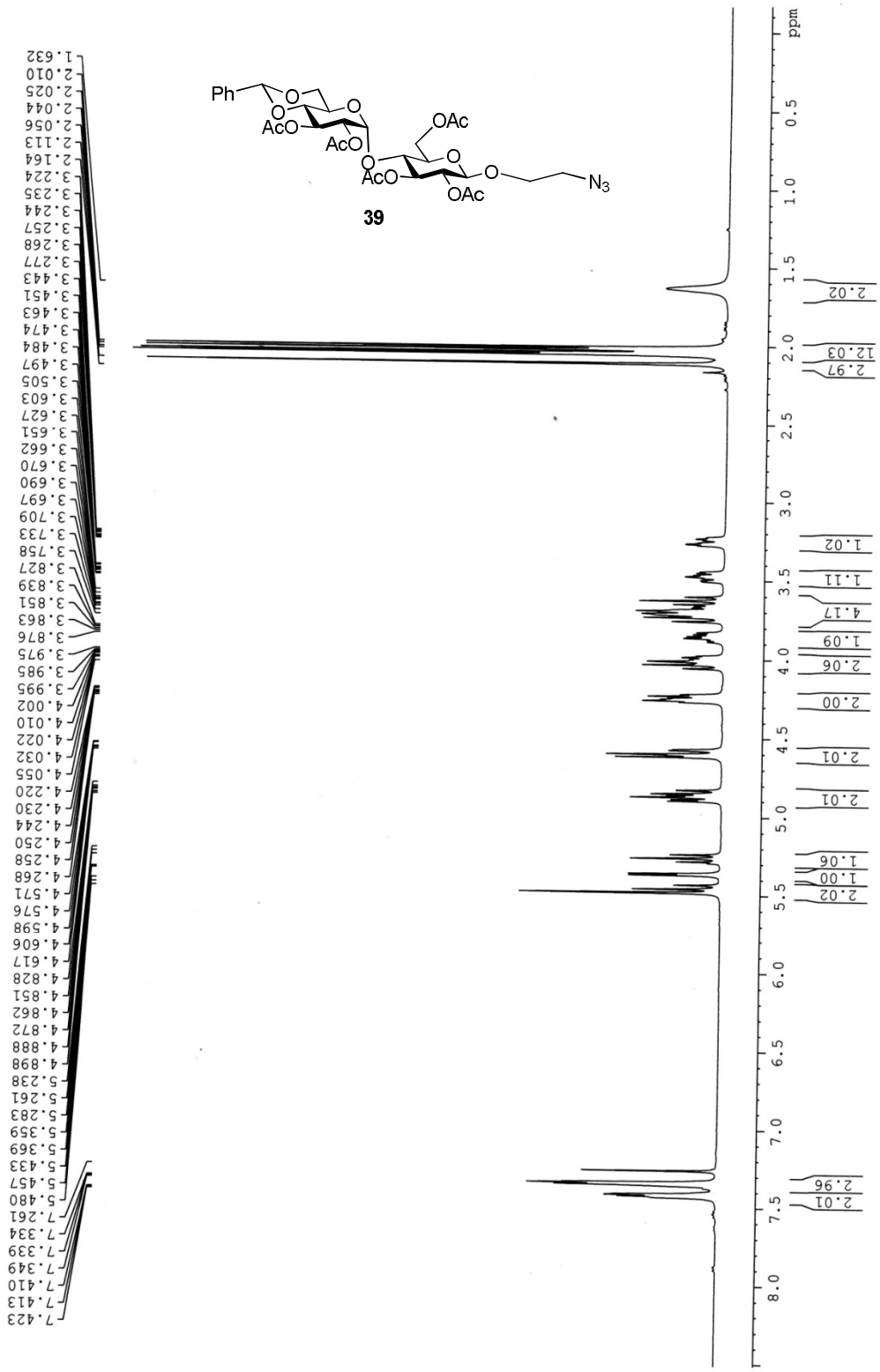
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3.670
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7.349
7.410
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7.423
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2.97
1.02
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1.00
2.02
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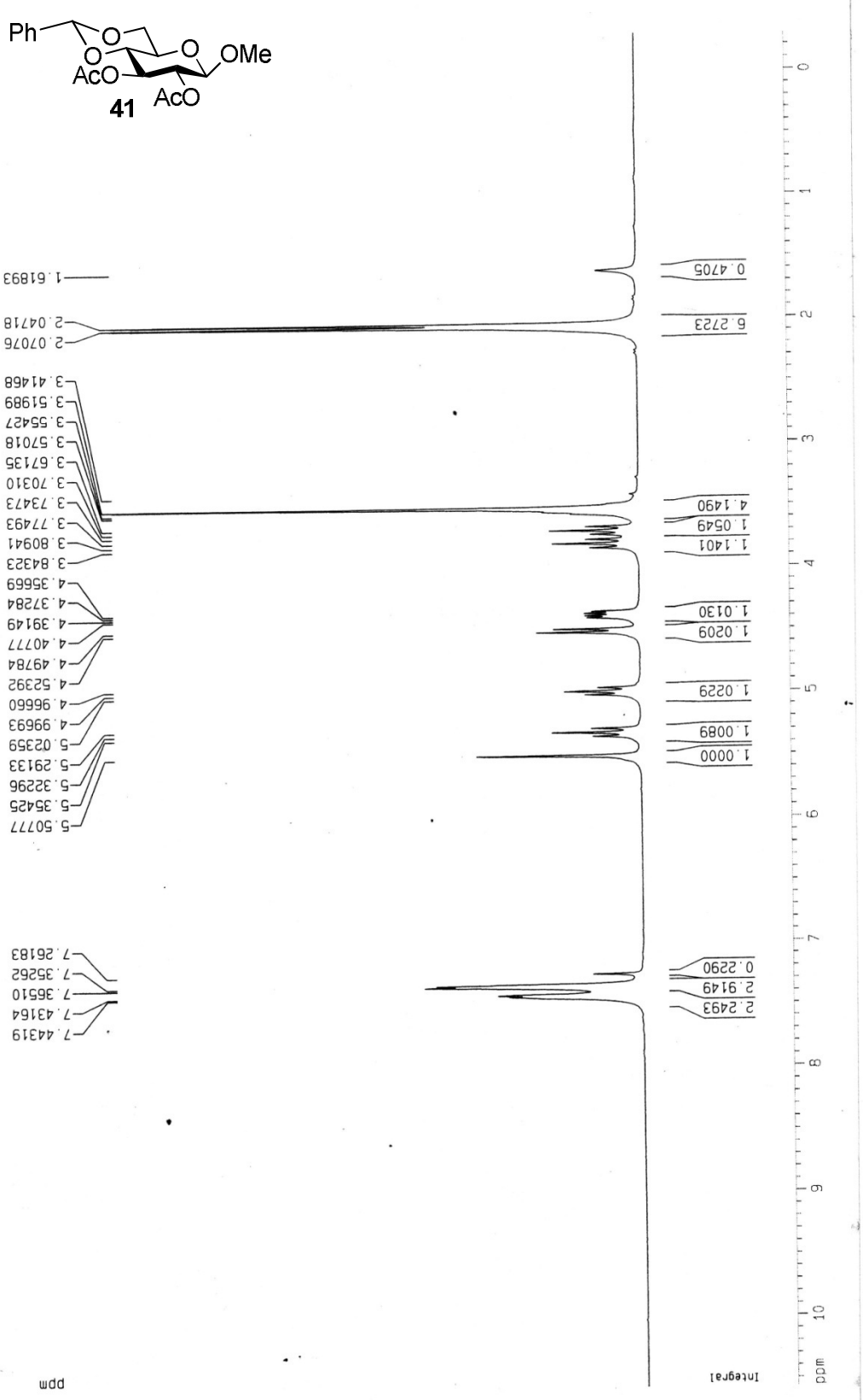
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OMe
AcO
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2.2493
ppm
Integral