10.1071/CH14144_AC

Australian Journal of Chemistry 2014, 67(7), 1017-1020

SUPPLEMENTARY MATERIAL

Development of Novel Guanidine–Bisurea Bifunctional Organocatalyst and its Application to Asymmetric a–Hydroxylation of Tetralone–Derived b–Keto Esters

Minami Odagi, Kan Takayama, Kota Furukori, Tatsuya Watanabe, and Kazuo Nagasawa

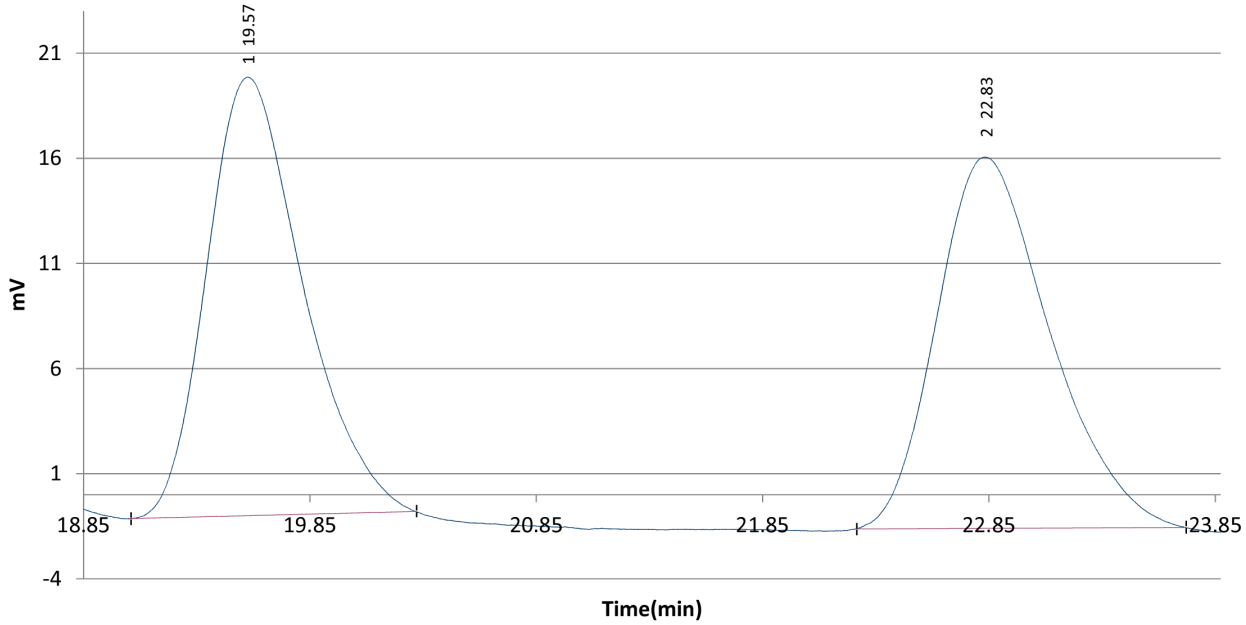
Table contents:

HPLC date for the products **3a-3g** S2–S

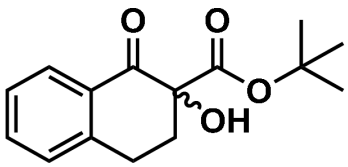
^{1}H and ^{13}C NMR date for new Guanidine-Urea bifunctional organocatalysts **4a–4e** S–S

^{1}H and ^{13}C NMR date for the product **7** S–S

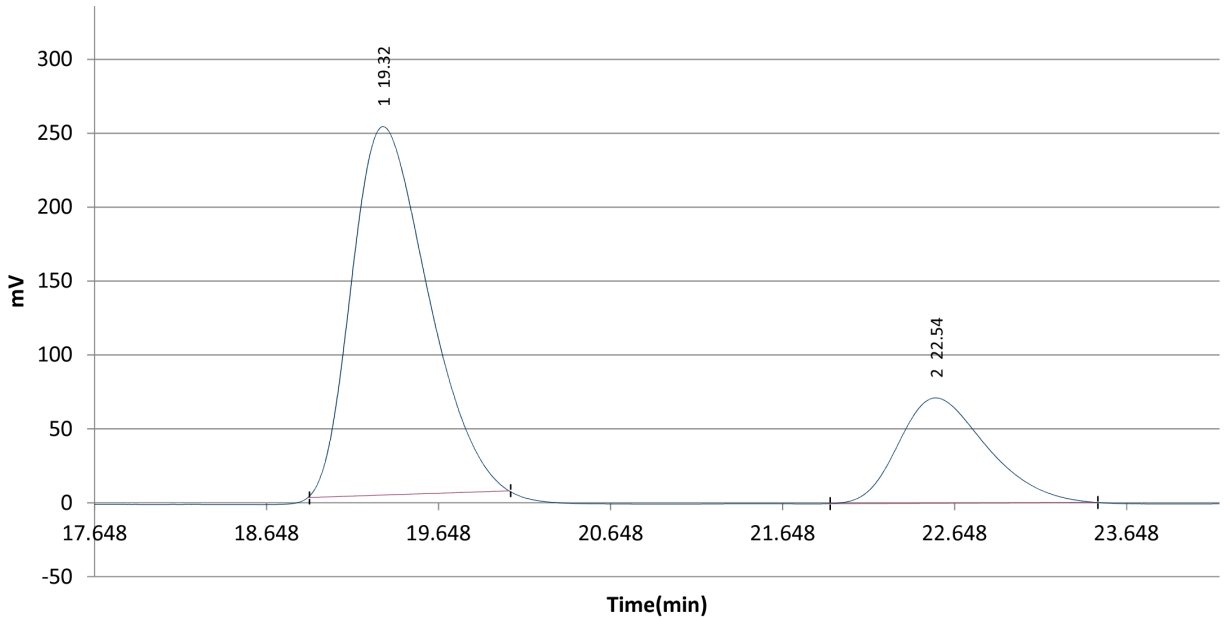
HPLC date for the products 3a-3g



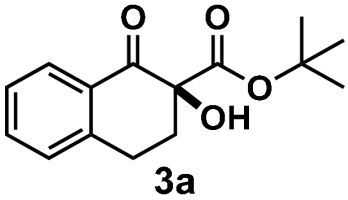
comment AD-H Hexane:IPA=95:5 1.0mL/min



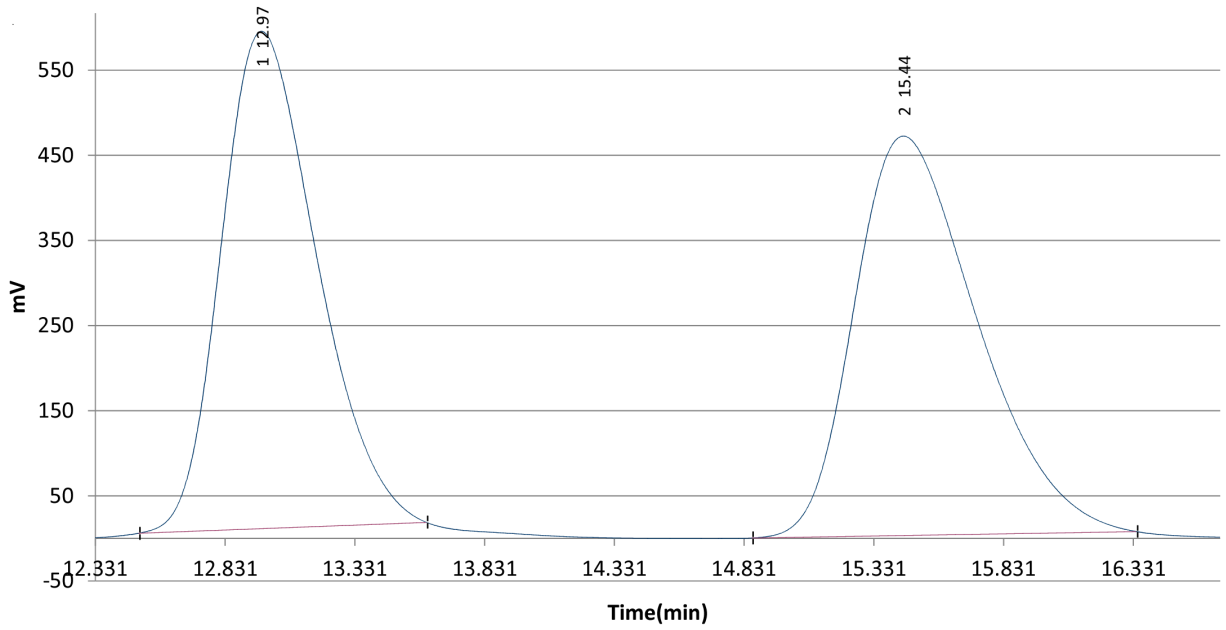
No.	Rt	Area	Area(%)
1	19.57	612786.4	50.0614
2	22.83	611284.2	49.9386



comment AD-H Hexane:IPA=95:5 1.0mL/min

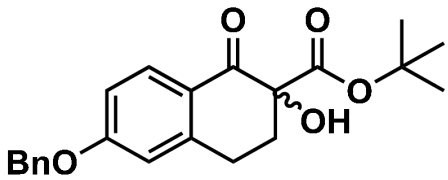


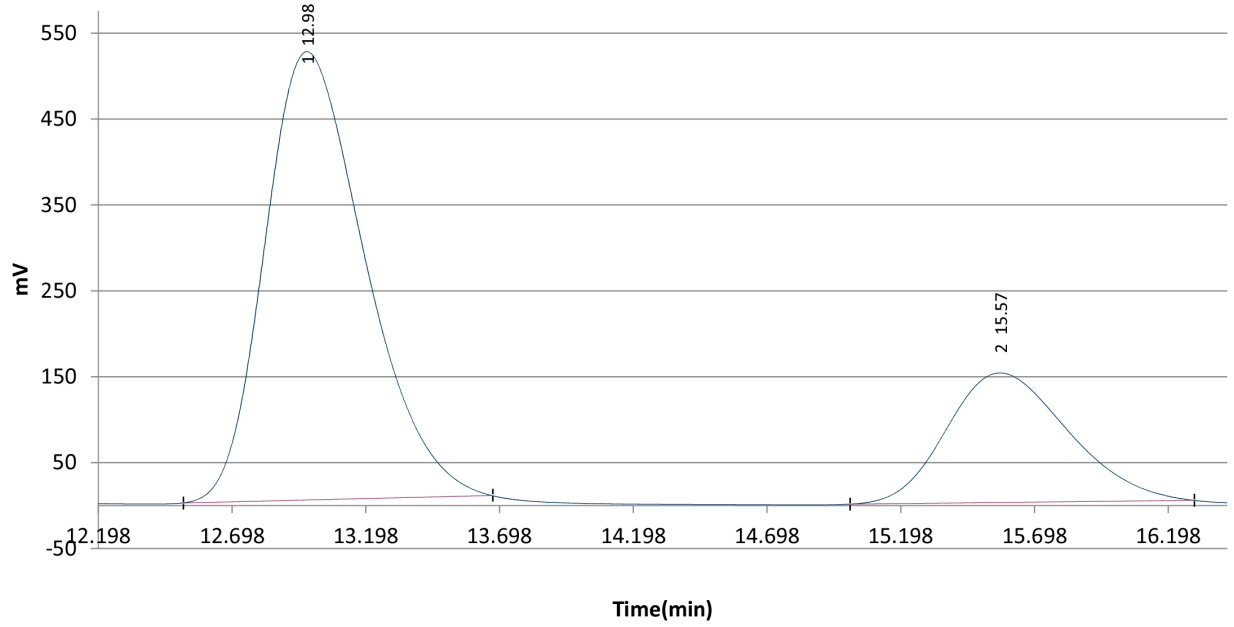
No.	Rt	Area	Area(%)
1	19.32	7675499	75.1047
2	22.54	2544231	24.8953



comment OD-H Hexane:IPA=90:10 1.0mL/min

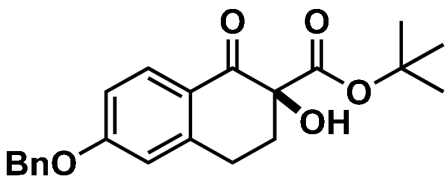
No.	Rt	Area	Area(%)
1	12.97	14934606	49.1886
2	15.44	15427302	50.8114



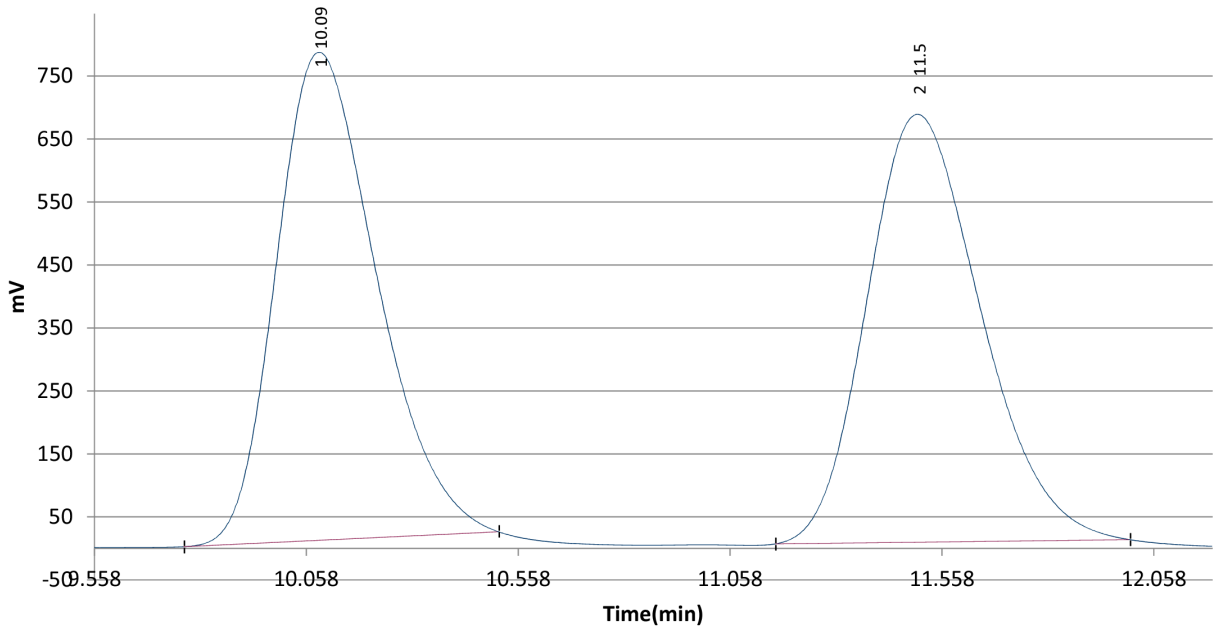


comment OD-H Hexane:IPA=90:10 1.0mL/min

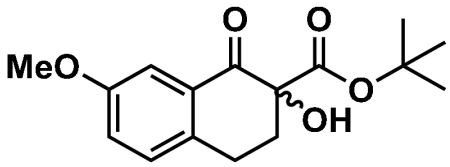
No.	Rt	Area	Area(%)
1	12.98	13568215	74.0817
2	15.57	4746995	25.9183



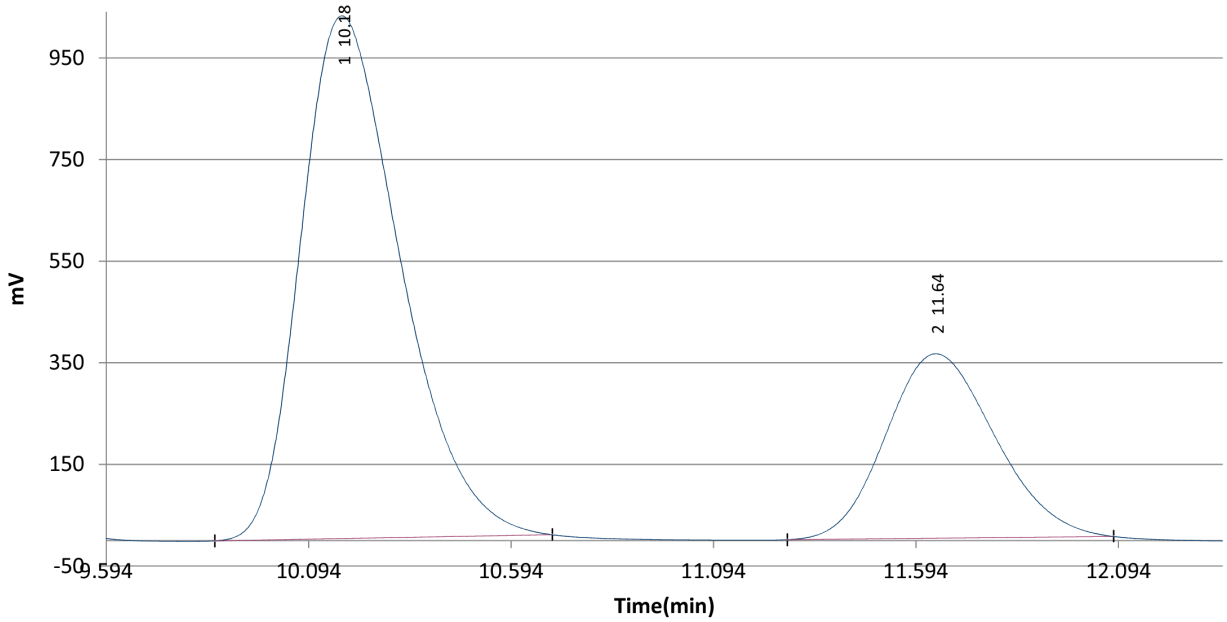
3b



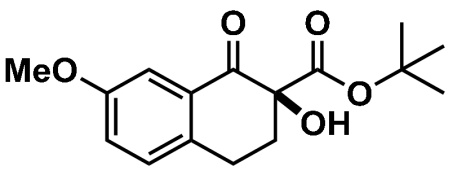
comment OD-H Hexane:IPA=97:3 1.0mL/min



No.	Rt	Area	Area(%)
1	10.09	12738601	50.14
2	11.5	12667450	49.86

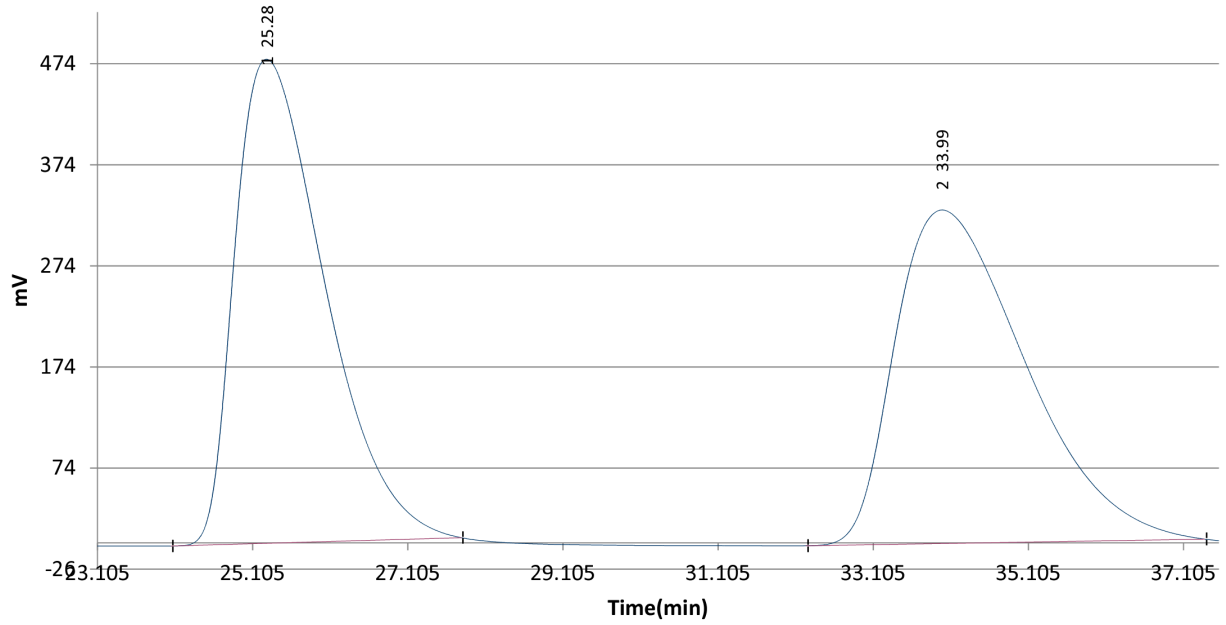


comment OD-H Hexane:IPA=97:3 1.0mL/min

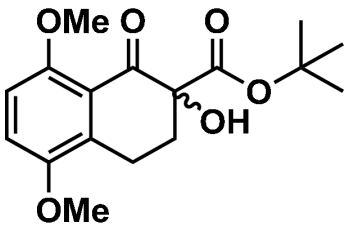


3c

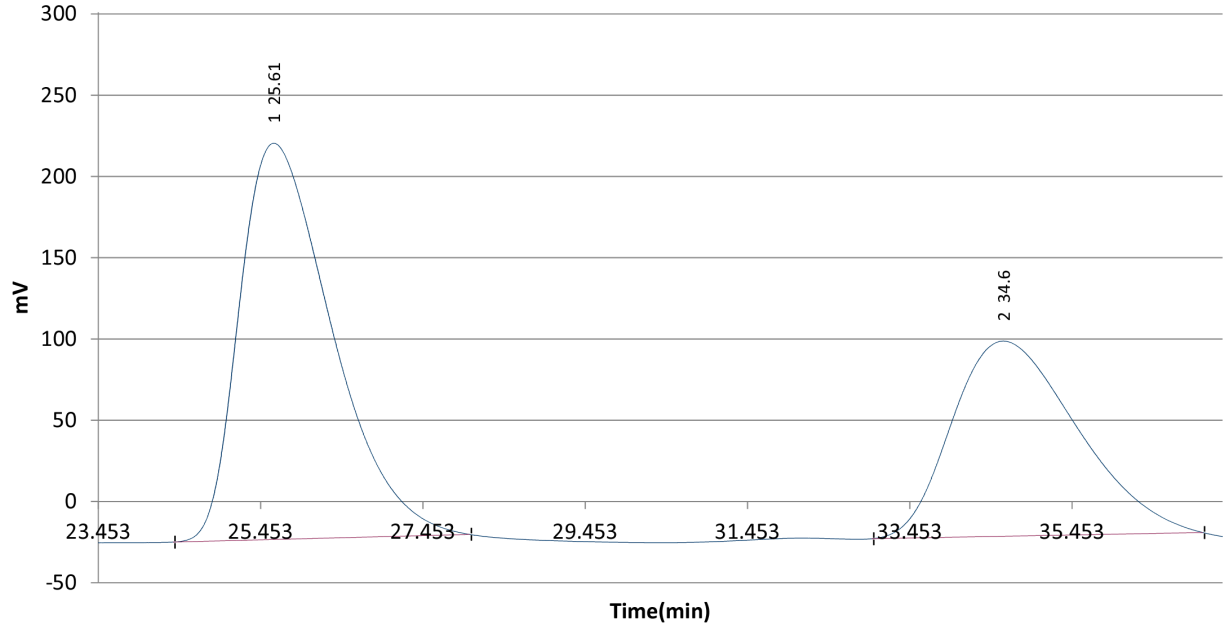
No.	Rt	Area	Area(%)
1	10.18	17611209	72.1853
2	11.64	6786016	27.8147



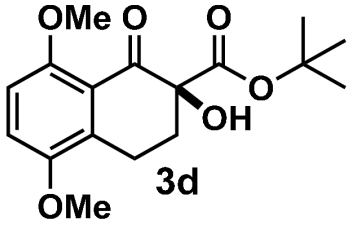
comment OJ-H Hexane:IPA=95:5 1.0mL/min



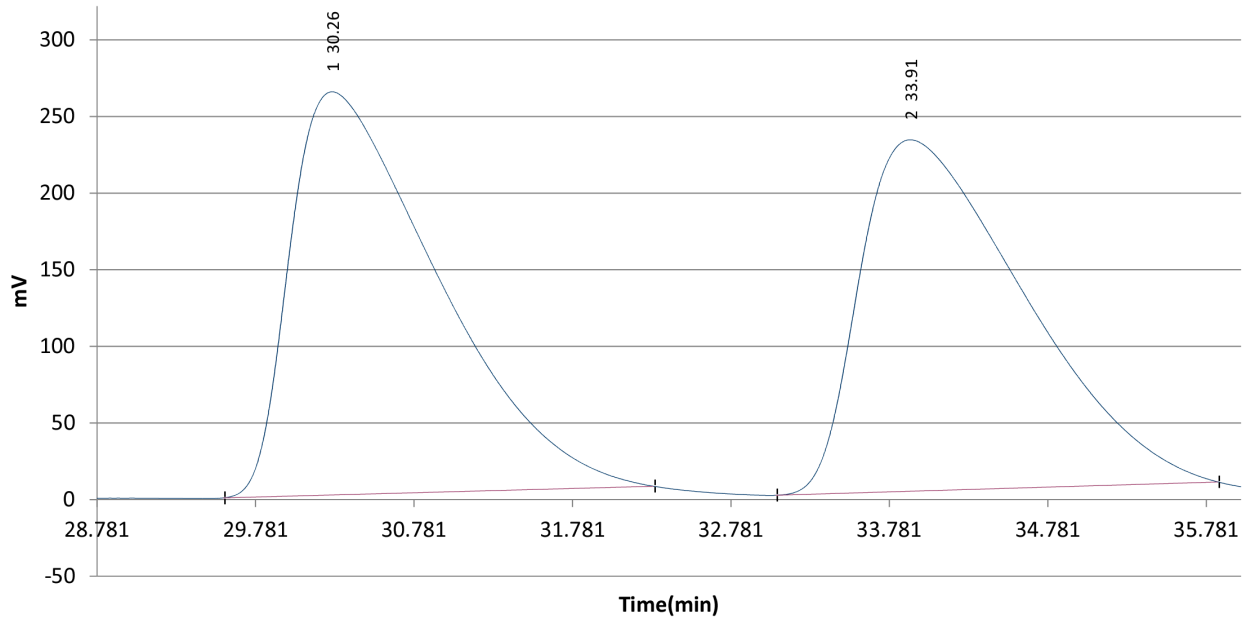
No.	Rt	Area	Area(%)
1	25.28	38493008	50.0504
2	33.99	38415558	49.9496



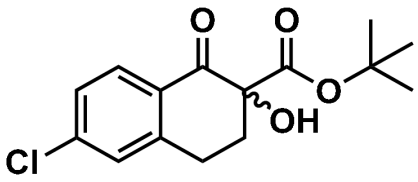
comment OJ-H Hexane:IPA=95:5 1.0mL/min



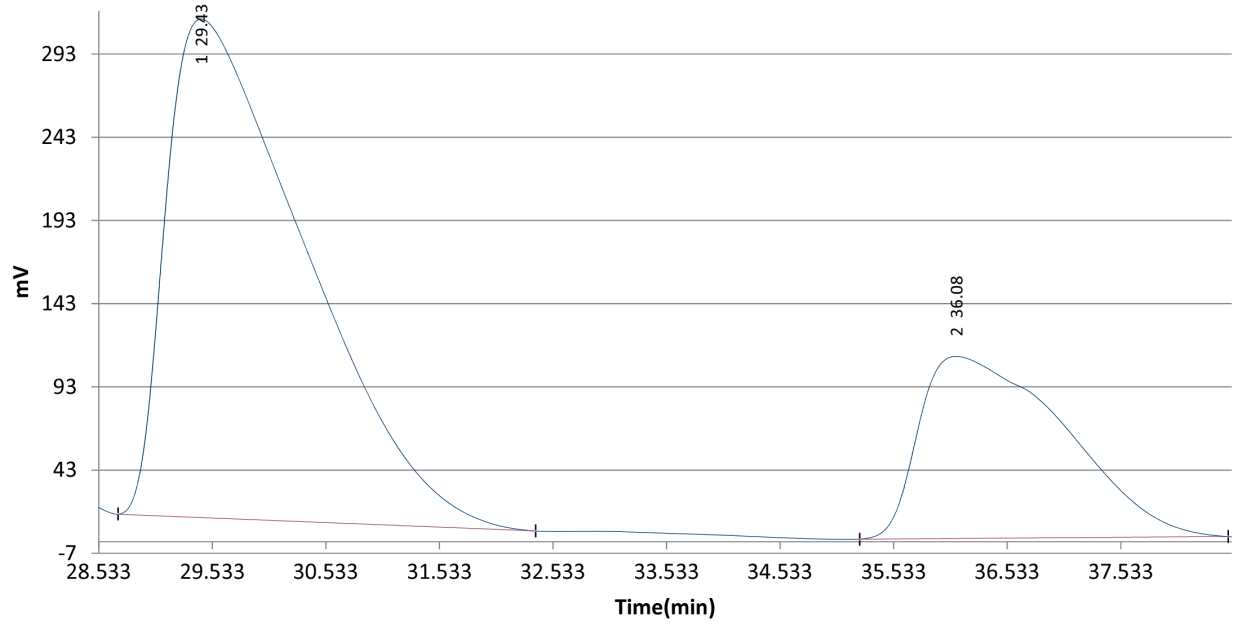
No.	Rt	Area	Area(%)
1	25.61	19245492	60.0254
2	34.6	12816731	39.9746



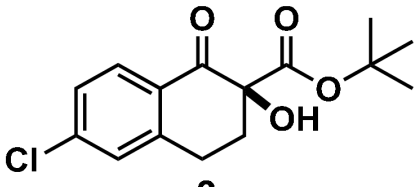
comment OD-H Hexane:IPA=99:0.3 0.993mL/min



No.	Rt	Area	Area(%)
1	30.26	17016404	50.7627
2	33.91	16505076	49.2373

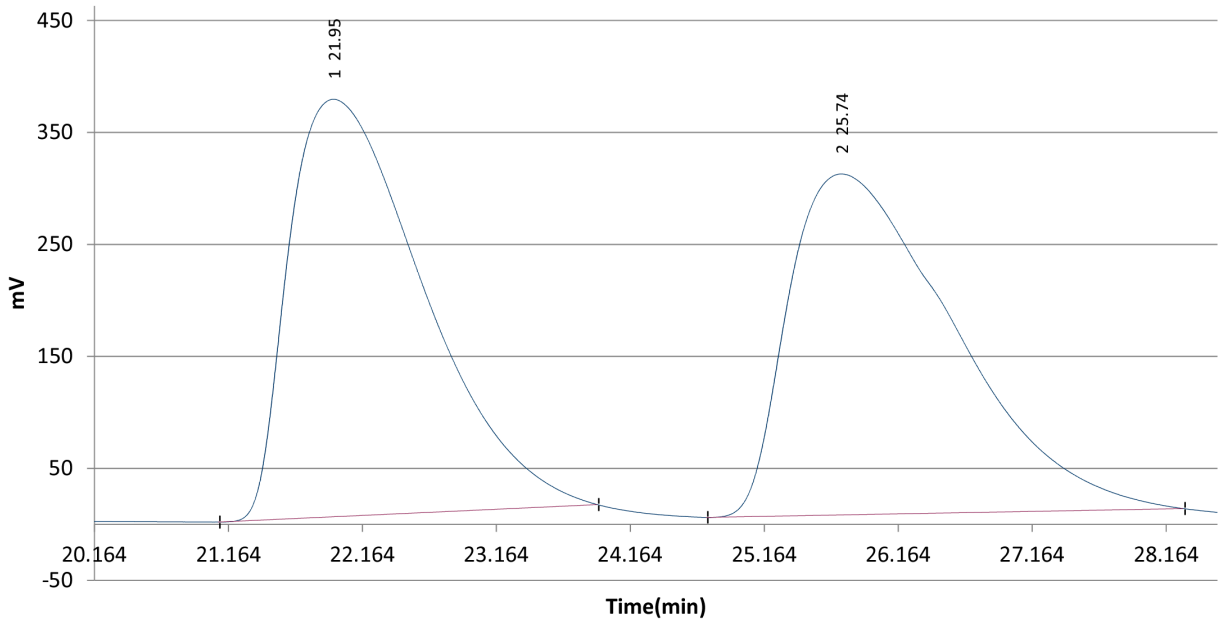


comment OD-H Hexane:IPA=99:0.3 0.993mL/min

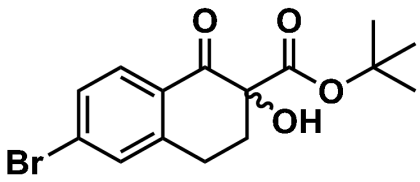


3e

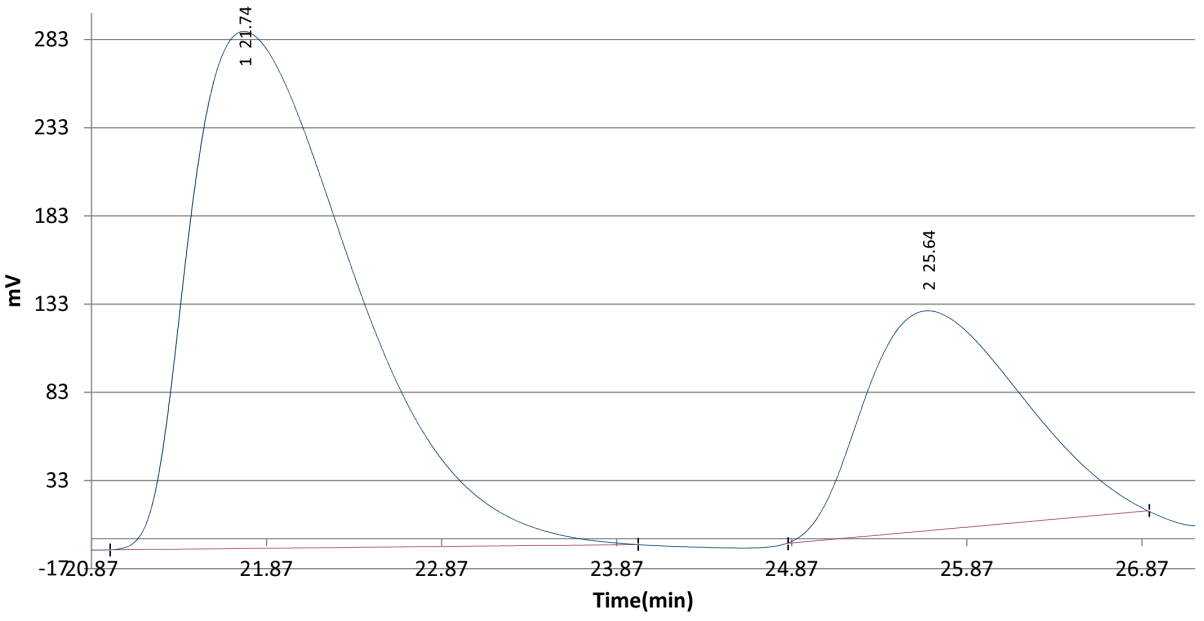
No.	Rt	Area	Area(%)
1	29.43	25566829	72.5026
2	36.08	9696522	27.4974



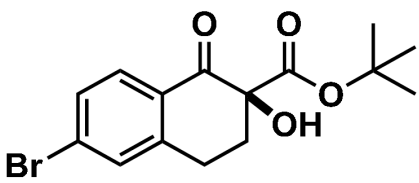
comment OJ-H Hexane:IPA=99:0.5 0.995mL/min



No.	Rt	Area	Area(%)
1	21.95	25997976	49.9749
2	25.74	26024125	50.0251

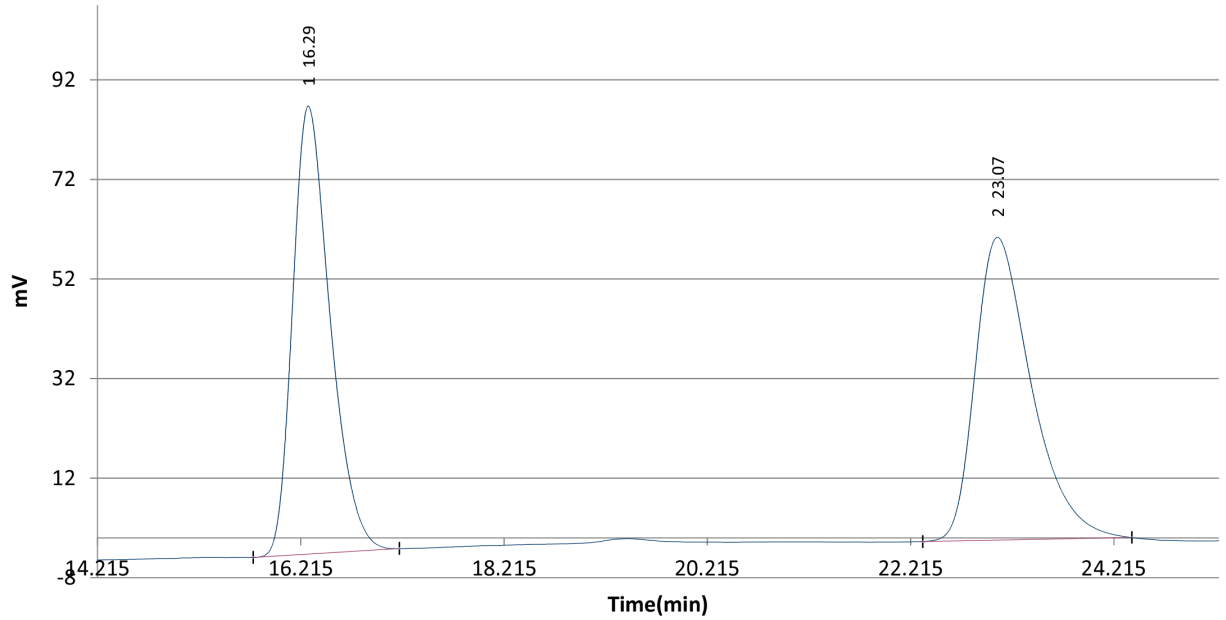


comment OJ-H Hexane:IPA=99:0.5 0.995mL/min



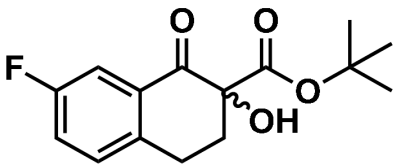
3f

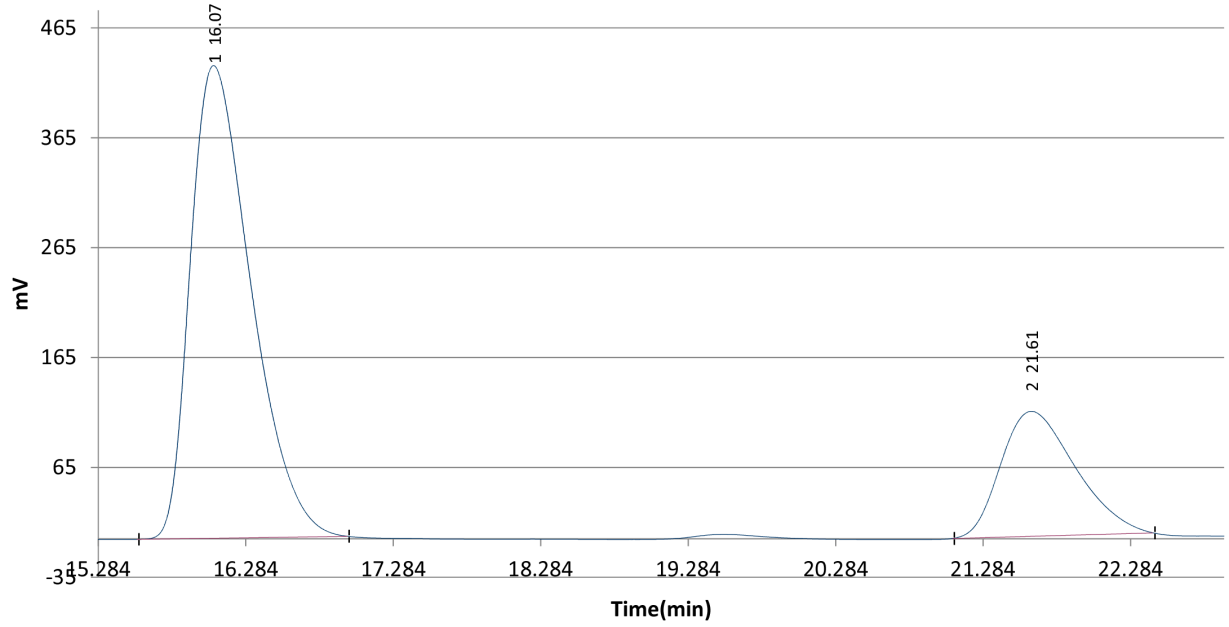
No.	Rt	Area	Area(%)
1	21.74	18917010	70.8897
2	25.64	7768139	29.1103



comment AD-H Hexane:IPA=95:5 1.0mL/min

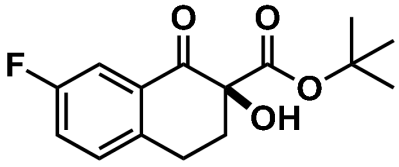
No.	Rt	Area	Area(%)
1	16.29	2307356	49.8171
2	23.07	2324301	50.1829





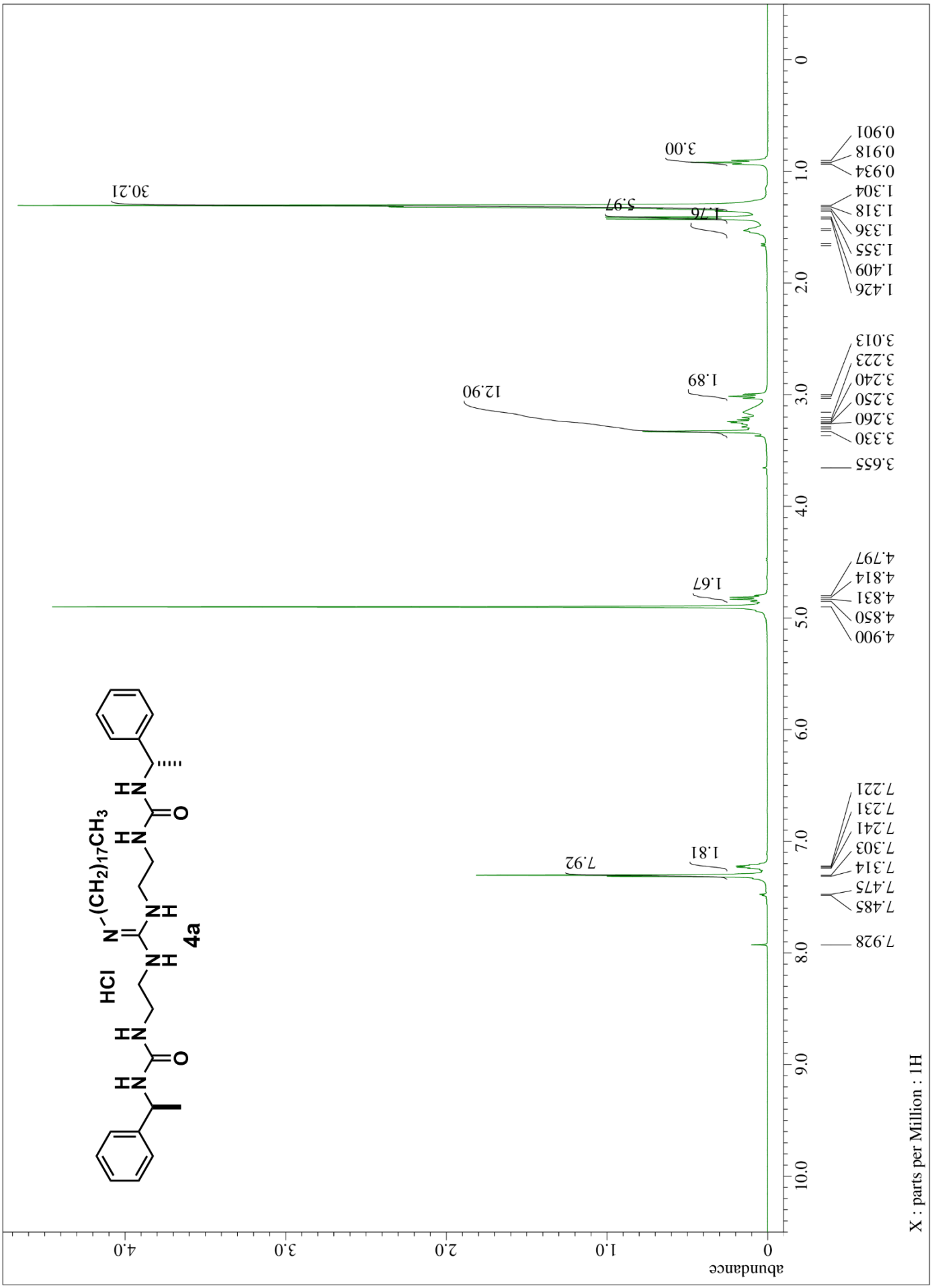
comment AD-H Hexane:IPA=95:5 1.0mL/min

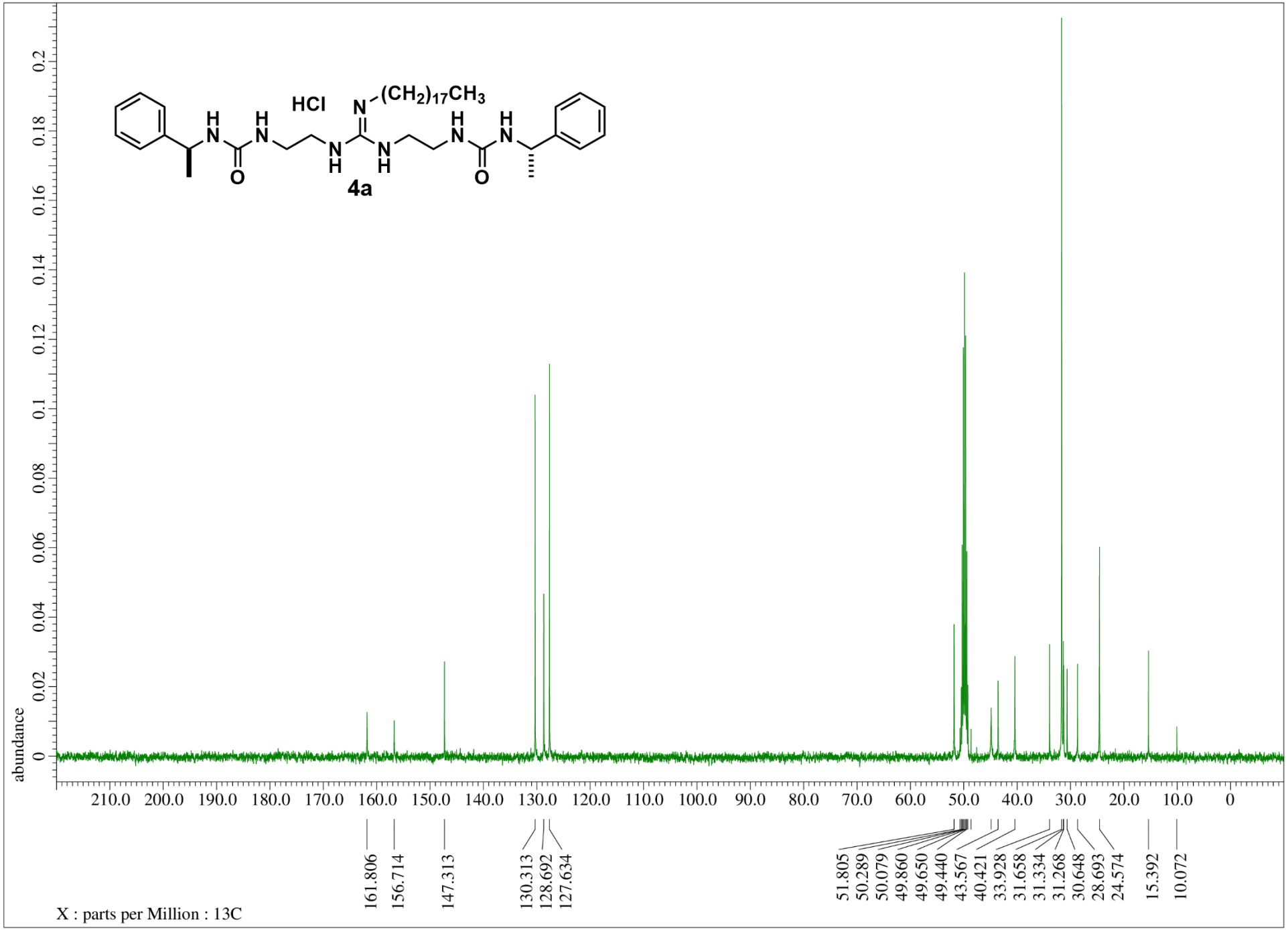
No.	Rt	Area	Area(%)
1	16.07	12278663	75.4993
2	21.61	3984607	24.5007

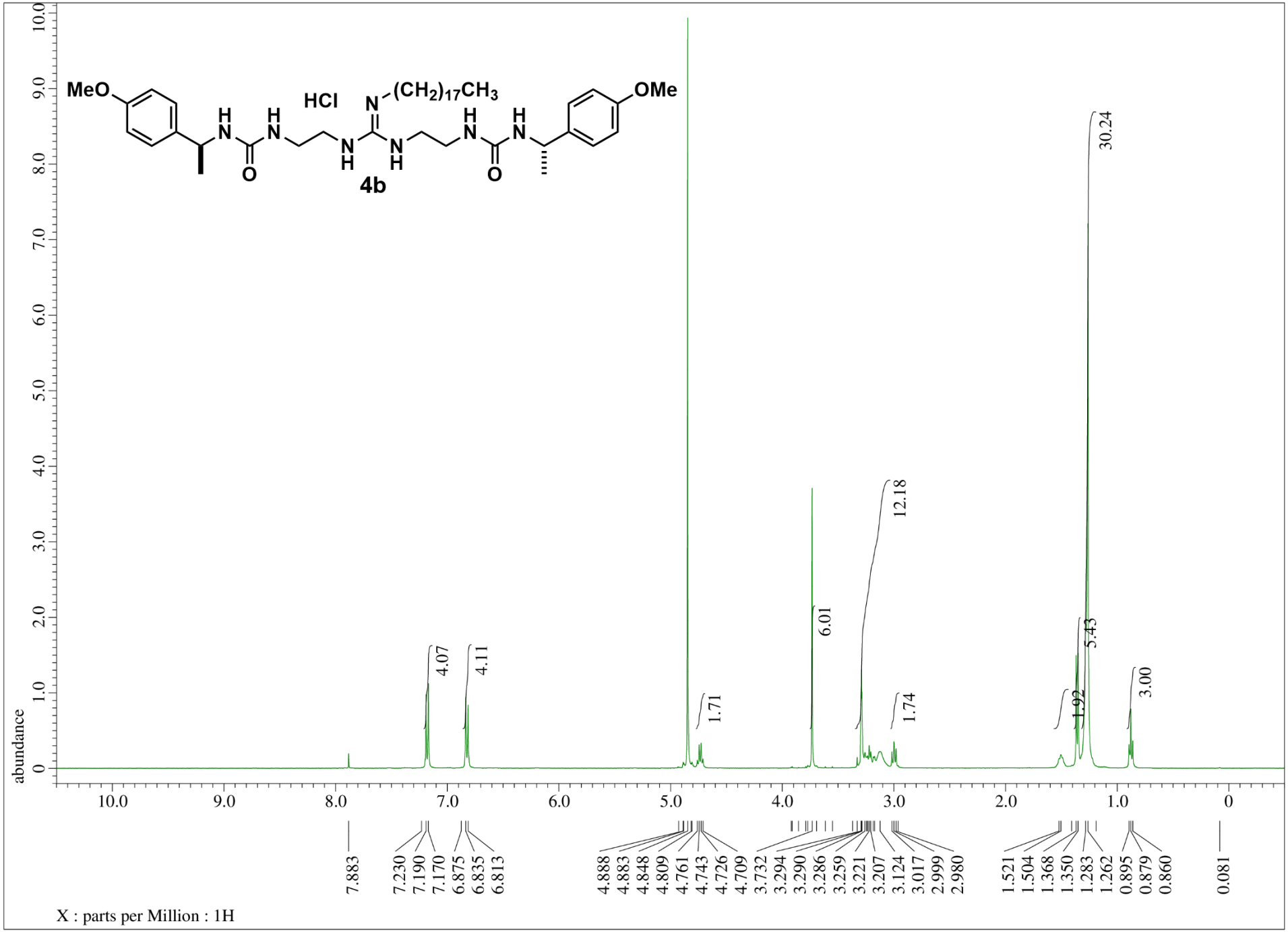


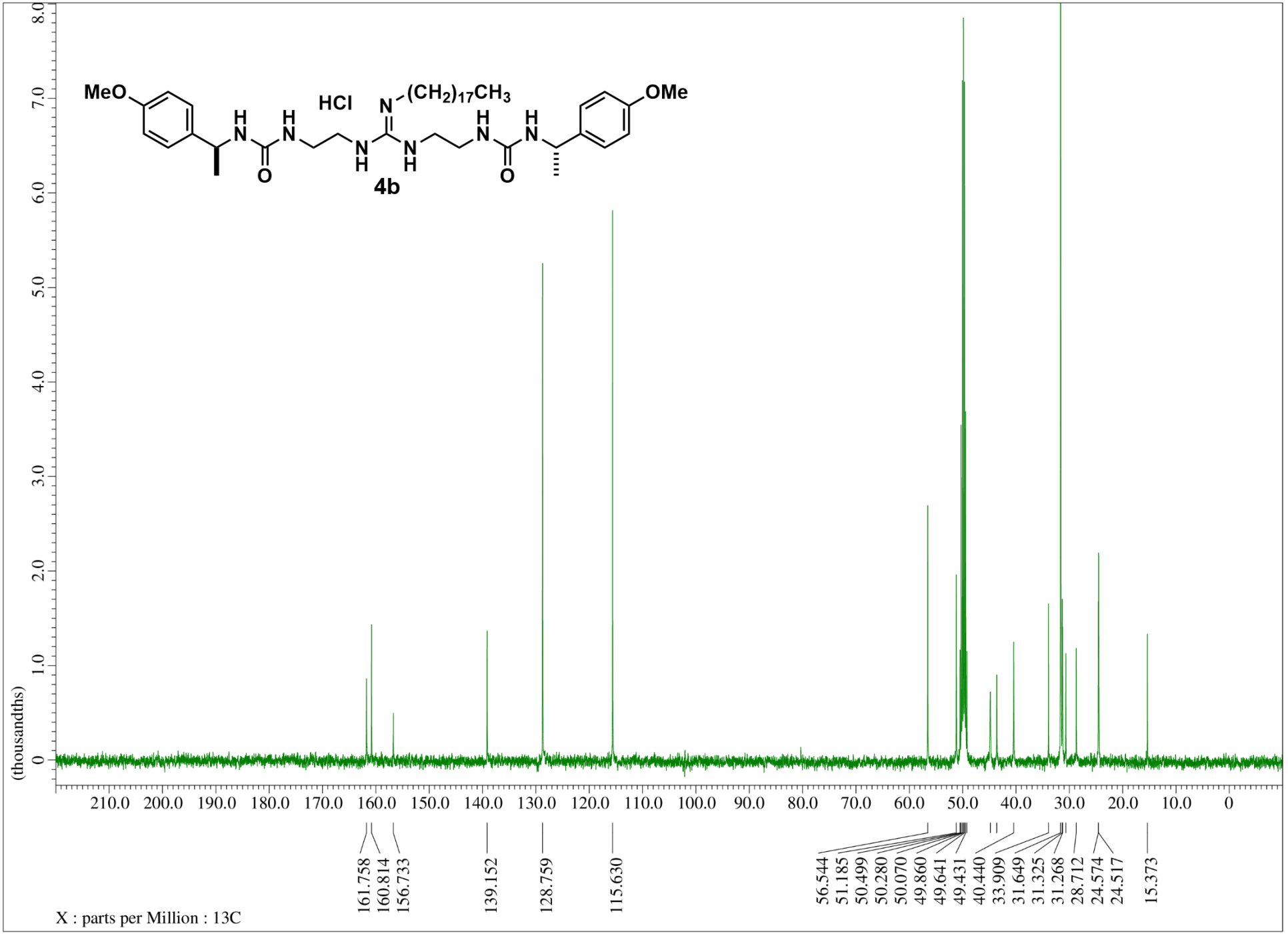
3g

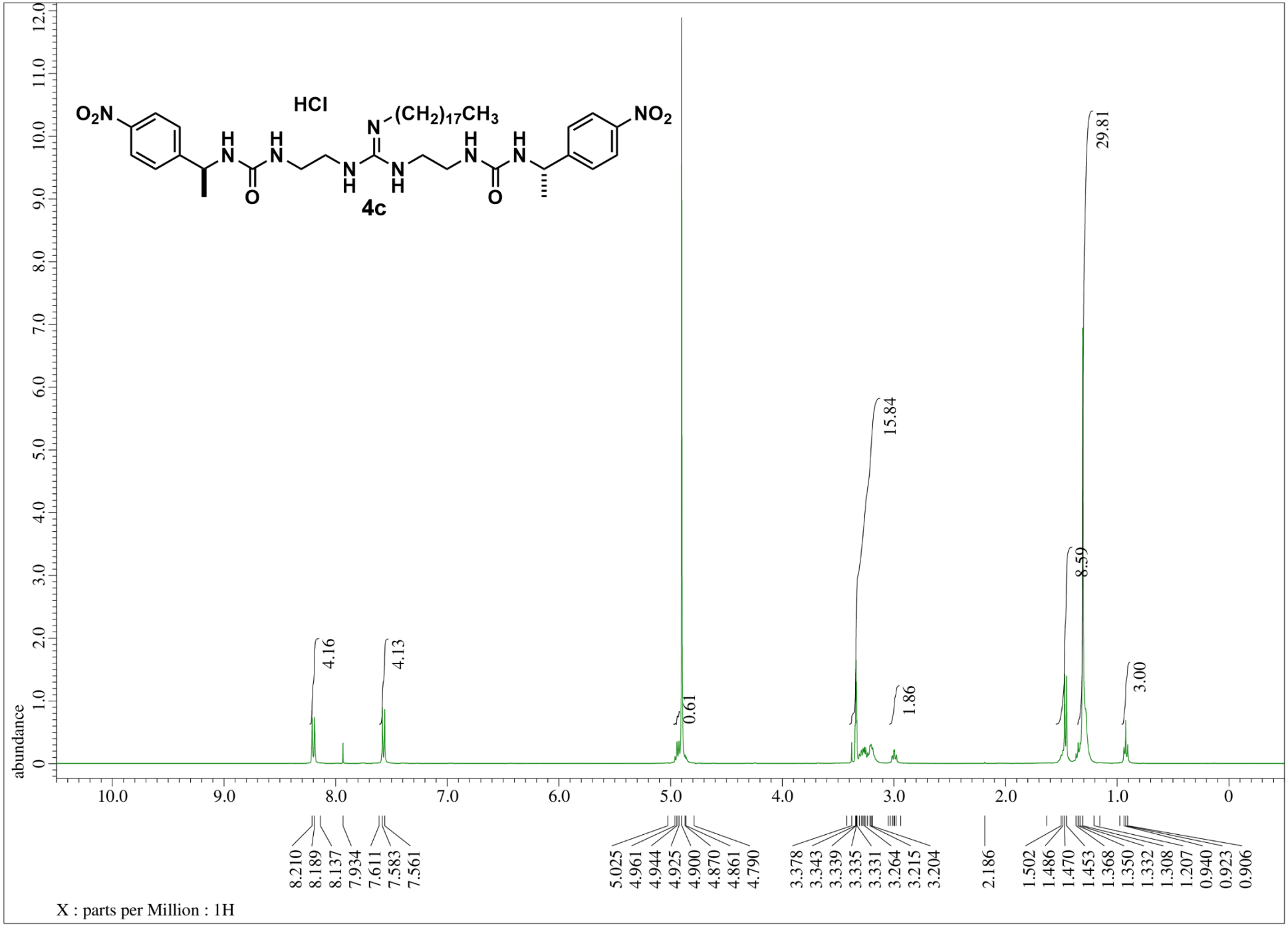
^{1}H and ^{13}C NMR date for new Guanidine-Urea bifunctional organocatalysts 4a–4e

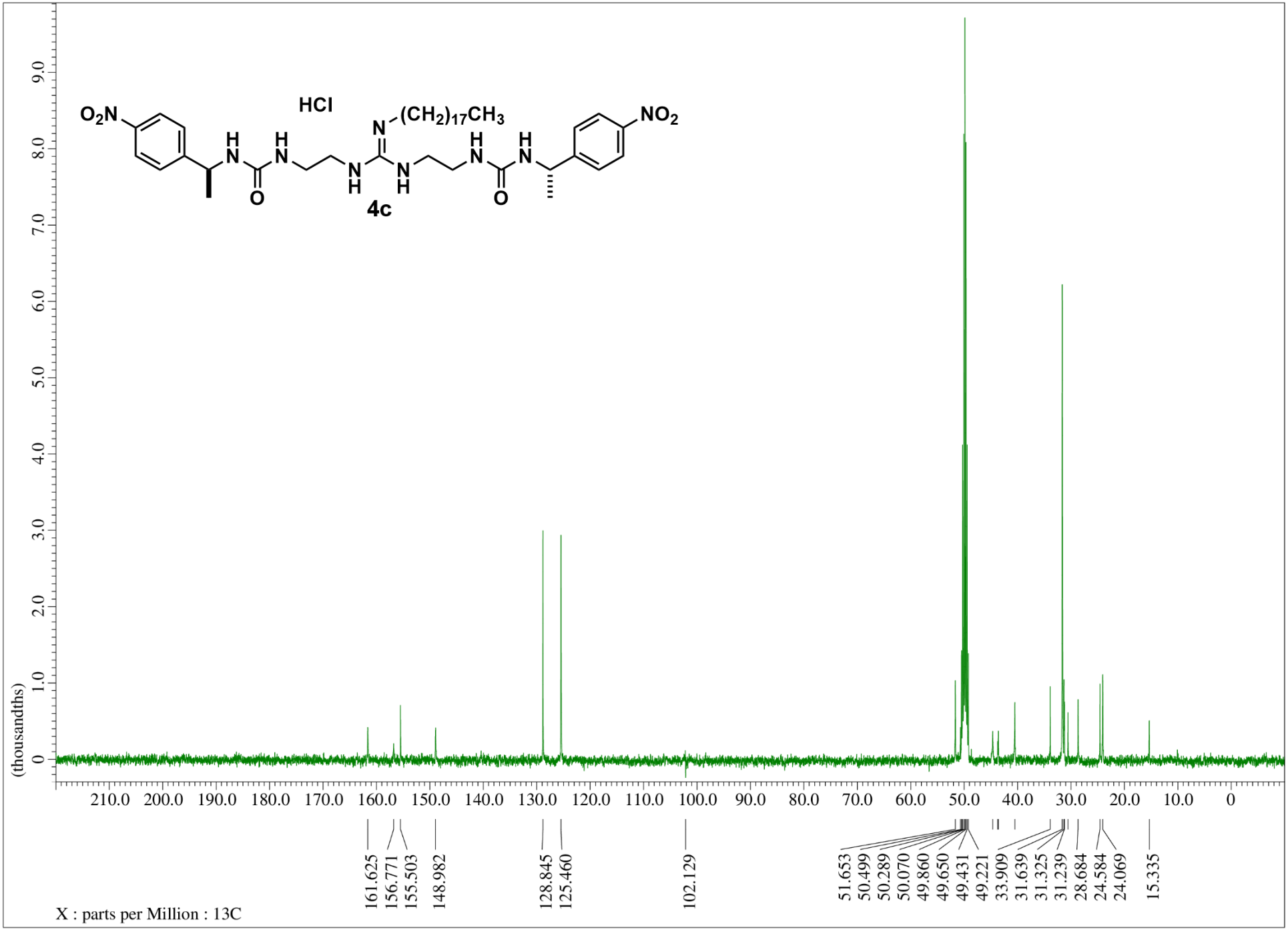


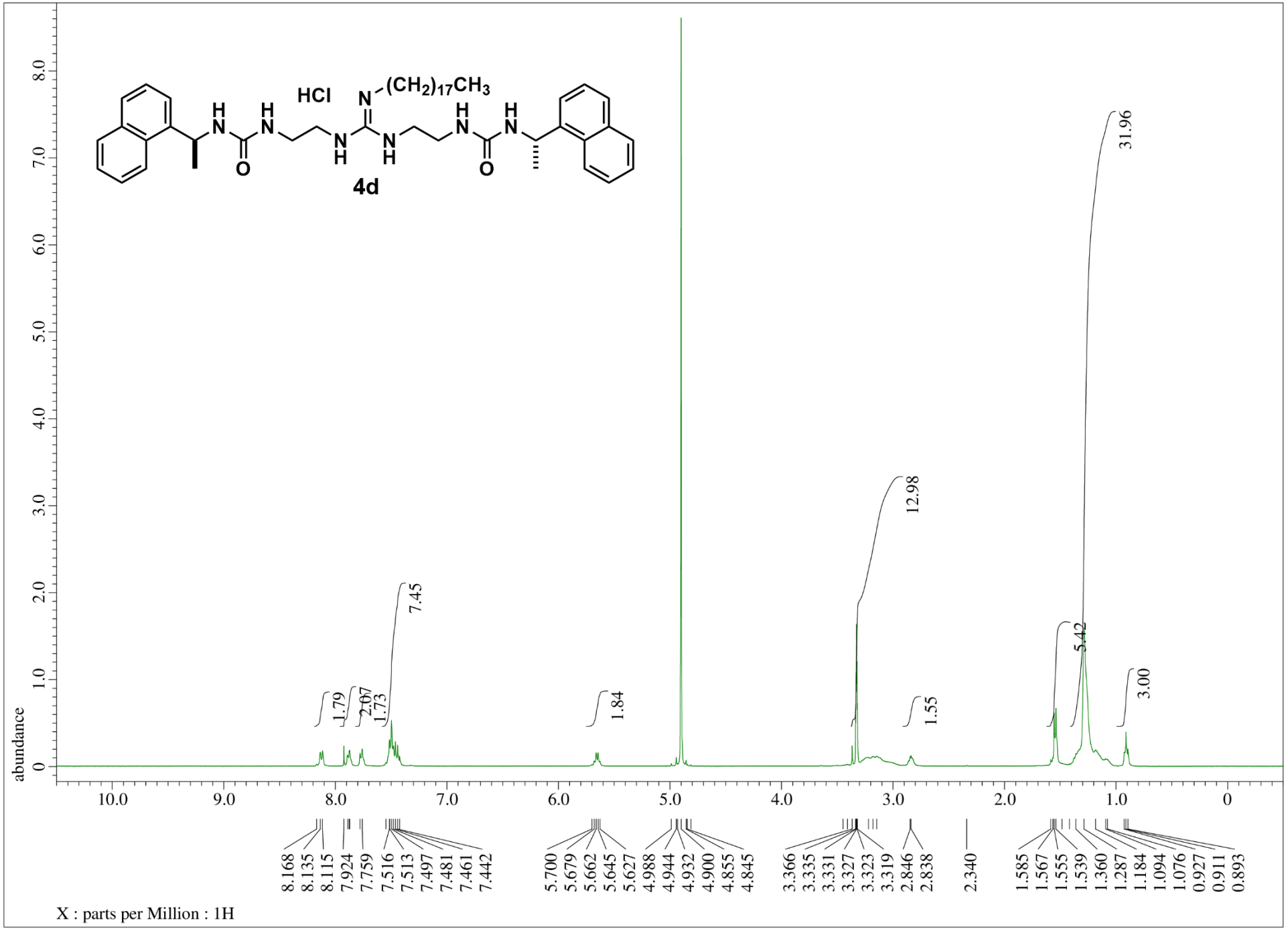

HCl
N
(CH2)17CH3
H
N
O
4a
abundance
0
0.02
0.04
0.06
0.08
0.1
0.12
0.14
0.16
0.18
0.2
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
161.806
156.714
147.313
130.313
128.692
127.634
51.805
50.289
50.079
49.860
49.650
49.440
43.567
40.421
33.928
31.658
31.334
31.268
30.648
28.693
24.574
15.392
10.072
X : parts per Million : 13C

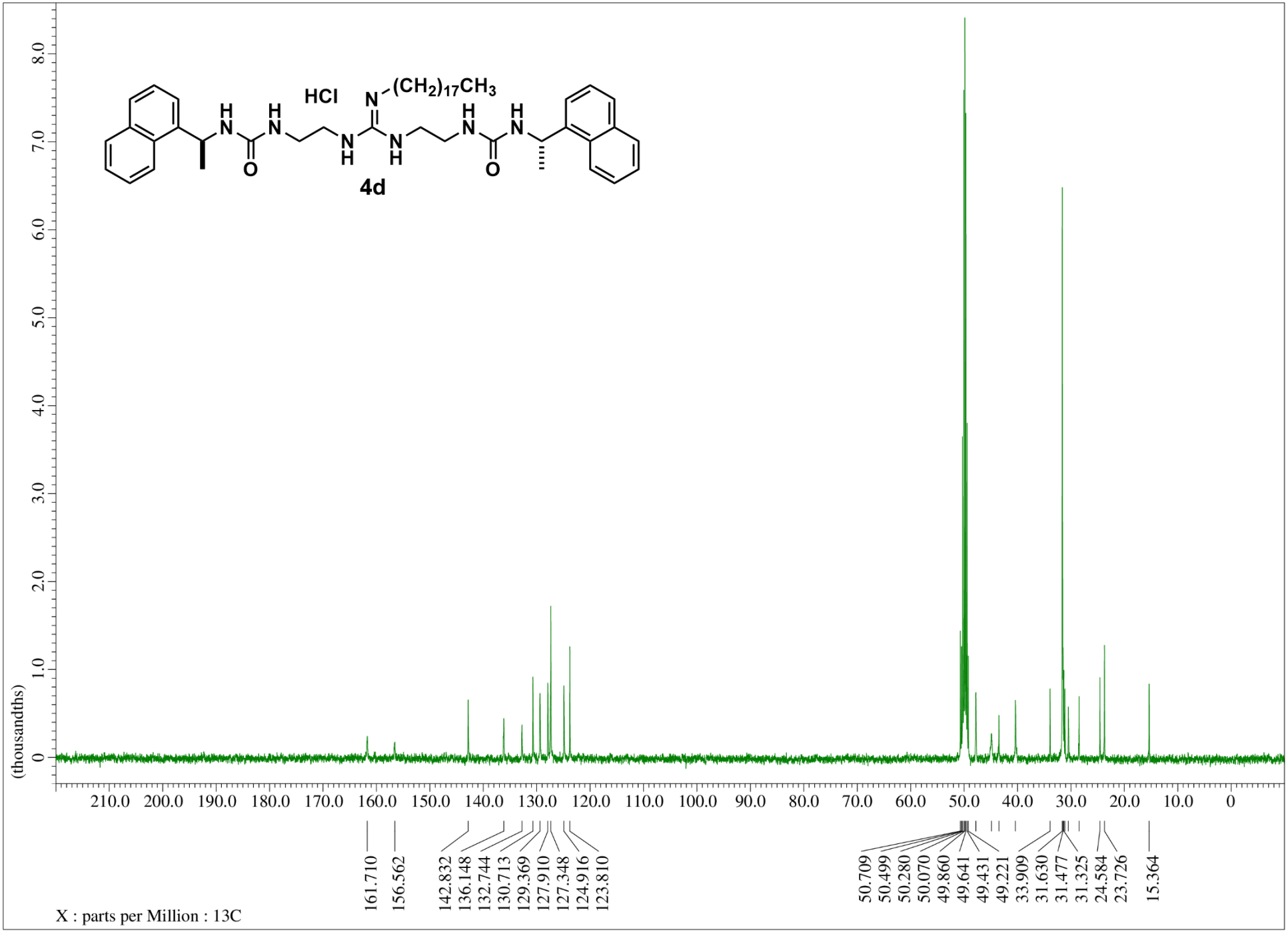

MeO
HCl
N
(CH2)17CH3
OMe
4b
abundance
30.24
12.18
6.01
5.43
4.07
4.11
1.71
1.74
1.92
3.00
7.883
7.230
7.190
7.170
6.875
6.835
6.813
4.888
4.883
4.848
4.809
4.761
4.743
4.726
4.709
3.732
3.294
3.290
3.286
3.259
3.221
3.207
3.124
3.017
2.999
2.980
1.521
1.504
1.368
1.350
1.283
1.262
0.895
0.879
0.860
0.081
X : parts per Million : 1H

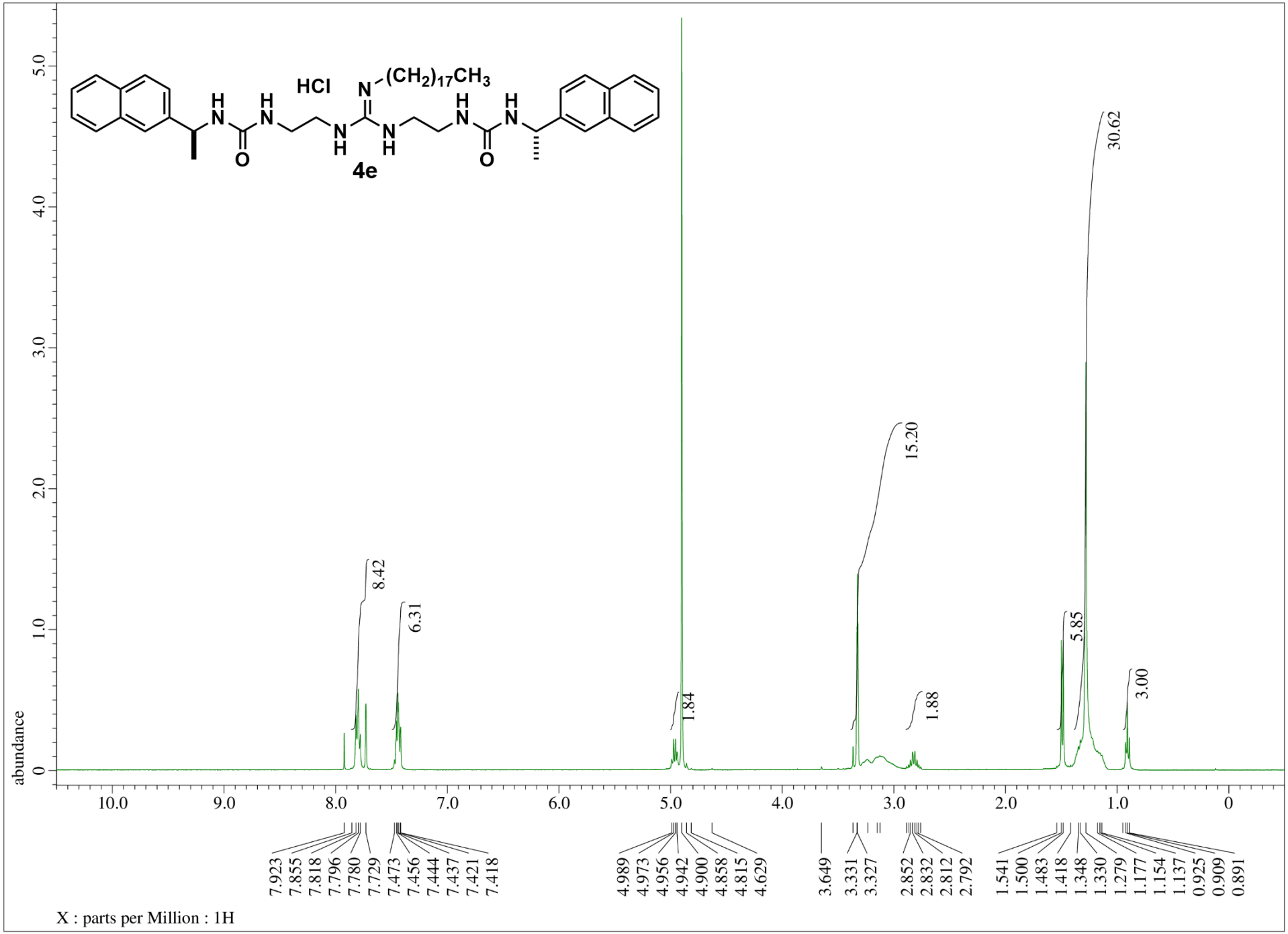

MeO
HCl
N
(CH2)17CH3
OMe
H
N
O
4b
(thousandths)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
161.758
160.814
156.733
139.152
128.759
115.630
56.544
51.185
50.499
50.280
50.070
49.860
49.641
49.431
40.440
33.909
31.649
31.325
31.268
28.712
24.574
24.517
15.373
X : parts per Million : 13C

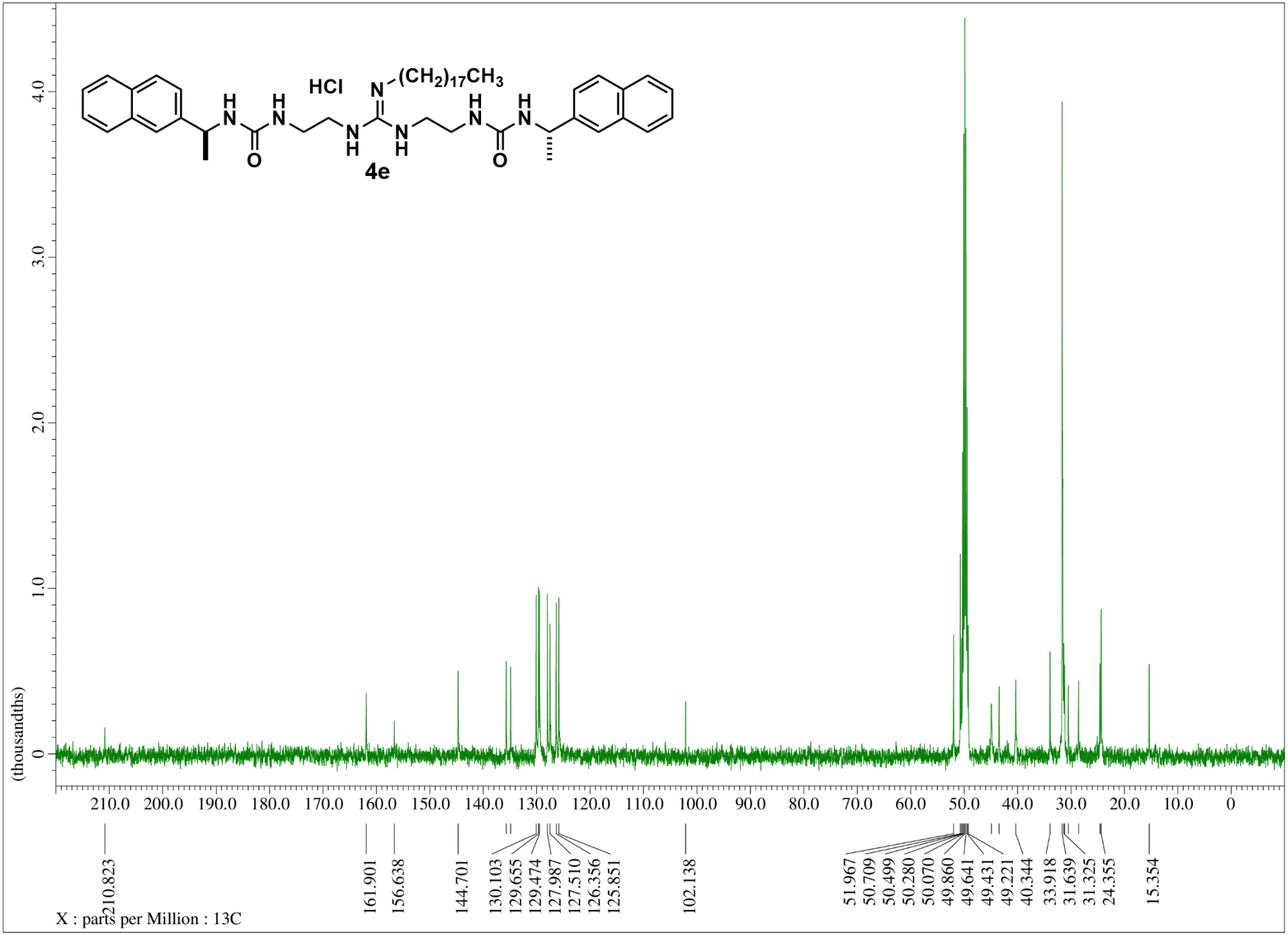

HCl
O_2N
NO_2
$(CH_2)_{17}CH_3$
4c
abundance
X : parts per Million : 1H
4.16
4.13
0.61
15.84
1.86
8.59
29.81
3.00
8.210
8.189
8.137
7.934
7.611
7.583
7.561
5.025
4.961
4.944
4.925
4.900
4.870
4.861
4.790
3.378
3.343
3.339
3.335
3.331
3.264
3.215
3.204
2.186
1.502
1.486
1.470
1.453
1.368
1.350
1.332
1.308
1.207
0.940
0.923
0.906


HCl
O_2N
NO_2
$(CH_2)_{17}CH_3$
4c
(thousandths)
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
161.625
156.771
155.503
148.982
128.845
125.460
102.129
51.653
50.499
50.289
50.070
49.860
49.650
49.431
49.221
33.909
31.639
31.325
31.239
28.684
24.584
24.069
15.335
X : parts per Million : 13C

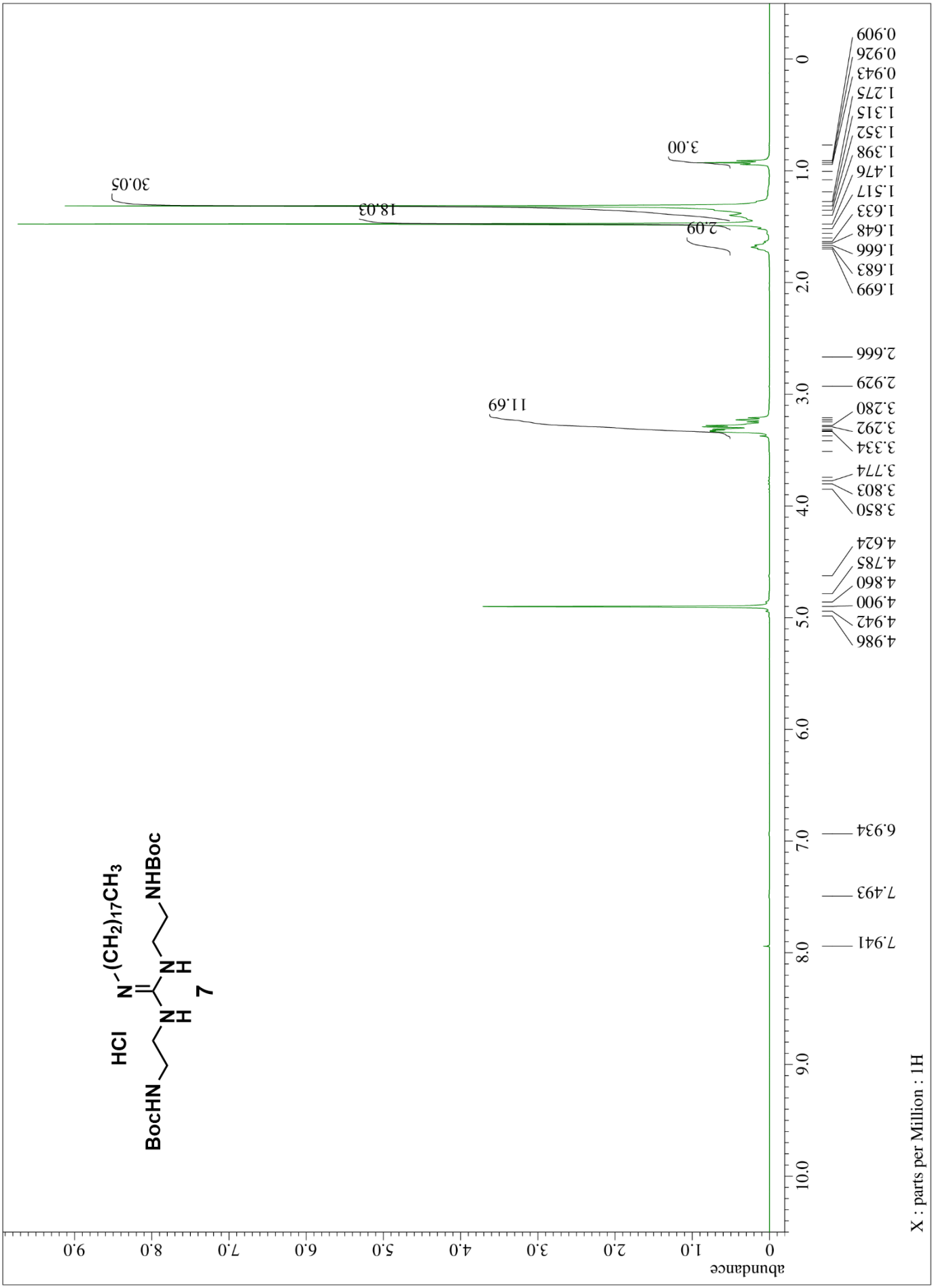

HCl
N
(CH2)17CH3
4d
abundance
X : parts per Million : 1H
1.79
2.07
1.73
7.45
1.84
12.98
1.55
5.42
31.96
3.00
8.168
8.135
8.115
7.924
7.759
7.516
7.513
7.497
7.481
7.461
7.442
5.700
5.679
5.662
5.645
5.627
4.988
4.944
4.932
4.900
4.855
4.845
3.366
3.335
3.331
3.327
3.323
3.319
2.846
2.838
2.340
1.585
1.567
1.555
1.539
1.360
1.287
1.184
1.094
1.076
0.927
0.911
0.893

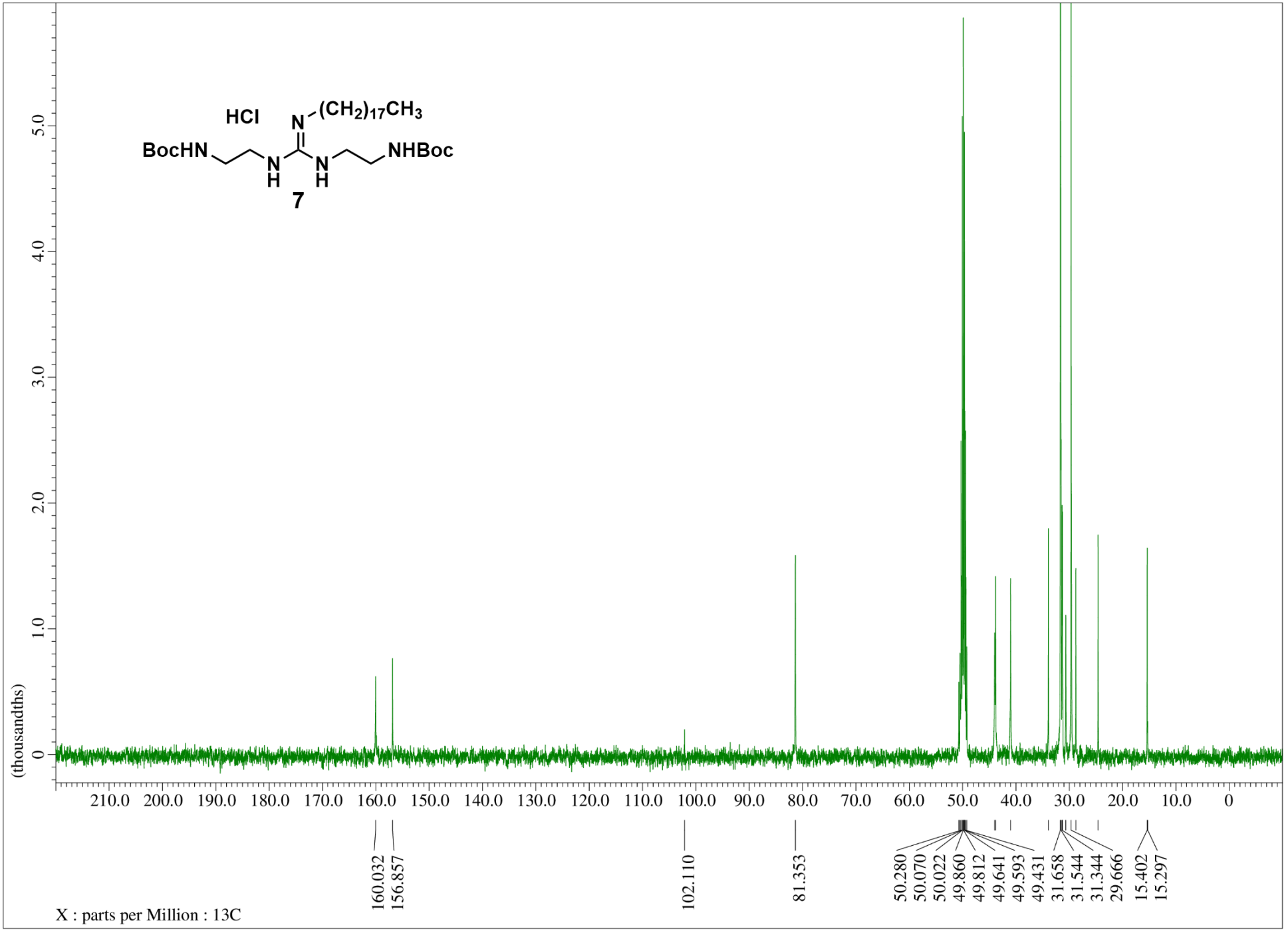

HCl
N
(CH2)17CH3
4d
(thousandths)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
161.710
156.562
142.832
136.148
132.744
130.713
129.369
127.910
127.348
124.916
123.810
50.709
50.499
50.280
50.070
49.860
49.641
49.431
49.221
33.909
31.630
31.477
31.325
24.584
23.726
15.364
X : parts per Million : 13C


HCl
N
(CH2)17CH3
H
N
O
N
H
4e
abundance
X : parts per Million : 1H
8.42
6.31
1.84
15.20
1.88
5.85
30.62
3.00
7.923
7.855
7.818
7.796
7.780
7.729
7.473
7.456
7.444
7.437
7.421
7.418
4.989
4.973
4.956
4.942
4.900
4.858
4.815
4.629
3.649
3.331
3.327
2.852
2.832
2.812
2.792
1.541
1.500
1.483
1.418
1.348
1.330
1.279
1.177
1.154
1.137
0.925
0.909
0.891


HCl
$(CH_2)_{17}CH_3$
4e
(thousandths)
4.0
3.0
2.0
1.0
0
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
210.823
161.901
156.638
144.701
130.103
129.655
129.474
127.987
127.510
126.356
125.851
102.138
51.967
50.709
50.499
50.280
50.070
49.860
49.641
49.431
49.221
40.344
33.918
31.639
31.325
24.355
15.354
X : parts per Million : 13C

^{1}H and ^{13}C NMR date for the product 7




HCl
N
(CH2)17CH3
BocHN
N
H
N
H
NHBoc
7
(thousandths)
5.0
4.0
3.0
2.0
1.0
0
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
160.032
156.857
102.110
81.353
50.280
50.070
50.022
49.860
49.812
49.641
49.593
49.431
31.658
31.544
31.344
29.666
15.402
15.297
X : parts per Million : 13C