Supporting Information for

Synthesis of Coumarins via PIDA/I_2-Mediated Oxidative Cyclization of Substituted Phenylacrylic Acids

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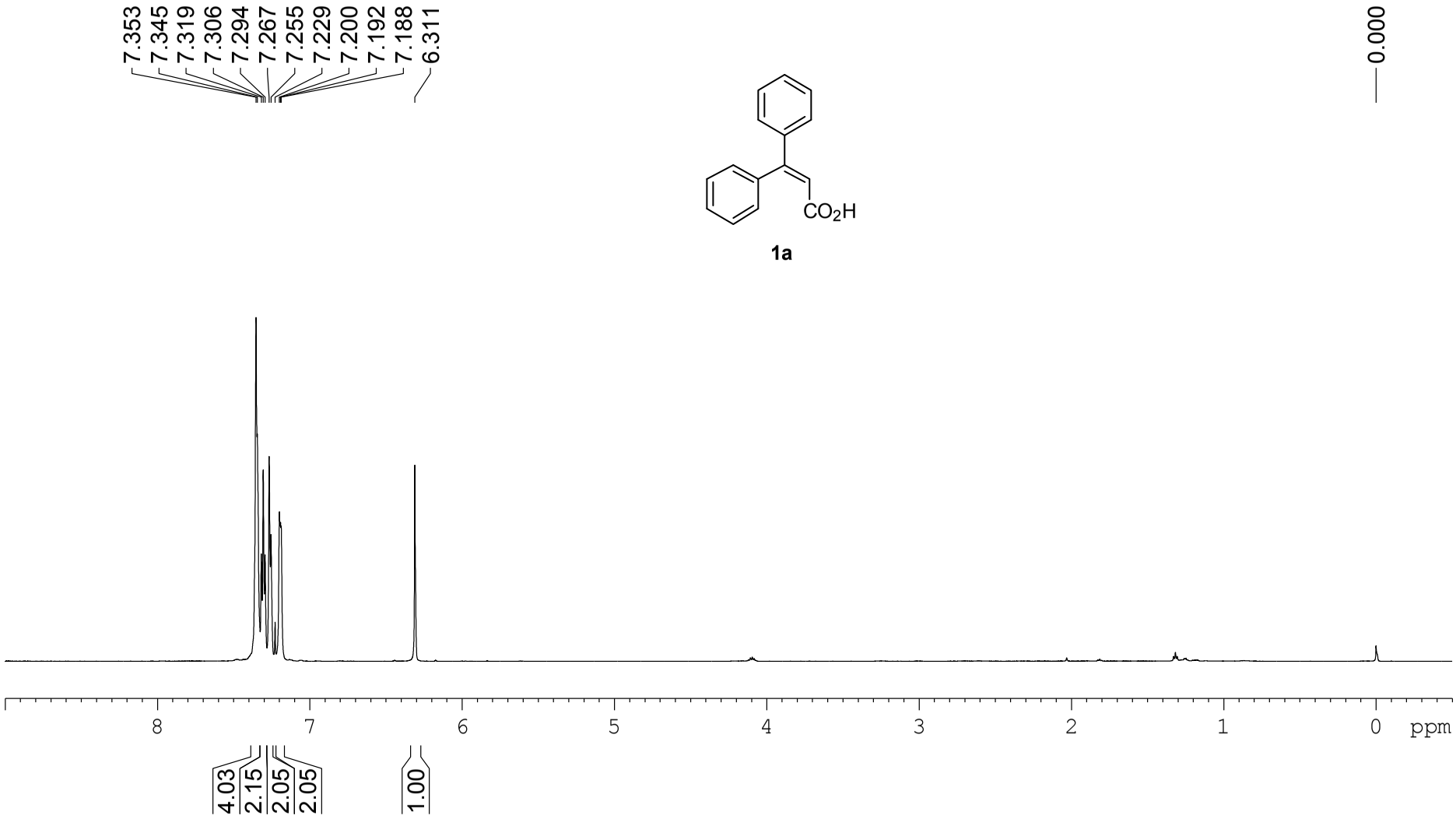
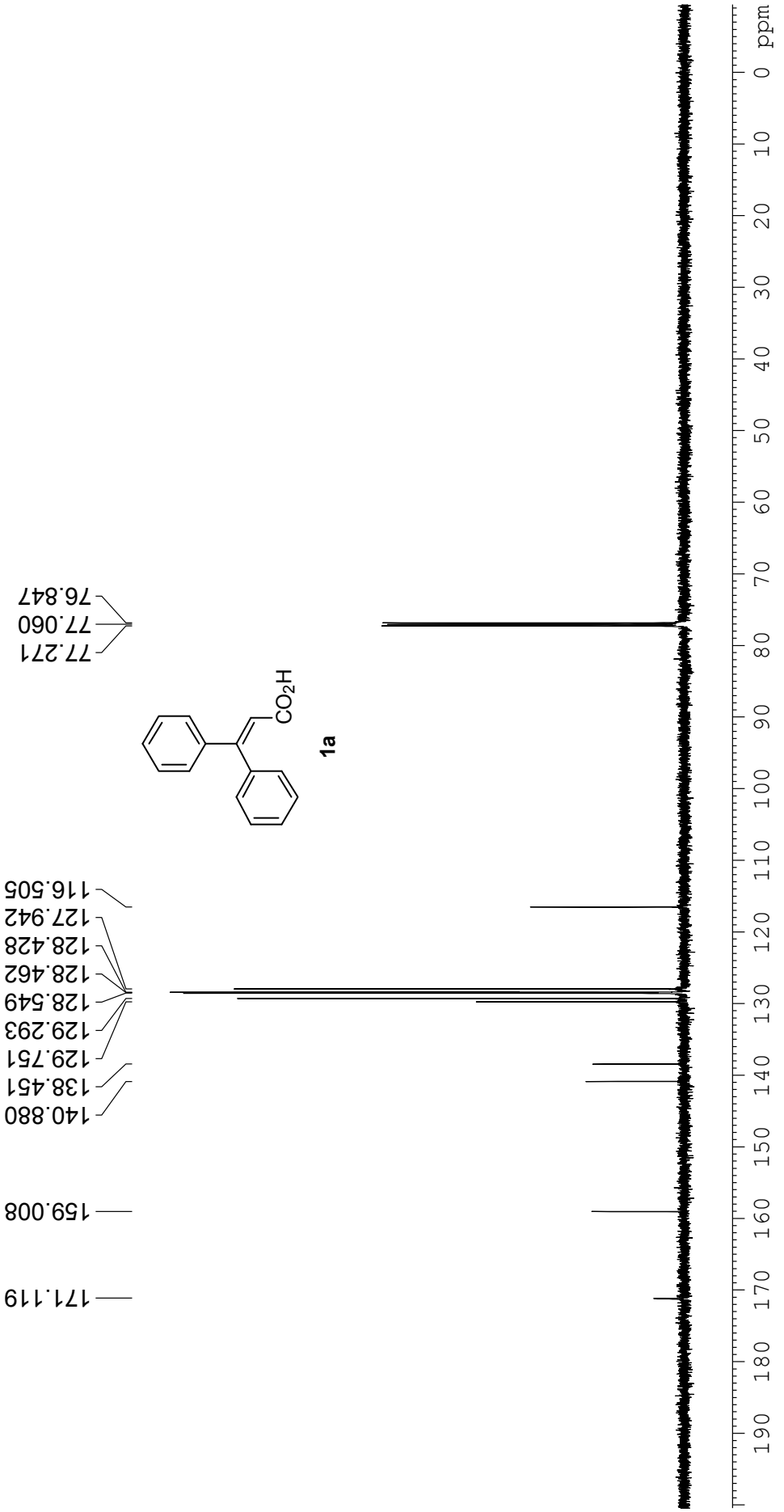
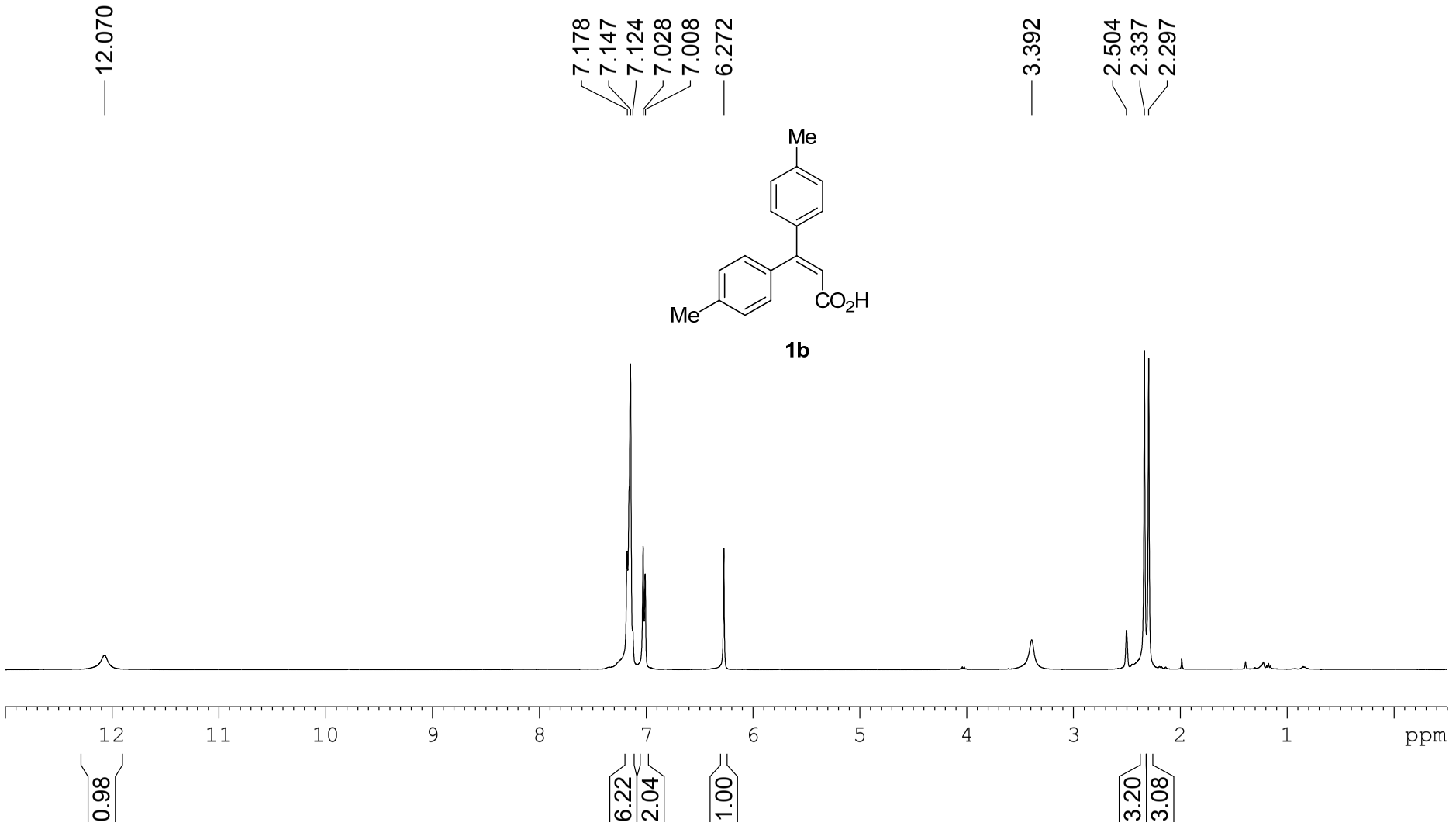
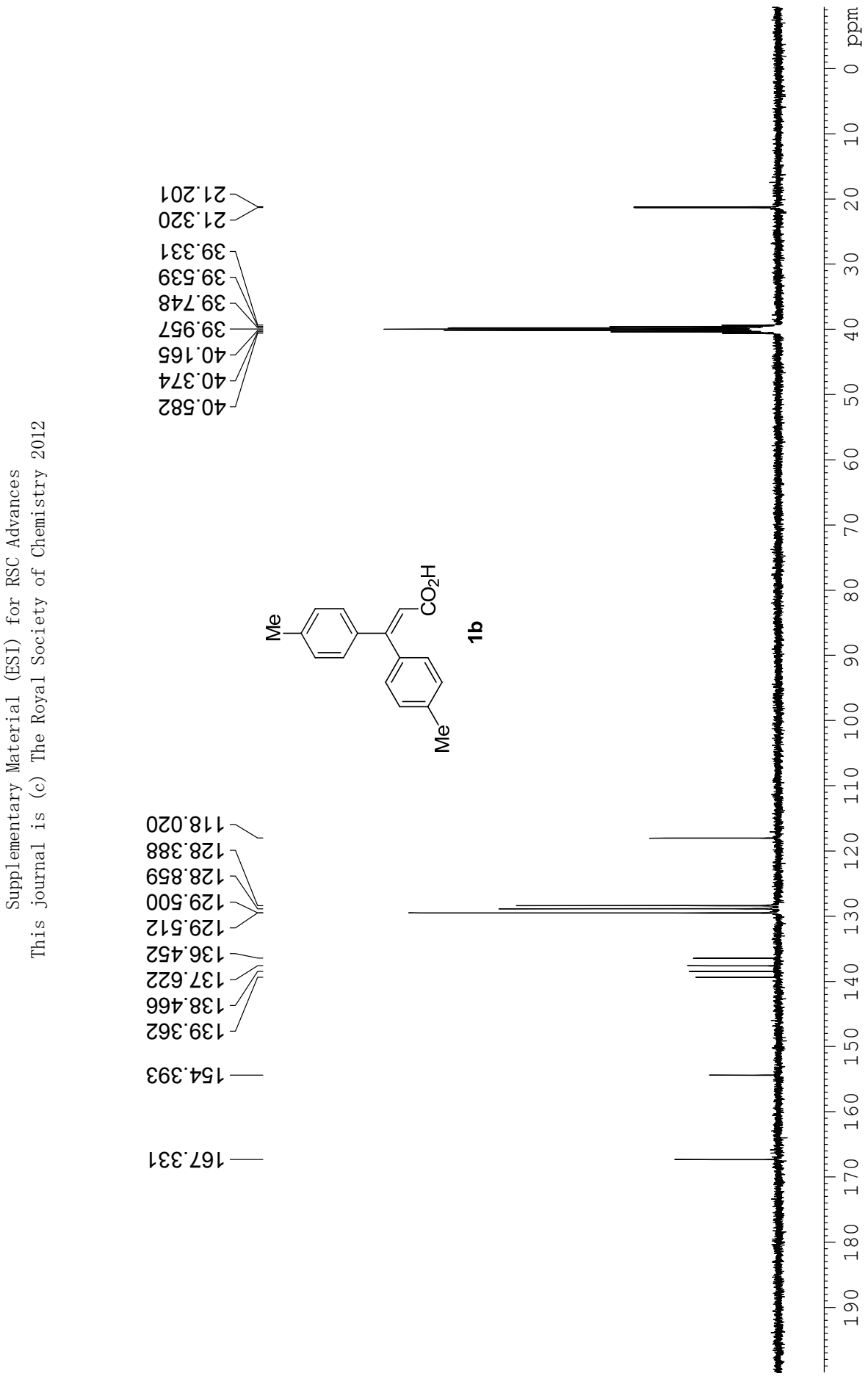
Supplementary Material








Supplementary Material (ESI) for RSC Advances
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21.201
21.320
39.331
39.539
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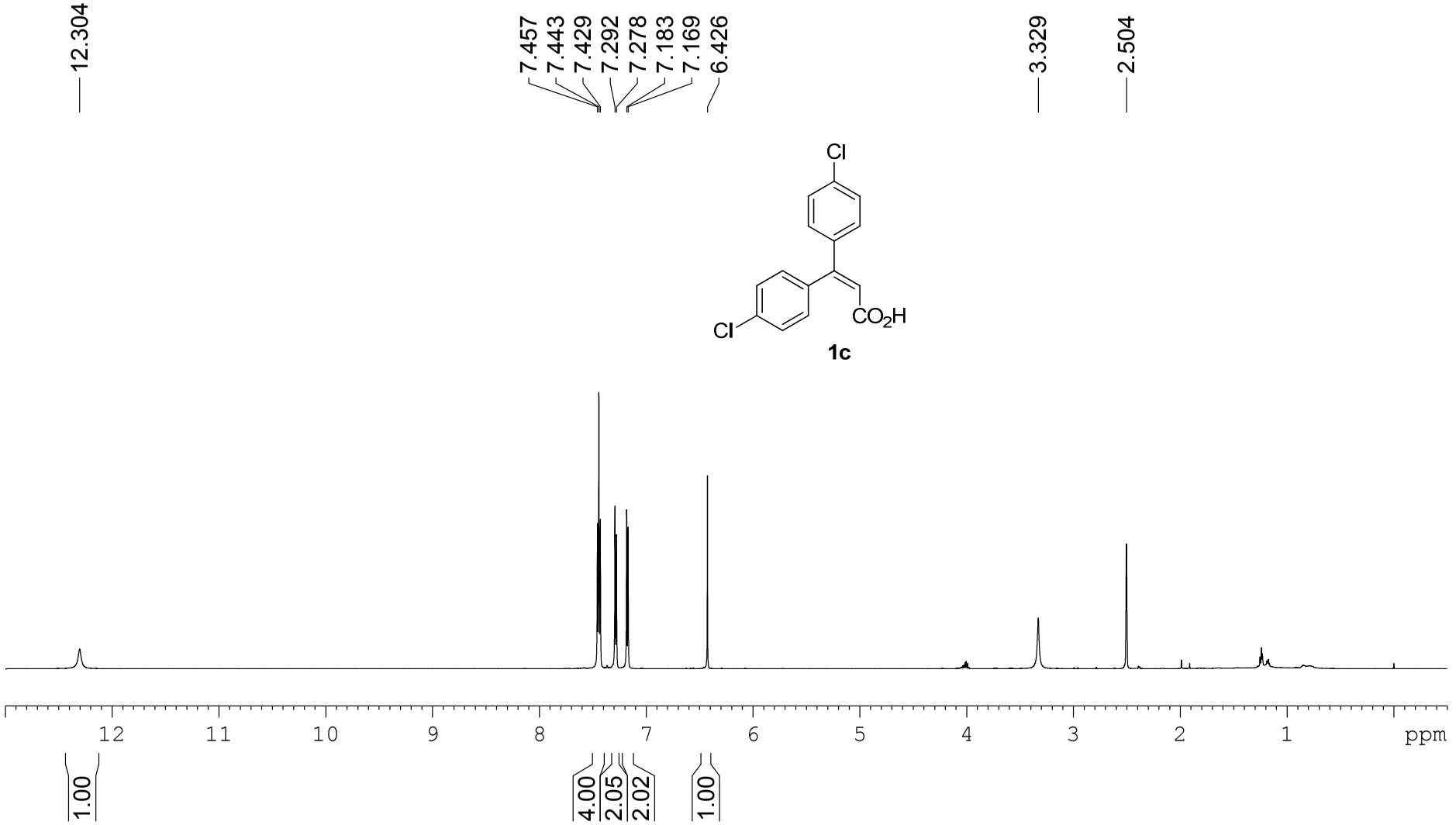
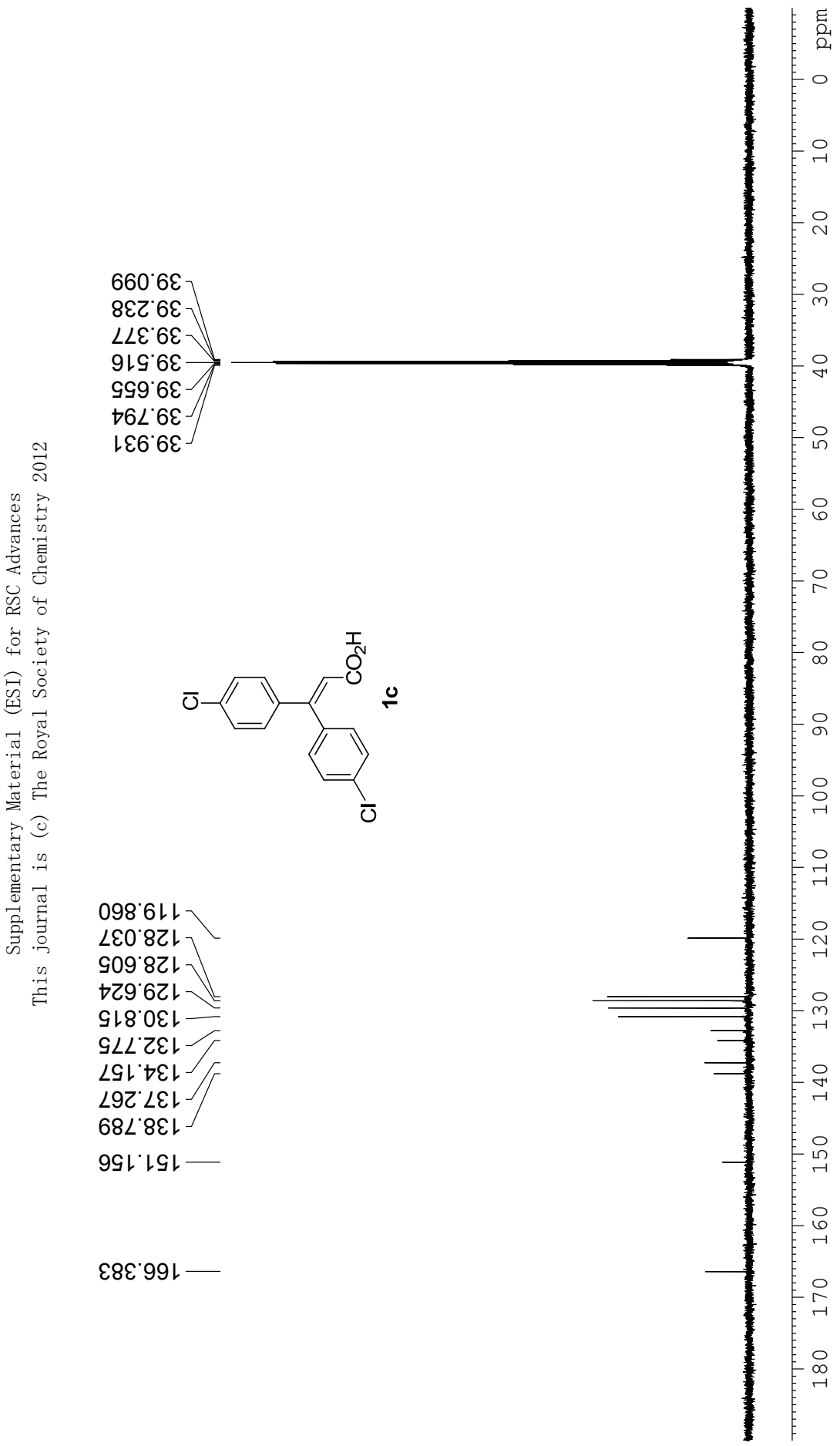
39.957
40.165
40.374
40.582
118.020
128.388
128.859
129.500
129.512
136.452
137.622
138.466
139.362
154.393
167.331
Me
CO2H
Me
1b
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

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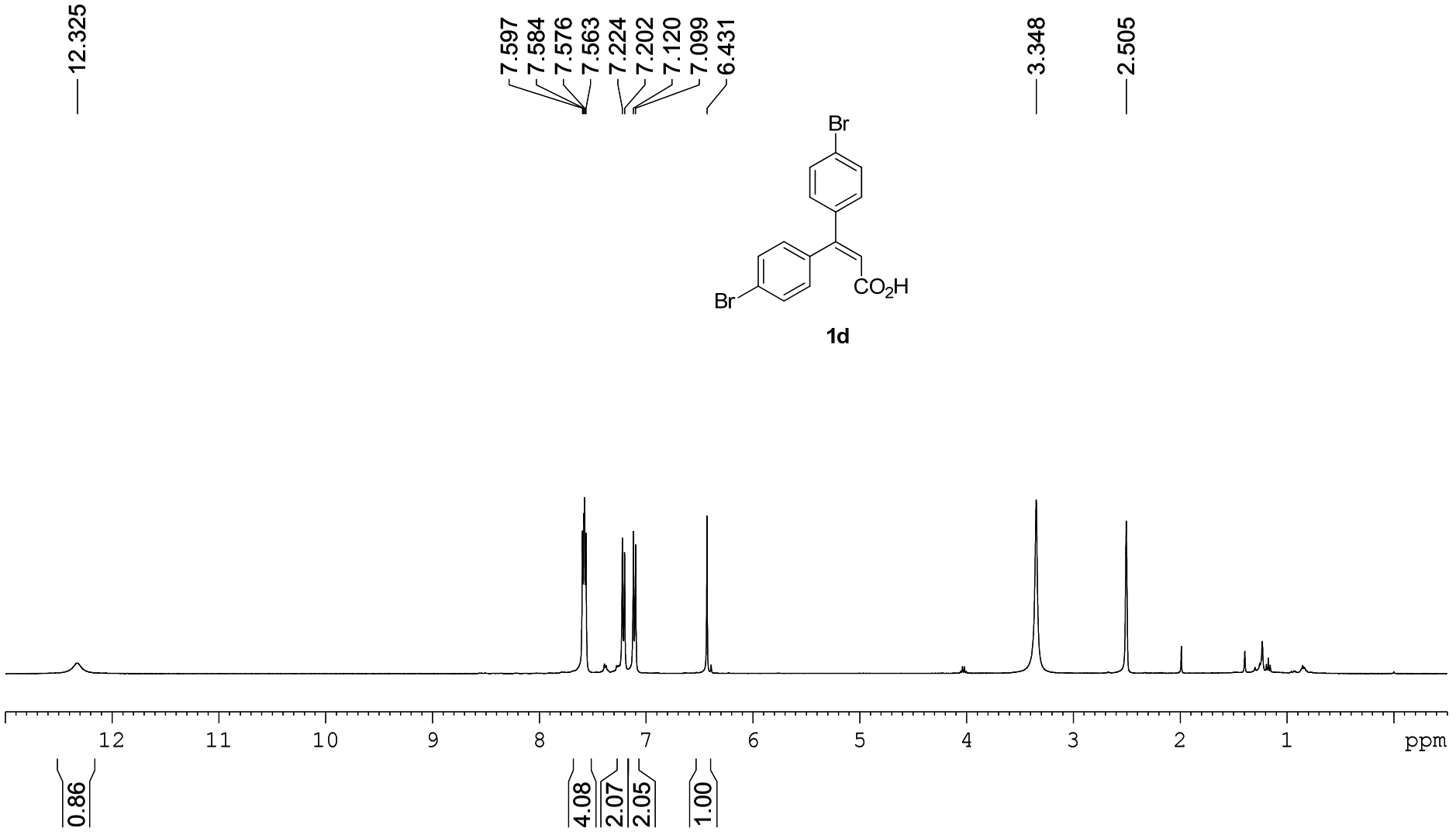
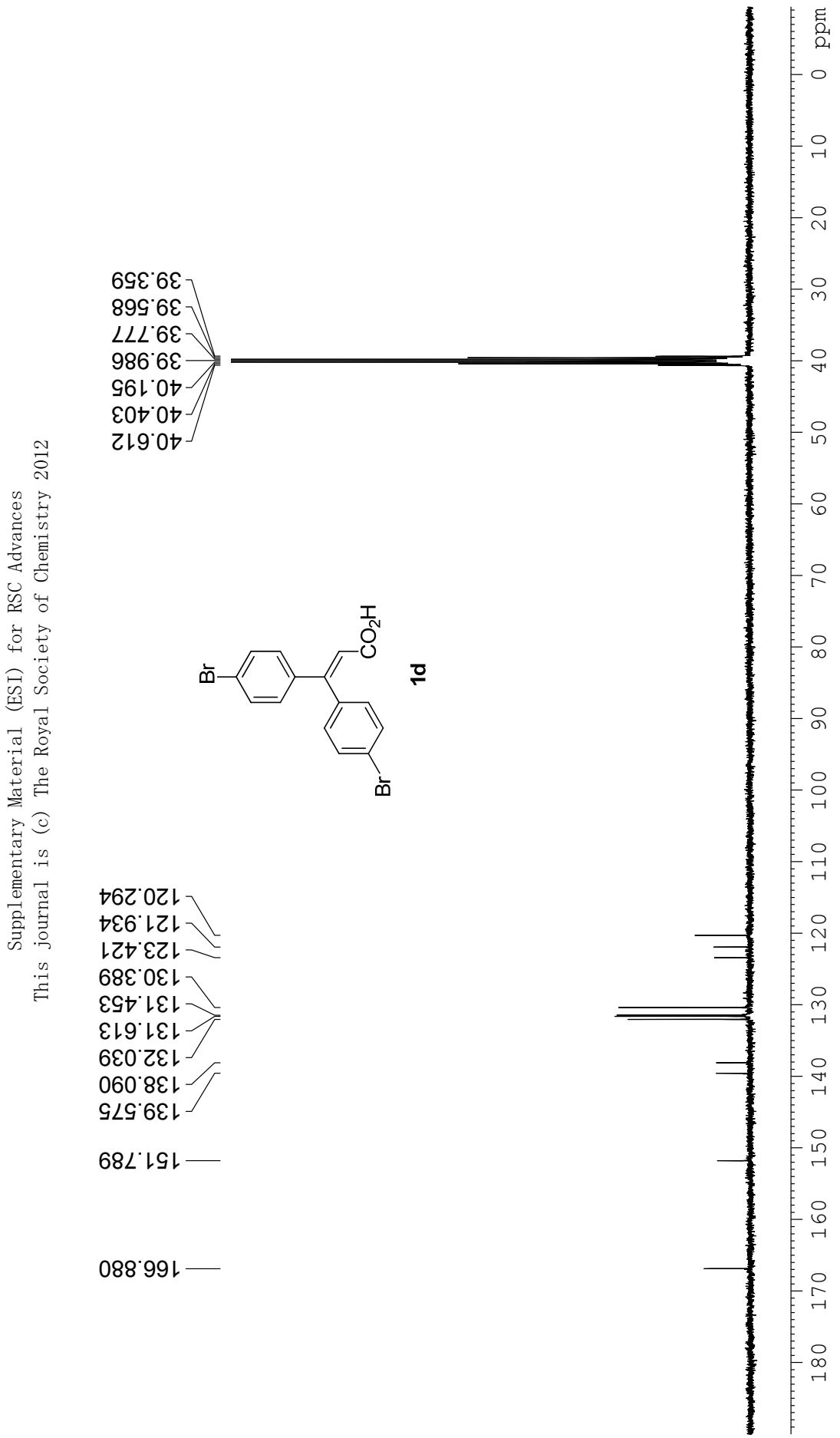
39.099
39.238
39.377
39.516
39.655
39.794
39.931
119.860
128.037
128.605
129.624
130.815
132.775
134.157
137.267
138.789
151.156
166.383
Cl
CO2H
Cl
1c
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

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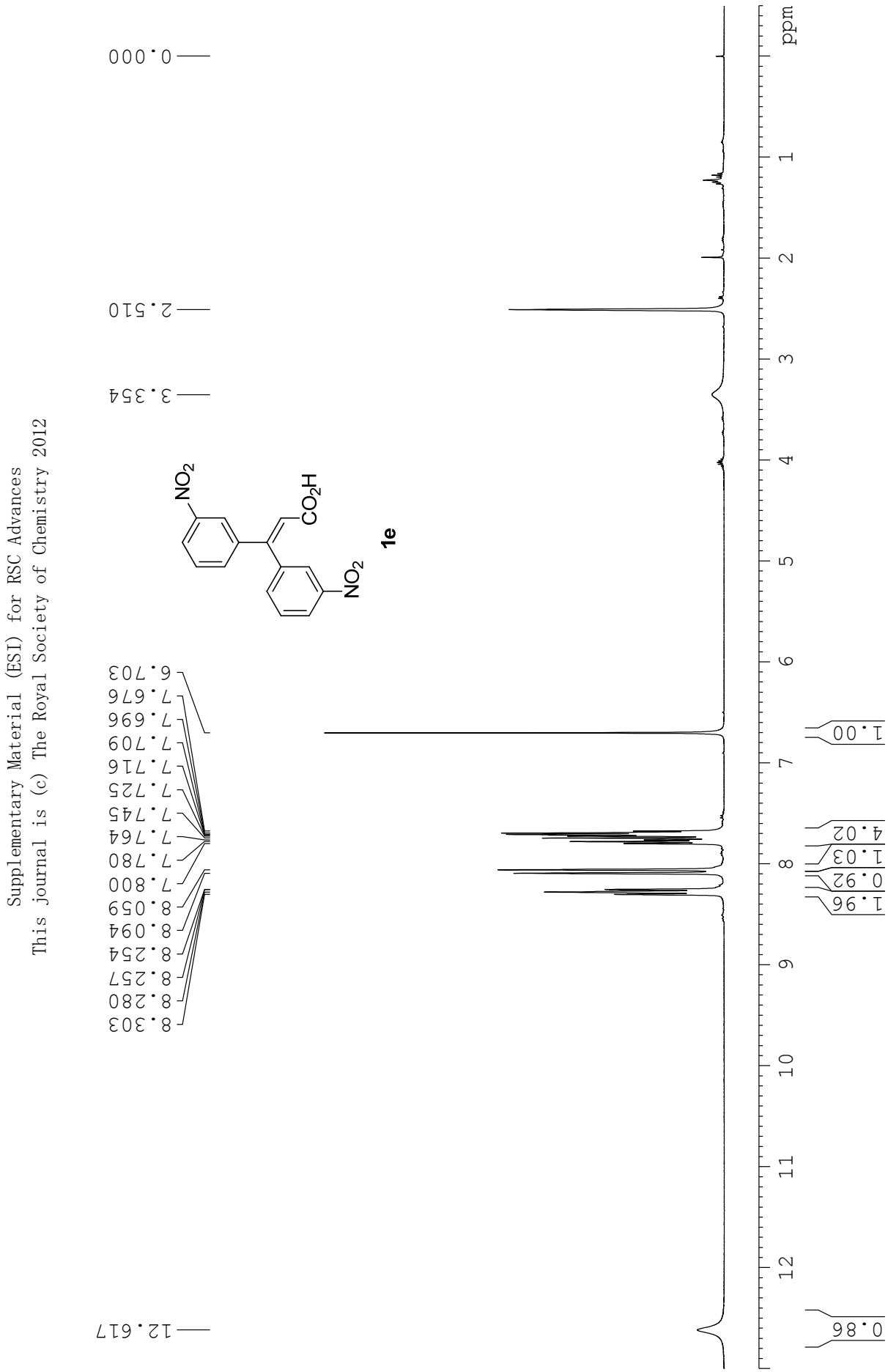
166.880
151.789
139.575
138.090
132.039
131.613
131.453
130.389
123.421
121.934
120.294
40.612
40.403
40.195
39.986
39.777
39.568
39.359
Br
CO2H
Br
1d
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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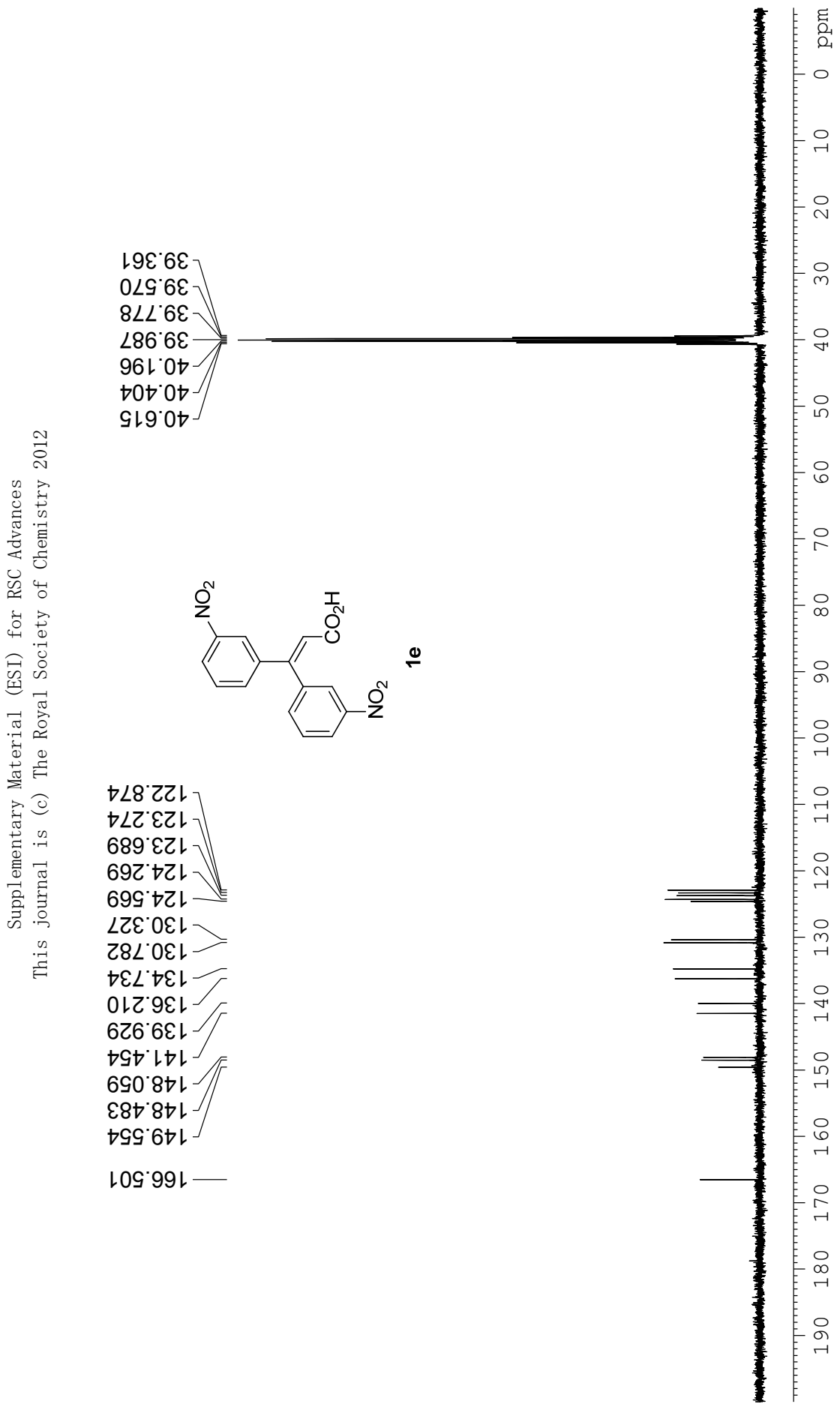
0.000
2.510
3.354
12.617
8.303
8.280
8.257
8.254
8.094
8.059
7.800
7.780
7.764
7.745
7.725
7.716
7.709
7.696
7.676
6.703
NO2
CO2H
NO2
1e
ppm
1 2 3 4 5 6 7 8 9 10 11 12
0.86
1.96
0.92
1.03
4.02
1.00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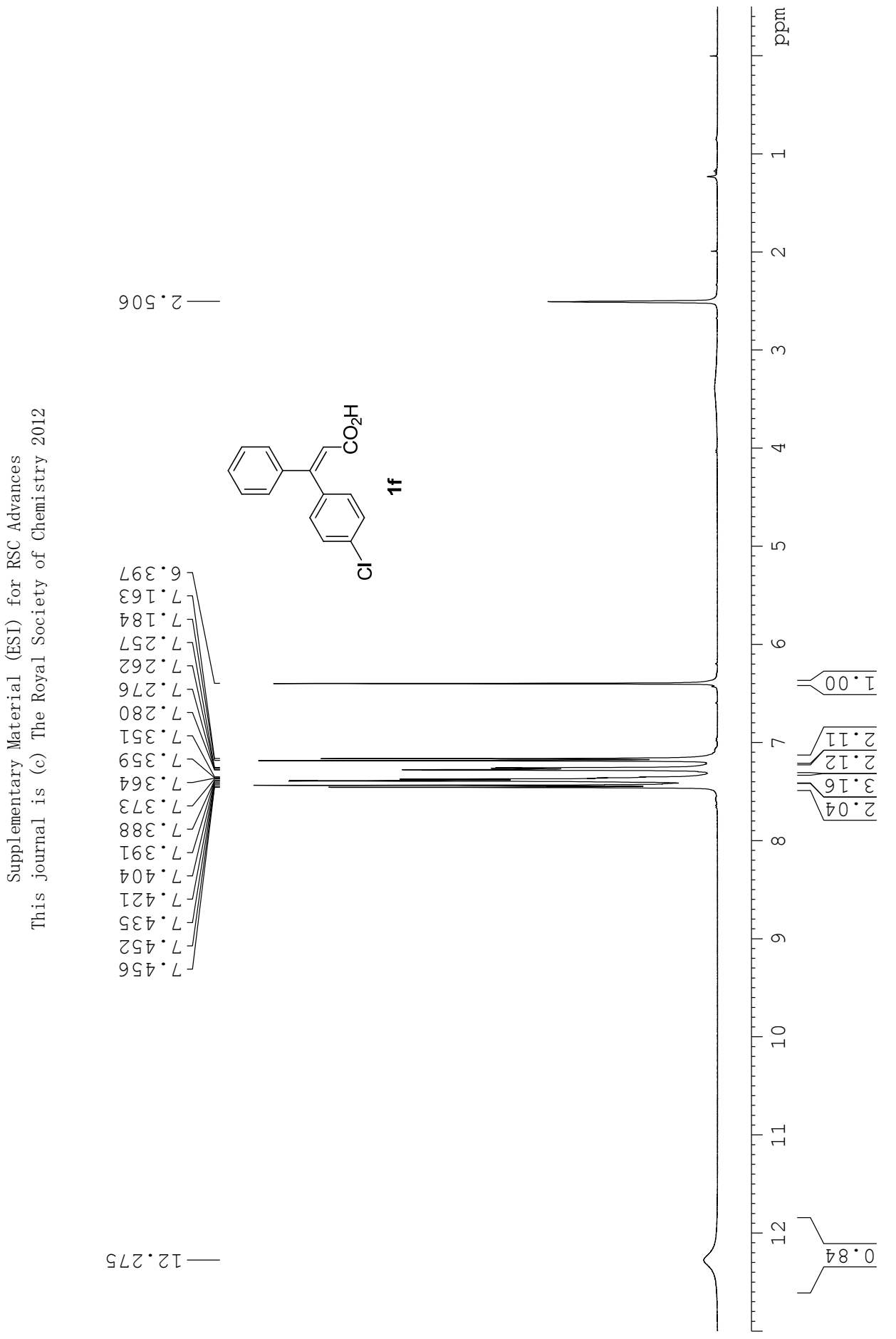
149.554
148.483
148.059
141.454
139.929
136.210
134.734
130.782
130.327
124.569
124.269
123.689
123.274
122.874
40.615
40.404
40.196
39.987
39.778
39.570
39.361
NO2
CO2H
NO2
1e
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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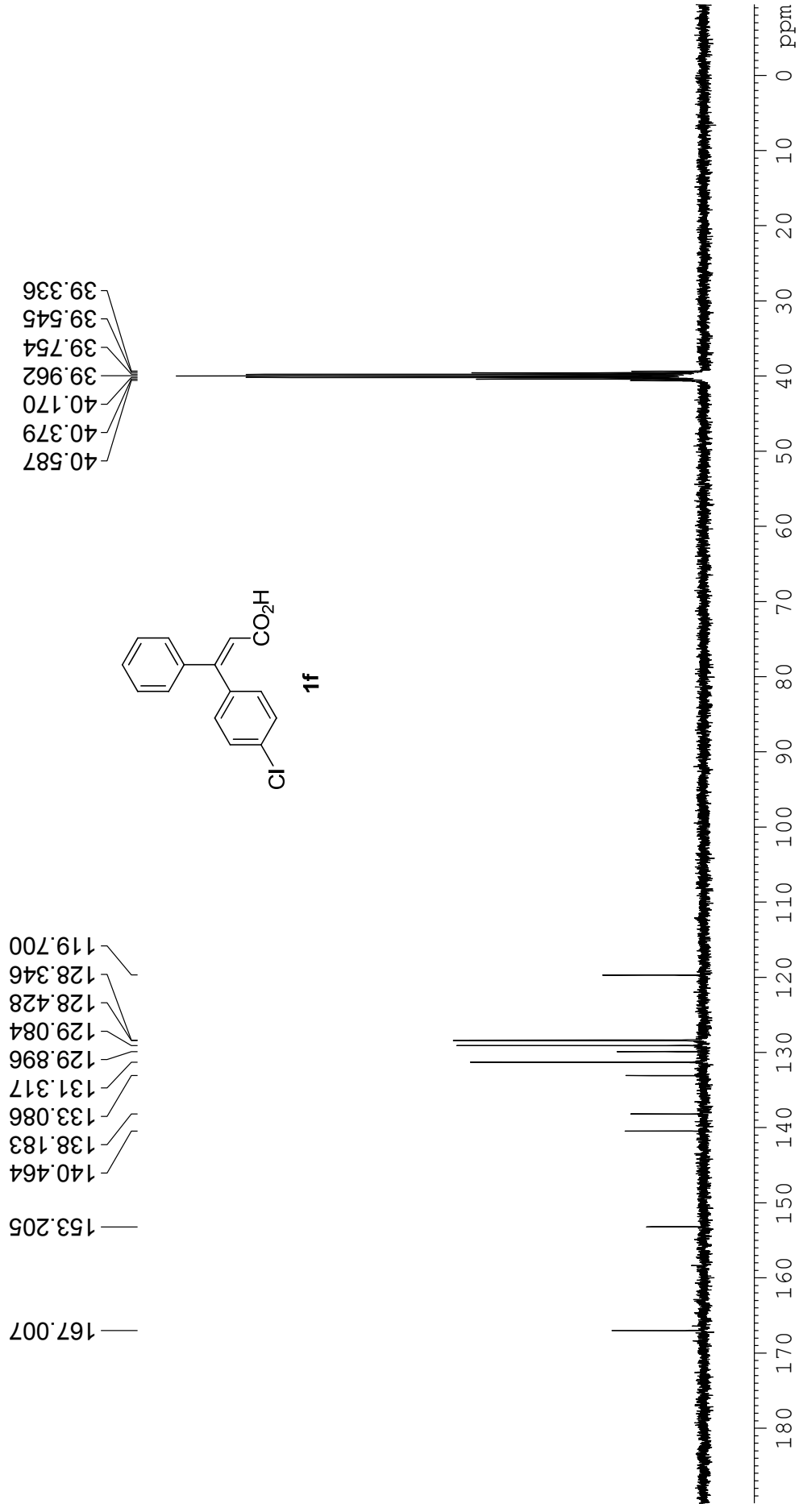
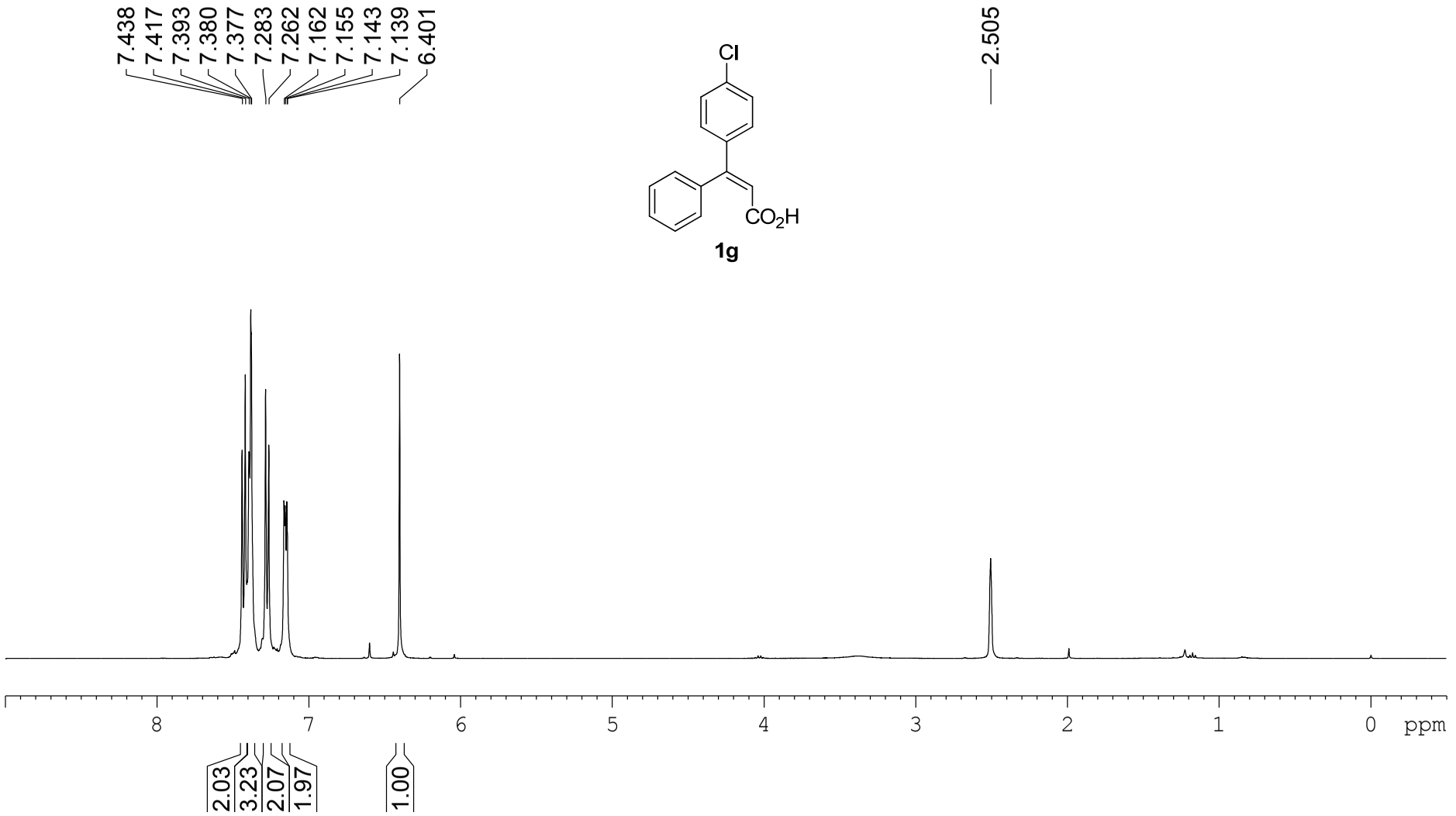
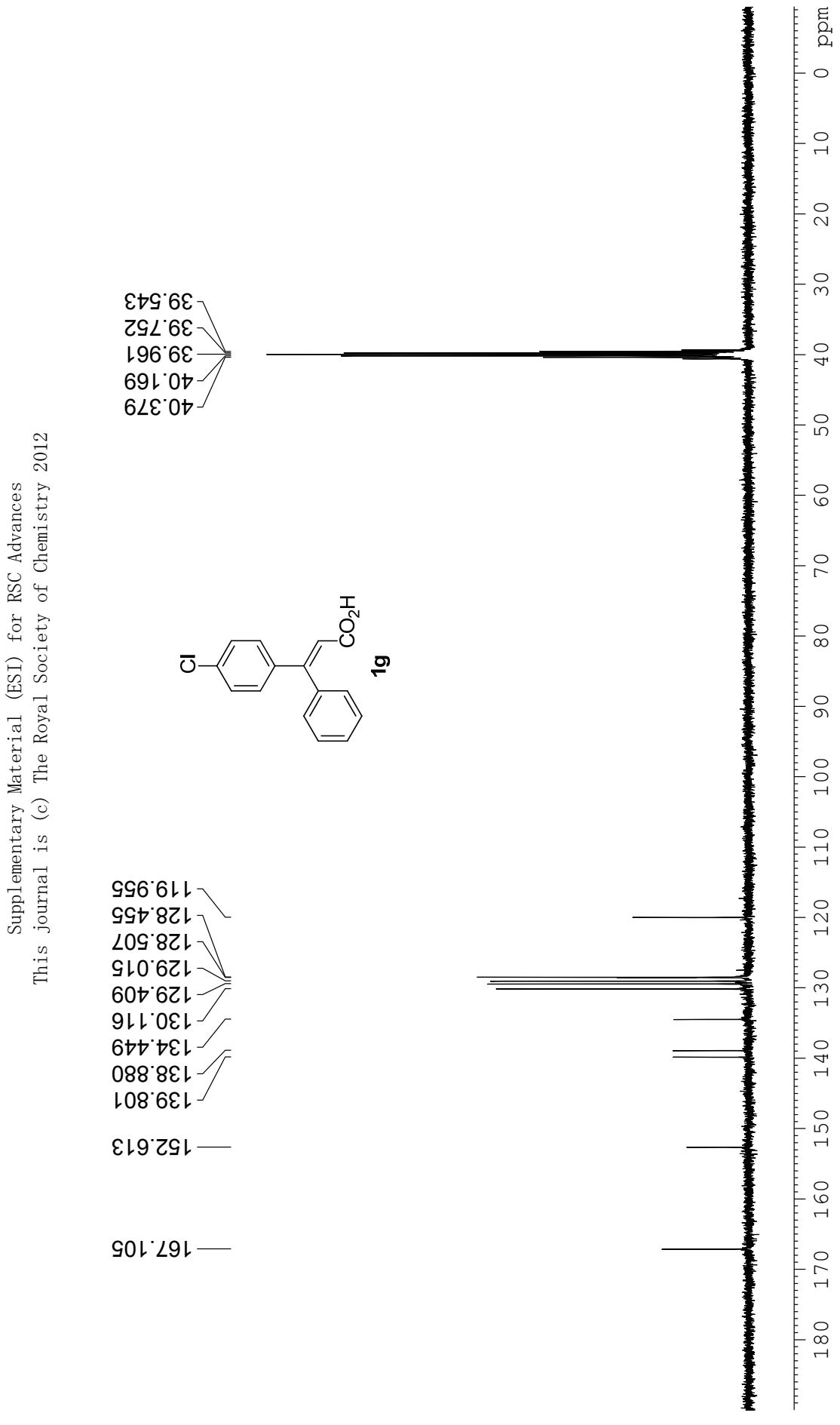
12.275
7.456
7.452
7.435
7.421
7.404
7.391
7.388
7.373
7.364
7.359
7.351
7.280
7.276
7.262
7.257
7.184
7.163
6.397
2.506
CO2H
Cl
1f
ppm
0.84
2.04
3.16
2.12
2.11
1.00



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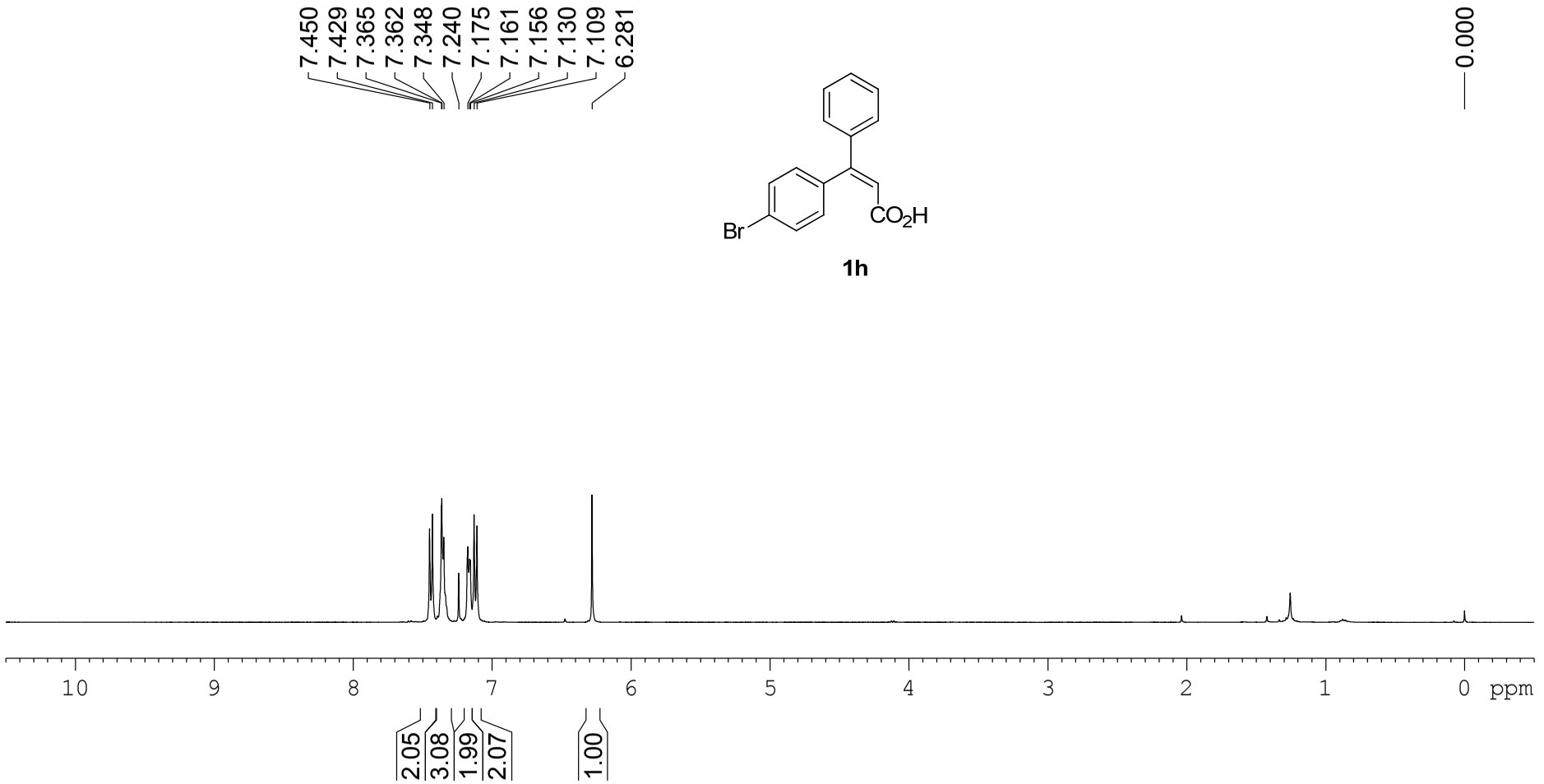
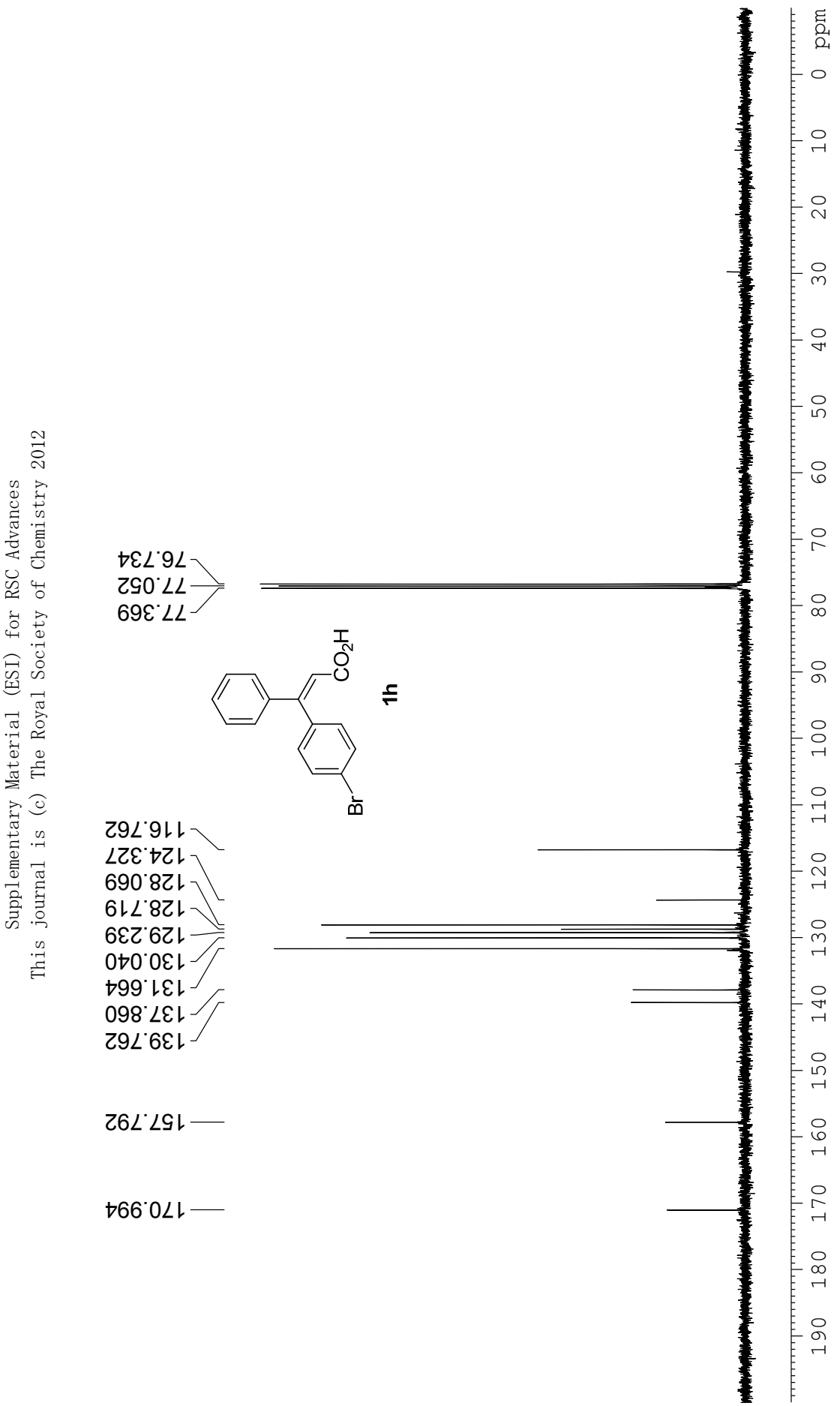
152.613
139.801
138.880
134.449
130.116
129.409
129.015
128.507
128.455
119.955
40.379
40.169
39.961
39.752
39.543
Cl
CO2H
1g
ppm

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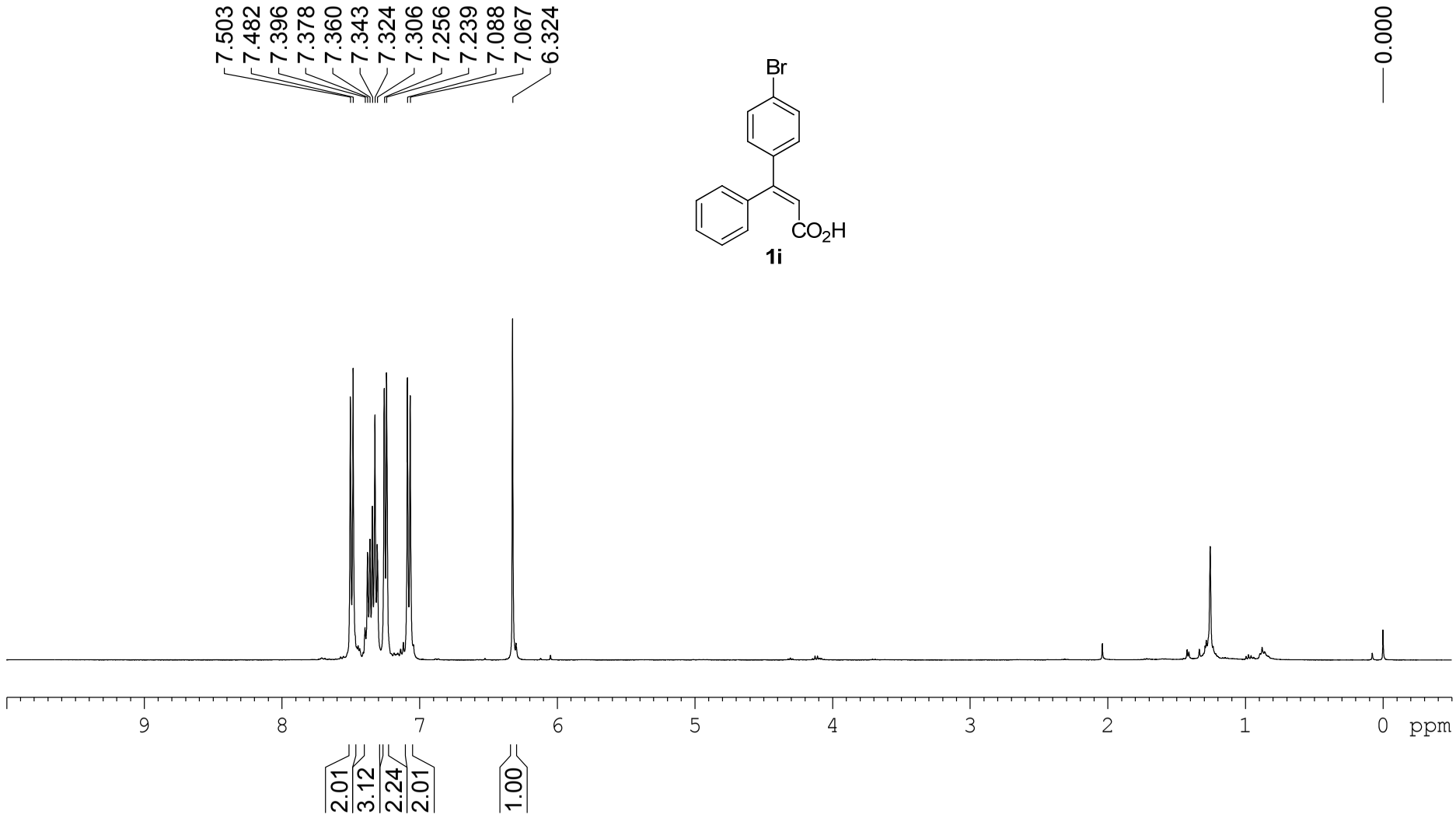
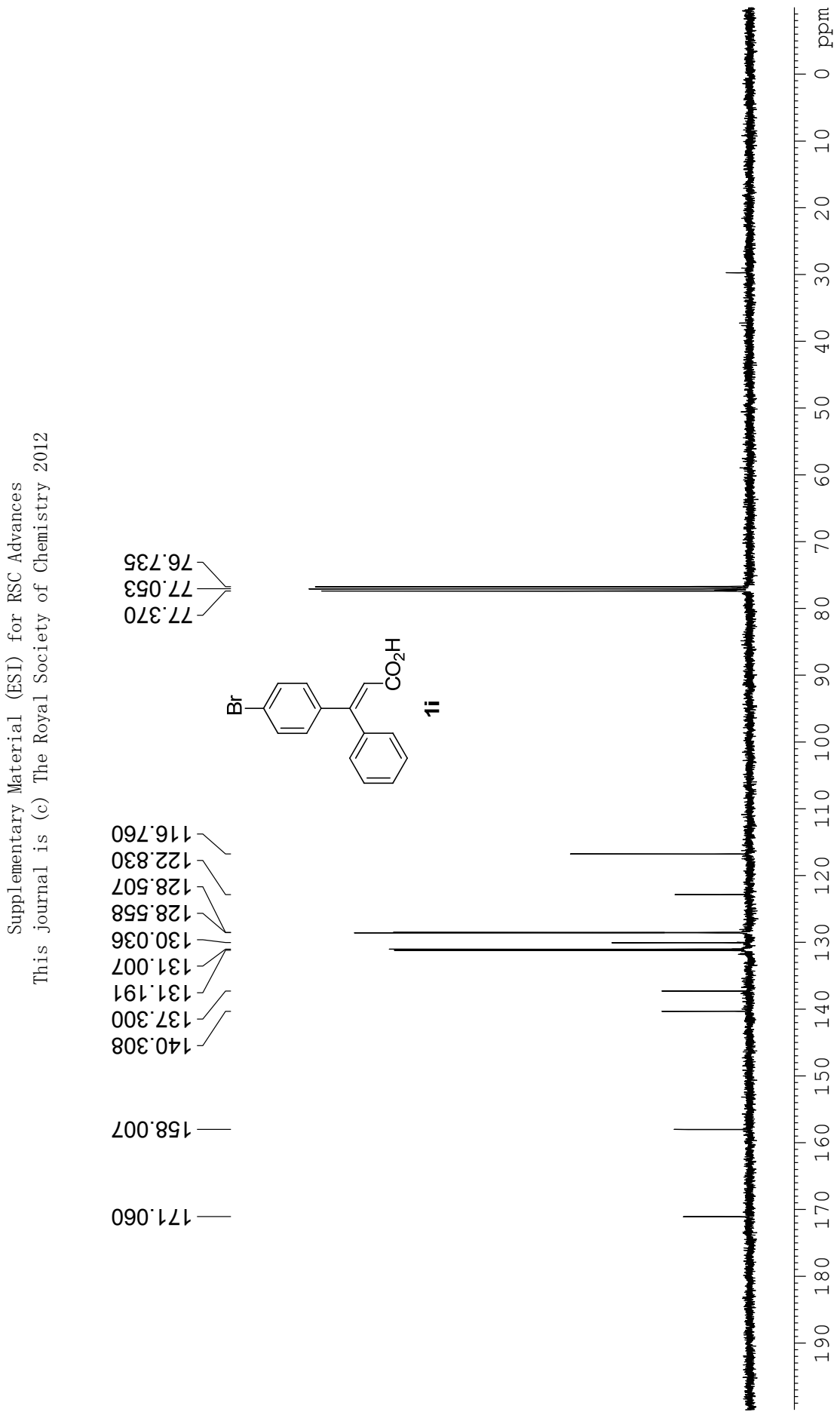
170.994
157.792
139.762
137.860
131.664
130.040
129.239
128.719
128.069
124.327
116.762
77.369
77.052
76.734
CO2H
Br
1h
ppm

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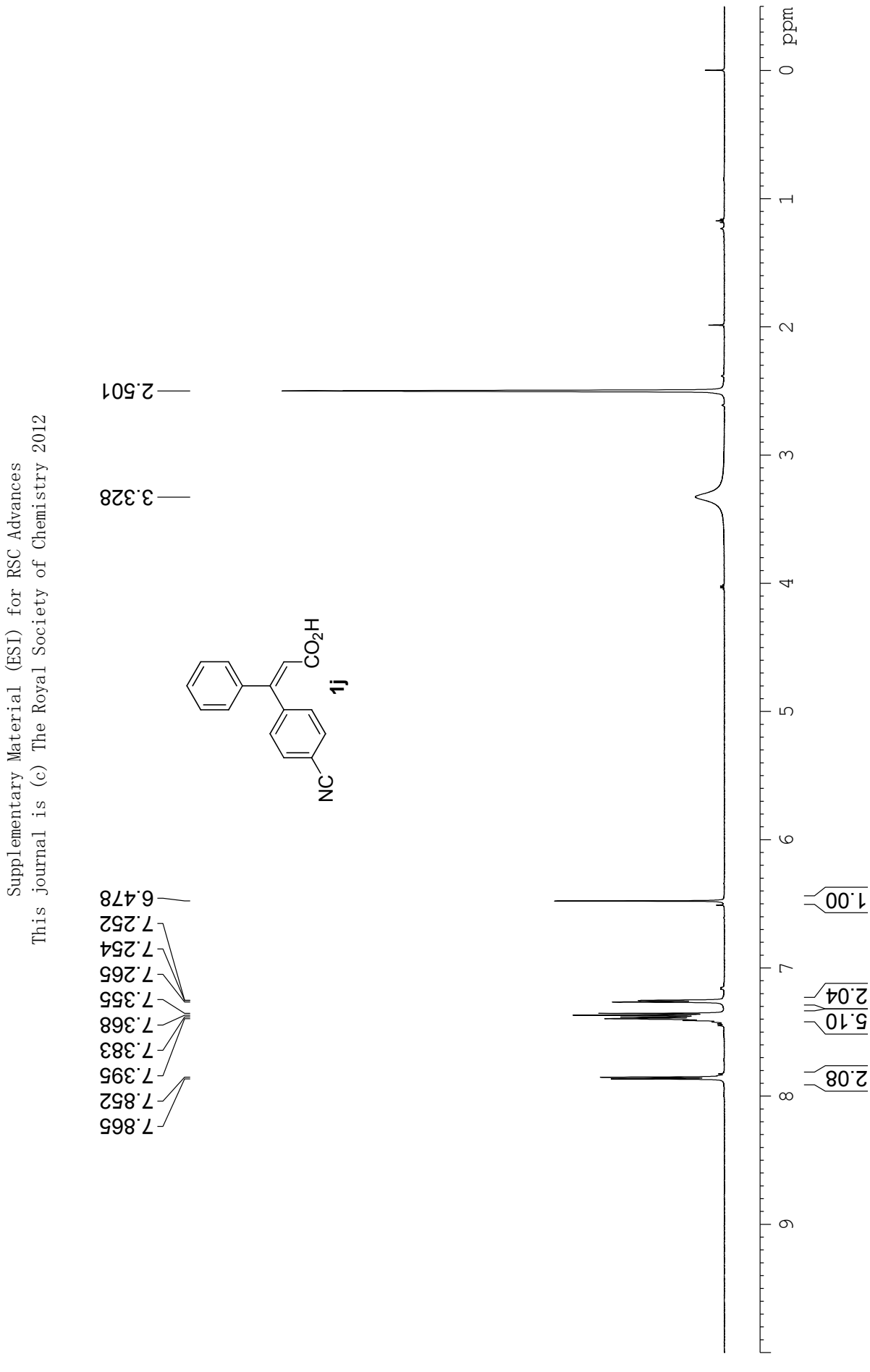
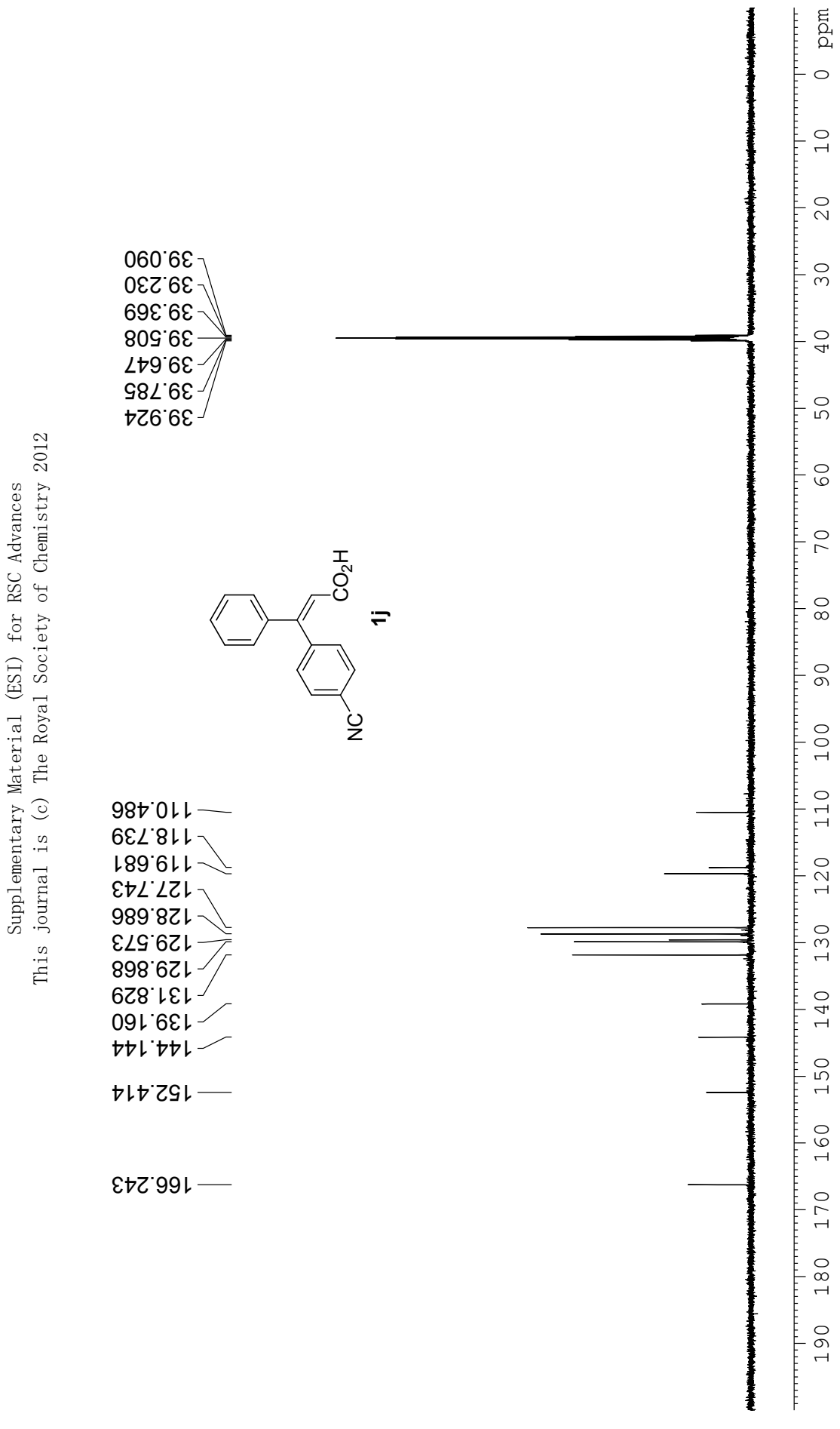
171.060
158.007
140.308
137.300
131.191
131.007
130.036
128.558
128.507
122.830
116.760
77.370
77.053
76.735
Br
CO2H
1i
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

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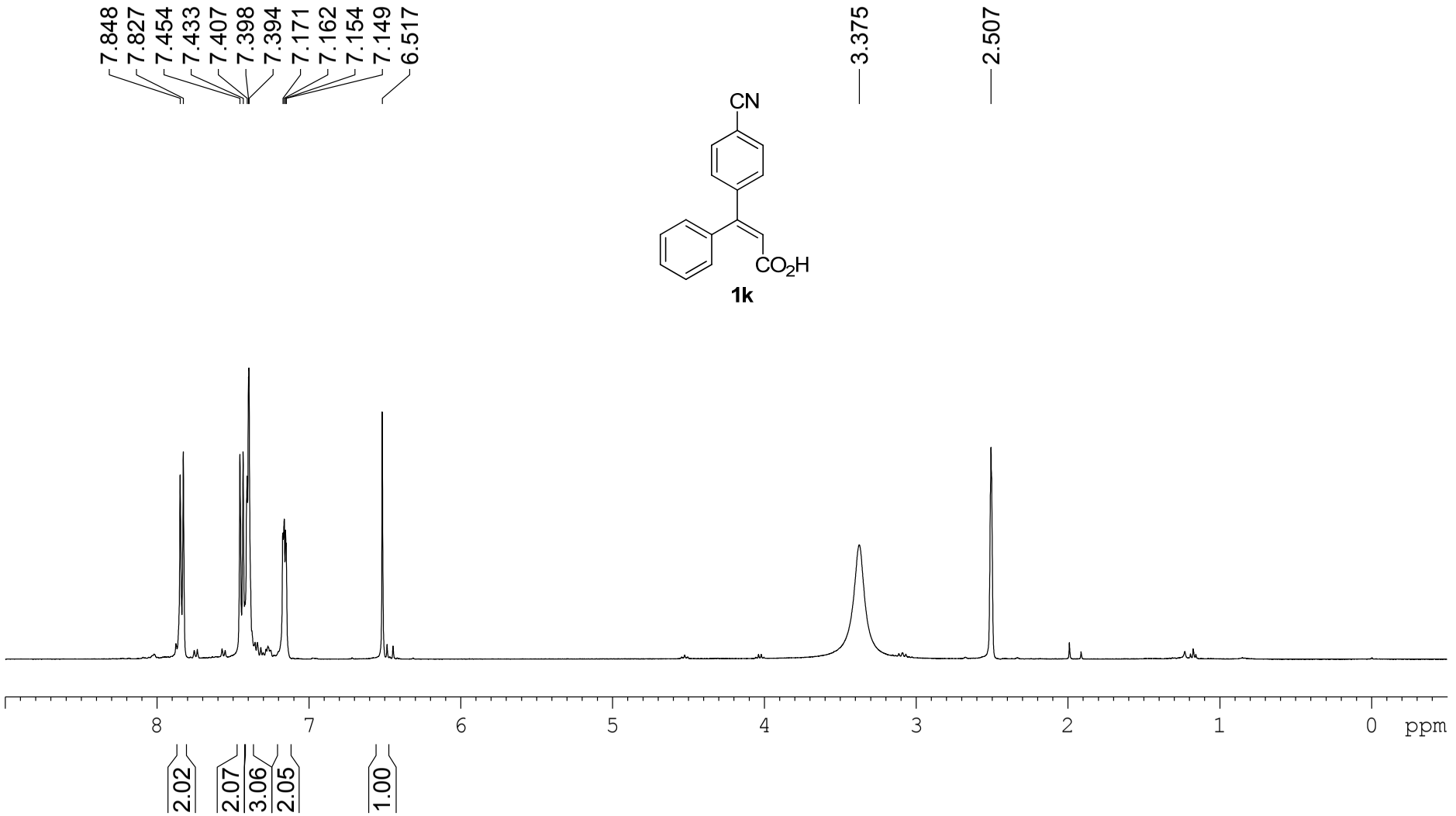
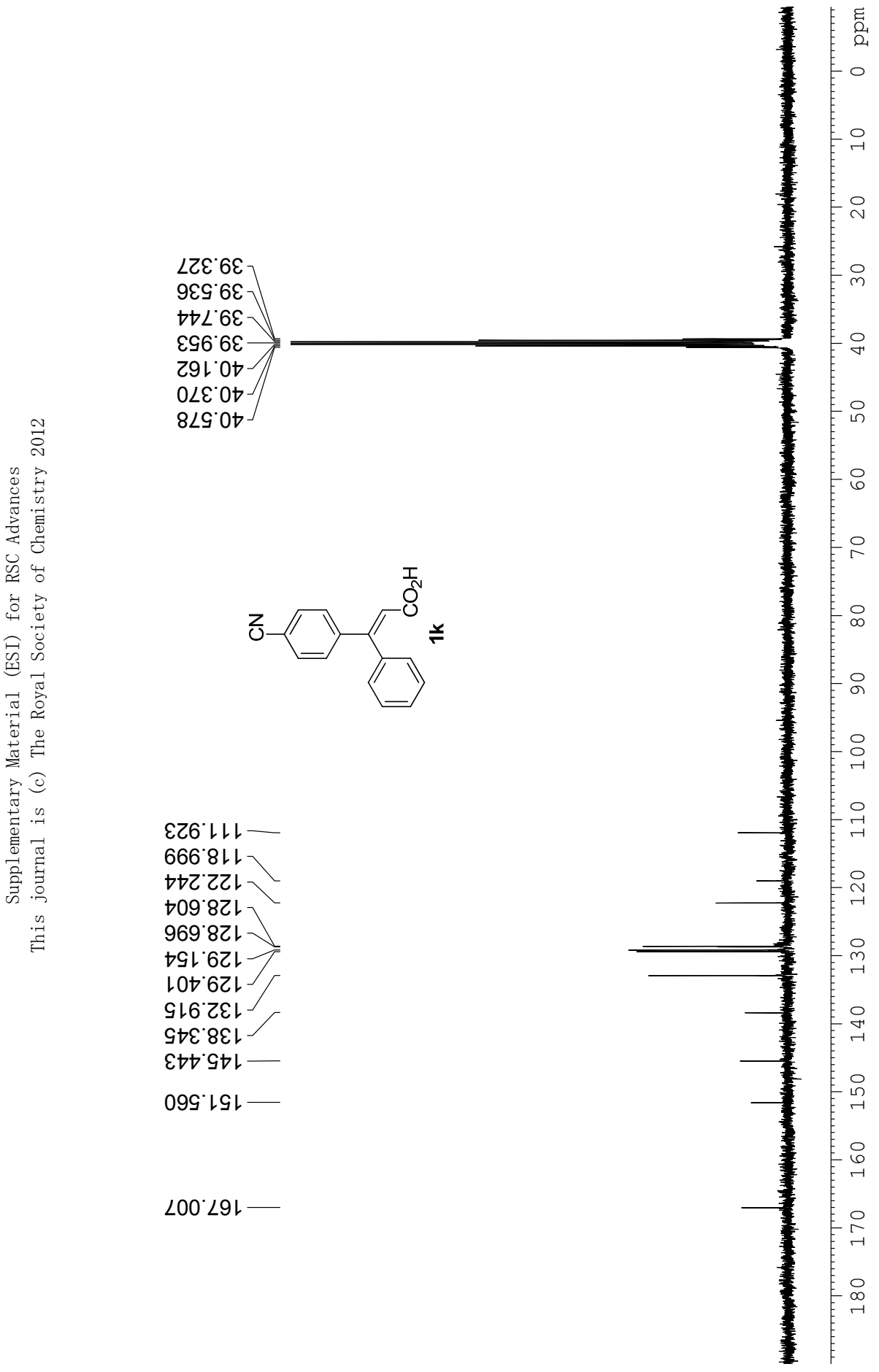
39.090
39.230
39.369
39.508
39.647
39.785
39.924
CO2H
NC
1j
110.486
118.739
119.681
127.743
128.686
129.573
129.868
131.829
139.160
144.144
152.414
166.243
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

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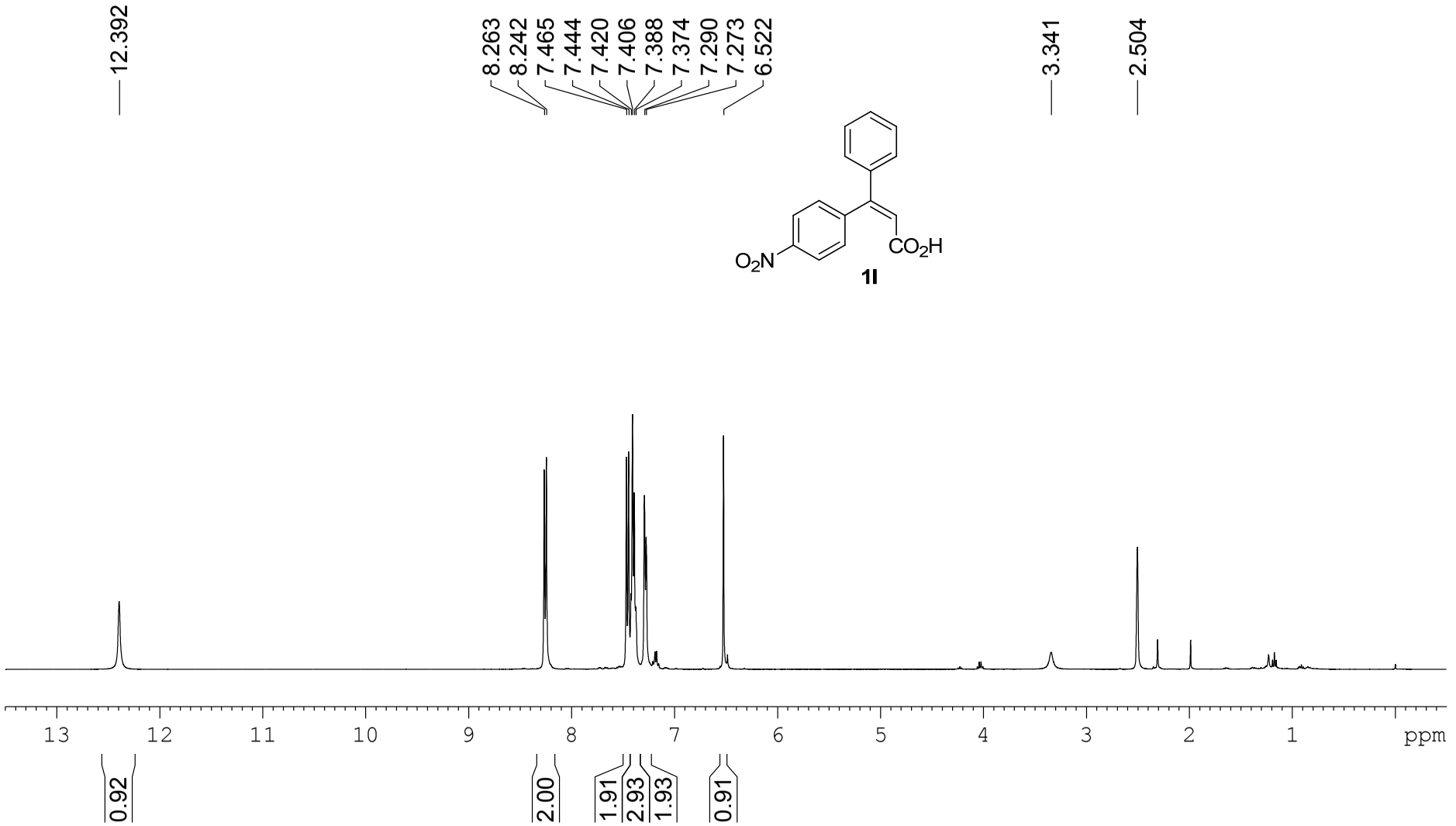
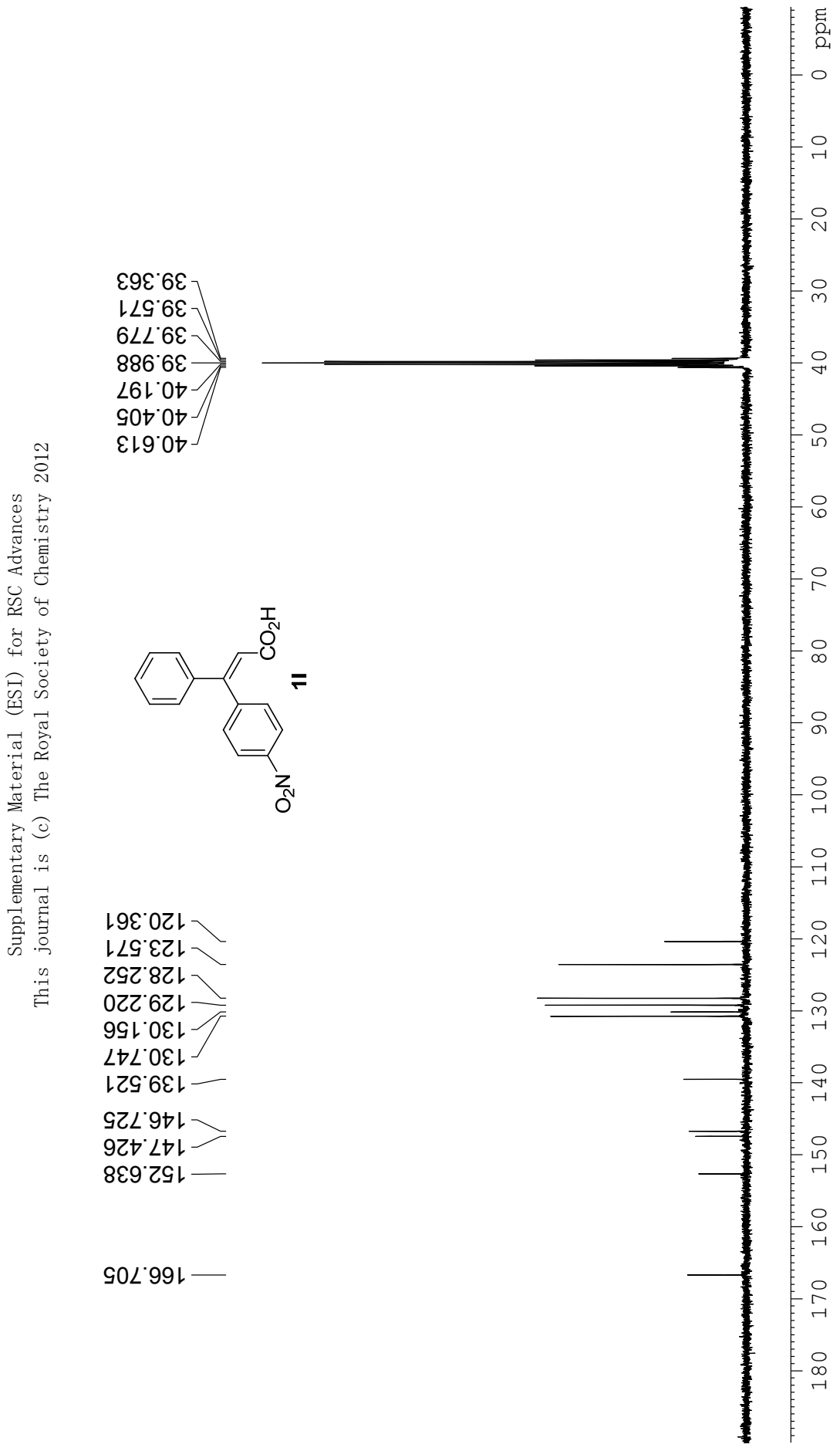
40.578
40.370
40.162
39.953
39.744
39.536
39.327
111.923
118.999
122.244
128.604
128.696
129.154
129.401
132.915
138.345
145.443
151.560
167.007
CN
CO2H
1k
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 ppm

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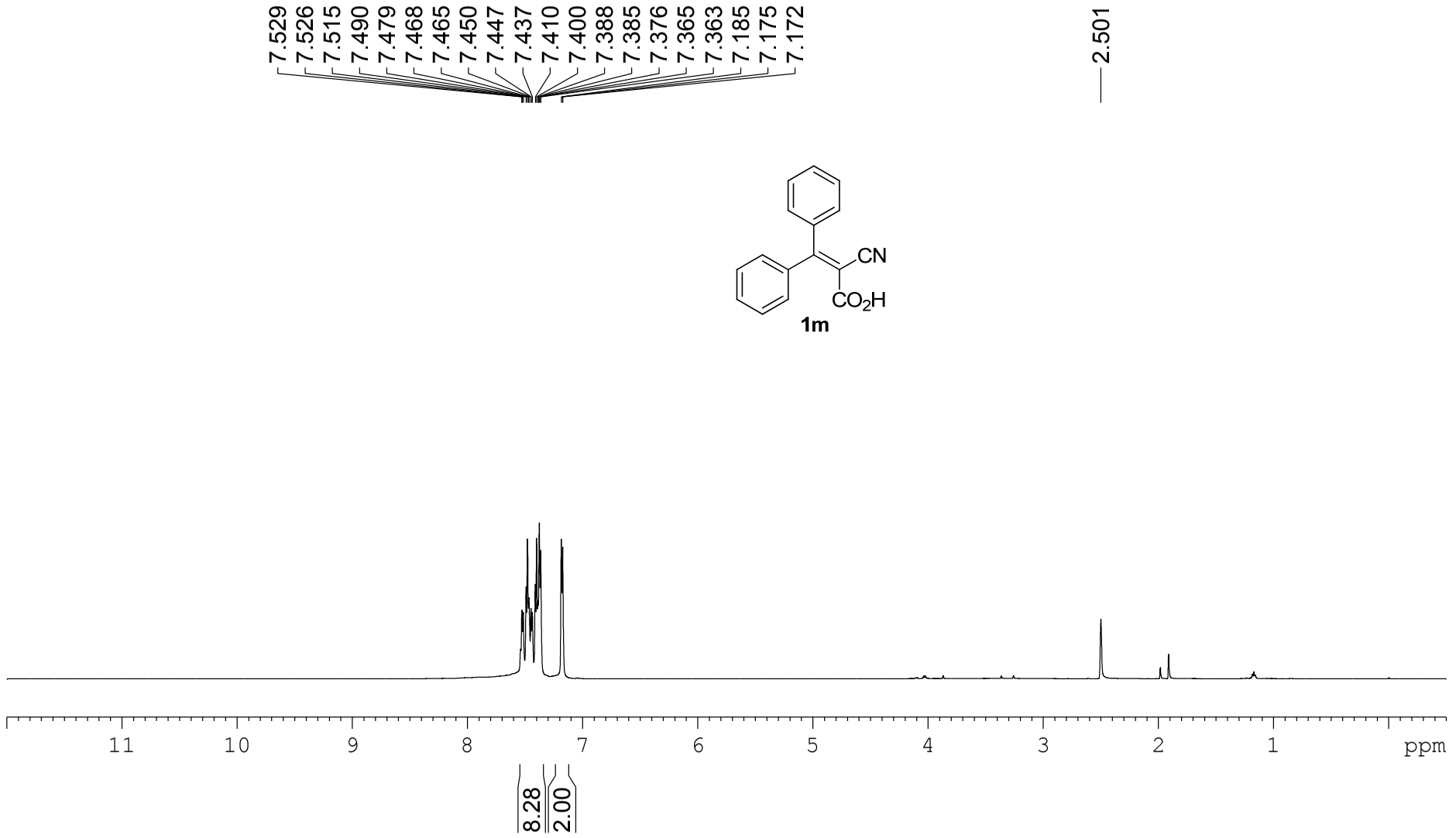
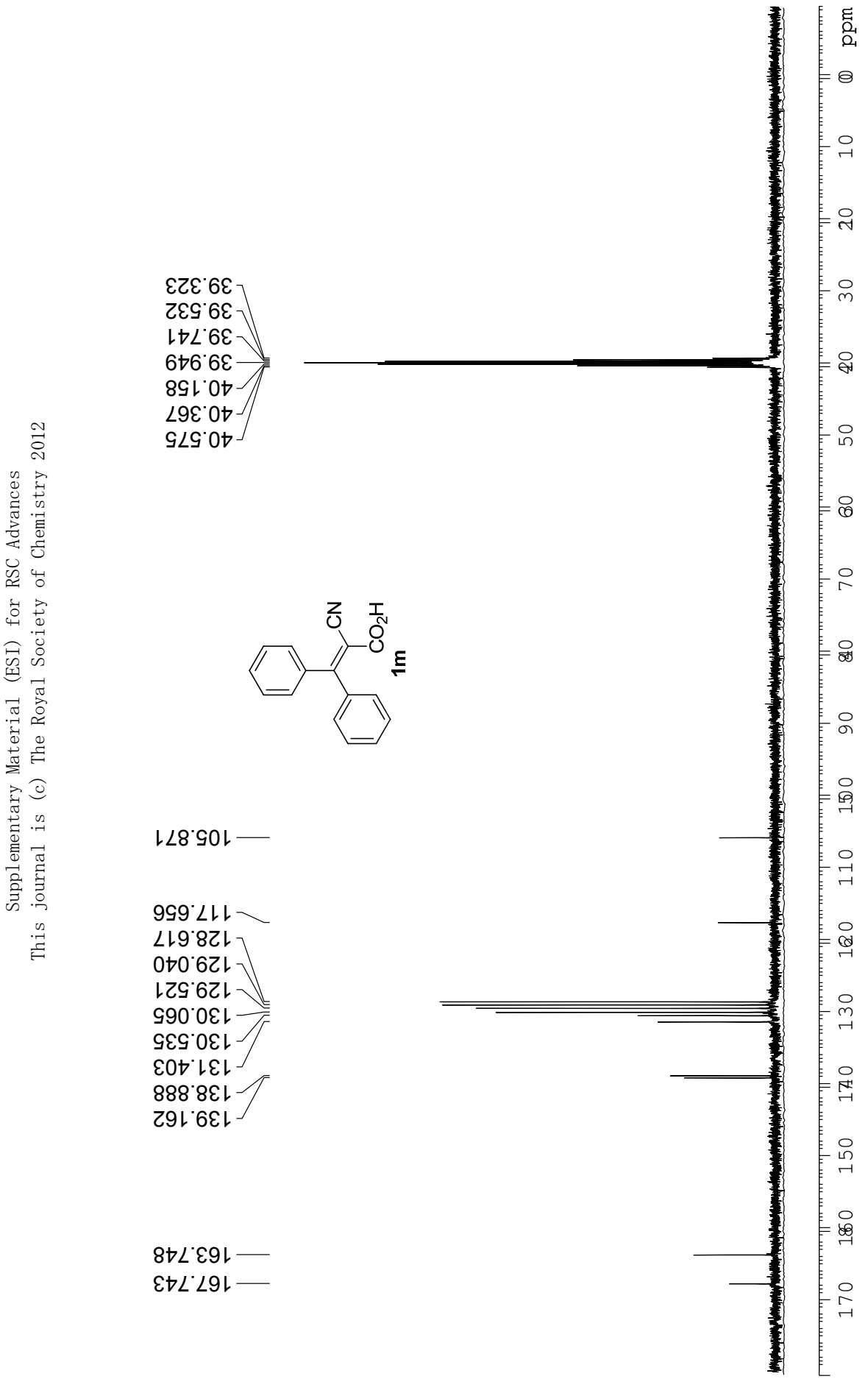
152.638
147.426
146.725
139.521
130.747
130.156
129.220
128.252
123.571
120.361
40.613
40.405
40.197
39.988
39.779
39.571
39.363
CO2H
O2N
1l
ppm

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39.741
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105.871
117.656
128.617
129.040
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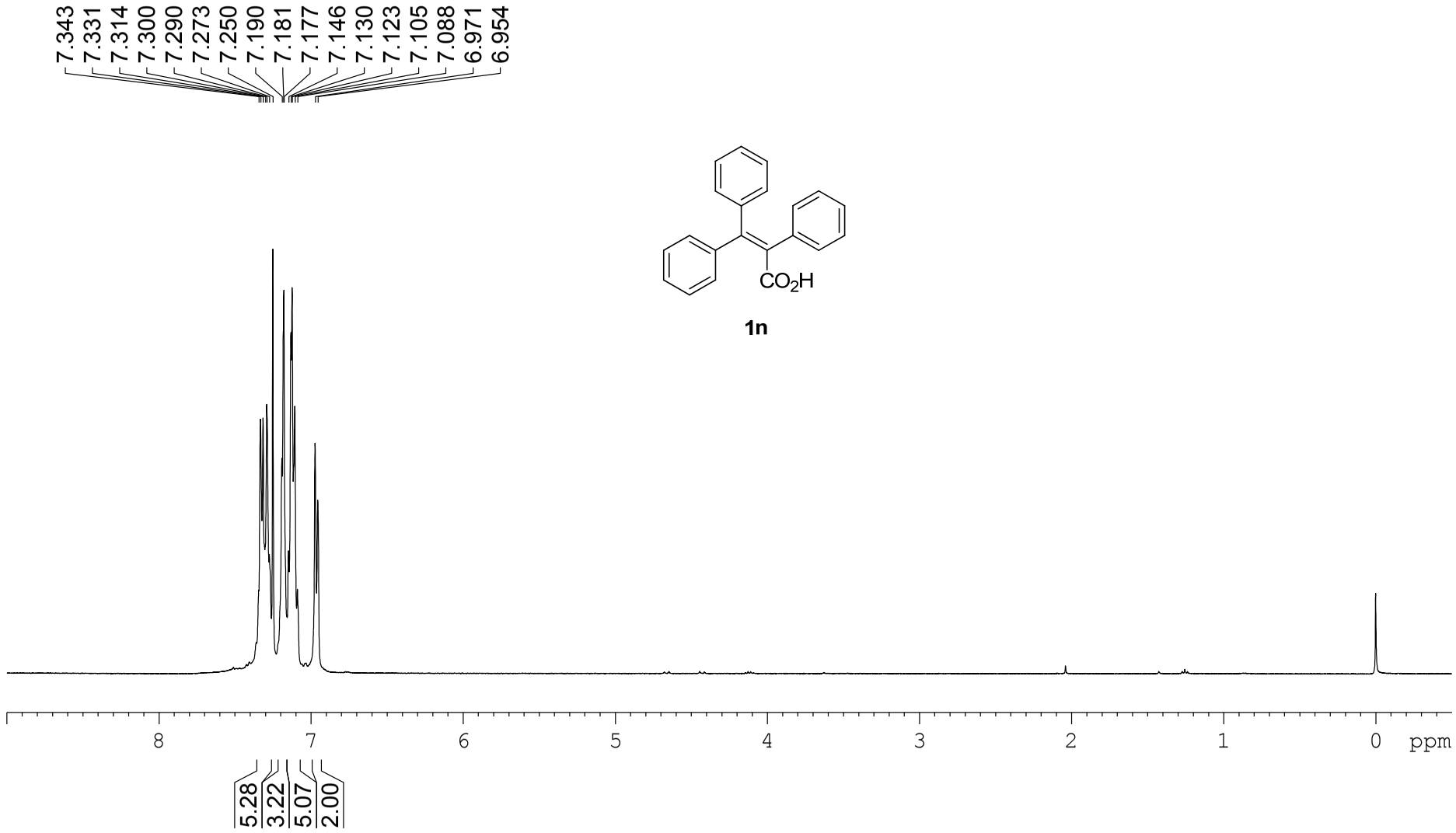
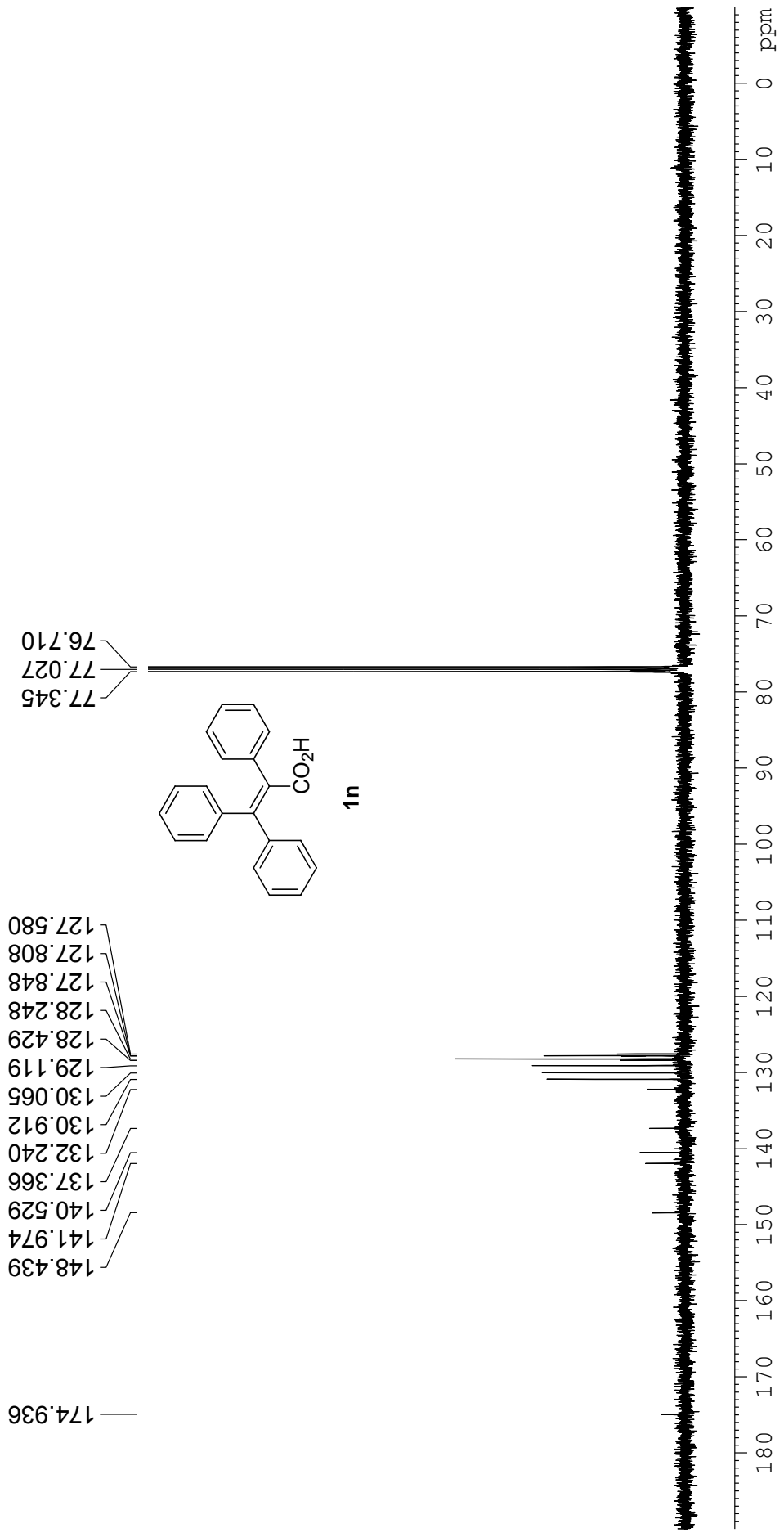
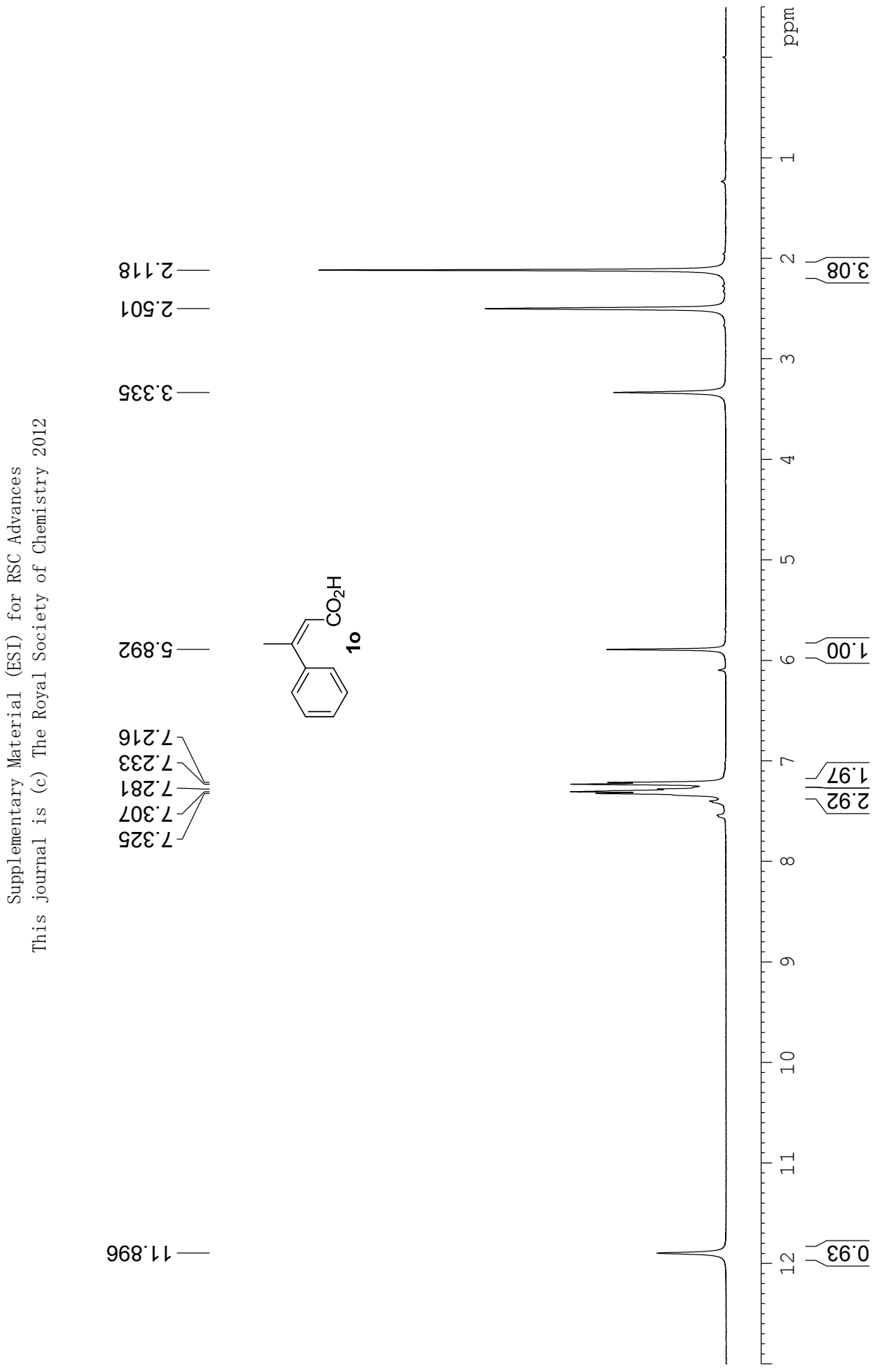
167.743
CN
CO2H
1m
ppm



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11.896
7.325
7.307
7.281
7.233
7.216
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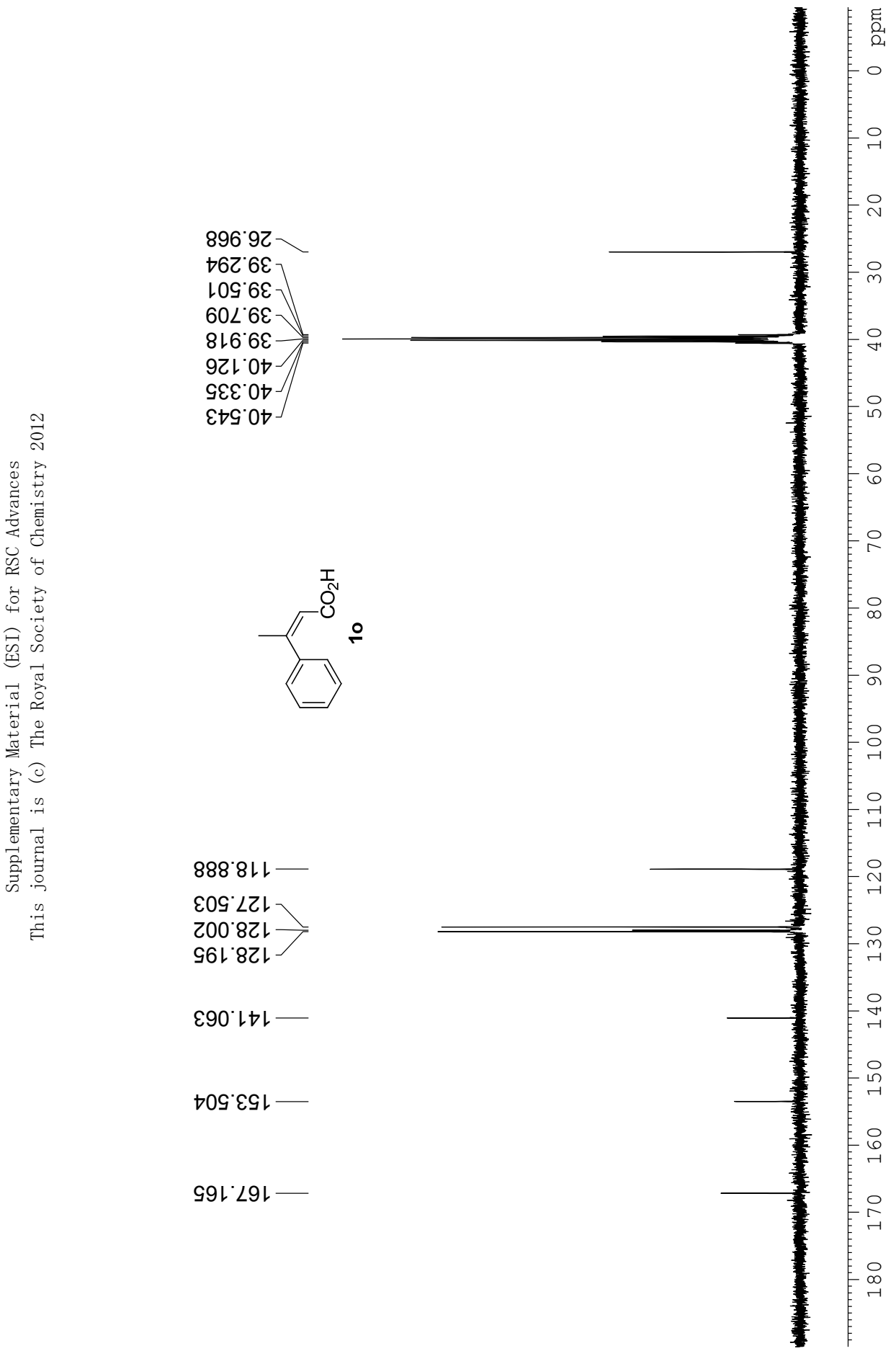
2.501
2.118
CO2H
1o
ppm
0.93
2.92
1.97
1.00
3.08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39.294
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39.709
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40.335
40.543
118.888
127.503
128.002
128.195
141.063
153.504
167.165
CO2H
1o
ppm
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20
30
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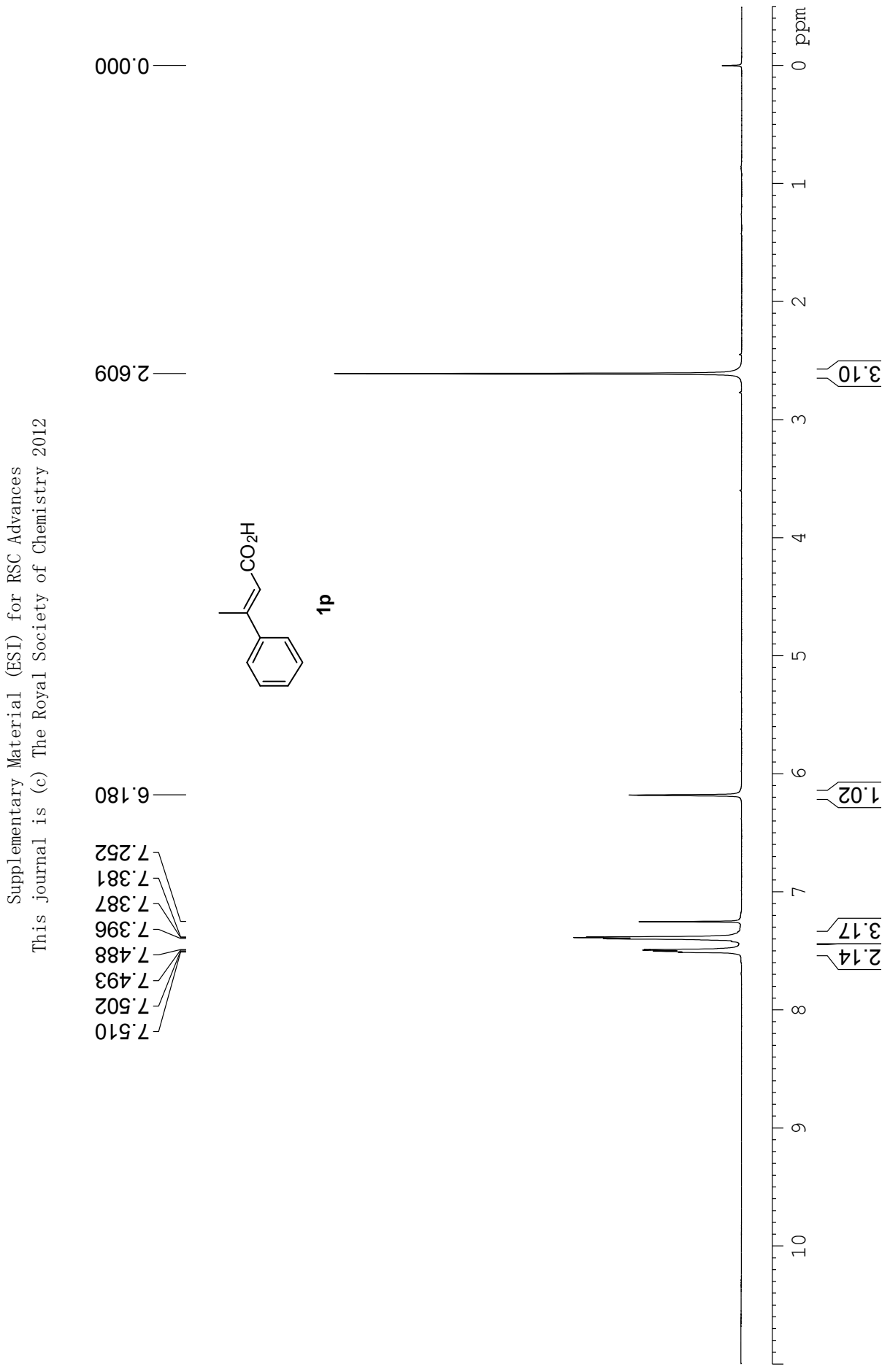
70
80
90
100
110
120
130
140
150
160
170
180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2.609
6.180
7.252
7.381
7.387
7.396
7.488
7.493
7.502
7.510
CO2H
1p
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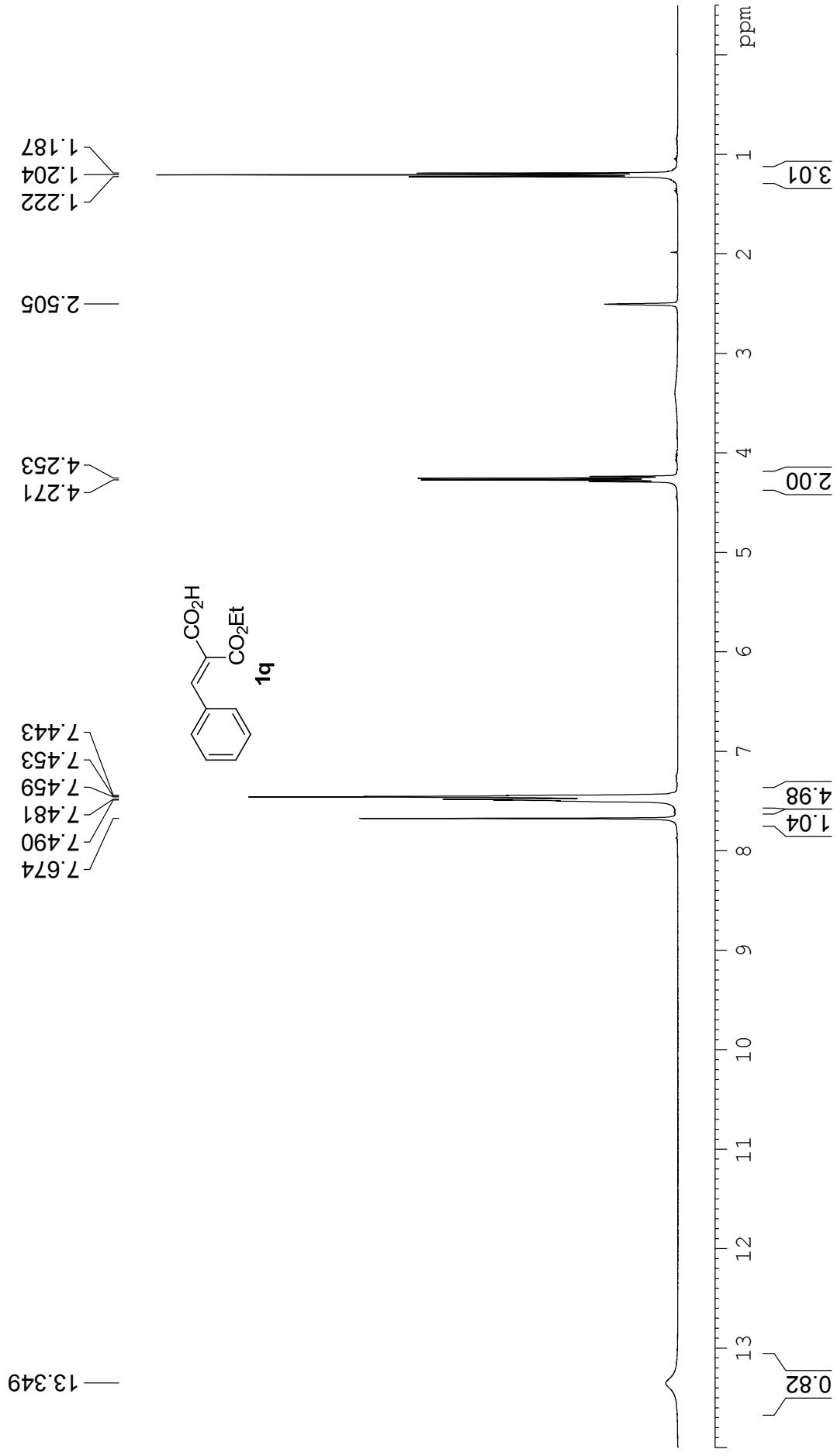
1.02
3.17
2.14
ppm
0
1
2
3
4
5
6
7
8
9
10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7.674
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7.481
7.459
7.453
7.443
4.271
4.253
2.505
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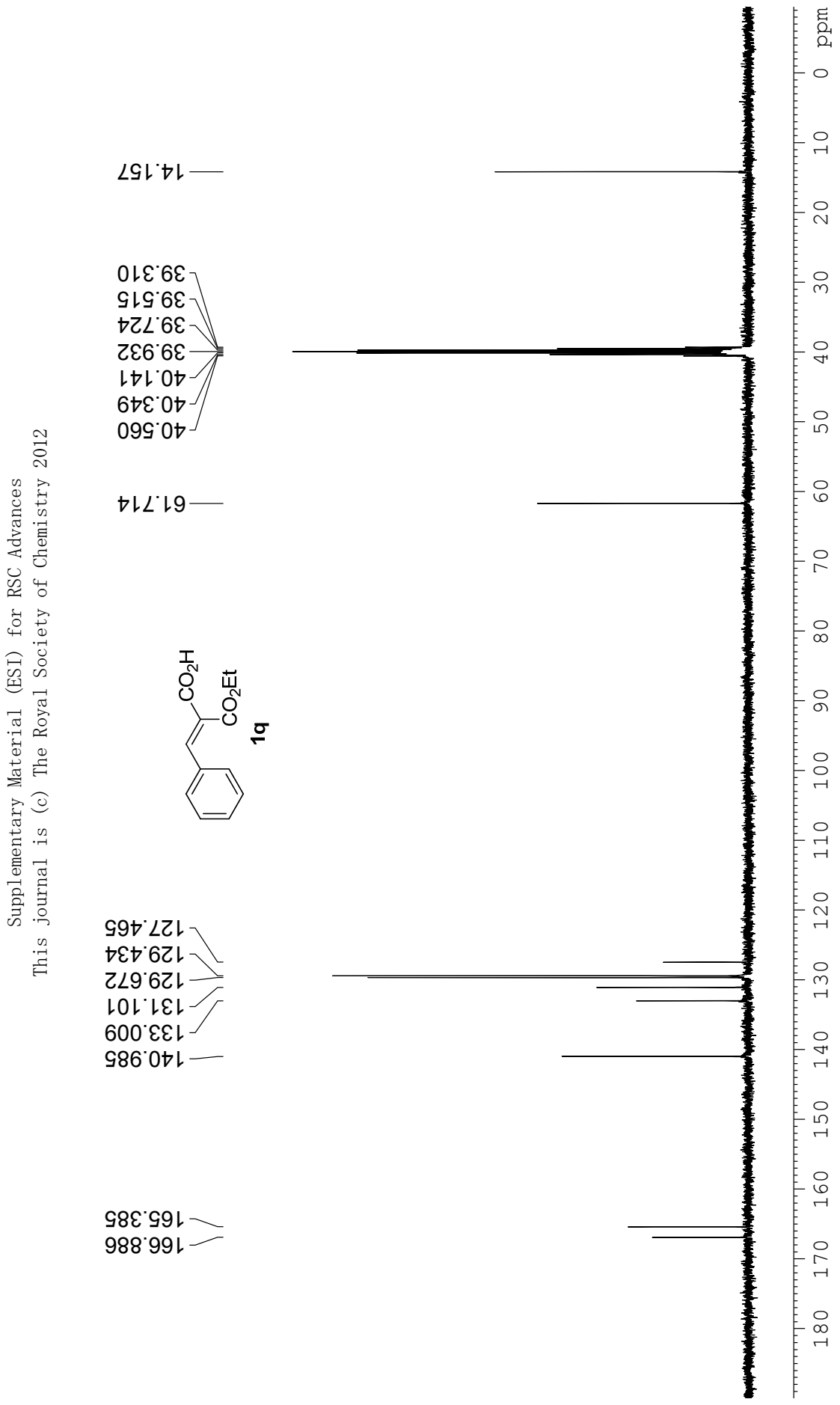
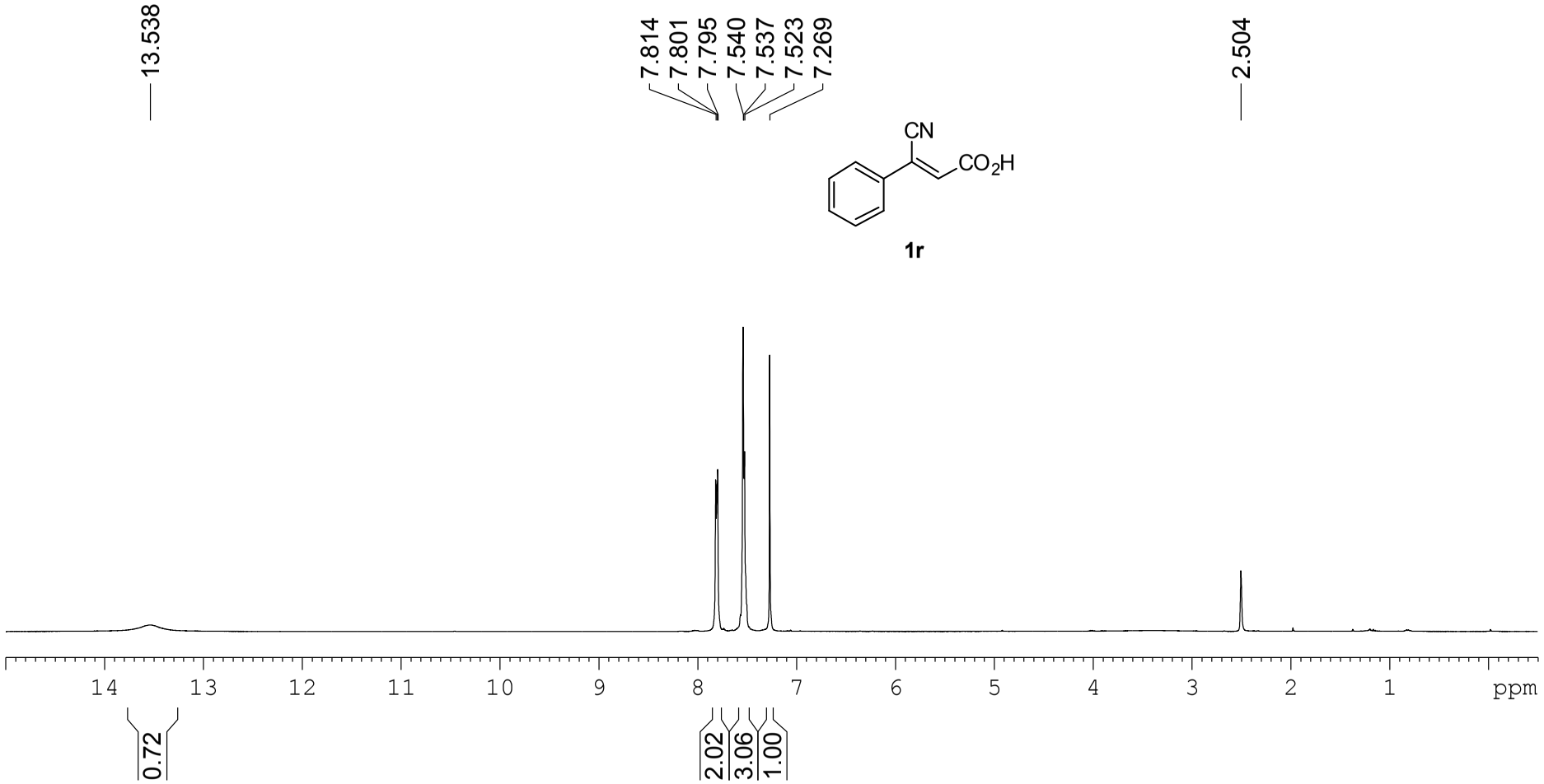
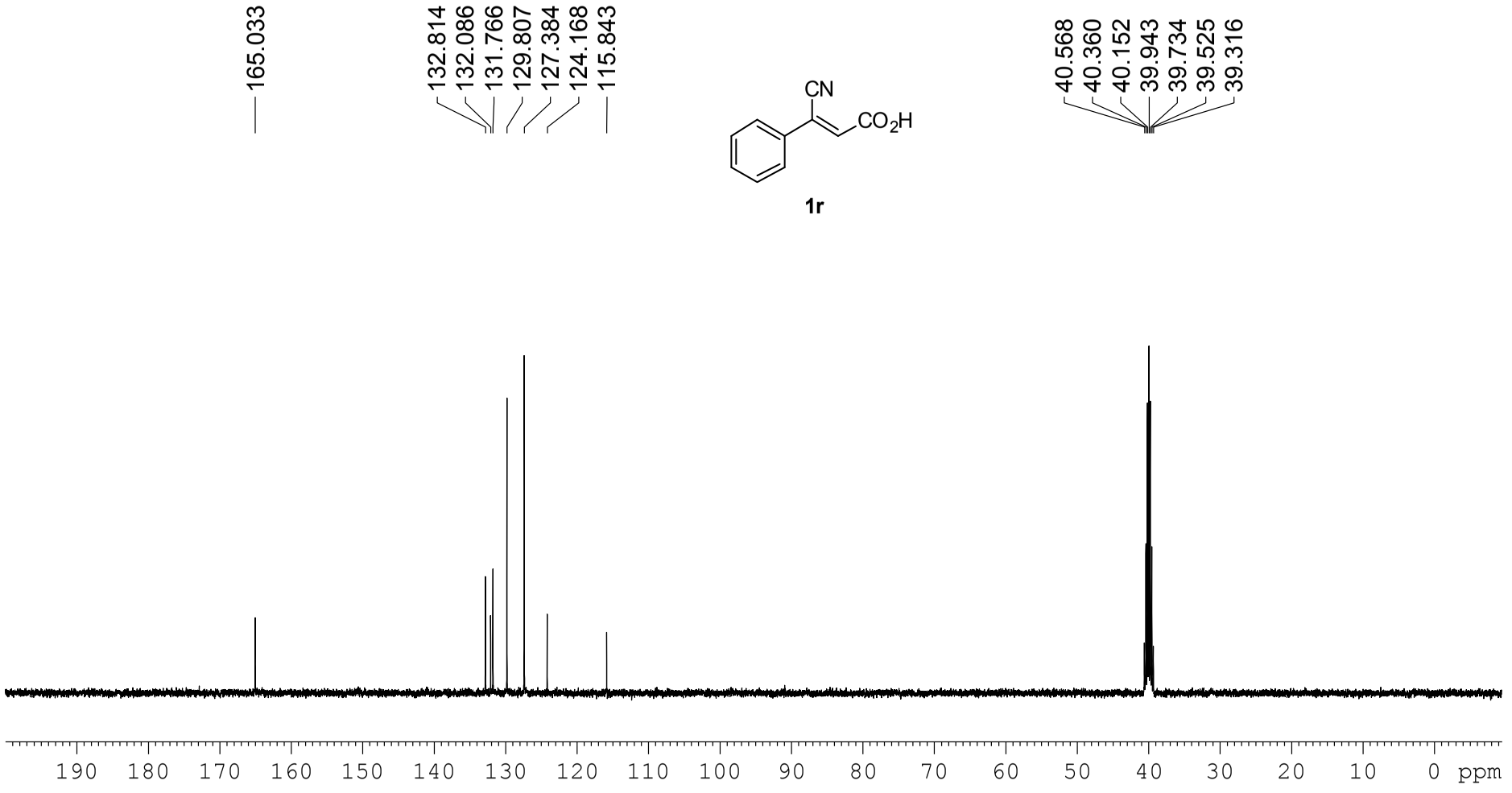
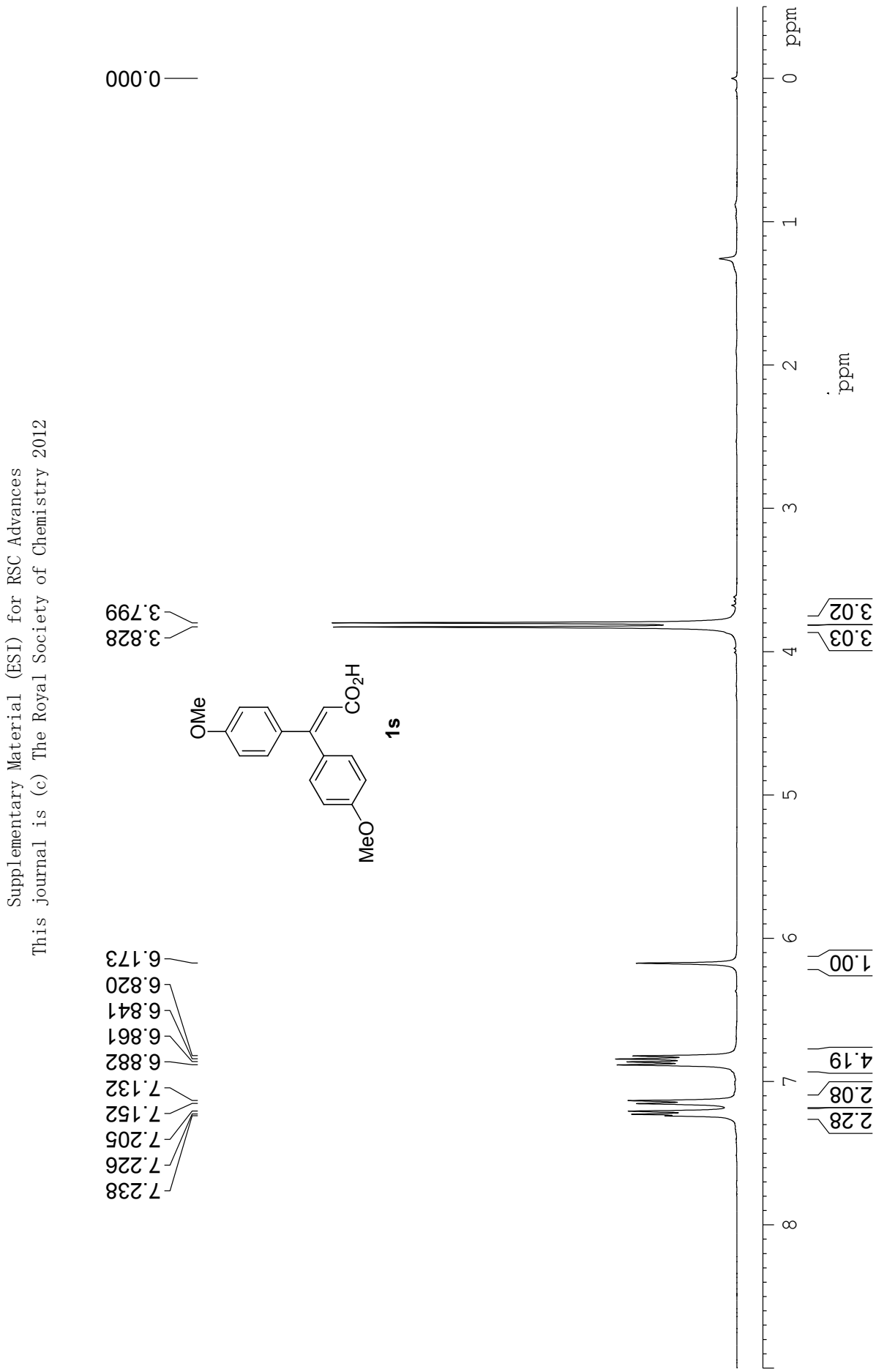
1.187
CO2H
CO2Et
1q
ppm
0.82
3.01
1.04
4.98
2.00





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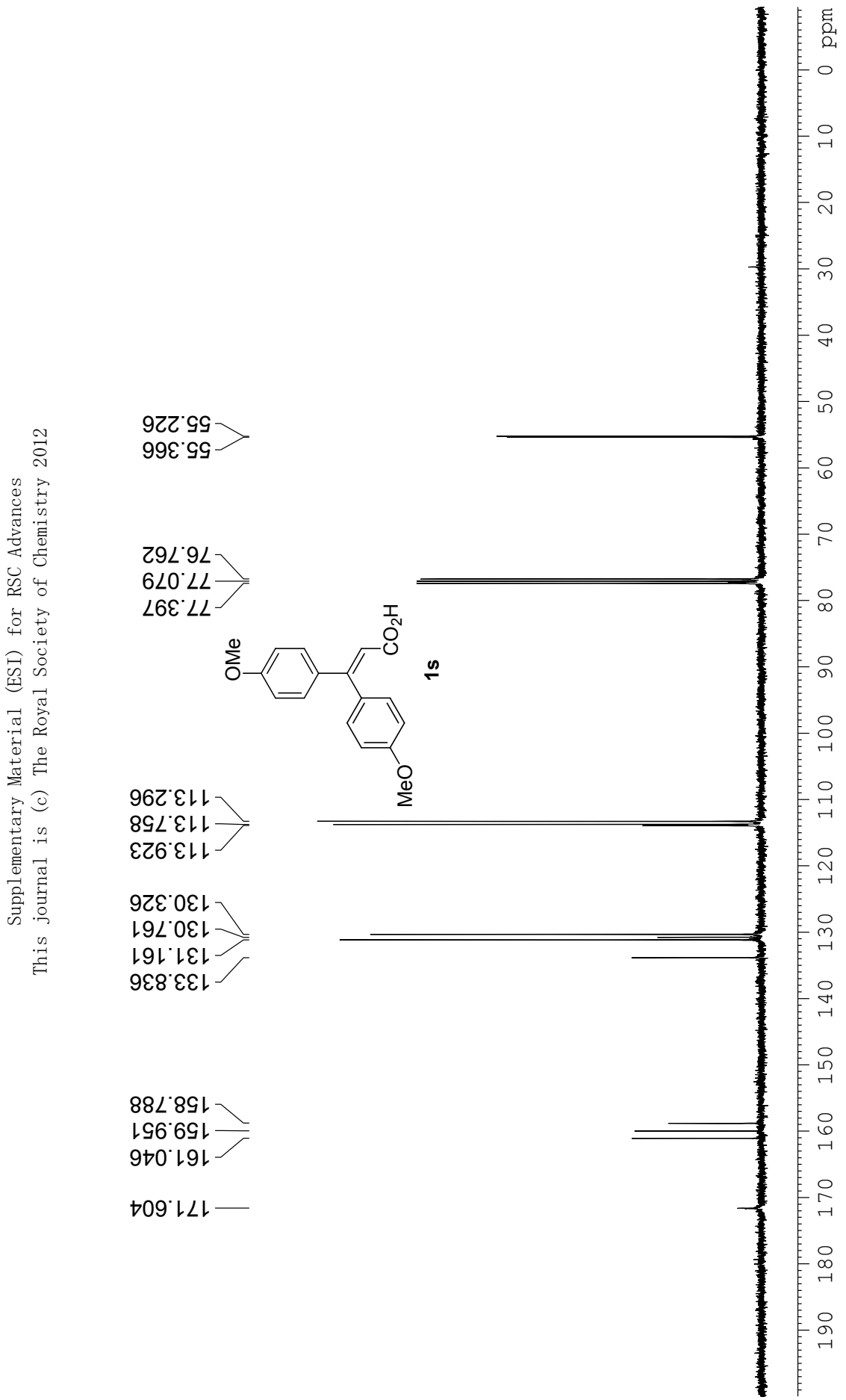
0.000
3.799
3.828
6.173
6.820
6.841
6.861
6.882
7.132
7.152
7.205
7.226
7.238
OMe
CO2H
MeO
1s
ppm
1.00
4.19
2.08
2.28
3.02
3.03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161.046
159.951
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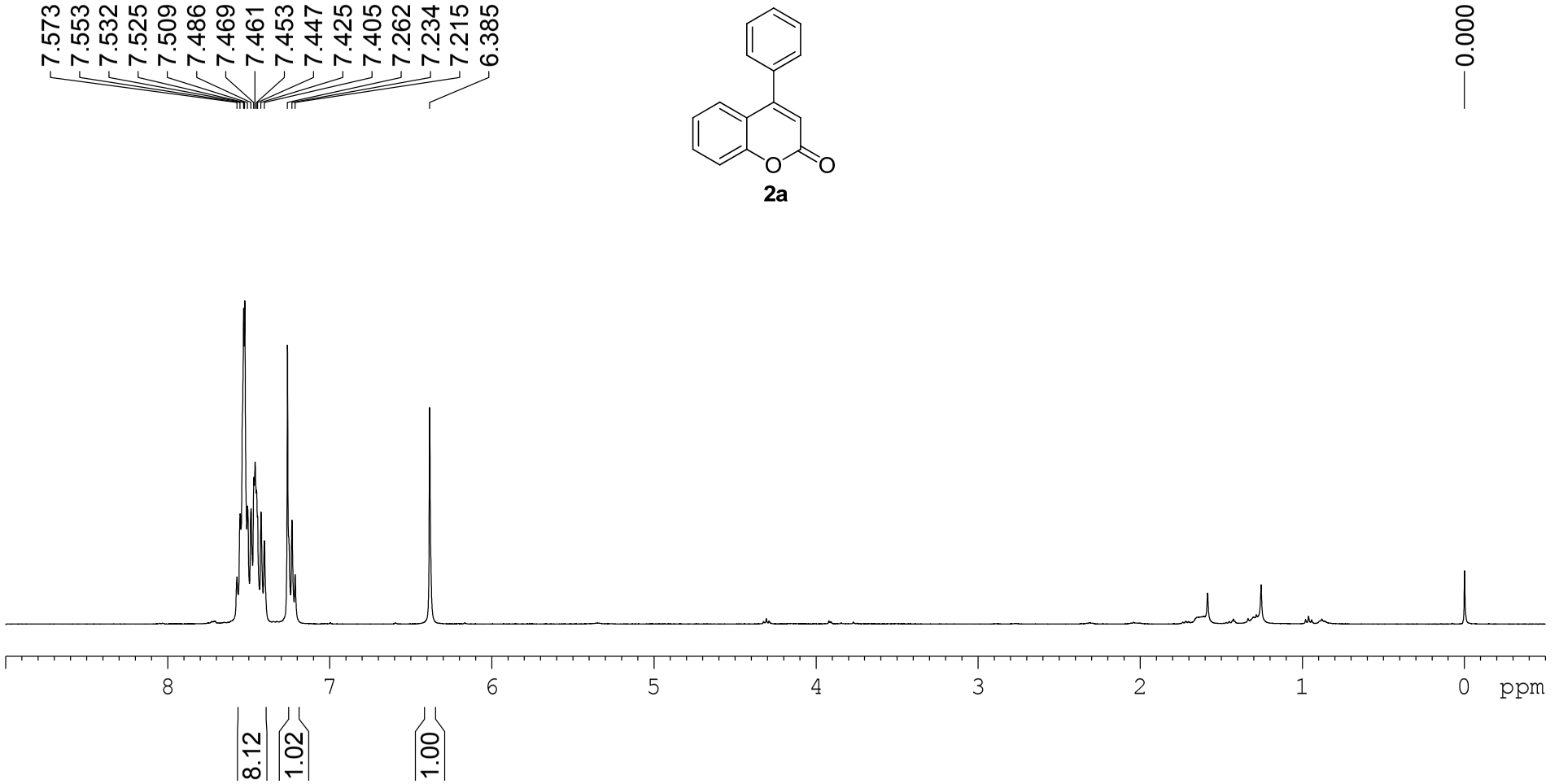
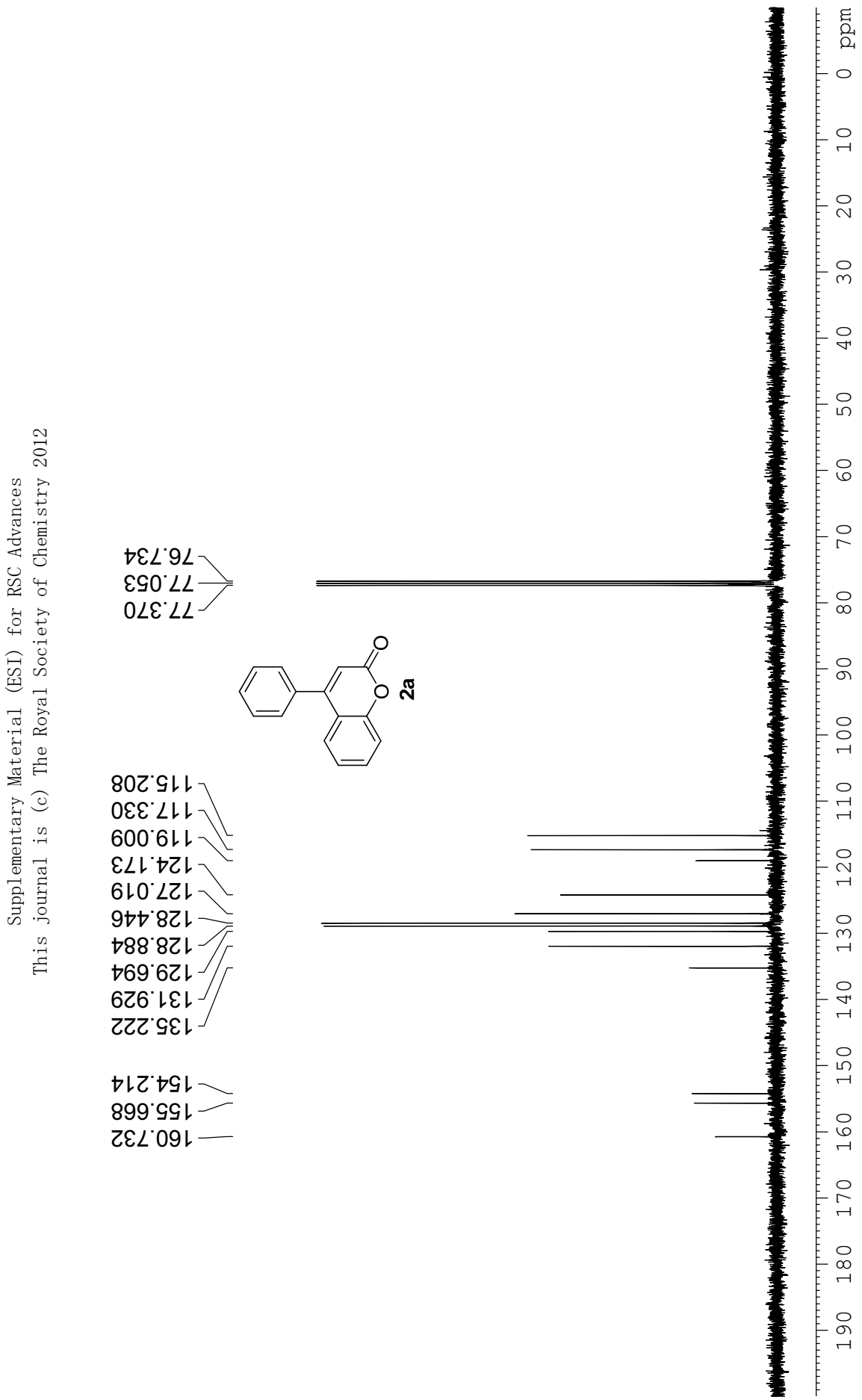
133.836
131.161
130.761
130.326
113.923
113.758
113.296
77.397
77.079
76.762
55.366
55.226
OMe
CO2H
MeO
1s
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm




Supplementary Material (ESI) for RSC Advances
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160.732
155.668
154.214
135.222
131.929
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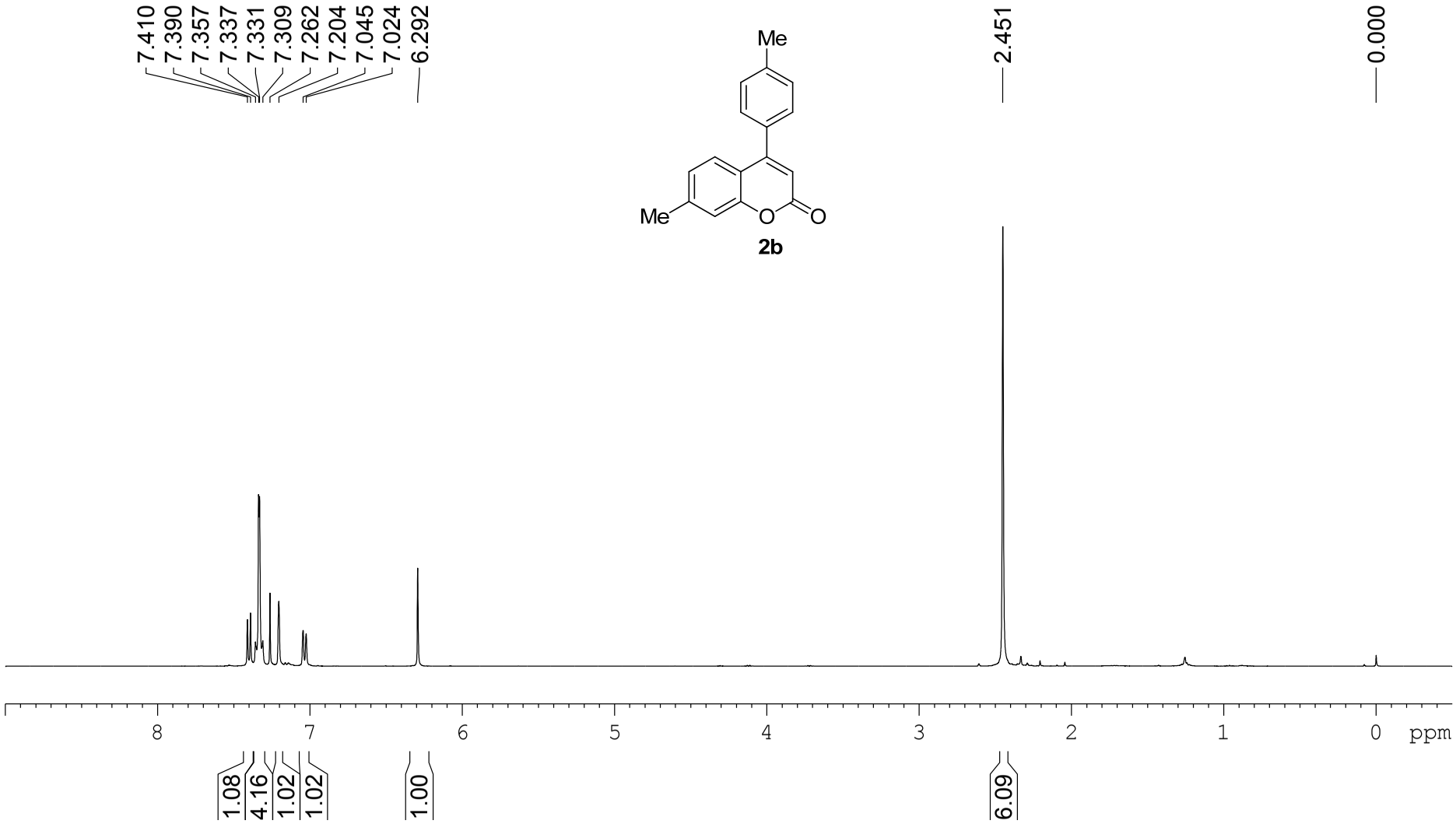

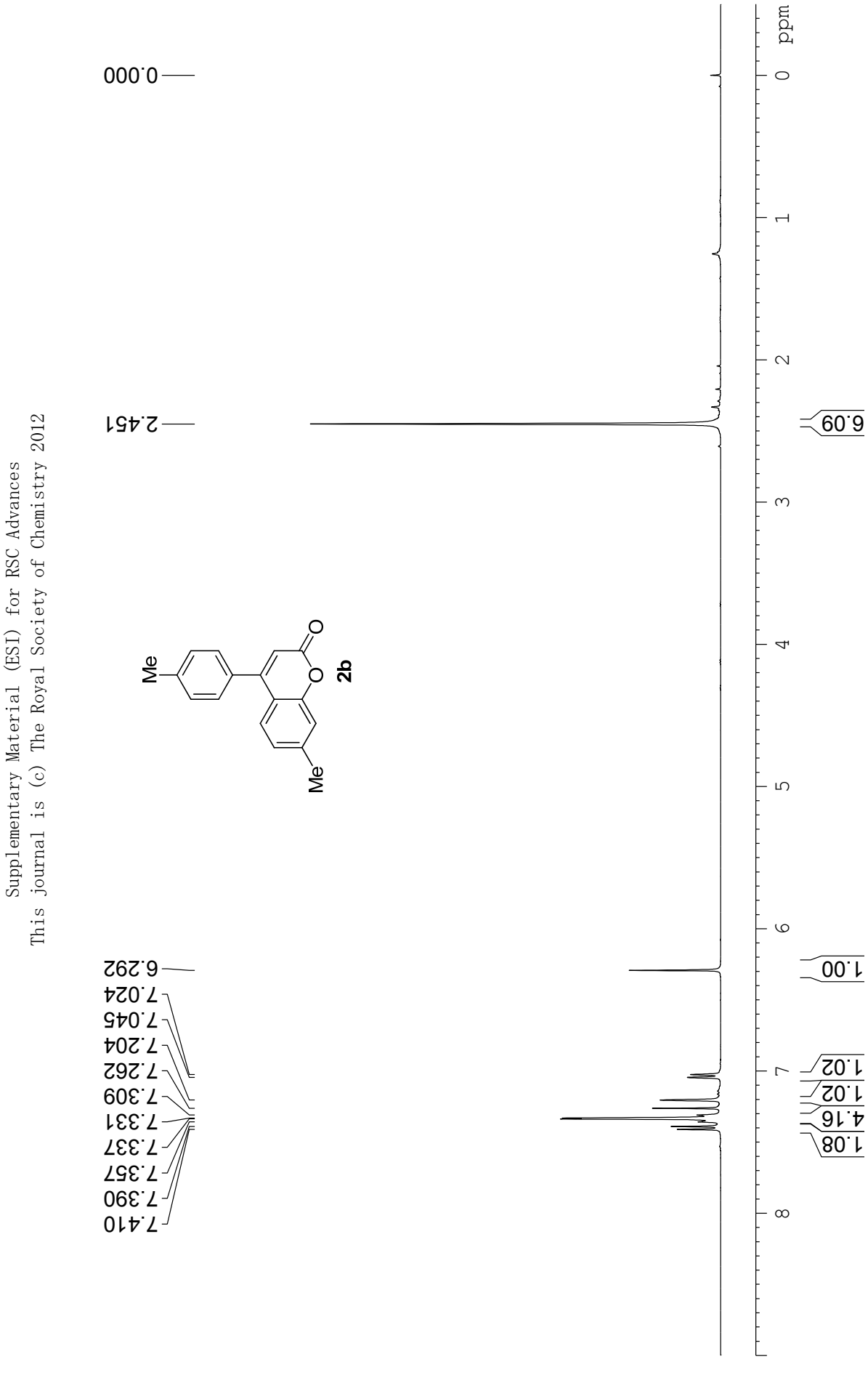
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7.045
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7.331
7.337
7.357
7.390
7.410
Me
Me
2b
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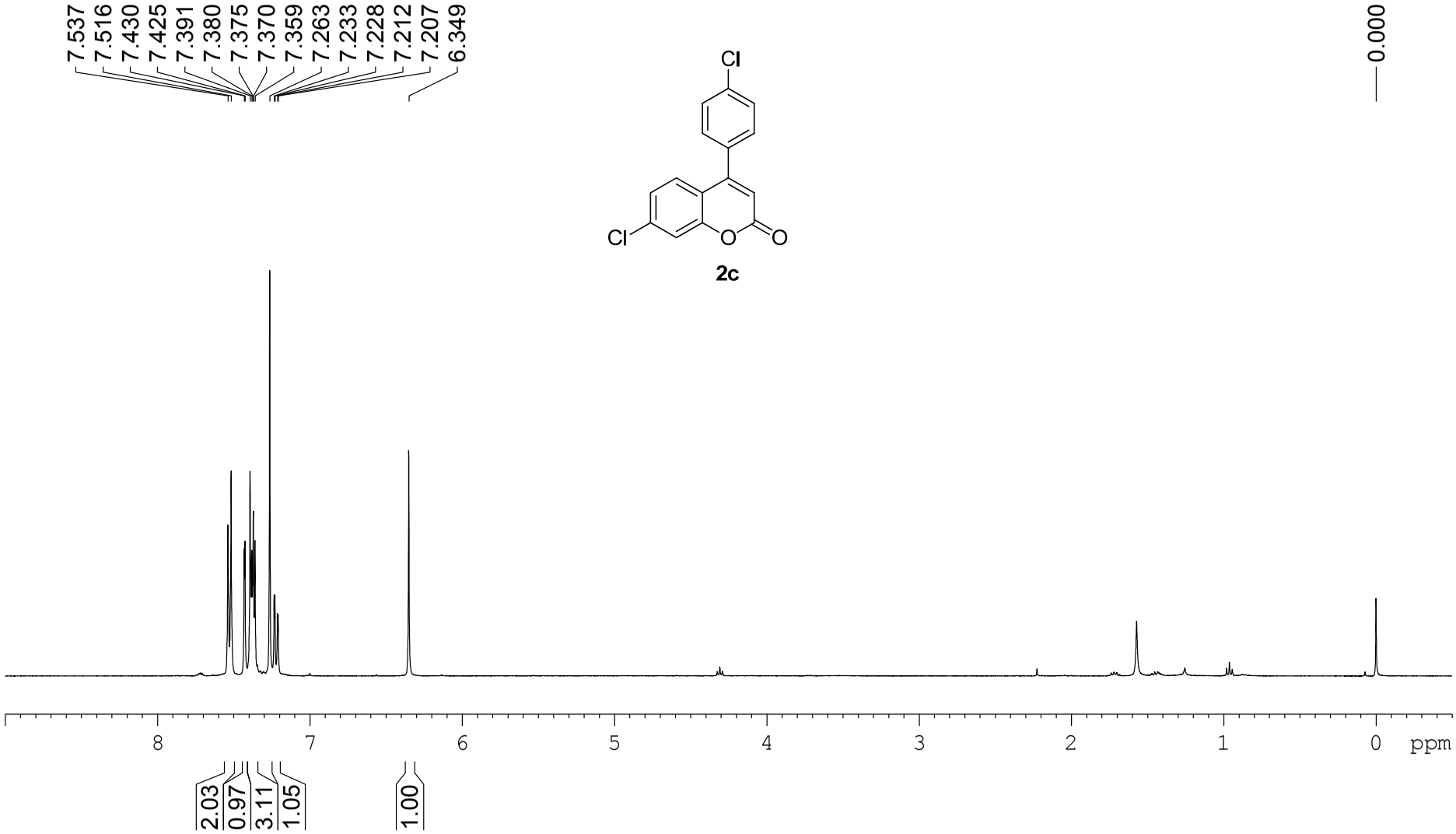
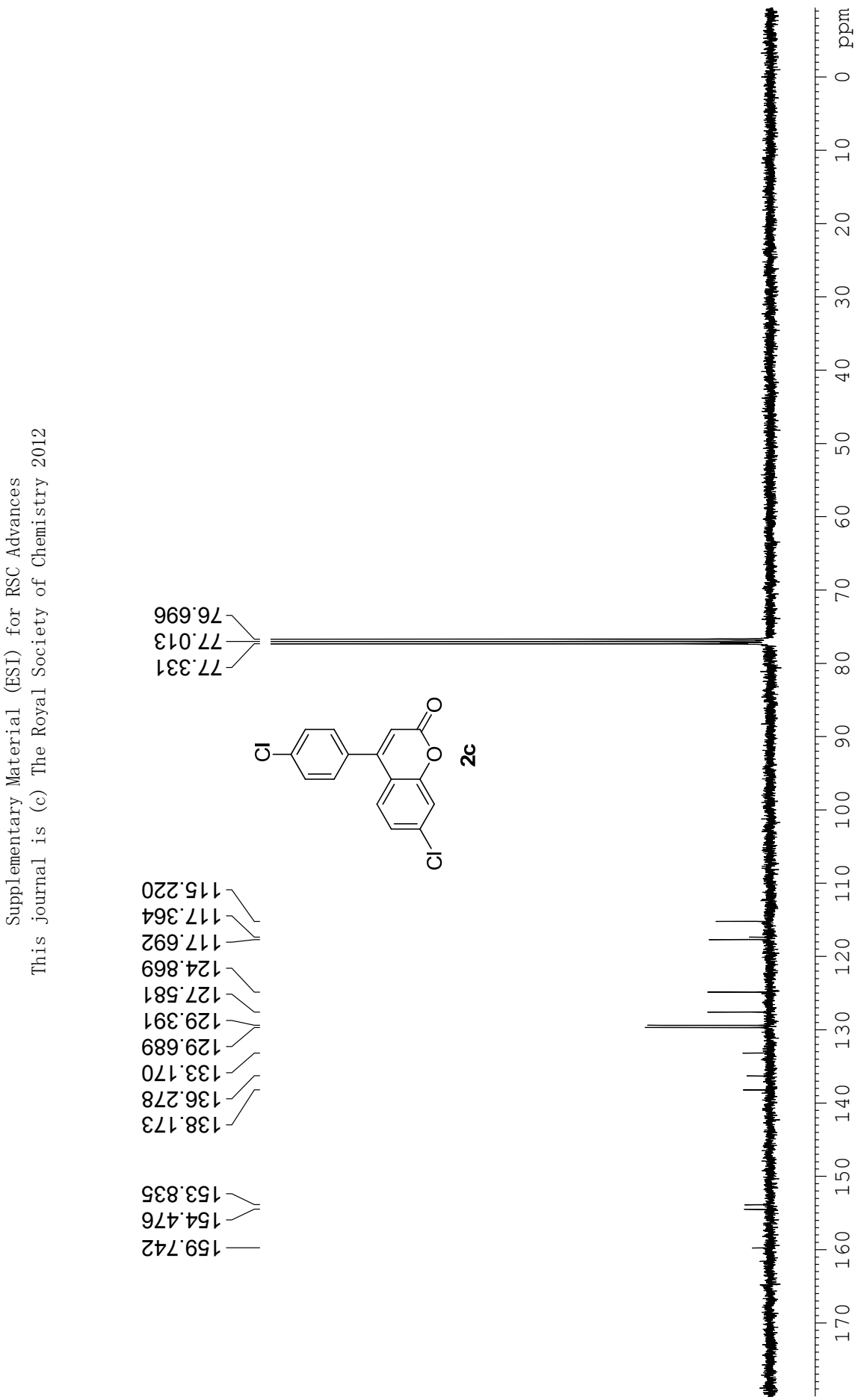
1.08




Supplementary Material (ESI) for RSC Advances
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127.581
129.391
129.689
133.170
136.278
138.173
153.835
154.476
159.742
Cl
O
O
Cl
2c
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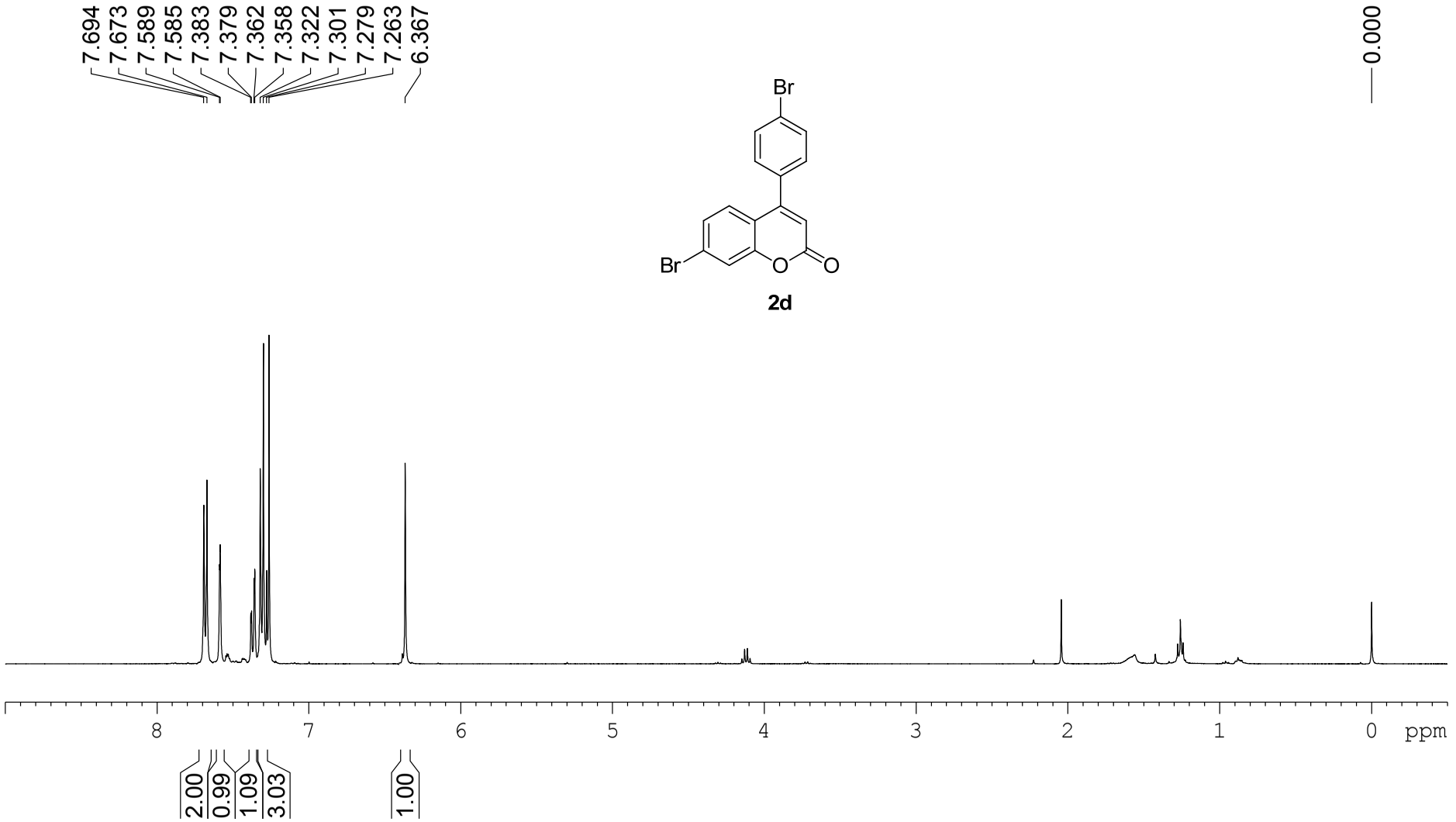

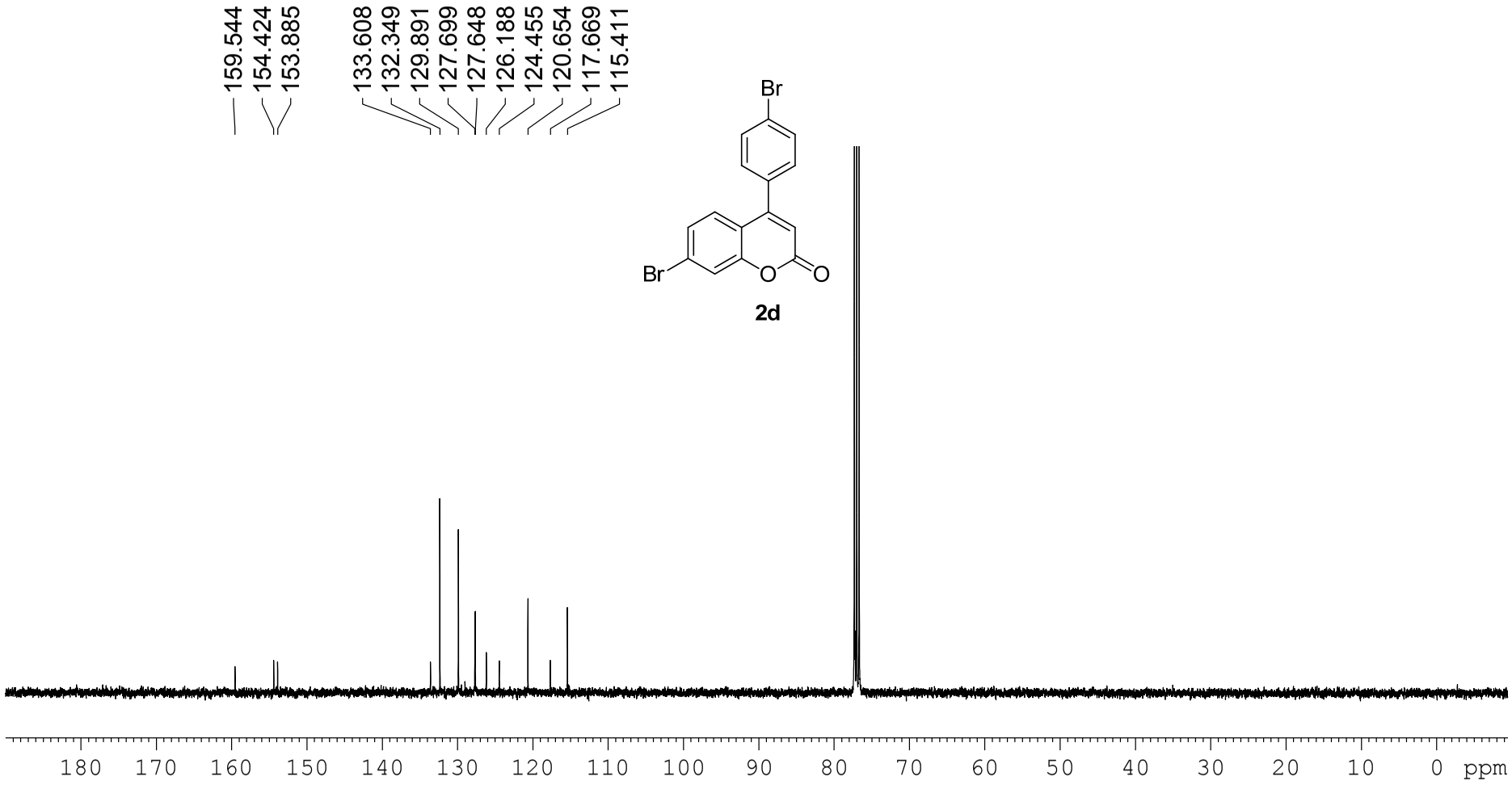

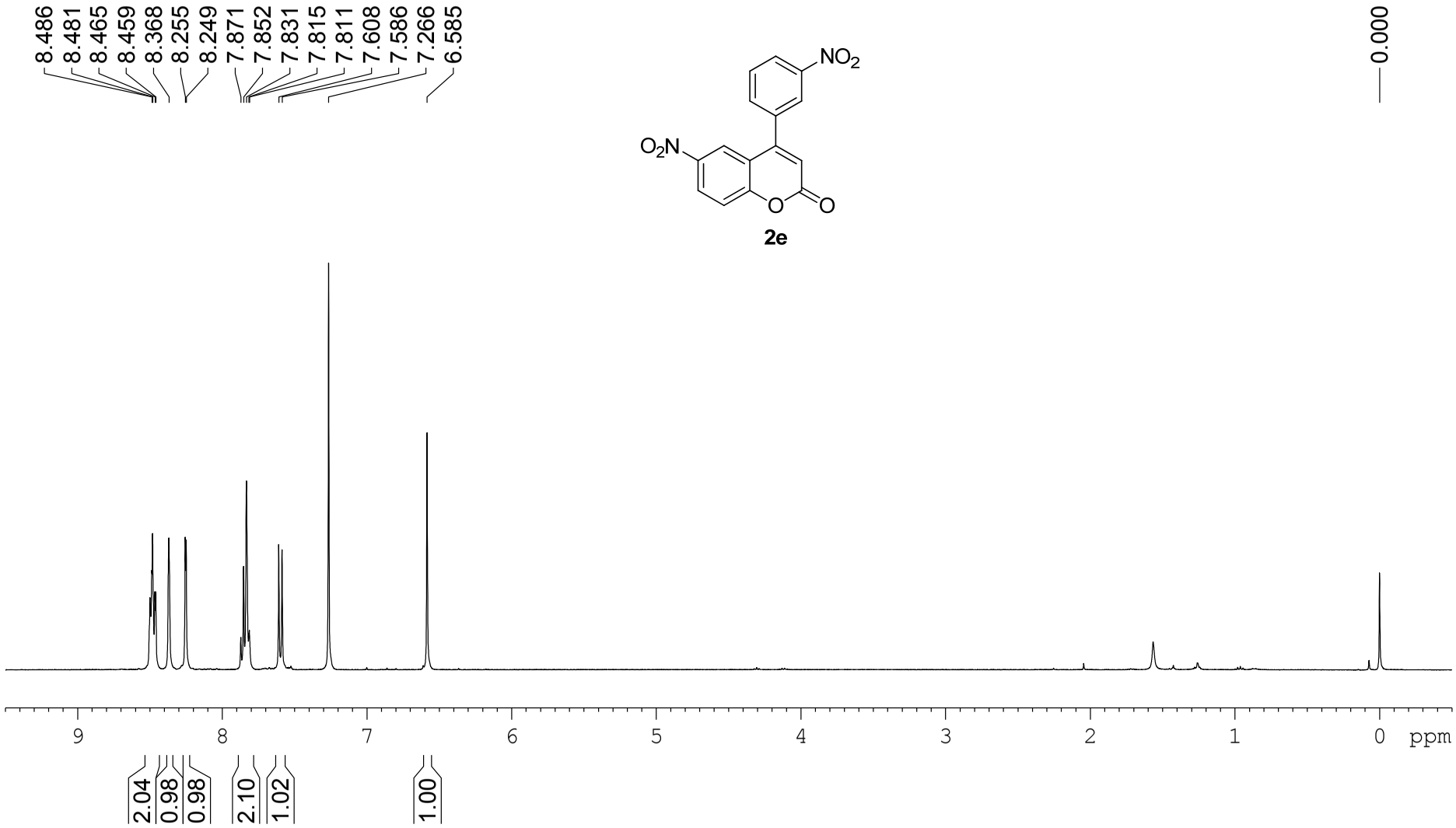

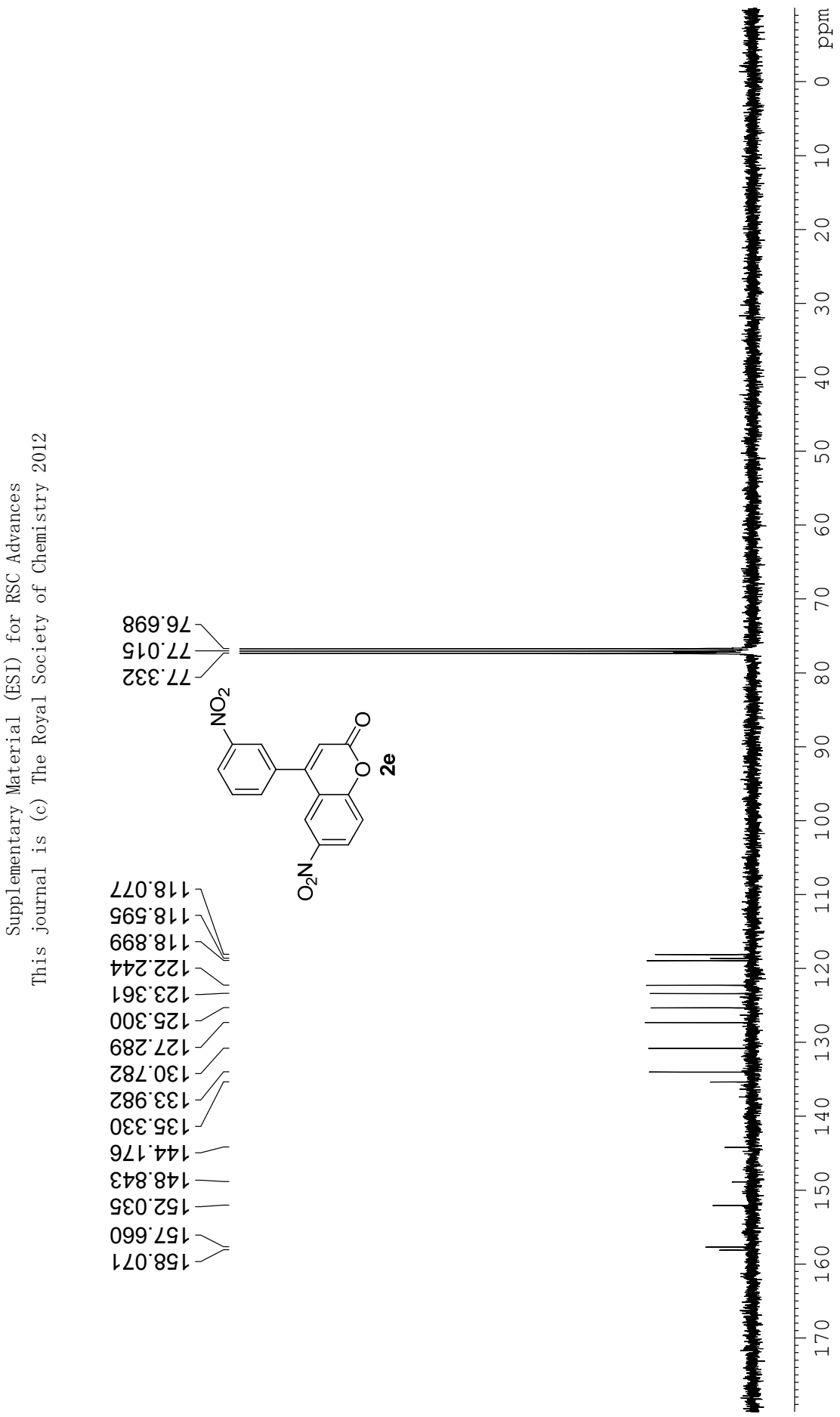
Supplementary Material (ESI) for RSC Advances
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130.782
127.289
125.300
123.361
122.244
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118.595
118.077
77.332
77.015
76.698
NO2
O2N
O
O
2e
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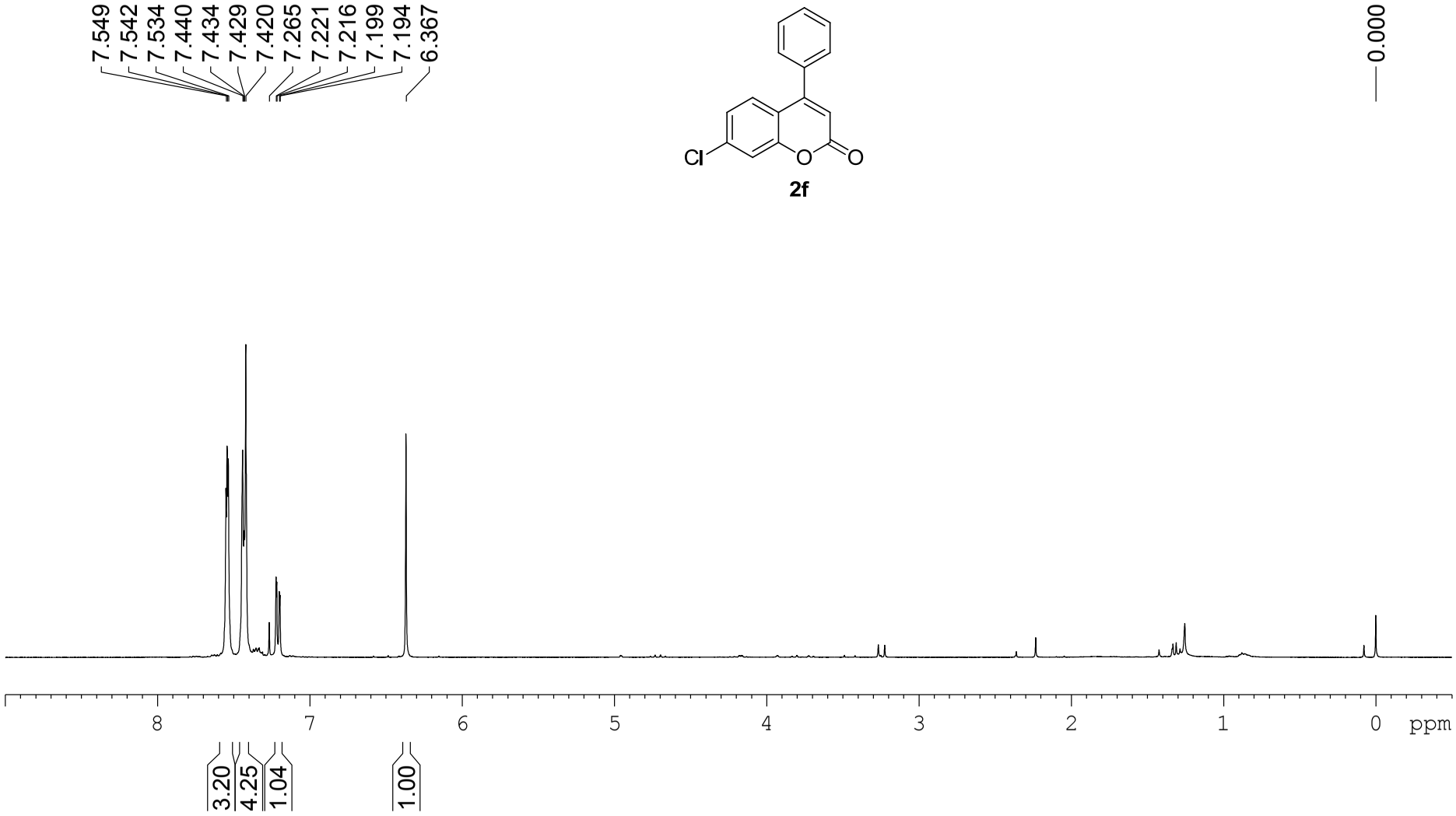

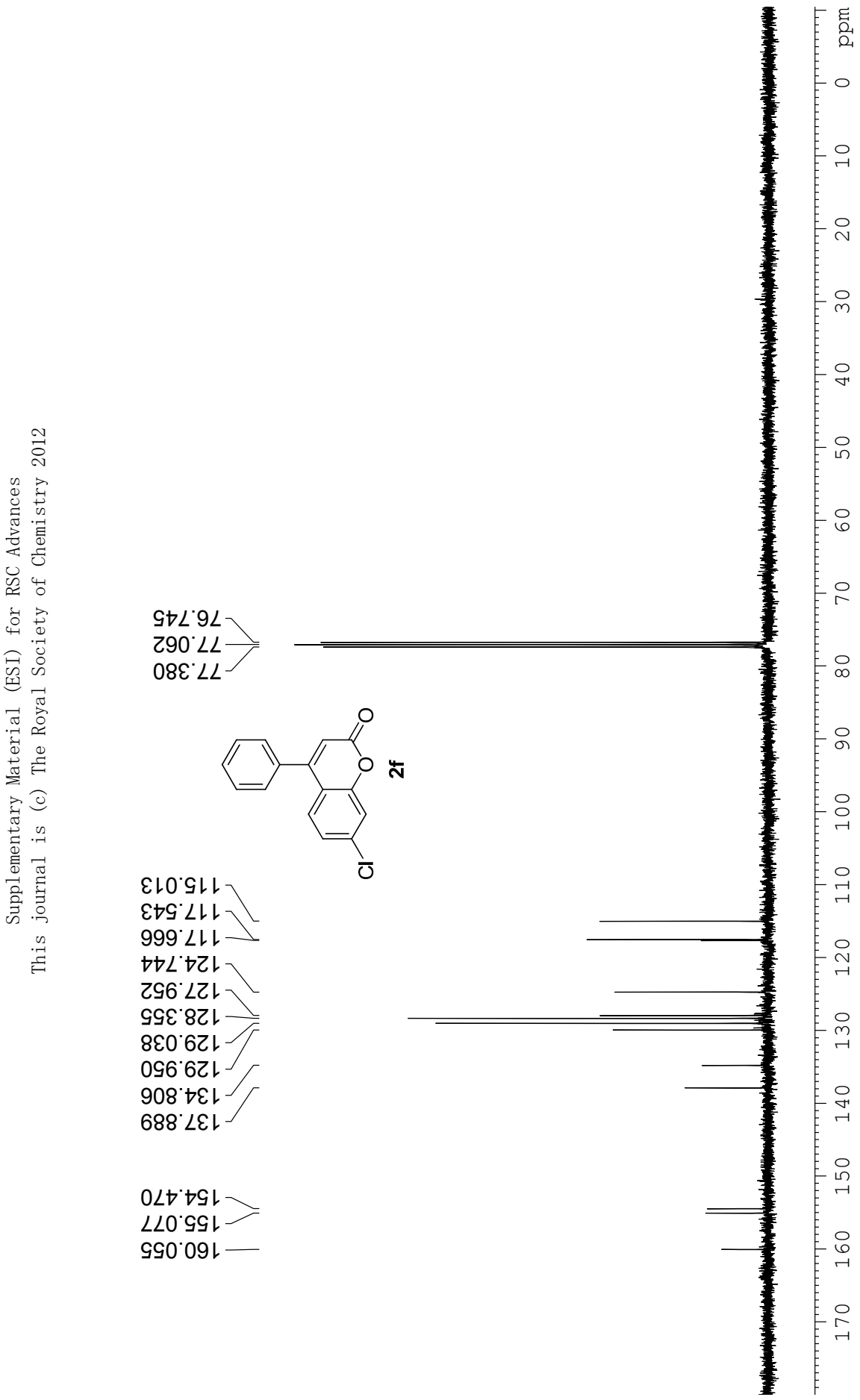
Supplementary Material (ESI) for RSC Advances
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129.950
134.806
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154.470
155.077
160.055
O
O
Cl
2f
ppm
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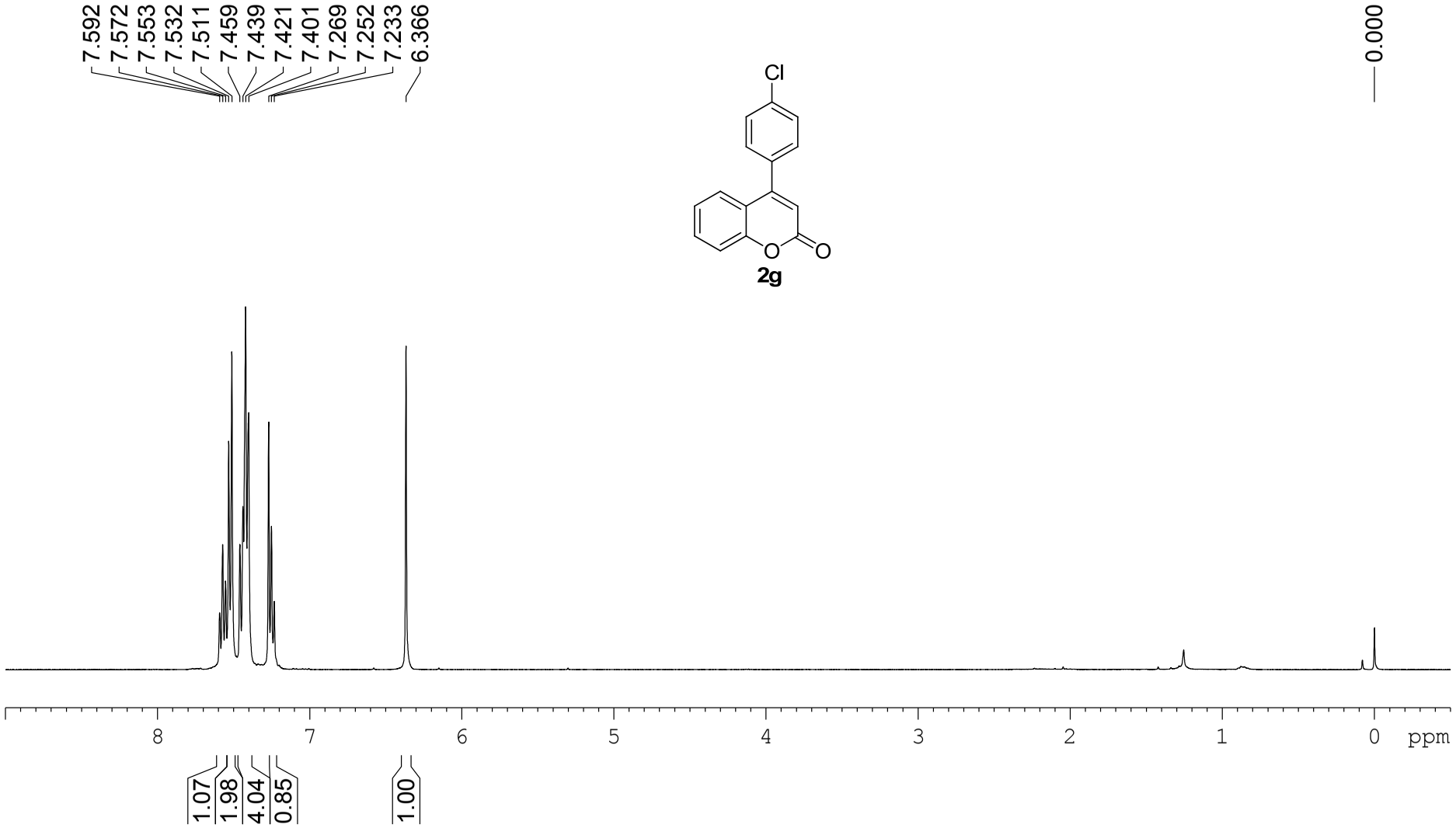

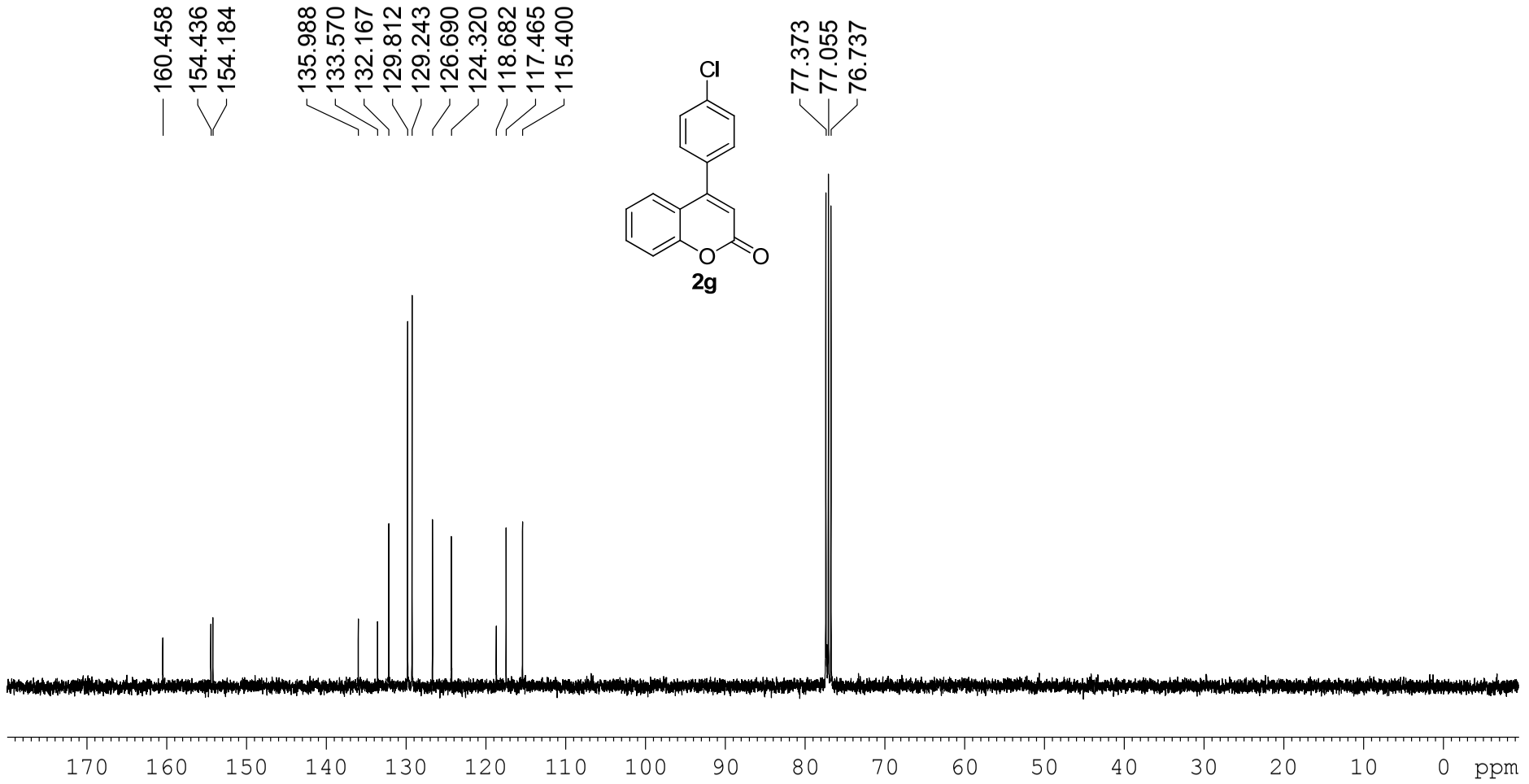

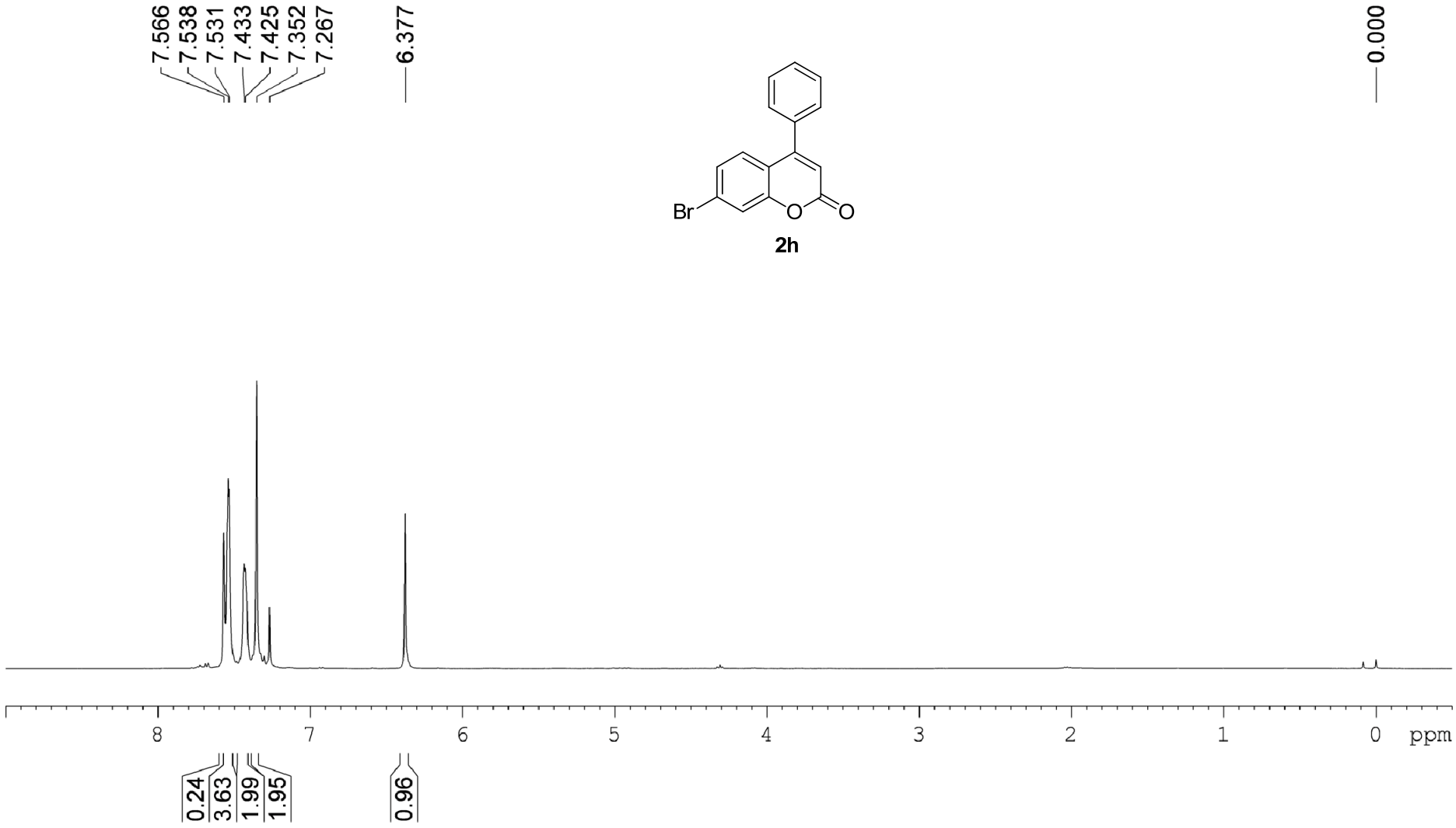

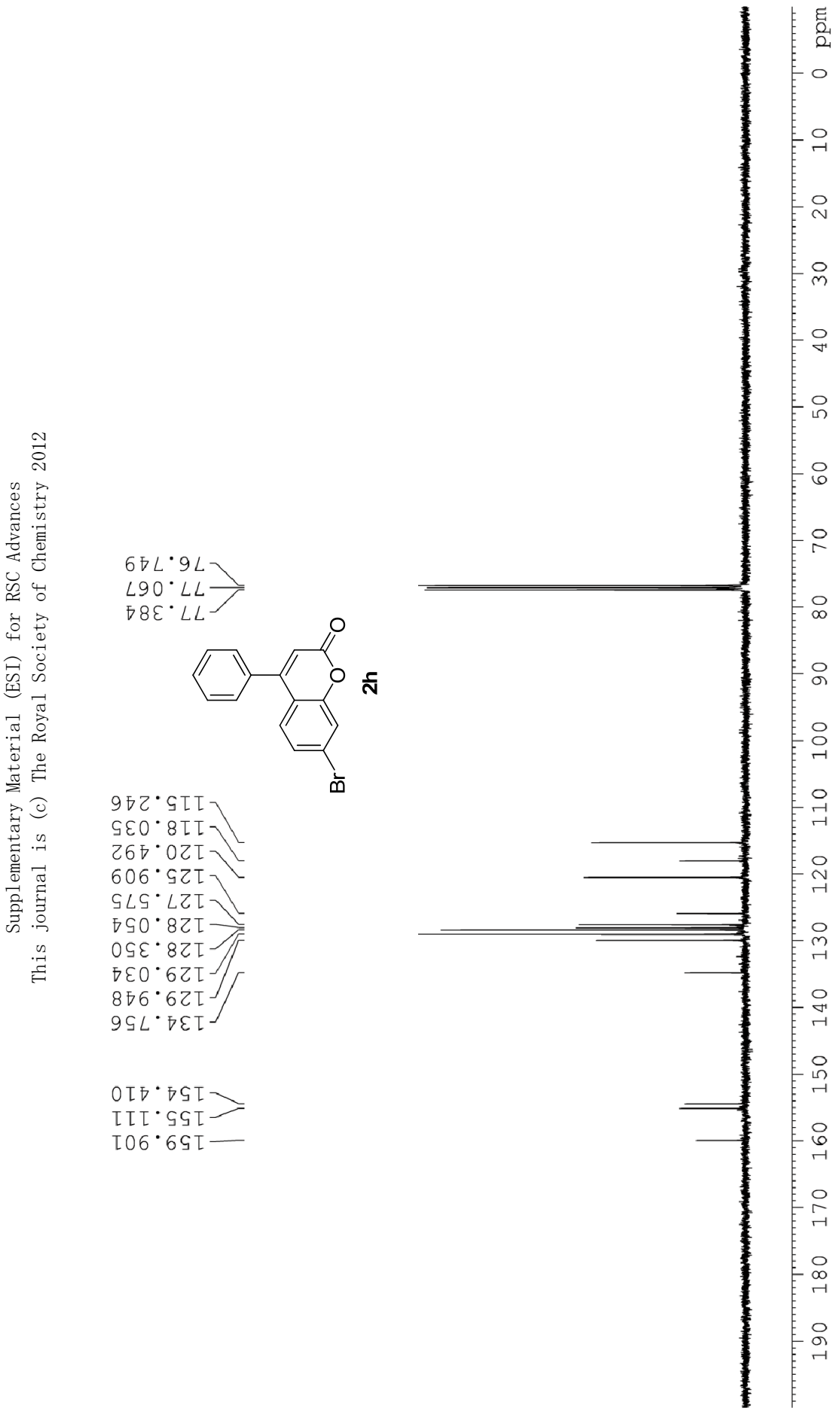
Supplementary Material (ESI) for RSC Advances
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O
O
Br
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118.035
120.492
125.909
127.575
128.054
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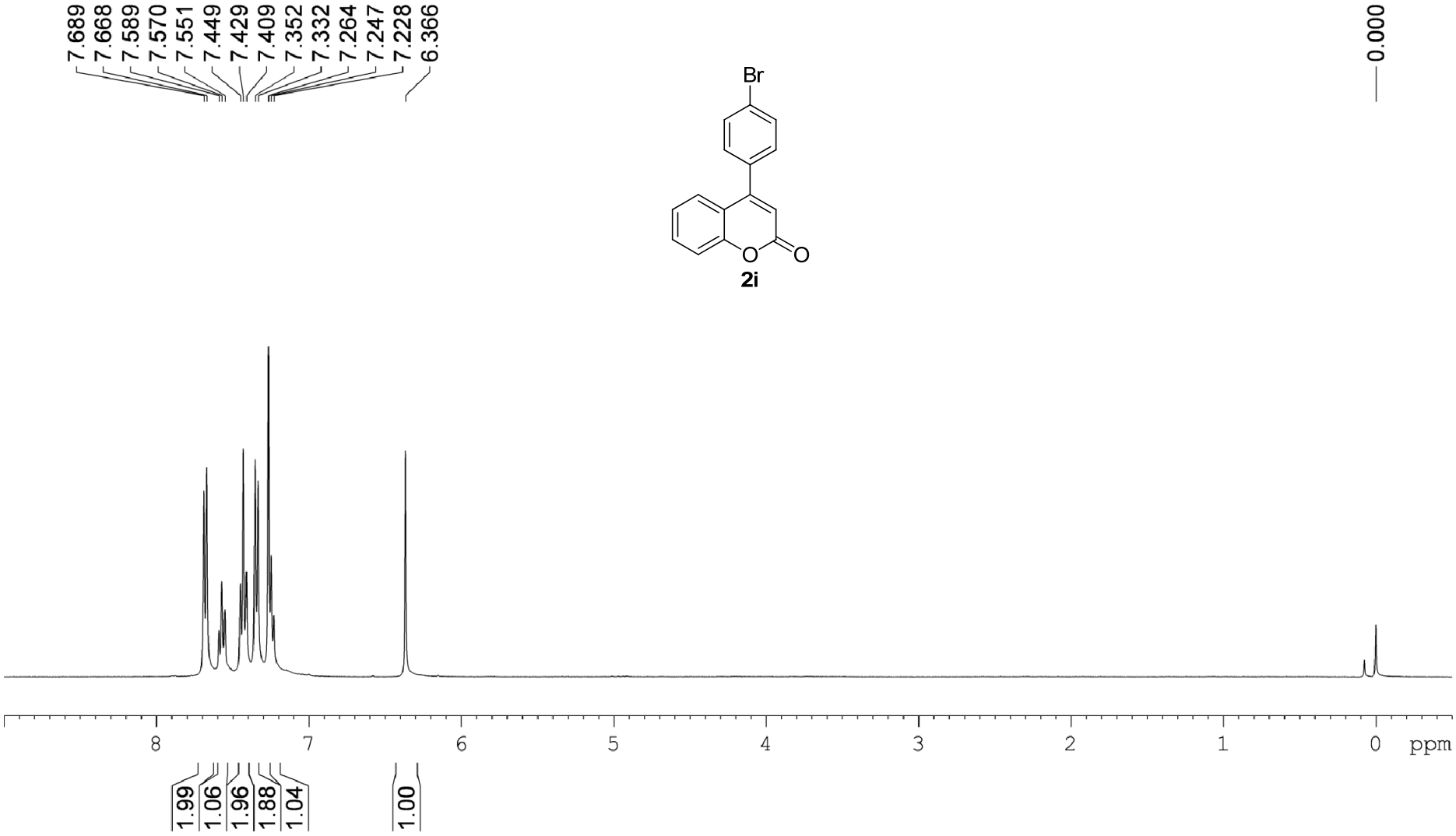

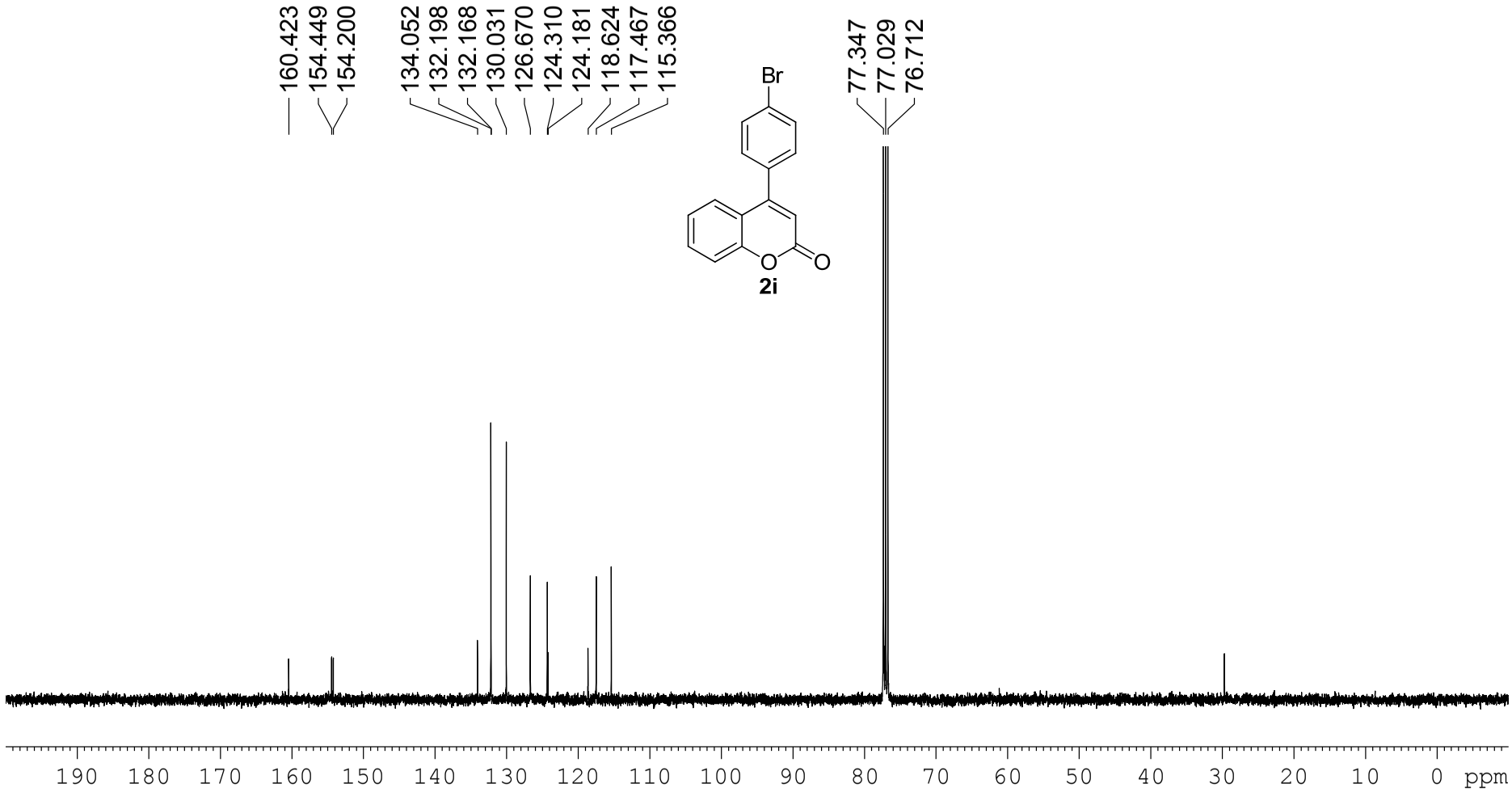

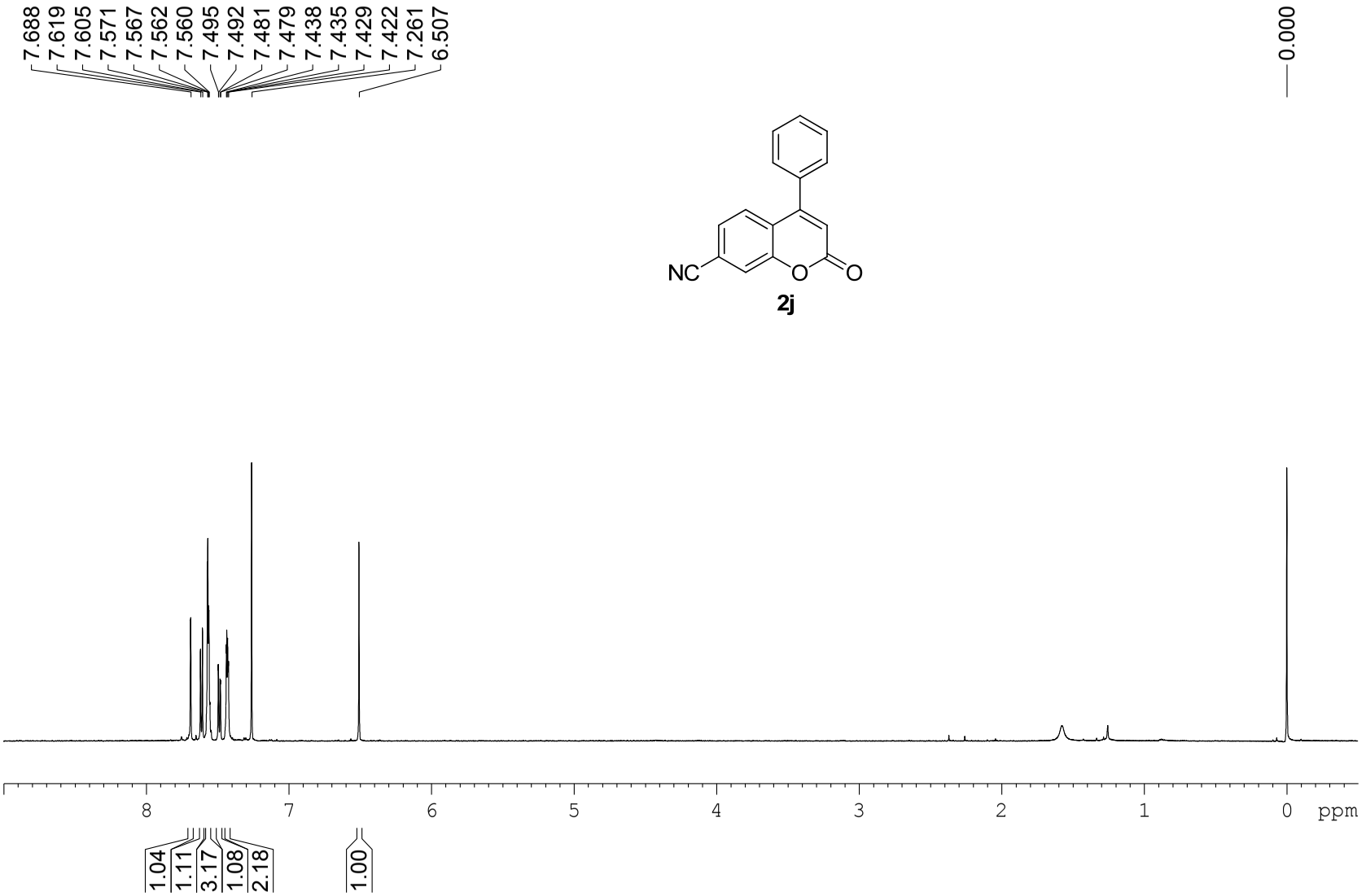

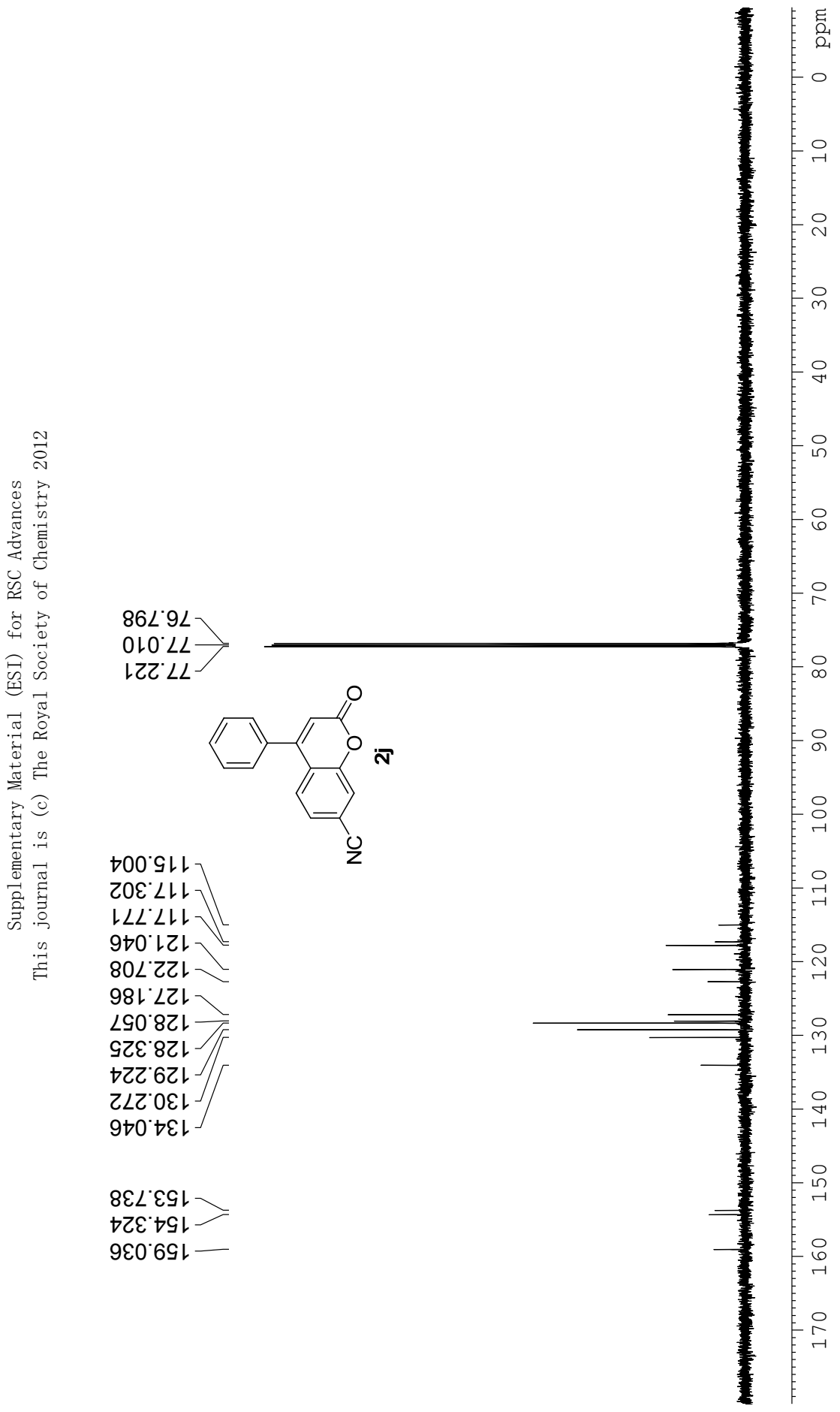
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128.057
127.186
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121.046
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115.004
77.221
77.010
76.798
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O
O
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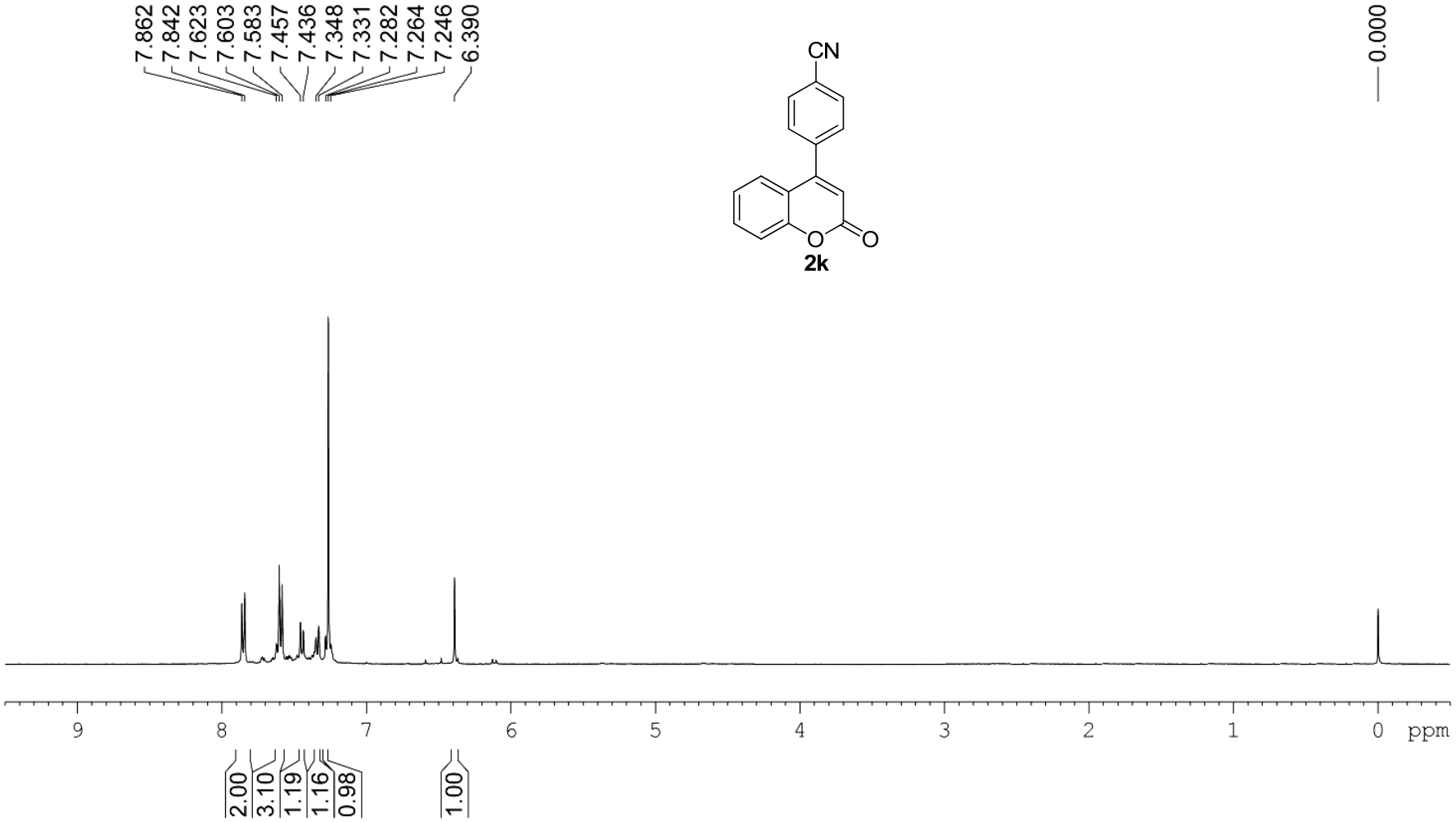

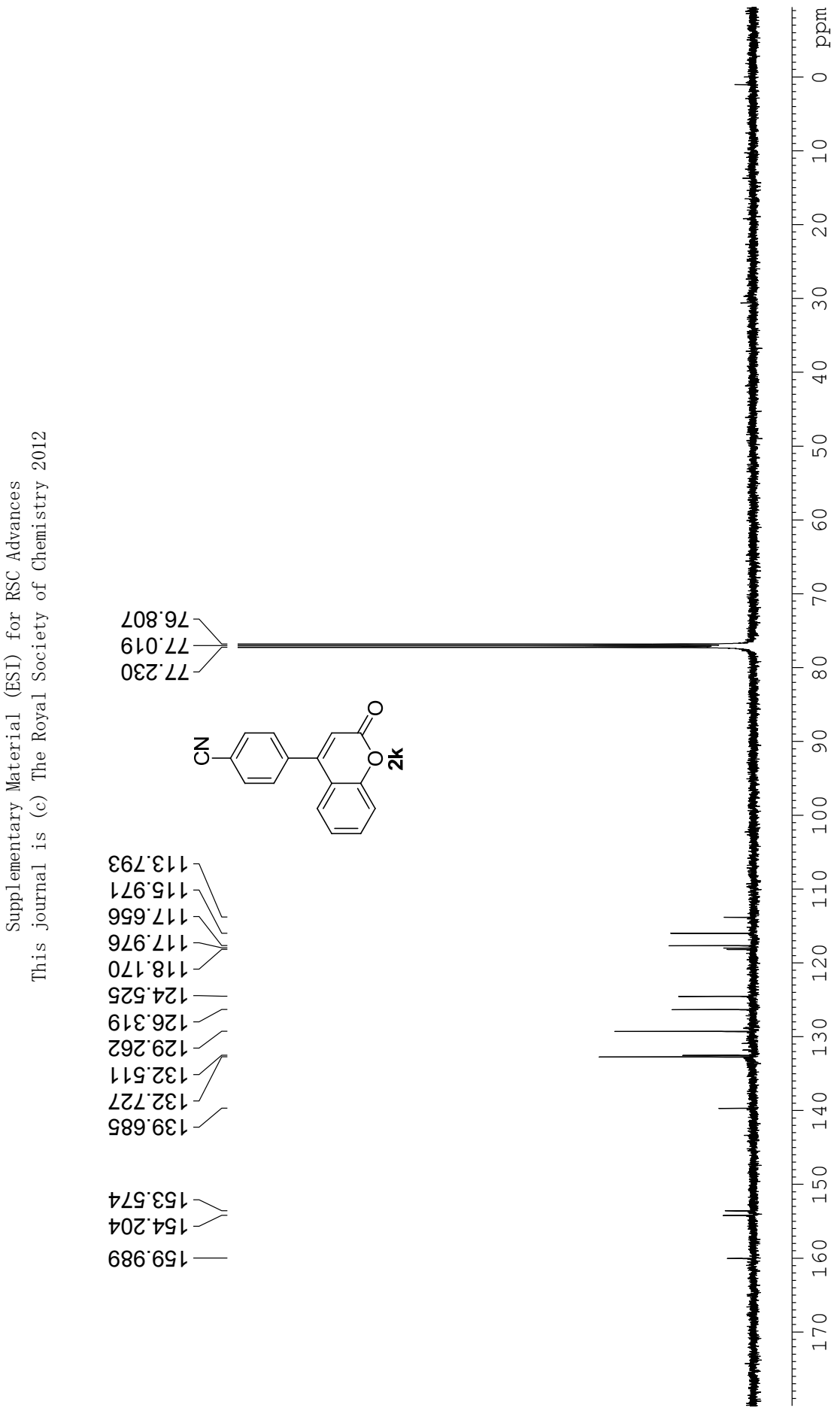
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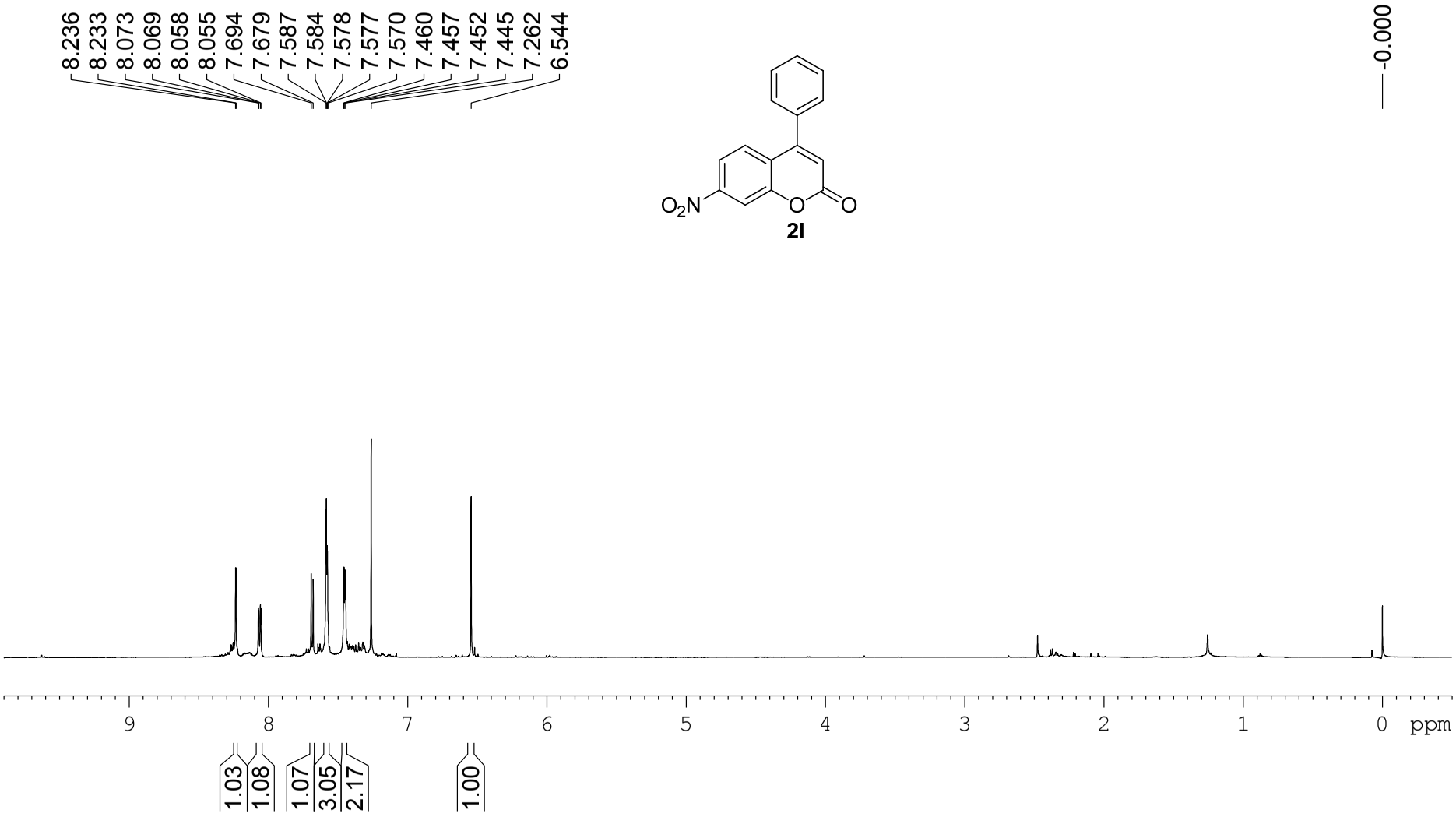

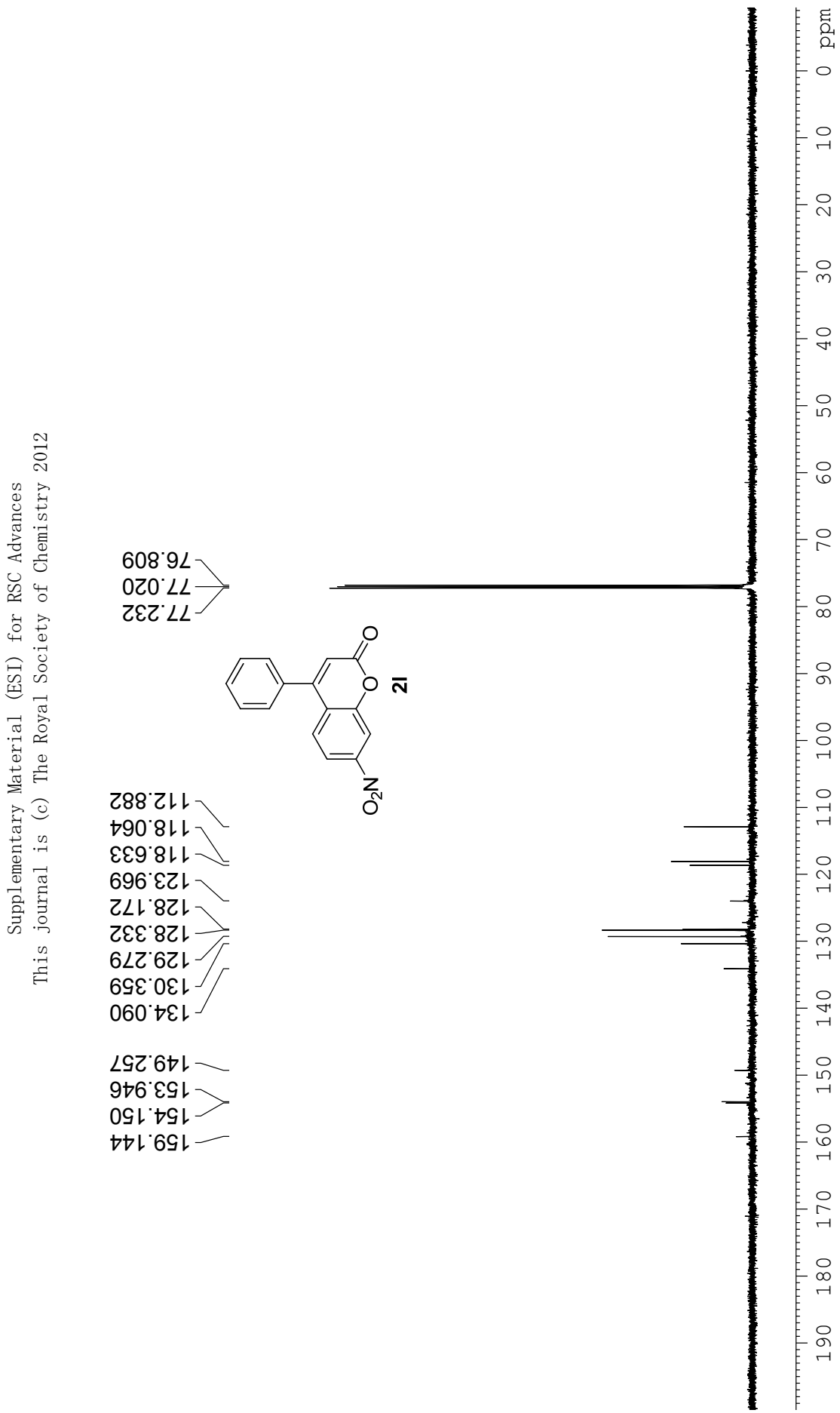
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123.969
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128.332
129.279
130.359
134.090
149.257
153.946
154.150
159.144
O
O
O
O2N
2l
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20
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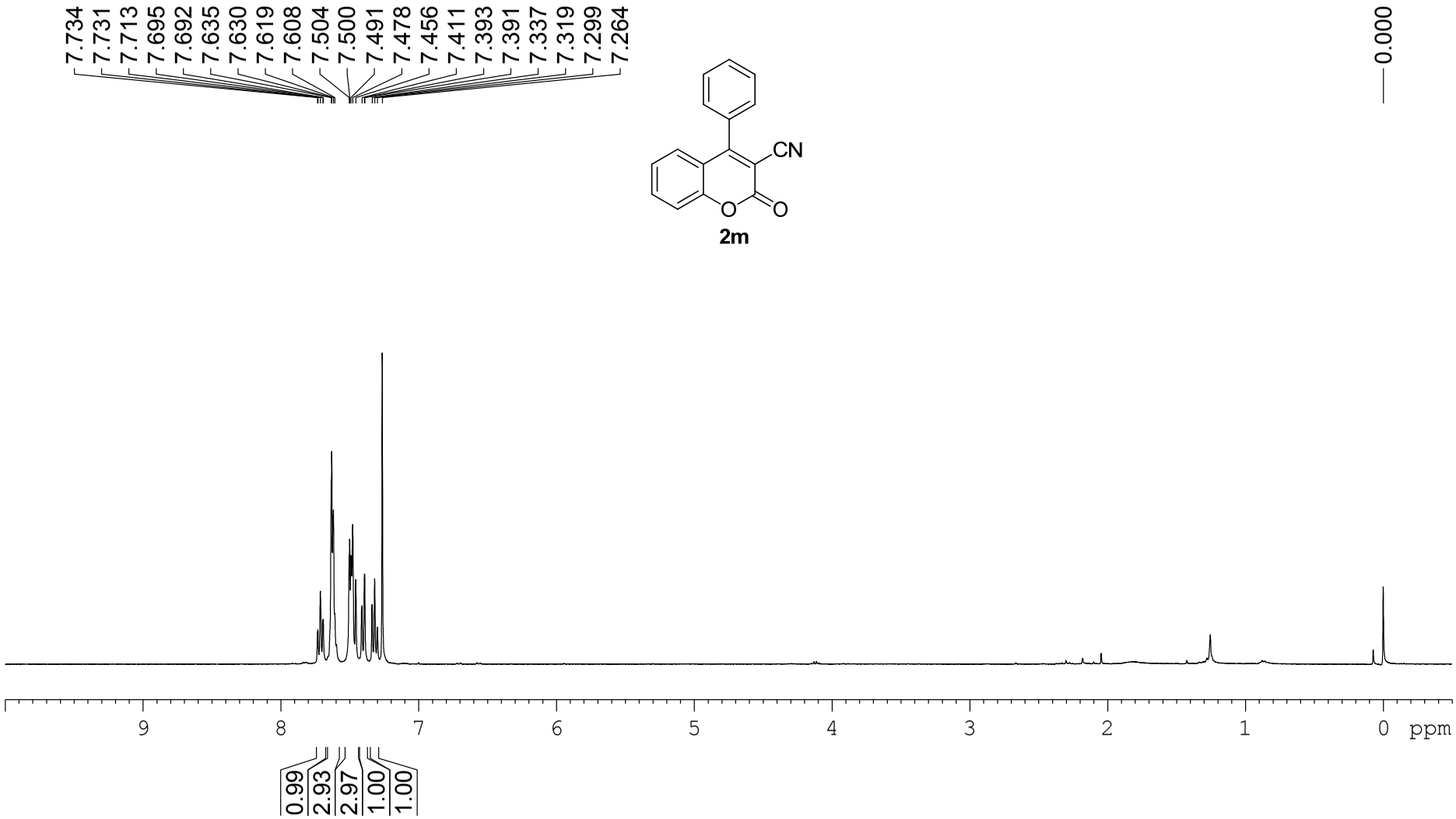

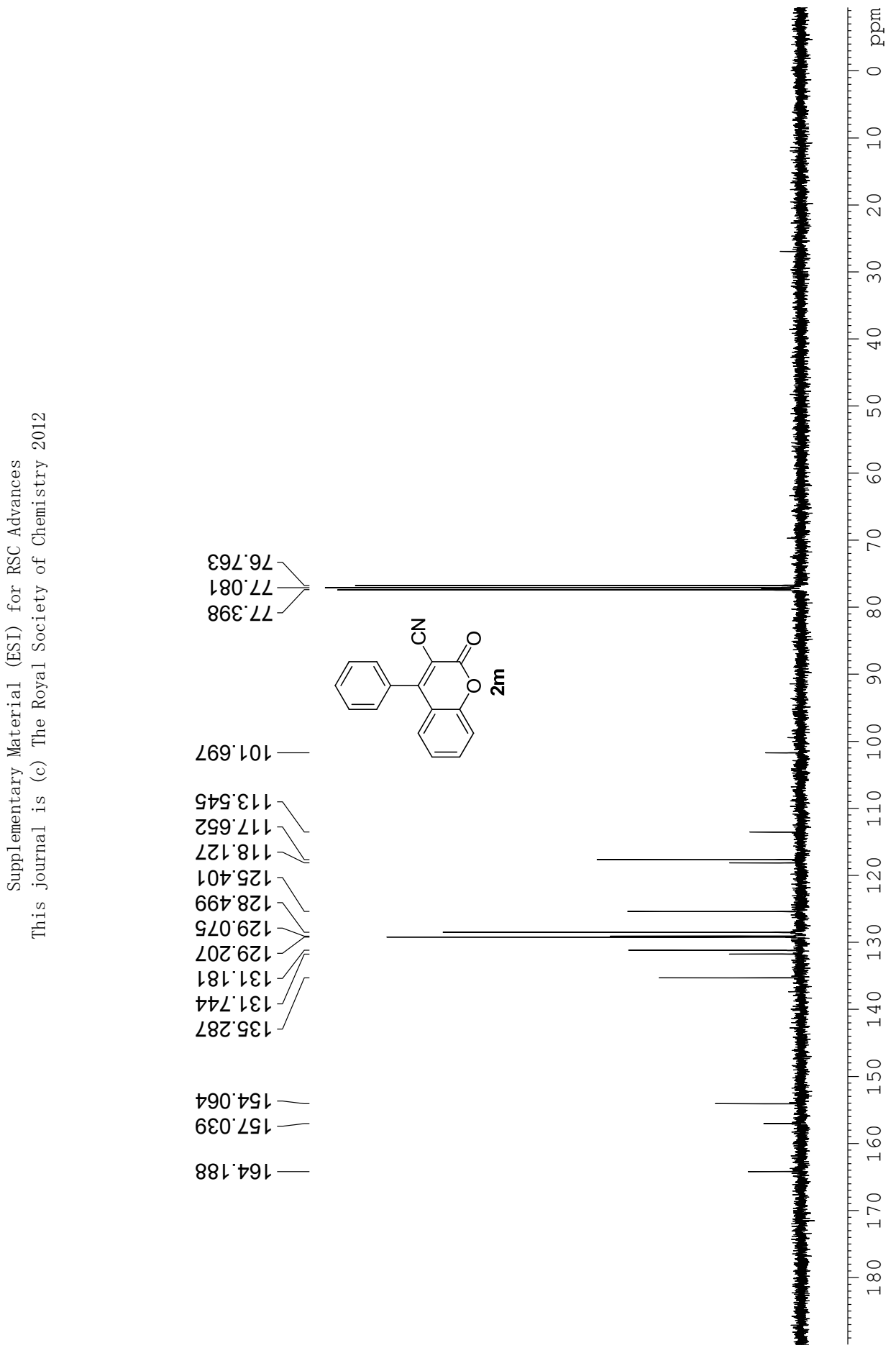
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129.207
131.181
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O
2m
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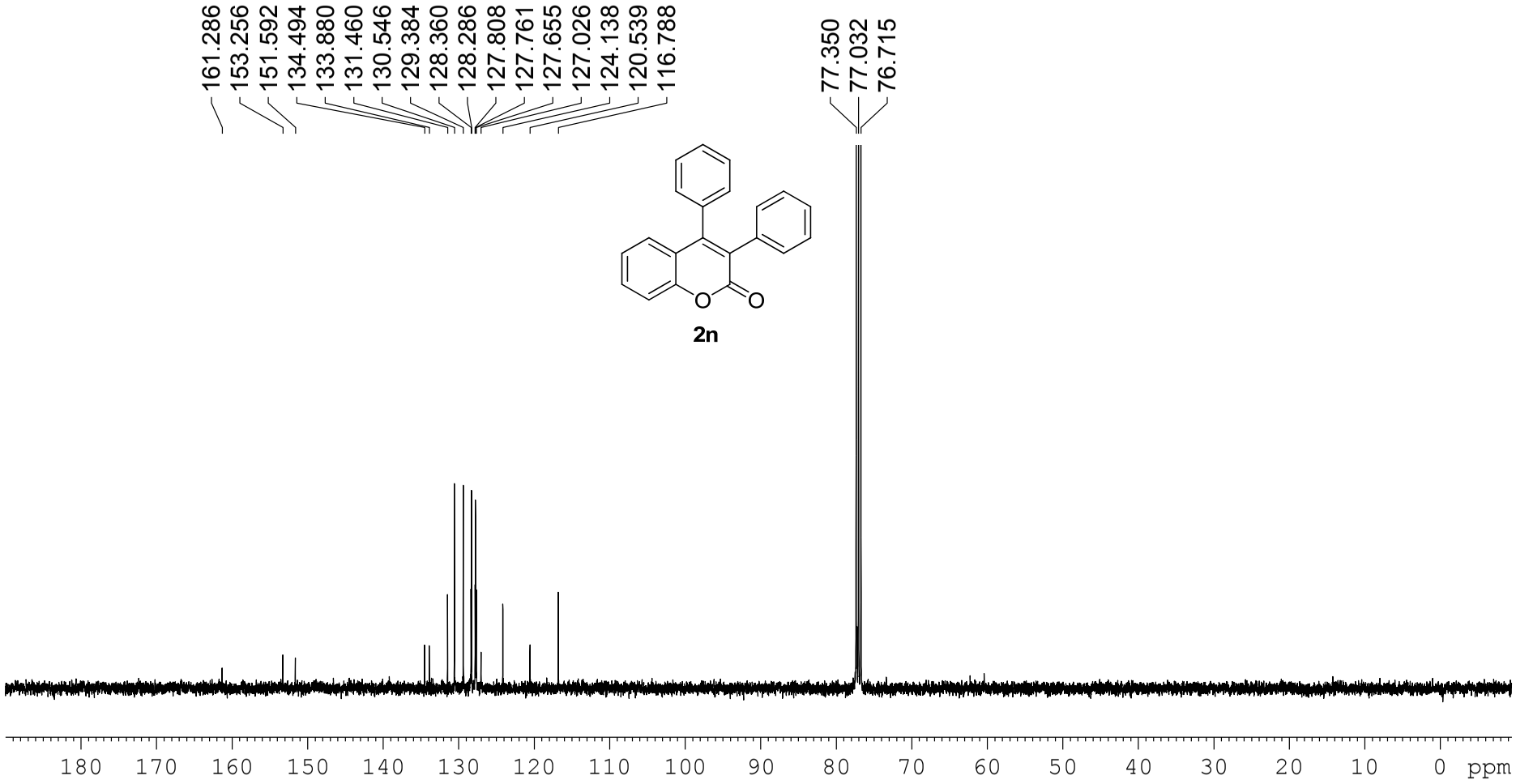

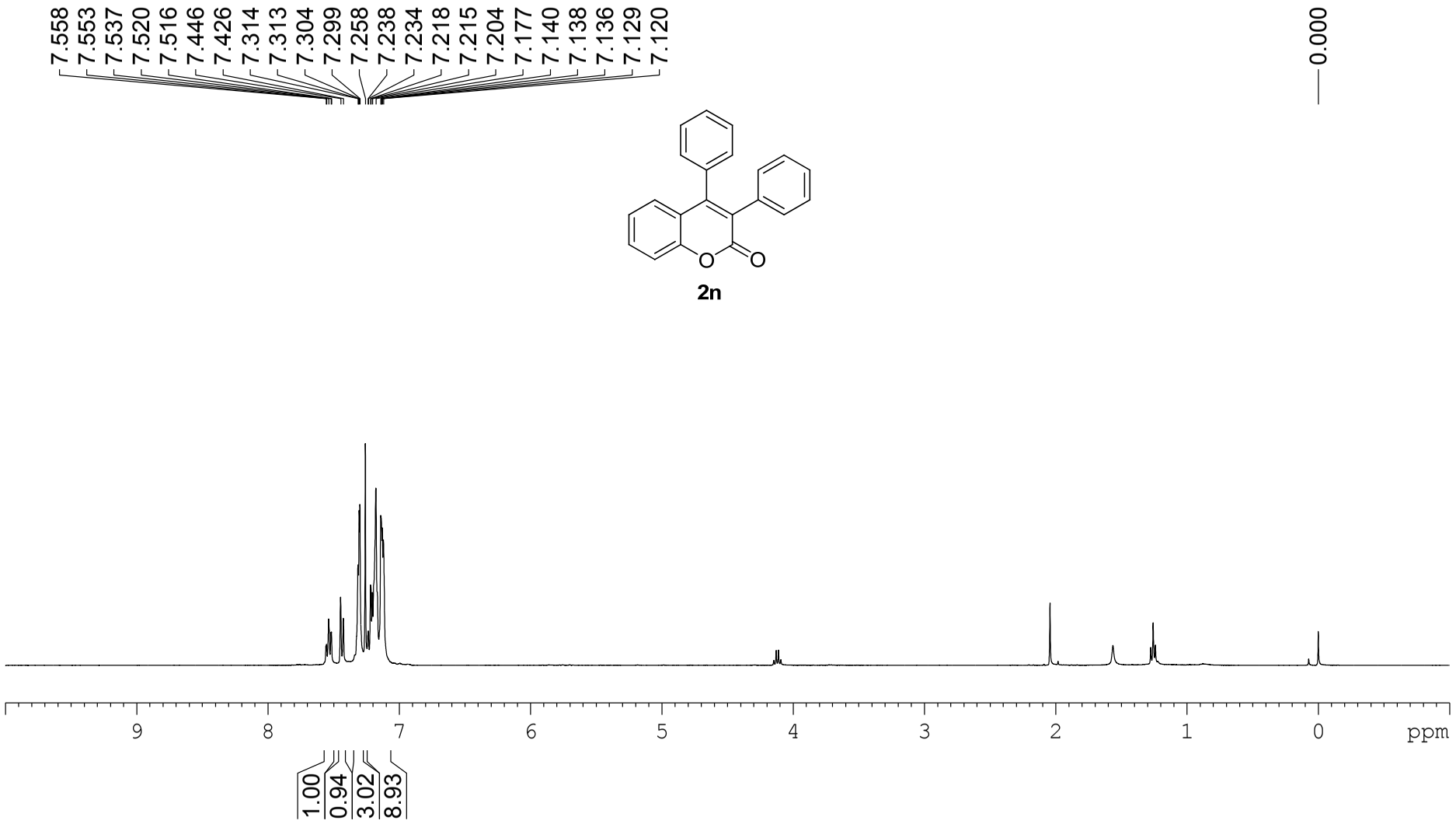

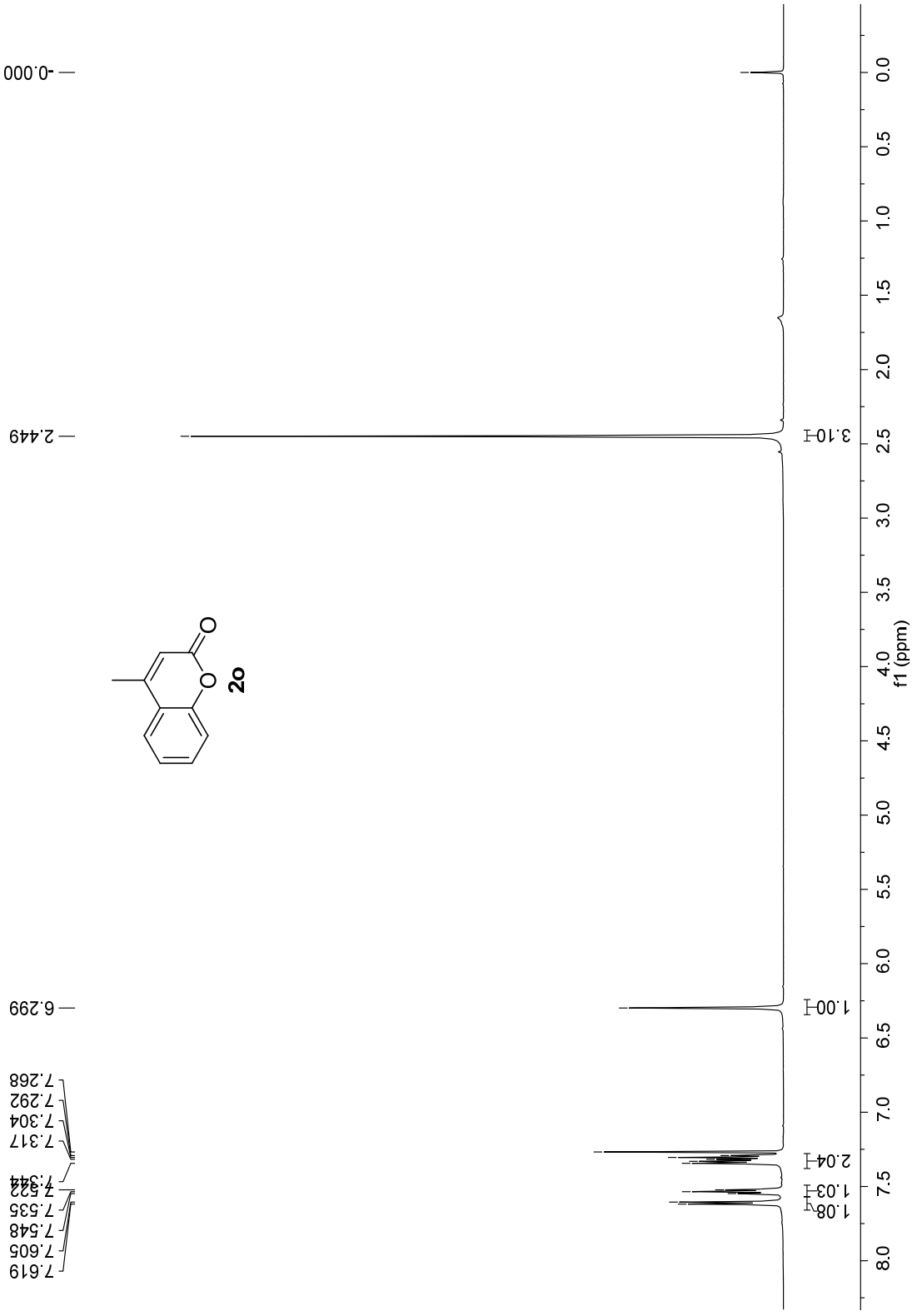

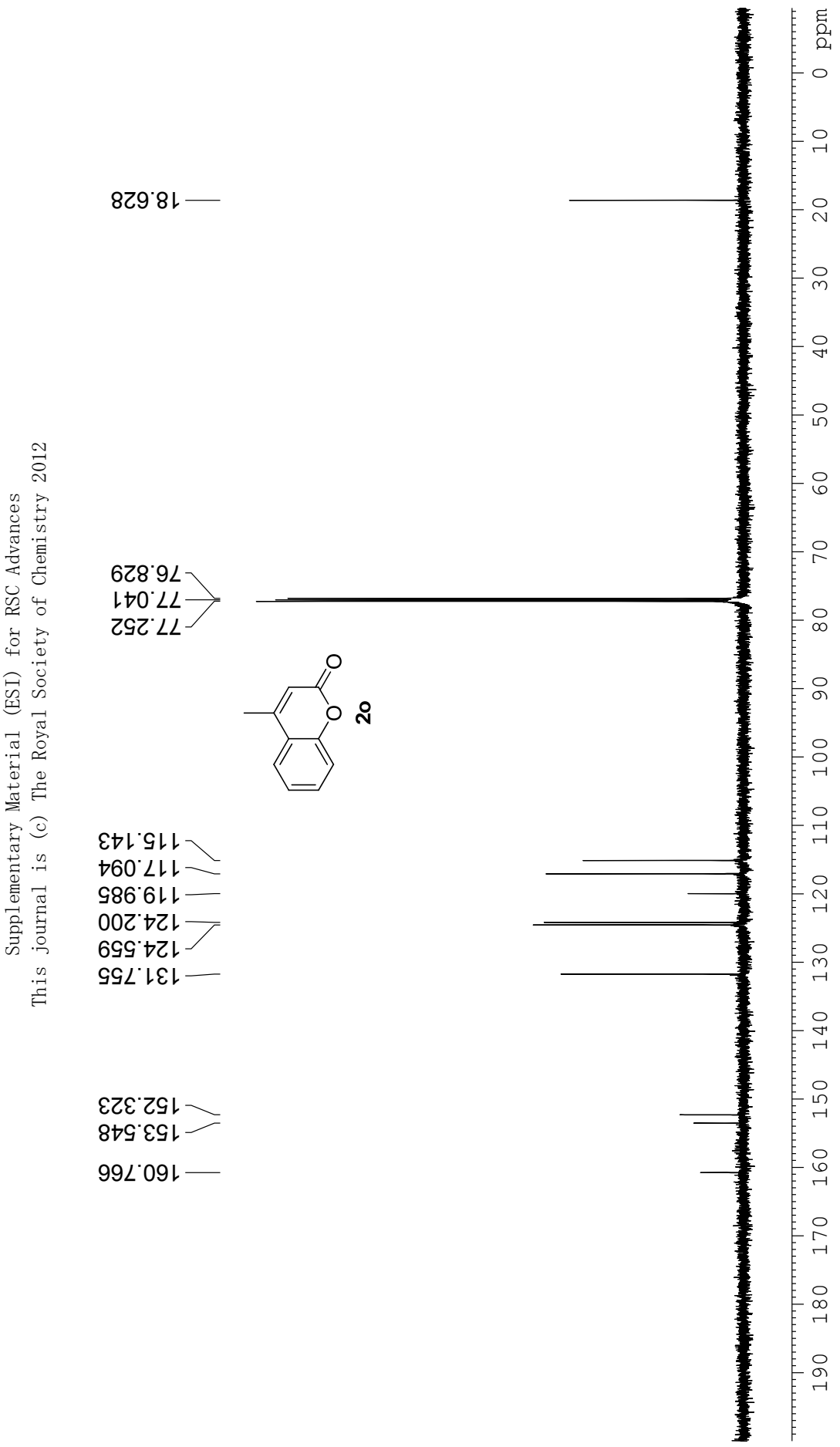
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115.143
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119.985
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153.548
160.766
2o
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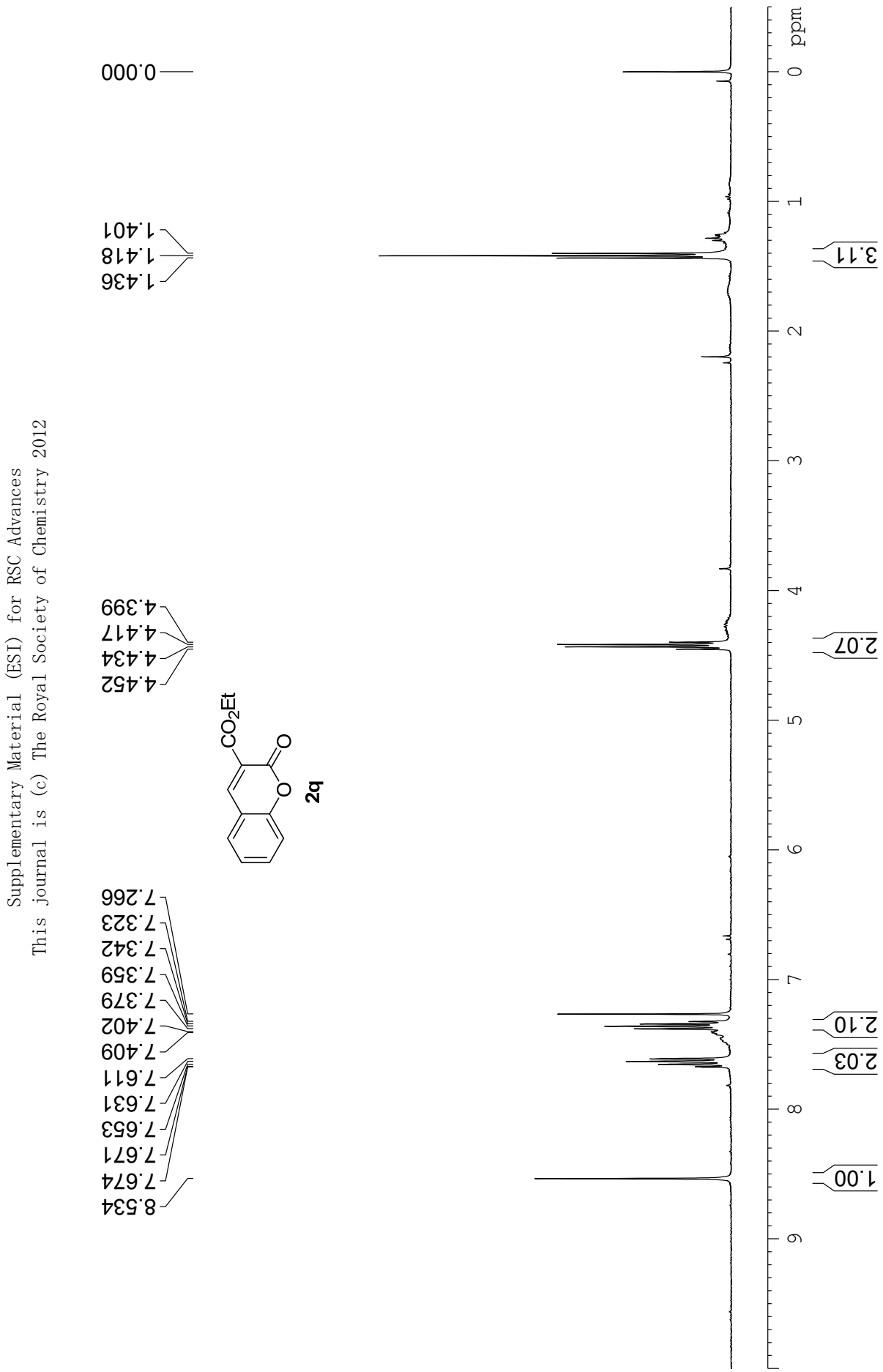
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7.266
7.323
7.342
7.359
7.379
7.402
7.409
7.611
7.631
7.653
7.671
7.674
8.534
CO2Et
O
O
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ppm
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3
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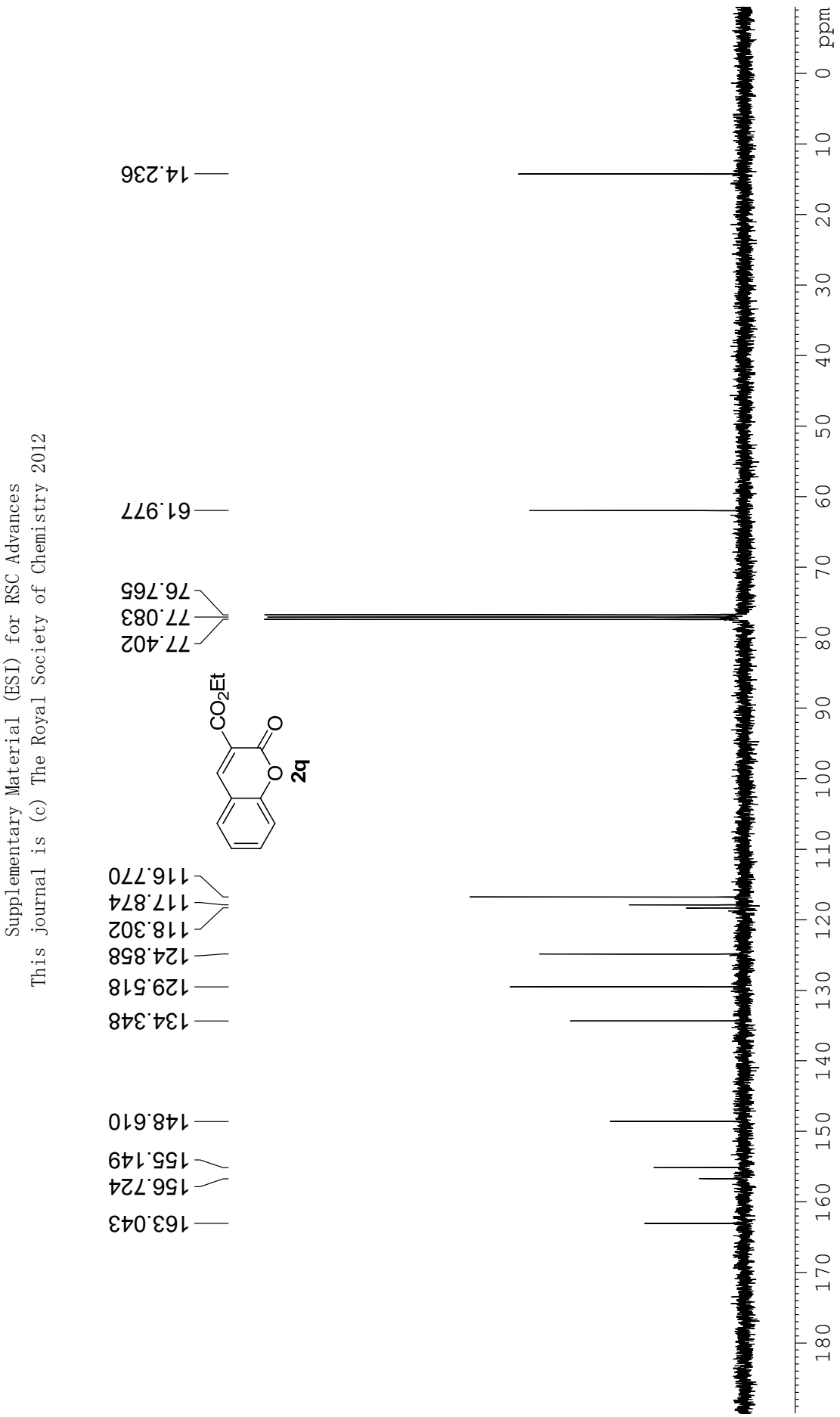
2.10
2.03
1.00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This journal is (c) The Royal Society of Chemistry 2012
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O
O
2q
116.770
117.874
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134.348
148.610
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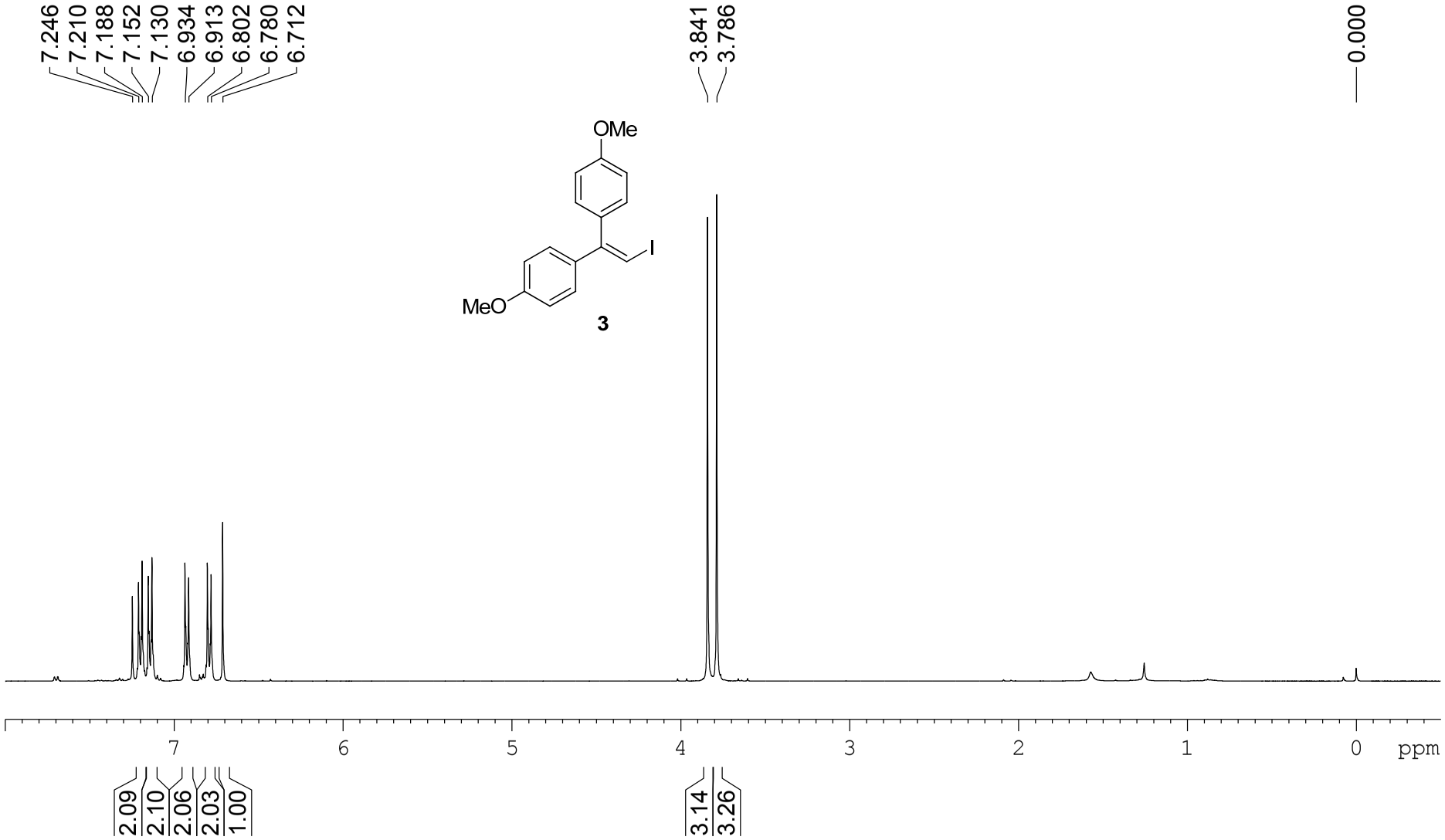

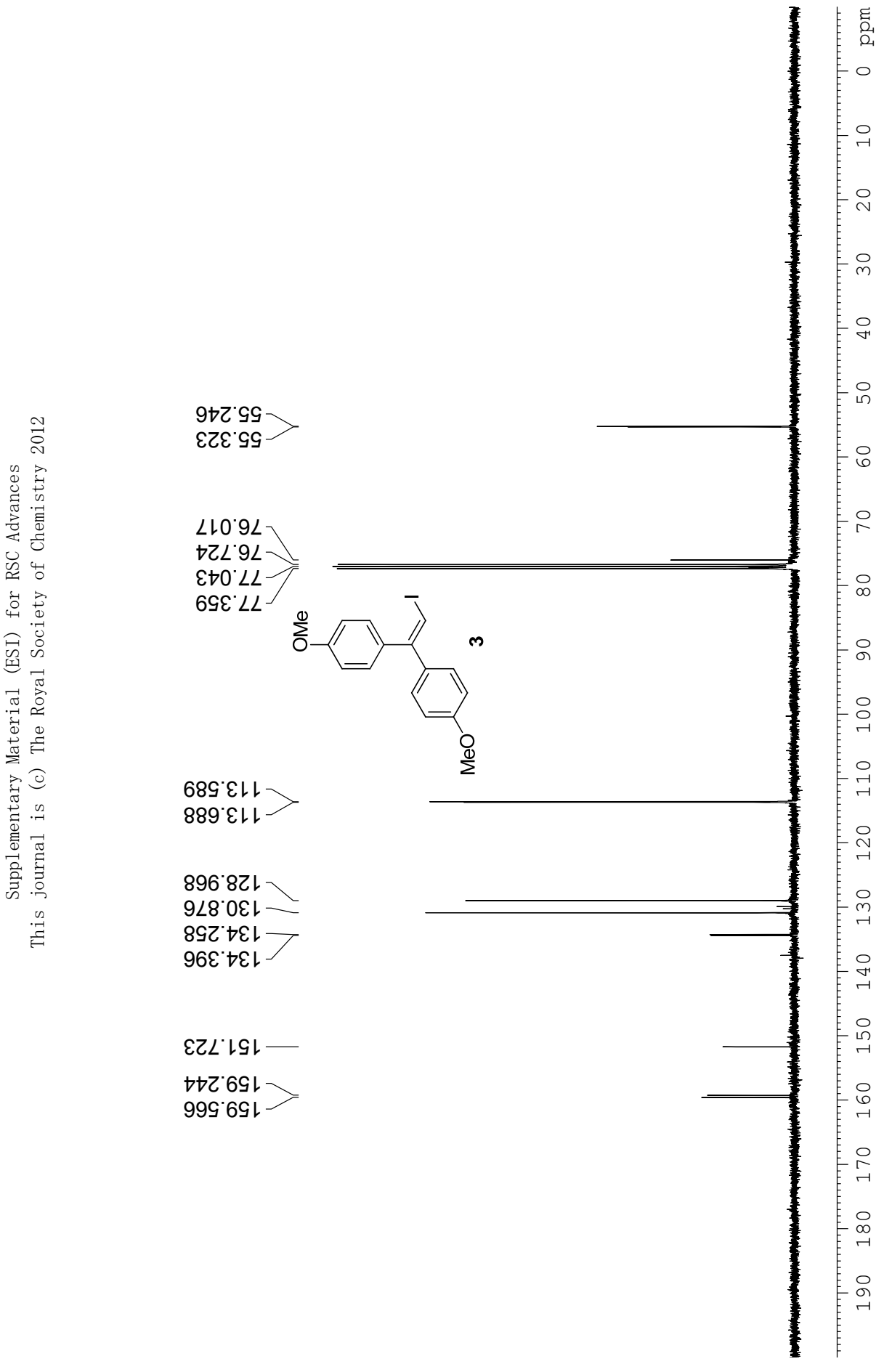
Supplementary Material (ESI) for RSC Advances
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134.258
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OMe
I
MeO
3
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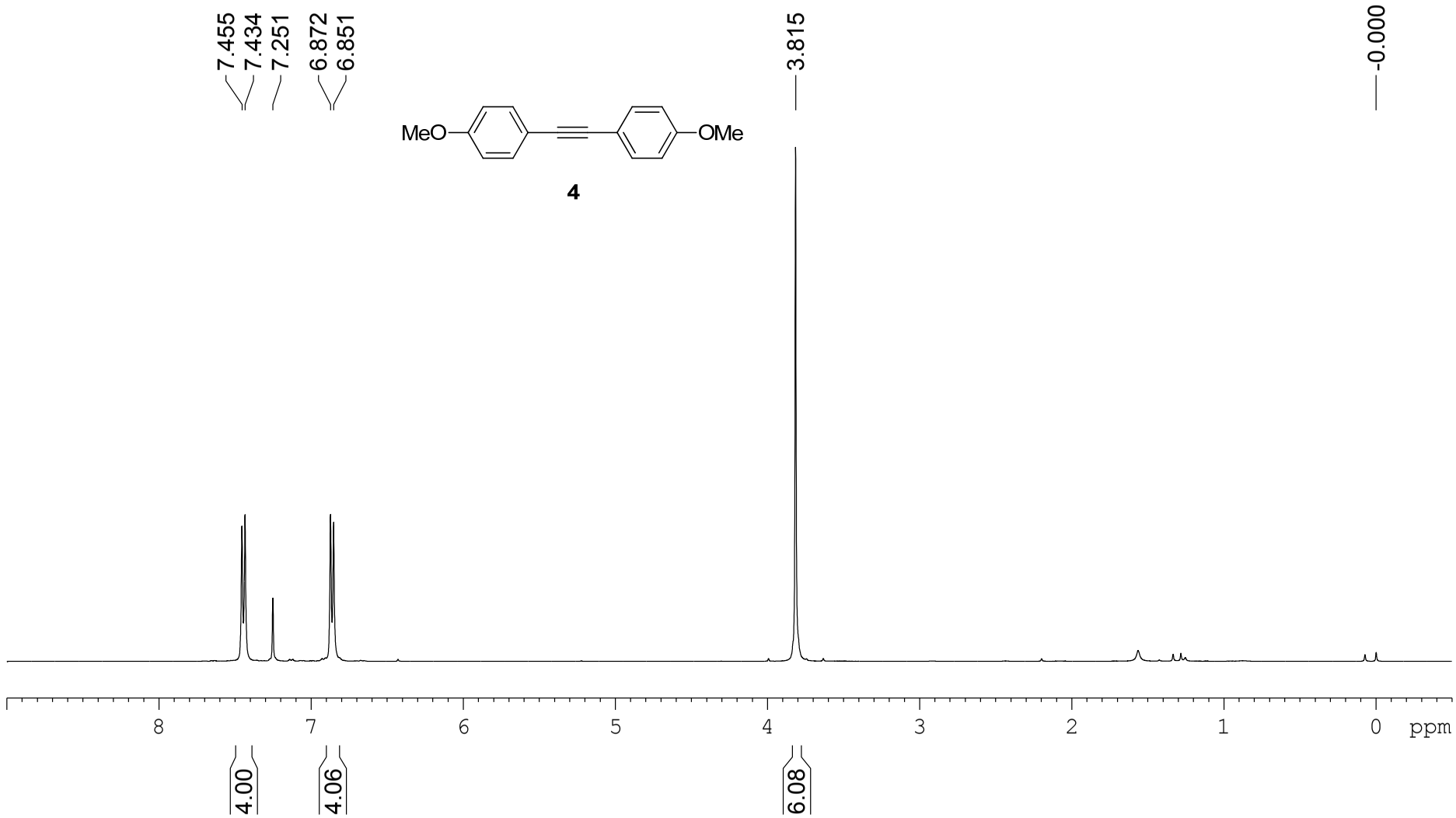

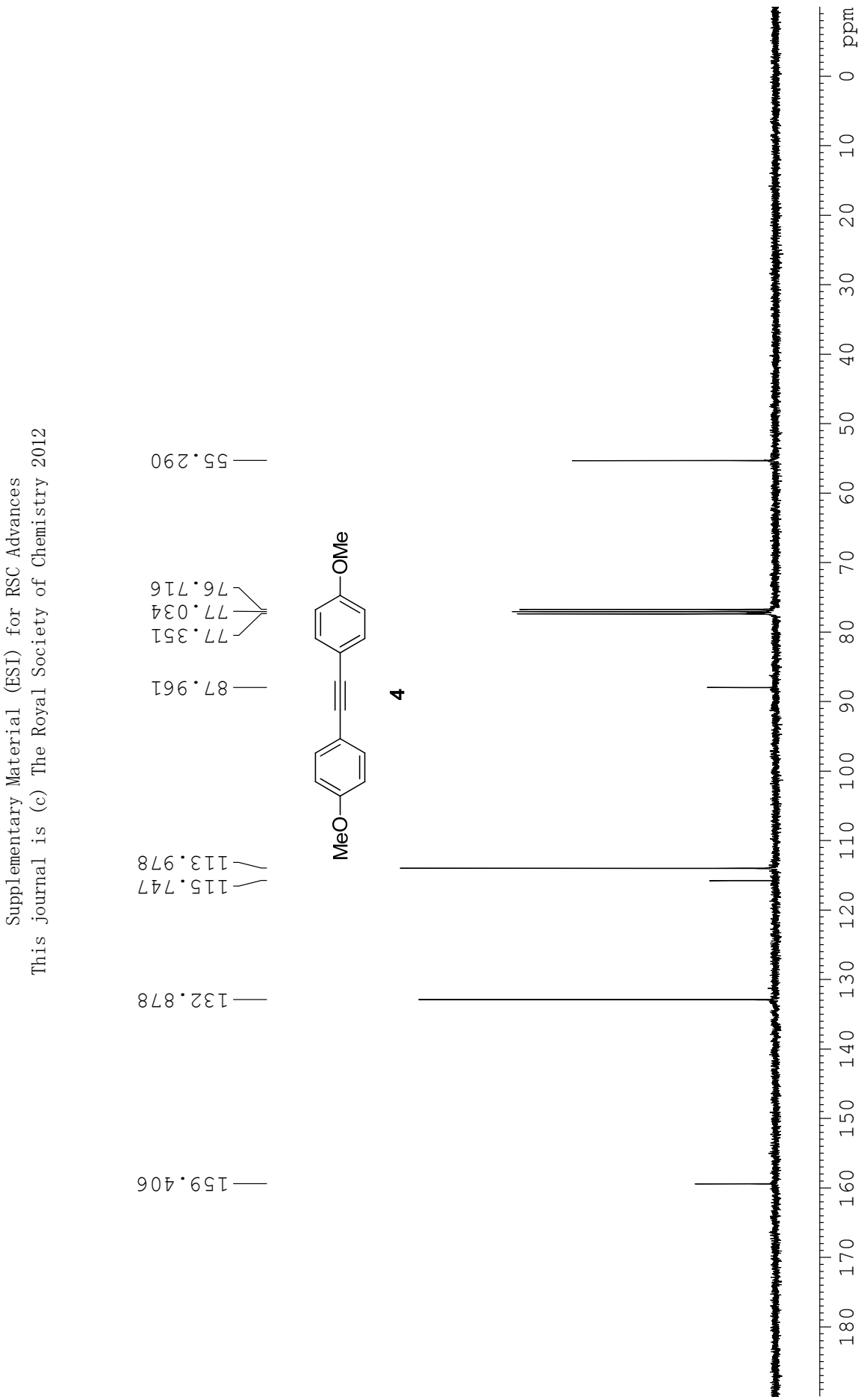
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This journal is (c) The Royal Society of Chemistry 2012
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V. X-ray structure and Data of 2f

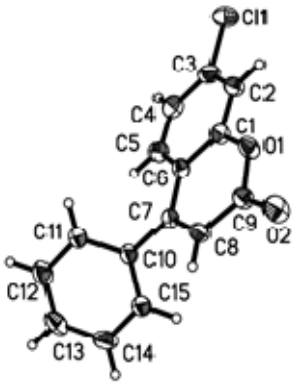

Figure 1. X-ray crystallography of courmarin **2f**.

Table 1. Crystal data and structure refinement for courmarin **2f**.

Identification code	2f
Empirical formula	$C_{15}H_9ClO_2$
Formula weight	256.67
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 9.6658(12) A alpha = 85.614(12) deg. b = 10.3292(14) A beta = 86.144(14) deg. c = 18.5201(18) A gamma = 81.723(10) deg.
Volume	1821.5(4) A^3
Z, Calculated density	6, 1.404 Mg/m^3
Absorption coefficient	0.303 mm^-1
F(000)	792
Crystal size	0.20 x 0.20 x 0.12 mm

Theta range for data collection	2.46 to 27.86 deg.
Limiting indices	-12<=h<=10, -13<=k<=13, -24<=l<=24
Reflections collected / unique	18731 / 8534 [R(int) = 0.0505]
Completeness to theta = 27.86	98.4 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9645 and 0.9418
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	8534 / 0 / 488
Goodness-of-fit on F^2	0.848
Final R indices [I>2sigma(I)]	R1 = 0.0435, wR2 = 0.0851
R indices (all data)	R1 = 0.1134, wR2 = 0.1095
Extinction coefficient	0.0205(11)
Largest diff. peak and hole	0.230 and -0.206 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **2f**.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Cl(1)	3365(1)	-1728(1)	2069(1)	67(1)
Cl(2)	6165(1)	12191(1)	10674(1)	70(1)
Cl(3)	292(1)	11923(1)	8538(1)	80(1)
O(1)	-415(1)	-1108(1)	4049(1)	49(1)
O(2)	-2236(2)	-1051(1)	4821(1)	67(1)
O(3)	5812(2)	7376(2)	10918(1)	59(1)
O(4)	5353(2)	5336(2)	11048(1)	87(1)
O(5)	4203(2)	11934(1)	6625(1)	58(1)
O(6)	5819(2)	12161(2)	5745(1)	72(1)
C(1)	465(2)	-485(2)	3570(1)	39(1)
C(2)	1363(2)	-1294(2)	3136(1)	46(1)
C(3)	2218(2)	-732(2)	2622(1)	45(1)
C(4)	2178(2)	619(2)	2536(1)	51(1)
C(5)	1292(2)	1402(2)	2984(1)	46(1)
C(6)	414(2)	871(2)	3525(1)	38(1)
C(7)	-589(2)	1627(2)	4005(1)	39(1)
C(8)	-1472(2)	988(2)	4446(1)	44(1)
C(9)	-1450(2)	-417(2)	4469(1)	47(1)
C(10)	-690(2)	3070(2)	4002(1)	41(1)
C(11)	476(2)	3696(2)	4066(1)	50(1)
C(12)	313(3)	5045(2)	4082(1)	60(1)
C(13)	-988(3)	5780(2)	4033(1)	60(1)
C(14)	-2145(2)	5167(2)	3973(1)	58(1)
C(15)	-1997(2)	3826(2)	3957(1)	50(1)
C(16)	6448(2)	8344(2)	10534(1)	47(1)
C(17)	6071(2)	9594(2)	10771(1)	51(1)
C(18)	6644(2)	10609(2)	10404(1)	51(1)
C(19)	7576(2)	10396(2)	9806(1)	61(1)
C(20)	7929(2)	9151(2)	9584(1)	58(1)
C(21)	7384(2)	8073(2)	9946(1)	45(1)
C(22)	7678(2)	6737(2)	9734(1)	49(1)
C(23)	6986(2)	5823(2)	10103(1)	57(1)
C(24)	6000(3)	6104(2)	10704(1)	61(1)
C(25)	8676(2)	6356(2)	9118(1)	51(1)
C(26)	10054(3)	6597(2)	9089(1)	63(1)
C(27)	10976(3)	6186(3)	8519(2)	74(1)
C(28)	10513(3)	5548(2)	7975(2)	78(1)

C(29)	9152(3)	5315(2)	7996(1)	76(1)
C(30)	8232(3)	5707(2)	8567(1)	62(1)
C(31)	3395(2)	11159(2)	7064(1)	48(1)
C(32)	2405(2)	11826(2)	7518(1)	56(1)
C(33)	1531(2)	11118(2)	7946(1)	54(1)
C(34)	1615(2)	9776(2)	7912(1)	57(1)
C(35)	2632(2)	9128(2)	7468(1)	49(1)
C(36)	3596(2)	9799(2)	7038(1)	43(1)
C(37)	4731(2)	9193(2)	6564(1)	43(1)
C(38)	5456(2)	9991(2)	6127(1)	51(1)
C(39)	5209(3)	11398(2)	6127(1)	55(1)
C(40)	5104(2)	7757(2)	6562(1)	44(1)
C(41)	5170(2)	6949(2)	7198(1)	52(1)
C(42)	5528(2)	5606(2)	7176(1)	62(1)
C(43)	5828(2)	5045(2)	6527(2)	66(1)
C(44)	5803(2)	5823(3)	5897(1)	63(1)
C(45)	5447(2)	7169(2)	5908(1)	52(1)

Table 3. Bond lengths [A] and angles [deg] for **2f**.

Cl(1)-C(3)	1.736(2)
Cl(2)-C(18)	1.735(2)
Cl(3)-C(33)	1.733(2)
O(1)-C(9)	1.375(2)
O(1)-C(1)	1.377(2)
O(2)-C(9)	1.201(2)
O(3)-C(16)	1.376(2)
O(3)-C(24)	1.385(3)
O(4)-C(24)	1.199(2)
O(5)-C(31)	1.381(2)
O(5)-C(39)	1.381(3)
O(6)-C(39)	1.208(2)
C(1)-C(2)	1.377(3)
C(1)-C(6)	1.390(2)
C(2)-C(3)	1.369(3)
C(2)-H(2)	0.9300
C(3)-C(4)	1.388(3)
C(4)-C(5)	1.372(3)
C(4)-H(4)	0.9300
C(5)-C(6)	1.401(2)
C(5)-H(5)	0.9300
C(6)-C(7)	1.454(3)
C(7)-C(8)	1.348(2)
C(7)-C(10)	1.479(3)
C(8)-C(9)	1.446(3)
C(8)-H(8)	0.9300
C(10)-C(15)	1.391(3)
C(10)-C(11)	1.393(3)
C(11)-C(12)	1.382(3)
C(11)-H(11)	0.9300
C(12)-C(13)	1.376(3)
C(12)-H(12)	0.9300
C(13)-C(14)	1.376(3)
C(13)-H(13)	0.9300
C(14)-C(15)	1.374(3)
C(14)-H(14)	0.9300
C(15)-H(15)	0.9300
C(16)-C(17)	1.387(3)
C(16)-C(21)	1.387(3)
C(17)-C(18)	1.370(3)
C(17)-H(17)	0.9300
C(18)-C(19)	1.390(3)

C(19)-C(20)	1.369(3)
C(19)-H(19)	0.9300
C(20)-C(21)	1.404(3)
C(20)-H(20)	0.9300
C(21)-C(22)	1.447(3)
C(22)-C(23)	1.352(3)
C(22)-C(25)	1.482(3)
C(23)-C(24)	1.435(3)
C(23)-H(23)	0.9300
C(25)-C(30)	1.386(3)
C(25)-C(26)	1.387(3)
C(26)-C(27)	1.386(3)
C(26)-H(26)	0.9300
C(27)-C(28)	1.377(3)
C(27)-H(27)	0.9300
C(28)-C(29)	1.368(3)
C(28)-H(28)	0.9300
C(29)-C(30)	1.382(3)
C(29)-H(29)	0.9300
C(30)-H(30)	0.9300
C(31)-C(32)	1.373(3)
C(31)-C(36)	1.395(3)
C(32)-C(33)	1.370(3)
C(32)-H(32)	0.9300
C(33)-C(34)	1.383(3)
C(34)-C(35)	1.370(3)
C(34)-H(34)	0.9300
C(35)-C(36)	1.408(3)
C(35)-H(35)	0.9300
C(36)-C(37)	1.457(3)
C(37)-C(38)	1.347(3)
C(37)-C(40)	1.475(3)
C(38)-C(39)	1.438(3)
C(38)-H(38)	0.9300
C(40)-C(41)	1.392(3)
C(40)-C(45)	1.395(3)
C(41)-C(42)	1.383(3)
C(41)-H(41)	0.9300
C(42)-C(43)	1.370(3)
C(42)-H(42)	0.9300
C(43)-C(44)	1.366(3)
C(43)-H(43)	0.9300
C(44)-C(45)	1.384(3)
C(44)-H(44)	0.9300

C(45)-H(45)	0.9300
C(9)-O(1)-C(1)	121.72(16)
C(16)-O(3)-C(24)	121.43(18)
C(31)-O(5)-C(39)	121.41(18)
O(1)-C(1)-C(2)	115.32(17)
O(1)-C(1)-C(6)	121.43(17)
C(2)-C(1)-C(6)	123.24(18)
C(3)-C(2)-C(1)	118.31(19)
C(3)-C(2)-H(2)	120.8
C(1)-C(2)-H(2)	120.8
C(2)-C(3)-C(4)	120.96(19)
C(2)-C(3)-Cl(1)	119.34(16)
C(4)-C(3)-Cl(1)	119.70(16)
C(5)-C(4)-C(3)	119.54(19)
C(5)-C(4)-H(4)	120.2
C(3)-C(4)-H(4)	120.2
C(4)-C(5)-C(6)	121.57(19)
C(4)-C(5)-H(5)	119.2
C(6)-C(5)-H(5)	119.2
C(1)-C(6)-C(5)	116.32(18)
C(1)-C(6)-C(7)	118.36(16)
C(5)-C(6)-C(7)	125.21(18)
C(8)-C(7)-C(6)	118.45(18)
C(8)-C(7)-C(10)	119.82(18)
C(6)-C(7)-C(10)	121.69(16)
C(7)-C(8)-C(9)	122.84(19)
C(7)-C(8)-H(8)	118.6
C(9)-C(8)-H(8)	118.6
O(2)-C(9)-O(1)	116.23(19)
O(2)-C(9)-C(8)	126.9(2)
O(1)-C(9)-C(8)	116.85(17)
C(15)-C(10)-C(11)	118.77(19)
C(15)-C(10)-C(7)	118.98(18)
C(11)-C(10)-C(7)	122.21(18)
C(12)-C(11)-C(10)	119.7(2)
C(12)-C(11)-H(11)	120.2
C(10)-C(11)-H(11)	120.2
C(13)-C(12)-C(11)	120.8(2)
C(13)-C(12)-H(12)	119.6
C(11)-C(12)-H(12)	119.6
C(12)-C(13)-C(14)	119.8(2)
C(12)-C(13)-H(13)	120.1
C(14)-C(13)-H(13)	120.1
C(15)-C(14)-C(13)	119.9(2)

C(15)-C(14)-H(14)	120.0
C(13)-C(14)-H(14)	120.0
C(14)-C(15)-C(10)	121.0(2)
C(14)-C(15)-H(15)	119.5
C(10)-C(15)-H(15)	119.5
O(3)-C(16)-C(17)	115.29(19)
O(3)-C(16)-C(21)	121.6(2)
C(17)-C(16)-C(21)	123.1(2)
C(18)-C(17)-C(16)	118.4(2)
C(18)-C(17)-H(17)	120.8
C(16)-C(17)-H(17)	120.8
C(17)-C(18)-C(19)	120.9(2)
C(17)-C(18)-Cl(2)	119.58(18)
C(19)-C(18)-Cl(2)	119.47(18)
C(20)-C(19)-C(18)	119.4(2)
C(20)-C(19)-H(19)	120.3
C(18)-C(19)-H(19)	120.3
C(19)-C(20)-C(21)	122.0(2)
C(19)-C(20)-H(20)	119.0
C(21)-C(20)-H(20)	119.0
C(16)-C(21)-C(20)	116.2(2)
C(16)-C(21)-C(22)	118.53(19)
C(20)-C(21)-C(22)	125.2(2)
C(23)-C(22)-C(21)	118.2(2)
C(23)-C(22)-C(25)	119.5(2)
C(21)-C(22)-C(25)	122.21(19)
C(22)-C(23)-C(24)	123.4(2)
C(22)-C(23)-H(23)	118.3
C(24)-C(23)-H(23)	118.3
O(4)-C(24)-O(3)	117.2(2)
O(4)-C(24)-C(23)	126.2(2)
O(3)-C(24)-C(23)	116.6(2)
C(30)-C(25)-C(26)	119.1(2)
C(30)-C(25)-C(22)	119.1(2)
C(26)-C(25)-C(22)	121.8(2)
C(27)-C(26)-C(25)	120.4(3)
C(27)-C(26)-H(26)	119.8
C(25)-C(26)-H(26)	119.8
C(28)-C(27)-C(26)	119.7(3)
C(28)-C(27)-H(27)	120.2
C(26)-C(27)-H(27)	120.2
C(29)-C(28)-C(27)	120.4(3)
C(29)-C(28)-H(28)	119.8
C(27)-C(28)-H(28)	119.8

C(28)-C(29)-C(30)	120.3(3)
C(28)-C(29)-H(29)	119.8
C(30)-C(29)-H(29)	119.8
C(29)-C(30)-C(25)	120.2(2)
C(29)-C(30)-H(30)	119.9
C(25)-C(30)-H(30)	119.9
C(32)-C(31)-O(5)	115.2(2)
C(32)-C(31)-C(36)	123.6(2)
O(5)-C(31)-C(36)	121.2(2)
C(33)-C(32)-C(31)	118.0(2)
C(33)-C(32)-H(32)	121.0
C(31)-C(32)-H(32)	121.0
C(32)-C(33)-C(34)	121.3(2)
C(32)-C(33)-Cl(3)	119.3(2)
C(34)-C(33)-Cl(3)	119.45(18)
C(35)-C(34)-C(33)	119.6(2)
C(35)-C(34)-H(34)	120.2
C(33)-C(34)-H(34)	120.2
C(34)-C(35)-C(36)	121.5(2)
C(34)-C(35)-H(35)	119.2
C(36)-C(35)-H(35)	119.2
C(31)-C(36)-C(35)	115.77(19)
C(31)-C(36)-C(37)	118.74(18)
C(35)-C(36)-C(37)	125.5(2)
C(38)-C(37)-C(36)	117.7(2)
C(38)-C(37)-C(40)	120.2(2)
C(36)-C(37)-C(40)	122.02(18)
C(37)-C(38)-C(39)	123.6(2)
C(37)-C(38)-H(38)	118.2
C(39)-C(38)-H(38)	118.2
O(6)-C(39)-O(5)	116.5(2)
O(6)-C(39)-C(38)	126.5(2)
O(5)-C(39)-C(38)	117.0(2)
C(41)-C(40)-C(45)	117.6(2)
C(41)-C(40)-C(37)	122.2(2)
C(45)-C(40)-C(37)	120.14(19)
C(42)-C(41)-C(40)	120.7(2)
C(42)-C(41)-H(41)	119.7
C(40)-C(41)-H(41)	119.7
C(43)-C(42)-C(41)	120.7(2)
C(43)-C(42)-H(42)	119.6
C(41)-C(42)-H(42)	119.6
C(44)-C(43)-C(42)	119.5(2)
C(44)-C(43)-H(43)	120.2

C(42)-C(43)-H(43)	120.2
C(43)-C(44)-C(45)	120.6(2)
C(43)-C(44)-H(44)	119.7
C(45)-C(44)-H(44)	119.7
C(44)-C(45)-C(40)	120.8(2)
C(44)-C(45)-H(45)	119.6
C(40)-C(45)-H(45)	119.6

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for **2f**.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
Cl(1)	61(1)	72(1)	62(1)	-13(1)	13(1)	7(1)
Cl(2)	87(1)	54(1)	71(1)	-11(1)	-9(1)	-6(1)
Cl(3)	67(1)	93(1)	75(1)	-20(1)	9(1)	7(1)
O(1)	57(1)	37(1)	52(1)	-4(1)	13(1)	-10(1)
O(2)	72(1)	50(1)	77(1)	1(1)	28(1)	-21(1)
O(3)	67(1)	57(1)	53(1)	-1(1)	17(1)	-15(1)
O(4)	107(2)	66(1)	85(1)	1(1)	37(1)	-31(1)
O(5)	67(1)	48(1)	56(1)	3(1)	2(1)	-7(1)
O(6)	92(1)	58(1)	65(1)	12(1)	7(1)	-25(1)
C(1)	41(1)	38(1)	39(1)	-1(1)	3(1)	-7(1)
C(2)	46(1)	37(1)	52(1)	-4(1)	2(1)	-1(1)
C(3)	38(1)	53(1)	44(1)	-8(1)	1(1)	0(1)
C(4)	48(2)	59(2)	46(1)	3(1)	5(1)	-13(1)
C(5)	44(1)	43(1)	51(1)	-1(1)	0(1)	-9(1)
C(6)	40(1)	35(1)	40(1)	-2(1)	-2(1)	-9(1)
C(7)	39(1)	35(1)	43(1)	-5(1)	-5(1)	-7(1)
C(8)	45(1)	36(1)	49(1)	-8(1)	5(1)	-4(1)
C(9)	49(2)	41(1)	49(1)	-4(1)	5(1)	-6(1)
C(10)	42(1)	37(1)	45(1)	-5(1)	-1(1)	-8(1)
C(11)	49(1)	40(1)	61(2)	-6(1)	-2(1)	-10(1)
C(12)	67(2)	43(1)	74(2)	-8(1)	-1(1)	-23(1)
C(13)	81(2)	34(1)	67(2)	-8(1)	3(1)	-9(1)
C(14)	56(2)	40(1)	75(2)	-3(1)	5(1)	3(1)
C(15)	45(1)	40(1)	66(2)	-7(1)	4(1)	-8(1)
C(16)	50(1)	52(1)	41(1)	2(1)	-3(1)	-10(1)
C(17)	48(1)	60(2)	44(1)	-6(1)	2(1)	-6(1)
C(18)	58(2)	49(1)	47(1)	-3(1)	-11(1)	-6(1)
C(19)	71(2)	59(2)	53(2)	2(1)	1(1)	-19(1)
C(20)	63(2)	59(2)	51(2)	-2(1)	9(1)	-13(1)
C(21)	43(1)	56(1)	37(1)	0(1)	-1(1)	-7(1)
C(22)	48(1)	55(2)	42(1)	-3(1)	-4(1)	-5(1)
C(23)	65(2)	51(2)	53(2)	-5(1)	5(1)	-6(1)
C(24)	70(2)	54(2)	58(2)	0(1)	11(1)	-12(1)
C(25)	54(2)	51(1)	46(1)	1(1)	5(1)	-3(1)
C(26)	59(2)	69(2)	62(2)	-10(1)	6(1)	-6(1)
C(27)	62(2)	72(2)	80(2)	4(2)	22(2)	-2(1)
C(28)	104(3)	54(2)	64(2)	1(1)	29(2)	7(2)
C(29)	106(2)	62(2)	57(2)	-14(1)	10(2)	-7(2)
C(30)	72(2)	58(2)	54(2)	-7(1)	2(1)	-9(1)

C(31)	48(2)	51(1)	46(1)	4(1)	-6(1)	-8(1)
C(32)	63(2)	51(1)	53(2)	-3(1)	-10(1)	1(1)
C(33)	49(2)	65(2)	47(1)	-8(1)	-6(1)	3(1)
C(34)	48(2)	69(2)	53(2)	-1(1)	-1(1)	-11(1)
C(35)	49(2)	50(1)	50(1)	-2(1)	-5(1)	-10(1)
C(36)	45(1)	45(1)	40(1)	0(1)	-9(1)	-4(1)
C(37)	43(1)	50(1)	39(1)	1(1)	-9(1)	-8(1)
C(38)	53(2)	55(2)	45(1)	1(1)	0(1)	-6(1)
C(39)	60(2)	54(2)	51(2)	4(1)	-9(1)	-9(1)
C(40)	40(1)	47(1)	45(1)	-2(1)	-3(1)	-9(1)
C(41)	57(2)	49(1)	50(2)	-4(1)	-3(1)	-6(1)
C(42)	62(2)	50(2)	71(2)	6(1)	-4(1)	-9(1)
C(43)	58(2)	51(2)	91(2)	-13(2)	0(2)	-6(1)
C(44)	53(2)	69(2)	70(2)	-26(2)	7(1)	-12(1)
C(45)	44(1)	65(2)	49(2)	-6(1)	3(1)	-11(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for **2f**.

	x	y	z	U(eq)
H(2)	1387	-2200	3191	55
H(4)	2747	992	2177	61
H(5)	1274	2306	2926	55
H(8)	-2124	1472	4749	52
H(11)	1359	3209	4099	59
H(12)	1091	5462	4125	72
H(13)	-1085	6688	4040	73
H(14)	-3026	5660	3943	70
H(15)	-2783	3419	3916	60
H(17)	5443	9739	11169	61
H(19)	7956	11092	9559	73
H(20)	8548	9015	9182	69
H(23)	7158	4968	9960	69
H(26)	10362	7037	9454	76
H(27)	11902	6341	8504	88
H(28)	11129	5273	7592	93
H(29)	8845	4892	7624	91
H(30)	7312	5534	8582	74
H(32)	2330	12731	7534	67
H(34)	984	9315	8189	68
H(35)	2687	8225	7449	59
H(38)	6155	9612	5807	62
H(41)	4972	7315	7643	63
H(42)	5565	5079	7606	74
H(43)	6048	4140	6516	80
H(44)	6026	5446	5456	76
H(45)	5437	7686	5474	63

Table 6. Torsion angles [deg] for **2f**.

C(9)-O(1)-C(1)-C(2)	-174.12(18)
C(9)-O(1)-C(1)-C(6)	4.6(3)
O(1)-C(1)-C(2)-C(3)	176.78(17)
C(6)-C(1)-C(2)-C(3)	-1.9(3)
C(1)-C(2)-C(3)-C(4)	-0.4(3)
C(1)-C(2)-C(3)-Cl(1)	179.79(16)
C(2)-C(3)-C(4)-C(5)	1.6(3)
Cl(1)-C(3)-C(4)-C(5)	-178.60(16)
C(3)-C(4)-C(5)-C(6)	-0.5(3)
O(1)-C(1)-C(6)-C(5)	-175.73(17)
C(2)-C(1)-C(6)-C(5)	2.9(3)
O(1)-C(1)-C(6)-C(7)	0.6(3)
C(2)-C(1)-C(6)-C(7)	179.27(19)
C(4)-C(5)-C(6)-C(1)	-1.6(3)
C(4)-C(5)-C(6)-C(7)	-177.7(2)
C(1)-C(6)-C(7)-C(8)	-3.3(3)
C(5)-C(6)-C(7)-C(8)	172.73(19)
C(1)-C(6)-C(7)-C(10)	178.77(18)
C(5)-C(6)-C(7)-C(10)	-5.2(3)
C(6)-C(7)-C(8)-C(9)	0.9(3)
C(10)-C(7)-C(8)-C(9)	178.92(19)
C(1)-O(1)-C(9)-O(2)	173.68(19)
C(1)-O(1)-C(9)-C(8)	-6.8(3)
C(7)-C(8)-C(9)-O(2)	-176.5(2)
C(7)-C(8)-C(9)-O(1)	4.0(3)
C(8)-C(7)-C(10)-C(15)	-47.6(3)
C(6)-C(7)-C(10)-C(15)	130.3(2)
C(8)-C(7)-C(10)-C(11)	130.2(2)
C(6)-C(7)-C(10)-C(11)	-51.8(3)
C(15)-C(10)-C(11)-C(12)	-0.2(3)
C(7)-C(10)-C(11)-C(12)	-178.08(19)
C(10)-C(11)-C(12)-C(13)	-0.1(3)
C(11)-C(12)-C(13)-C(14)	0.4(4)
C(12)-C(13)-C(14)-C(15)	-0.4(3)
C(13)-C(14)-C(15)-C(10)	0.1(3)
C(11)-C(10)-C(15)-C(14)	0.2(3)
C(7)-C(10)-C(15)-C(14)	178.2(2)
C(24)-O(3)-C(16)-C(17)	174.47(19)
C(24)-O(3)-C(16)-C(21)	-4.7(3)
O(3)-C(16)-C(17)-C(18)	-178.81(19)
C(21)-C(16)-C(17)-C(18)	0.3(3)

C(16)-C(17)-C(18)-C(19)	0.4(3)
C(16)-C(17)-C(18)-Cl(2)	178.62(16)
C(17)-C(18)-C(19)-C(20)	-0.4(3)
Cl(2)-C(18)-C(19)-C(20)	-178.60(18)
C(18)-C(19)-C(20)-C(21)	-0.4(4)
O(3)-C(16)-C(21)-C(20)	178.1(2)
C(17)-C(16)-C(21)-C(20)	-1.0(3)
O(3)-C(16)-C(21)-C(22)	0.6(3)
C(17)-C(16)-C(21)-C(22)	-178.5(2)
C(19)-C(20)-C(21)-C(16)	1.0(3)
C(19)-C(20)-C(21)-C(22)	178.3(2)
C(16)-C(21)-C(22)-C(23)	2.4(3)
C(20)-C(21)-C(22)-C(23)	-174.8(2)
C(16)-C(21)-C(22)-C(25)	-179.2(2)
C(20)-C(21)-C(22)-C(25)	3.5(3)
C(21)-C(22)-C(23)-C(24)	-1.5(3)
C(25)-C(22)-C(23)-C(24)	-179.9(2)
C(16)-O(3)-C(24)-O(4)	-176.3(2)
C(16)-O(3)-C(24)-C(23)	5.4(3)
C(22)-C(23)-C(24)-O(4)	179.5(2)
C(22)-C(23)-C(24)-O(3)	-2.4(3)
C(23)-C(22)-C(25)-C(30)	52.8(3)
C(21)-C(22)-C(25)-C(30)	-125.5(2)
C(23)-C(22)-C(25)-C(26)	-125.2(3)
C(21)-C(22)-C(25)-C(26)	56.4(3)
C(30)-C(25)-C(26)-C(27)	-0.4(4)
C(22)-C(25)-C(26)-C(27)	177.7(2)
C(25)-C(26)-C(27)-C(28)	0.7(4)
C(26)-C(27)-C(28)-C(29)	-0.1(4)
C(27)-C(28)-C(29)-C(30)	-0.7(4)
C(28)-C(29)-C(30)-C(25)	1.0(4)
C(26)-C(25)-C(30)-C(29)	-0.4(4)
C(22)-C(25)-C(30)-C(29)	-178.6(2)
C(39)-O(5)-C(31)-C(32)	-177.94(19)
C(39)-O(5)-C(31)-C(36)	2.2(3)
O(5)-C(31)-C(32)-C(33)	177.36(18)
C(36)-C(31)-C(32)-C(33)	-2.8(3)
C(31)-C(32)-C(33)-C(34)	-1.6(3)
C(31)-C(32)-C(33)-Cl(3)	178.72(17)
C(32)-C(33)-C(34)-C(35)	3.1(3)
Cl(3)-C(33)-C(34)-C(35)	-177.21(17)
C(33)-C(34)-C(35)-C(36)	-0.4(3)
C(32)-C(31)-C(36)-C(35)	5.2(3)
O(5)-C(31)-C(36)-C(35)	-174.90(18)

C(32)-C(31)-C(36)-C(37)	-176.6(2)
O(5)-C(31)-C(36)-C(37)	3.2(3)
C(34)-C(35)-C(36)-C(31)	-3.6(3)
C(34)-C(35)-C(36)-C(37)	178.4(2)
C(31)-C(36)-C(37)-C(38)	-5.6(3)
C(35)-C(36)-C(37)-C(38)	172.3(2)
C(31)-C(36)-C(37)-C(40)	173.62(19)
C(35)-C(36)-C(37)-C(40)	-8.4(3)
C(36)-C(37)-C(38)-C(39)	2.8(3)
C(40)-C(37)-C(38)-C(39)	-176.4(2)
C(31)-O(5)-C(39)-O(6)	176.80(19)
C(31)-O(5)-C(39)-C(38)	-5.0(3)
C(37)-C(38)-C(39)-O(6)	-179.6(2)
C(37)-C(38)-C(39)-O(5)	2.4(3)
C(38)-C(37)-C(40)-C(41)	136.5(2)
C(36)-C(37)-C(40)-C(41)	-42.7(3)
C(38)-C(37)-C(40)-C(45)	-41.5(3)
C(36)-C(37)-C(40)-C(45)	139.3(2)
C(45)-C(40)-C(41)-C(42)	-1.7(3)
C(37)-C(40)-C(41)-C(42)	-179.8(2)
C(40)-C(41)-C(42)-C(43)	0.2(4)
C(41)-C(42)-C(43)-C(44)	1.4(4)
C(42)-C(43)-C(44)-C(45)	-1.4(4)
C(43)-C(44)-C(45)-C(40)	-0.2(3)
C(41)-C(40)-C(45)-C(44)	1.7(3)
C(37)-C(40)-C(45)-C(44)	179.83(19)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for **2f** [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(38)-H(38)...O(2)#1	0.93	2.39	3.311(3)	169.1
C(13)-H(13)...O(1)#2	0.93	2.46	3.343(3)	159.2

Symmetry transformations used to generate equivalent atoms:
#1 x+1, y+1, z #2 x, y+1, z