

Supplementary Information

Synthesis of functionalized 2-aryl-4-(indol-3-yl)-4*H*-chromenes via iodine-catalyzed domino Michael addition-intramolecular cyclization reaction

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Anxin Wu,^b and Nengfang She^{*,b}

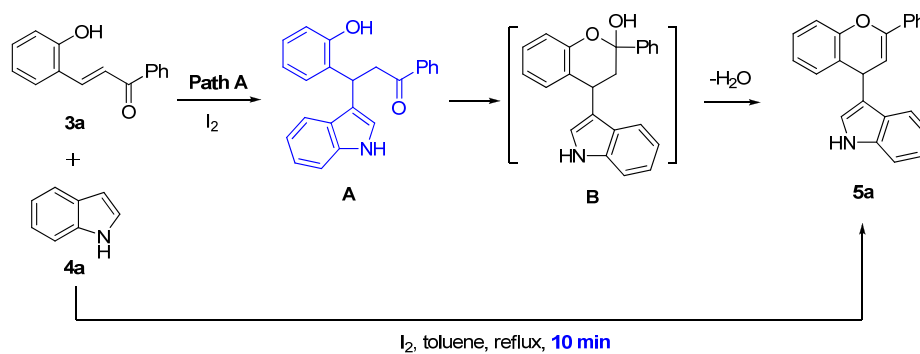
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E-mail: gdyin@hbnu.edu.cn

^bKey Laboratory of Pesticides and Chemical Biology of the Ministry of Education, College of Chemistry, Central China Normal University, Wuhan 430079, China.
E-mail: nfshe@mail.ccnu.edu.cn

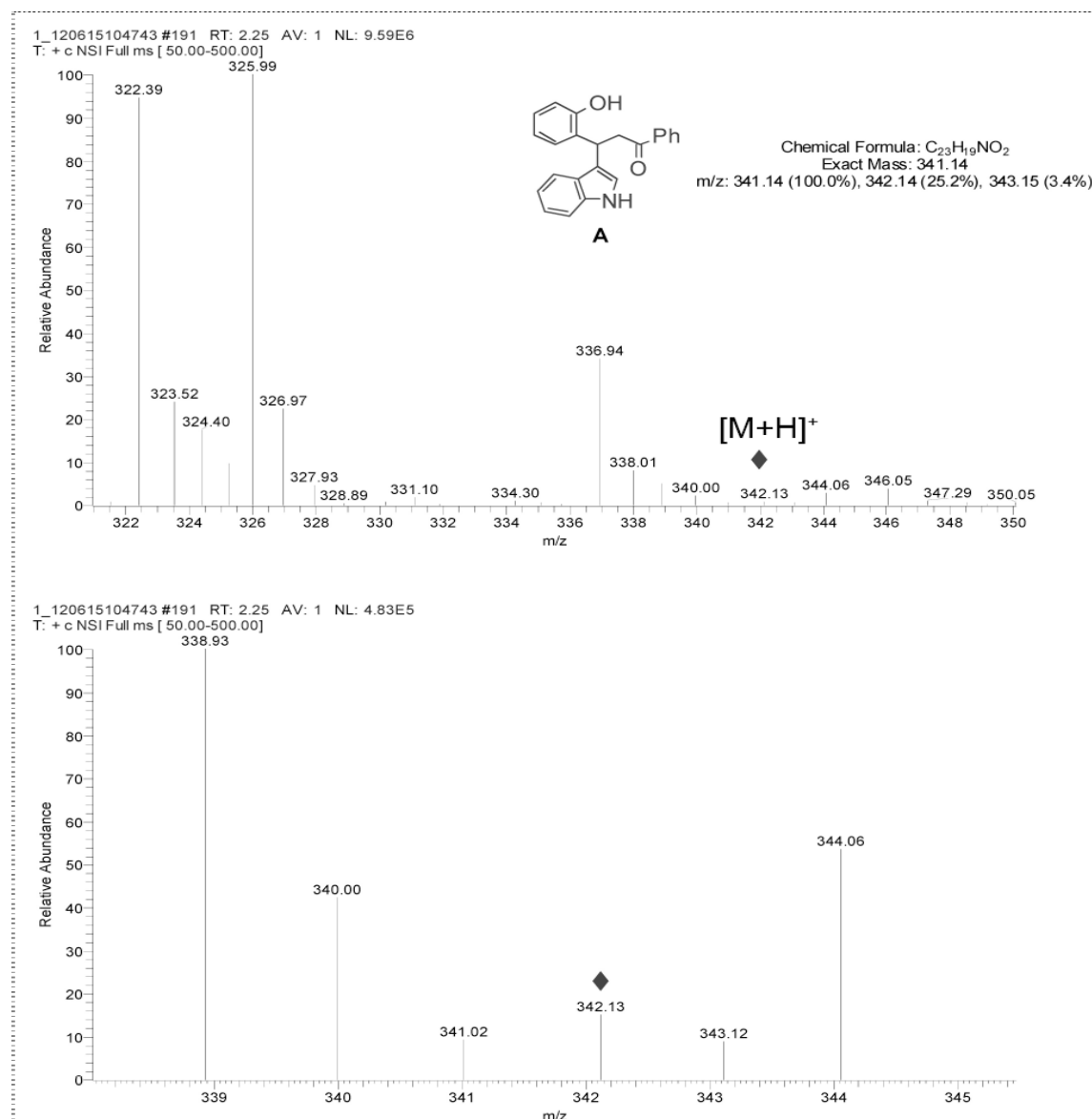
Outline

◆ Mechanism study.....	S2
◆ Spectral copies of ¹ H NMR, ¹³ C NMR of all compounds.....	S3-S50
◆ HRMS for compounds 5 , 7-9	S51-S66
◆ HSQC Spectrum of compound 7	S67

◆ Mechanism study



ESI-MS of the reaction mixture after 10min, and intermediate **A** was observed.

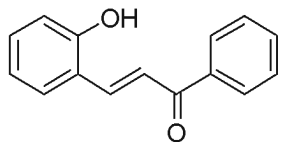


^1H NMR (300MHz, CDCl_3)

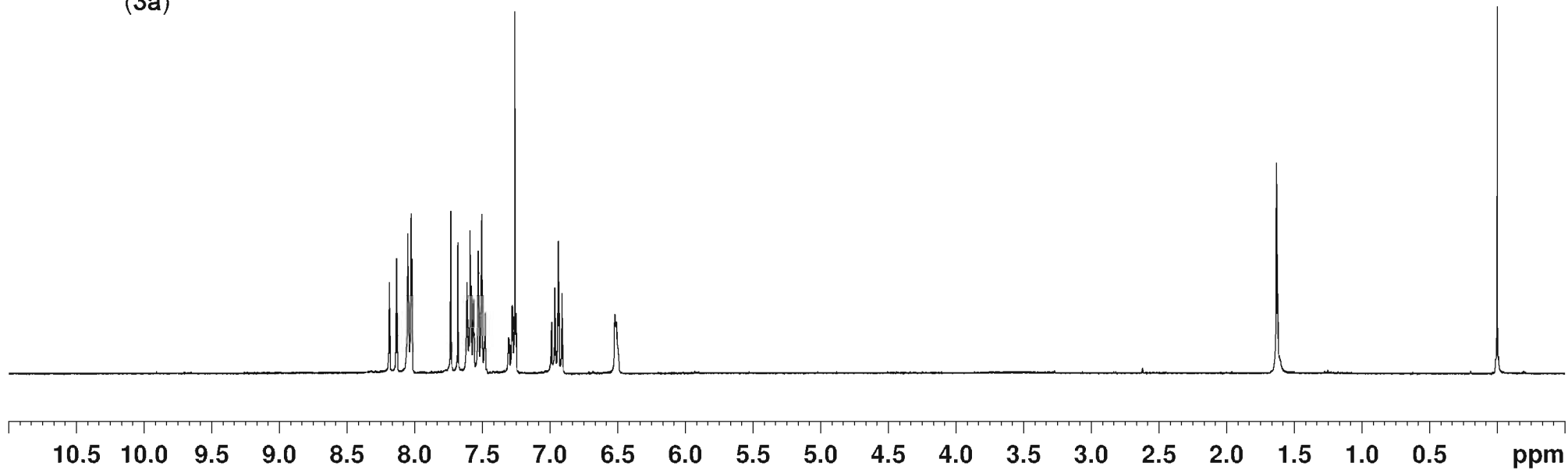
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8.0538
8.0508
8.0271
8.0218
7.7341
7.6813
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7.6108
7.6001
7.5911
7.5845
7.5717
7.5671
7.5631
7.5320
7.5104
7.5057
7.4875
7.4822
7.4775
7.3079
7.3027
7.2809
7.2781
7.2609
7.2598
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6.9895
6.9652
6.9395
6.9124
6.5206
6.5111

—1.6307

—-0.0001

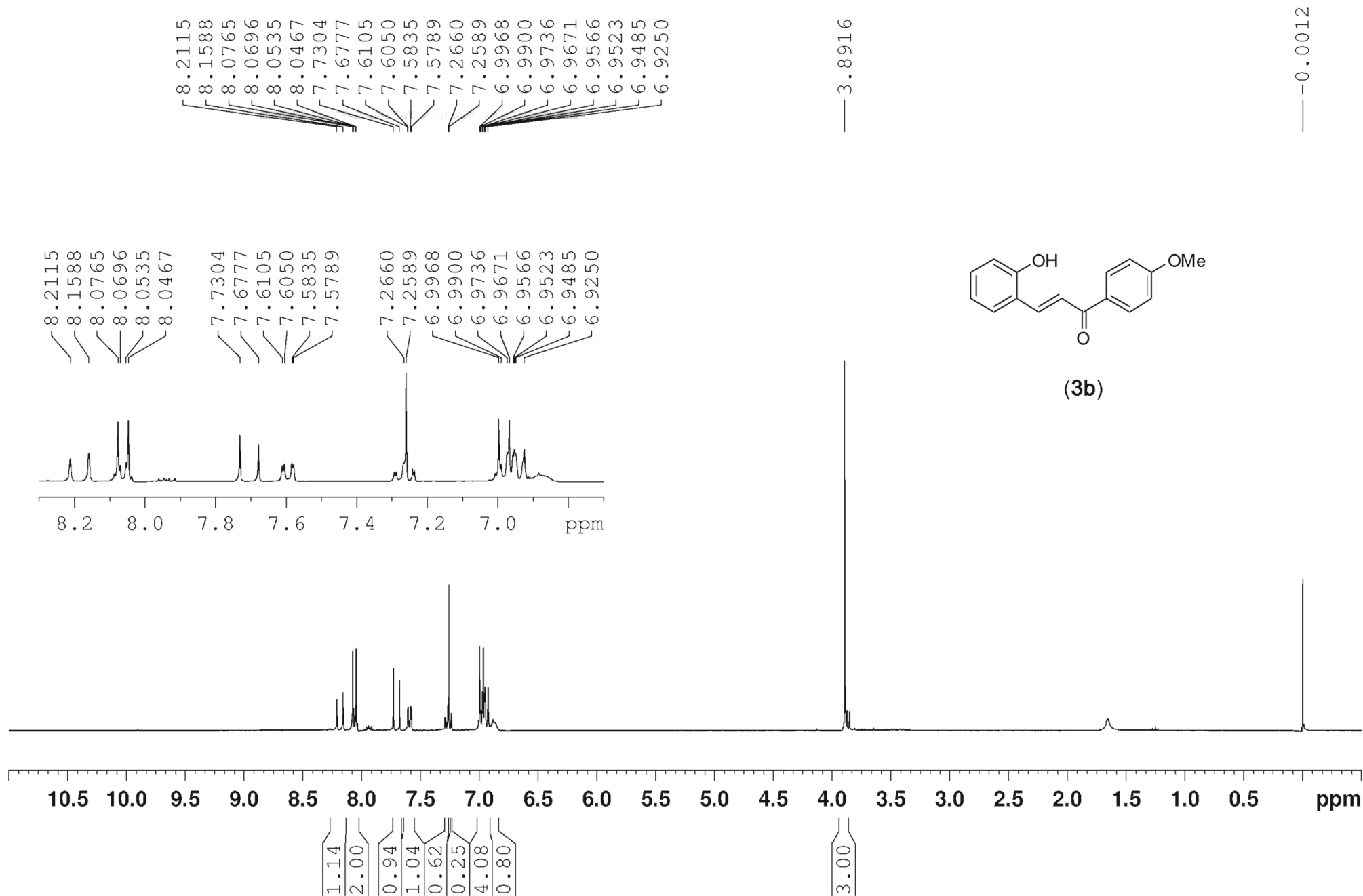


(3a)



1.31
2.31
1.31
2.37
2.28
0.96
2.37
1.00

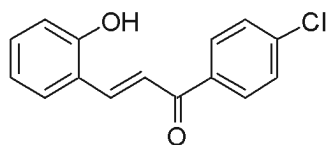
^1H NMR (300MHz, CDCl_3)



^1H NMR (300MHz, CDCl_3)

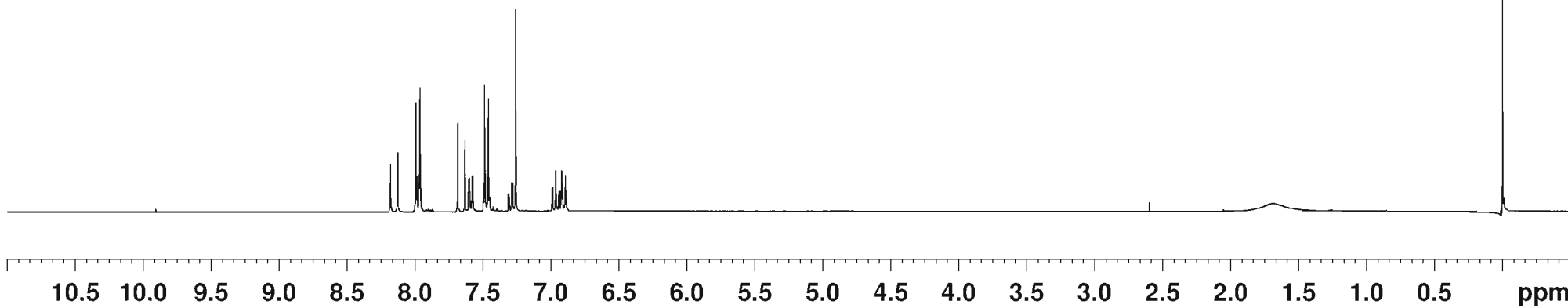
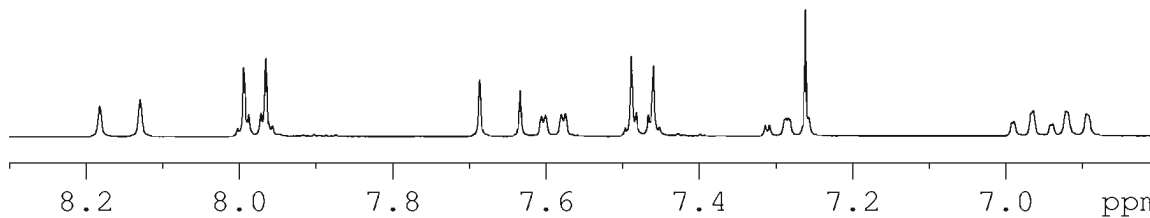
8.1818
8.1291
7.9940
7.9873
7.9716
7.9649
7.9571
7.6868
7.6340
7.6062
7.6008
7.5803
7.5750
7.4969
7.4890
7.4823
7.4666
7.4599
7.4521
7.3140
7.3085
7.2870
7.2840
7.2820
7.2618
7.2573
6.9921
6.9896
6.9641
6.9419
6.9392
6.9220
6.9196
6.8950
6.8924

0.0000



(3c)

8.1818
8.1291
7.9940
7.9873
7.9716
7.9649
7.9571
7.6868
7.6340
7.6062
7.6008
7.5803
7.5750
7.4890
7.4823
7.4666
7.4599
7.4521
7.3140
7.3085
7.2870
7.2840
7.2820
7.2618
7.2573
6.9921
6.9896
6.9641
6.9419
6.9392
6.9220
6.9196
6.8950
6.8924



1.00
2.01
1.02
0.47
0.55
1.97
0.72
2.21

^{13}C NMR (75MHz, CDCl_3)

— 190.4862

— 155.7926

141.3824

139.1137

136.5596

131.9218

129.9859

129.5900

128.8422

122.1885

121.9936

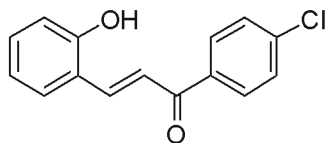
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116.5484

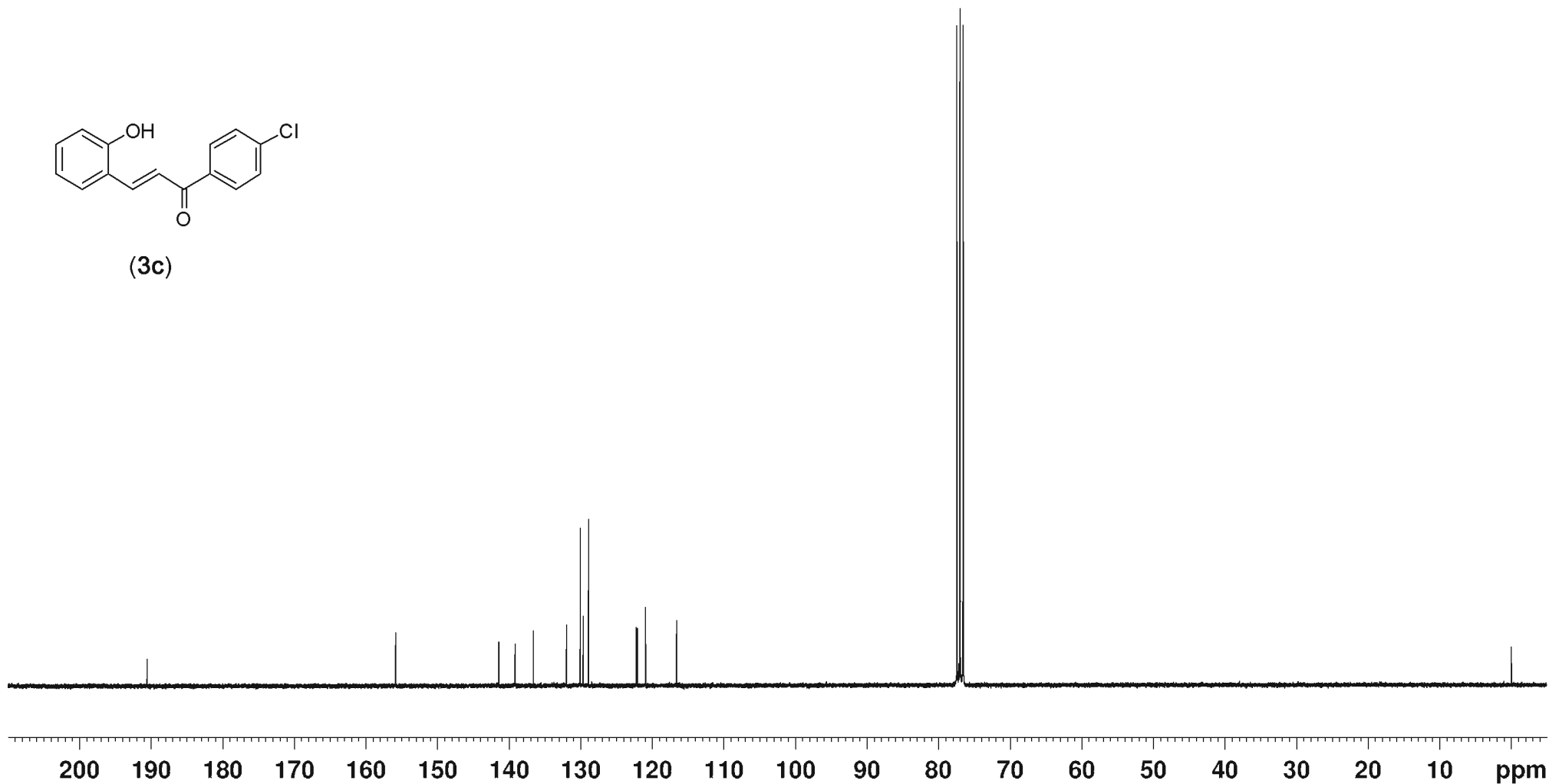
77.3680

76.9447

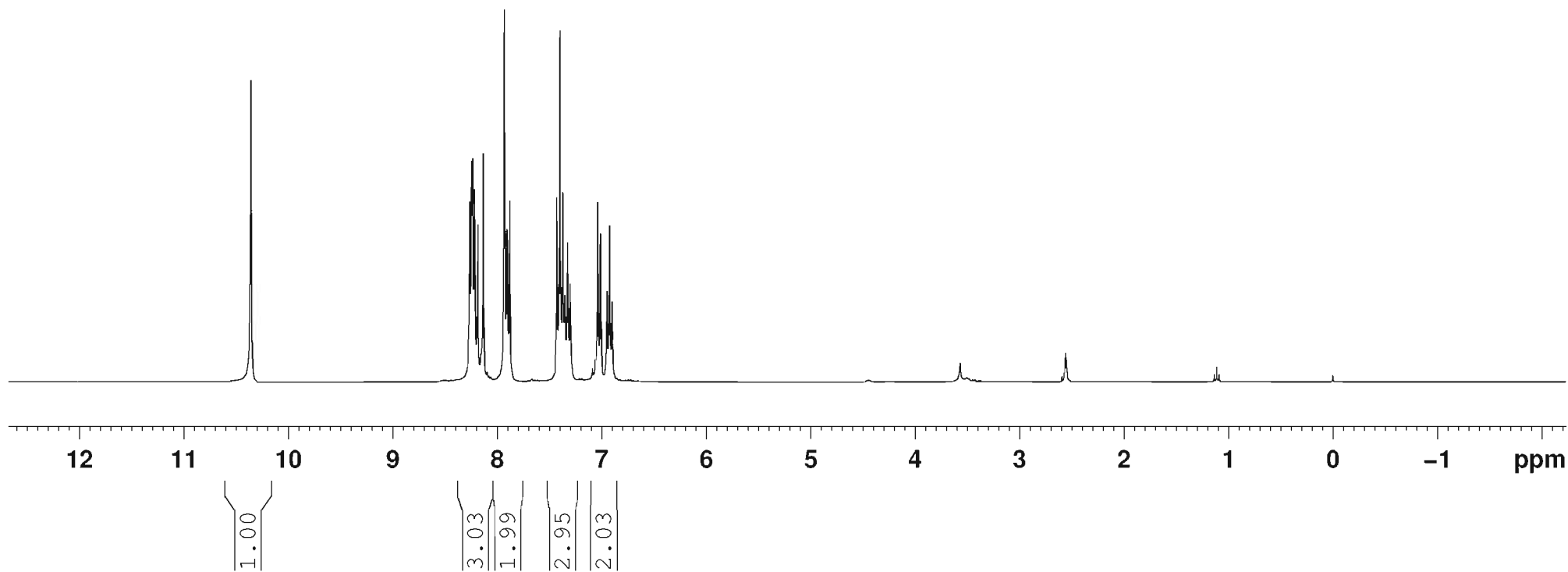
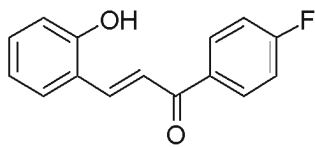
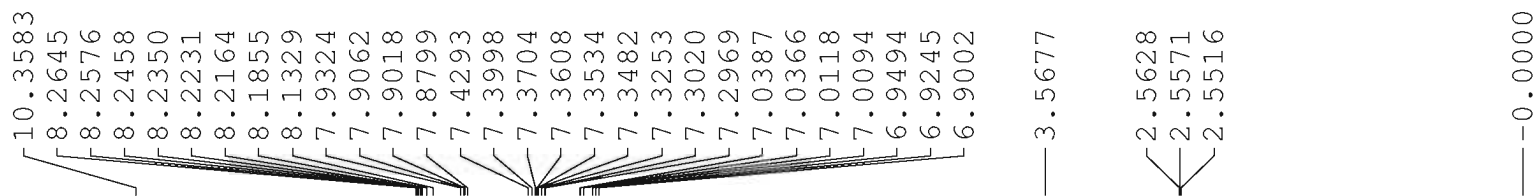
76.5213



(3c)



^1H NMR (300MHz, DMSO-d_6)



^{13}C NMR (75MHz, DMSO- d_6)

— 187.9875

— 166.5949

— 163.2572

— 157.3503

139.7294

134.6125

134.5758

132.1020

131.3458

131.2220

128.7022

121.4069

120.6197

119.4148

116.2936

115.8599

115.5717

40.3269

40.0562

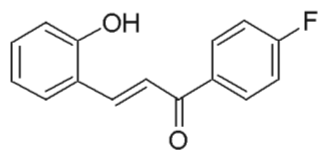
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39.5006

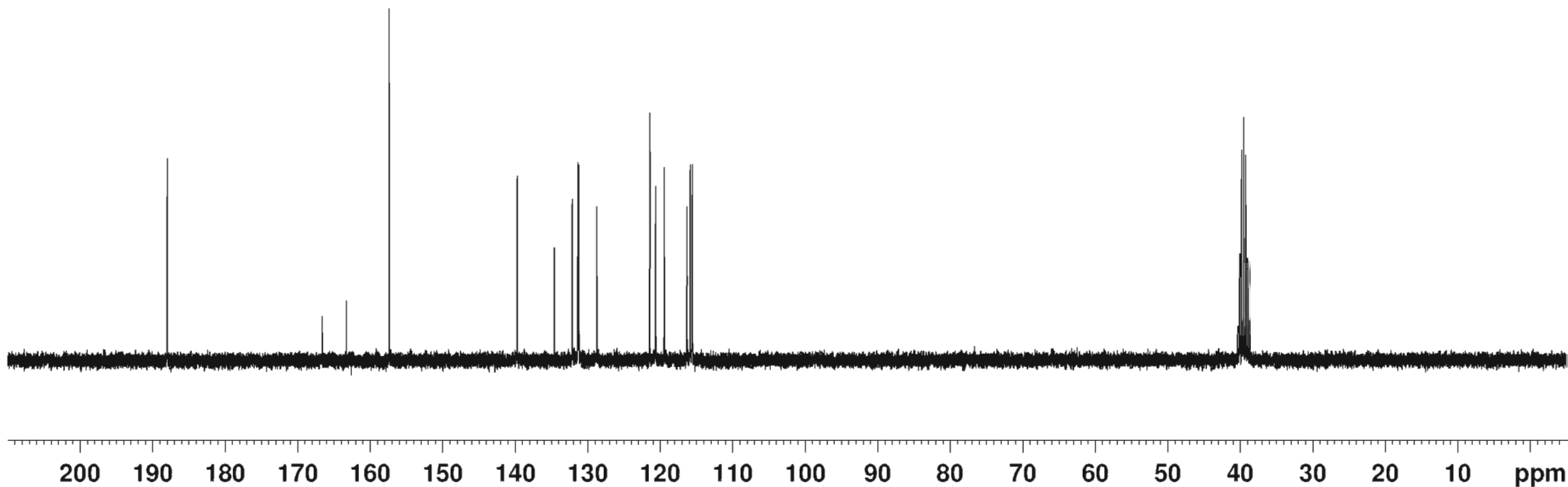
39.2211

38.9481

38.6636

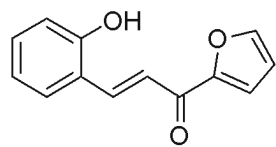


(3d)

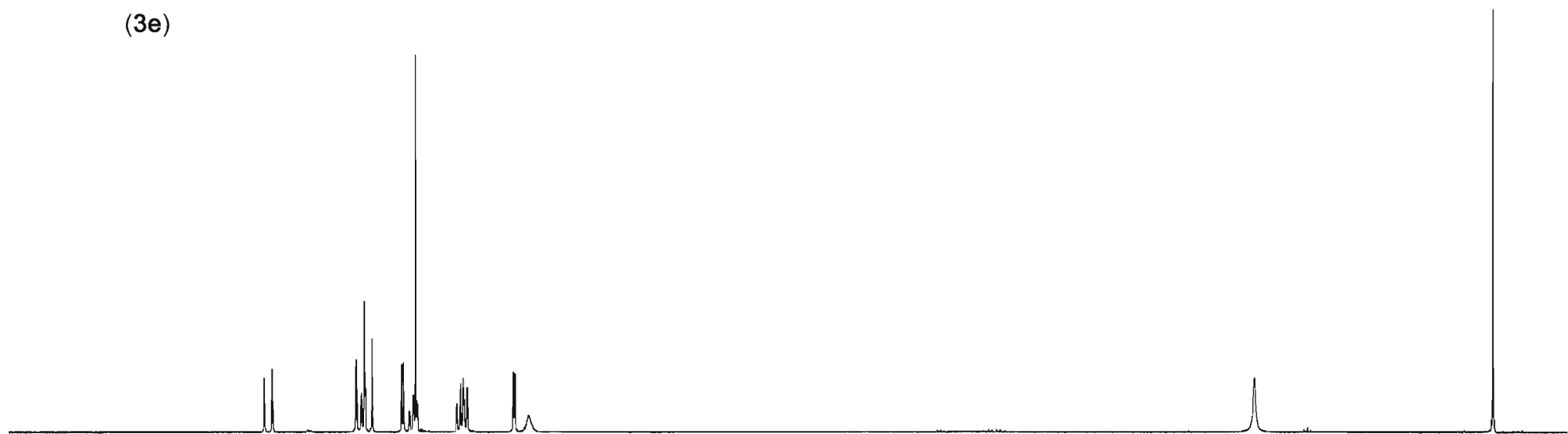


^1H NMR (300MHz, CDCl_3)

8.2266
7.6642
7.6619
7.6586
7.6563
7.6285
7.6236
7.6052
7.5978
7.5523
7.3547
7.3525
7.3428
7.3406
7.3035
7.2980
7.2766
7.2729
7.2614
7.2520
7.2465
6.9822
6.9567
6.9404
6.9136
6.6046
6.5989
6.5927
6.5870
6.4996



(3e)



— 1.6079

0.0109
0.0001
-0.0110

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

1.00
2.92
0.88
2.20
1.98
0.84
0.69

^{13}C NMR (75MHz, CDCl_3)

— 178.9468

— 155.9351

— 153.7813

— 146.6317

— 139.8820

— 131.8957

— 129.3214

— 122.0378

— 121.6033

— 120.8269

— 117.7722

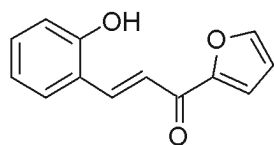
— 116.6730

— 112.5199

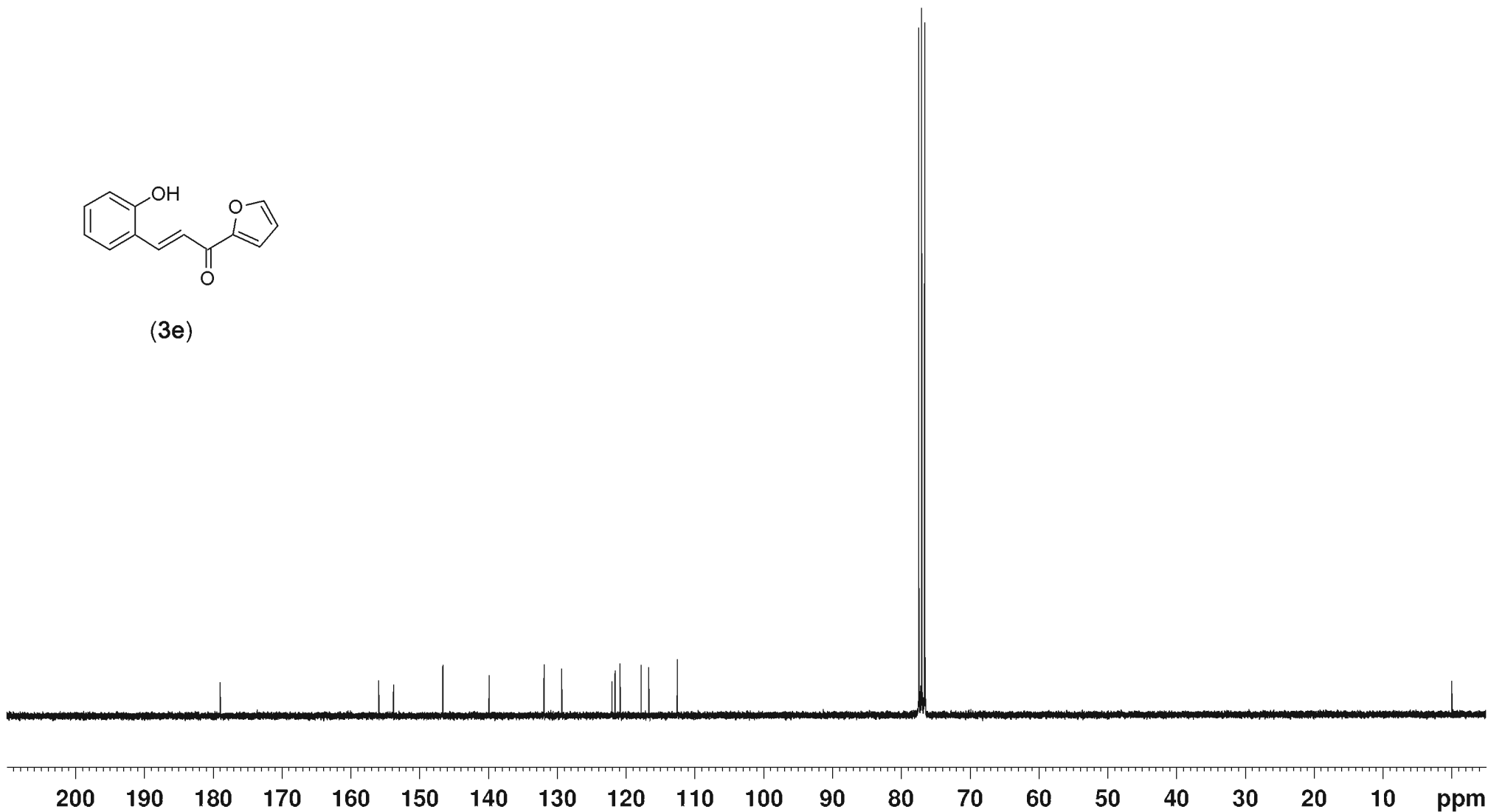
— 77.4265

— 77.0034

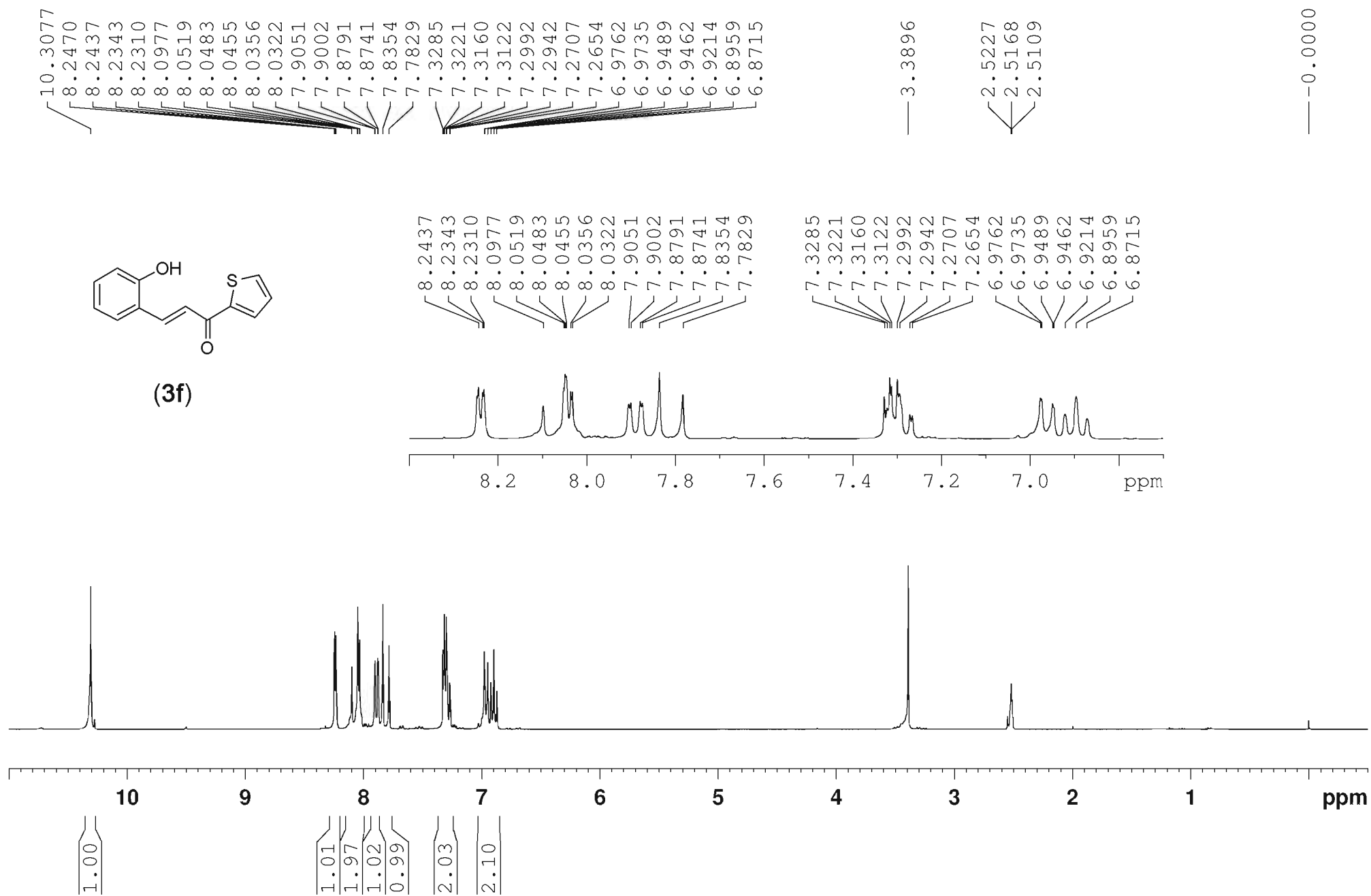
— 76.5798



(3e)



¹H NMR (300MHz, DMSO-d₆)



^{13}C NMR (75MHz, DMSO- d_6)

— 181.8306

— 157.2981

— 145.7356

— 138.5689

— 135.1371

— 133.0850

— 132.1649

— 128.9001

— 128.5908

— 121.2353

— 120.6593

— 119.4292

— 116.2906

40.3760

40.1041

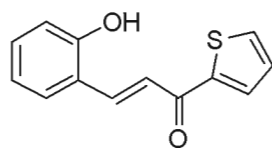
39.8279

39.5491

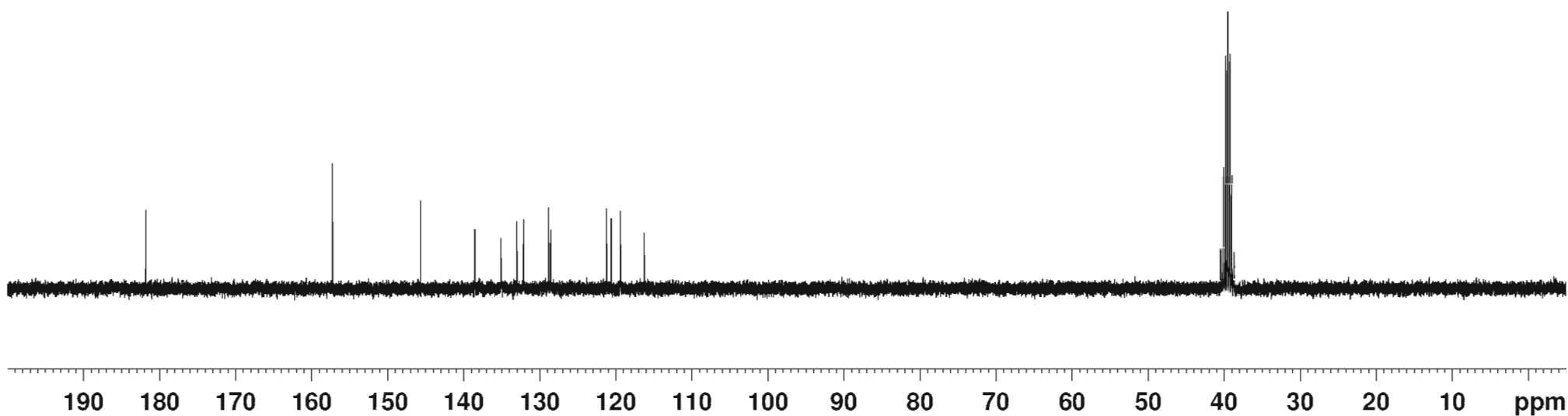
39.2724

38.9929

38.7073



(3f)

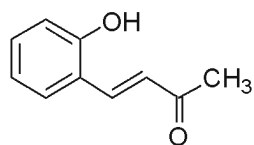


^1H NMR (300MHz, CDCl_3)

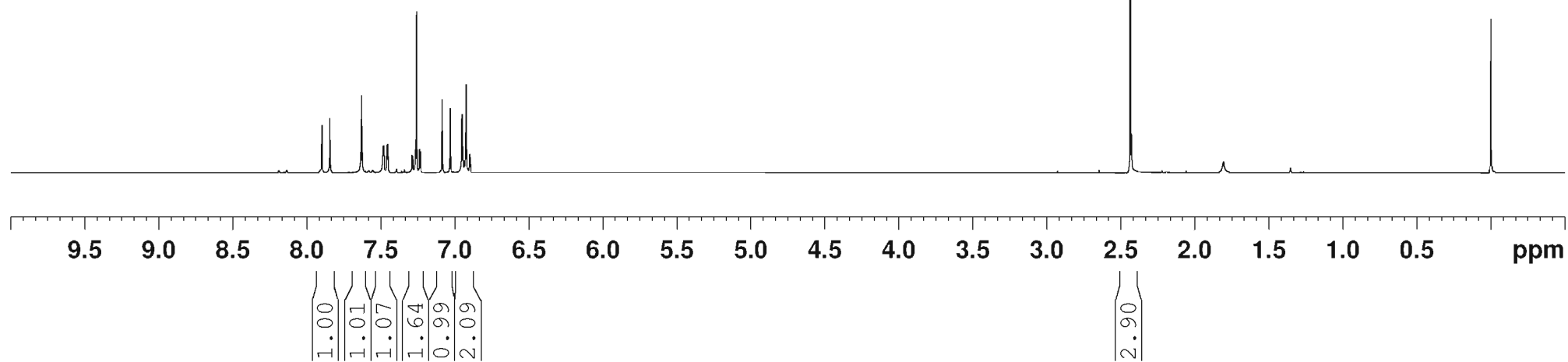
7.8995
7.8447
7.6311
7.4850
7.4805
7.4598
7.4546
7.2901
7.2846
7.2599
7.2386
7.2331
7.0860
7.0313
6.9520
6.9251
6.9001
6.8967

2.4366

0.0000



(3g)



^{13}C NMR (75MHz, CDCl_3)

— 201.3680

— 156.1141

— 141.0112

— 131.9369

— 129.7447

— 127.6877

— 121.4674

— 120.6256

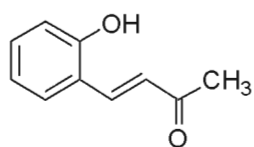
— 116.6008

— 77.4207

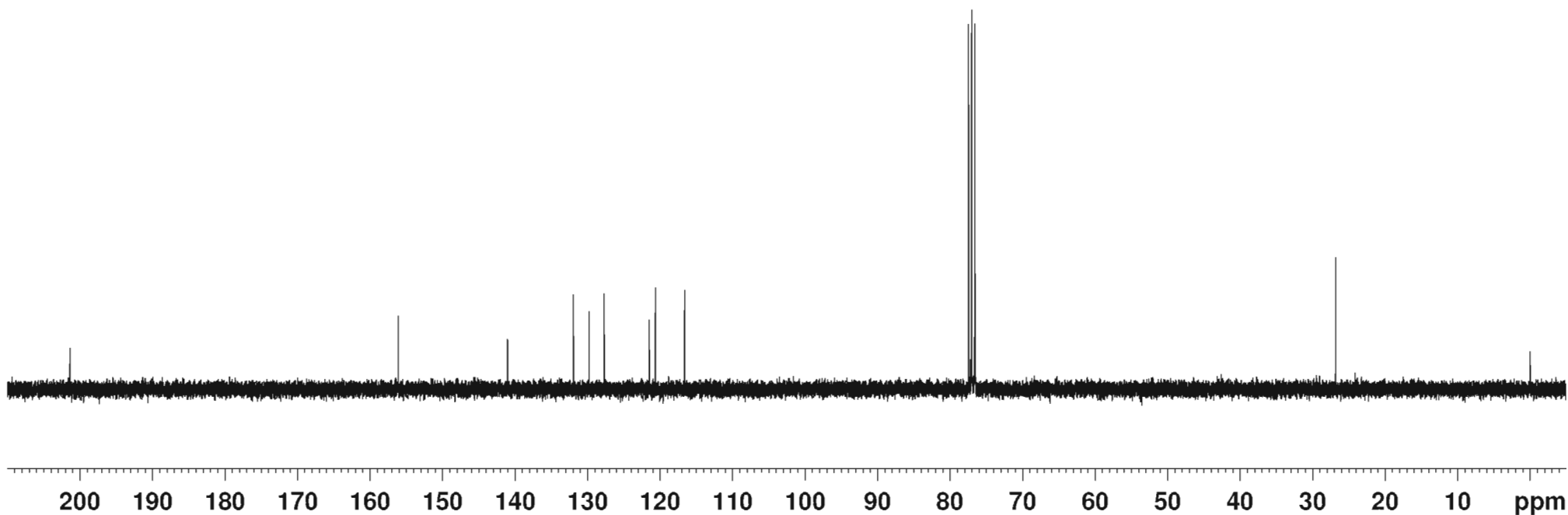
— 76.9969

— 76.5741

— 26.7821



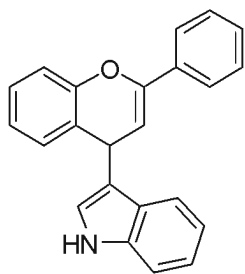
(3g)



^1H NMR (300MHz, CDCl_3)

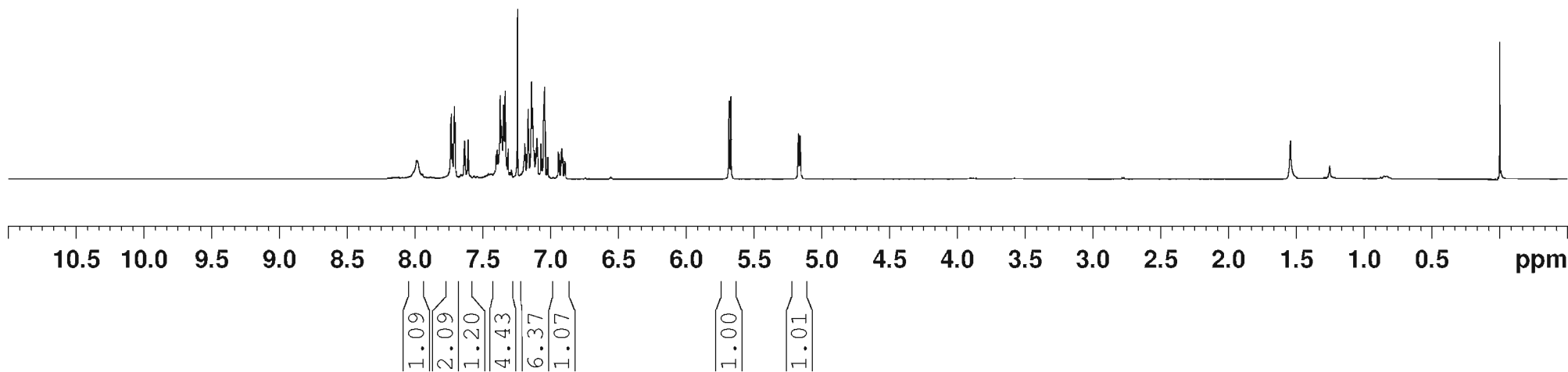
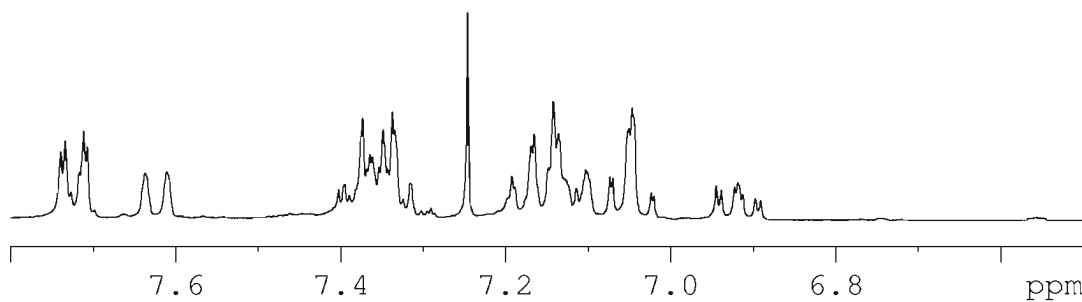
7.9868
7.7388
7.7333
7.7109
7.7065
7.6364
7.6103
7.3733
7.3486
7.3373
7.3345
7.3155
7.2460
7.1923
7.1693
7.1653
7.1478
7.1421
7.1360
7.0735
7.0702
7.0504
7.0466
7.0235
7.0202
6.9445
6.9384
6.9223
6.9185
6.9124
6.8971
6.8907
5.6851
5.6718
5.1729
5.1597

— 0.0001

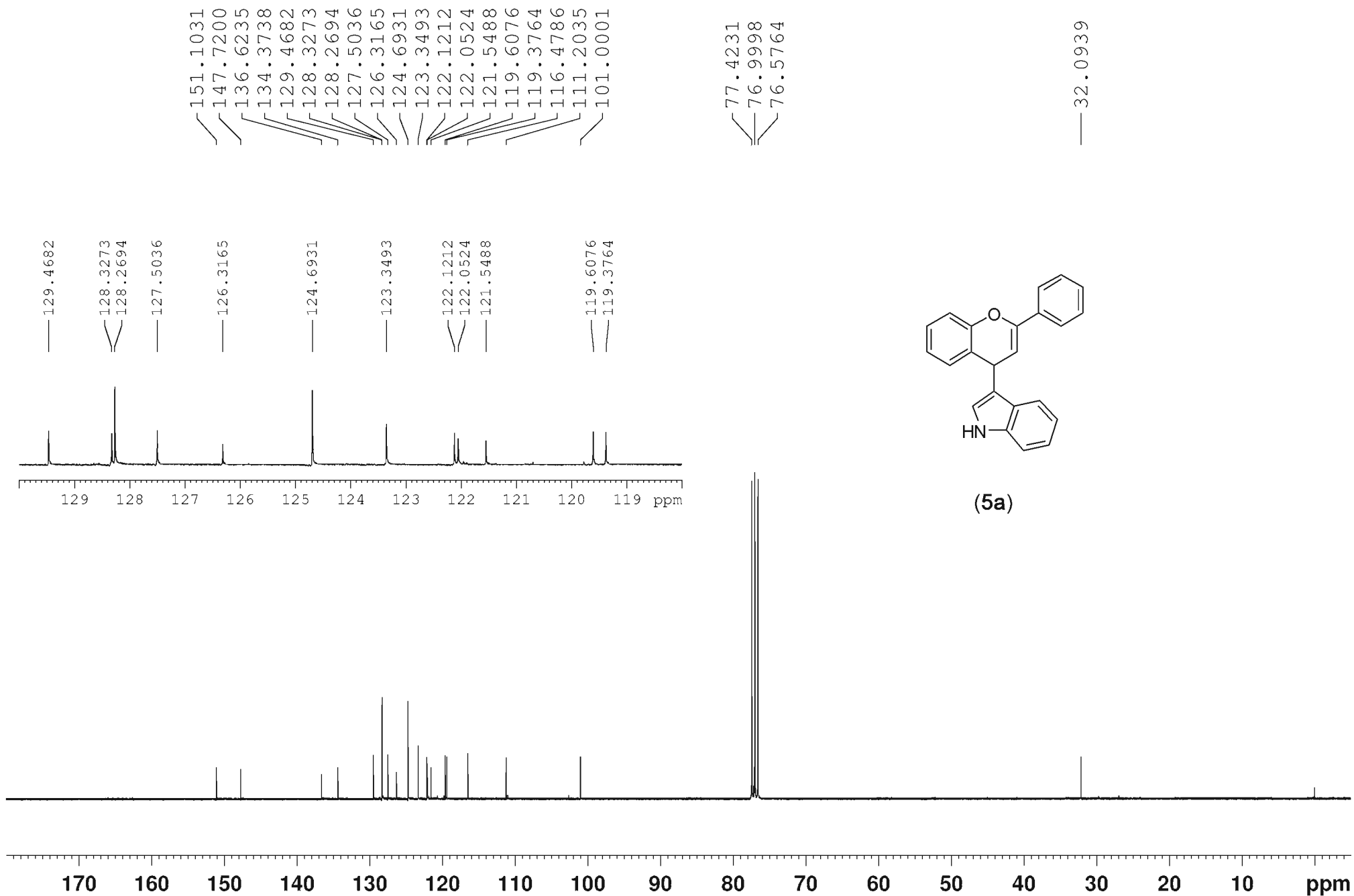


(5a)

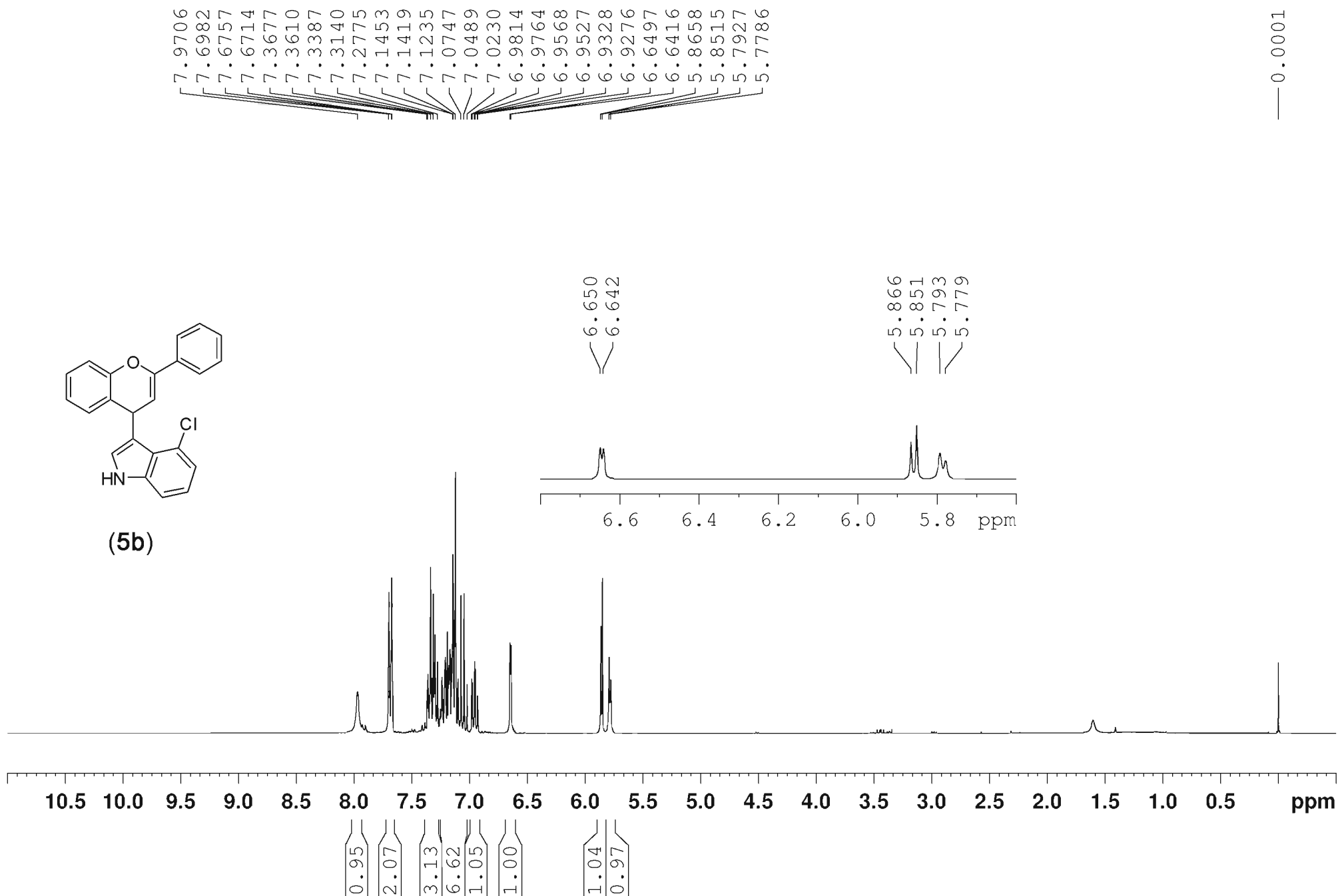
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7.707
7.636
7.610
7.373
7.349
7.337
7.335
7.316
7.246
7.192
7.169
7.165
7.148
7.142
7.136
7.074
7.070
7.050
7.047
7.024
7.020
6.944
6.938
6.922
6.918
6.912
6.897
6.891



^{13}C NMR (75MHz, CDCl_3)



^1H NMR (300MHz, CDCl_3)

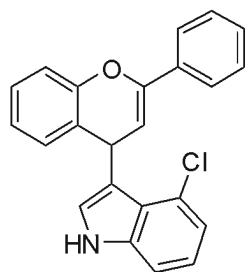


^{13}C NMR (75MHz, CDCl_3)

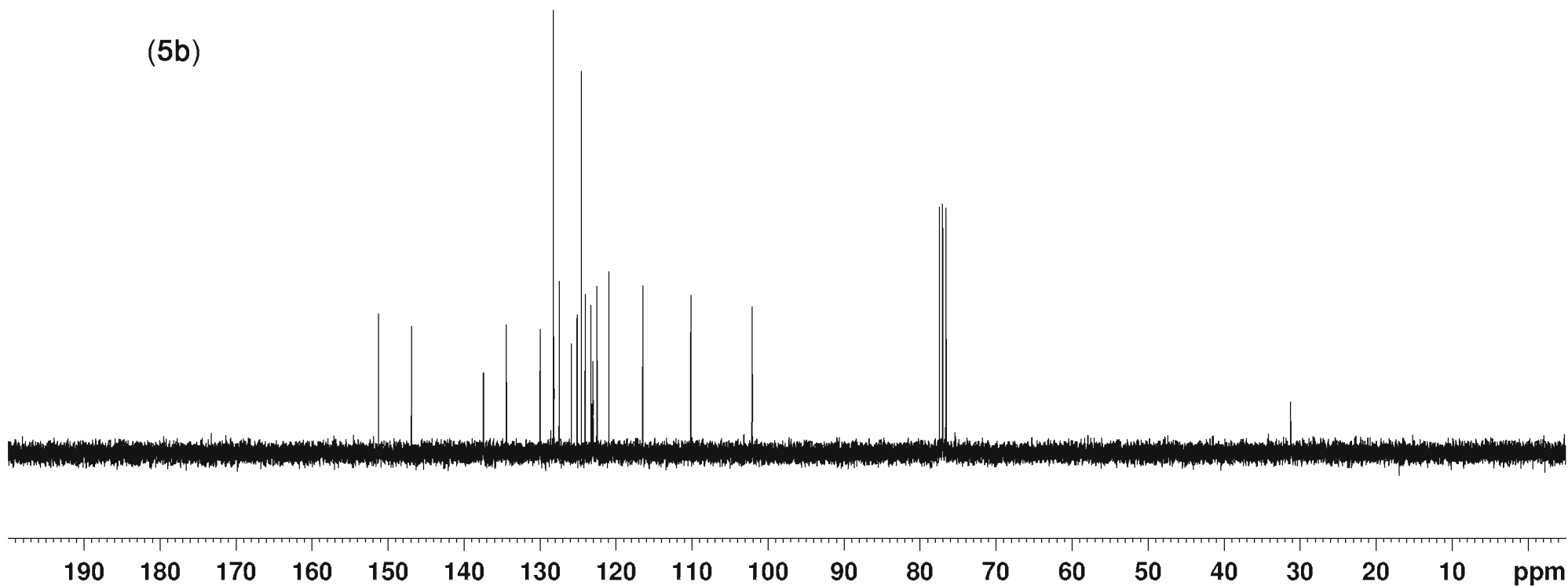
151.2518
146.8882
137.4772
134.3846
129.9707
128.2448
127.4279
125.8871
125.0943
124.5721
124.0372
123.2919
123.0801
123.0197
122.4735
120.9199
116.4469
110.1458
102.0614

77.4222
76.9985
76.5753

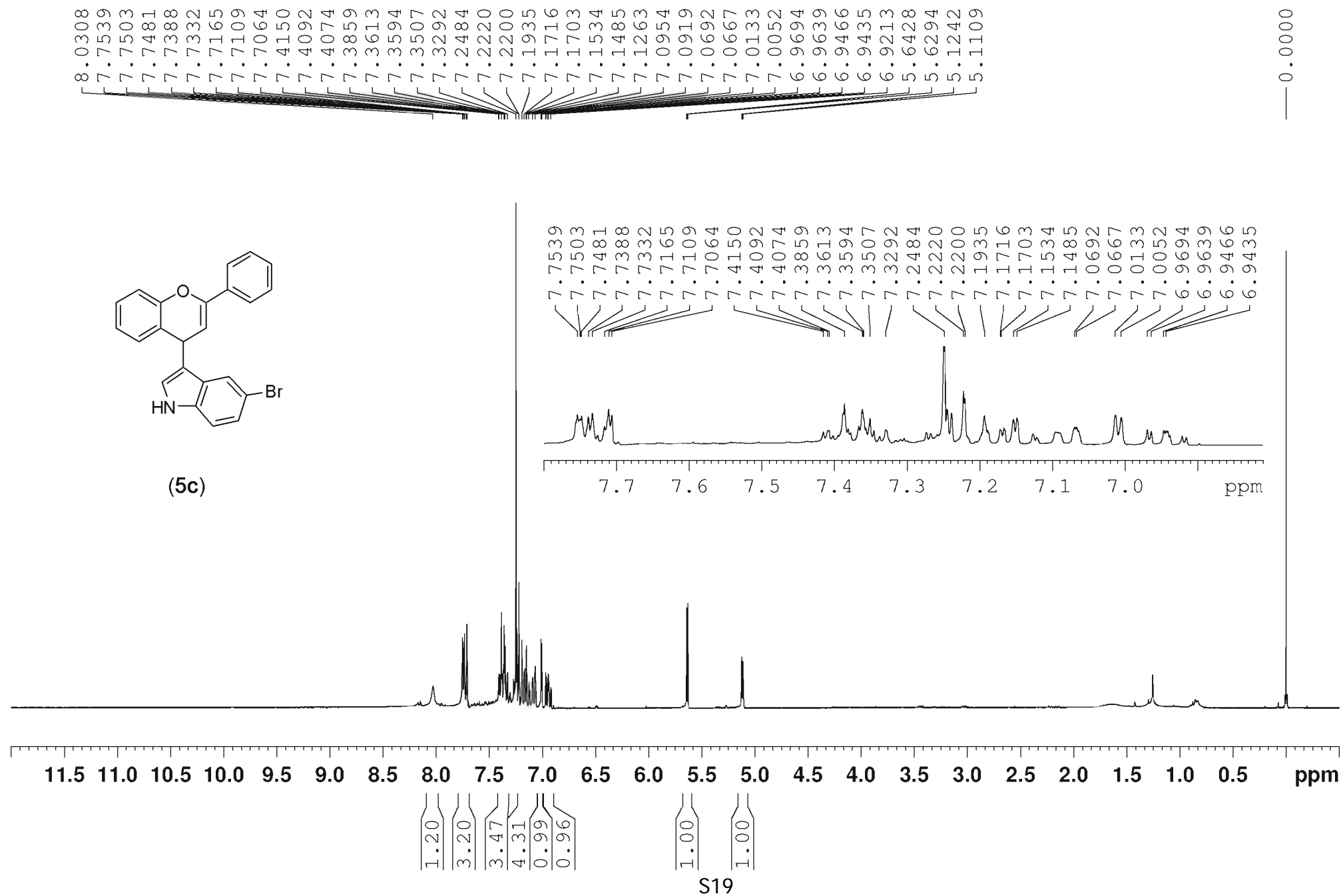
31.2073



(5b)



^1H NMR (300 MHz, CDCl_3)



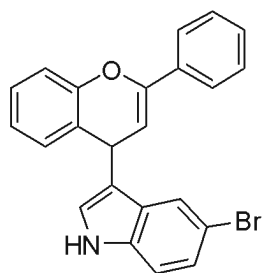
^{13}C NMR (75 MHz, CDCl_3)

151.0827
148.0093
135.1526
134.2537
129.2773
128.4500
128.3151
128.0400
127.7070
125.0671
124.7292
123.4904
123.4558
122.9685
121.7883
121.4333
116.6535
112.9584
112.6662
100.5395

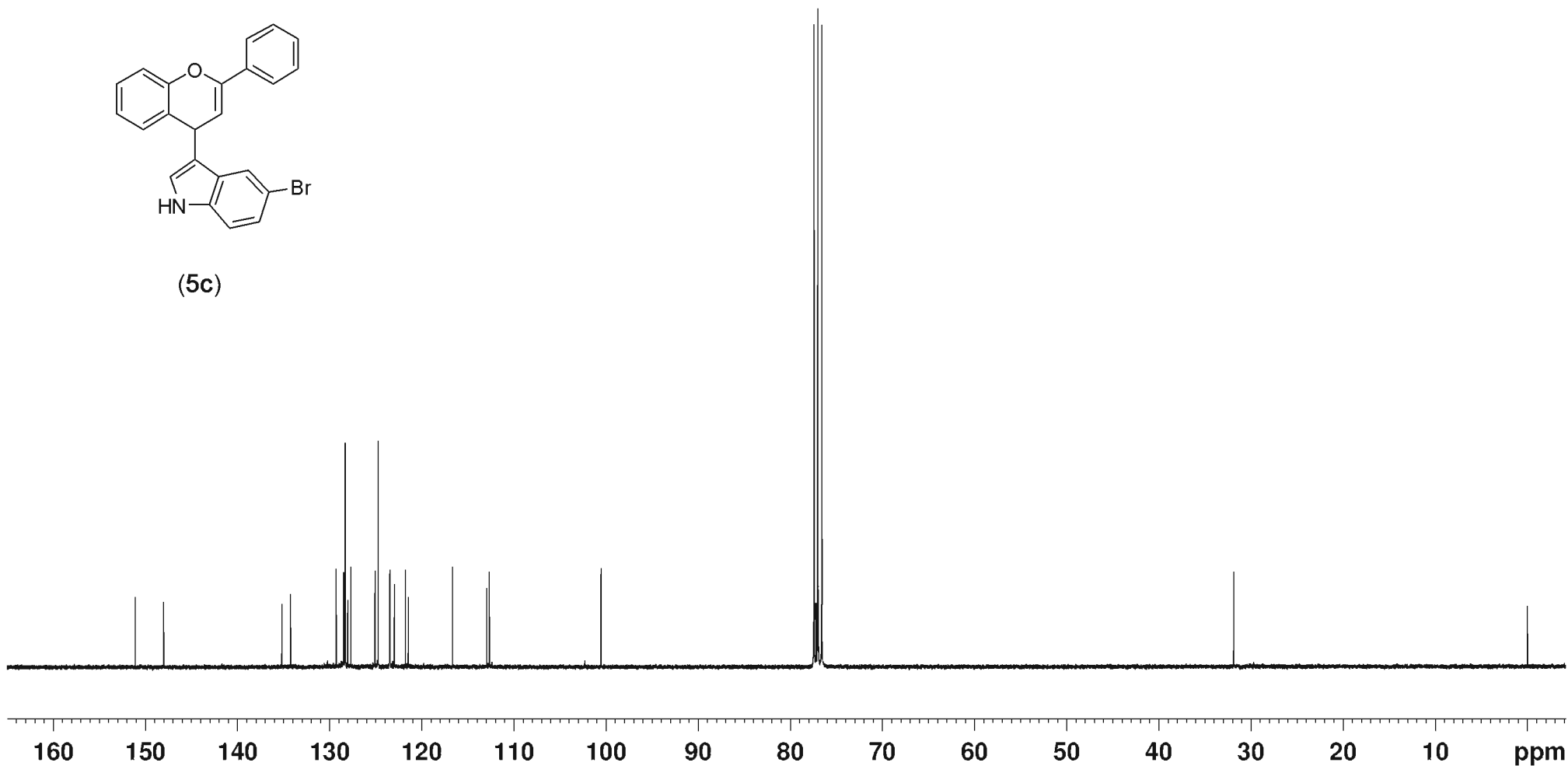
77.4207
76.9974
76.5740

— 31.8365

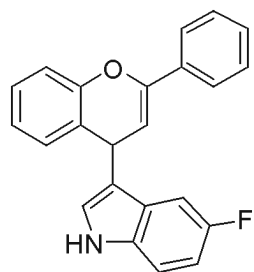
— -0.0180



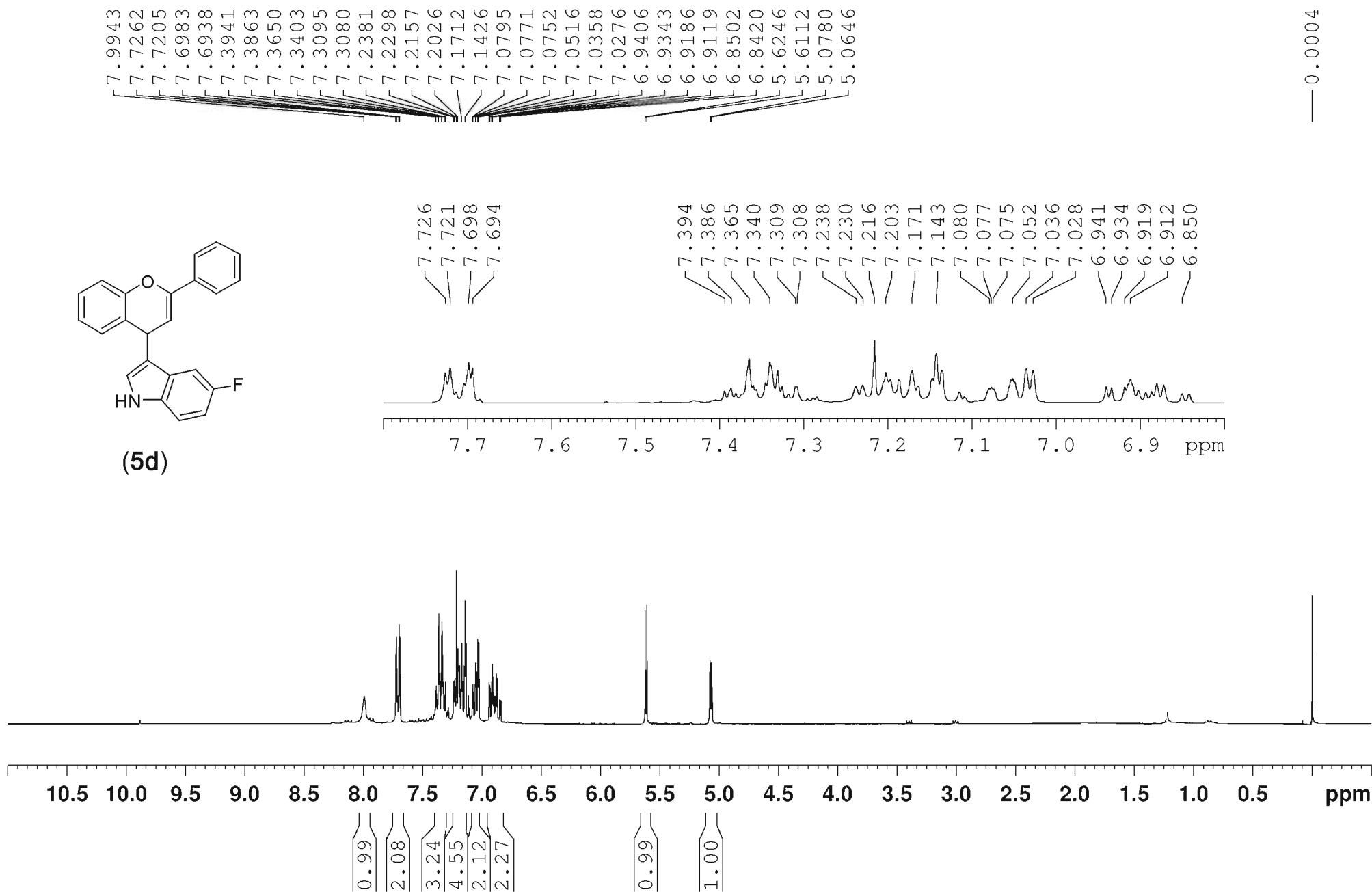
(5c)



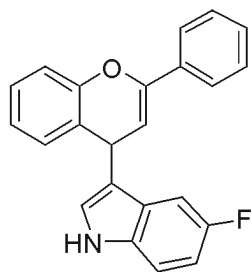
^1H NMR (300MHz, CDCl_3)



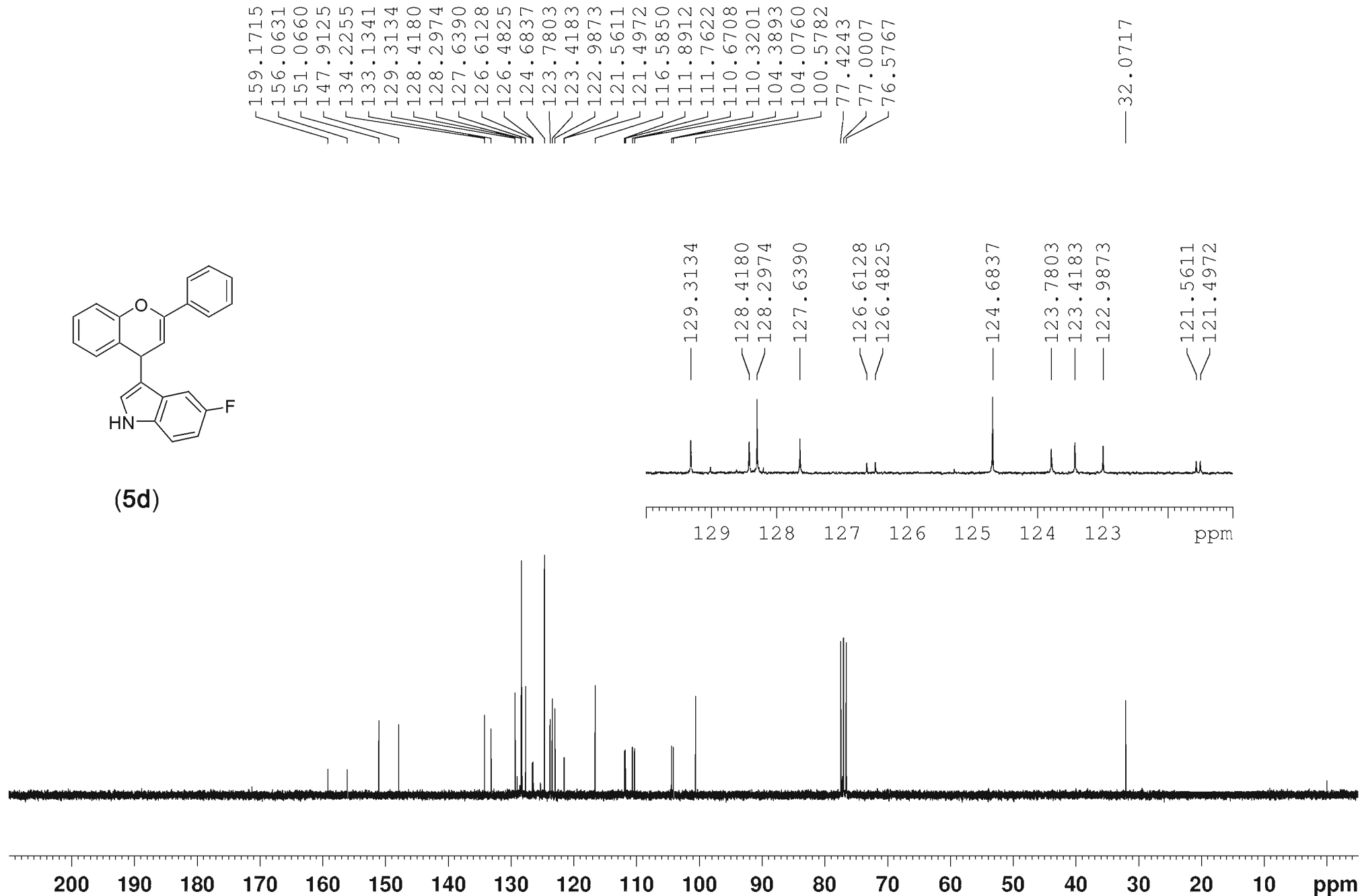
(5d)



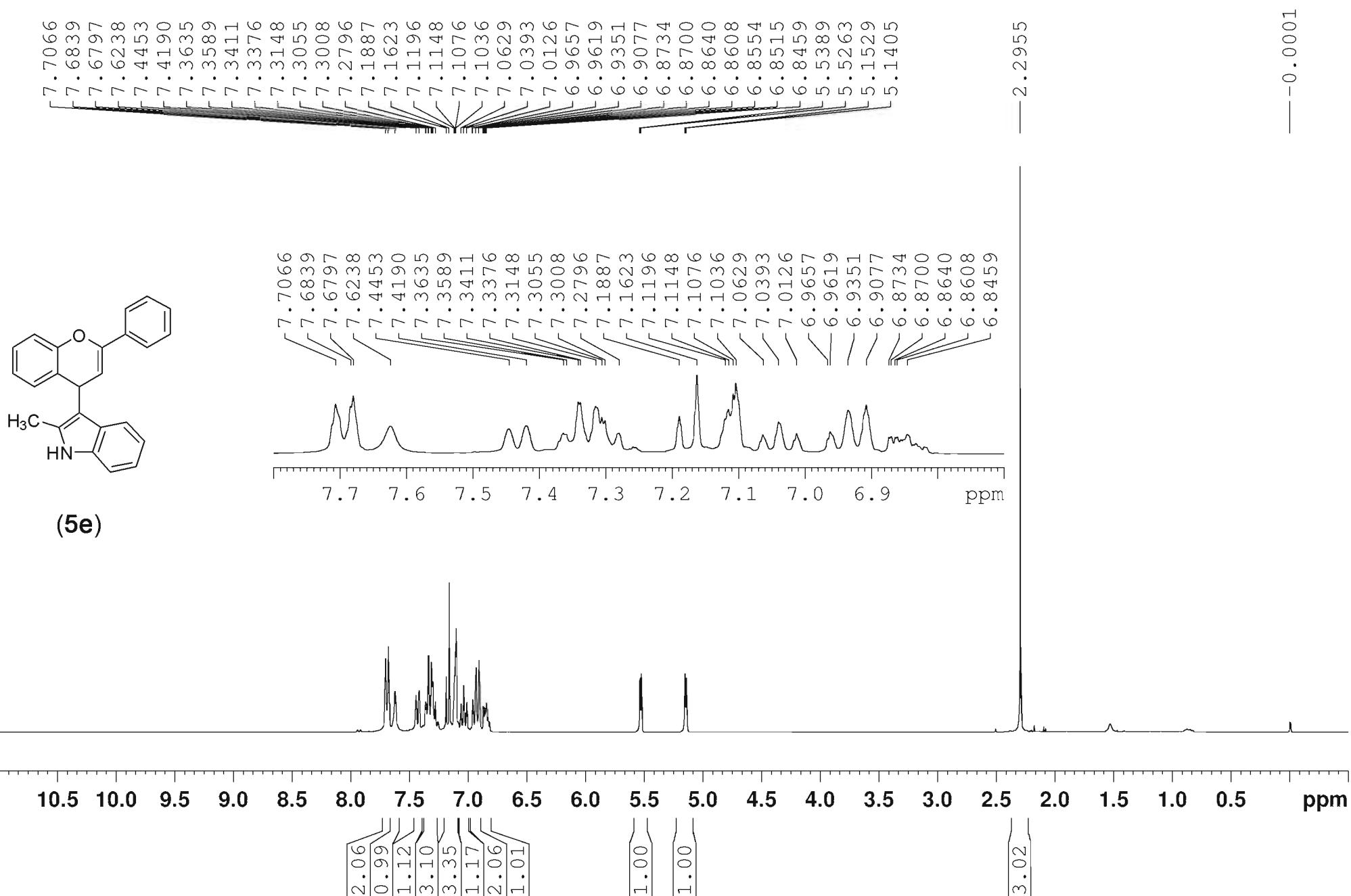
^{13}C NMR (75MHz, CDCl_3)



(5d)



^1H NMR (300MHz, CDCl_3)



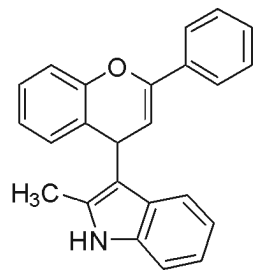
^{13}C NMR (75MHz, CDCl_3)

151.1531
147.5887
135.0711
134.3339
131.4115
129.3703
128.2555
127.7594
127.3864
124.5932
123.3810
123.2707
120.9726
119.3577
118.4998
116.2590
115.8721
110.1805
100.8642

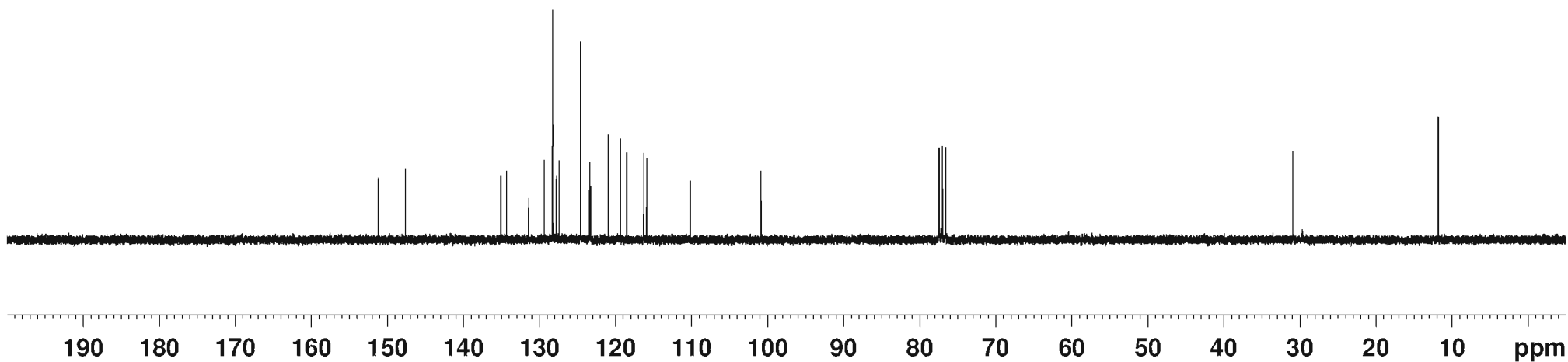
77.4225
77.0013
76.5760

30.9146

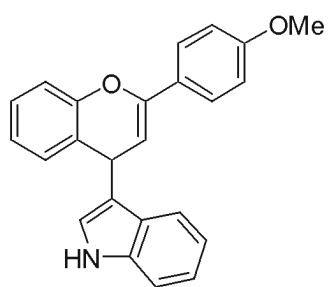
11.7897



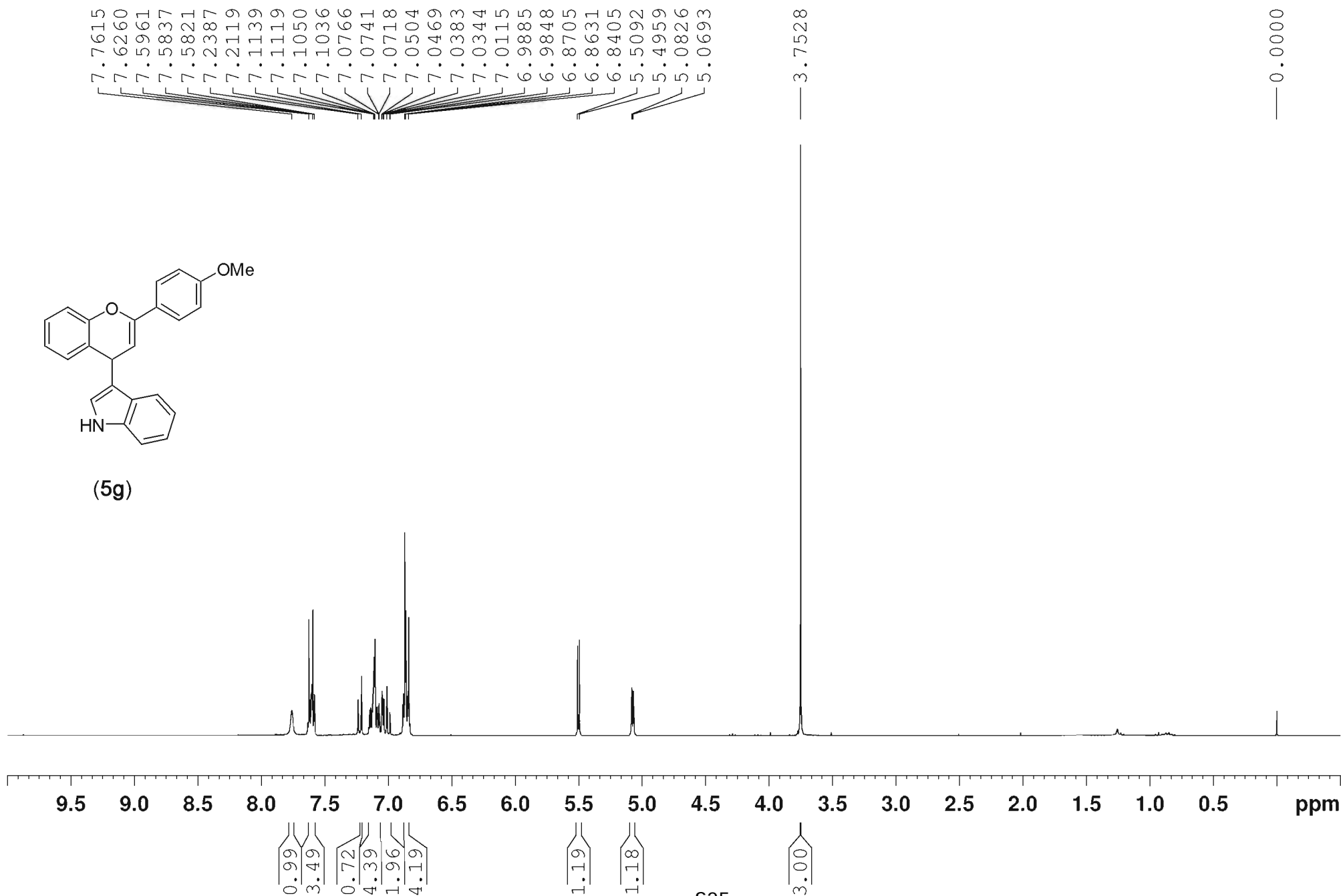
(5e)



^1H NMR (300MHz, CDCl_3)



(5g)



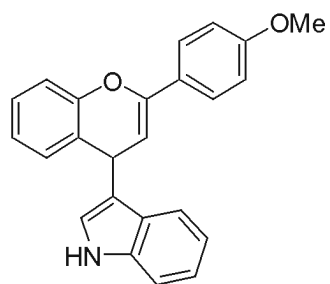
^{13}C NMR (75 MHz, CDCl_3)

159.6913
151.0862
147.4475
136.5252
129.4269
127.4004
127.0272
126.2380
126.0100
123.4696
123.2347
122.0214
121.9835
121.5057
119.4756
119.2984
116.3845
113.6192
111.2004
99.3423

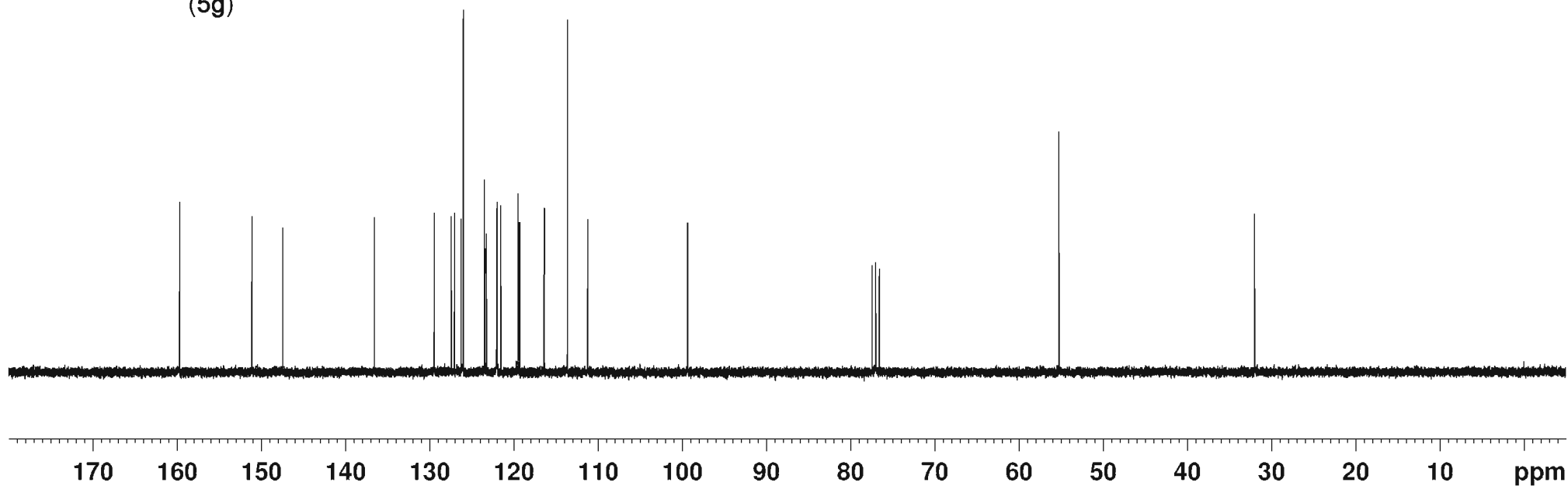
77.4177
76.9934
76.5697

55.2199

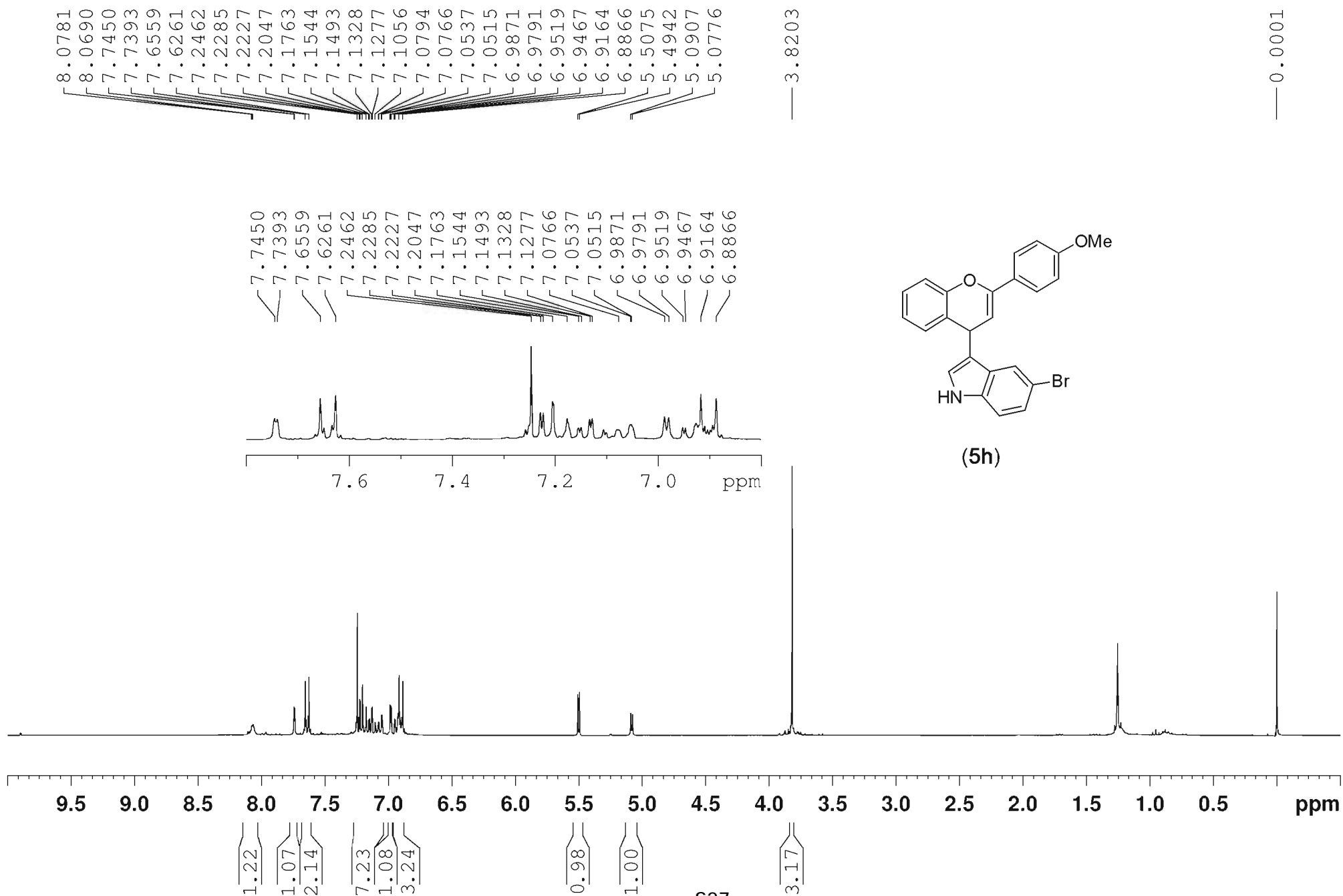
31.9783



(5g)



^1H NMR (300MHz, CDCl_3)



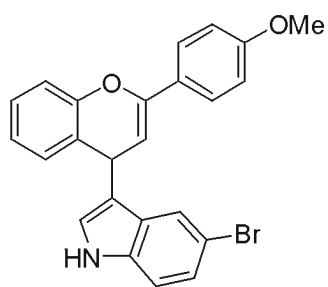
^{13}C NMR (75MHz, CDCl_3)

159.8529
151.1542
147.8435
135.1823
129.2662
128.0710
127.6265
126.9877
126.1065
125.0085
123.4580
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123.1107
121.8174
121.5905
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113.6989
112.9065
112.6663
98.9113

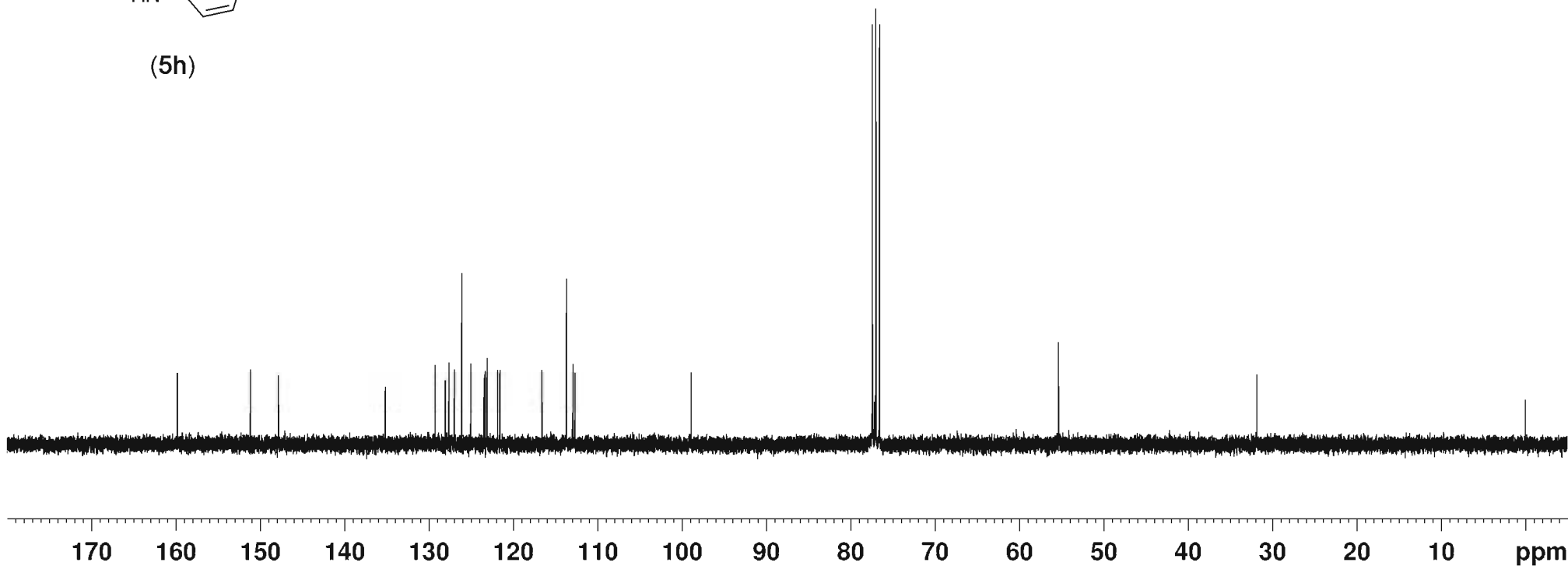
77.4223
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76.5758

55.3150

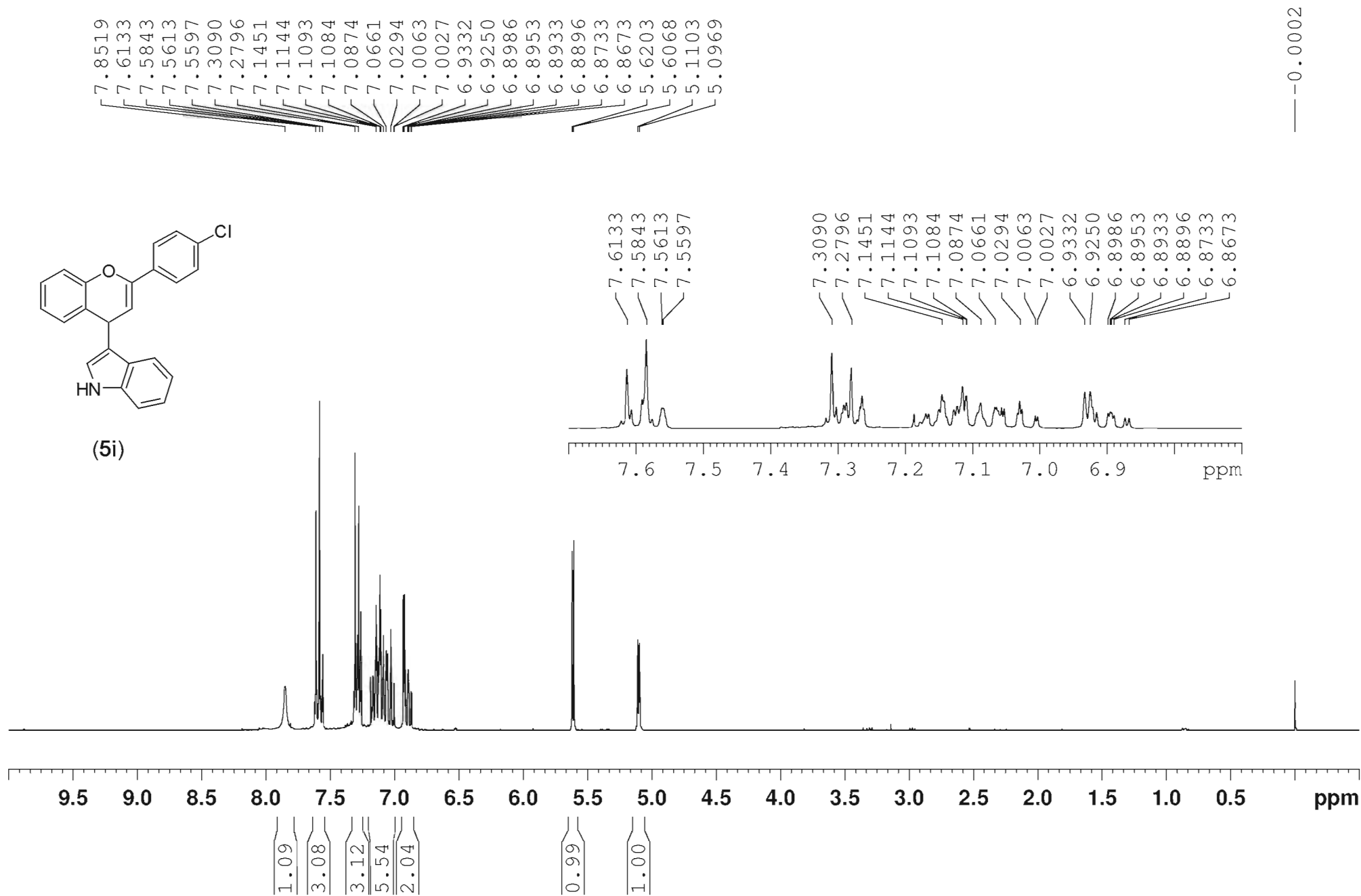
31.8282



(5h)



^1H NMR (300MHz, CDCl_3)

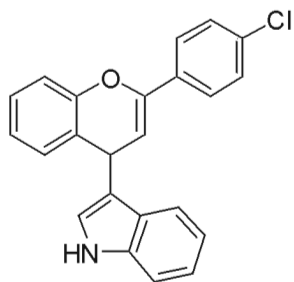


^{13}C NMR (75MHz, CDCl_3)

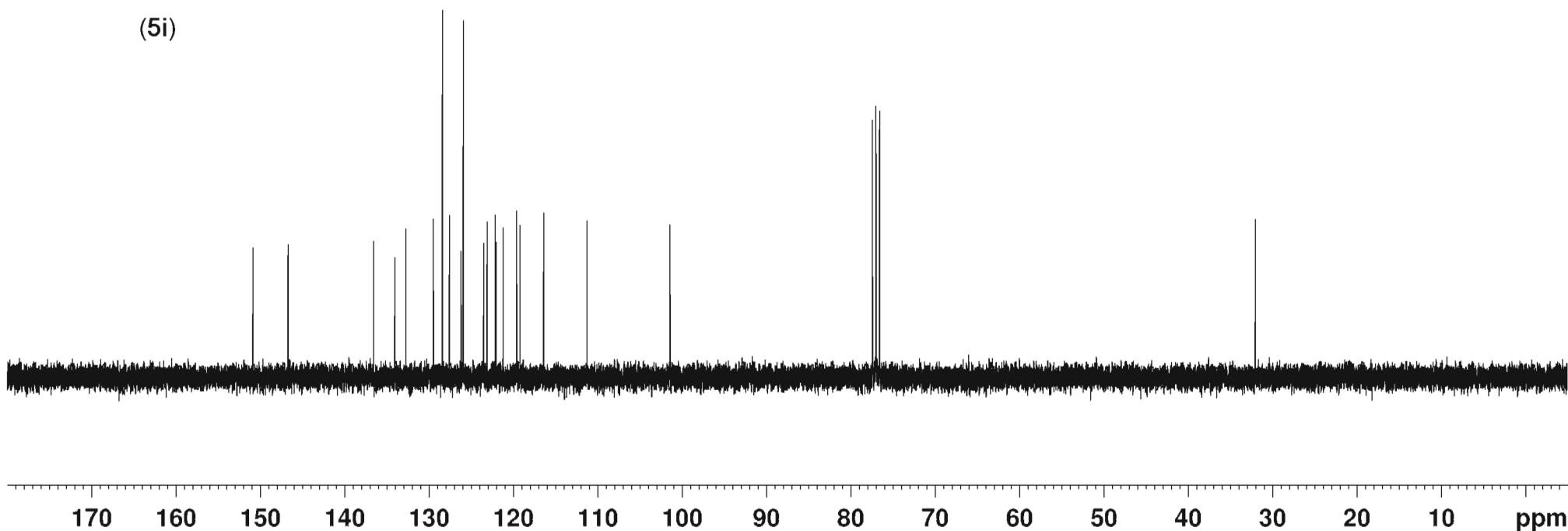
150.8683
146.7054
136.5410
134.0328
132.7369
129.4671
128.4006
127.5699
126.1742
125.9133
123.5003
123.1077
122.1285
122.0387
121.1840
119.6041
119.2069
116.3932
111.2468
101.4113

77.4141
76.9906
76.5682

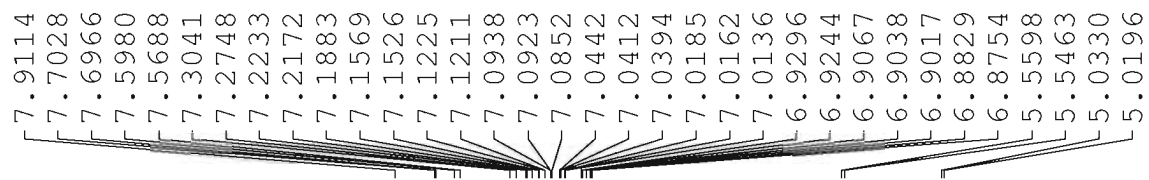
32.0047



(5i)

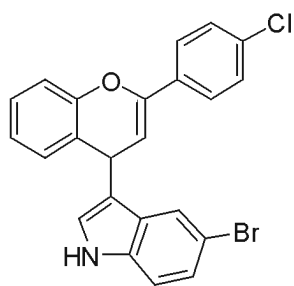


^1H NMR (300MHz, CDCl_3)

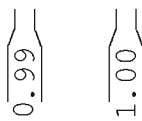
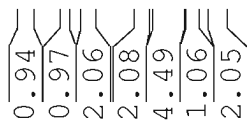
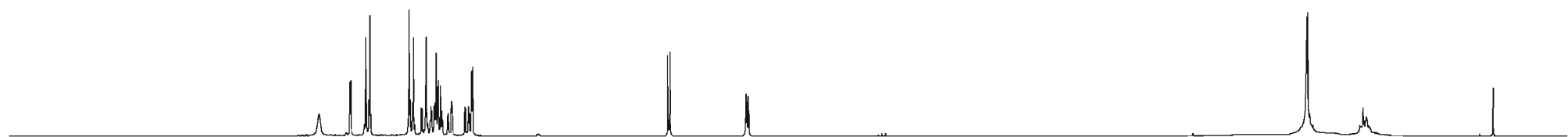
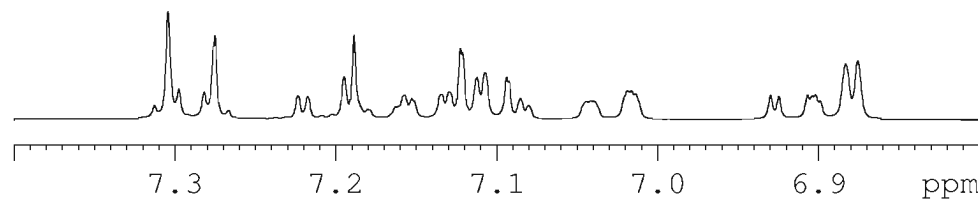
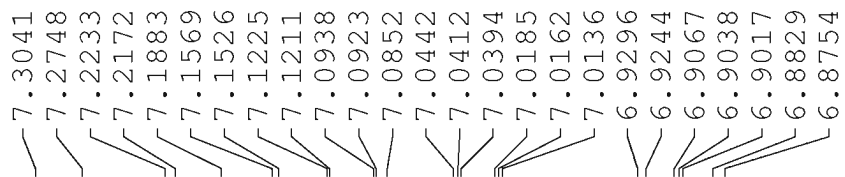


— 1.2524

— -0.0001



(5j)

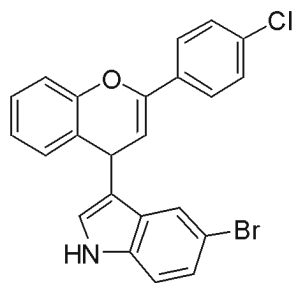


^{13}C NMR (75MHz, CDCl_3)

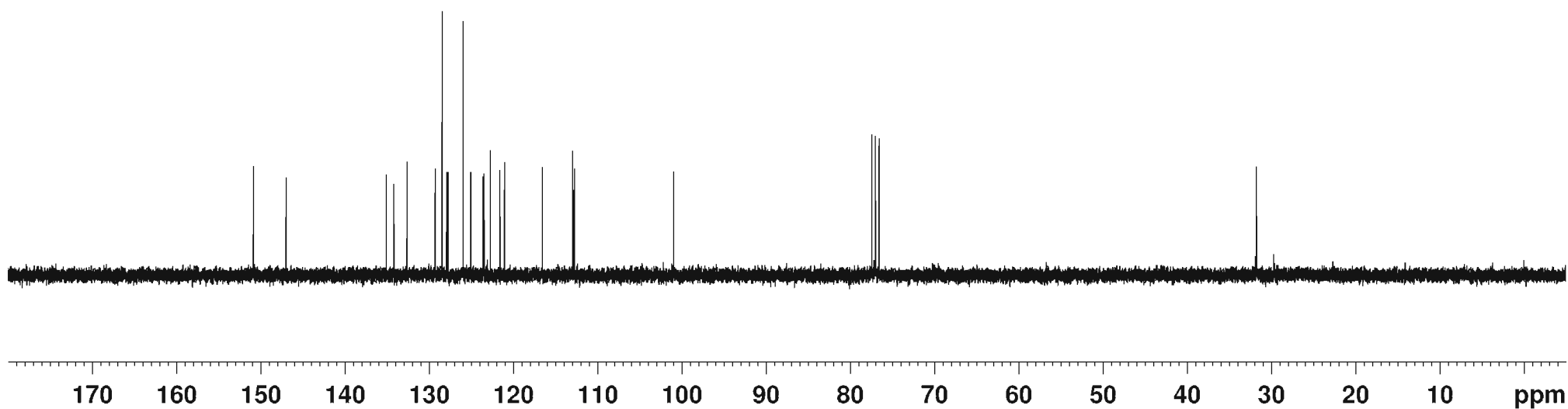
150.8410
146.9827
135.0855
134.1630
132.6034
129.2740
128.4388
127.9057
127.7629
125.9354
125.0355
123.5992
123.4676
122.7407
121.6150
121.0389
116.5553
112.9320
112.7217
100.9449

77.4242
77.0007
76.5764

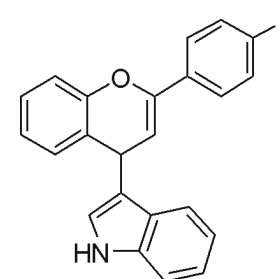
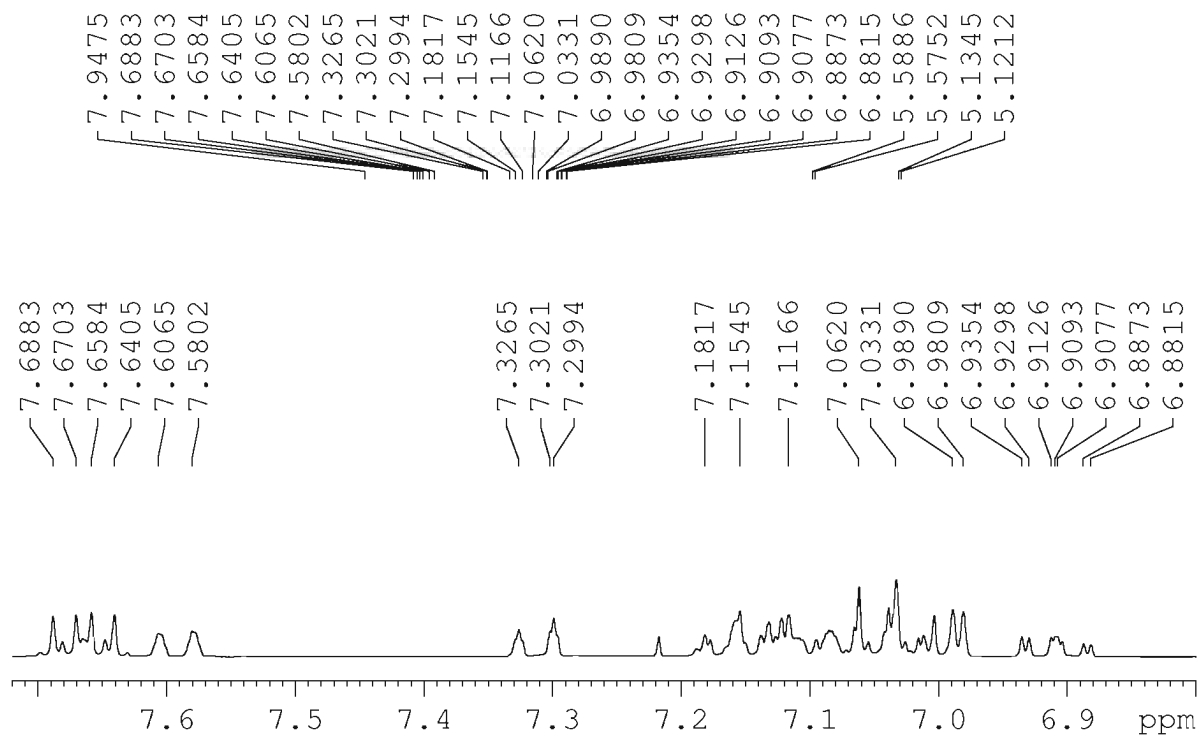
31.7502



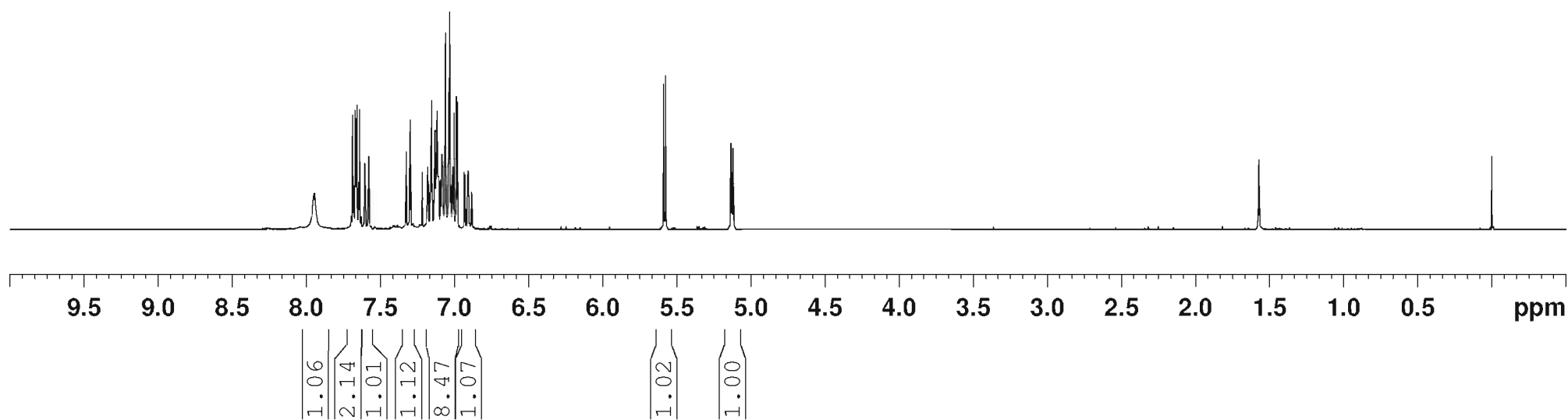
(5j)



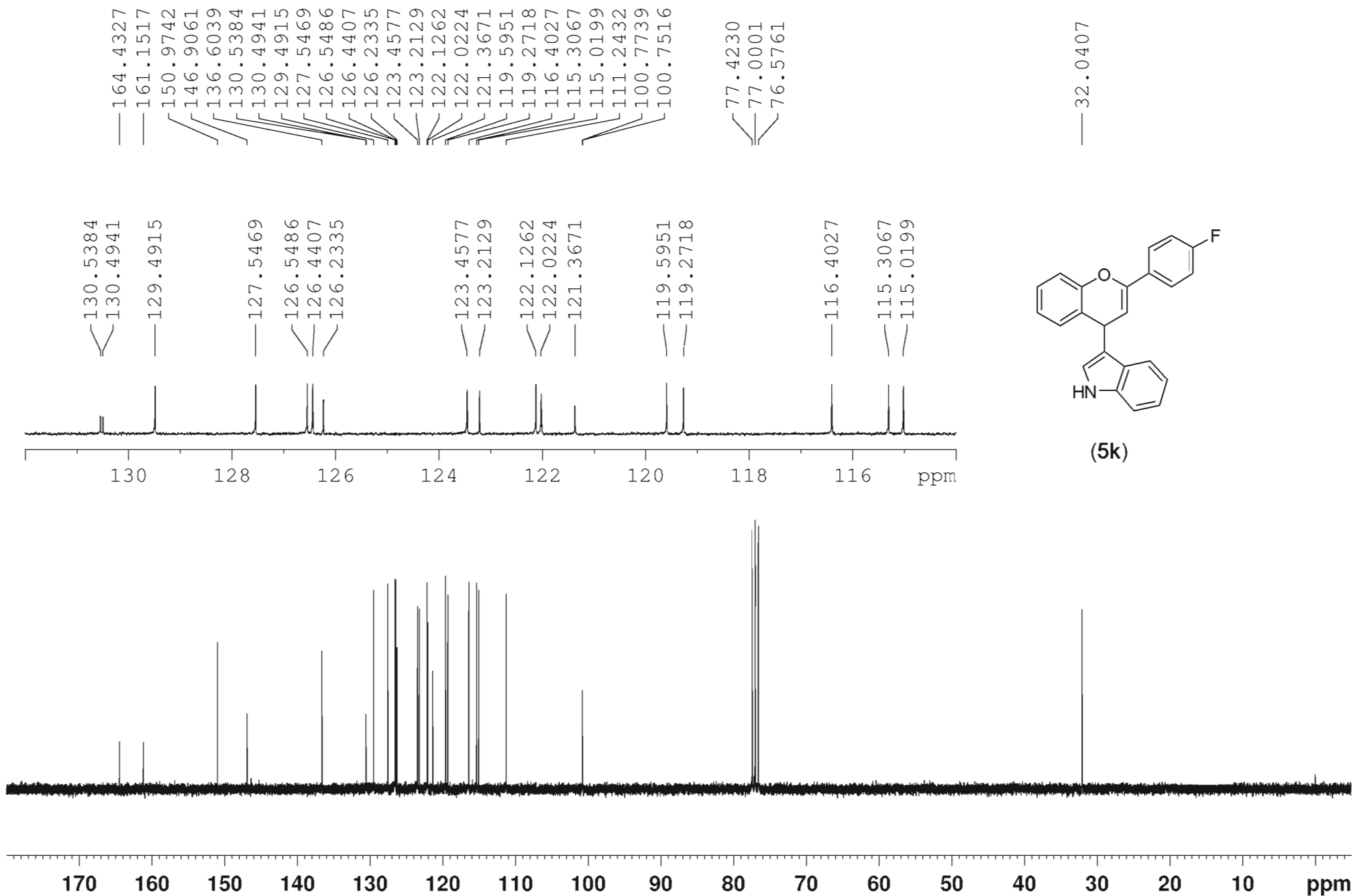
^1H NMR (300MHz, CDCl_3)



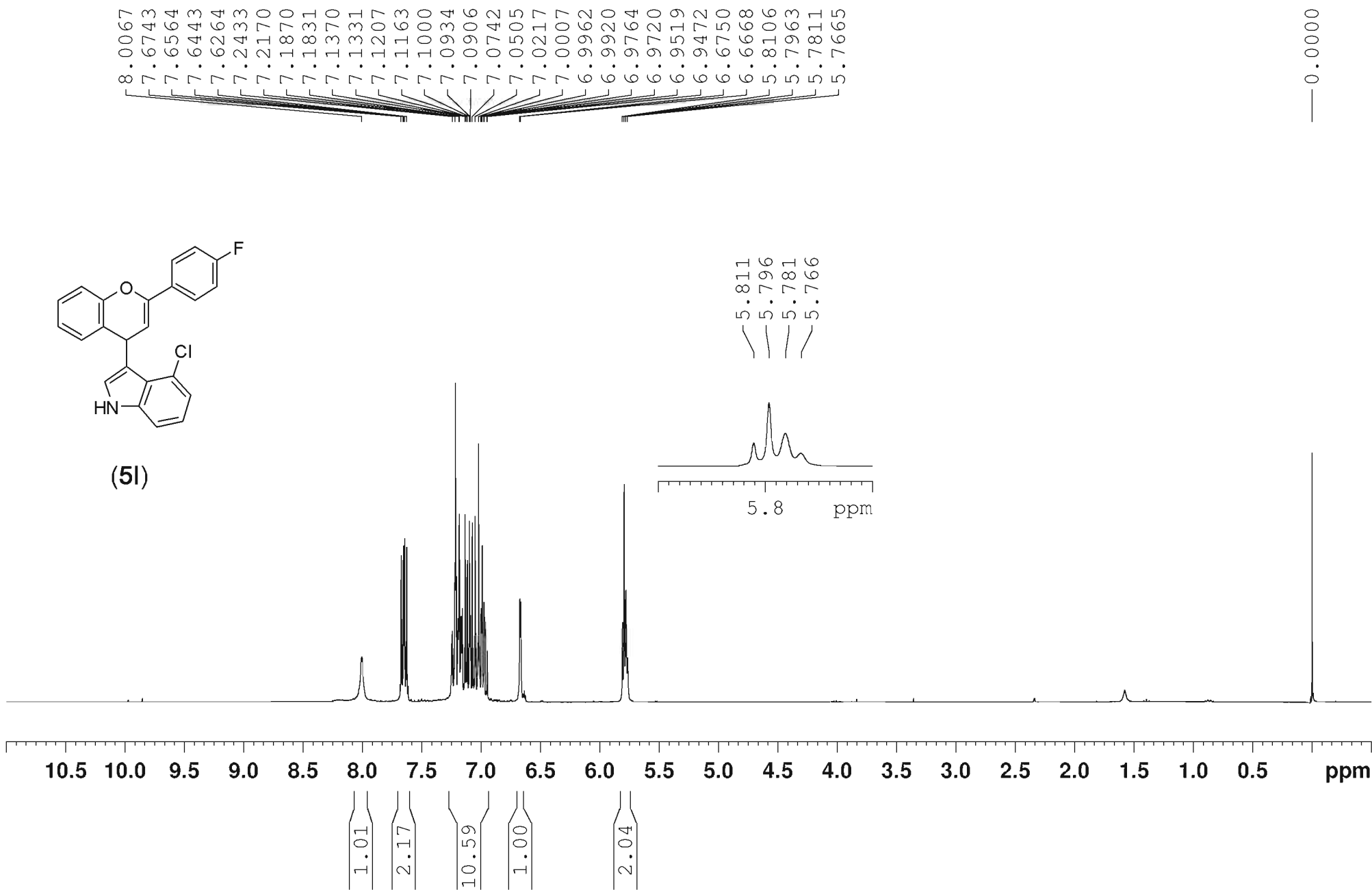
(5k)



^{13}C NMR (75 MHz, CDCl_3)



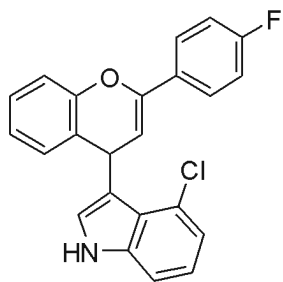
^1H NMR (300MHz, CDCl_3)



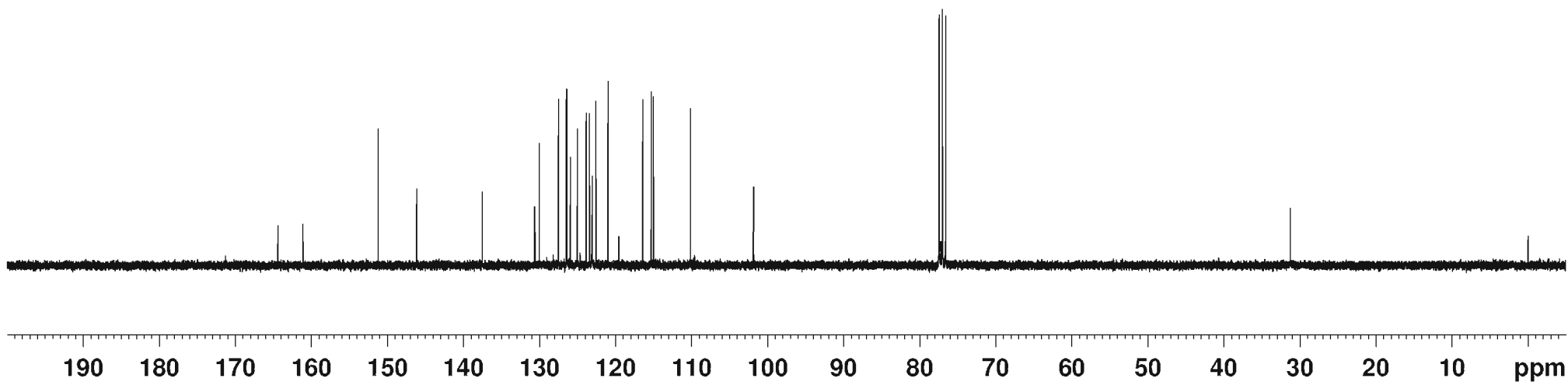
^{13}C NMR (75MHz, CDCl_3)

164.3846
161.1050
151.2231
146.1451
137.5308
130.6162
130.5737
130.0032
127.5075
126.4680
126.3601
125.9220
125.0060
123.8702
123.4029
123.0455
122.5765
120.9879
119.5353
116.4136
115.2799
114.9931
110.1573
101.8580
101.8354
77.4227
76.9997
76.5766

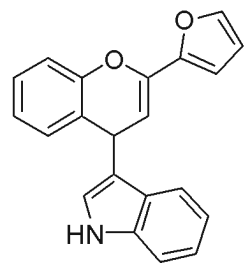
— 31.2555



(5I)

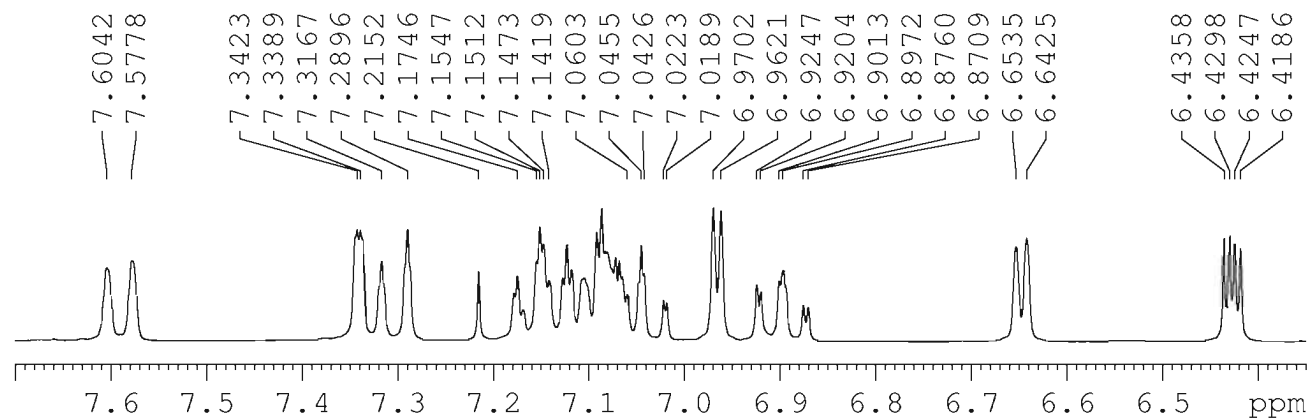


¹H NMR (300MHz, CDCl₃)

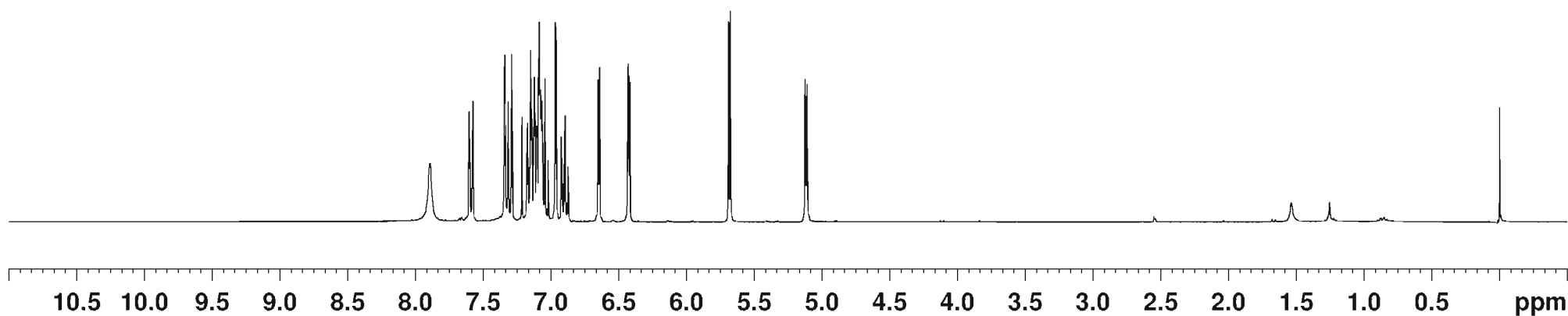


(5m)

7.8937
7.6042
7.5778
7.3423
7.3389
7.3167
7.2896
7.2152
7.1746
7.1547
7.1512
7.1473
7.1419
7.0603
7.0455
7.0426
7.0223
7.0189
6.9702
6.9621
6.9247
6.9204
6.9013
6.8972
6.8760
6.8709
6.6535
6.6425
6.4358
6.4298
6.4247
6.4186
5.6898
5.6762
5.1245
5.1110



— 0.0000

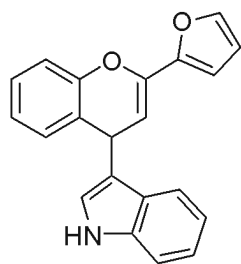


^{13}C NMR (75MHz, CDCl_3)

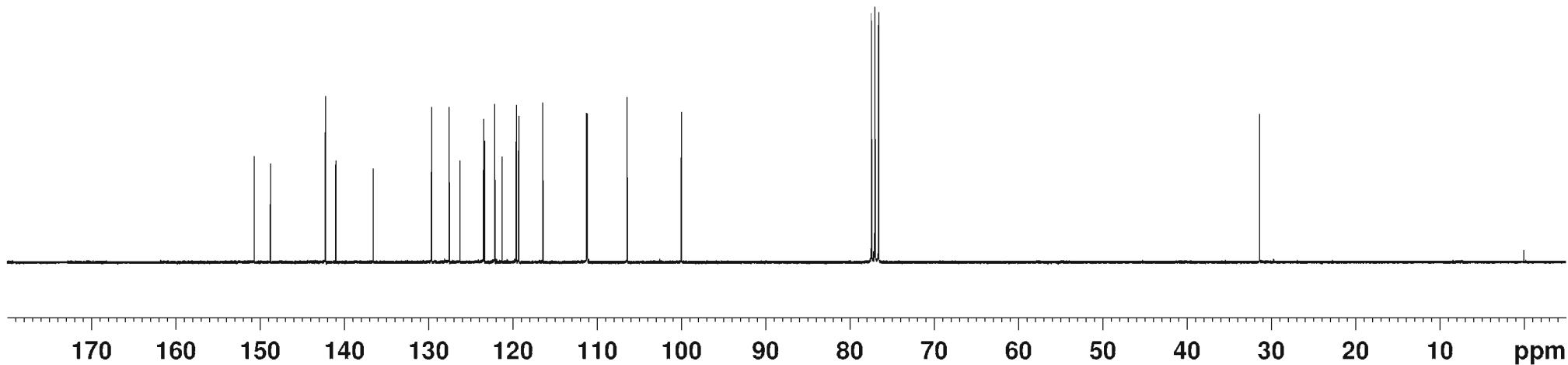
150.6757
148.7455
142.2269
140.9936
136.5708
129.6208
127.5418
126.2474
123.4548
123.3323
122.1199
122.0961
121.2498
119.5752
119.2777
116.4130
111.2085
111.1317
106.4053
99.9746

77.4241
77.0008
76.5772

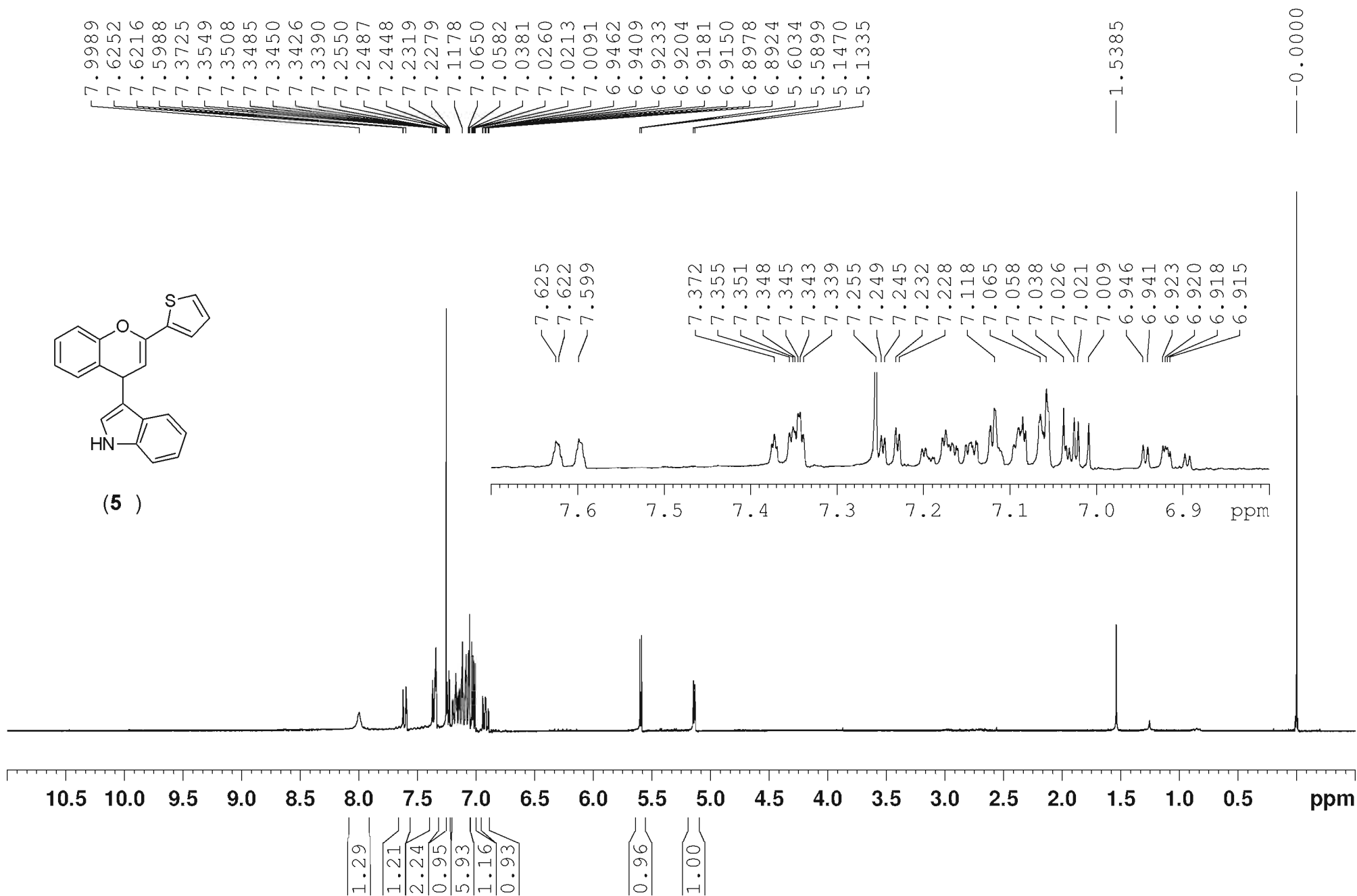
31.3717



(5m)



^1H NMR (300 MHz, CDCl_3)



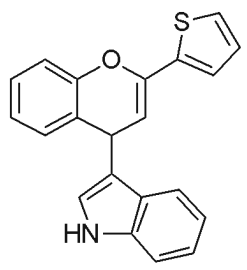
^{13}C NMR (75 MHz, CDCl_3)

150.8420
143.8621
138.2001
136.5860
129.4782
127.5381
127.2487
126.2712
124.7423
123.4829
123.4553
123.2182
122.1547
122.1118
121.2635
119.6464
119.3500
116.4470
111.1886
100.3547

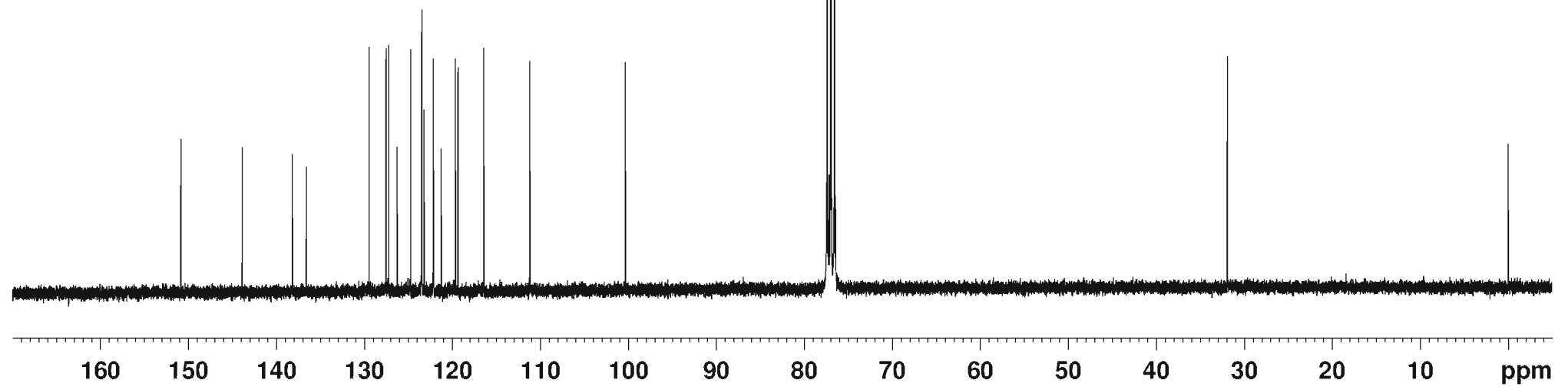
77.4072
76.9838
76.5605

31.9145

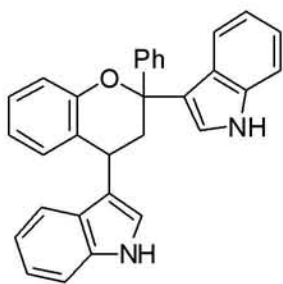
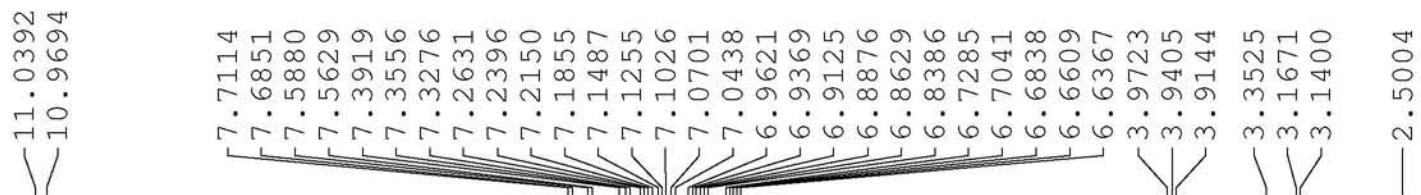
0.0297



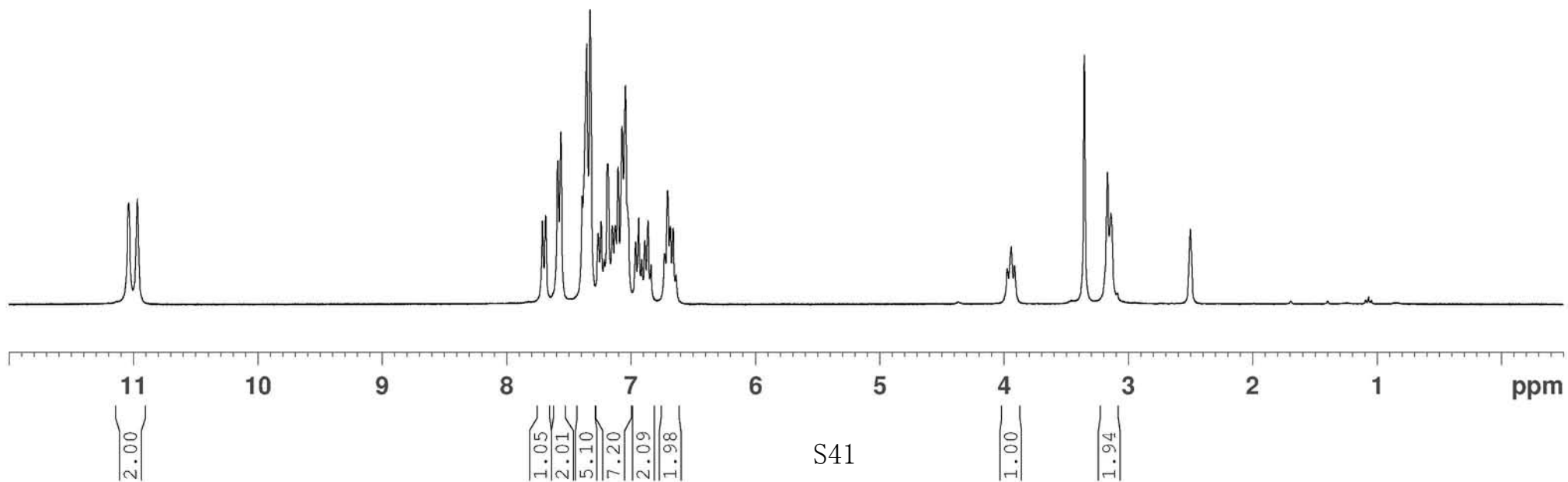
(5n)



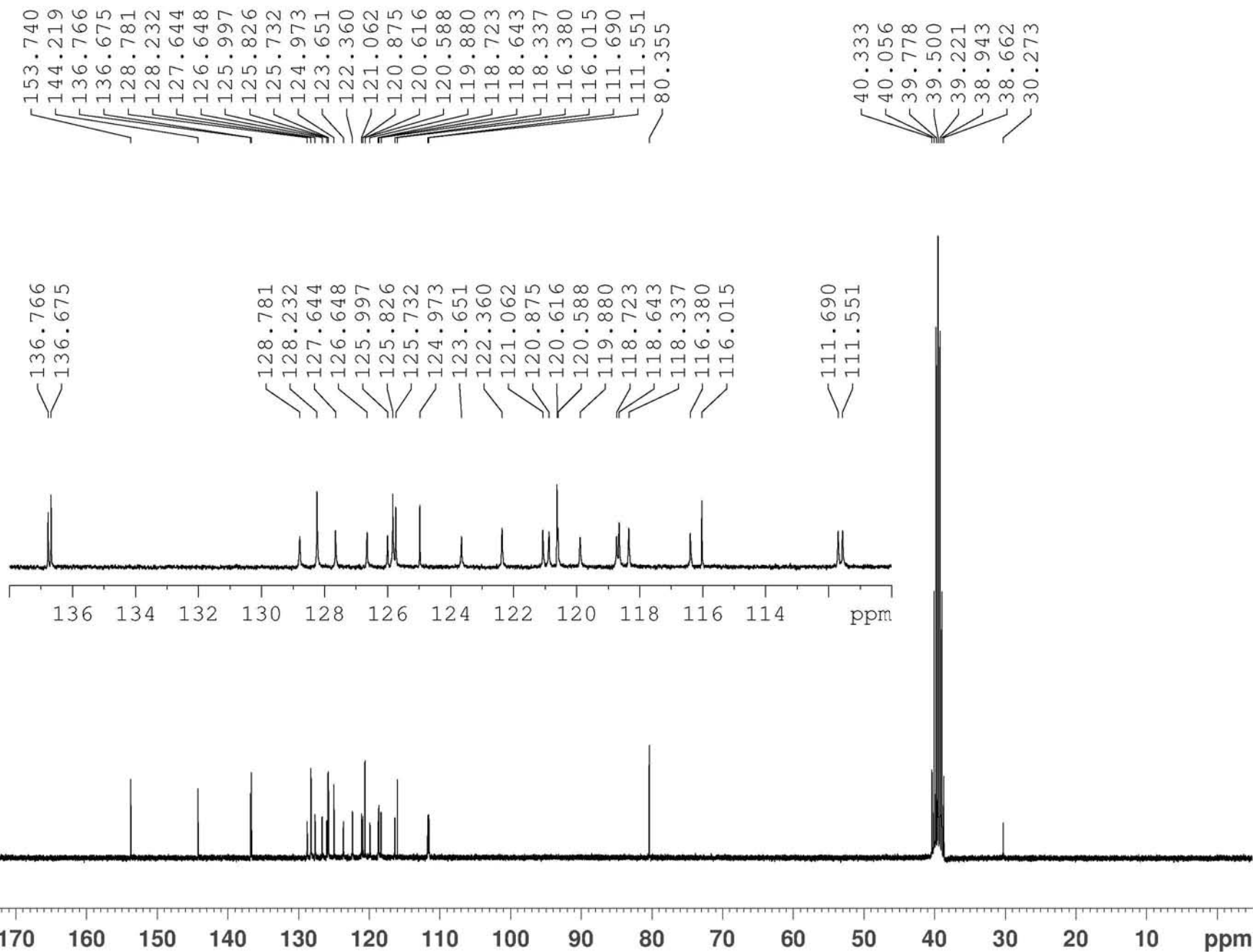
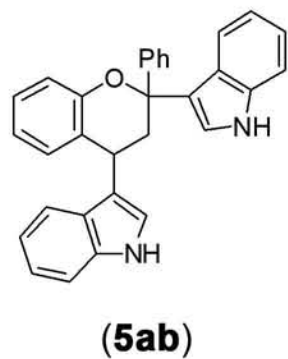
^1H NMR (300 MHz, DMSO-d_6)



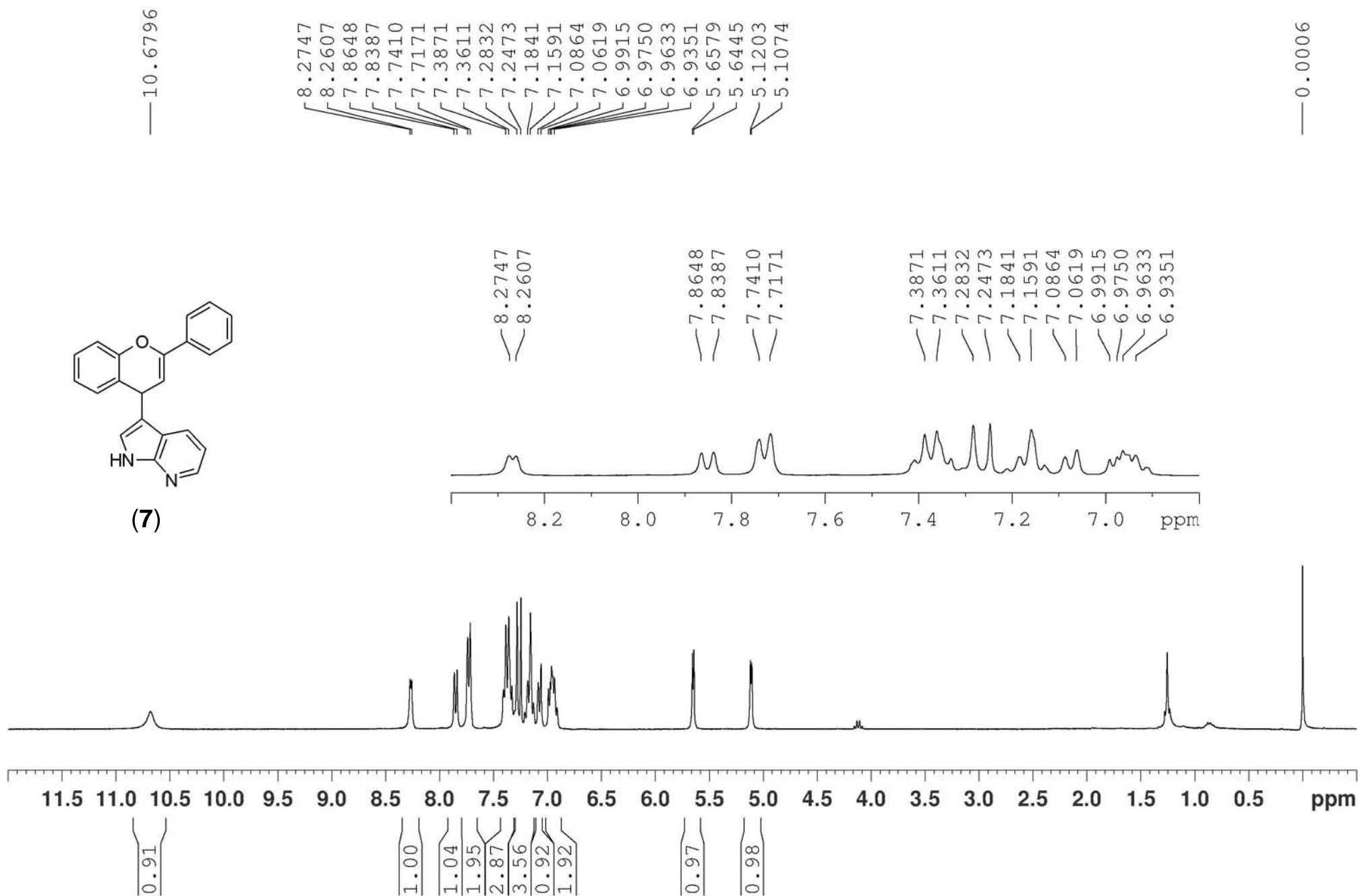
(5ab)



^{13}C NMR (75 MHz, DMSO- d_6)



^1H NMR (300MHz, CDCl_3)

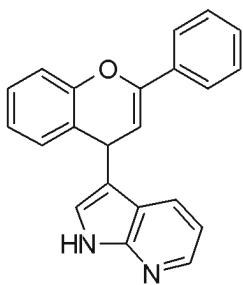


^{13}C NMR (75MHz, CDCl_3)

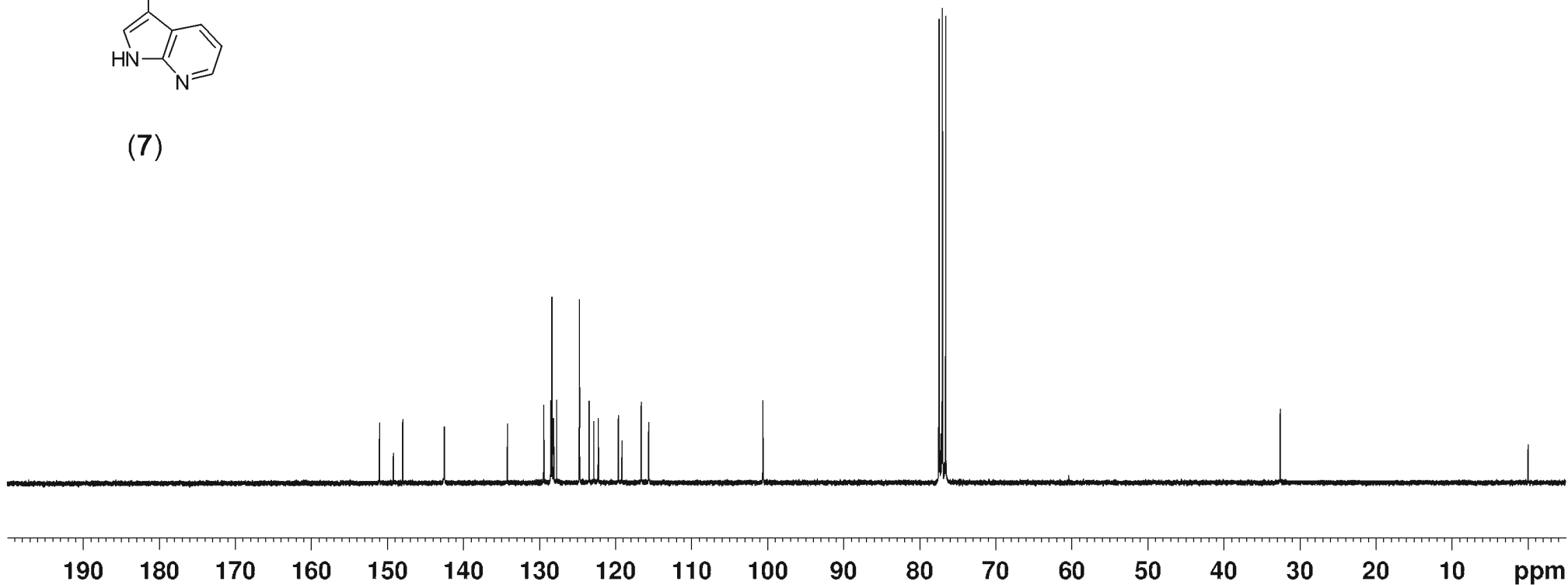
151.0329
149.1920
147.9783
142.5150
134.1930
129.4326
128.4969
128.3345
128.1334
127.7176
124.7197
123.4785
122.8517
122.2762
119.6143
119.1315
116.5952
115.6119
100.5830

77.4243
77.0005
76.5769

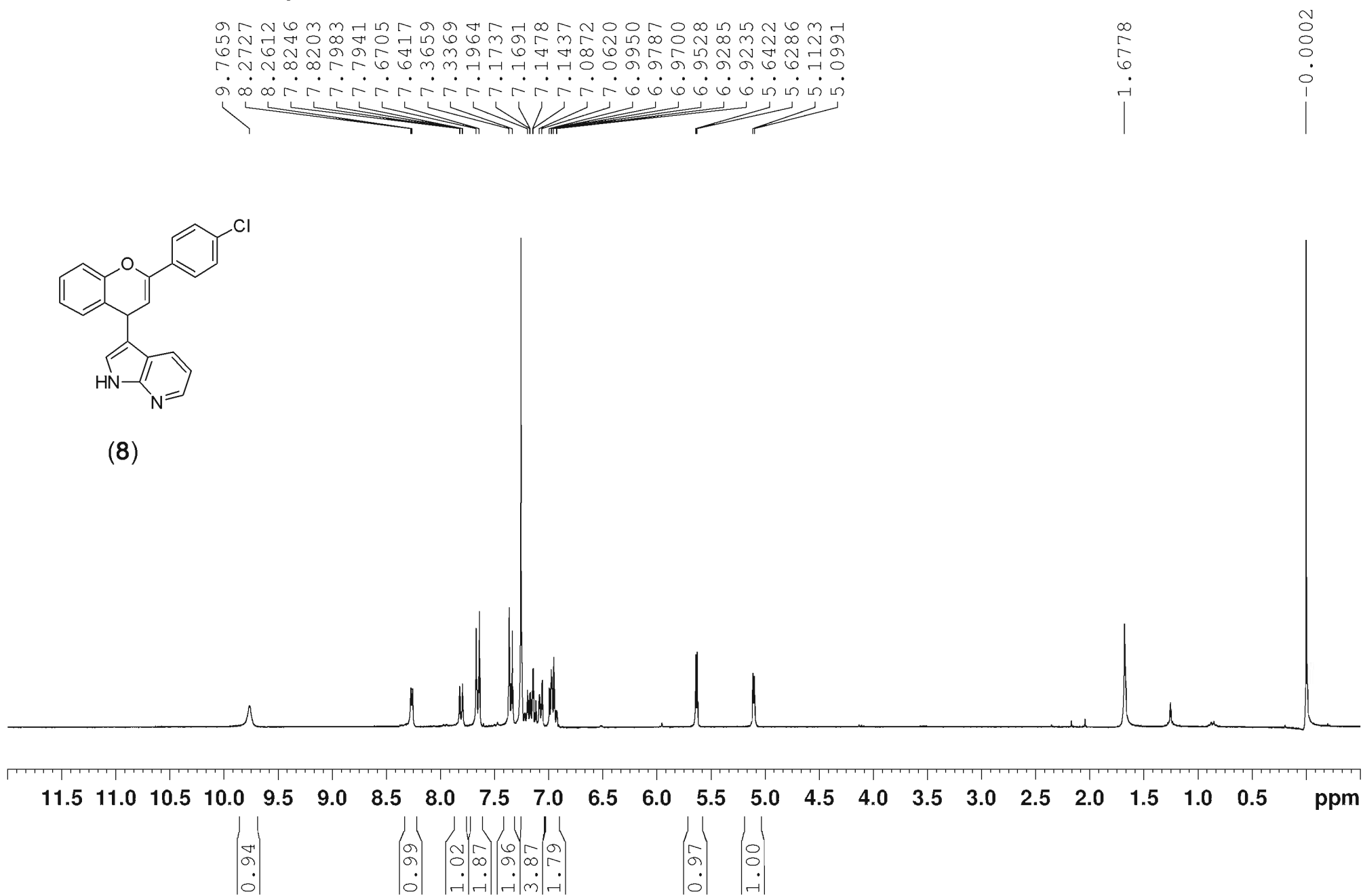
32.5830



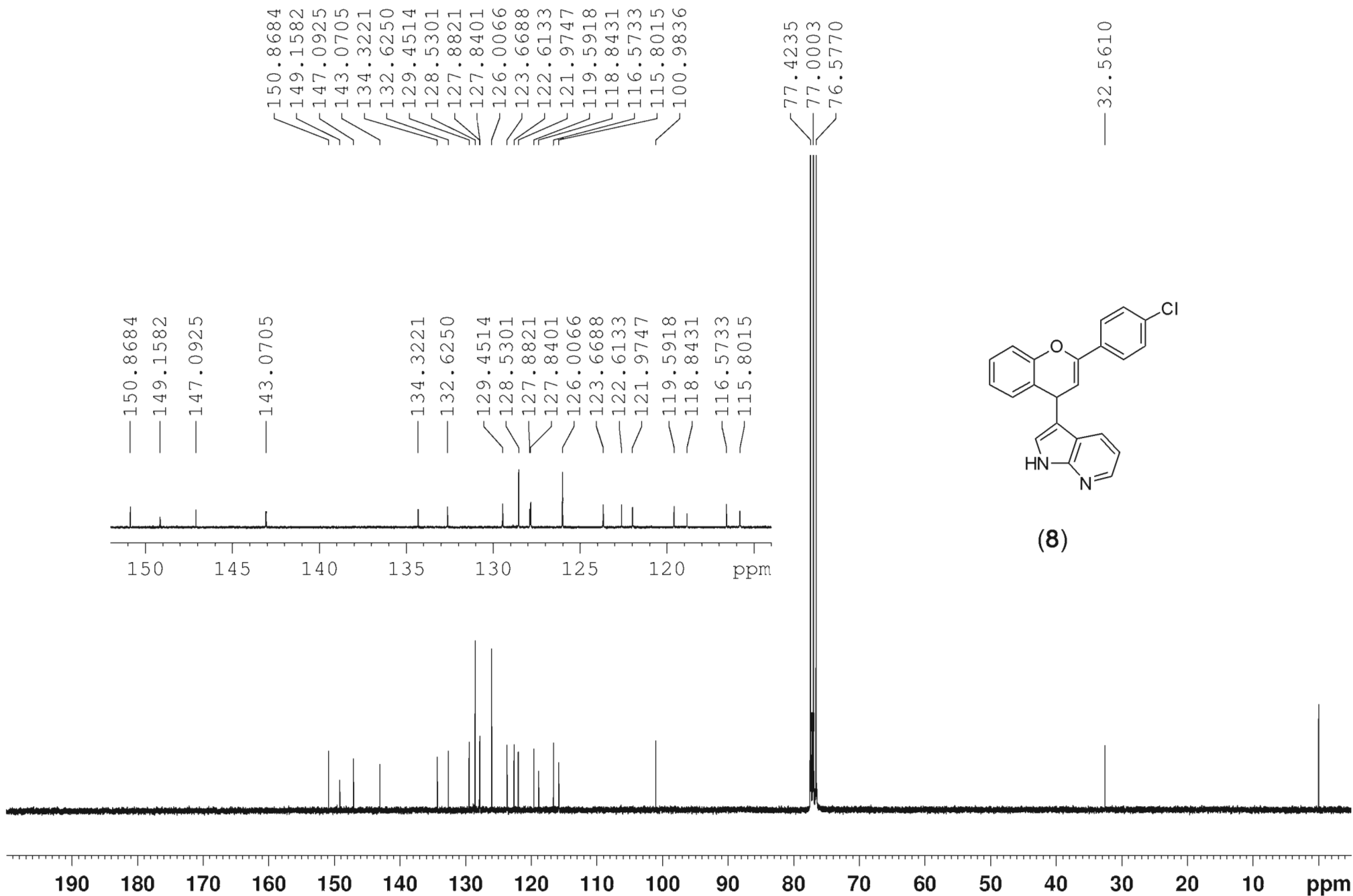
(7)



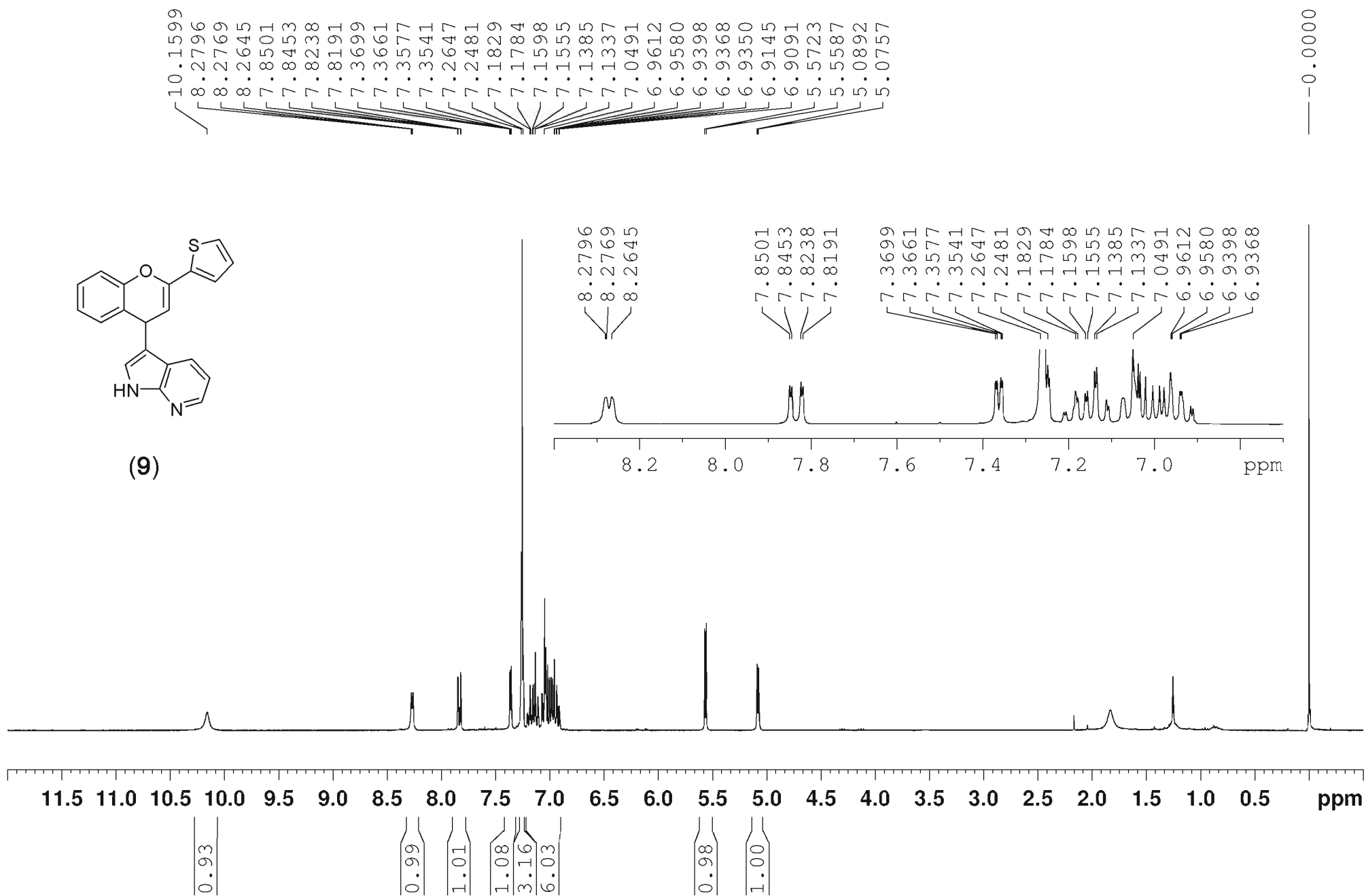
^1H NMR (300MHz, CDCl_3)



^{13}C NMR (75MHz, CDCl_3)



^1H NMR (300MHz, CDCl_3)

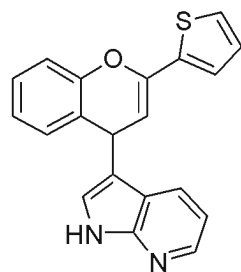


^{13}C NMR (75MHz, CDCl_3)

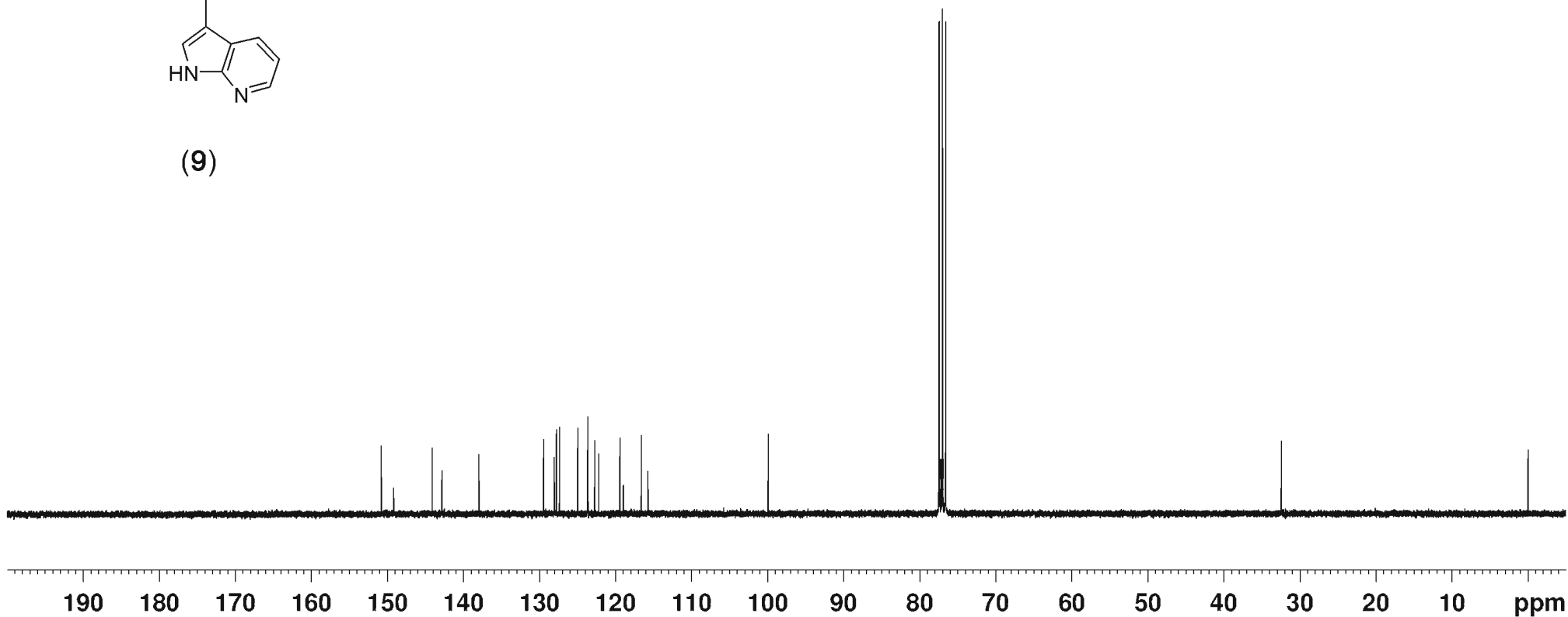
150.7895
149.1636
144.1166
142.8168
137.9716
129.4480
128.0276
127.7759
127.3298
124.9603
123.6537
123.6212
122.7168
122.1805
119.4194
118.9781
116.5831
115.7378
99.9007

77.4230
76.9998
76.5765

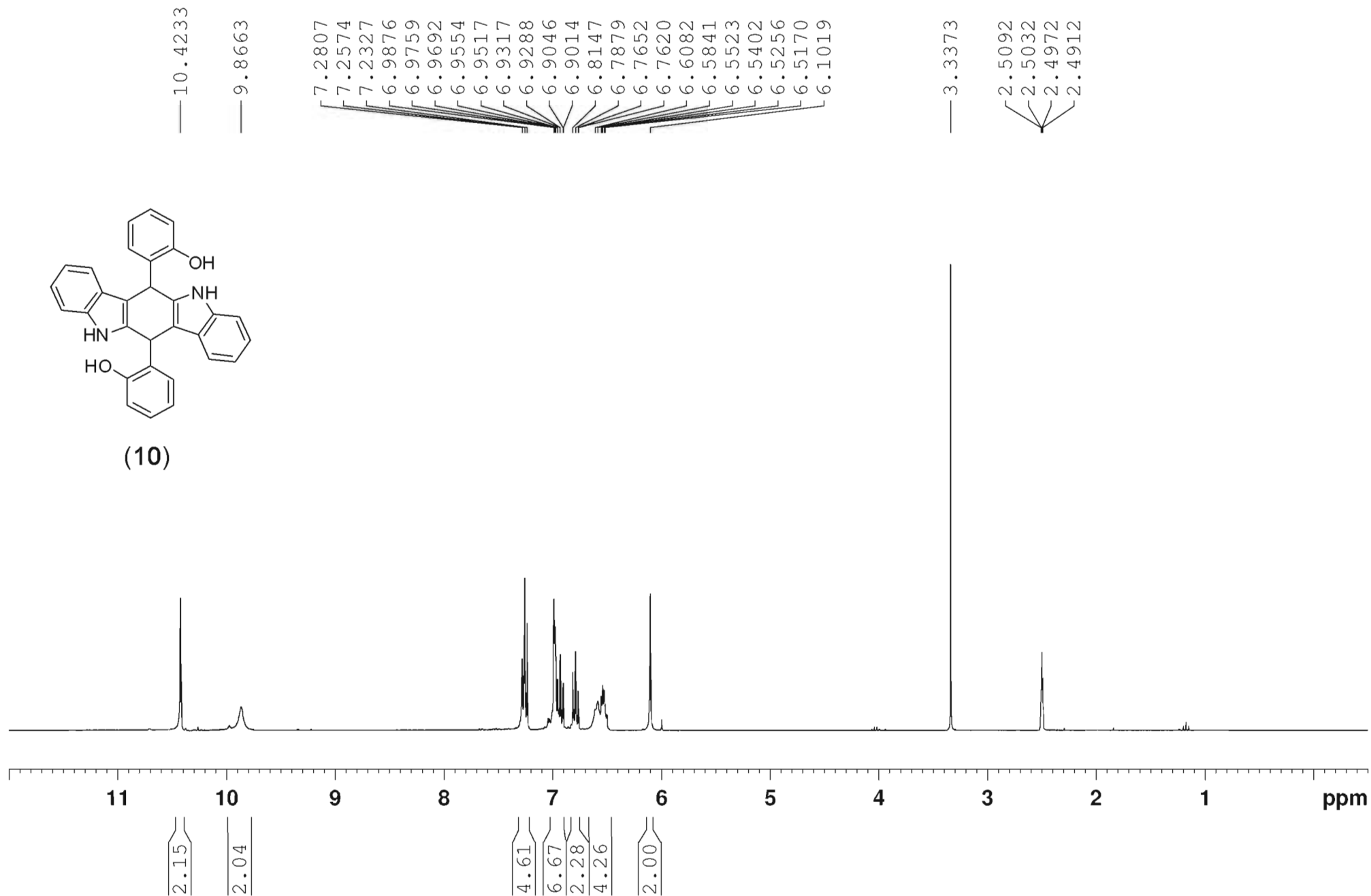
32.4163



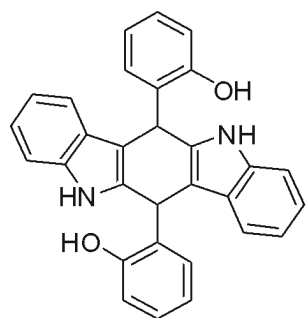
(9)



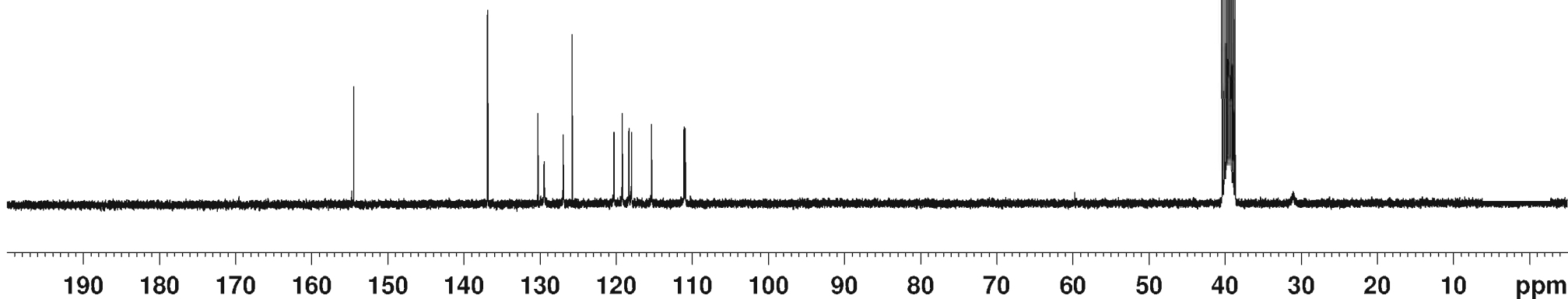
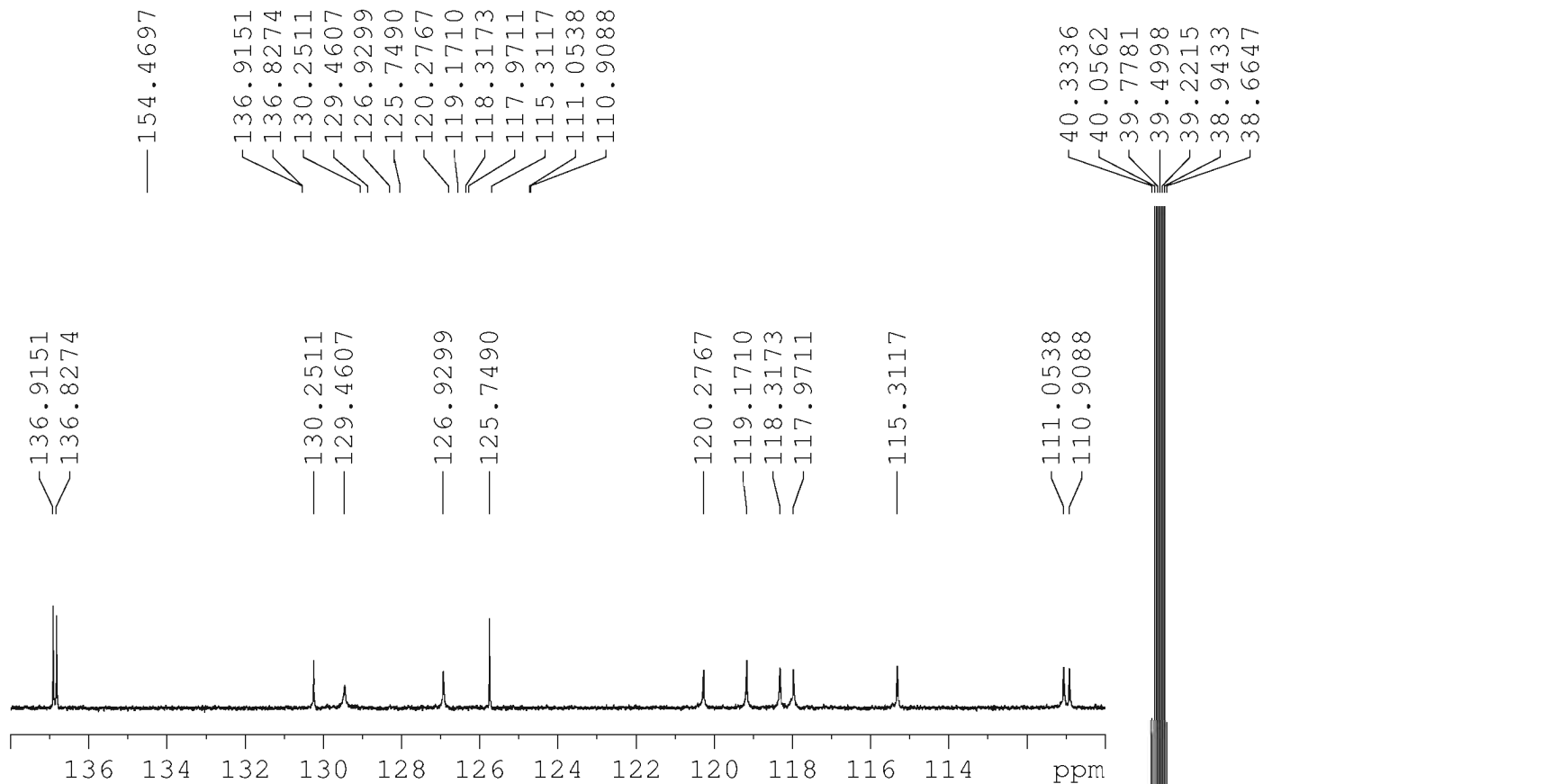
^1H NMR (300MHz, DMSO- d_6)



^{13}C NMR (75MHz, DMSO- d_6)



(10)



Mass Spectrum SmartFormula Report

Analysis Info

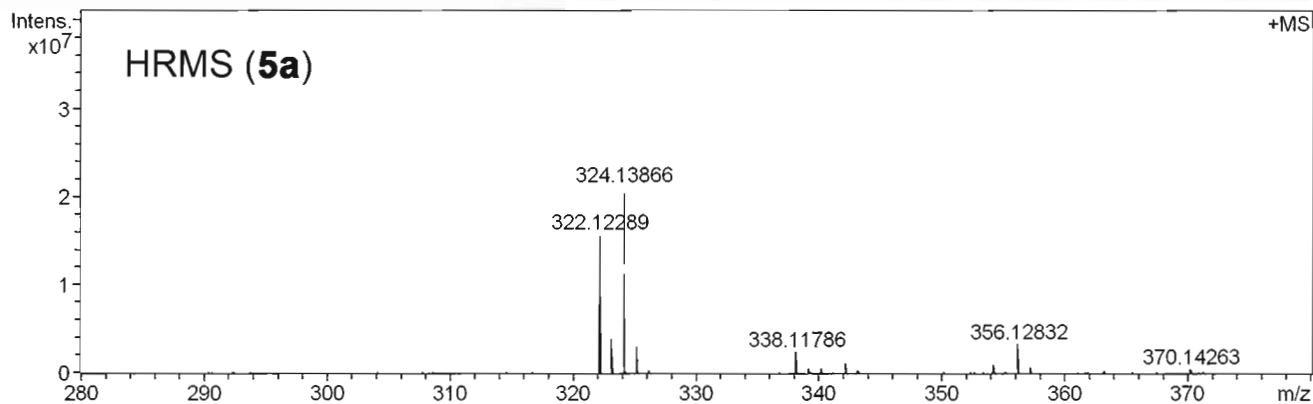
Analysis Name D:\20120619\YIN_1_APCI_POS_000002.d
Method Metal_Trypsin digestion
Sample Name YIN_1_APCI_POS
Comment

Acquisition Date 2012-6-19 19:00:33

Operator
Instrument apex-Ultra

Acquisition Parameter

Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	2	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	600.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Wed Jan 4 05:51:21 2012
Source Accumulation	0.0 sec	Drying Gas Flow Rate	4.0 L/min	Data Acquisition Size	131072
Ion Accumulation Time	0.0 sec	Nebulizer Gas Flow Rate	1.0 L/min	Apodization	Sine-Bell Multiplication
Flight Time to Acq. Cell	0.0 sec				



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e ₁	Conf	N-Rule
324.13866	1	C ₂₃ H ₁₈ N ₂ O	100.00	324.13829	-0.4	-1.1	11.8	15.5		even	ok

Mass Spectrum SmartFormula Report

Analysis Info

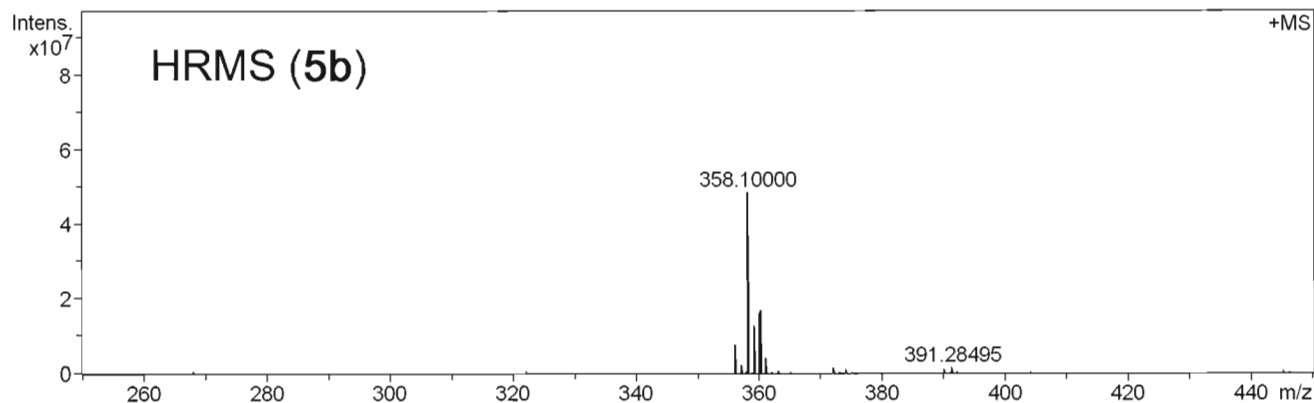
Analysis Name D:\20120619\YIN_2_APCI_POS_000002.d
Method Metal_Trypsin digestion
Sample Name YIN_2_APCI_POS
Comment

Acquisition Date 2012-6-19 19:01:42

Operator
Instrument apex-Ultra

Acquisition Parameter

Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	2	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	600.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Wed Jan 4 05:51:21 2012
Source Accumulation	0.0 sec	Drying Gas Flow Rate	4.0 L/min	Data Acquisition Size	131072
Ion Accumulation Time	0.0 sec	Nebulizer Gas Flow Rate	1.0 L/min	Apodization	Sine-Bell Multiplication
Flight Time to Acq. Cell	0.0 sec				



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	ej	Conf	N-Rule
358.10000	1	C ₂₃ H ₁₇ ClN ₂ O	100.00	358.09932	-0.7	-1.9	3.6	15.5		even	ok

Mass Spectrum SmartFormula Report

Analysis Info

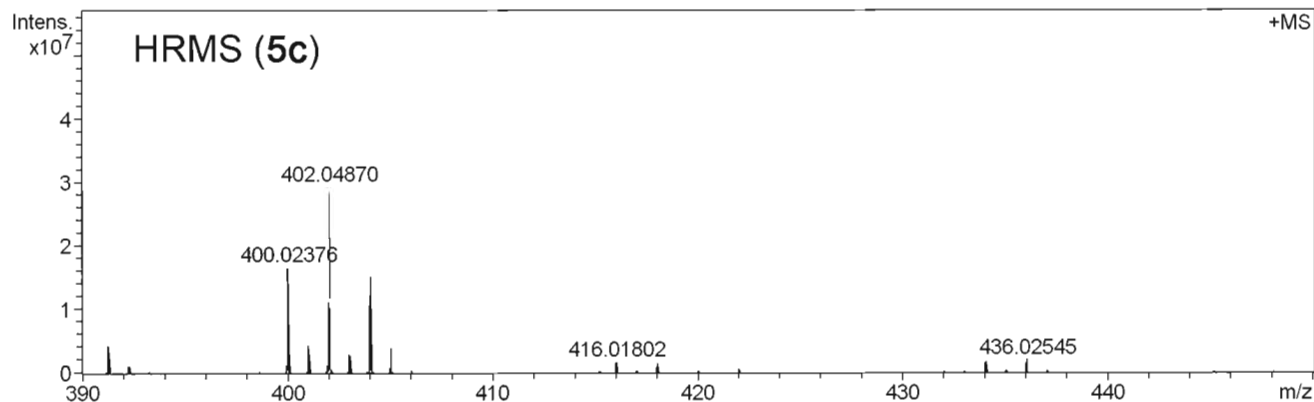
Analysis Name D:\20120619\YIN_3_APCI_POS_000002.d
Method Metal_Trypsin digestion
Sample Name YIN_3_APCI_POS
Comment

Acquisition Date 2012-6-19 19:02:53

Operator
Instrument apex-Ultra

Acquisition Parameter

Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	2	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	600.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Wed Jan 4 05:51:21 2012
Source Accumulation	0.0 sec	Drying Gas Flow Rate	4.0 L/min	Data Acquisition Size	131072
Ion Accumulation Time	0.0 sec	Nebulizer Gas Flow Rate	1.0 L/min	Apodization	Sine-Bell Multiplication
Flight Time to Acq. Cell	0.0 sec				



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e ⁻ Conf	N-Rule
402.01318	1	C ₂₂ H ₁₃ BrNO ₂	100.00	402.01242	-0.8	-1.9	459.8	16.5	even	ok
402.04870	1	C ₂₃ H ₁₇ BrNO	100.00	402.04880	0.1	0.3	148.3	15.5	even	ok

Mass Spectrum SmartFormula Report

Analysis Info

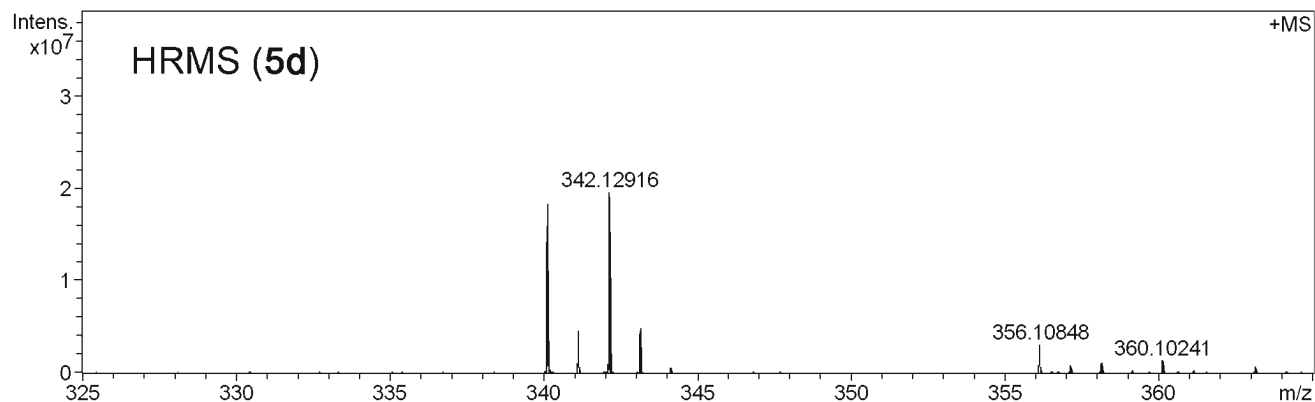
Analysis Name D:\20120619\YIN_4_APCI_POS_000002.d
Method Metal_Trypsin digestion
Sample Name YIN_4_APCI_POS
Comment

Acquisition Date 2012-6-19 19:04:08

Operator
Instrument apex-Ultra

Acquisition Parameter

Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	2	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	600.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Wed Jan 4 05:51:21 2012
Source Accumulation	0.0 sec	Drying Gas Flow Rate	4.0 L/min	Data Acquisition Size	131072
Ion Accumulation Time	0.0 sec	Nebulizer Gas Flow Rate	1.0 L/min	Apodization	Sine-Bell Multiplication
Flight Time to Acq. Cell	0.0 sec				



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e ⁻	Conf	N-Rule
342.12916	1	C ₂₃ H ₁₇ FNO	100.00	342.12887	-0.3	-0.9	0.8	15.5	even		ok

Mass Spectrum SmartFormula Report

Analysis Info

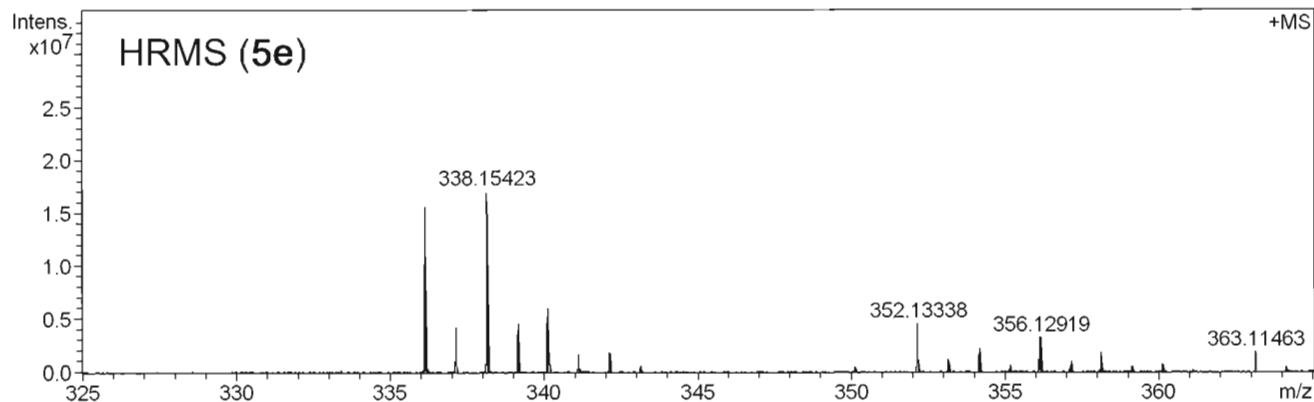
Analysis Name D:\20120619\YIN_5_APCI_POS_000002.d
Method Metal_Trypsin digestion
Sample Name YIN_5_APCI_POS
Comment

Acquisition Date 2012-6-19 19:05:11

Operator
Instrument apex-Ultra

Acquisition Parameter

Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	4	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	600.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Wed Jan 4 05:51:21 2012
Source Accumulation	0.0 sec	Drying Gas Flow Rate	4.0 L/min	Data Acquisition Size	131072
Ion Accumulation Time	0.0 sec	Nebulizer Gas Flow Rate	1.0 L/min	Apodization	Sine-Bell Multiplication
Flight Time to Acq. Cell	0.0 sec				



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e _i	Conf	N-Rule
338.15423	1	C ₂₄ H ₂₀ N ₂ O	100.00	338.15394	-0.3	-0.8	42.5	15.5		even	ok

Mass Spectrum SmartFormula Report

Analysis Info

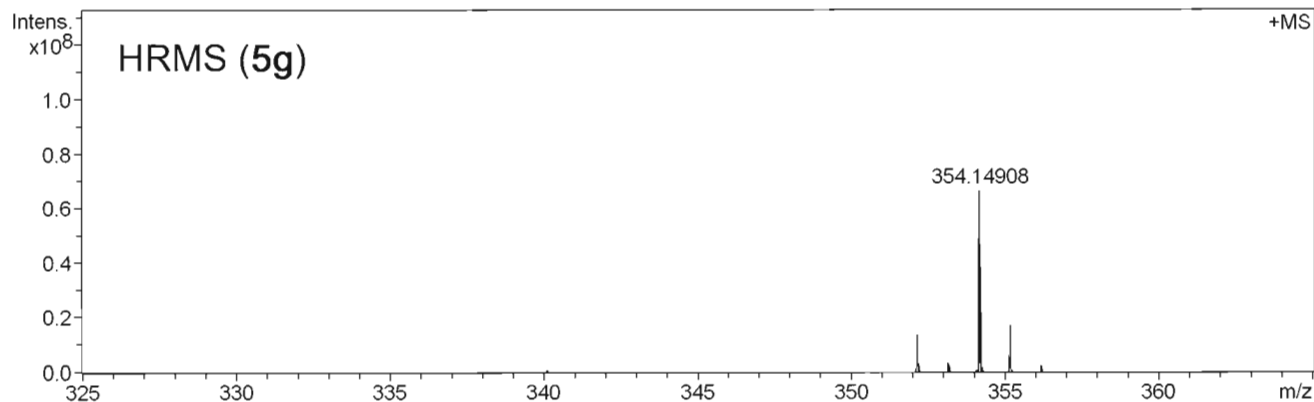
Analysis Name D:\20120619\YIN_6_APCI_POS_000002.d
Method Metal_Trypsin digestion
Sample Name YIN_6_APCI_POS
Comment

Acquisition Date 2012-6-19 19:06:18

Operator
Instrument apex-Ultra

Acquisition Parameter

Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	2	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	600.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Wed Jan 4 05:51:21 2012
Source Accumulation	0.0 sec	Drying Gas Flow Rate	4.0 L/min	Data Acquisition Size	131072
Ion Accumulation Time	0.0 sec	Nebulizer Gas Flow Rate	1.0 L/min	Apodization	Sine-Bell Multiplication
Flight Time to Acq. Cell	0.0 sec				



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	ej	Conf	N-Rule
354.14908	1	C ₂₄ H ₂₀ N ₂ O ₂	100.00	354.14886	-0.2	-0.6	5.7	15.5		even	ok

Mass Spectrum SmartFormula Report

Analysis Info

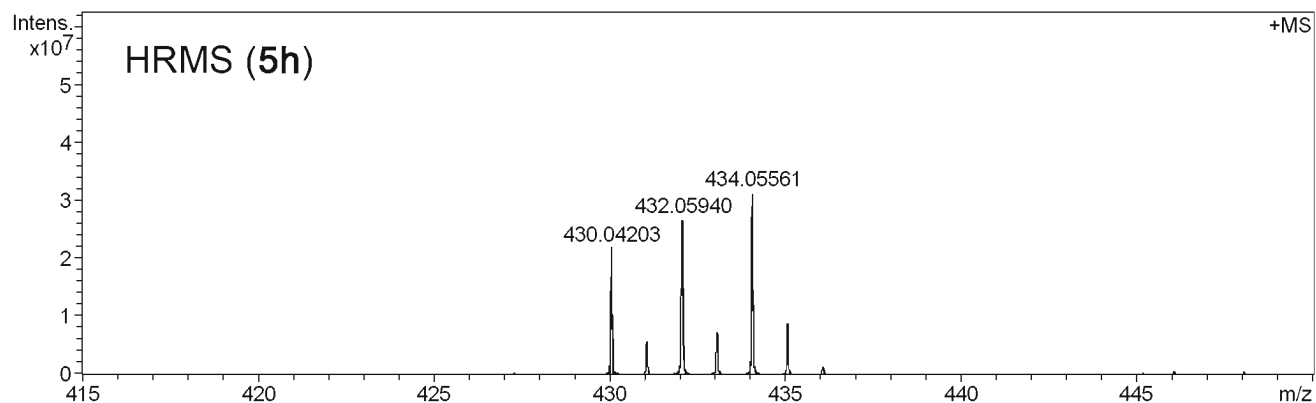
Analysis Name D:\20120619\YIN_7_APCI_POS_000002.d
Method Metal_Trypsin digestion
Sample Name YIN_7_APCI_POS
Comment

Acquisition Date 2012-6-19 19:07:22

Operator
Instrument apex-Ultra

Acquisition Parameter

Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	2	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	600.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Wed Jan 4 05:51:21 2012
Source Accumulation	0.0 sec	Drying Gas Flow Rate	4.0 L/min	Data Acquisition Size	131072
Ion Accumulation Time	0.0 sec	Nebulizer Gas Flow Rate	1.0 L/min	Apodization	Sine-Bell Multiplication
Flight Time to Acq. Cell	0.0 sec				



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e ₄ Conf	N-Rule
432.05940	1	C ₂₄ H ₁₉ BrNO ₂	100.00	432.05937	-0.0	-0.1	64.0	15.5	even	ok

Mass Spectrum SmartFormula Report

Analysis Info

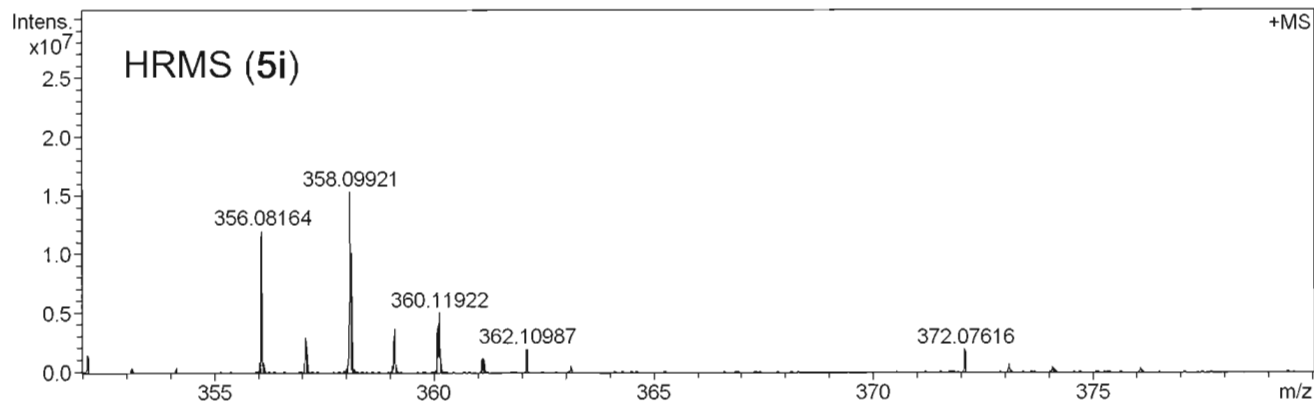
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Method Metal_Trypsin digestion
Sample Name YIN_8_APCI_POS
Comment

Acquisition Date 2012-6-19 19:08:26

Operator
Instrument apex-Ultra

Acquisition Parameter

Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	2	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	600.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Wed Jan 4 05:51:21 2012
Source Accumulation	0.0 sec	Drying Gas Flow Rate	4.0 L/min	Data Acquisition Size	131072
Ion Accumulation Time	0.0 sec	Nebulizer Gas Flow Rate	1.0 L/min	Apodization	Sine-Bell Multiplication
Flight Time to Acq. Cell	0.0 sec				



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e _j	Conf	N-Rule
358.09921	1	C ₂₃ H ₁₇ ClNO	100.00	358.09932	0.1	0.3	67.1	15.5		even	ok

Mass Spectrum SmartFormula Report

Analysis Info

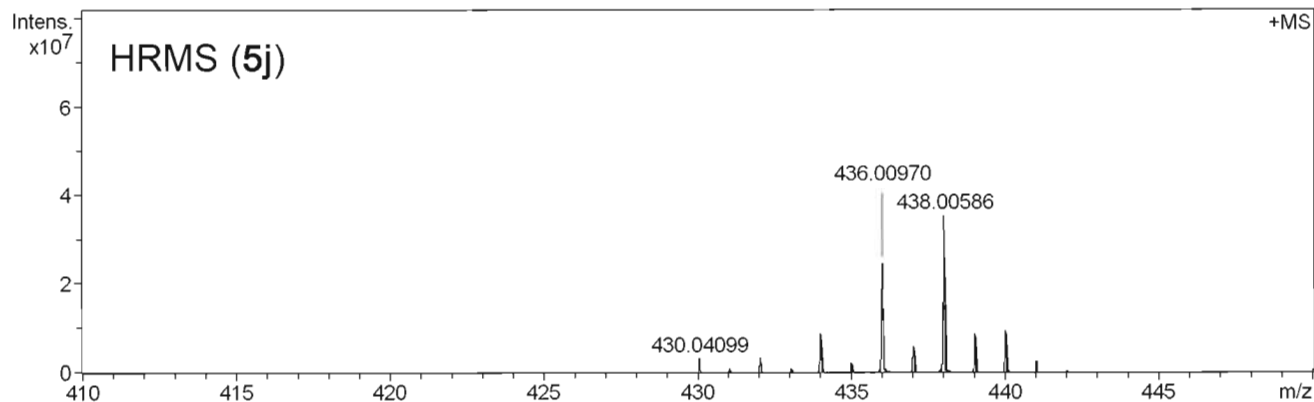
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Method Metal_Trypsin digestion
Sample Name YIN_9_APCI_POS
Comment

Acquisition Date 2012-6-19 19:09:23

Operator
Instrument apex-Ultra

Acquisition Parameter

Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	2	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	600.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Wed Jan 4 05:51:21 2012
Source Accumulation	0.0 sec	Drying Gas Flow Rate	4.0 L/min	Data Acquisition Size	131072
Ion Accumulation Time	0.0 sec	Nebulizer Gas Flow Rate	1.0 L/min	Apodization	Sine-Bell Multiplication
Flight Time to Acq. Cell	0.0 sec				



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e _j	Conf	N-Rule
436.00970	1	C ₂₃ H ₁₆ BrClNO	100.00	436.00983	0.1	0.3	27.6	15.5	even		ok

Mass Spectrum SmartFormula Report

Analysis Info

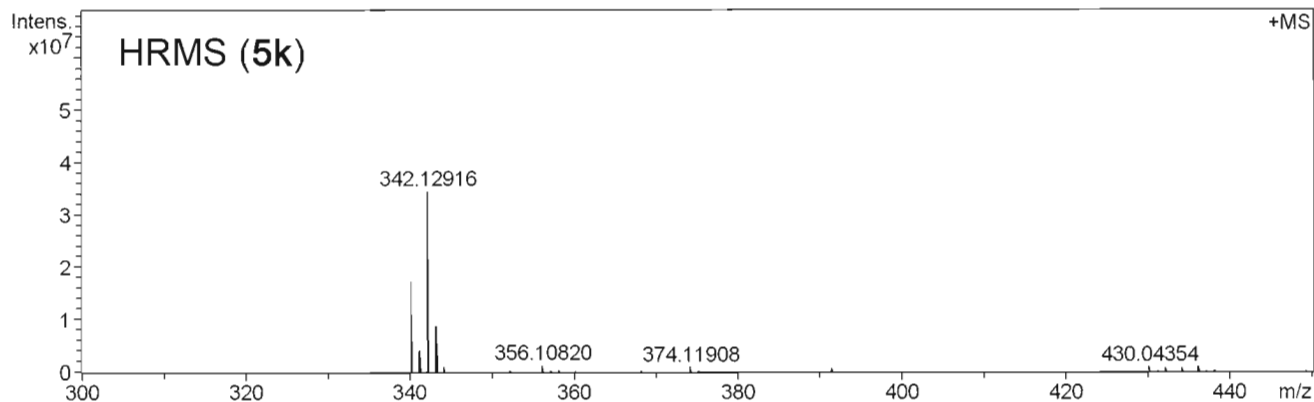
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Method Metal_Trypsin digestion
Sample Name YIN_10_APCI_POS
Comment

Acquisition Date 2012-6-19 19:10:20

Operator
Instrument apex-Ultra

Acquisition Parameter

Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	2	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	600.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Wed Jan 4 05:51:21 2012
Source Accumulation	0.0 sec	Drying Gas Flow Rate	4.0 L/min	Data Acquisition Size	131072
Ion Accumulation Time	0.0 sec	Nebulizer Gas Flow Rate	1.0 L/min	Apodization	Sine-Bell Multiplication
Flight Time to Acq. Cell	0.0 sec				



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	ej	Conf	N-Rule
342.12916	1	C ₂₃ H ₁₇ F ₂ N ₂ O	100.00	342.12887	-0.3	-0.9	3.8	15.5	even		ok

Mass Spectrum SmartFormula Report

Analysis Info

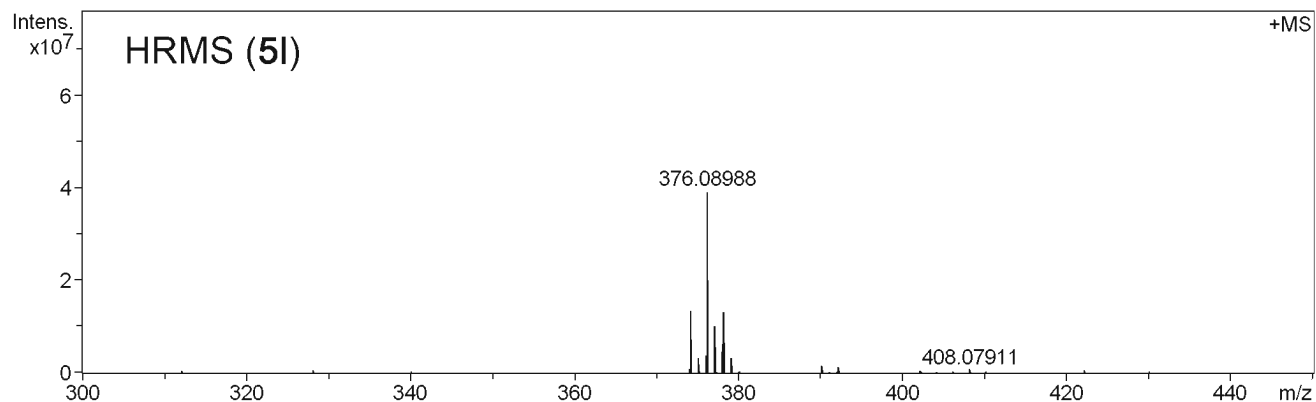
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Method Metal_Trypsin digestion
Sample Name YIN_16_APCI_POS
Comment

Acquisition Date 2012-6-19 19:17:11

Operator
Instrument apex-Ultra

Acquisition Parameter

Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	2	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	600.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Wed Jan 4 05:51:21 2012
Source Accumulation	0.0 sec	Drying Gas Flow Rate	4.0 L/min	Data Acquisition Size	131072
Ion Accumulation Time	0.0 sec	Nebulizer Gas Flow Rate	1.0 L/min	Apodization	Sine-Bell Multiplication
Flight Time to Acq. Cell	0.0 sec				



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSig ma	rdb	e;¥ Conf	N-R ule
376.08988	1	C 23 H 16 Cl F N O	100.00	376.08990	0.0	0.0	7.0	15.5	even	ok
	2	C 20 H 17 Cl F 2 N O 2	43.28	376.09104	1.2	3.1	20.7	11.5	even	ok
	3	C 18 H 16 Cl F 5 N	31.79	376.08859	-1.3	-3.4	30.8	8.5	even	ok

Mass Spectrum SmartFormula Report

Analysis Info

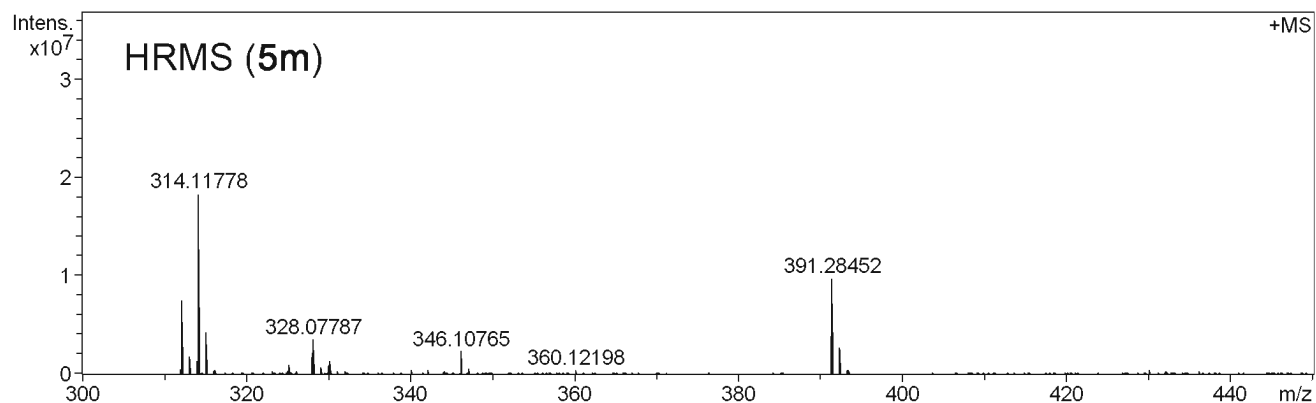
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Method Metal_Trypsin digestion
Sample Name YIN_15_APCI_POS
Comment

Acquisition Date 2012-6-19 19:15:22

Operator
Instrument apex-Ultra

Acquisition Parameter

Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	2	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	600.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Wed Jan 4 05:51:21 2012
Source Accumulation	0.0 sec	Drying Gas Flow Rate	4.0 L/min	Data Acquisition Size	131072
Ion Accumulation Time	0.0 sec	Nebulizer Gas Flow Rate	1.0 L/min	Apodization	Sine-Bell Multiplication
Flight Time to Acq. Cell	0.0 sec				



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e ₁	Conf	N-Rule
314.11778	1	C ₂₁ H ₁₆ N ₂ O ₂	100.00	314.11756	-0.2	-0.7	3.2	14.5	even		ok

Mass Spectrum SmartFormula Report

Analysis Info

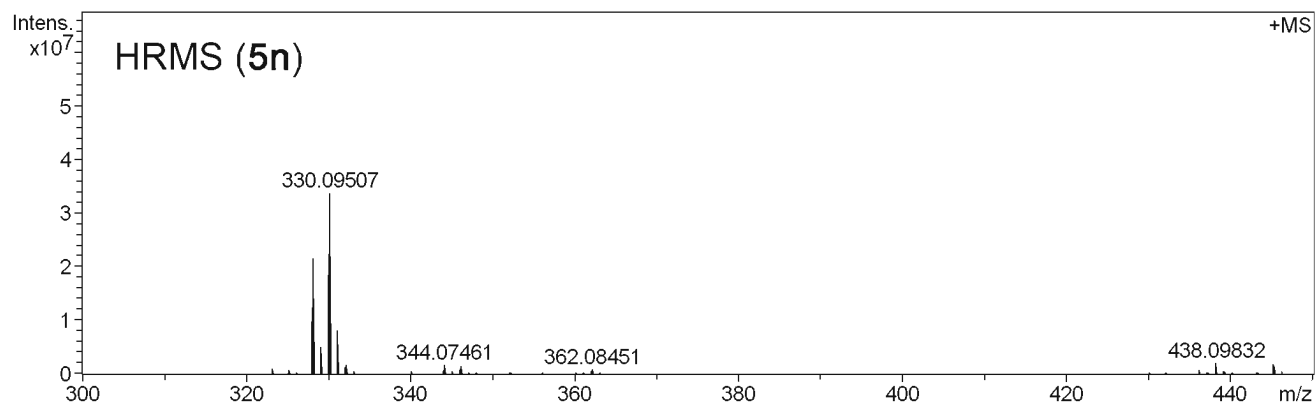
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Method Metal_Trypsin digestion
Sample Name YIN_14_APCI_POS
Comment

Acquisition Date 2012-6-19 19:14:18

Operator
Instrument apex-Ultra

Acquisition Parameter

Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	2	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	600.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Wed Jan 4 05:51:21 2012
Source Accumulation	0.0 sec	Drying Gas Flow Rate	4.0 L/min	Data Acquisition Size	131072
Ion Accumulation Time	0.0 sec	Nebulizer Gas Flow Rate	1.0 L/min	Apodization	Sine-Bell Multiplication
Flight Time to Acq. Cell	0.0 sec				



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e _j	Conf	N-Rule
330.09507	1	C ₂₁ H ₁₆ N ₁ O ₅	100.00	330.09471	-0.4	-1.1	19.2	14.5		even	ok

Mass Spectrum SmartFormula Report

Analysis Info

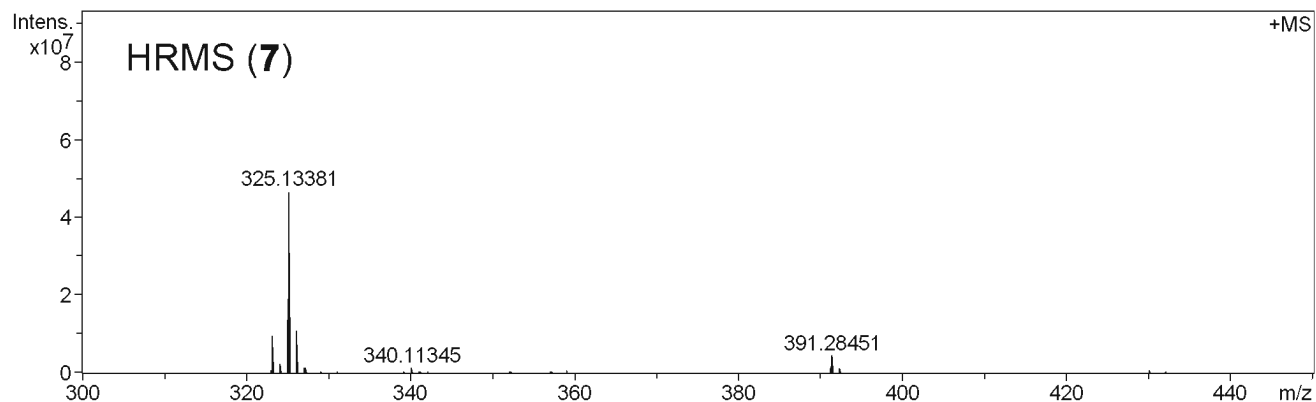
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Method Metal_Trypsin digestion
Sample Name YIN_13_APCI_POS
Comment

Acquisition Date 2012-6-19 19:13:22

Operator
Instrument apex-Ultra

Acquisition Parameter

Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	2	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	600.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Wed Jan 4 05:51:21 2012
Source Accumulation	0.0 sec	Drying Gas Flow Rate	4.0 L/min	Data Acquisition Size	131072
Ion Accumulation Time	0.0 sec	Nebulizer Gas Flow Rate	1.0 L/min	Apodization	Sine-Bell Multiplication
Flight Time to Acq. Cell	0.0 sec				



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e _i	Conf	N-Rule
325.13381	1	C ₂₂ H ₁₇ N ₂ O	100.00	325.13354	-0.3	-0.8	5.0	15.5		even	ok

Mass Spectrum SmartFormula Report

Analysis Info

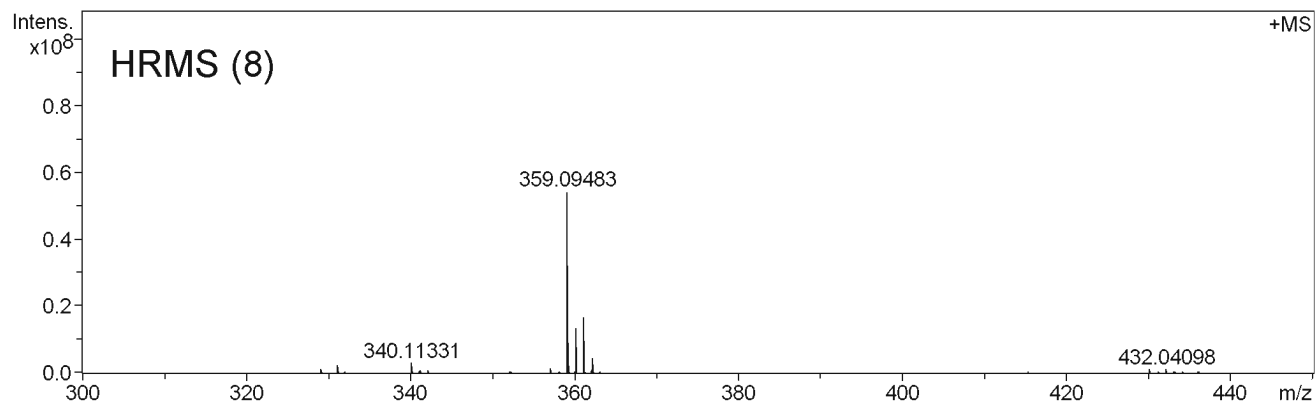
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Method Metal_Trypsin digestion
Sample Name YIN_12_APCI_POS
Comment

Acquisition Date 2012-6-19 19:12:27

Operator
Instrument apex-Ultra

Acquisition Parameter

Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	2	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	600.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Wed Jan 4 05:51:21 2012
Source Accumulation	0.0 sec	Drying Gas Flow Rate	4.0 L/min	Data Acquisition Size	131072
Ion Accumulation Time	0.0 sec	Nebulizer Gas Flow Rate	1.0 L/min	Apodization	Sine-Bell Multiplication
Flight Time to Acq. Cell	0.0 sec				



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e ₁	Conf	N-Rule
359.09483	1	C ₂₂ H ₁₆ ClN ₂ O	100.00	359.09457	-0.3	-0.7	15.5	15.5	even		ok

Mass Spectrum SmartFormula Report

Analysis Info

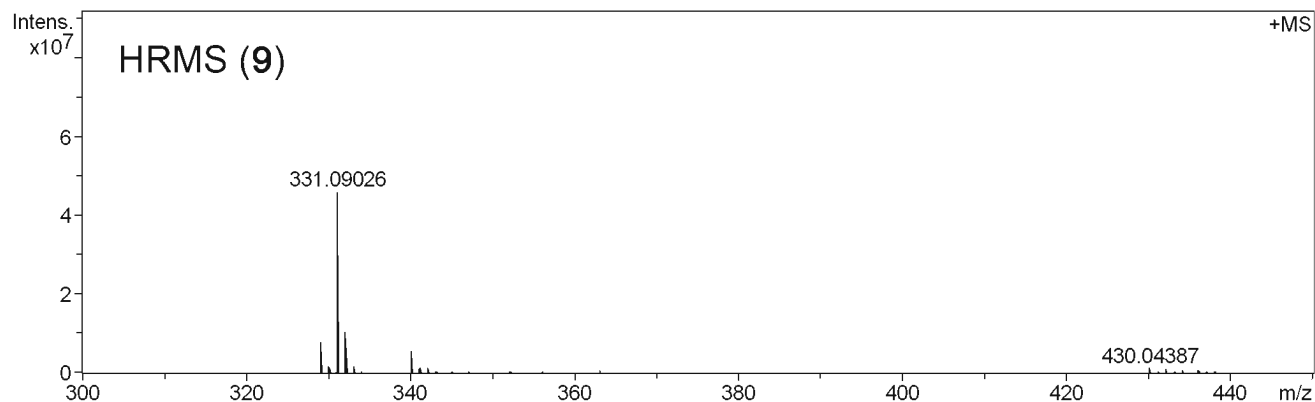
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Method Metal_Trypsin digestion
Sample Name YIN_11_APCI_POS
Comment

Acquisition Date 2012-6-19 19:11:31

Operator
Instrument apex-Ultra

Acquisition Parameter

Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	2	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	600.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Wed Jan 4 05:51:21 2012
Source Accumulation	0.0 sec	Drying Gas Flow Rate	4.0 L/min	Data Acquisition Size	131072
Ion Accumulation Time	0.0 sec	Nebulizer Gas Flow Rate	1.0 L/min	Apodization	Sine-Bell Multiplication
Flight Time to Acq. Cell	0.0 sec				



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e _j Conf	N-Rule
331.09026	1	C ₂₀ H ₁₅ N ₂ O ₅	100.00	331.08996	-0.3	-0.9	17.3	14.5	even	ok

HSQC spectrum of compound 7 (600MHz, CDCl₃)

