Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
This journal is © The Royal Society of Chemistry 2012

Supplementary Information

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

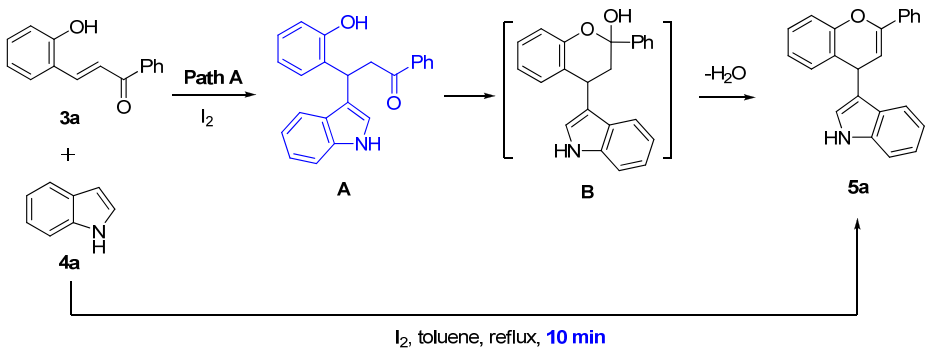
Guodong Yin,*[,a] Ling Fan,[a] Tianbing Ren,[a] Chunyang Zheng,[a] Qing Tao,[a] Anxin Wu,[b] and Nengfang She*[,b]

[a]Hubei Key Laboratory of Pollutant Analysis & Reuse Technology, College of Chemistry and Environmental Engineering, Hubei Normal University, Huangshi 435002, China. E-mail: gdyin@hbnu.edu.cn

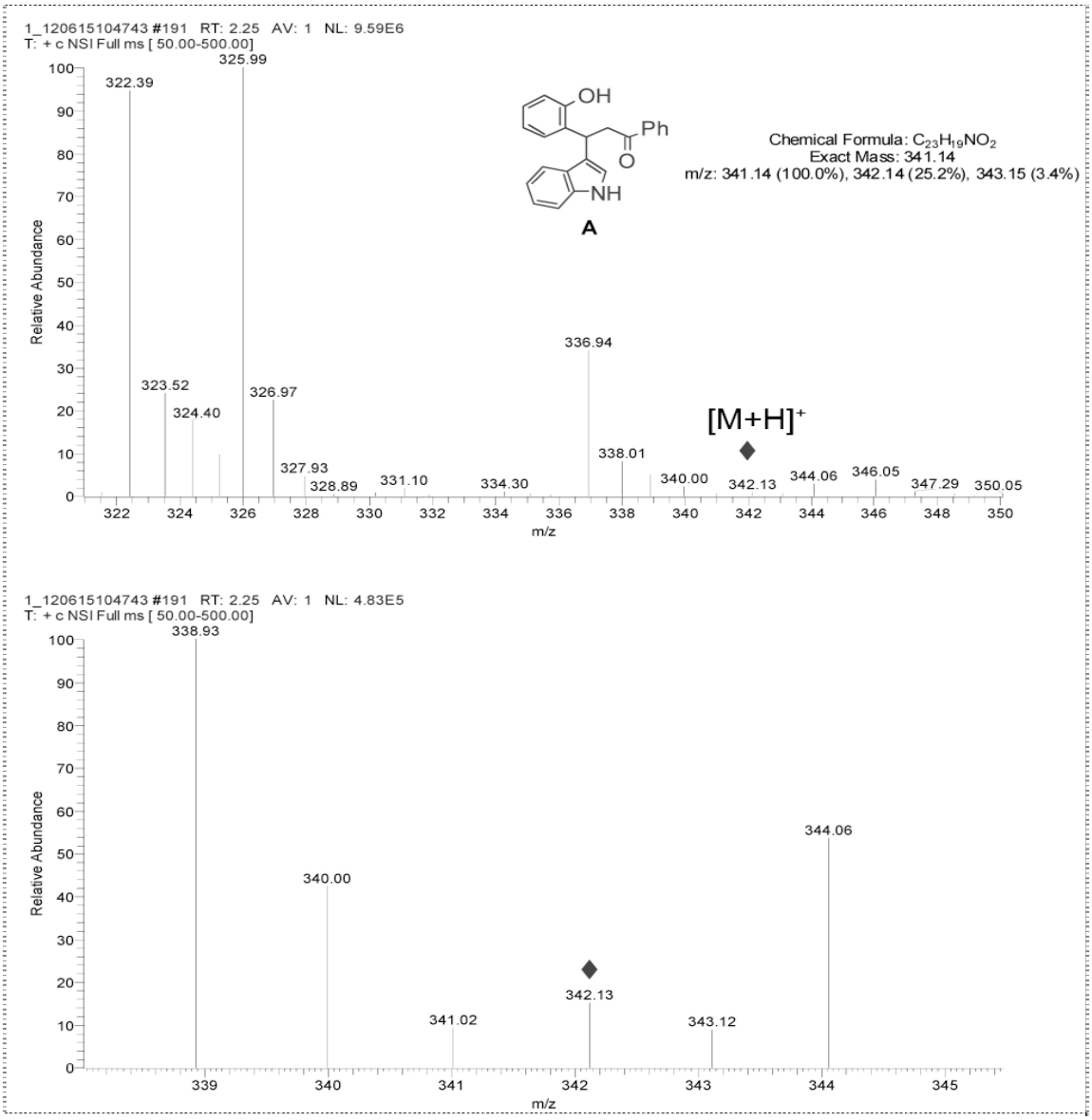
[b]Key Laboratory of Pesticides and Chemical Biology of the Ministry of Education, College of Chemistry, Central China Normal University, Wuhan 430079, China. E-mail: nfshe@mail.ccnu.edu.cn

Outline

◆ Mechanism study

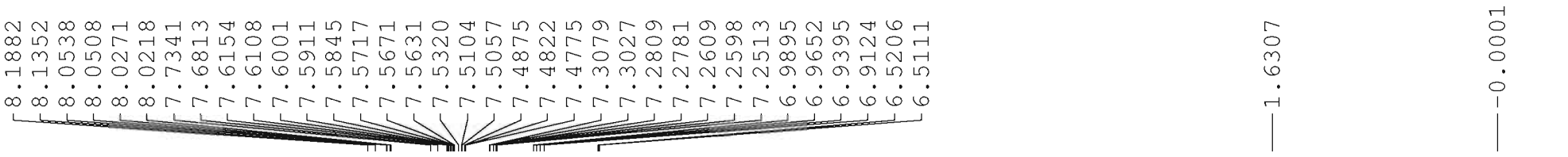


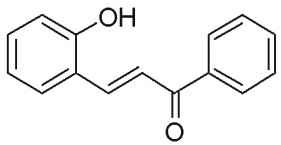
ESI-MS of the reaction mixture after 10min, and intermediate **A** was observed.



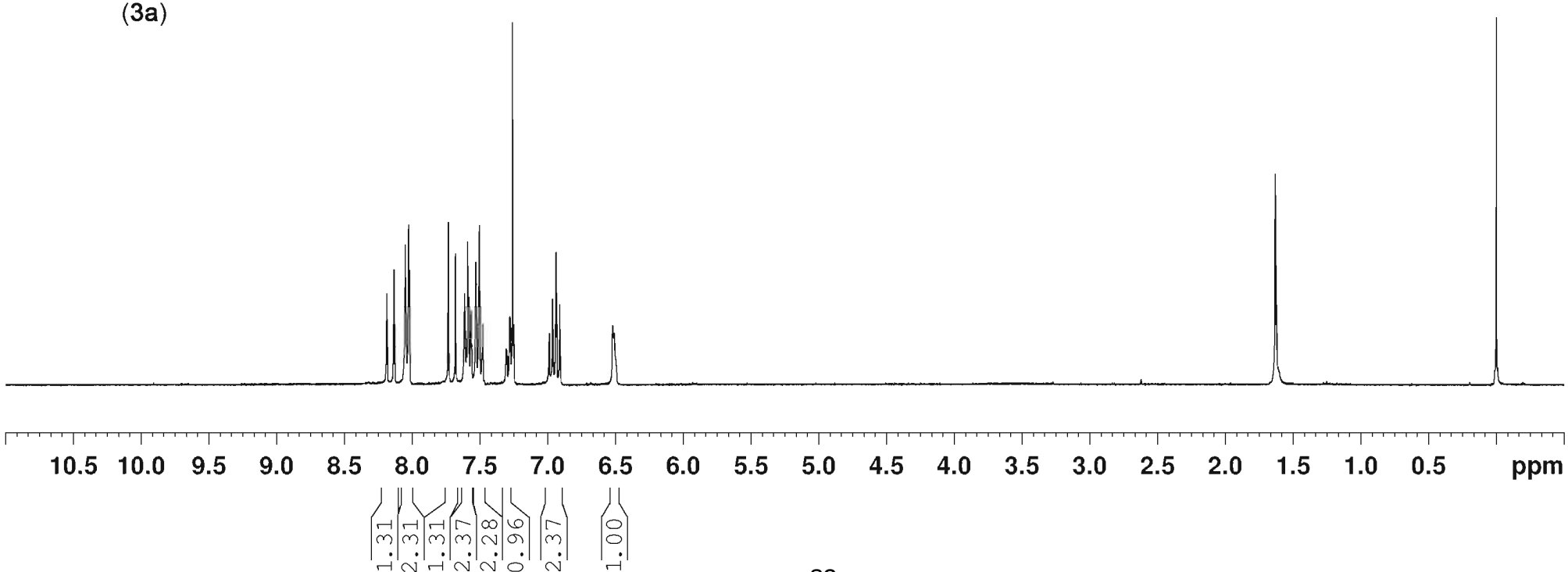
This journal is © The Royal Society of Chemistry 2012

^{1}H NMR (300MHz, $CDCl_3$)



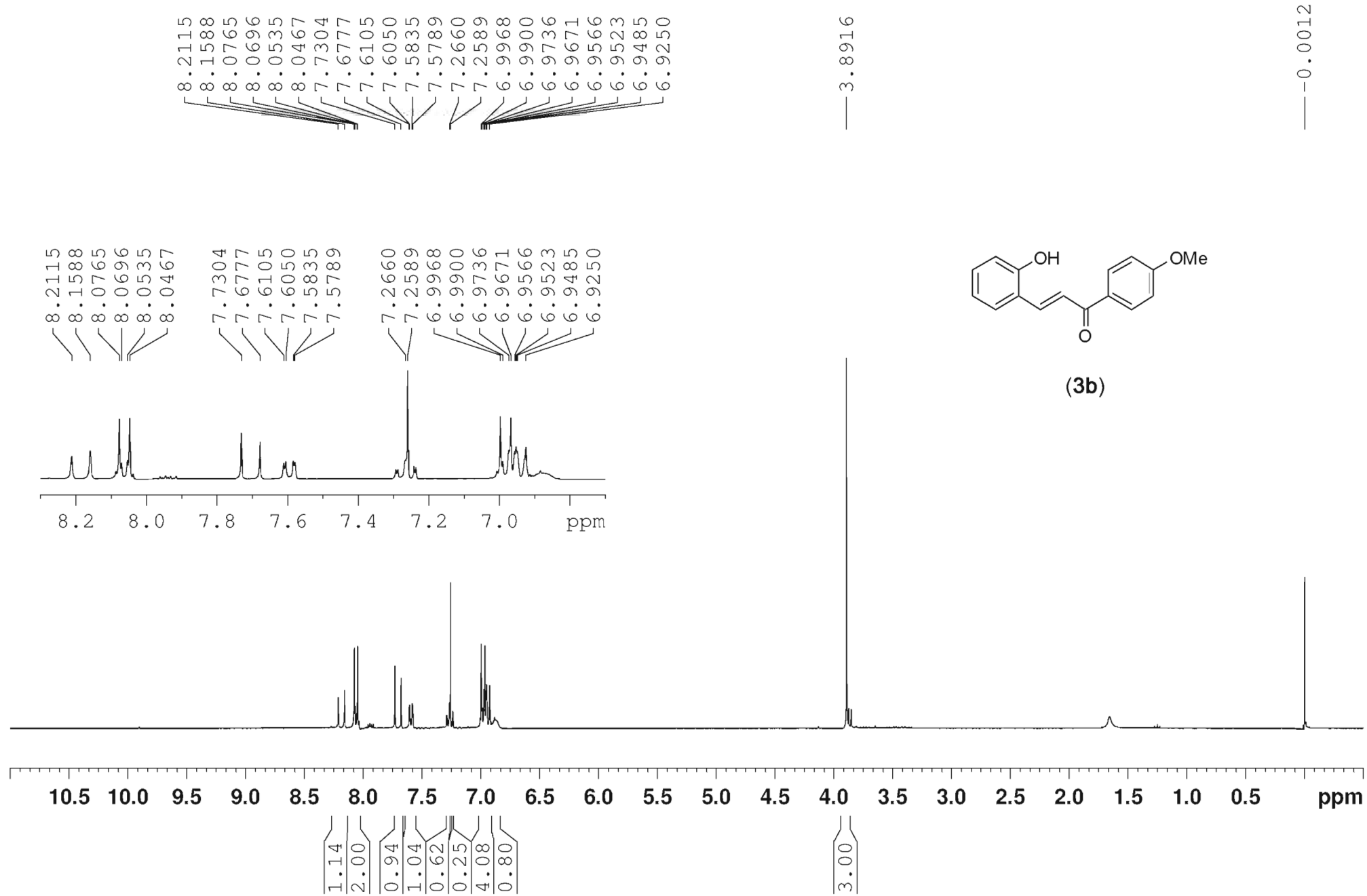


(**3a**)



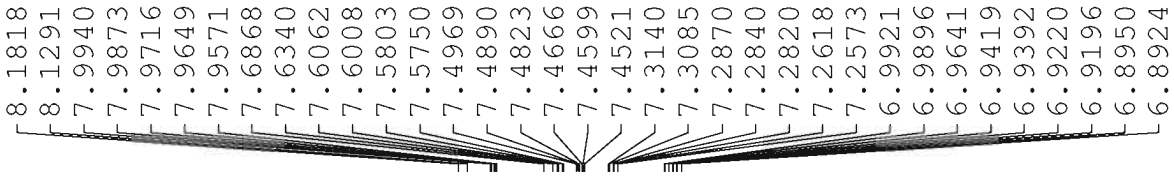
This journal is © The Royal Society of Chemistry 2012

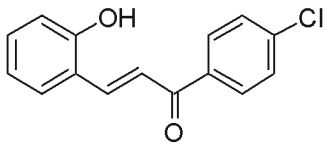
^{1}H NMR (300MHz, $CDCl_3$)



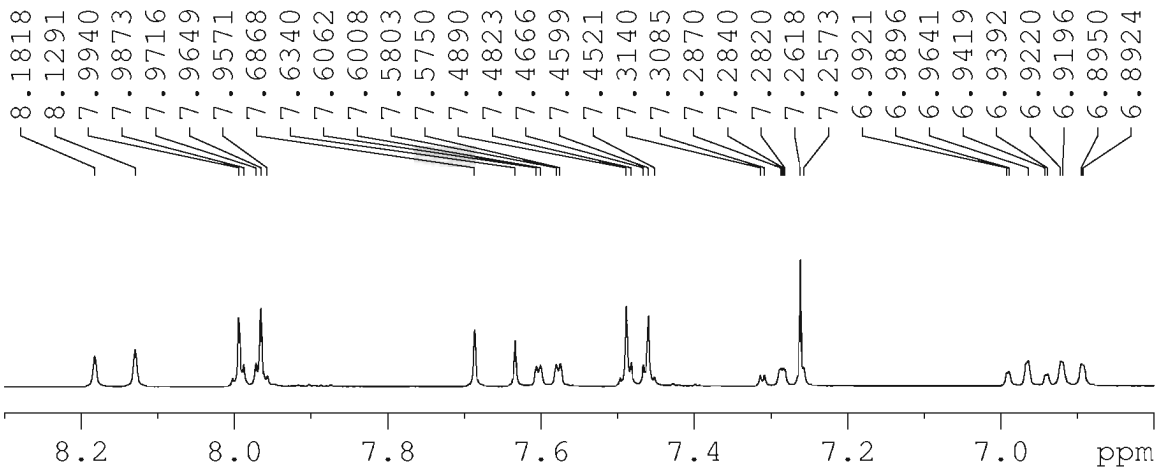
This journal is © The Royal Society of Chemistry 2012

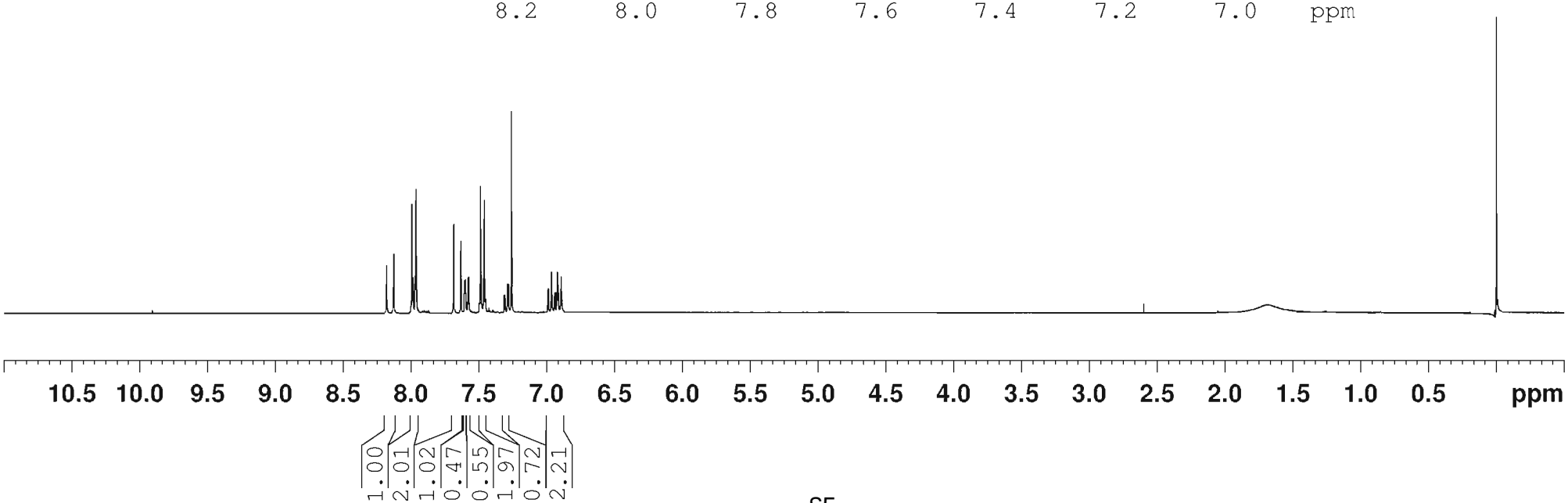
^{1}H NMR (300MHz, $CDCl_3$)





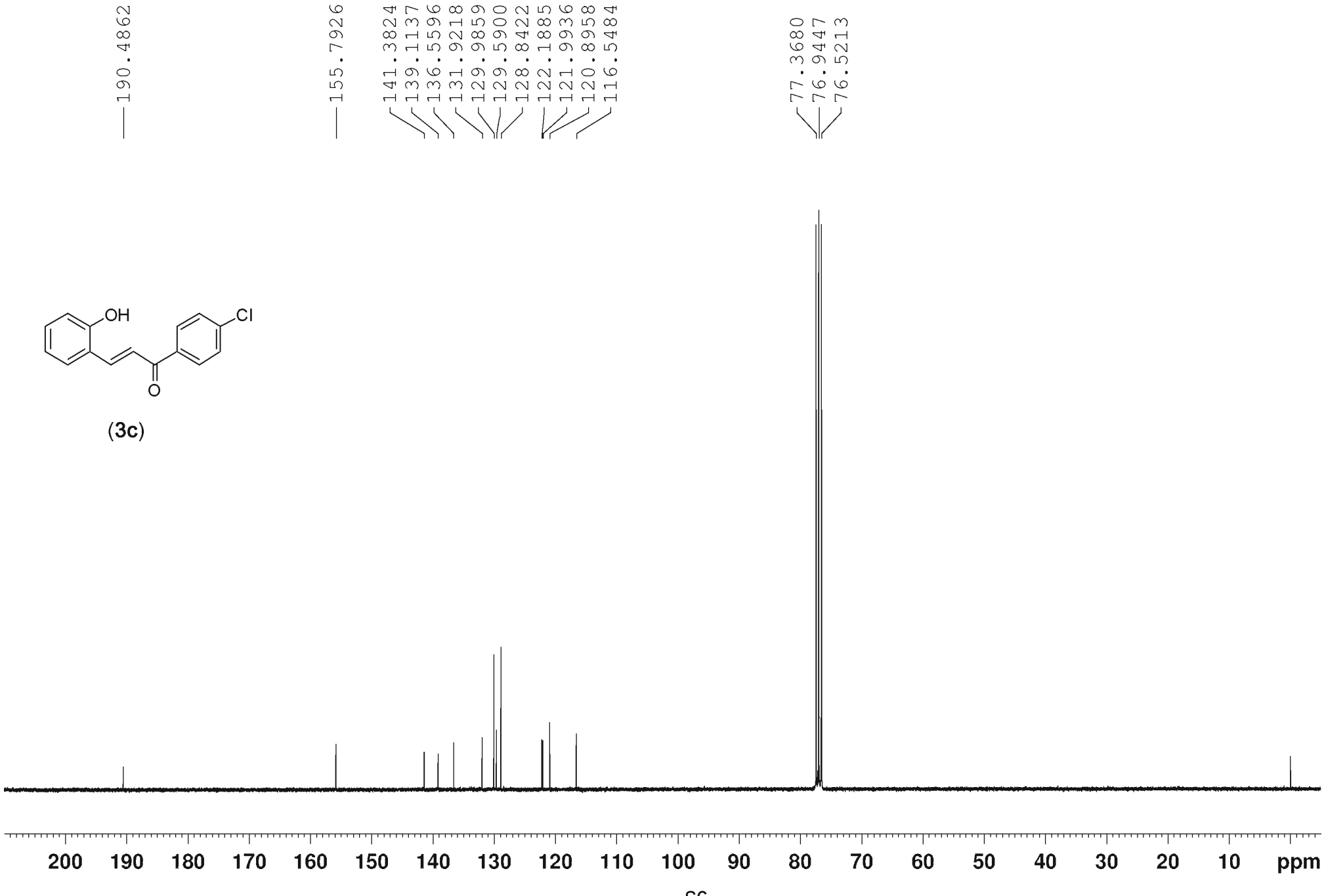
(3c)





This journal is © The Royal Society of Chemistry 2012

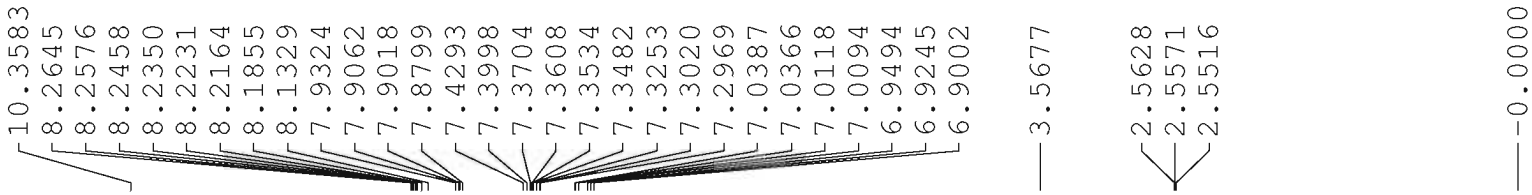
^{13}C NMR (75MHz, $CDCl_3$)

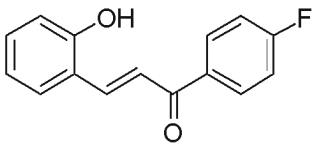


(**3c**)

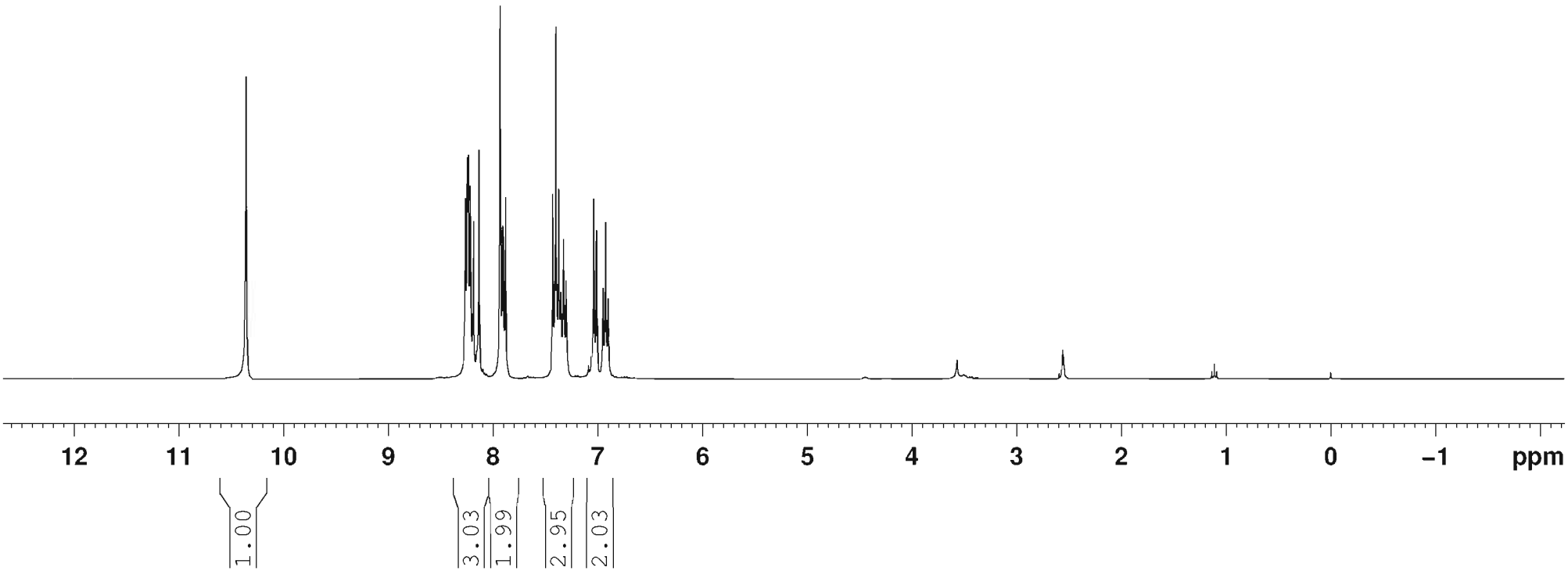
This journal is © The Royal Society of Chemistry 2012

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





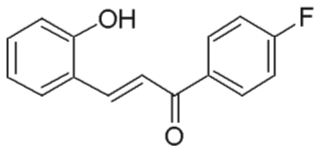
(3d)



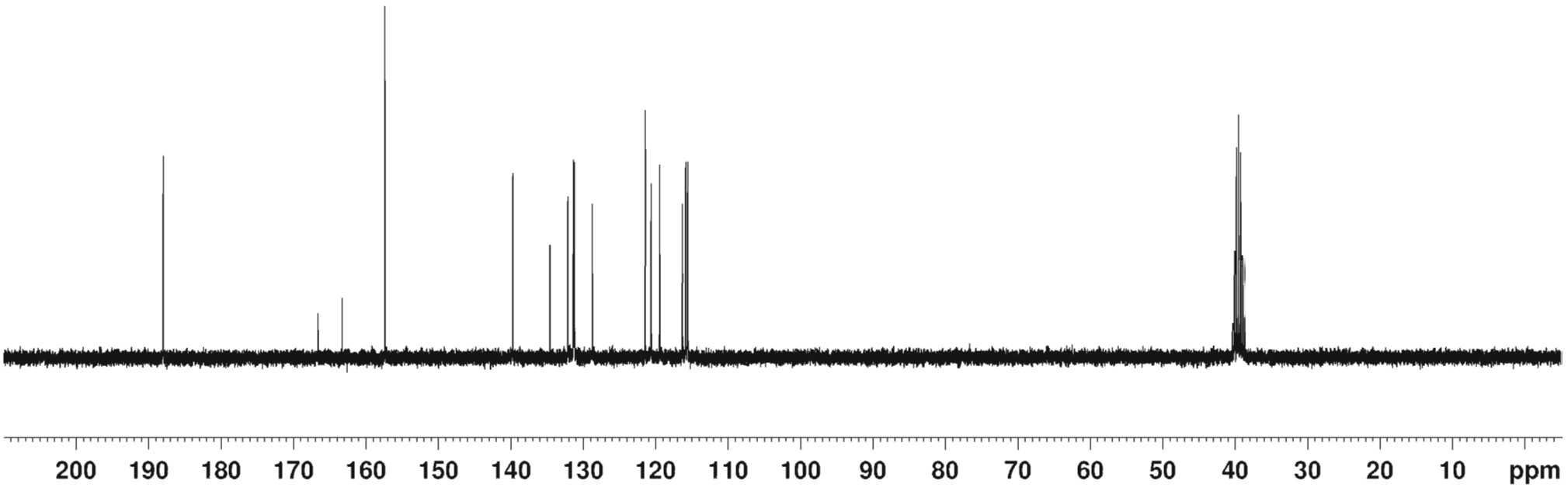
This journal is © The Royal Society of Chemistry 2012

^{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
39.7774
39.5006
39.2211
38.9481
38.6636

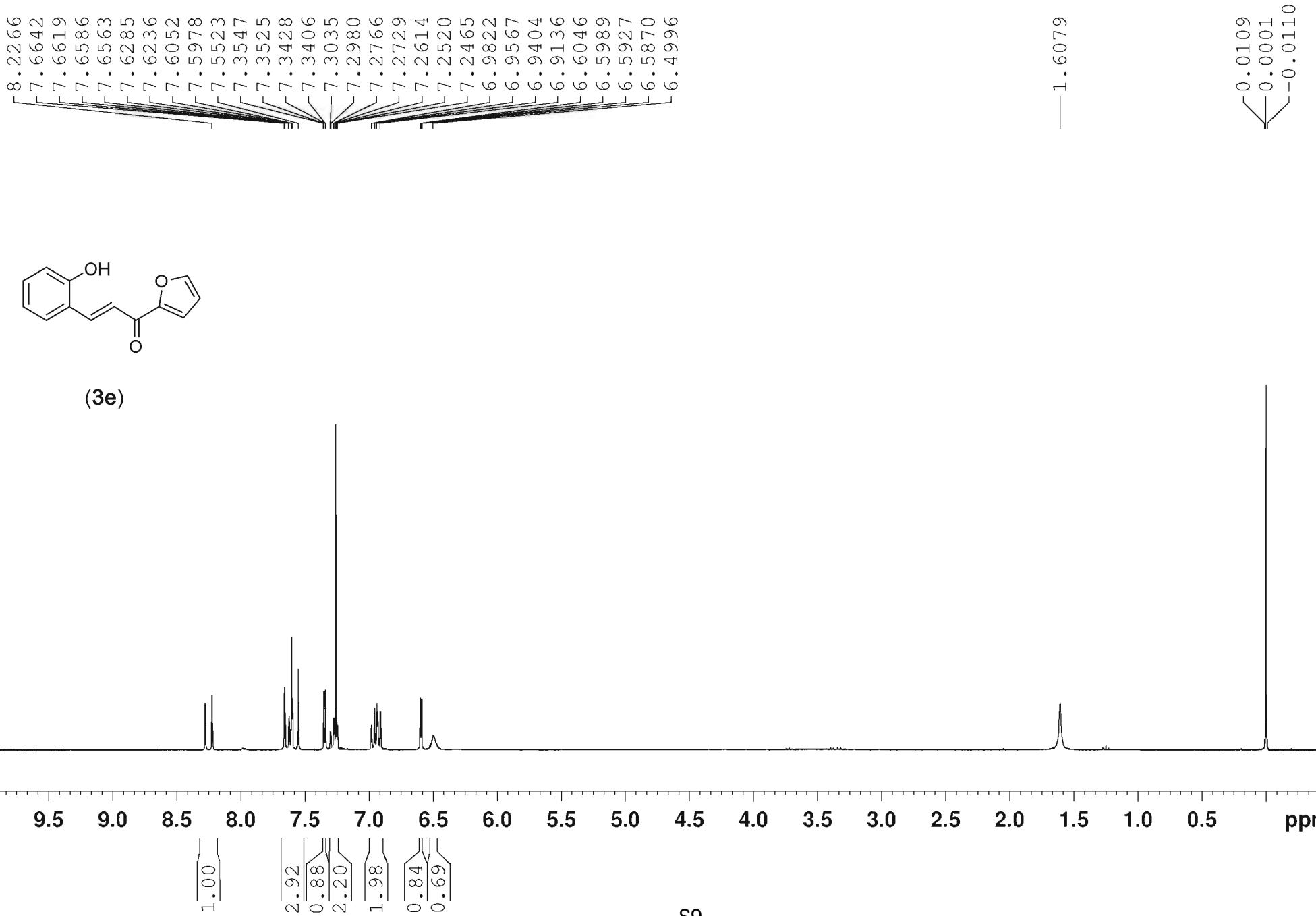


(3d)



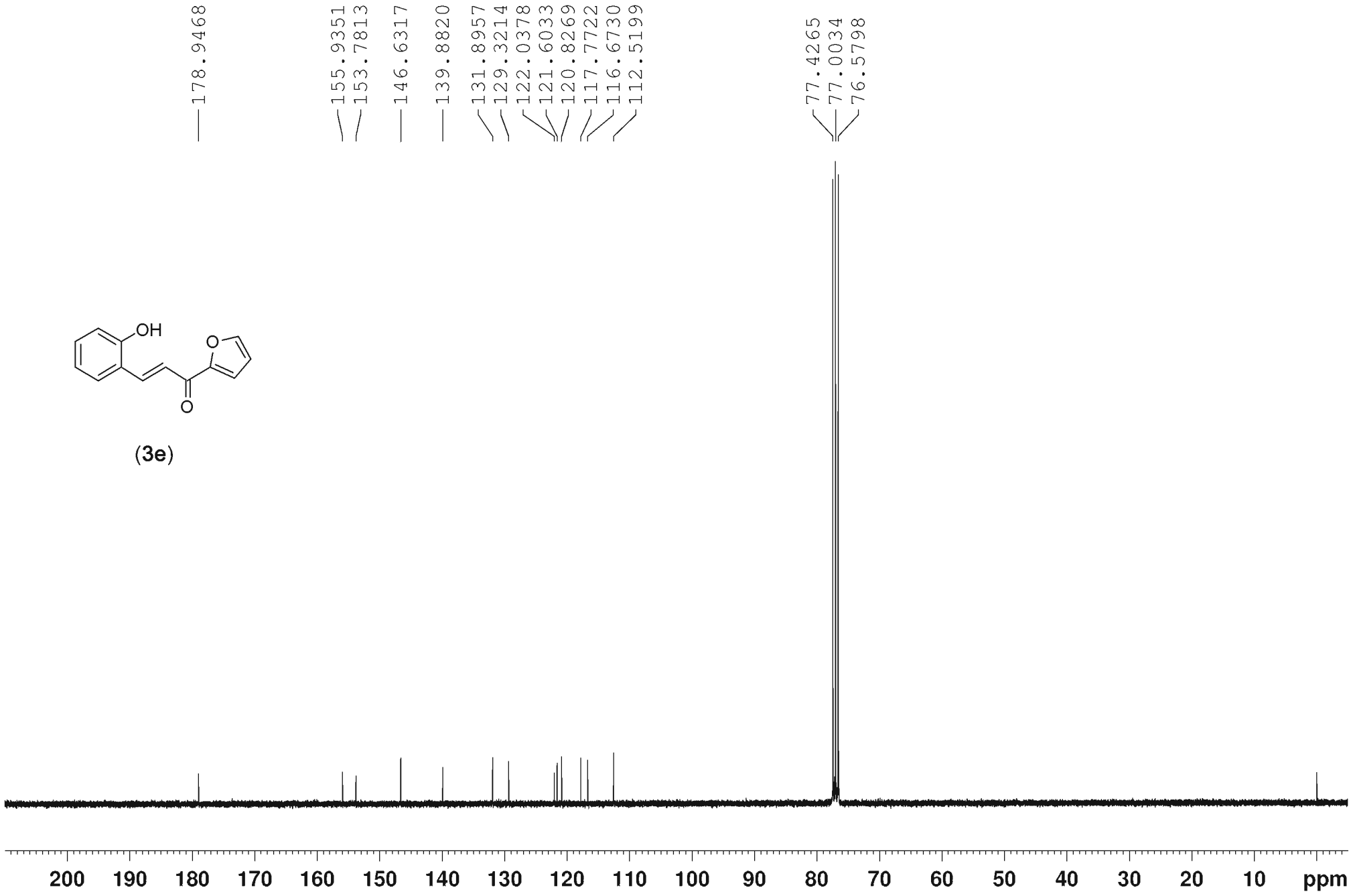
This journal is © The Royal Society of Chemistry 2012

^{1}H NMR (300MHz, $CDCl_3$)



This journal is © The Royal Society of Chemistry 2012

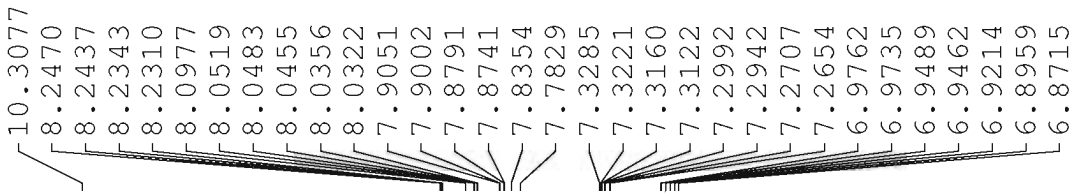
^{13}C NMR (75MHz, $CDCl_3$)

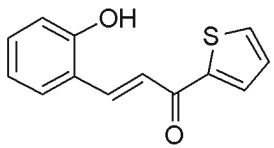


(**3e**)

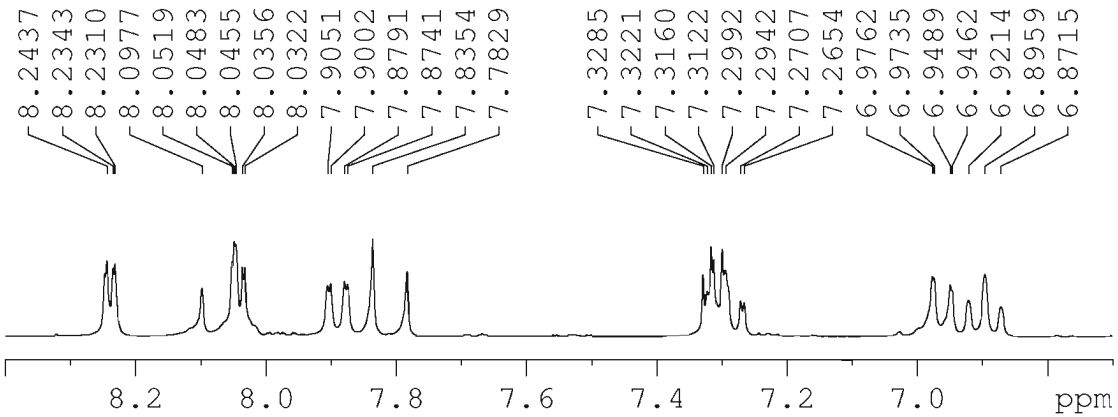
This journal is © The Royal Society of Chemistry 2012

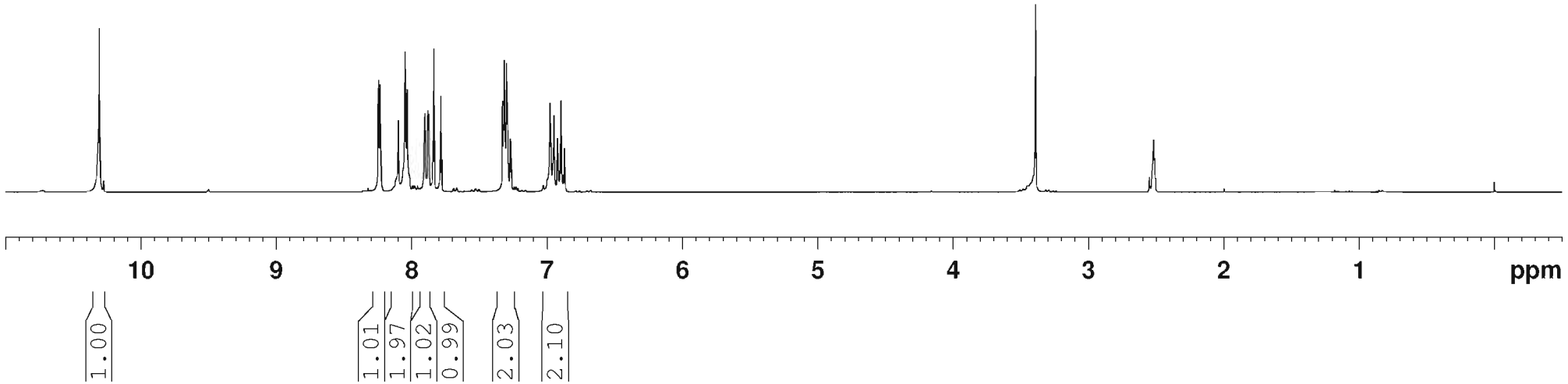
^{1}H NMR (300MHz, DMSO-d_6)





(3f)

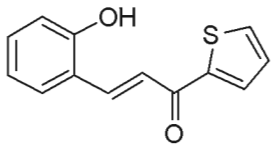




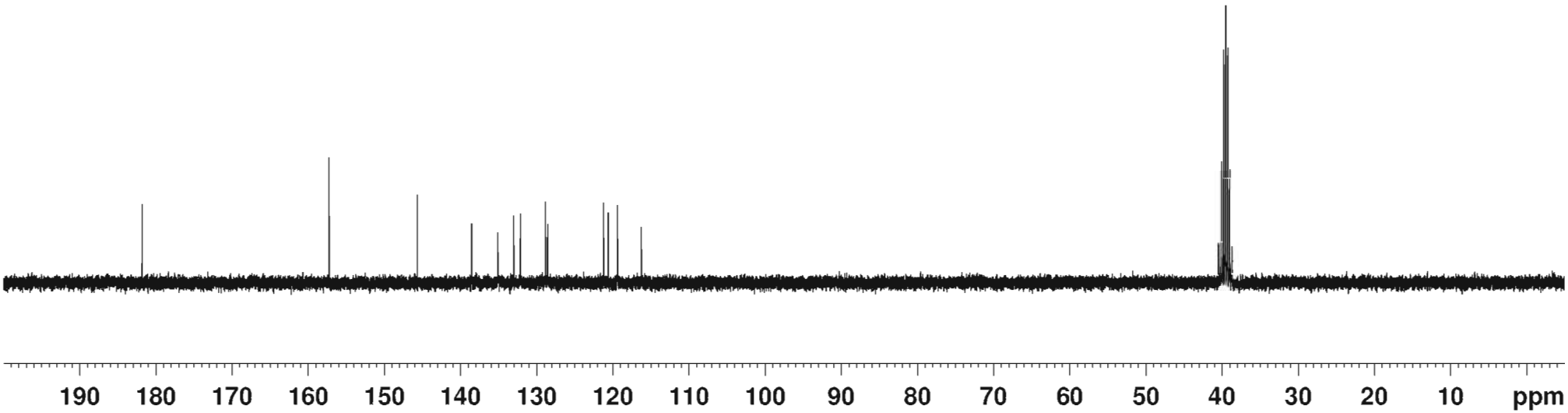
This journal is © The Royal Society of Chemistry 2012

^{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

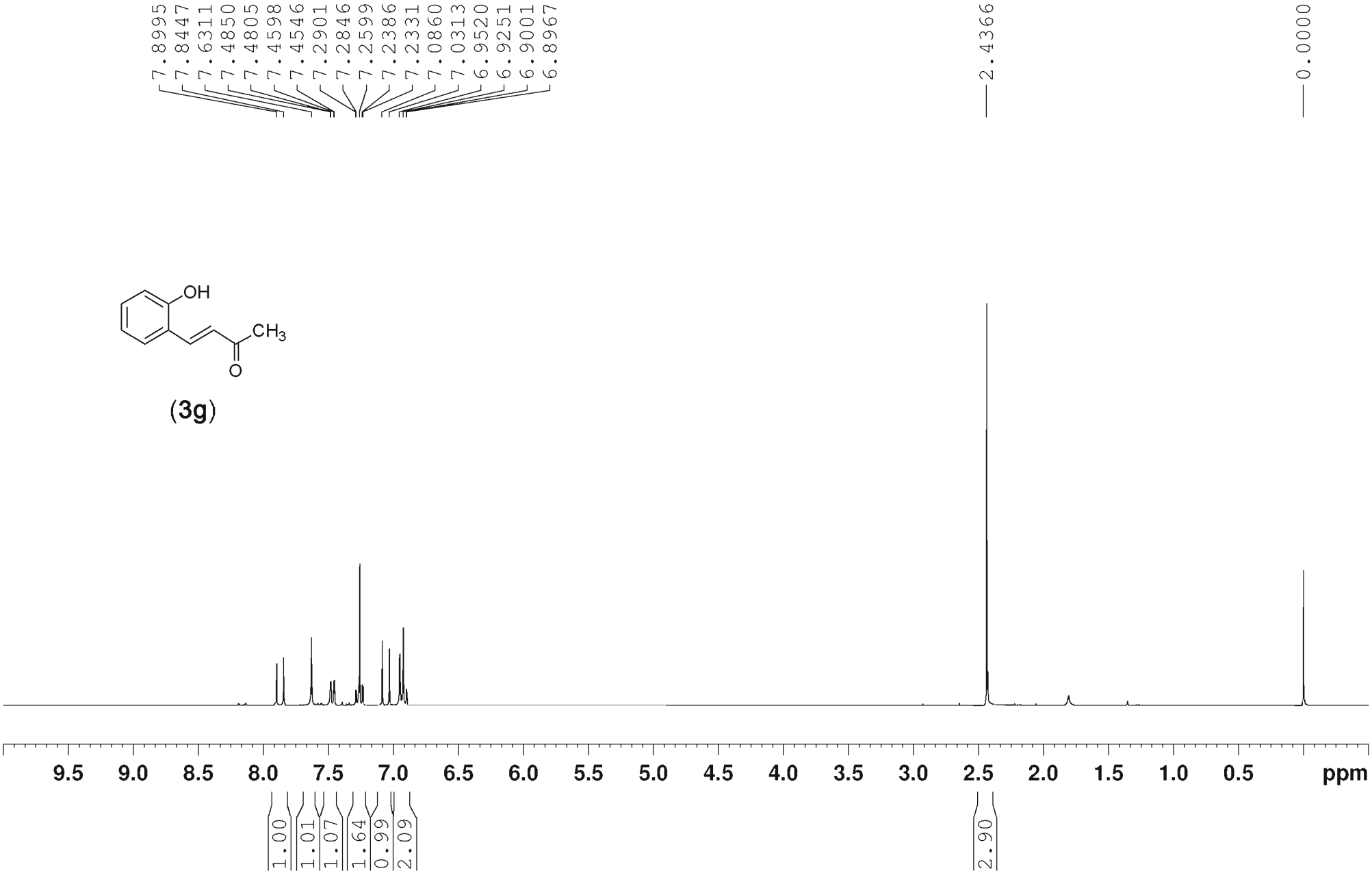


(3f)



This journal is © The Royal Society of Chemistry 2012

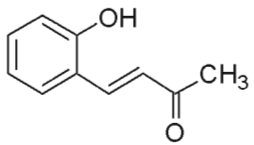
^{1}H NMR (300MHz, $CDCl_3$)



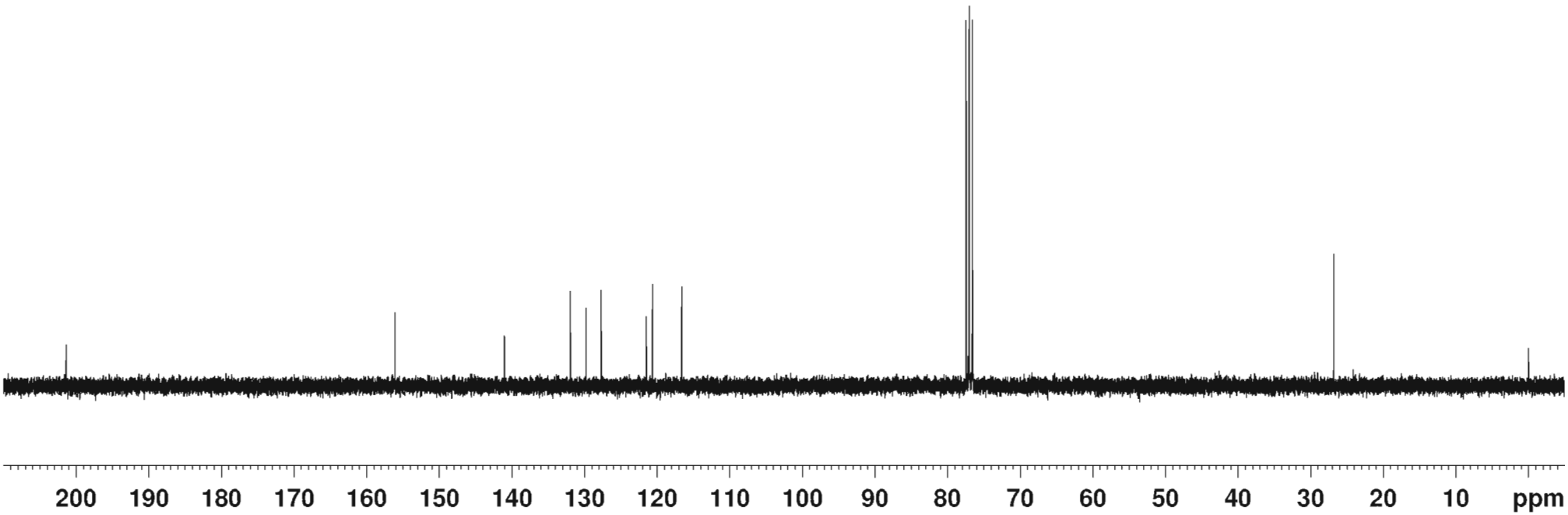
This journal is © The Royal Society of Chemistry 2012

^{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

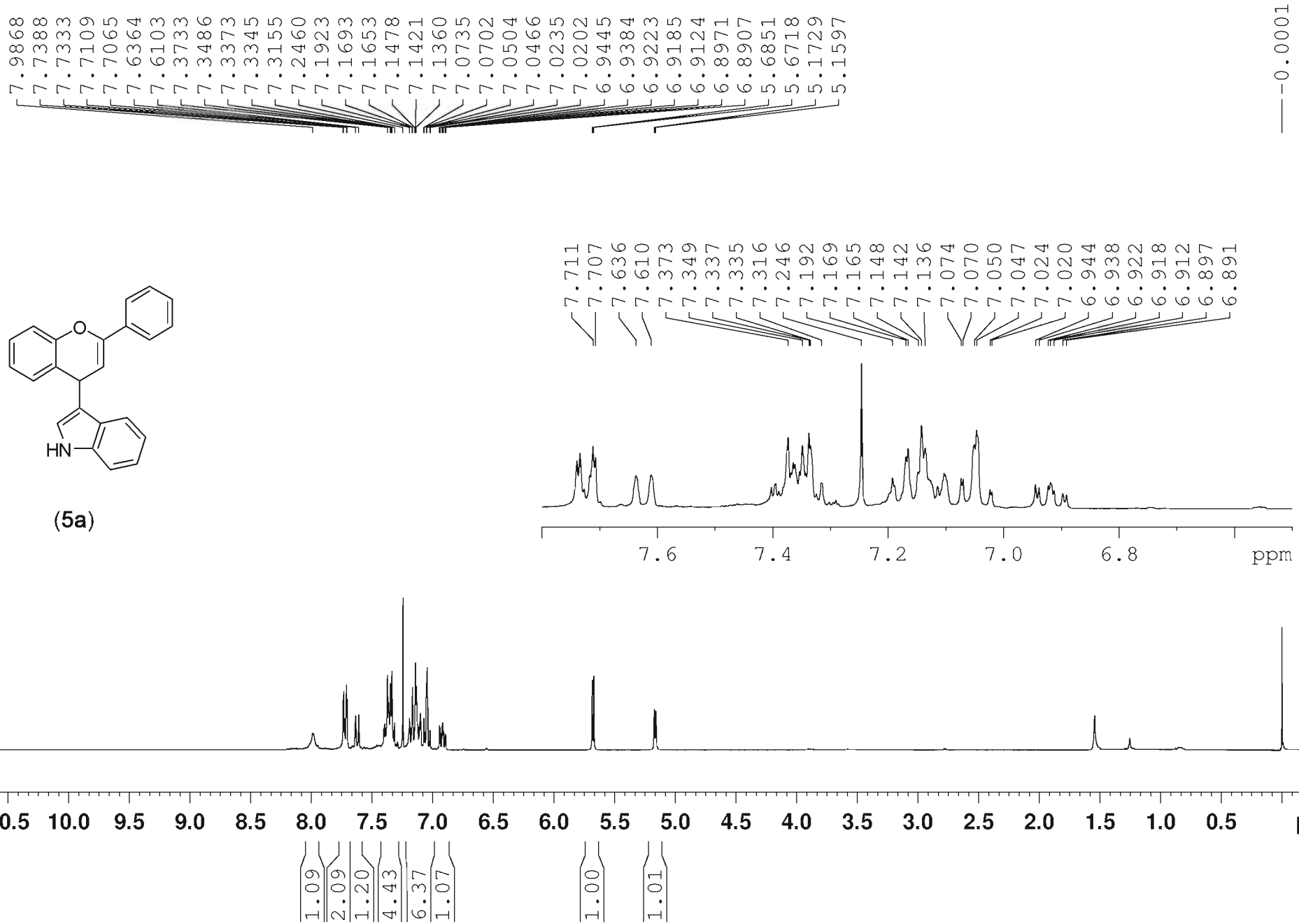


(3g)



This journal is © The Royal Society of Chemistry 2012

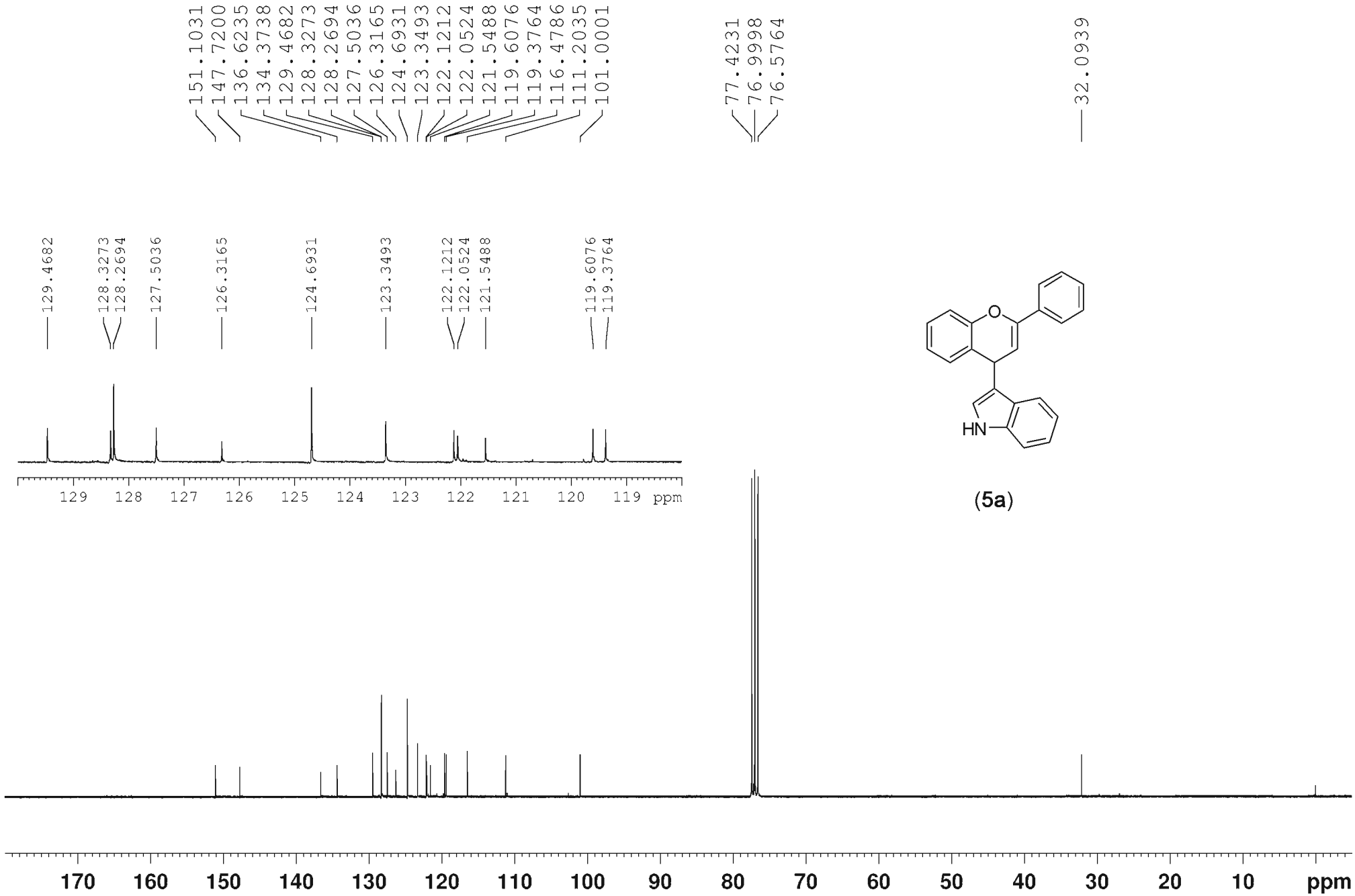
^{1}H NMR (300MHz, $CDCl_3$)



(5a)

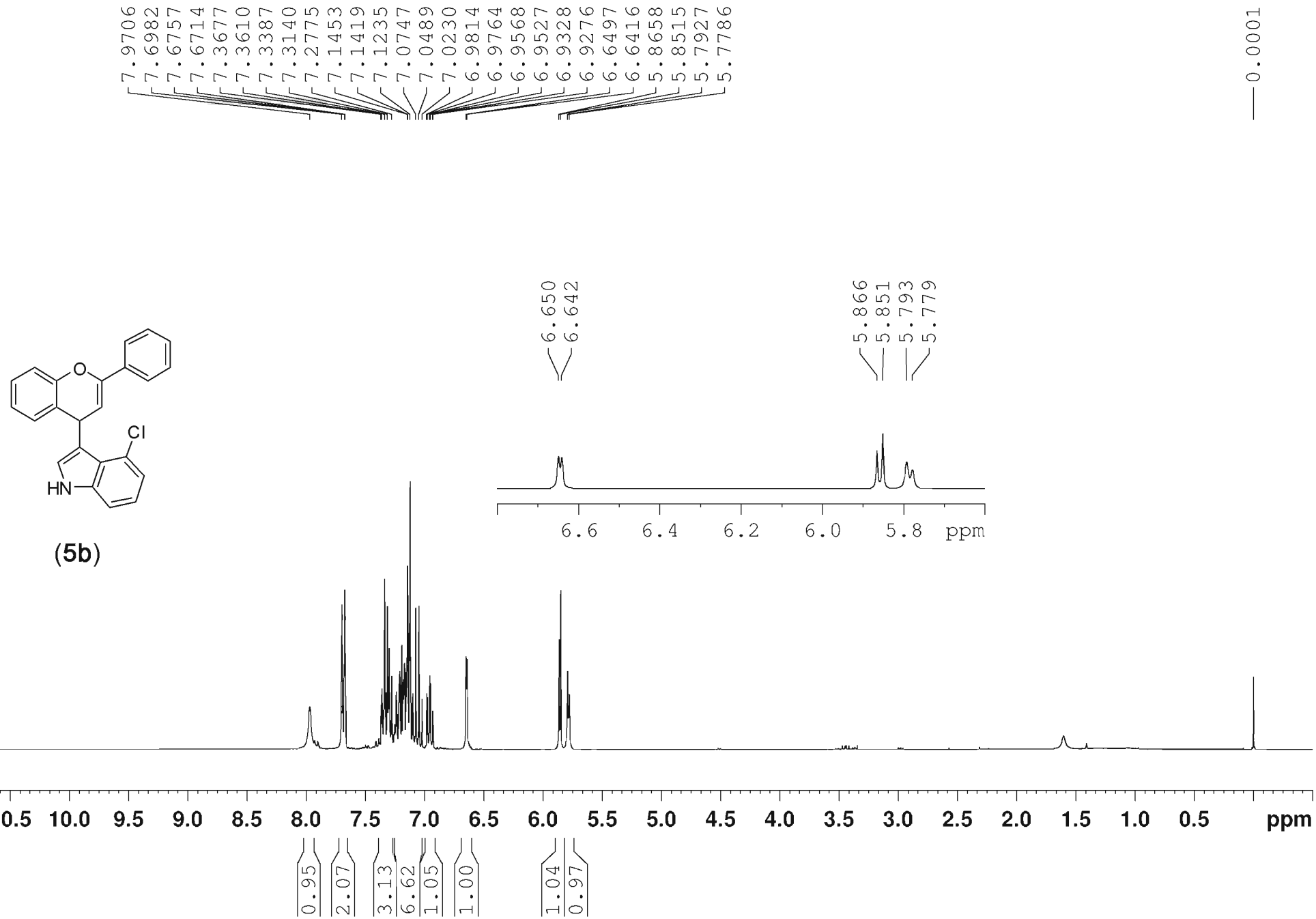
This journal is © The Royal Society of Chemistry 2012

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



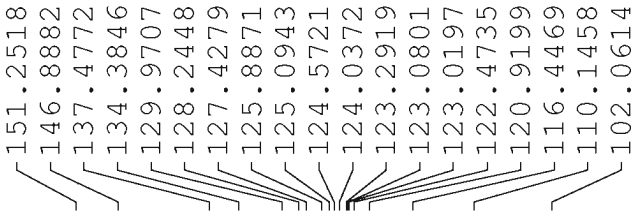
This journal is © The Royal Society of Chemistry 2012

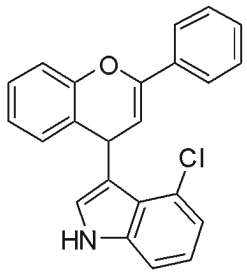
^{1}H NMR (300MHz, $CDCl_3$)



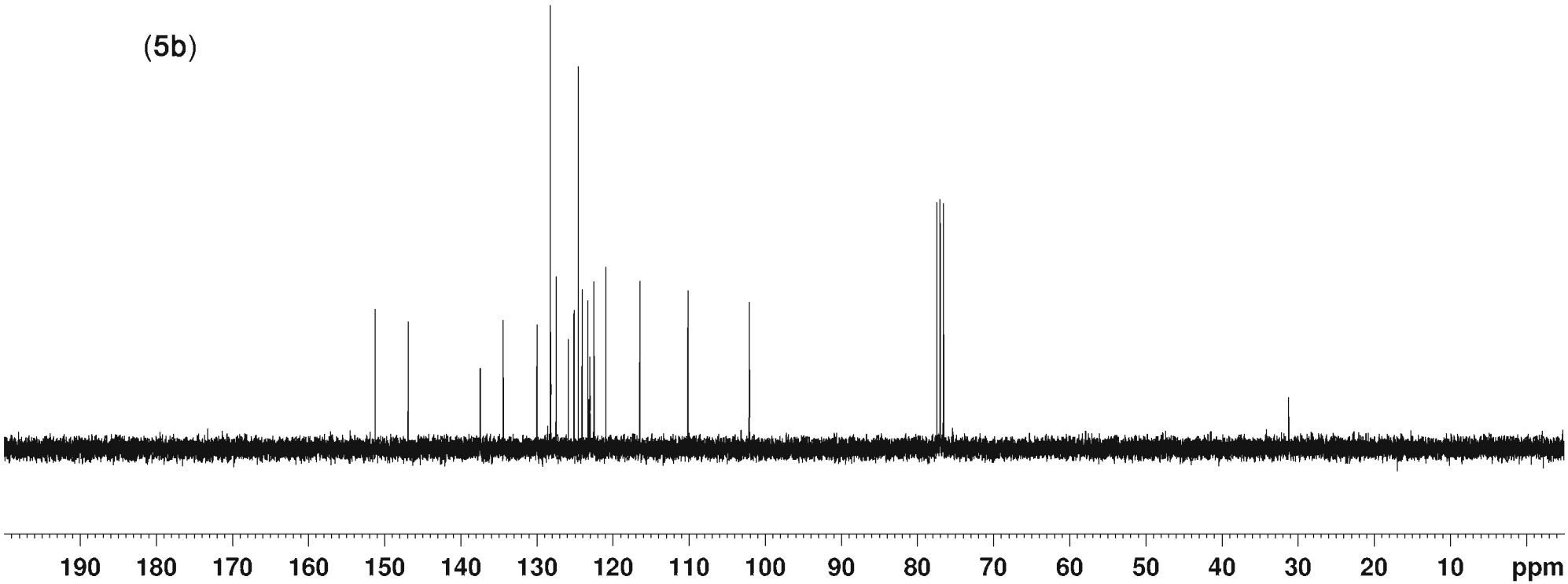
This journal is © The Royal Society of Chemistry 2012

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



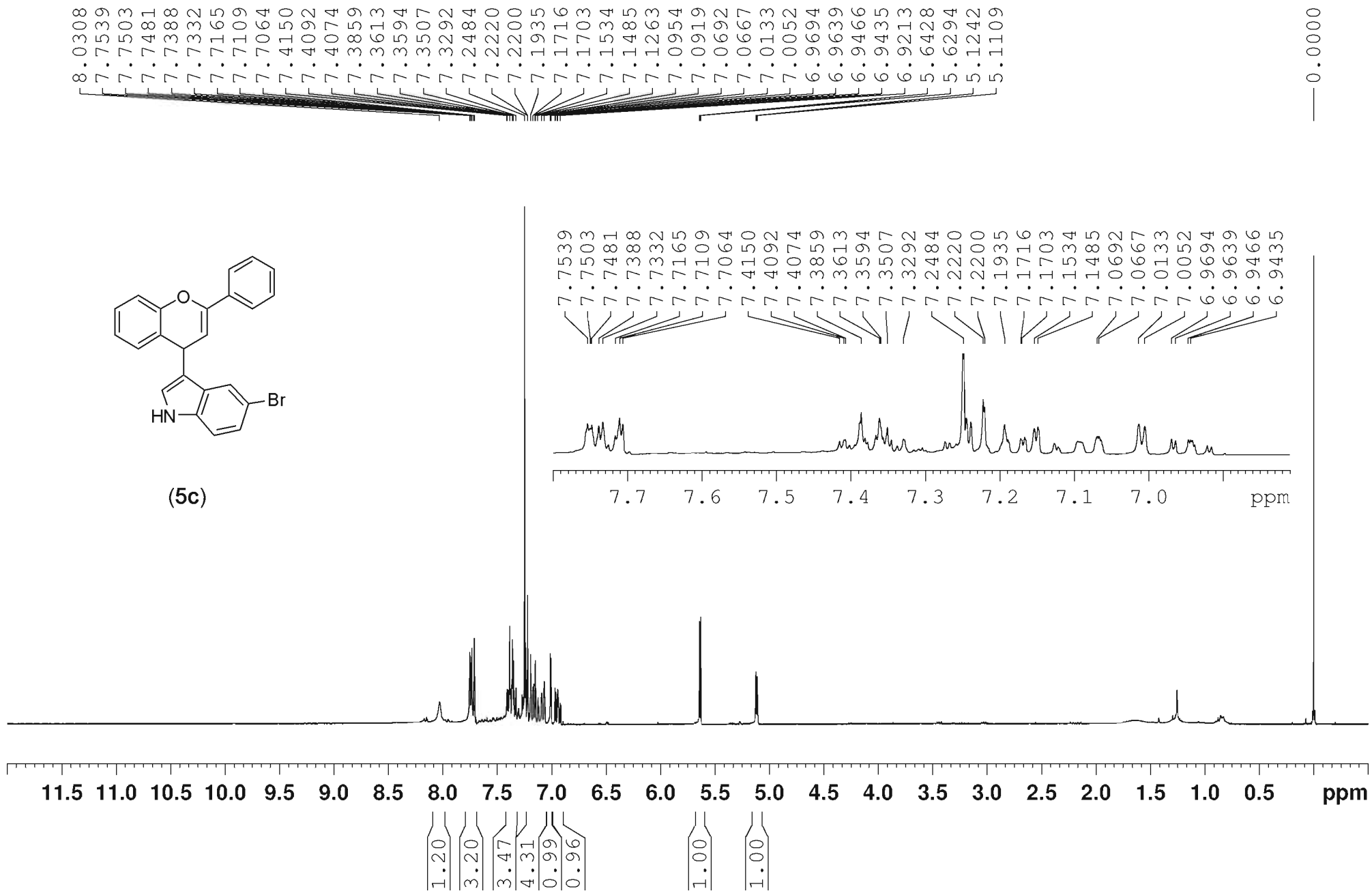


(5b)



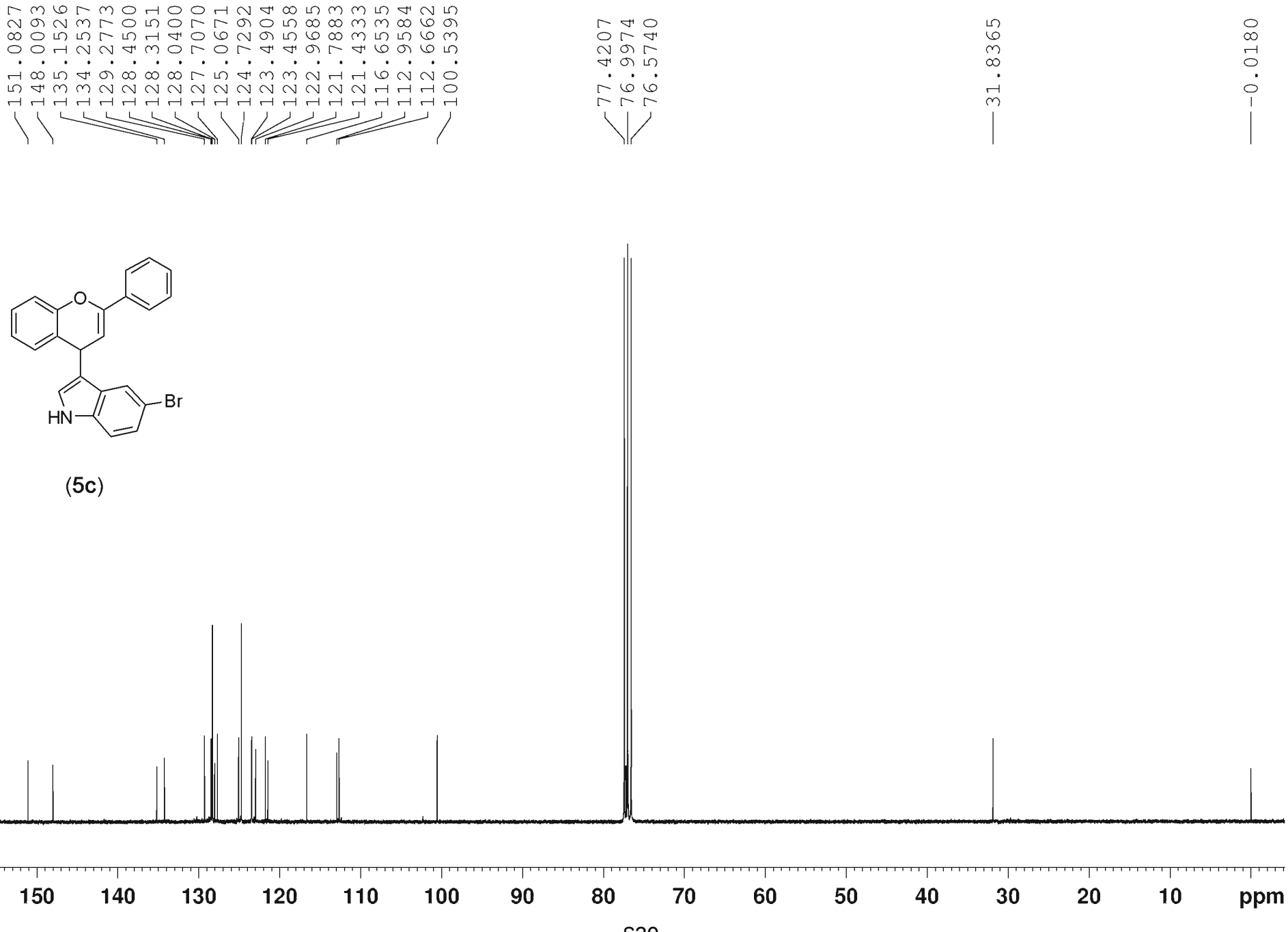
This journal is © The Royal Society of Chemistry 2012

^{1}H NMR (300 MHz, $CDCl_3$)



This journal is © The Royal Society of Chemistry 2012

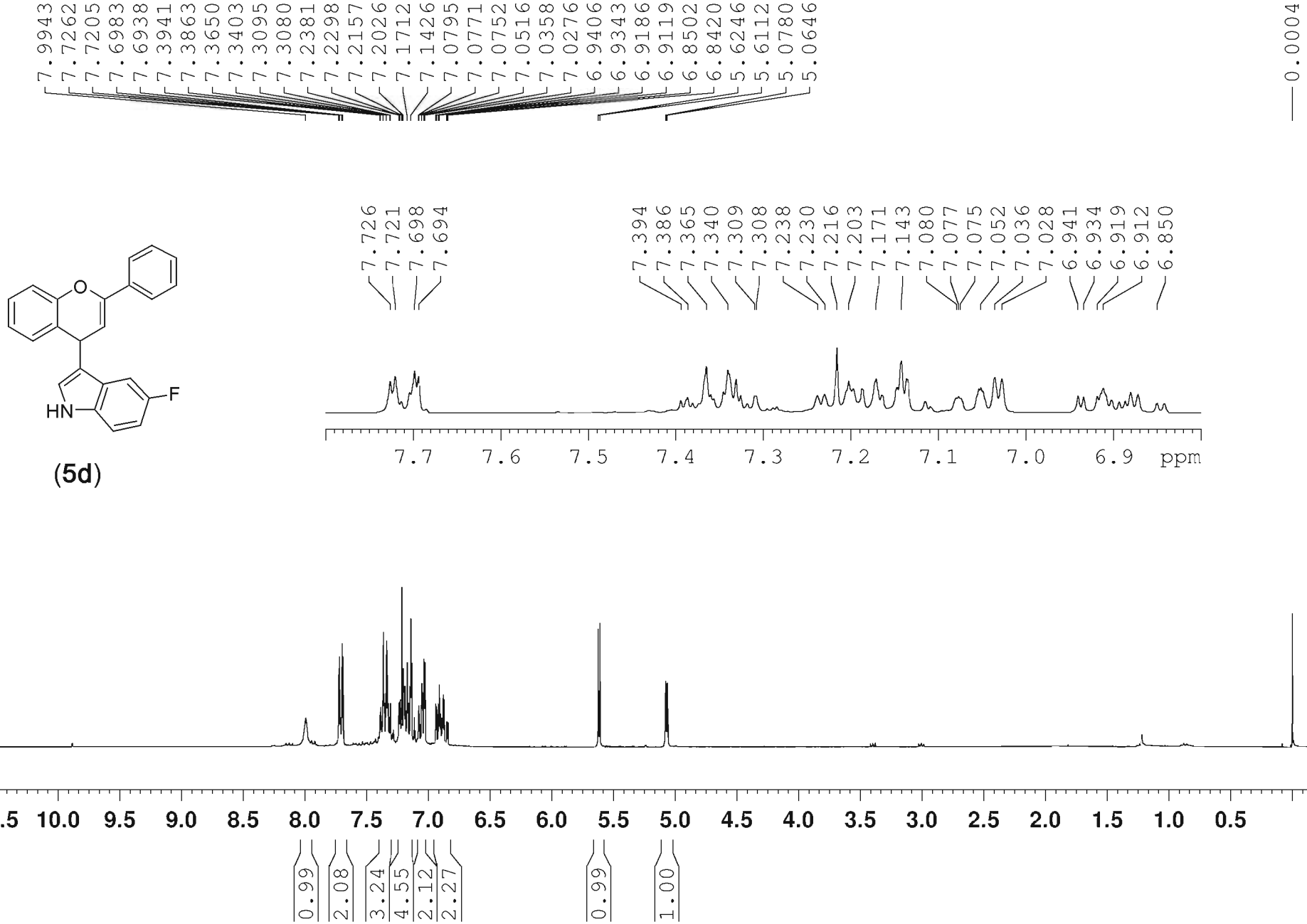
^{13}C NMR (75 MHz, $CDCl_3$)



(5c)

This journal is © The Royal Society of Chemistry 2012

^{1}H NMR (300MHz, $CDCl_3$)

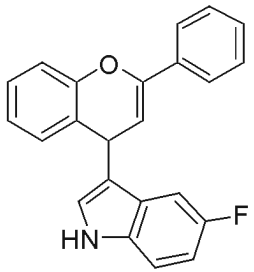


(5d)

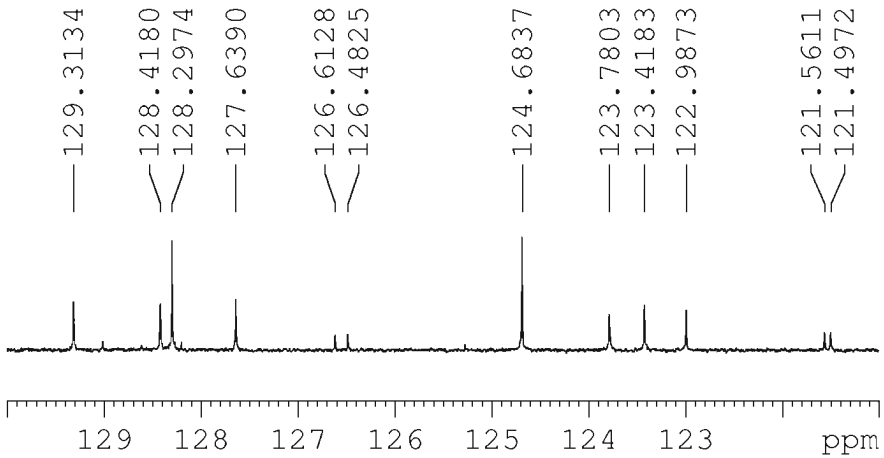
This journal is © The Royal Society of Chemistry 2012

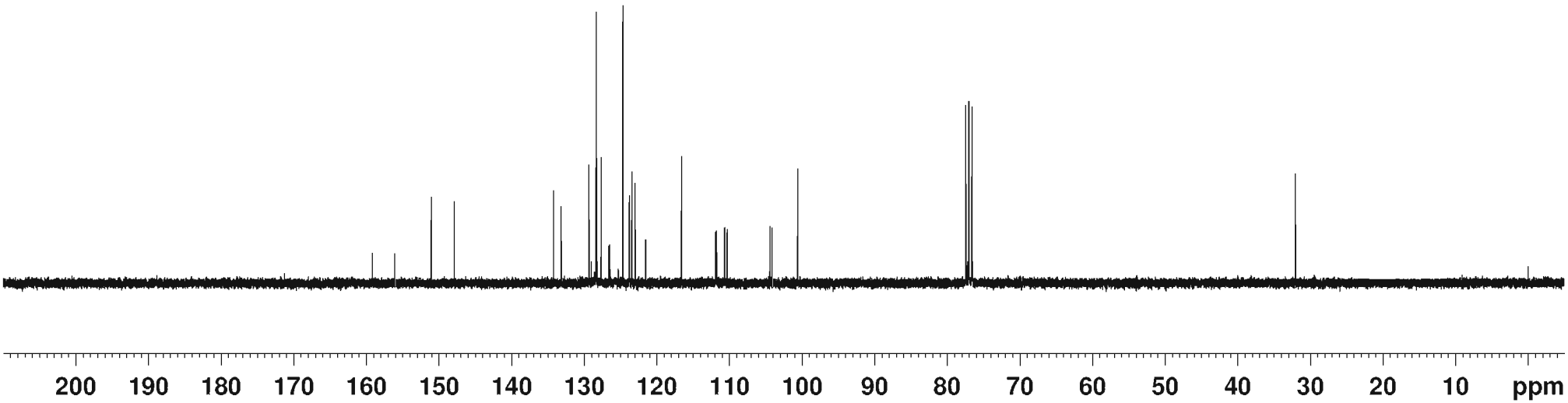
^{13}C NMR (75MHz, $CDCl_3$)

159.1715
156.0631
151.0660
147.9125
134.2255
133.1341
129.3134
128.4180
128.2974
127.6390
126.6128
126.4825
124.6837
123.7803
123.4183
122.9873
121.5611
121.4972
116.5850
111.8912
111.7622
110.6708
110.3201
104.3893
104.0760
100.5782
77.4243
77.0007
76.5767
32.0717



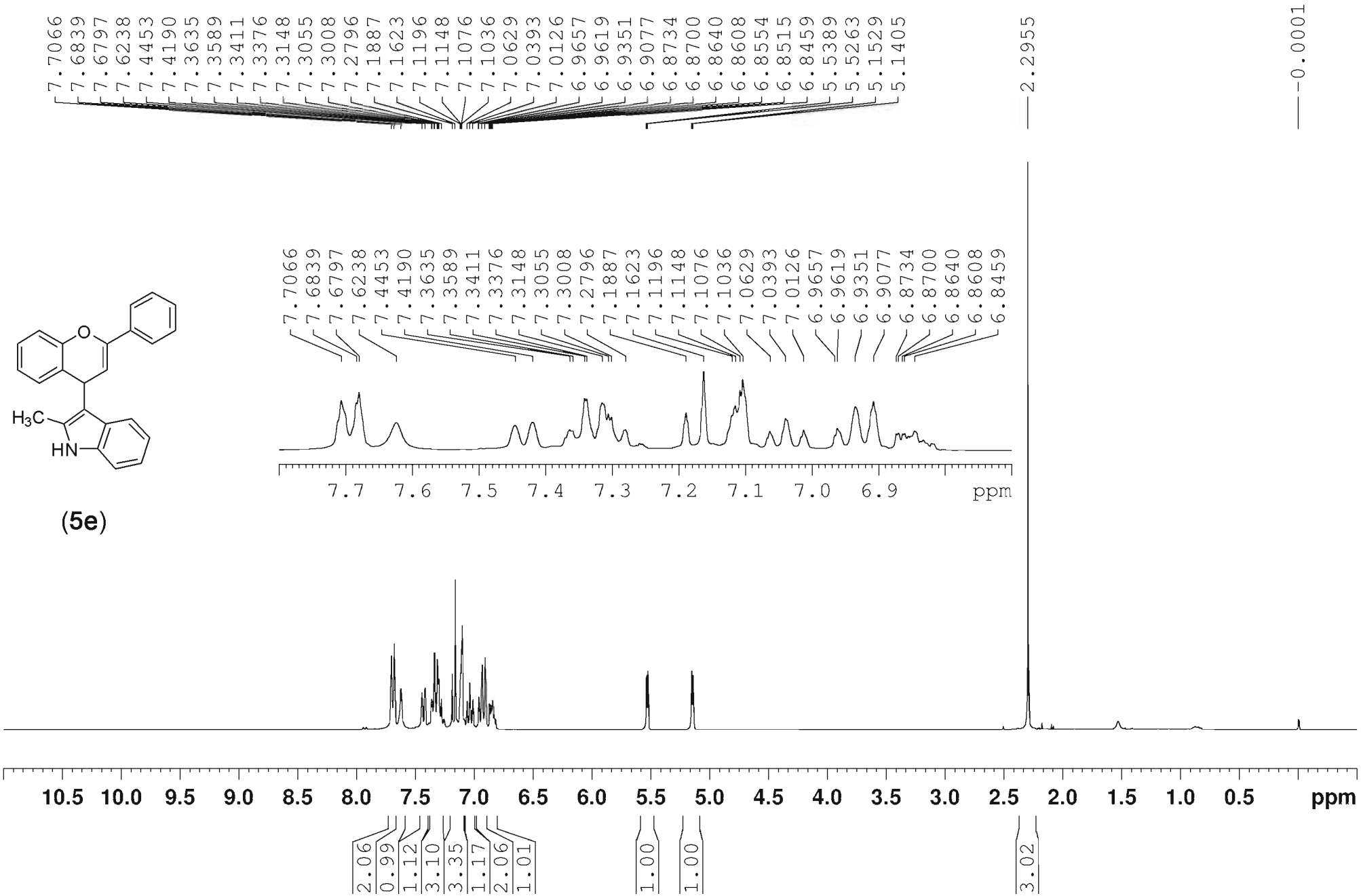
(5d)





This journal is © The Royal Society of Chemistry 2012

^{1}H NMR (300MHz, $CDCl_3$)

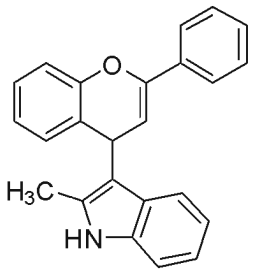


(5e)

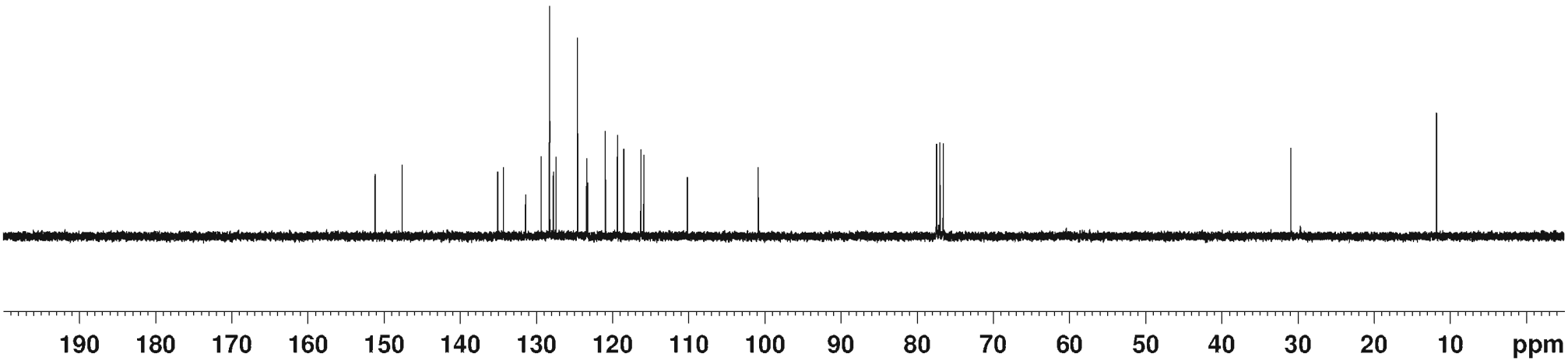
This journal is © The Royal Society of Chemistry 2012

^{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

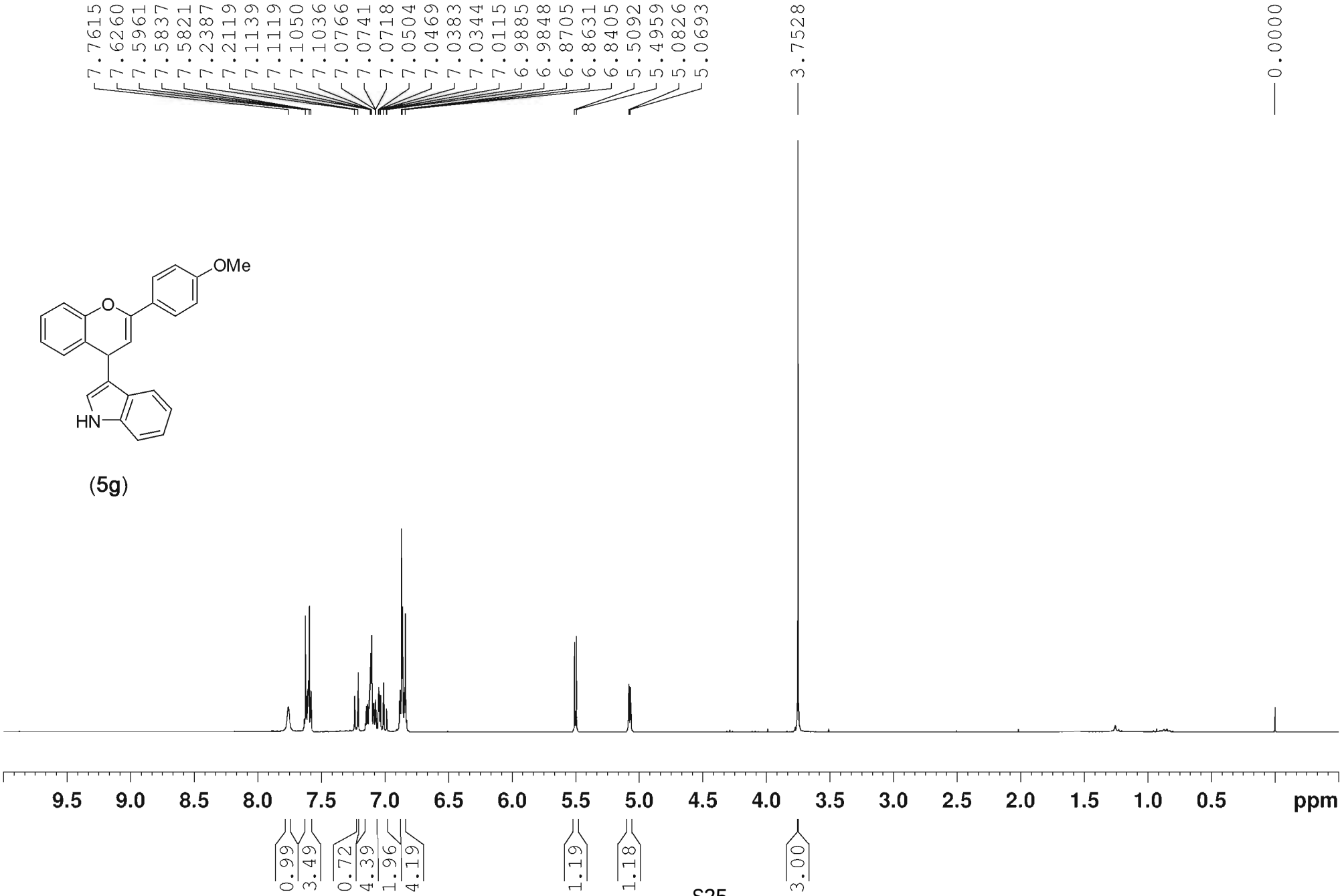


(5e)



This journal is © The Royal Society of Chemistry 2012

^{1}H NMR (300MHz, $CDCl_3$)

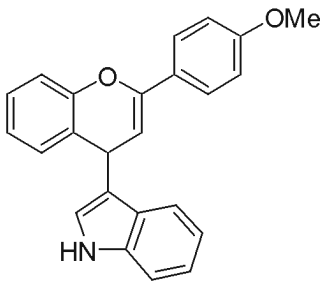


(5g)

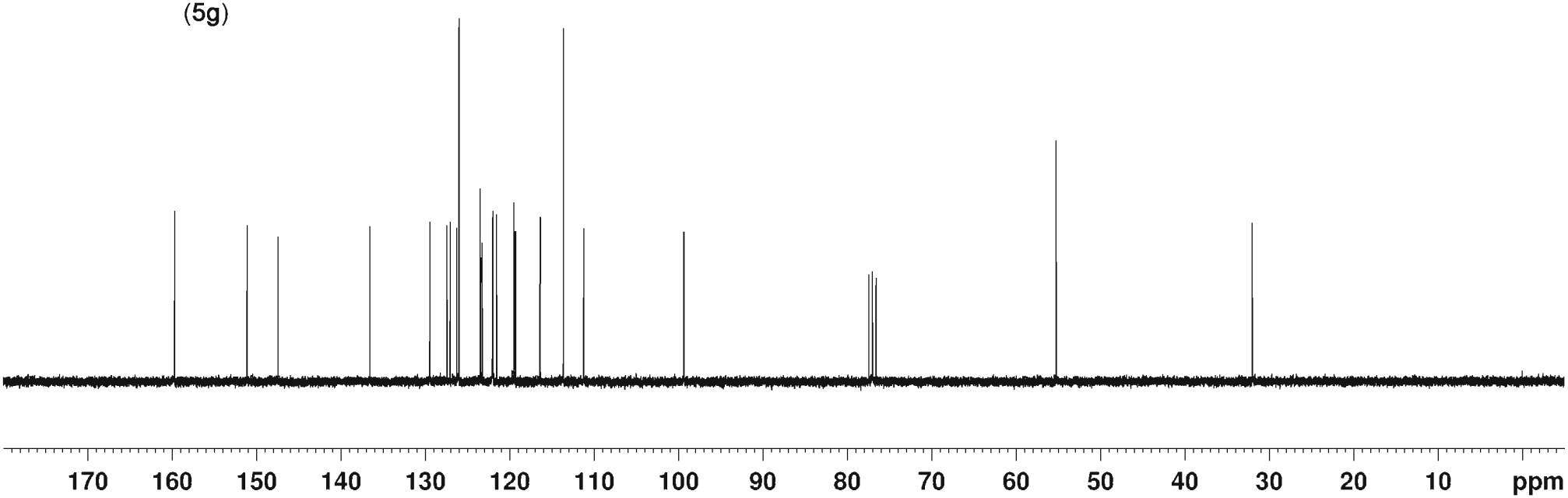
This journal is © The Royal Society of Chemistry 2012

^{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

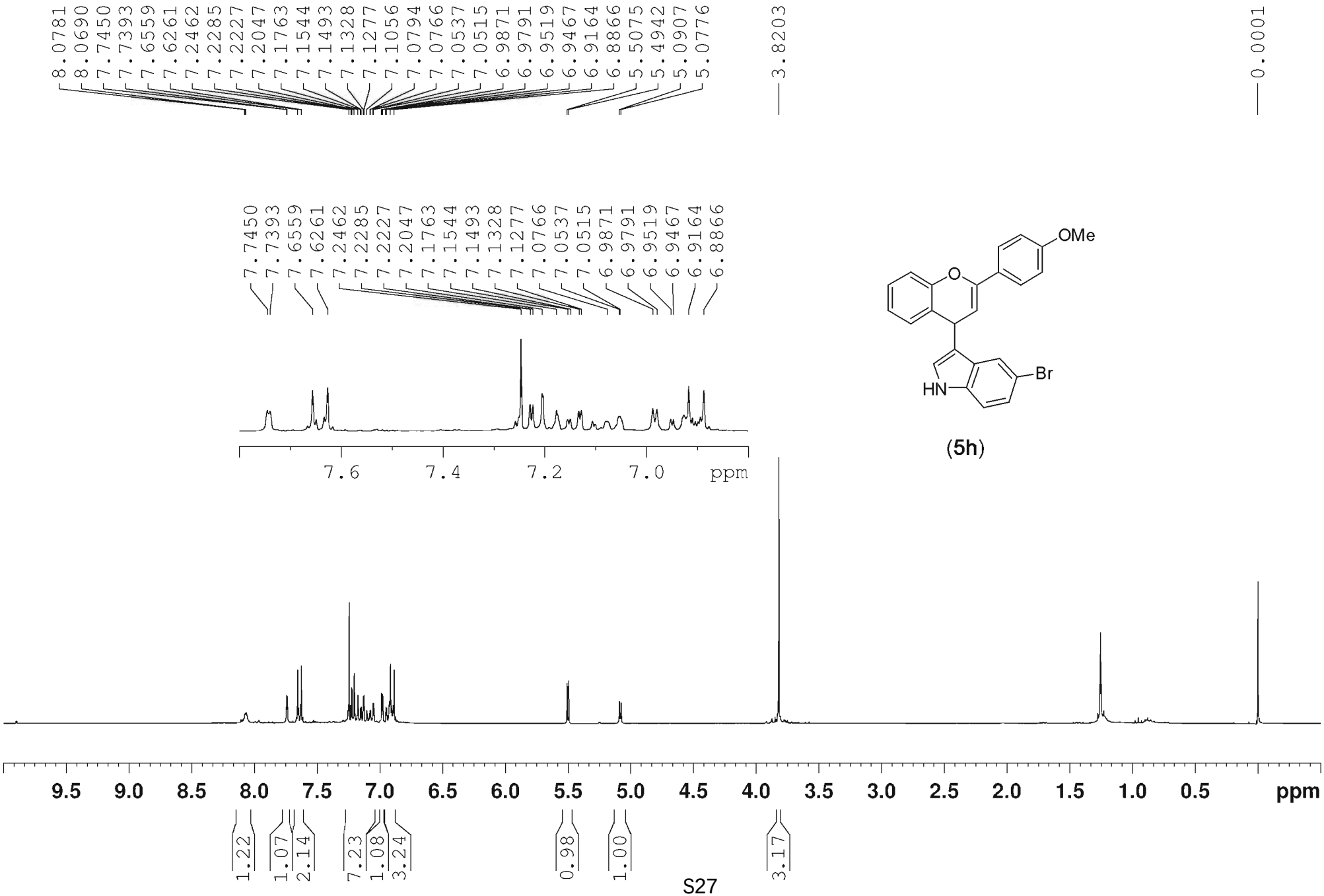


(5g)



This journal is © The Royal Society of Chemistry 2012

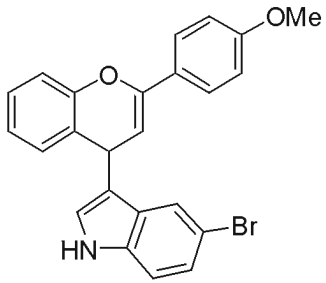
^{1}H NMR (300MHz, $CDCl_3$)



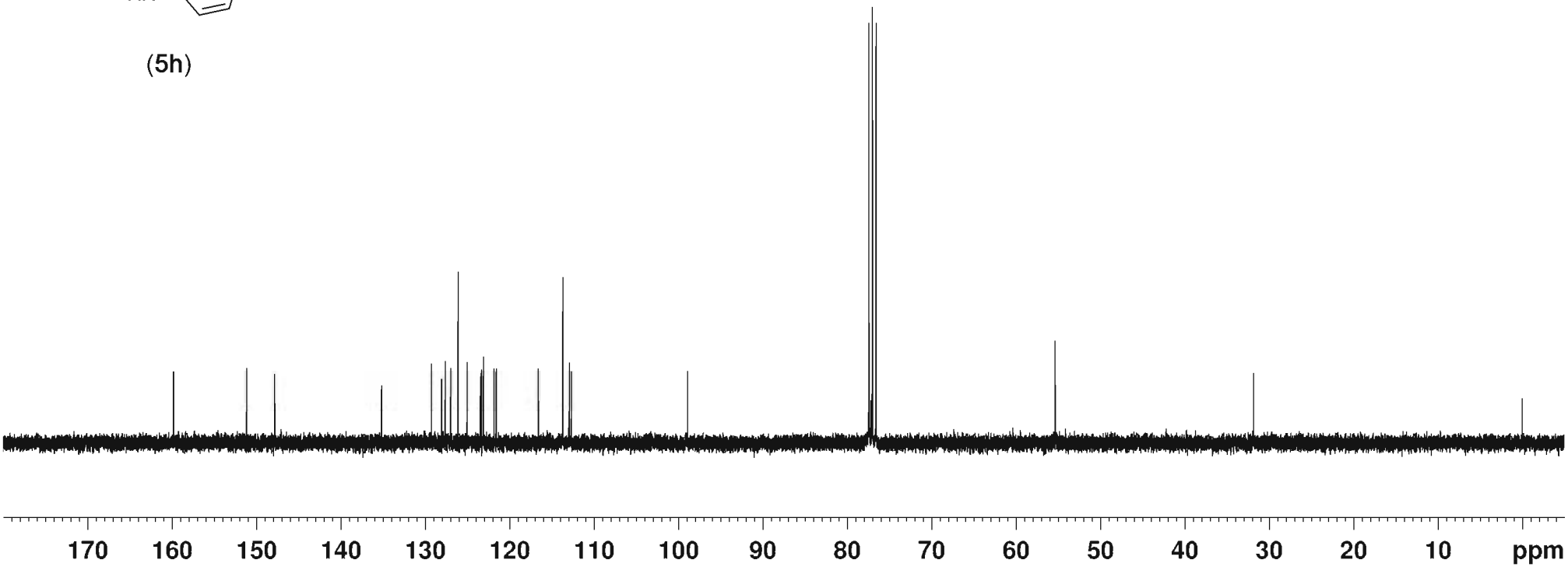
This journal is © The Royal Society of Chemistry 2012

^{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
123.3505
123.1107
121.8174
121.5905
116.6107
113.6989
112.9065
112.6663
98.9113
77.4223
76.9990
76.5758
55.3150
31.8282

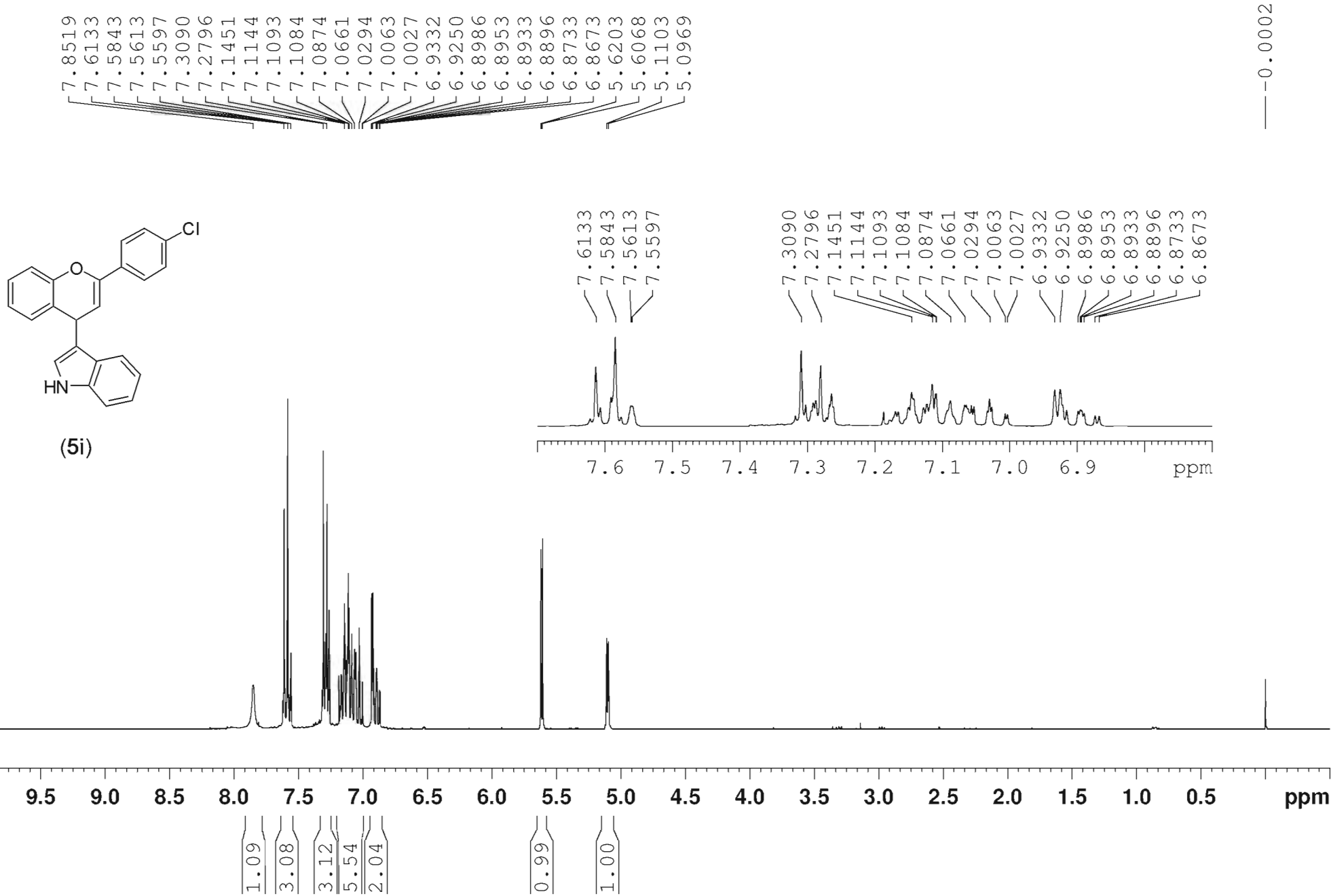


(5h)



This journal is © The Royal Society of Chemistry 2012

^{1}H NMR (300MHz, $CDCl_3$)



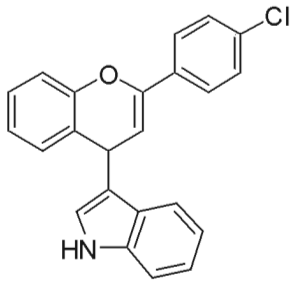
This journal is © The Royal Society of Chemistry 2012

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

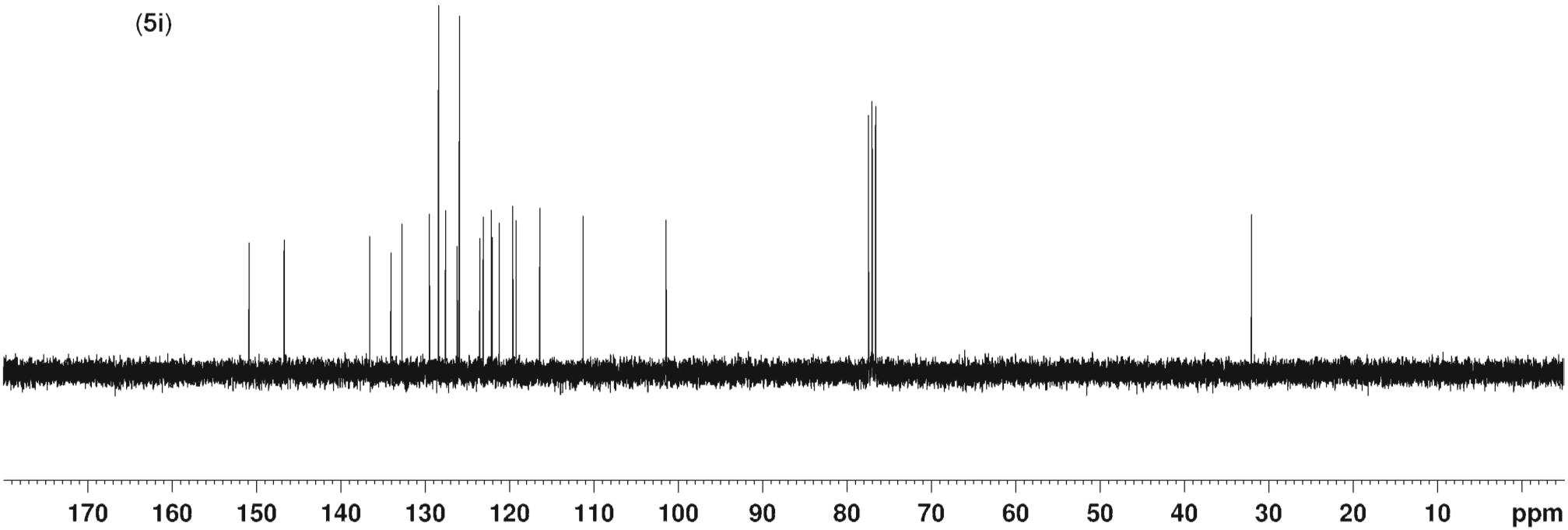
150.8683
146.7054
136.5410
134.0328
132.7369
129.4671
128.4006
127.5699
126.1742
125.9133
123.5003
123.1077
122.1285
122.0387
121.1840
119.6041
119.2069
116.3932
111.2468
101.4113

77.4141
76.9906
76.5682

32.0047

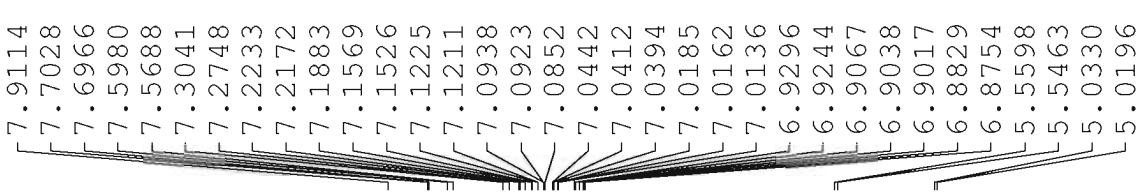


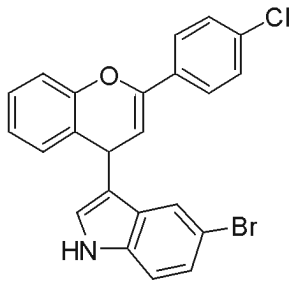
(5i)



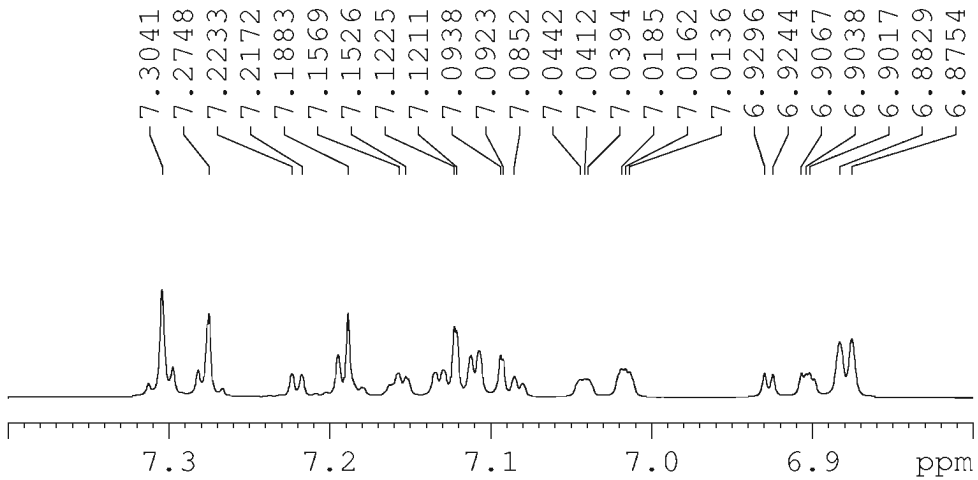
This journal is © The Royal Society of Chemistry 2012

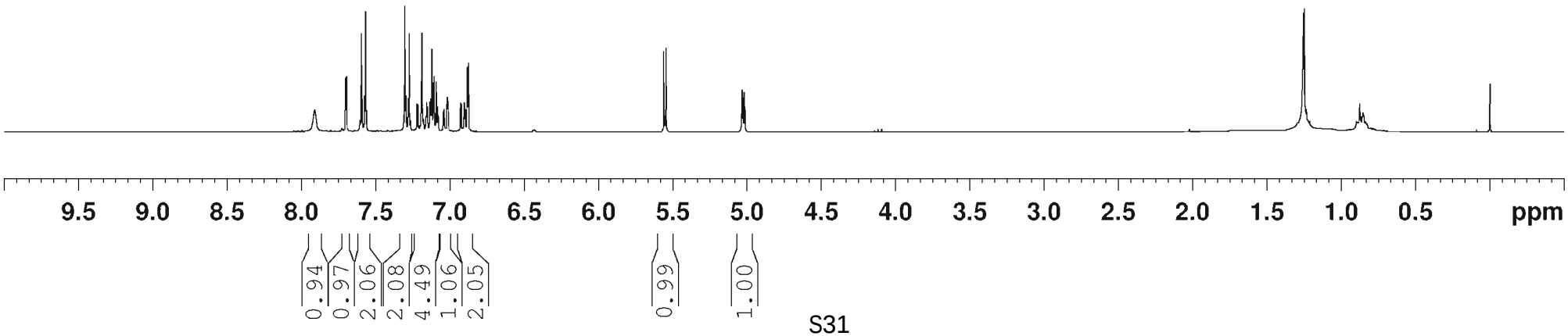
^{1}H NMR (300MHz, $CDCl_3$)





(5j)





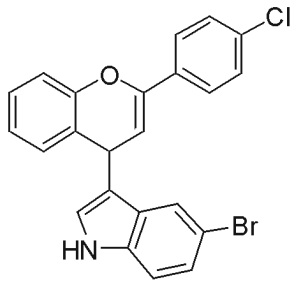
This journal is © The Royal Society of Chemistry 2012

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

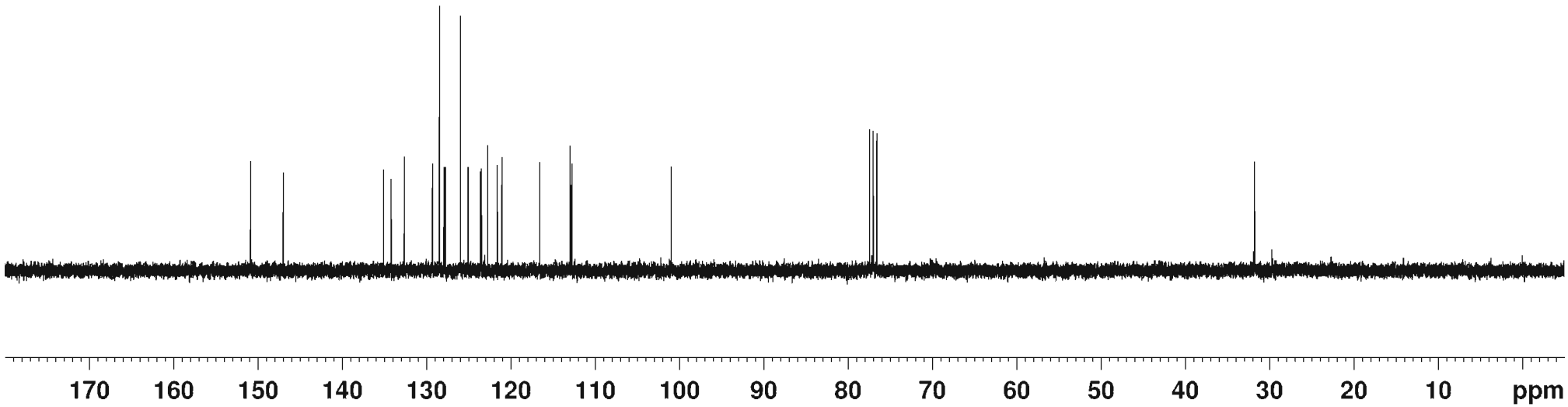
150.8410
146.9827
135.0855
134.1630
132.6034
129.2740
128.4388
127.9057
127.7629
125.9354
125.0355
123.5992
123.4676
122.7407
121.6150
121.0389
116.5553
112.9320
112.7217
100.9449

77.4242
77.0007
76.5764

31.7502

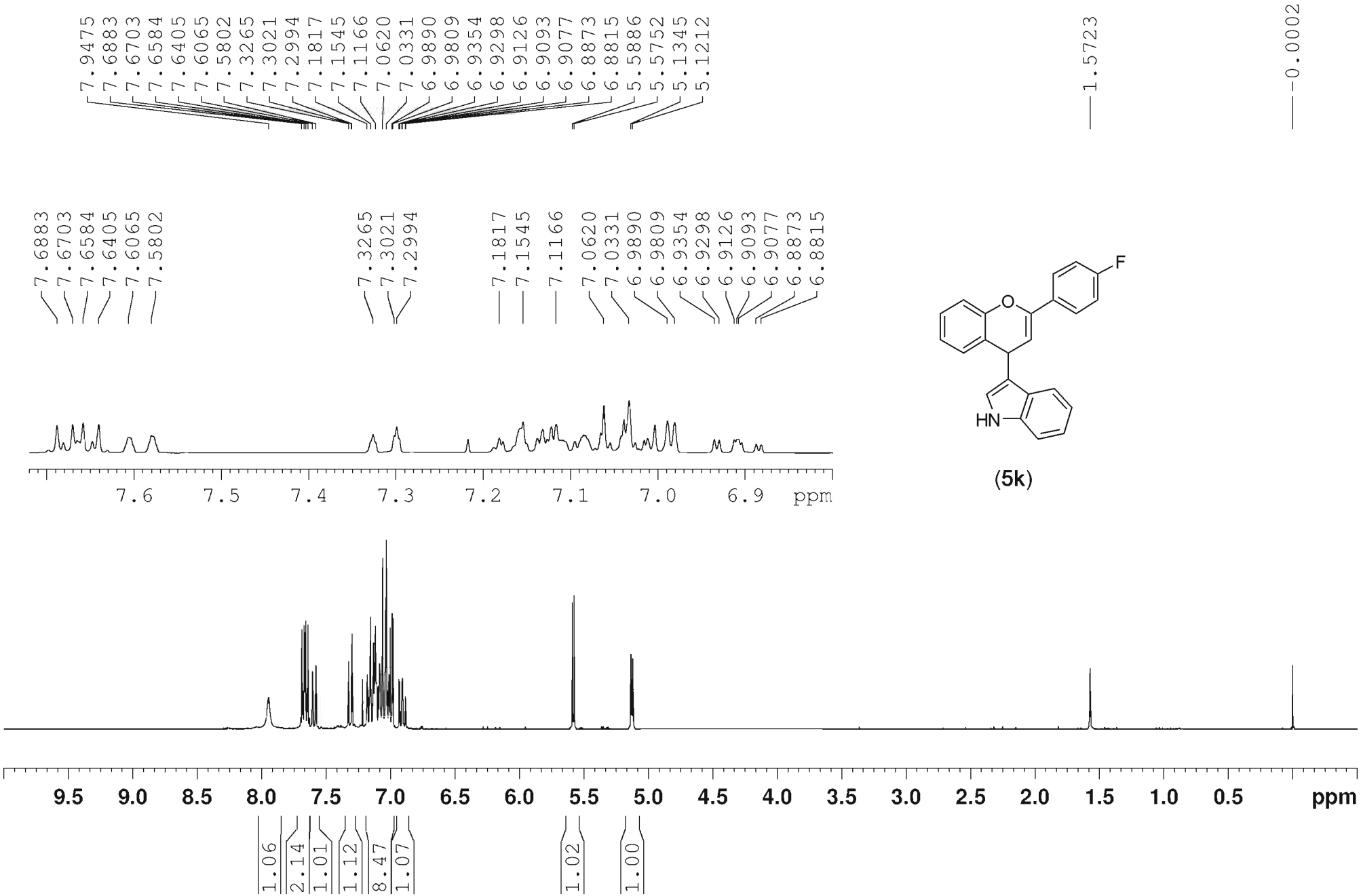


(5j)



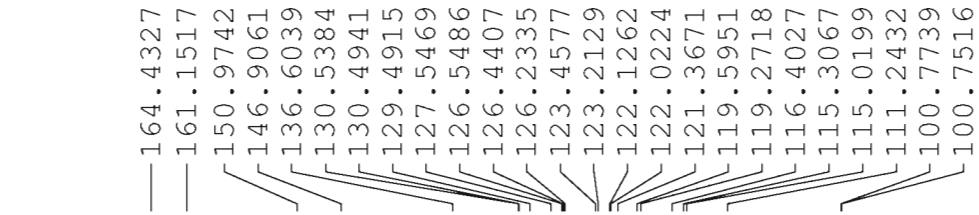
This journal is © The Royal Society of Chemistry 2012

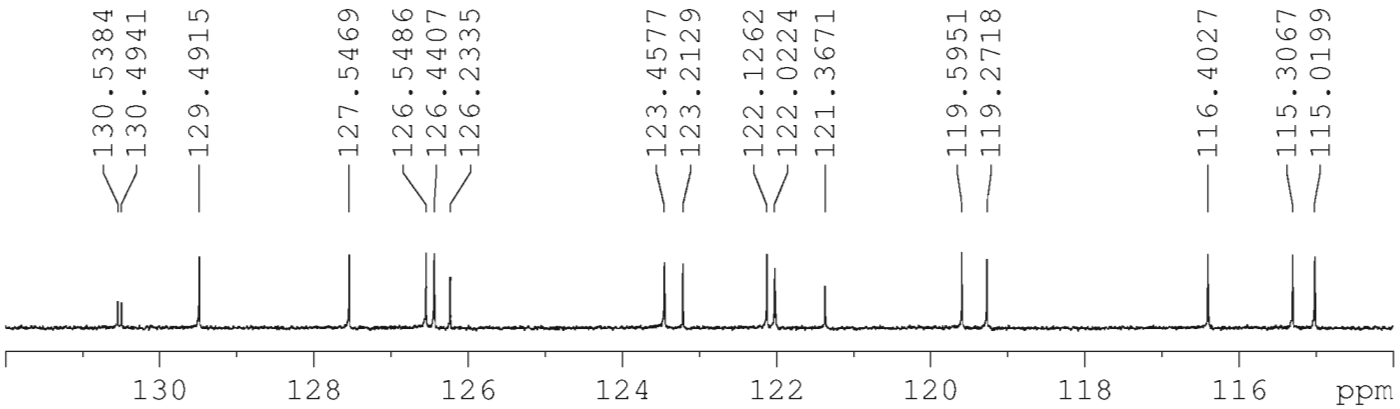
^{1}H NMR (300MHz, $CDCl_3$)

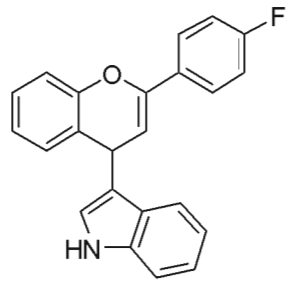


This journal is © The Royal Society of Chemistry 2012

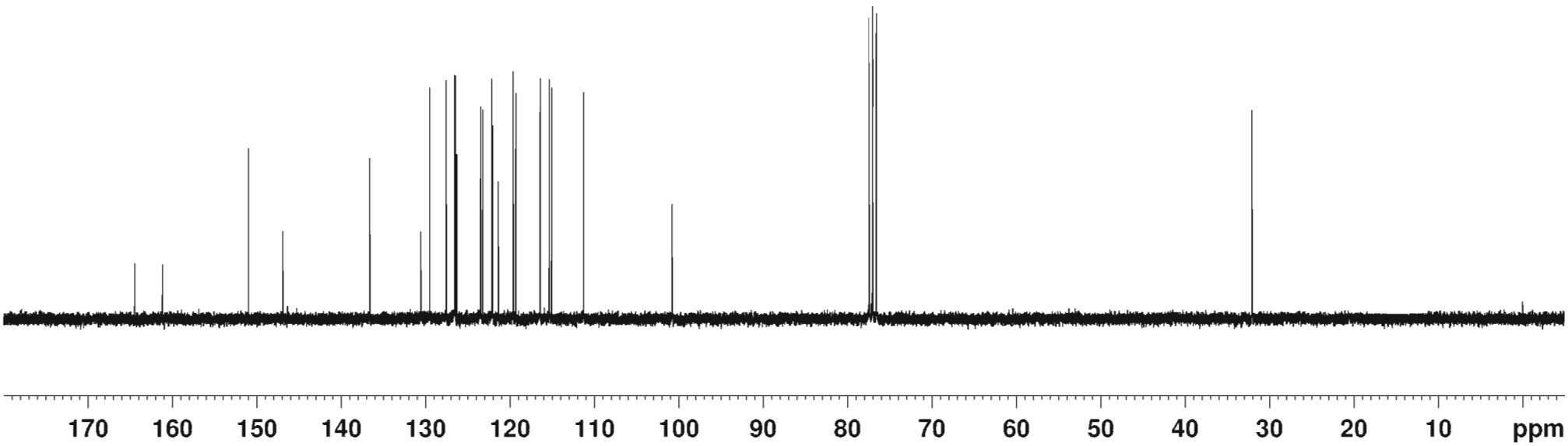
^{13}C NMR (75 MHz, $CDCl_3$)





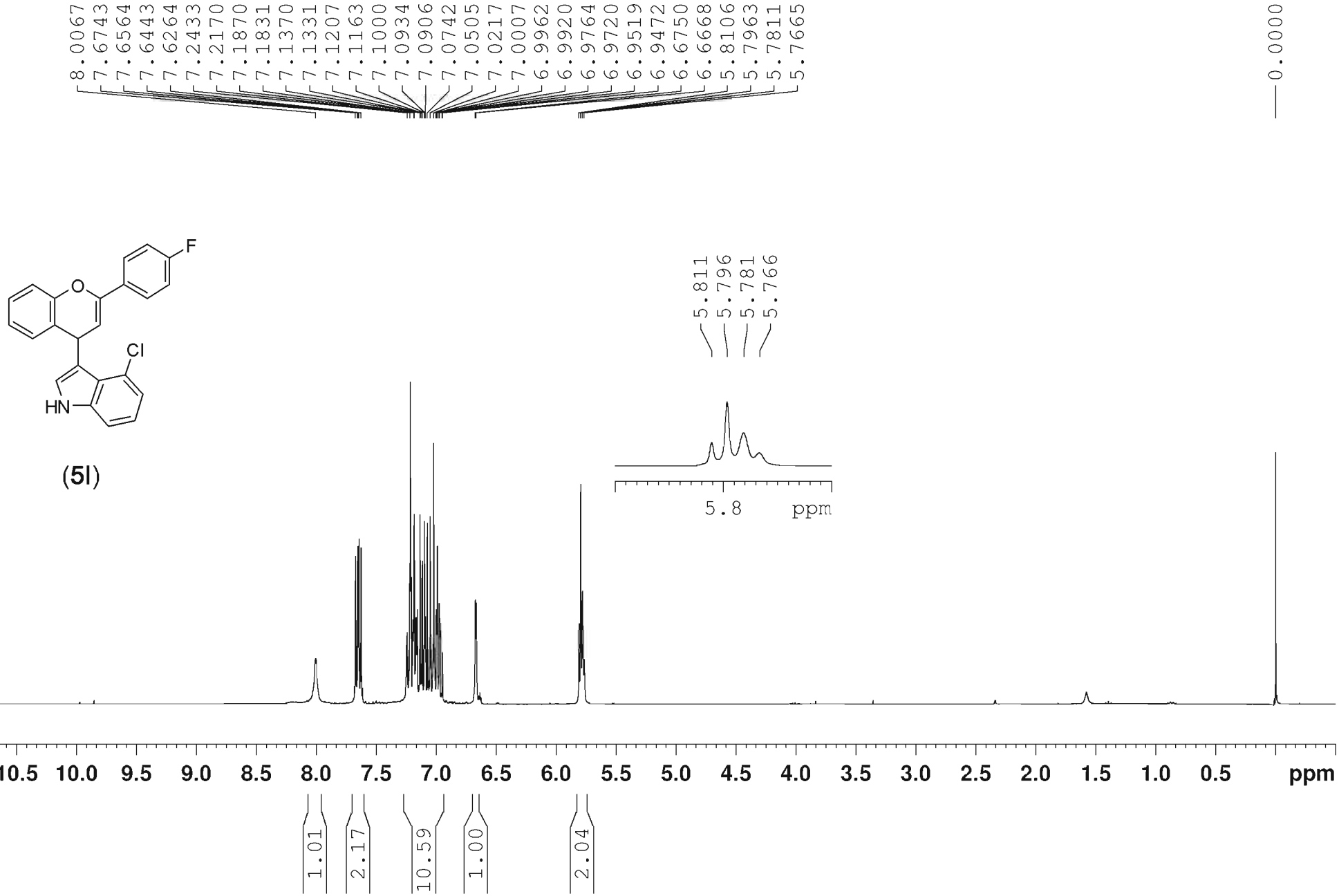


(5k)



This journal is © The Royal Society of Chemistry 2012

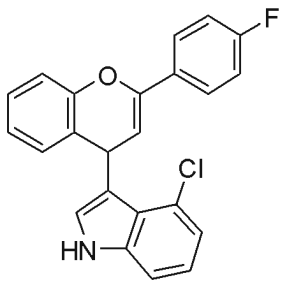
^{1}H NMR (300MHz, $CDCl_3$)



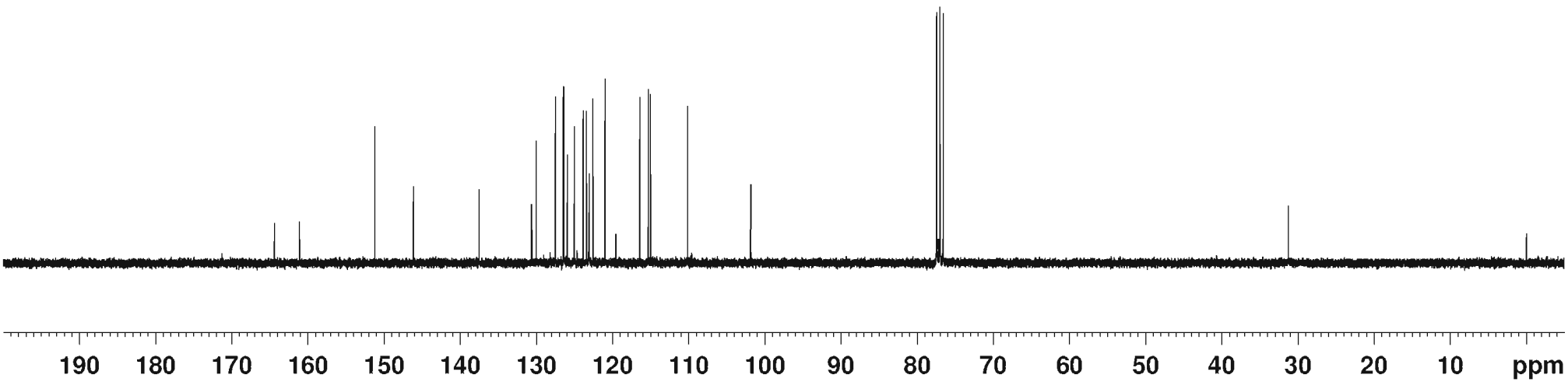
This journal is © The Royal Society of Chemistry 2012

^{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

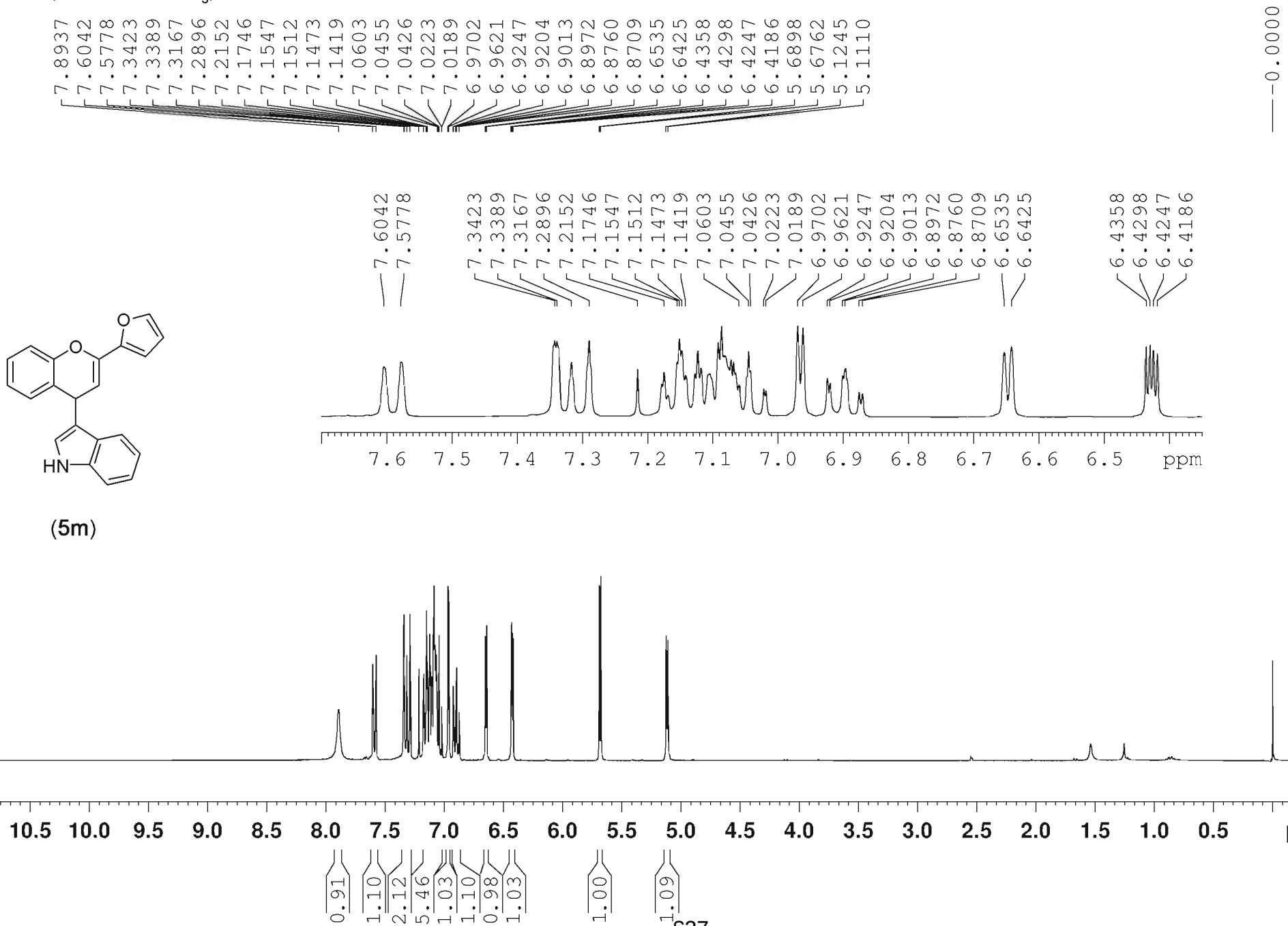


(5l)



This journal is © The Royal Society of Chemistry 2012

^{1}H NMR (300MHz, $CDCl_3$)



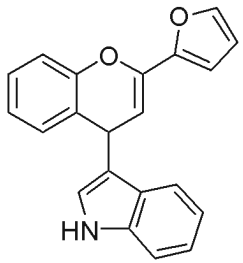
This journal is © The Royal Society of Chemistry 2012

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

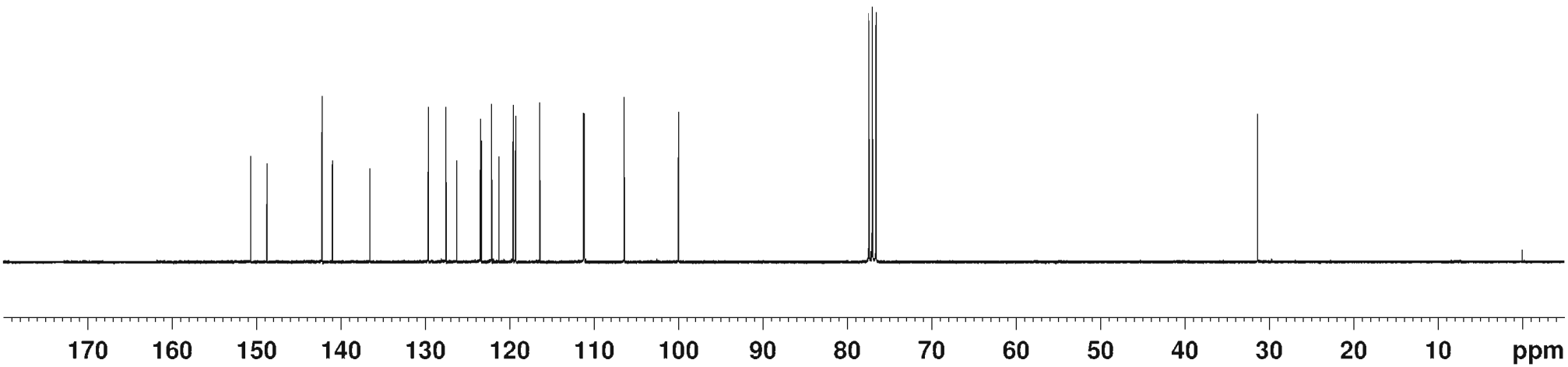
150.6757
148.7455
142.2269
140.9936
136.5708
129.6208
127.5418
126.2474
123.4548
123.3323
122.1199
122.0961
121.2498
119.5752
119.2777
116.4130
111.2085
111.1317
106.4053
99.9746

77.4241
77.0008
76.5772

31.3717

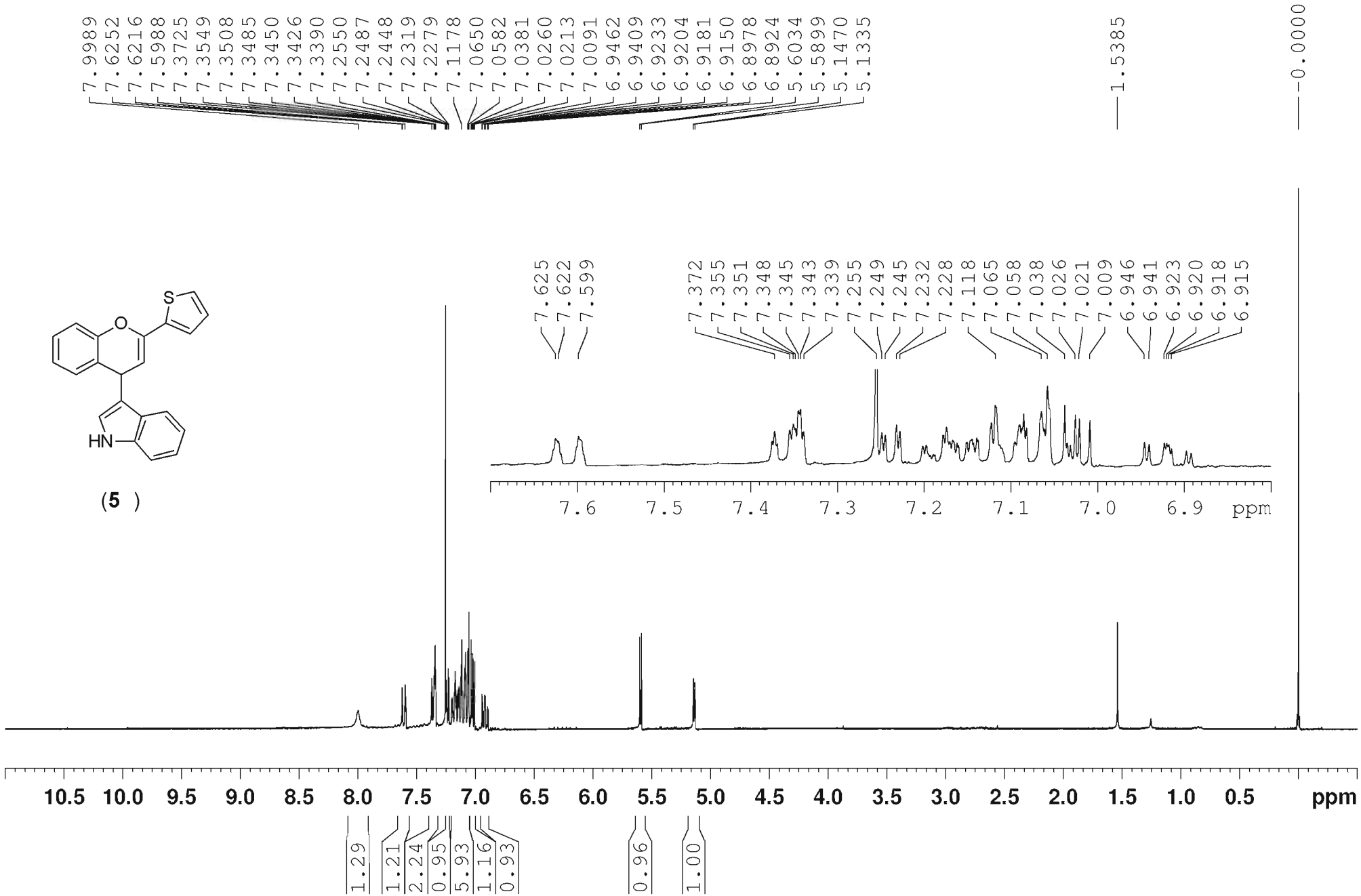


(5m)



This journal is © The Royal Society of Chemistry 2012

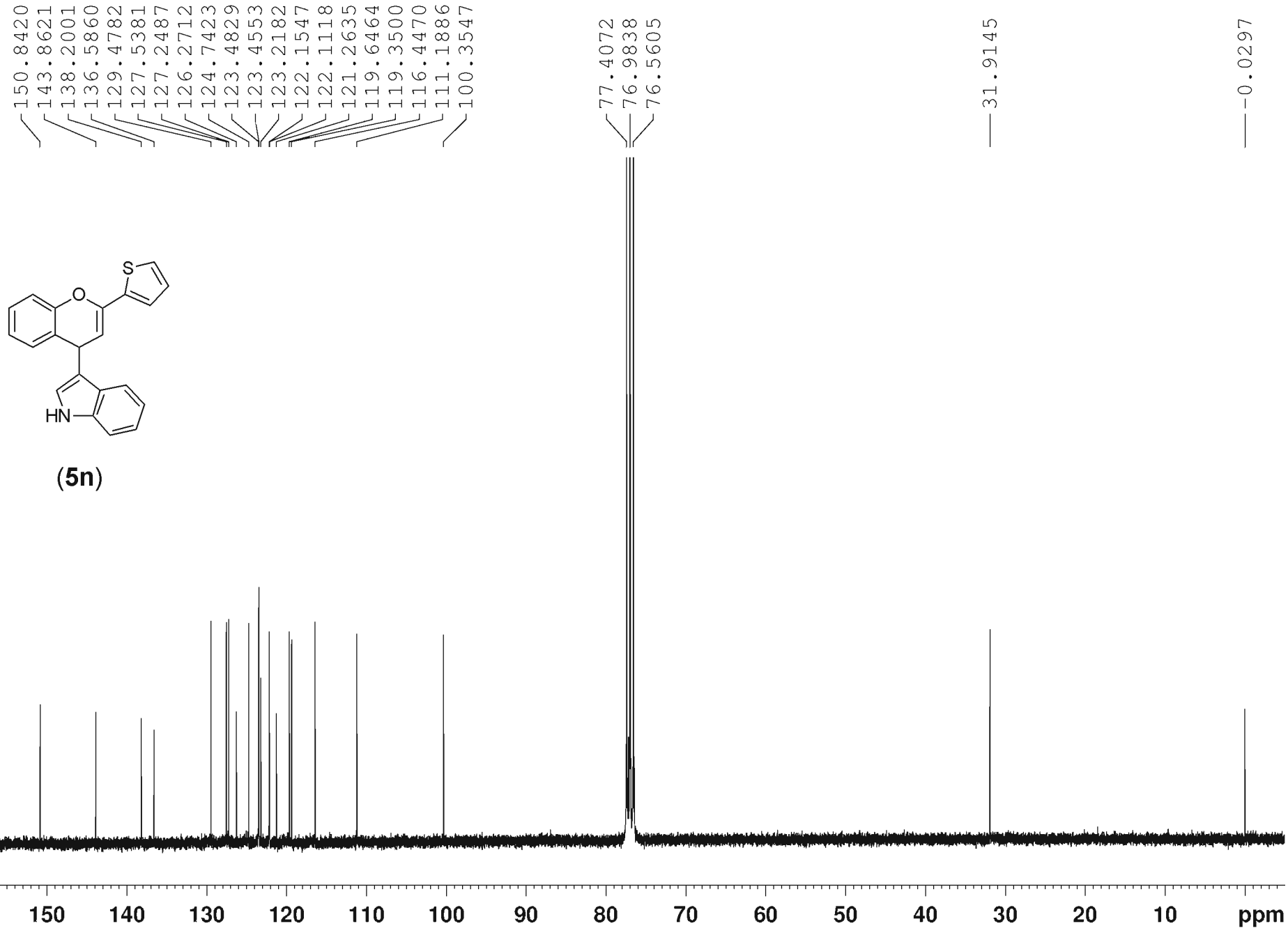
^{1}H NMR (300 MHz, $CDCl_3$)



(**5**)

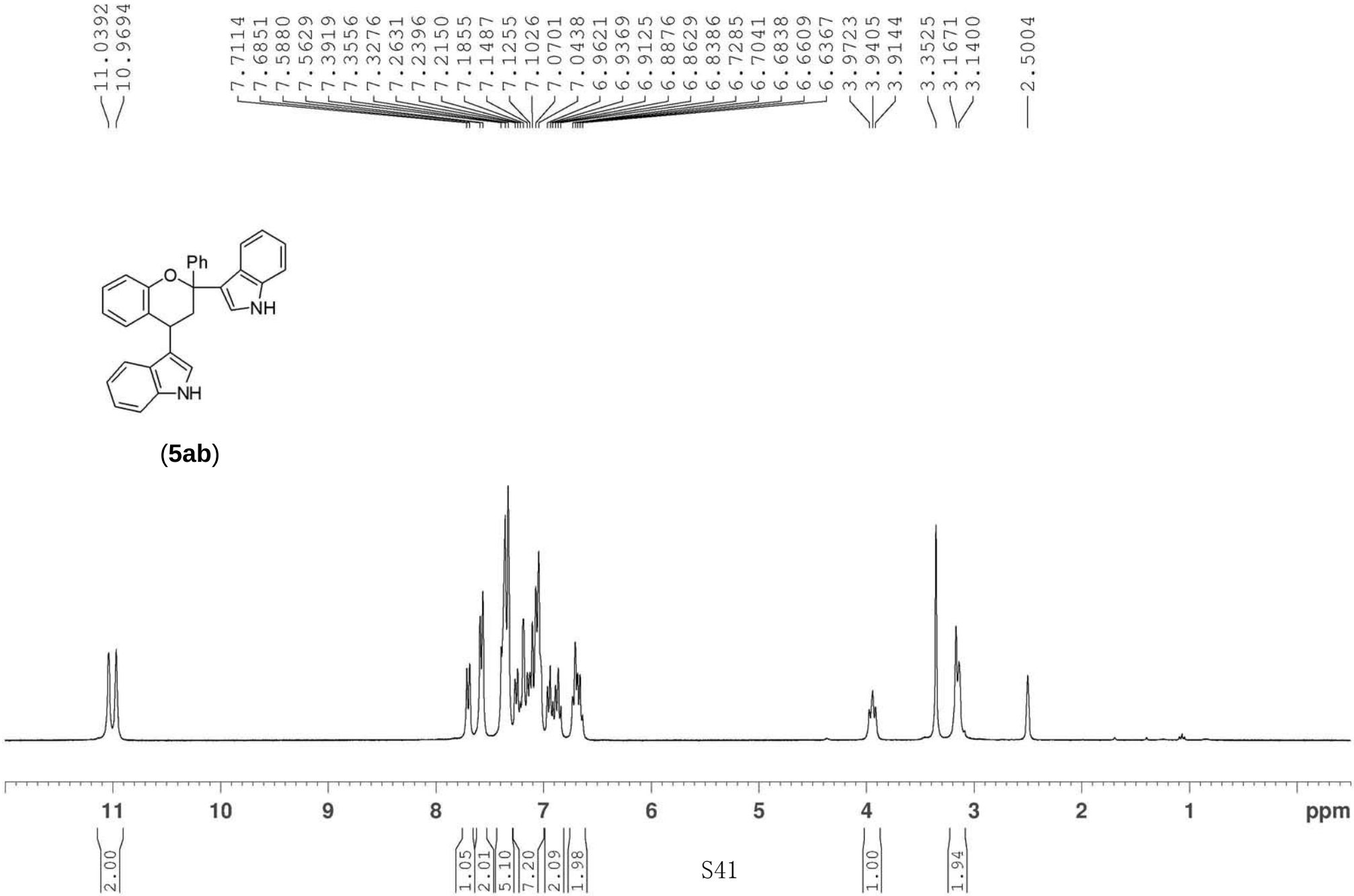
This journal is © The Royal Society of Chemistry 2012

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



(5n)

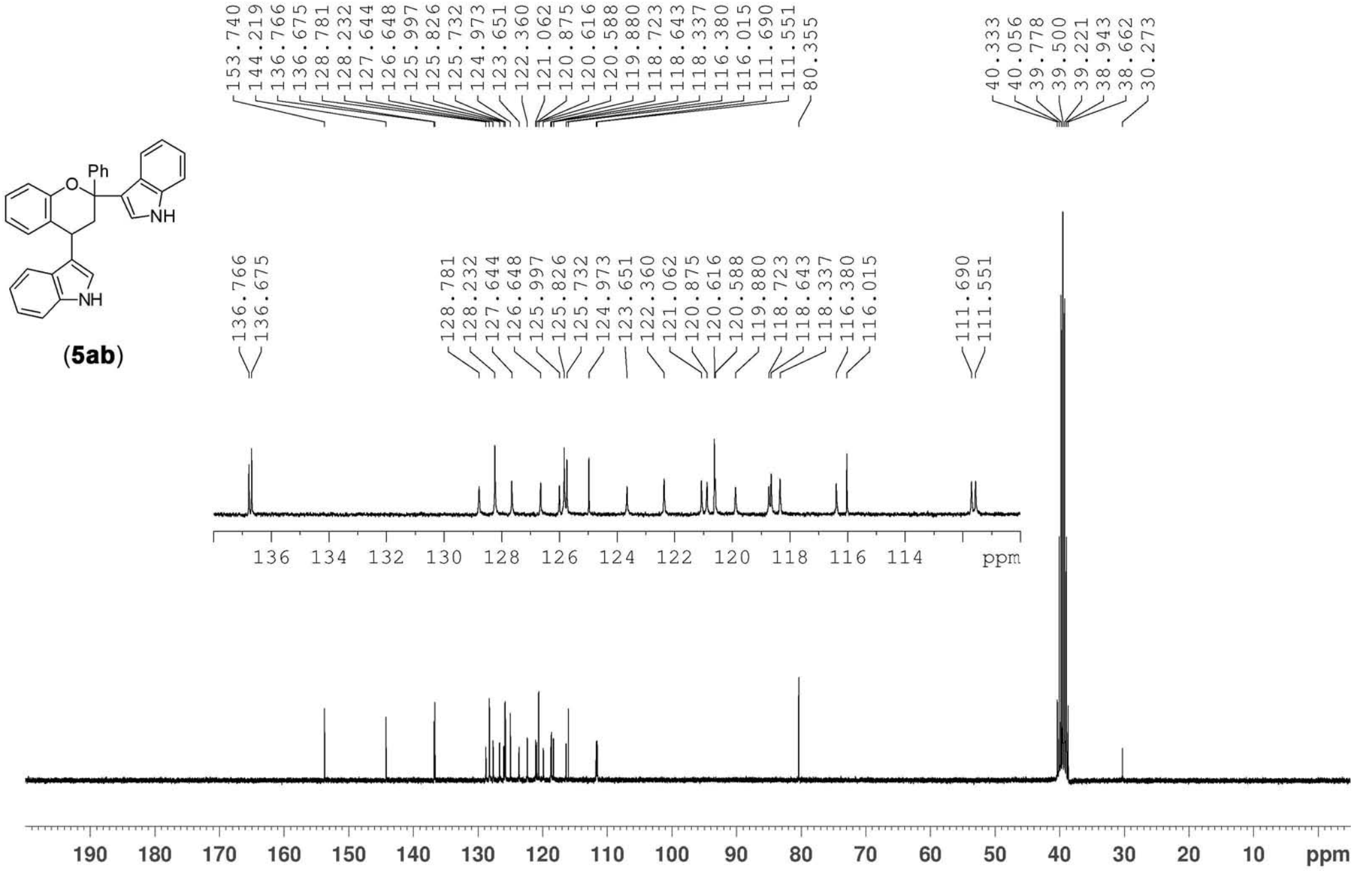
^{1}H NMR (300 MHz, DMSO-d_6)



(**5ab**)

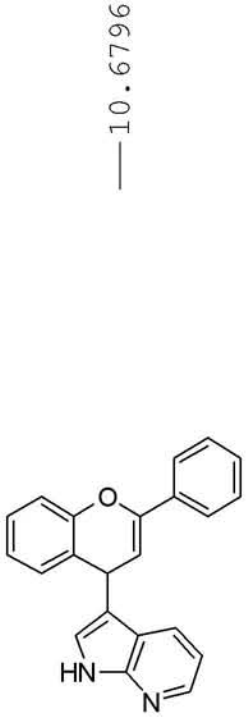
This journal is © The Royal Society of Chemistry 2012

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



This journal is © The Royal Society of Chemistry 2012

^{1}H NMR (300MHz, $CDCl_3$)

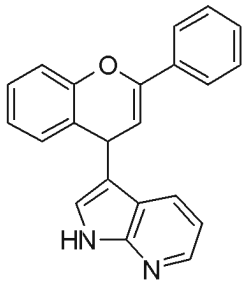


(**7**)

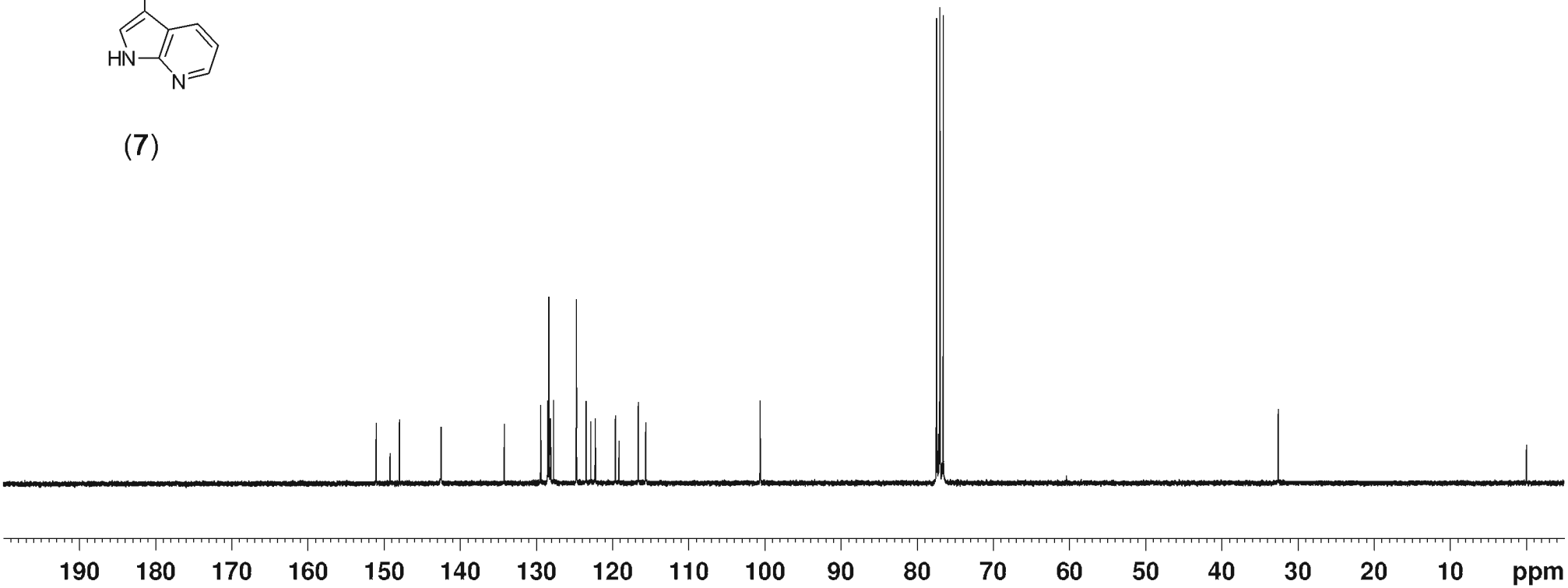
This journal is © The Royal Society of Chemistry 2012

^{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

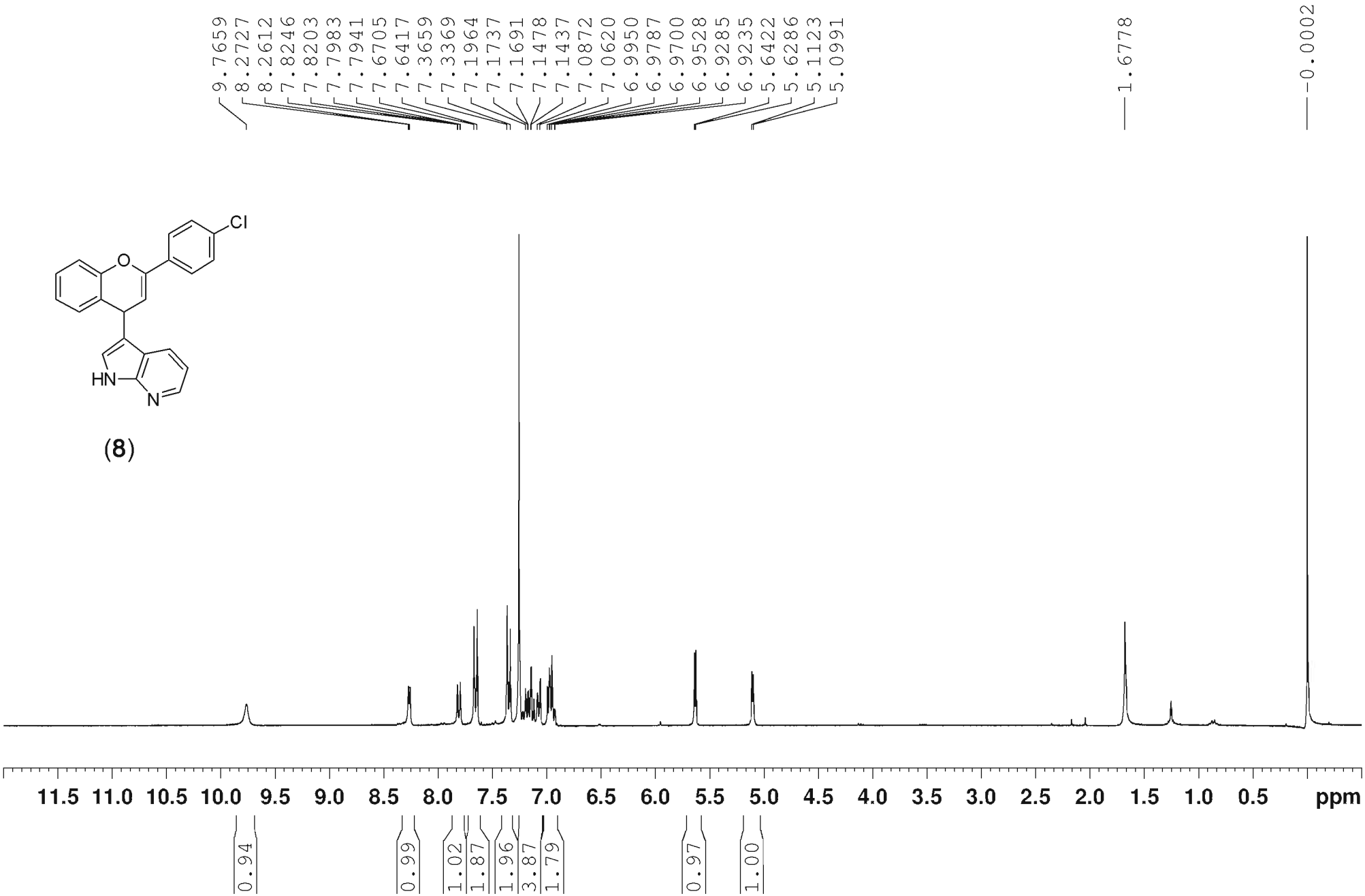


(7)



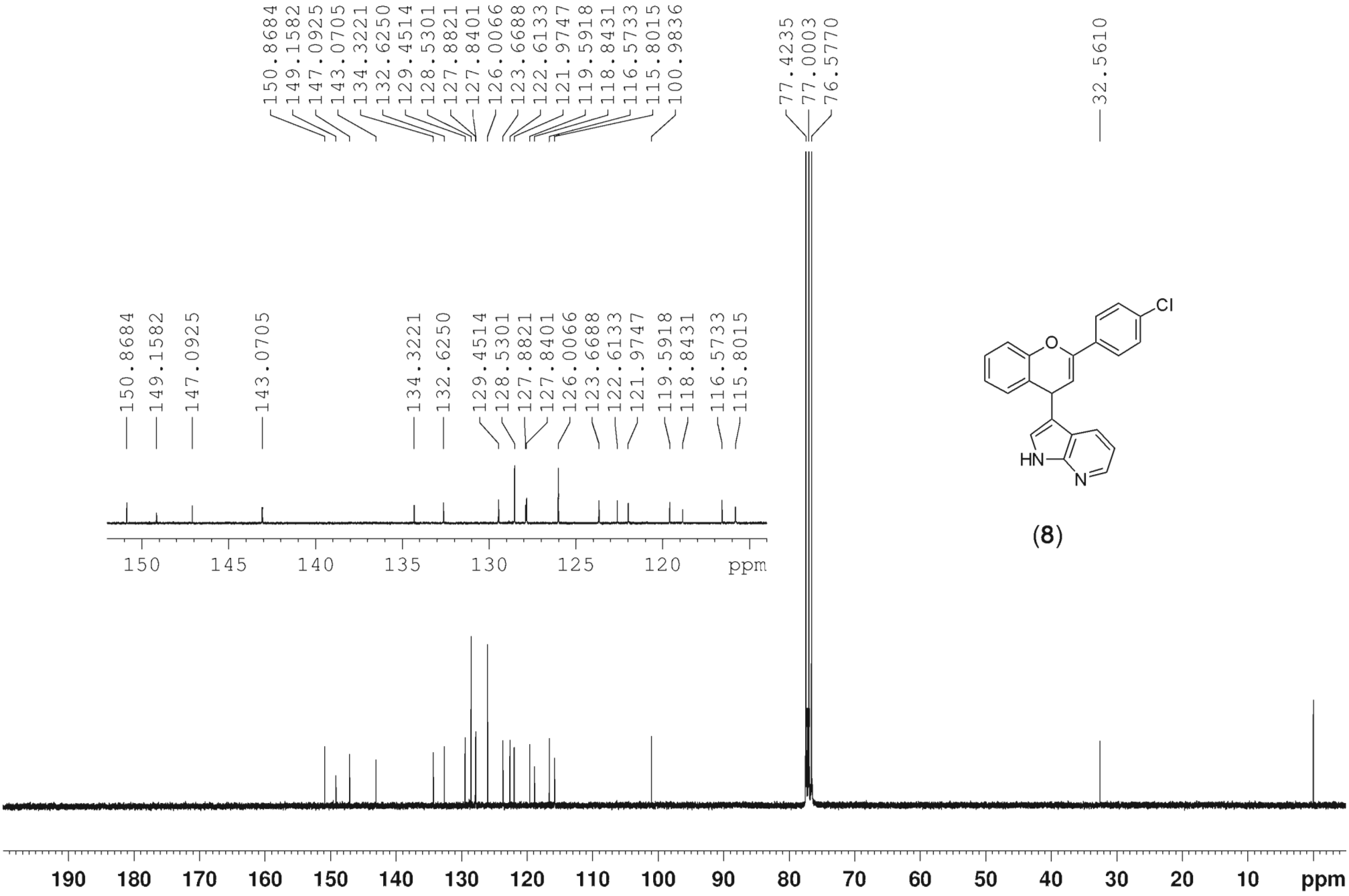
This journal is © The Royal Society of Chemistry 2012

^{1}H NMR (300MHz, $CDCl_3$)



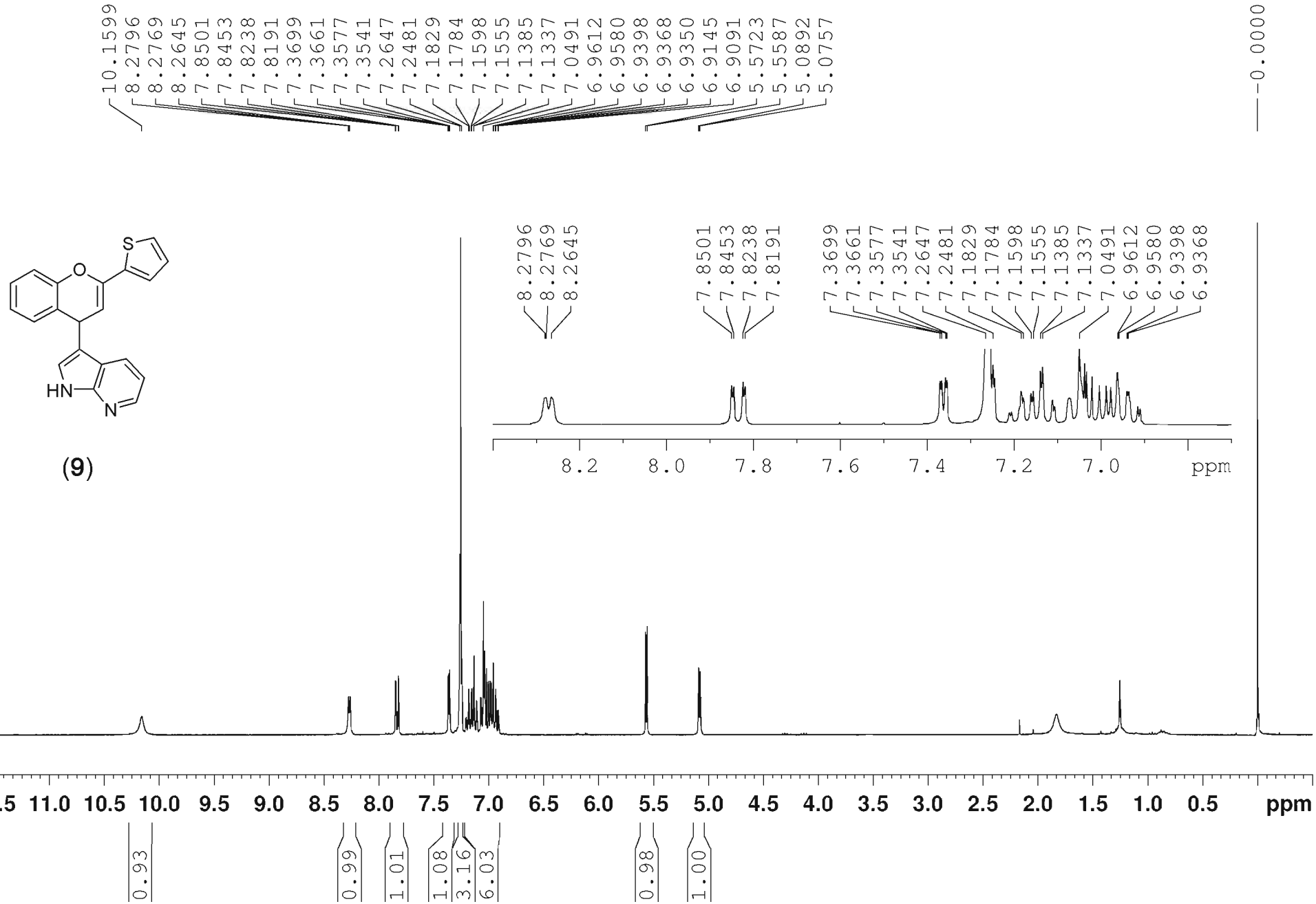
(**8**)

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



This journal is © The Royal Society of Chemistry 2012

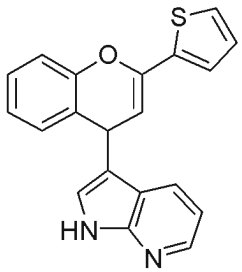
^{1}H NMR (300MHz, $CDCl_3$)



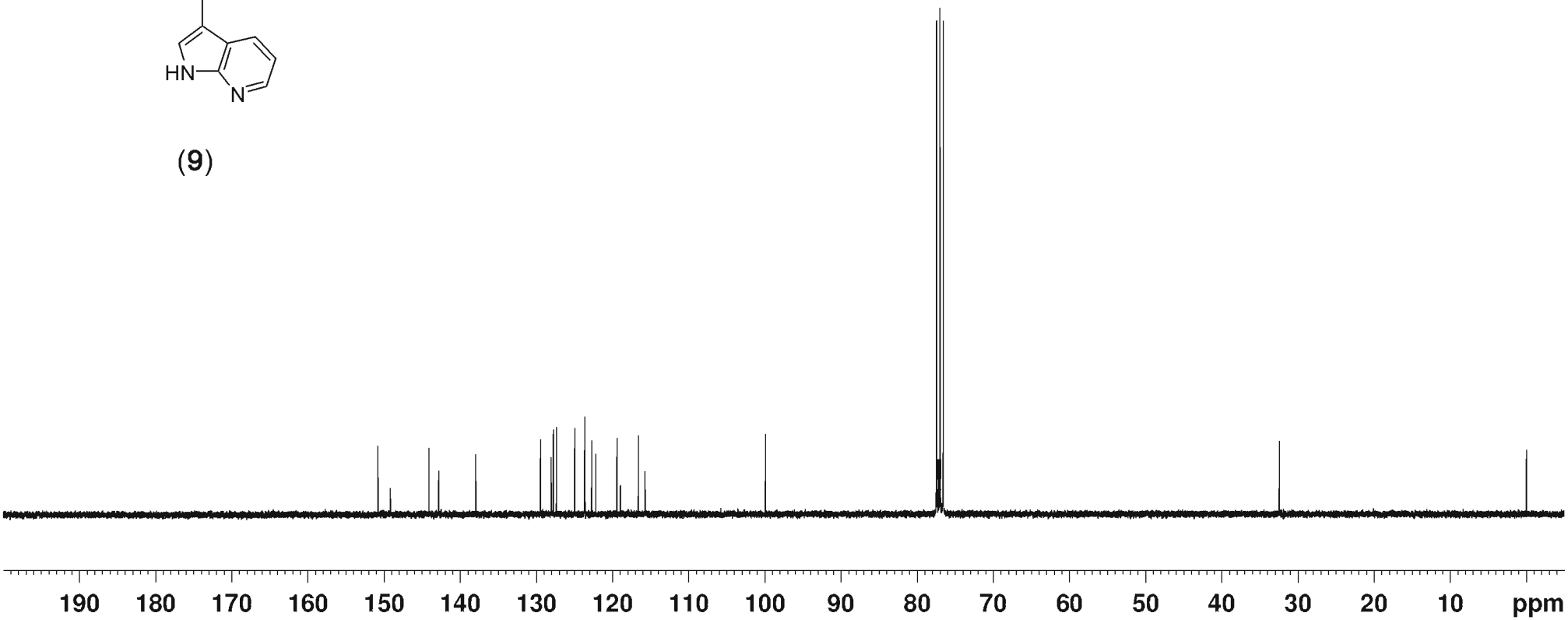
This journal is © The Royal Society of Chemistry 2012

^{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

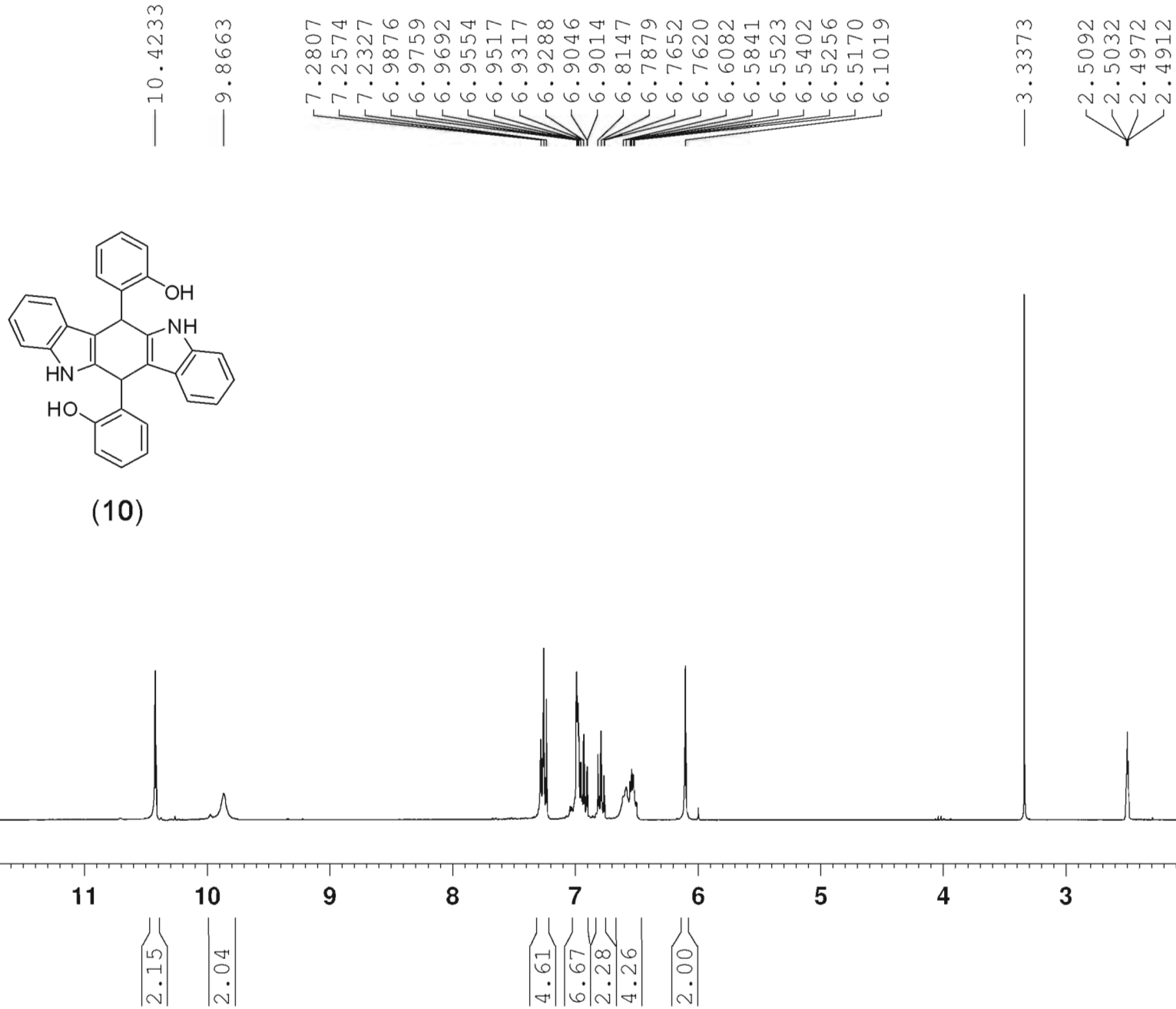


(**9**)



This journal is © The Royal Society of Chemistry 2012

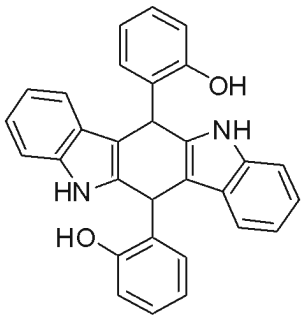
^{1}H NMR (300MHz, DMSO-d_6)



(10)

This journal is © The Royal Society of Chemistry 2012

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



(10)

This journal is © The Royal Society of Chemistry 2012

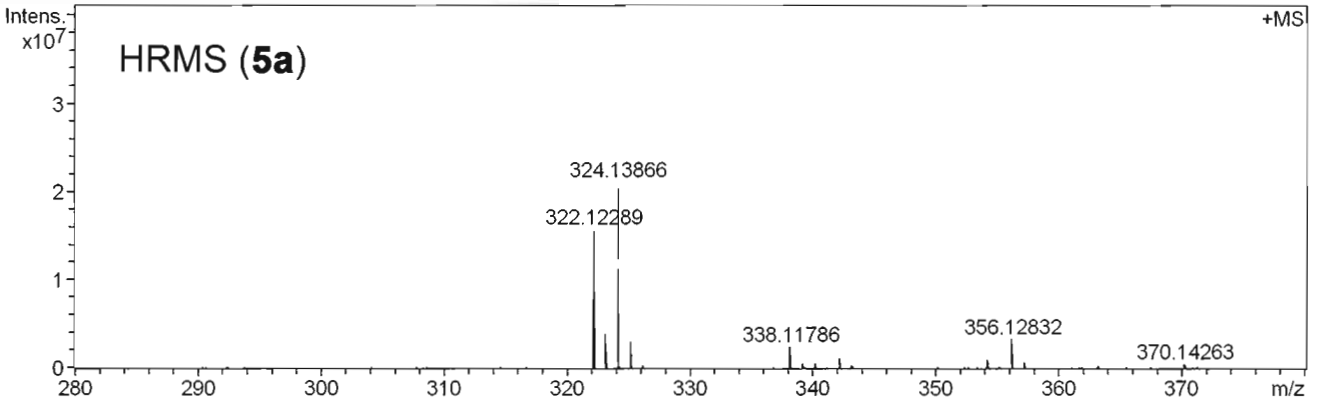
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\20120619\YIN_1_APCI_POS_000002.d	Acquisition Date	2012-6-19 19:00:33
Method	Metal_Trypsin digestion	Operator	
Sample Name	YIN_1_APCI_POS	Instrument	apex-Ultra
Comment			

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
324.13866	1	C 23 H 18 N O	100.00	324.13829	-0.4	-1.1	11.8	15.5	even	ok

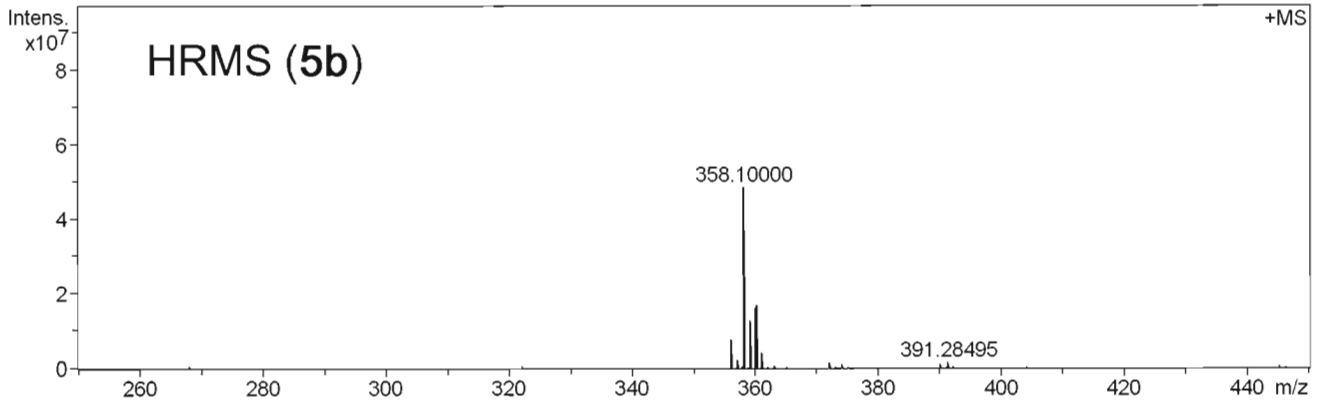
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\20120619\YIN_2_APCI_POS_000002.d	Acquisition Date	2012-6-19 19:01:42
Method	Metal_Trypsin digestion	Operator	
Sample Name	YIN_2_APCI_POS	Instrument	apex-Ultra
Comment			

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
358.10000	1	C 23 H 17 Cl N O	100.00	358.09932	-0.7	-1.9	3.6	15.5	even	ok

This journal is © The Royal Society of Chemistry 2012

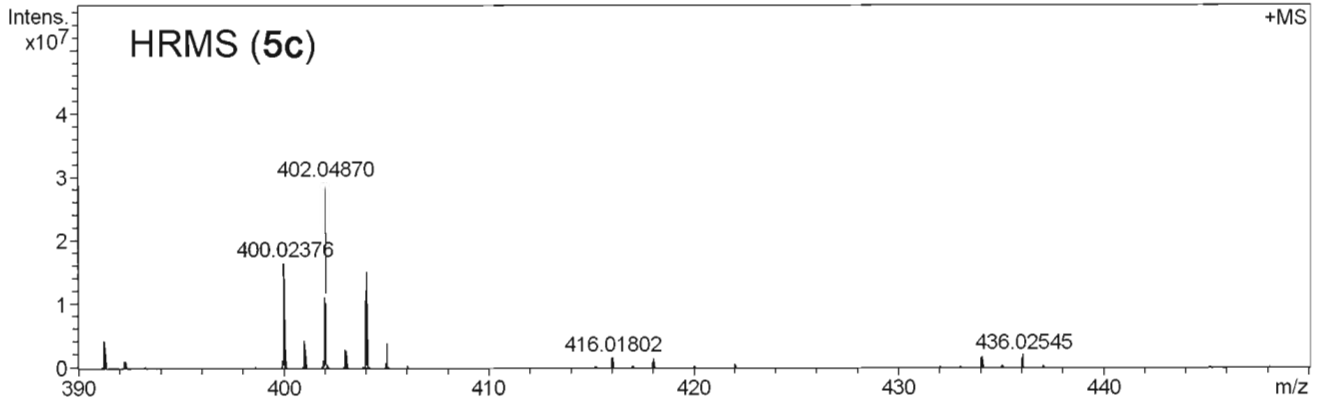
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\20120619\YIN_3_APCI_POS_000002.d	Acquisition Date	2012-6-19 19:02:53
Method	Metal_Trypsin digestion	Operator	
Sample Name	YIN_3_APCI_POS	Instrument	apex-Ultra
Comment			

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 Br N O 2	100.00	402.01242	-0.8	-1.9	459.8	16.5	even	ok
402.04870	1	C 23 H 17 Br N O	100.00	402.04880	0.1	0.3	148.3	15.5	even	ok

This journal is © The Royal Society of Chemistry 2012

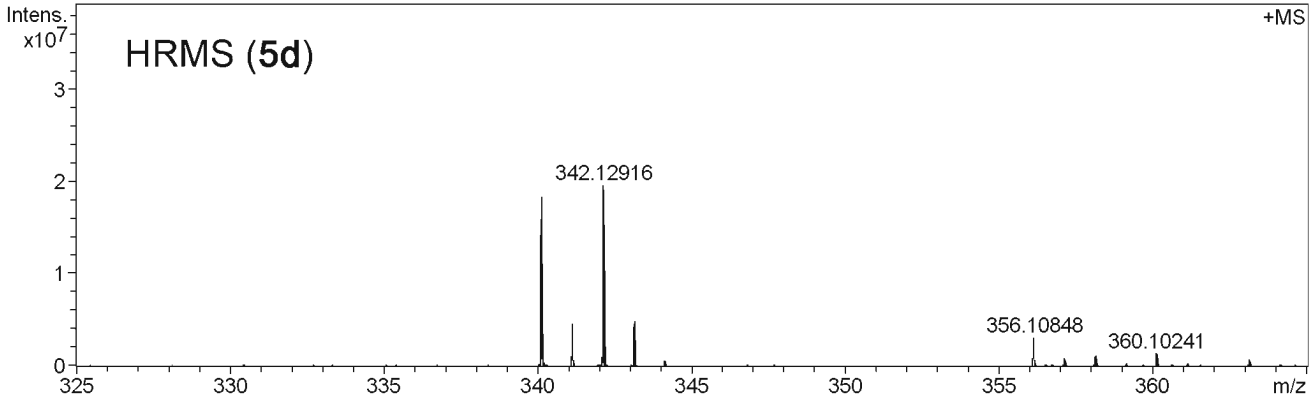
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\20120619\YIN_4_APCI_POS_000002.d	Acquisition Date	2012-6-19 19:04:08
Method	Metal_Trypsin digestion	Operator	
Sample Name	YIN_4_APCI_POS	Instrument	apex-Ultra
Comment			

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 F N O	100.00	342.12887	-0.3	-0.9	0.8	15.5	even	ok

This journal is © The Royal Society of Chemistry 2012

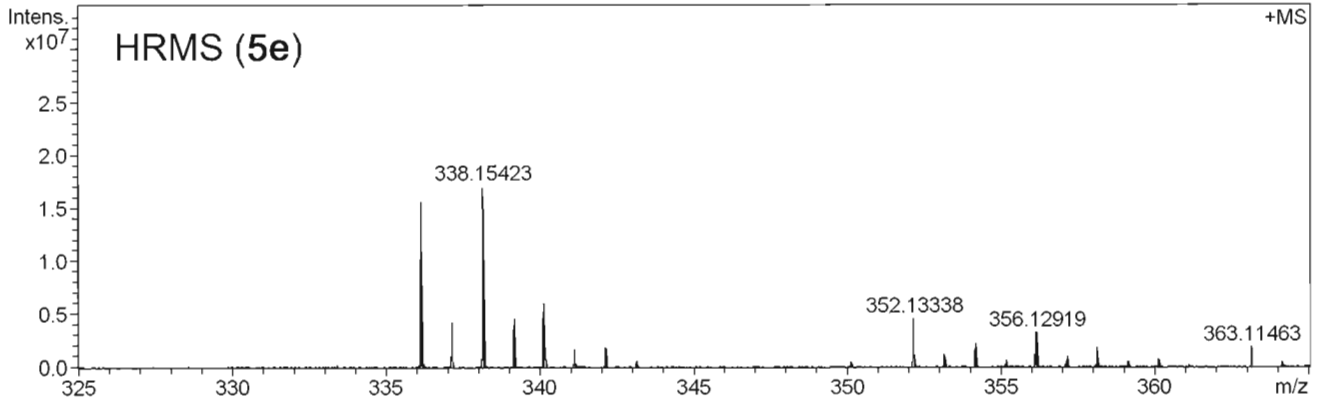
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\20120619\YIN_5_APCI_POS_000002.d	Acquisition Date	2012-6-19 19:05:11
Method	Metal_Trypsin digestion	Operator	
Sample Name	YIN_5_APCI_POS	Instrument	apex-Ultra
Comment			

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¡¥Conf	N-Rule
338.15423	1	C 24 H 20 N O	100.00	338.15394	-0.3	-0.8	42.5	15.5	even	ok

This journal is © The Royal Society of Chemistry 2012

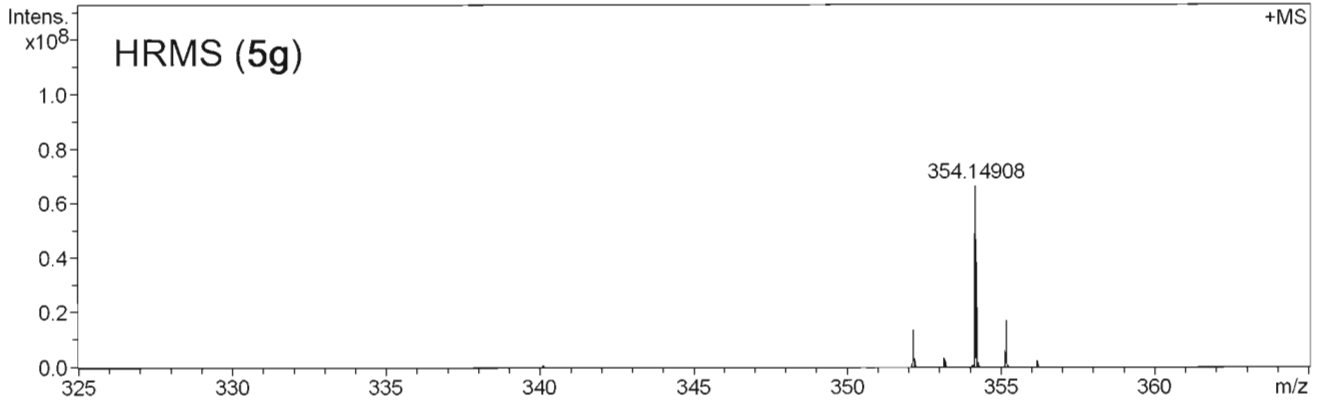
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\20120619\YIN_6_APCI_POS_000002.d	Acquisition Date	2012-6-19 19:06:18
Method	Metal_Trypsin digestion	Operator	
Sample Name	YIN_6_APCI_POS	Instrument	apex-Ultra
Comment			

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
354.14908	1	C 24 H 20 N O 2	100.00	354.14886	-0.2	-0.6	5.7	15.5	even	ok

This journal is © The Royal Society of Chemistry 2012

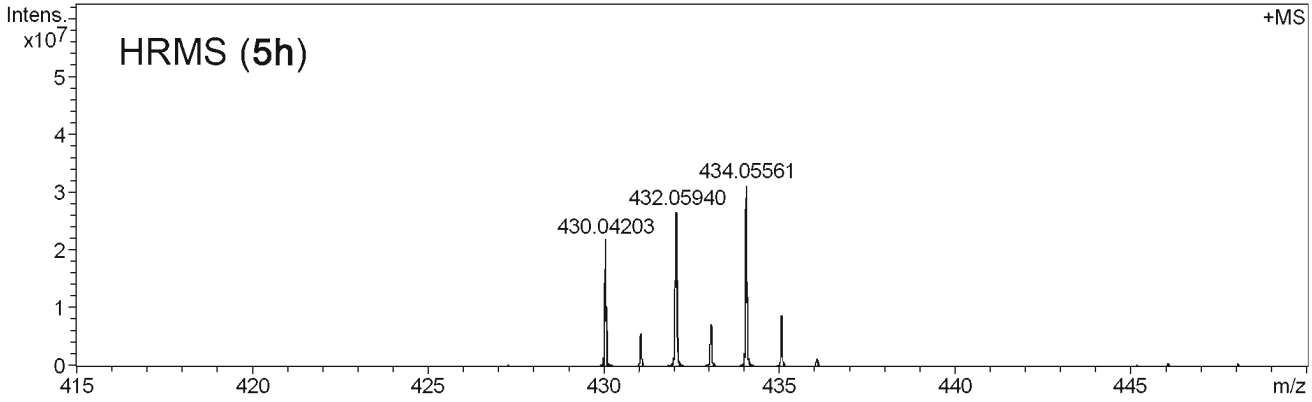
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\20120619\YIN_7_APCI_POS_000002.d	Acquisition Date	2012-6-19 19:07:22
Method	Metal_Trypsin digestion	Operator	
Sample Name	YIN_7_APCI_POS	Instrument	apex-Ultra
Comment			

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
432.05940	1	C 24 H 19 Br N O 2	100.00	432.05937	-0.0	-0.1	64.0	15.5	even	ok

This journal is © The Royal Society of Chemistry 2012

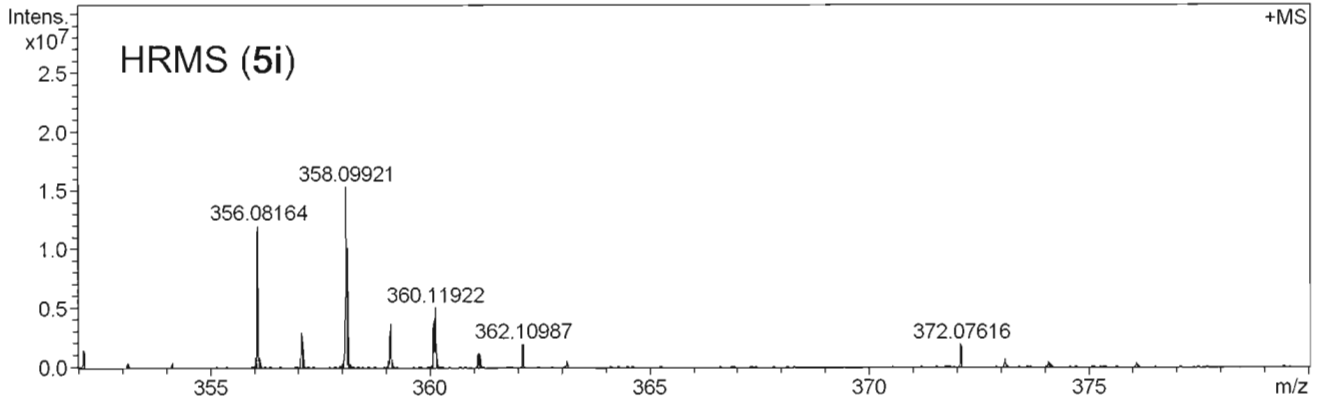
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\20120619\YIN_8_APCI_POS_000002.d	Acquisition Date	2012-6-19 19:08:26
Method	Metal_Trypsin digestion	Operator	
Sample Name	YIN_8_APCI_POS	Instrument	apex-Ultra
Comment			

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
358.09921	1	C 23 H 17 Cl N O	100.00	358.09932	0.1	0.3	67.1	15.5	even	ok

This journal is © The Royal Society of Chemistry 2012

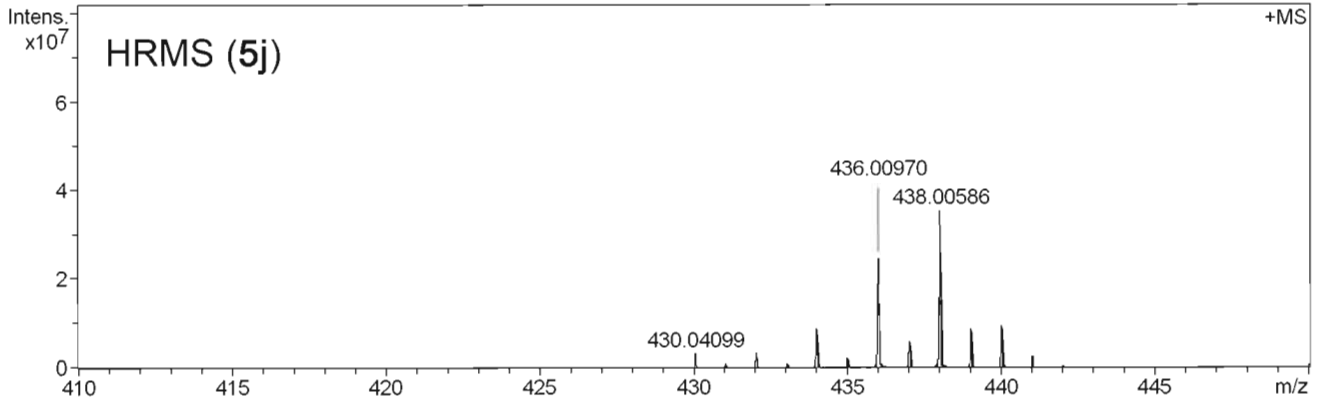
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\20120619\YIN_9_APCI_POS_000002.d	Acquisition Date	2012-6-19 19:09:23
Method	Metal_Trypsin digestion	Operator	
Sample Name	YIN_9_APCI_POS	Instrument	apex-Ultra
Comment			

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
436.00970	1	C 23 H 16 Br Cl N O	100.00	436.00983	0.1	0.3	27.6	15.5	even	ok

This journal is © The Royal Society of Chemistry 2012

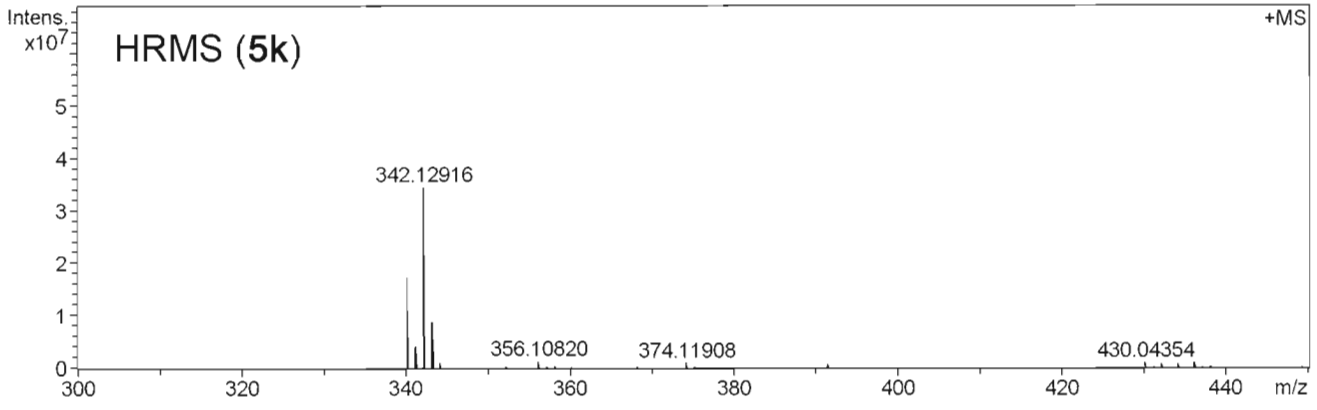
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\20120619\YIN_10_APCI_POS_000002.d	Acquisition Date	2012-6-19 19:10:20
Method	Metal_Trypsin digestion	Operator	
Sample Name	YIN_10_APCI_POS	Instrument	apex-Ultra
Comment			

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 F N O	100.00	342.12887	-0.3	-0.9	3.8	15.5	even	ok

This journal is © The Royal Society of Chemistry 2012

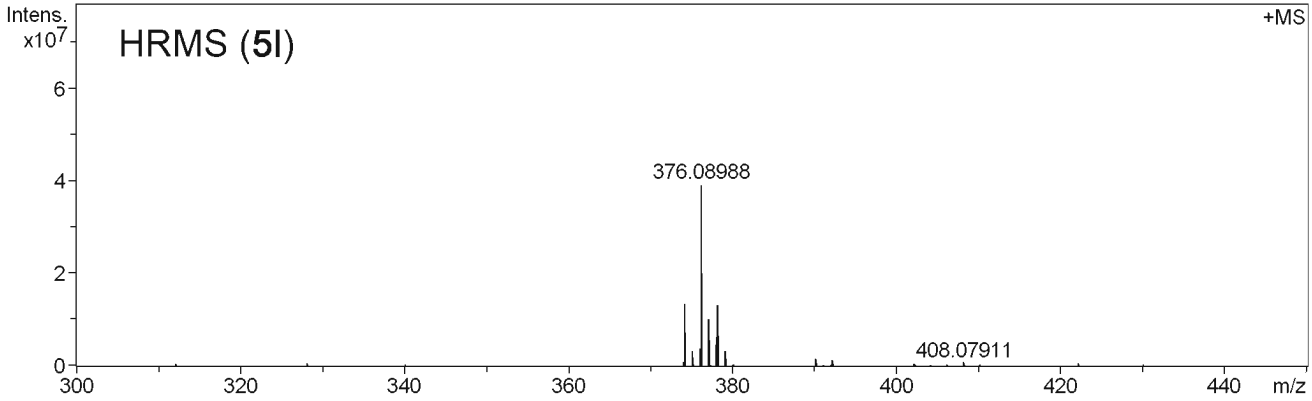
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\20120619\YIN_16_APCI_POS_000002.d	Acquisition Date	2012-6-19 19:17:11
Method	Metal_Trypsin digestion	Operator	
Sample Name	YIN_16_APCI_POS	Instrument	apex-Ultra
Comment			

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
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

This journal is © The Royal Society of Chemistry 2012

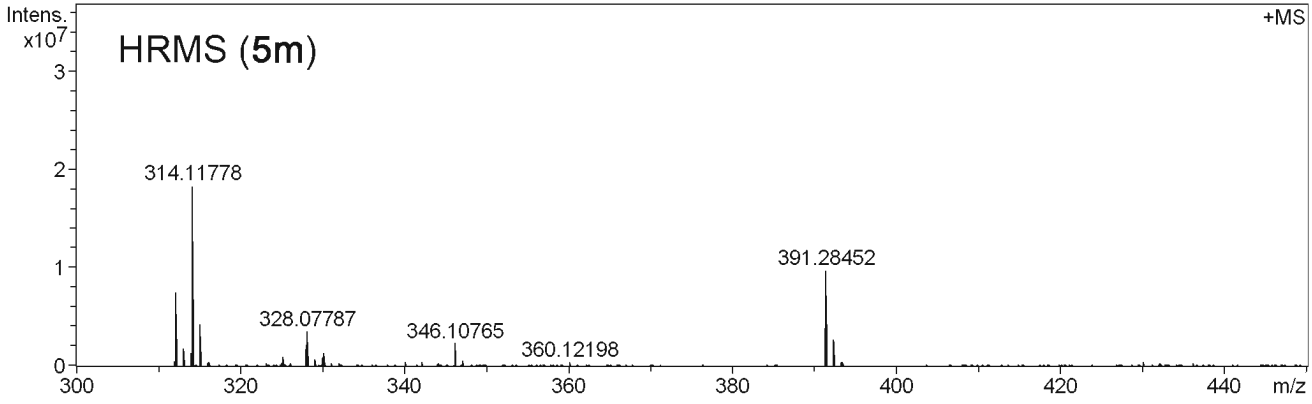
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\20120619\YIN_15_APCI_POS_000002.d	Acquisition Date	2012-6-19 19:15:22
Method	Metal_Trypsin digestion	Operator	
Sample Name	YIN_15_APCI_POS	Instrument	apex-Ultra
Comment			

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
314.11778	1	C 21 H 16 N O 2	100.00	314.11756	-0.2	-0.7	3.2	14.5	even	ok

This journal is © The Royal Society of Chemistry 2012

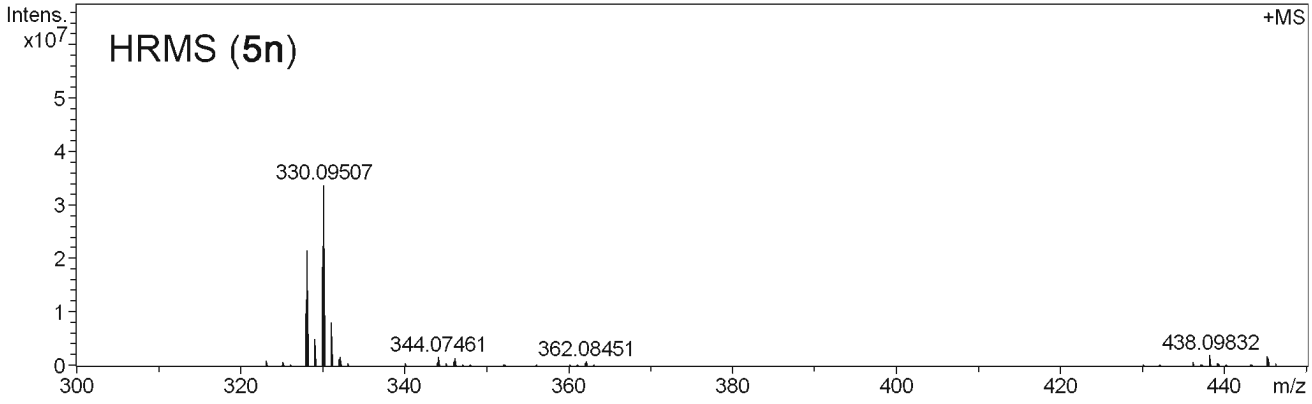
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\20120619\YIN_14_APCI_POS_000002.d	Acquisition Date	2012-6-19 19:14:18
Method	Metal_Trypsin digestion	Operator	
Sample Name	YIN_14_APCI_POS	Instrument	apex-Ultra
Comment			

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
330.09507	1	C 21 H 16 N O S	100.00	330.09471	-0.4	-1.1	19.2	14.5	even	ok

This journal is © The Royal Society of Chemistry 2012

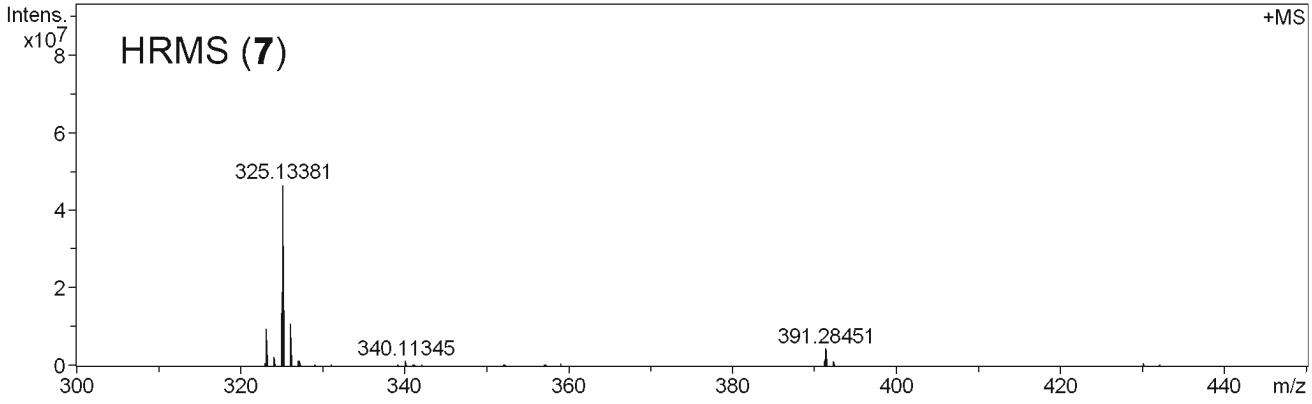
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\20120619\YIN_13_APCI_POS_000002.d	Acquisition Date	2012-6-19 19:13:22
Method	Metal_Trypsin digestion	Operator	
Sample Name	YIN_13_APCI_POS	Instrument	apex-Ultra
Comment			

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
325.13381	1	C 22 H 17 N 2 O	100.00	325.13354	-0.3	-0.8	5.0	15.5	even	ok

This journal is © The Royal Society of Chemistry 2012

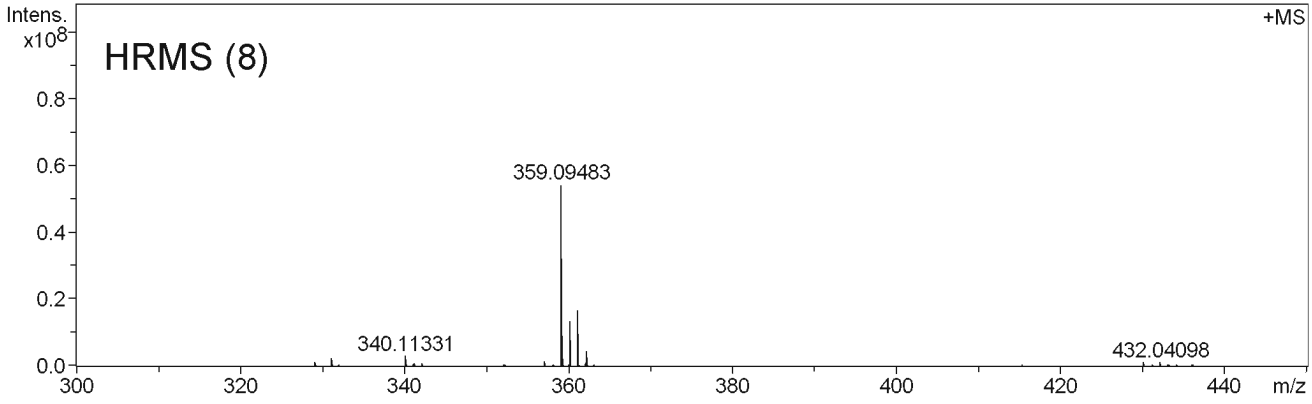
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\20120619\YIN_12_APCI_POS_000002.d	Acquisition Date	2012-6-19 19:12:27
Method	Metal_Trypsin digestion	Operator	
Sample Name	YIN_12_APCI_POS	Instrument	apex-Ultra
Comment			

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
359.09483	1	C 22 H 16 Cl N 2 O	100.00	359.09457	-0.3	-0.7	15.5	15.5	even	ok

This journal is © The Royal Society of Chemistry 2012

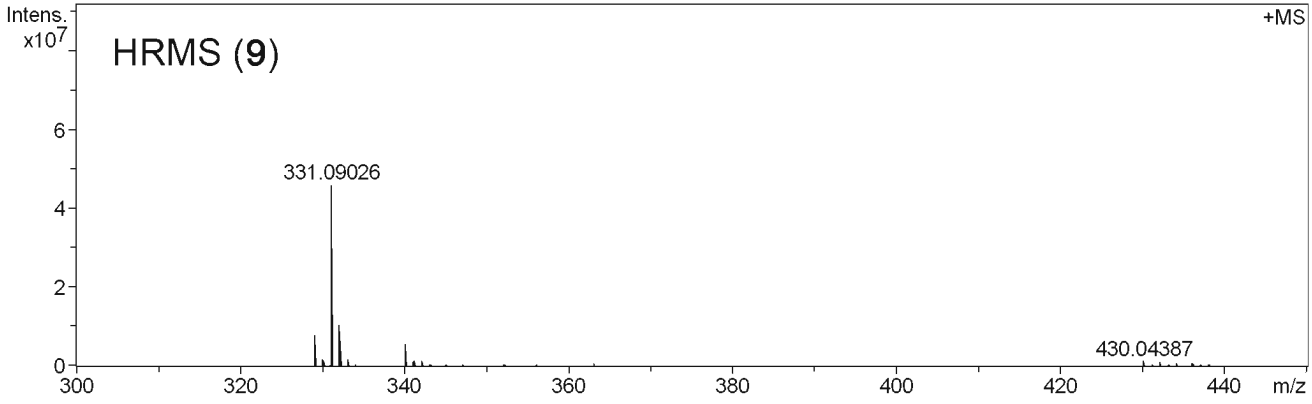
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\20120619\YIN_11_APCI_POS_000002.d	Acquisition Date	2012-6-19 19:11:31
Method	Metal_Trypsin digestion	Operator	
Sample Name	YIN_11_APCI_POS	Instrument	apex-Ultra
Comment			

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
331.09026	1	C 20 H 15 N 2 O S	100.00	331.08996	-0.3	-0.9	17.3	14.5	even	ok

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
This journal is © The Royal Society of Chemistry 2012

HSQC spectrum of compound 7 (600MHz, $CDCl_3$)