Electronic Supplementary Material (ESI) for Green Chemistry.

Green Chemistry

An ammonium molybdate deposited amorphous silica coated iron oxide magnetic core-shell nanocomposite for the efficient synthesis of 2-benzimidazoles using hydrogen peroxide

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Electronic Supplementary Information (ESI)

Supplementary Information Available: complete catalyst characterization data, product characterization data, analytical details.

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General Considerations and Instrumentation

Catalyst characterization

Scanning electron microscopy (SEM) was obtained using a JEOL JSM-7500 electron microscope. Surface compositions were identified by energy-dispersive X-ray (EDX) using a Thermo NORAN System 7 spectrometer.

Thermogravimetric (TG) and differential thermogravimetric (DTG) measurements were carried out on a Netzsch TG 209 F3 thermogravimetric analyzer at a constant heating rate of 30/10.0 ($K \cdot min^{-1}$)/200 under a nitrogen atmosphere with a gas flow of 25 $mL \cdot min^{-1}$.

Product characterization

The structures of 2-benzimidazoles were confirmed by IR, NMR, and HRMS. IR spectra were collected on a Bruker Vertex 70 spectrophotometer. ^{1}H NMR and ^{13}C NMR spectra were recorded at 600 MHz and 150 MHz, respectively, on a Bruker spectrometer in DMSO-d_6 using TMS as the internal standard at room temperature. Mass spectrometric data were determined on an IonSpec QFT-MALDI MS (Varian).

SEM images and EDX spectrum

SEM images of the $Fe_3O_4@SiO_2@(NH_4)_6Mo_7O_{24}$ core-shell nanocomposites showed that the particles were slightly agglomerated (Figure S1 b), due to the Van der Waals force being strengthened in the preparation process, although they still keeping in a nanometer size. EDX from the obtained nanomaterials demonstrated the presence of the expected Mo on the surface of this catalyst (Figure S2), and further supported the fact that the ammonium molybdate species were highly dispersed on the surface.

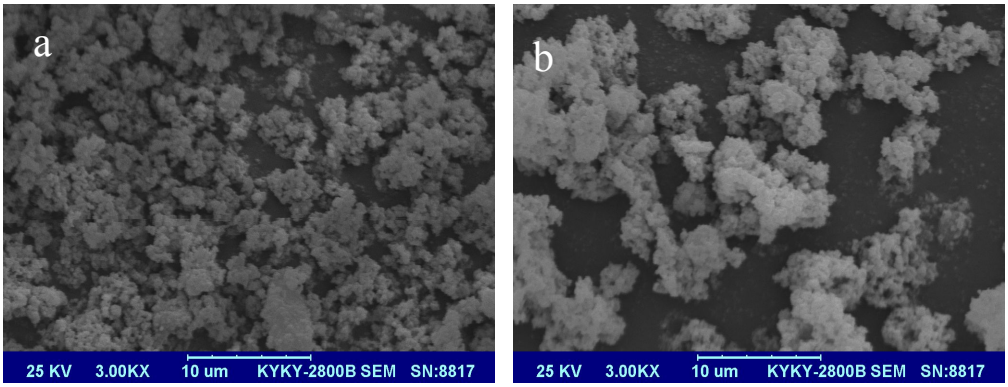


Figure S1. SEM images of (a) $Fe_3O_4@SiO_2$ and (b) $Fe_3O_4@SiO_2@(NH_4)_6Mo_7O_{24}$.

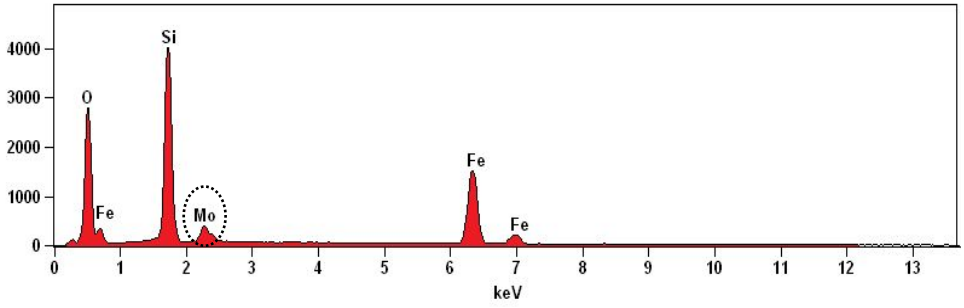


Figure S2. EDX spectrum of $Fe_3O_4@SiO_2@(NH_4)_6Mo_7O_{24}$.

TG and DTG analyses

The thermal stability of $Fe_3O_4@SiO_2@(NH_4)_6Mo_7O_{24}$ was evaluated by TG and DTG measurements and compared with those of the unsupported $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$. Figure 3A exhibits the relationship between the residual masses (RM, %) of the two samples and the temperature. It illustrated that the unsupported $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ has a significant mass loss in the range of 353-415 K due to the release of crystal water;[1] whereas, the $Fe_3O_4@SiO_2@(NH_4)_6Mo_7O_{24}$ has less mass loss in the same range, demonstrating that $(NH_4)_6Mo_7O_{24}$ has been integrally deposited on the surface of $Fe_3O_4@SiO_2$, together with the loss of crystal water during preparation, as also supported by the DTG results of $Fe_3O_4@SiO_2@(NH_4)_6Mo_7O_{24}$ (Figure 3 B, b).

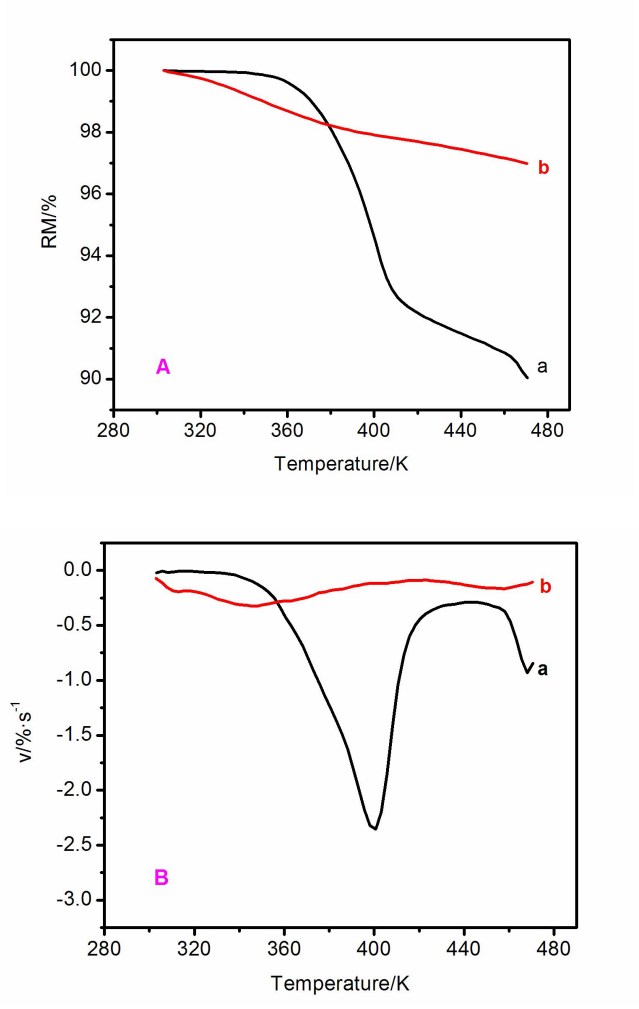
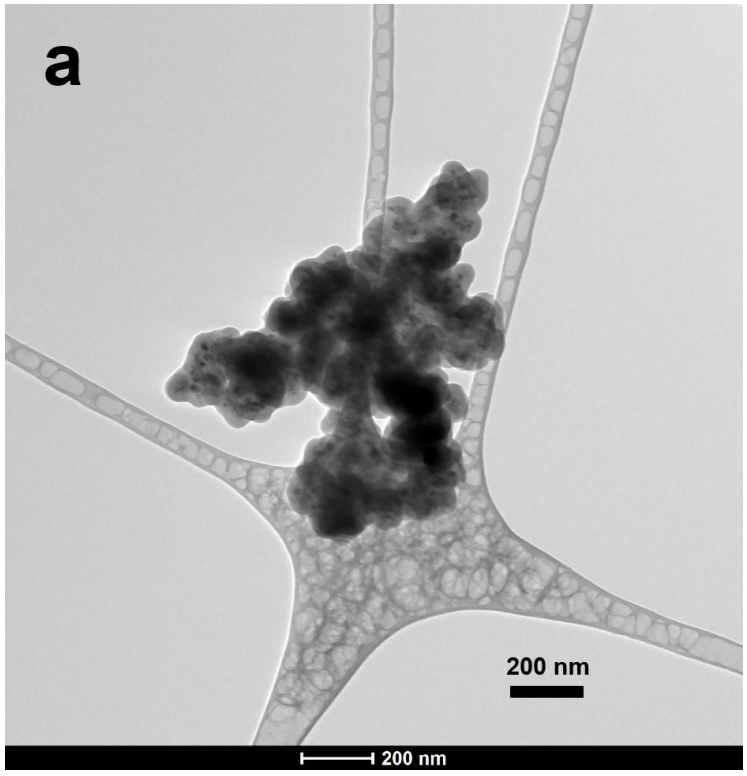


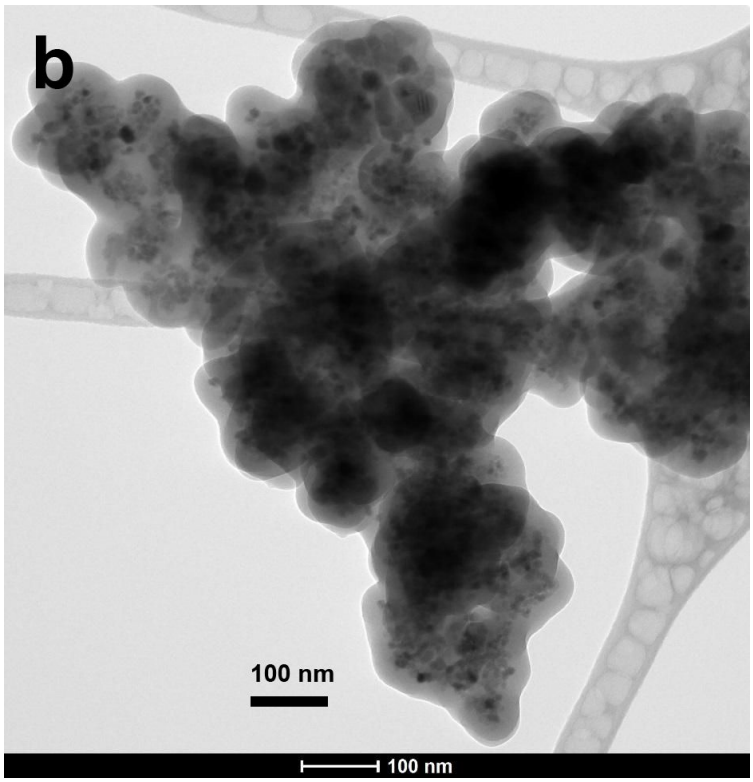
Figure S3. TG (A) and DTG (B) profiles of

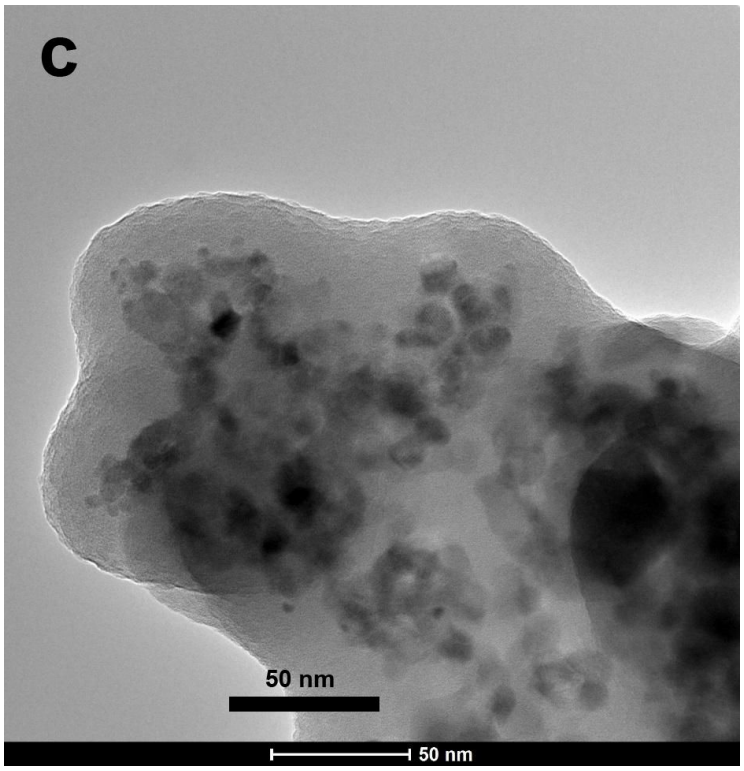
(a) $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ and (b) $Fe_3O_4@SiO_2@(NH_4)_6Mo_7O_{24}$.

TEM images

The surface morphology and the particle distributions of the corresponding magnetic core-shell nanocomposites of Fe_3O_4@SiO_2 (Figure S4 a-c) and Fe_3O_4@SiO_2@$(NH_4)_6Mo_7O_{24}$ (Figure S4 a'-c') were recorded by transmission electron microscope (TEM) at different bar sizes.



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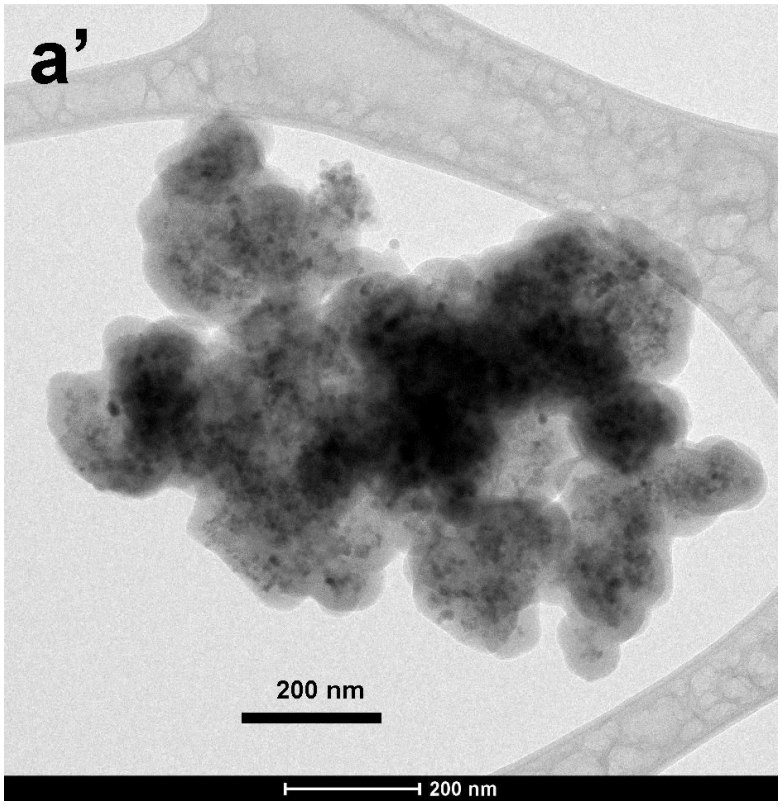
a’
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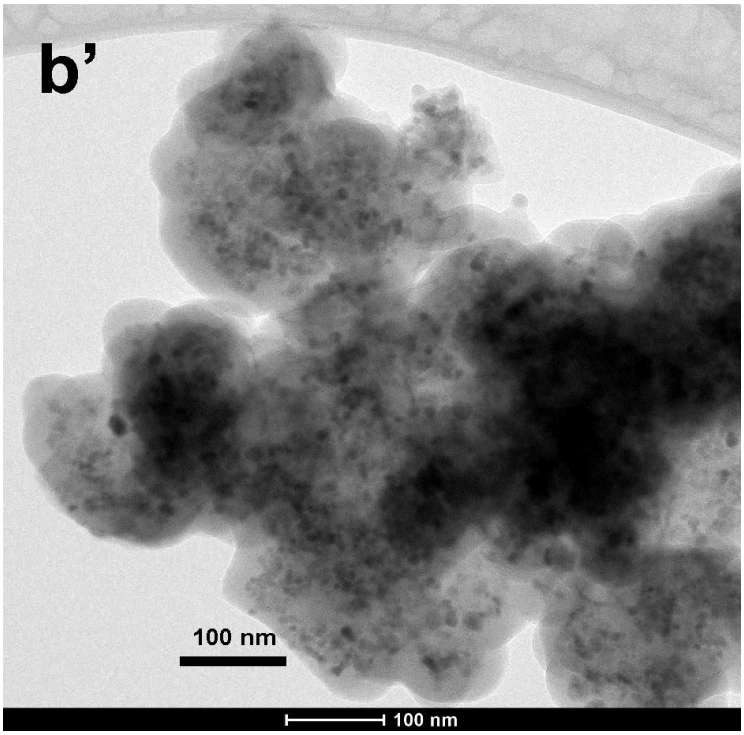


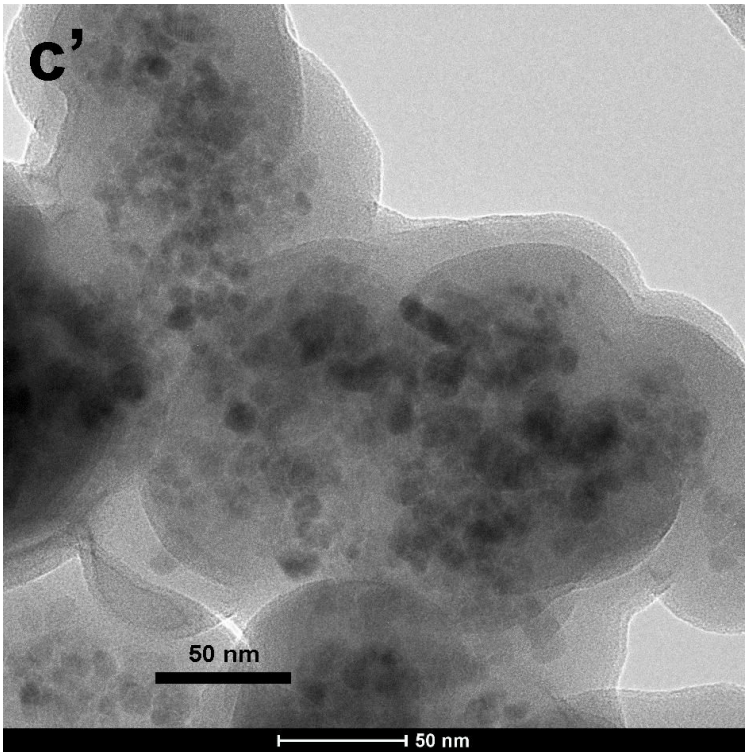


Figure S4. TEM images of (a-c) $Fe_3O_4@SiO_2$ and (a’-c’) $Fe_3O_4@SiO_2@(NH_4)_6Mo_7O_{24}$ nanocomposites at different bar sizes.

Product characterization data(IR, ^{1}H NMR, ^{13}C NMR, HRMS)

2-Phenyl-1*H*-benzimidazole:

Pale yellow crystals, IR (KBr): 1626 (C=N), 3436 (NH) cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 12.96 (s, 1H), 8.21-8.20 (t, J = 9.0 Hz, 2H), 7.62-7.49 (m, 5H), 7.23-7.20 (m, 2H). ^{13}C NMR (150 MHz, DMSO-d_6) δ 151.70, 130.65, 130.31, 129.42, 126.91, 122.58. HRMS (ESI) Calc. for $C_{13}H_{11}N_2$ $[M+H]^+$: 195.0917, found: 195.0916.

2-(4-Chlorophenyl)-1*H*-benzimidazole:

White crystals, IR (KBr): 1623 (C=N), 3442 (NH) cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 13.00 (s, 1H), 8.20 (dd, J_1 = 6.6 Hz, J_2 = 1.8 Hz, 2H), 7.65-7.63 (m, 4H), 7.23 (d, J = 3.0 Hz, 2H). ^{13}C NMR (150 MHz, DMSO-d_6) δ 150.63, 134.96, 129.53, 128.61, 123.20, 122.29, 119.44, 111.88. HRMS (ESI) Calc. for $C_{13}H_{10}ClN_2$ $[M+H]^+$: 229.0527, found: 229.0523.

2-(3-Chlorophenyl)-1*H*-benzimidazole:

Pale yellow crystals, IR (KBr): 1623 (C=N), 3439 (NH) cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 13.05 (s, 1H), 8.24 (s, 1H), 8.16-8.15 (m, 1H), 7.61-7.56 (m, 4H), 7.24 (s, 2H). ^{13}C NMR (150 MHz, DMSO-d_6) δ 150.20, 134.24, 132.68, 131.41, 130.01, 126.49, 125.48. HRMS (ESI) Calc. for $C_{13}H_{10}ClN_2$ $[M+H]^+$: 229.0527, found: 229.0523.

2-(2-Chlorophenyl)-1*H*-benzimidazole:

White crystals, IR (KBr): 1623 (C=N), 3433 (NH) cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 12.73 (s, 1H), 7.92 (dd, J_1 = 7.8 Hz, J_2 = 1.8 Hz, 1H), 7.67 (dd, J_1 = 8.4 Hz, J_2 = 1.2 Hz, 2H), 7.57-7.52

(m, 3H), 7.25 (s, 2H). ^{13}C NMR (150 MHz, DMSO-d_6) δ 149.57, 132.56, 132.11, 131.67, 130.82, 130.45, 127.91. HRMS (ESI) Calc. for $C_{13}H_{10}ClN_2$ $[M+H]^+$: 229.0527, found: 229.0523.

2-(4-Nitrophenyl)-1*H*-benzimidazole:

Yellow crystals, IR (KBr): 1338, 1516 (NO_2), 1607 (C=N), 3436 (NH) cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 13.31 (s, 1H), 8.44-8.41 (m, 4H), 7.73-7.66 (m, 2H), 7.28 (s, 2H).^{13}C NMR (150 MHz, DMSO-d_6) δ 149.46, 148.26, 136.50, 127.85, 124.76. HRMS (ESI) Calc. for $C_{13}H_{10}N_3O_2$ $[M+H]^+$: 240.0768, found: 240.0768.

2-(4-Methoxyphenyl)-1*H*-benzimidazole:

White crystals, IR (KBr): 1244 (C-O), 1613 (C=N), 2965 (CH_3), 3439 (NH) cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 12.76 (s, 1H), 8.13 (d, J = 3.0 Hz, 2H), 7.56 (s, 2H), 7.18 (dd, J_1=5.4 Hz, J_2 = 3.0 Hz, 2H), 7.13 (d, J = 2.4 Hz, 2H), 3.85 (s, 3H). ^{13}C NMR (150 MHz, DMSO-d_6) δ 161.07, 151.82, 128.48, 123.18, 122.21, 114.83, 55.79. HRMS (ESI) Calc. for $C_{14}H_{13}N_2O$ $[M+H]^+$: 225.1022, found: 225.1021.

2-(4-Methylphenyl)-1*H*-benzimidazole:

Yellow crystals, IR (KBr): 1623 (C=N), 2965 (CH_3), 3449 (NH) cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 12.83 (s, 1H), 8.08 (d, J = 8.4 Hz, 2H), 7.58 (s, 2H), 7.37 (d, J = 7.8 Hz, 2H), 7.20 (dd, J_1=6.0 Hz, J_2 = 3.0 Hz, 2H), 2.39 (s, 3H). ^{13}C NMR (150 MHz, DMSO-d_6) δ 151.84, 140.04, 129.98, 127.90, 126.87, 122.43, 21.44. HRMS (ESI) Calc. for $C_{14}H_{13}N_2$ $[M+H]^+$: 209.1073, found: 209.1072.

2-(4-Hydroxyphenyl)-1*H*-benzimidazole:

Orange crystals, IR (KBr): 1610 (C=N), 3308 (OH, NH) cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 12.65 (s, 1H), 9.96 (s, 1H), 8.01 (d, J = 2.4 Hz, 2H), 7.53 (s, 2H), 7.16 (dd, J_1 = 6.0 Hz, J_2 = 3.0 Hz, 2H), 6.92 (d, J = 3.6 Hz, 2H). ^{13}C NMR (150 MHz, DMSO-d_6) δ 159.59, 152.25, 128.62, 122.07, 121.62, 116.15. HRMS (ESI) Calc. for $C_{13}H_{11}N_2O$ $[M+H]^+$: 211.0866, found: 211.0863.

2-(1,3-Benzodioxol-5-yl)-1*H*-benzimidazole:

Yellow crystals, IR (KBr): 1226, 1245 (C-O), 1626 (C=N), 2879 (CH_2) 3452 (NH) cm^{-1}. ^{1}H NMR (600 MHz, DMSO-d_6) δ 12.75 (s, 1H), 7.73 (dd, J_1 = 2.4 Hz, J_2 = 1.2 Hz, 1H), 7.69 (d, J = 1.8 Hz, 1H), 7.56 (s, 2H), 7.20-7.18 (m, 2H), 7.11 (d, J = 1.8 Hz, 1H), 6.13 (s, 2H). ^{13}C NMR (150 MHz, DMSO-d_6) δ 151.62, 149.20, 148.34, 124.79, 122.36, 121.37, 109.18, 106.98. 102.03. HRMS (ESI) Calc. for $C_{14}H_{11}N_2O_2$ $[M+H]^+$: 239.0815, found: 239.0811.

5-Methyl-2-phenyl-1*H*-benzimidazole:

White crystals, IR (KBr): 1631 (C=N), 2919 (CH_3), 3435 (NH). ^{1}H NMR (600 MHz, DMSO-d_6) δ 12.78 (s, 1H), 8.17 (d, J=7.2 Hz, 2H), 7.56-7.38 (m, 5H), 7.04 (d, J=8.4, 1H), 2.43 (s, 3H). ^{13}C NMR (150 MHz, DMSO-d_6) δ 135.46, 134.78, 134.04, 131.44, 26.48. HRMS (ESI) Calc. for $C_{14}H_{13}N_2$$[M+H]^+$: 209.1073, found: 209.1070.

5-Bromo-2-phenyl-1*H*-benzimidazole:

Yellow crystals, IR (KBr): 1617 (C=N), 3435 (NH). ^{1}H NMR (600 MHz, DMSO-d_6) δ 13.15 (s,

1H), 8.19-8.17 (m, 2H), 7.80 (s, 1H), 7.59-7.51 (m, 4H), 7.36-7.35 (dd, J_1=9.0 Hz, J_2=8.4 Hz, 1H). ^{13}C NMR (150 MHz, DMSO-d_6) δ 152.93, 130.71, 130.11, 129.49, 127.08, 125.45, 114.74. HRMS (ESI) Calc. for $C_{13}H_{10}BrN_2[M+H]^+$: 273.0022, found: 273.0019.

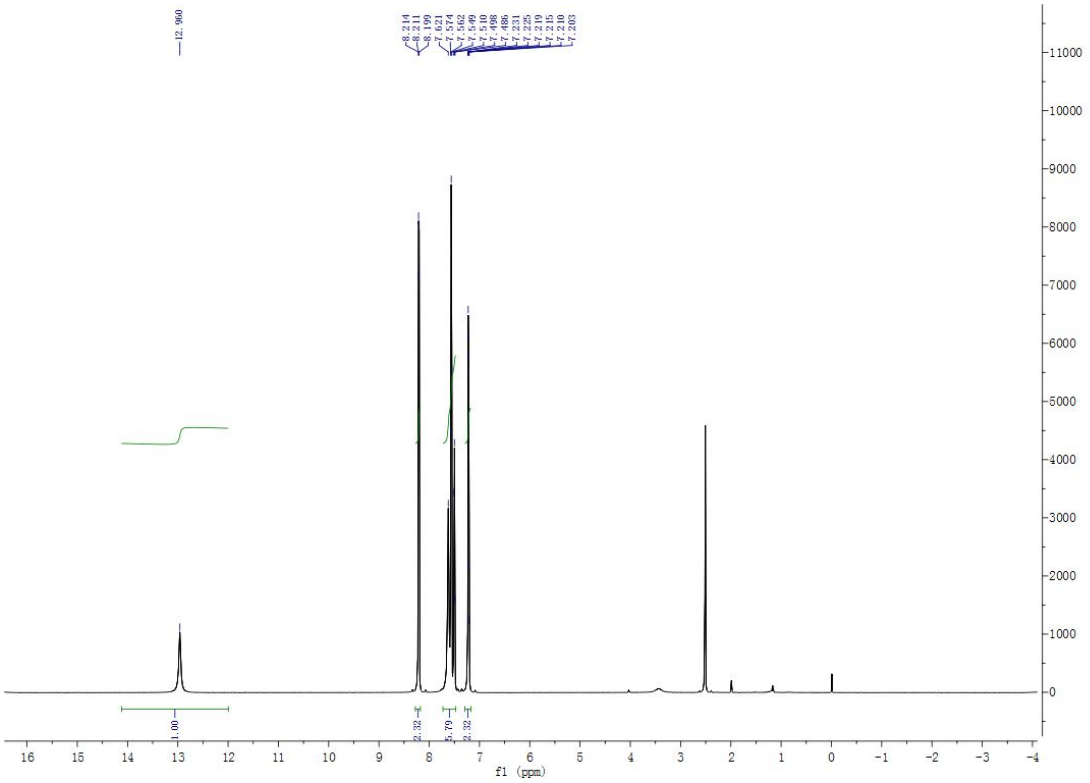
5-Nitro-2-phenyl-1*H*-benzimidazole:

Yellow crystals, IR (KBr): 1334, 1512 (NO_2), 1624 (C=N), 3436 (NH). ^{1}H NMR (600 MHz, DMSO-d_6) δ 13.64 (s, 1H), 8.51 (s, 1H), 8.23-8.14 (m, 3H), 7.78 (s, 1H), 7.62 (d, *J*=6.6 Hz, 3H). ^{13}C NMR (150 MHz, DMSO-d_6) δ 143.18, 131.43, 129.62, 129.51, 127.46, 118.44. HRMS (ESI) Calc. for $C_{13}H_{10}N_3O_2[M+H]^+$: 240.0768, found: 240.0763.

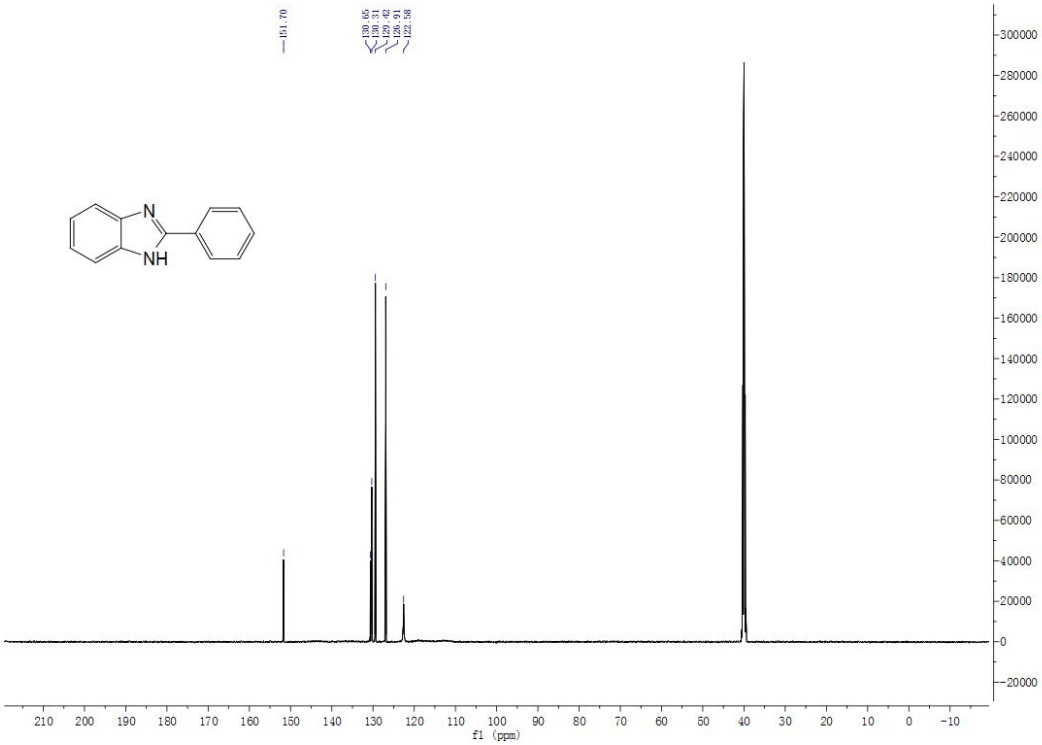
Reference:

[1] L. X. Song, M. Wang, Z. Dang, F. Y. Du, *J. Phys. Chem. B* **2010**, 114, 3404-3410.

2-Phenyl-1*H*-benzimidazole characterization spectrum



^{1}H NMR Spectrum of 2-phenyl-1*H*-benzimidazole



^{13}C NMR Spectrum of 2-phenyl-1*H*-benzimidazole

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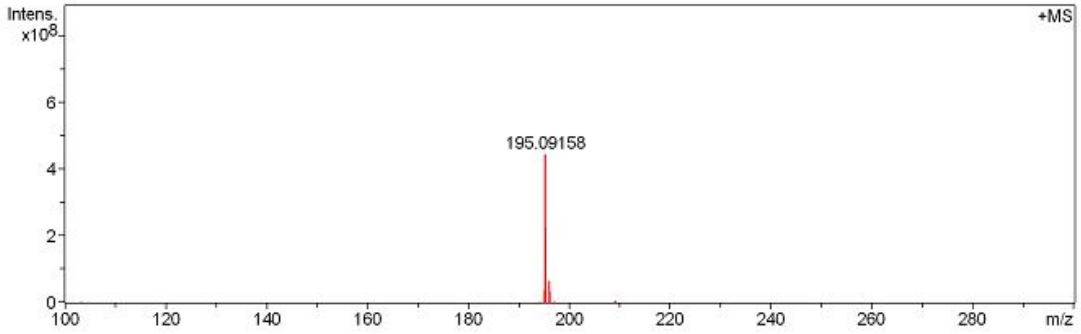

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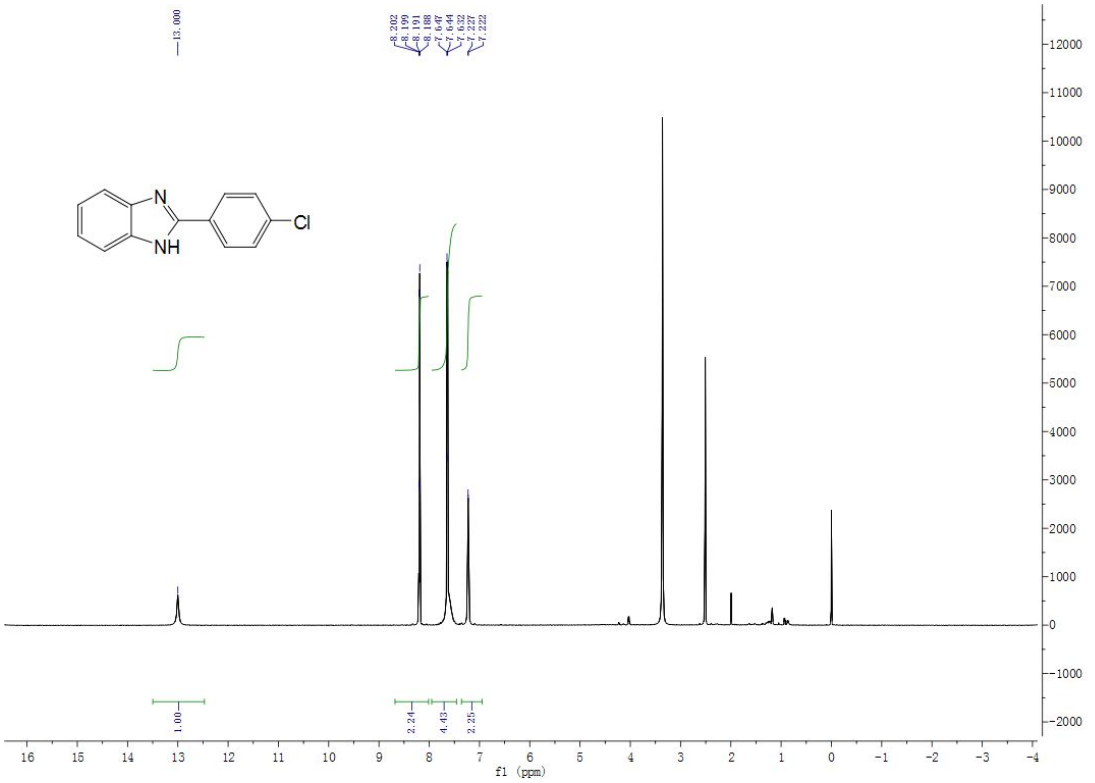
HRMS Spectrum of 2-phenyl-1*H*-benzimidazole

2-(4-Chlorophenyl)-1*H*-benzimidazole characterization spectrum



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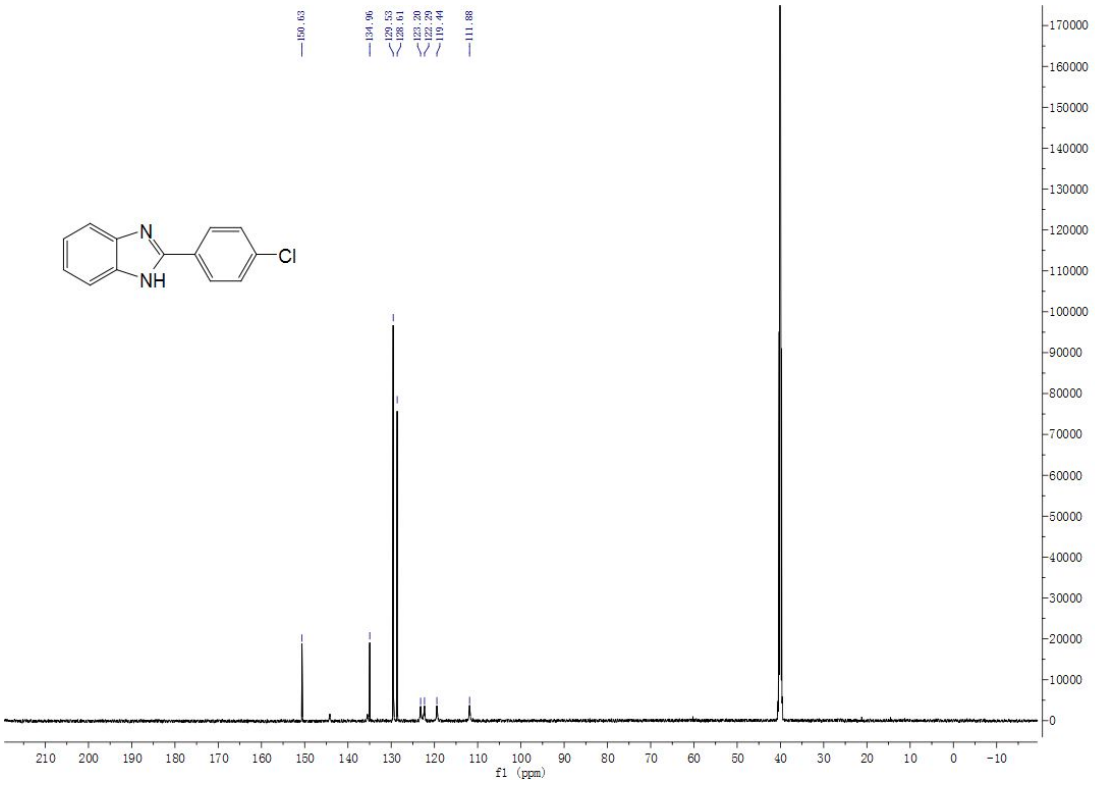

^{13}C NMR Spectrum of 2-(4-chlorophenyl)-1*H*-benzimidazole

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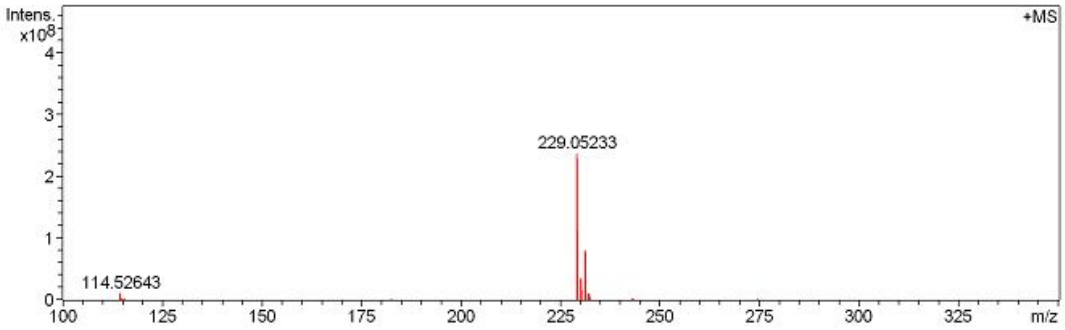

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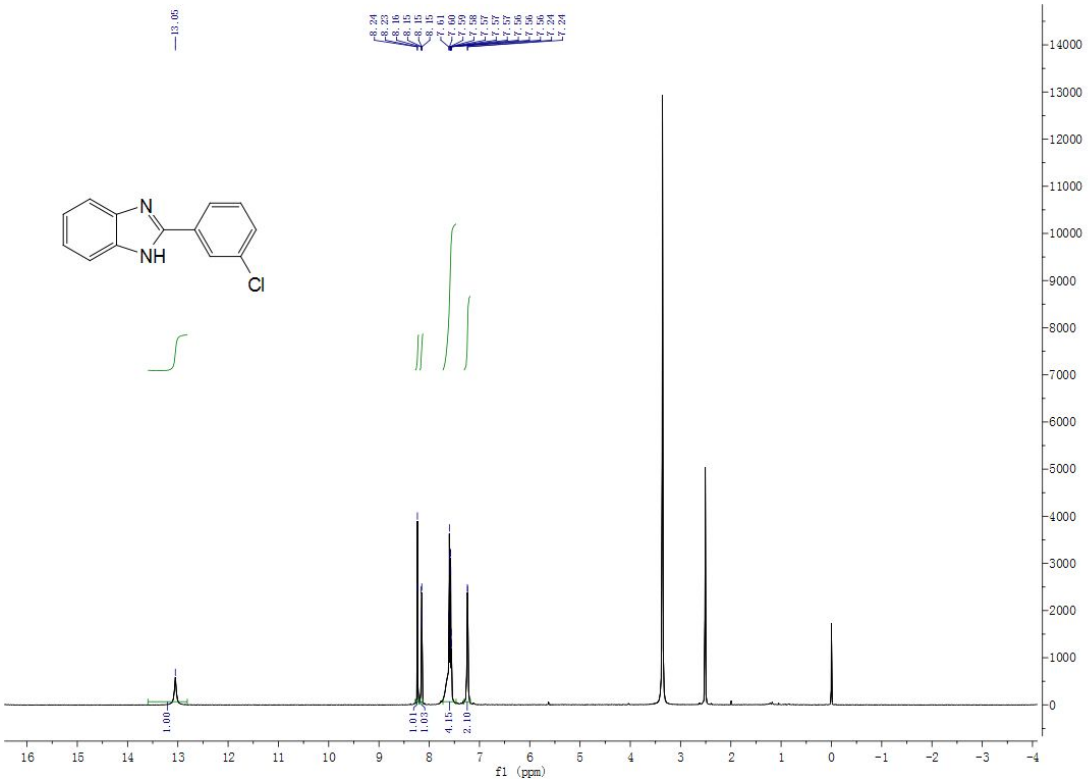
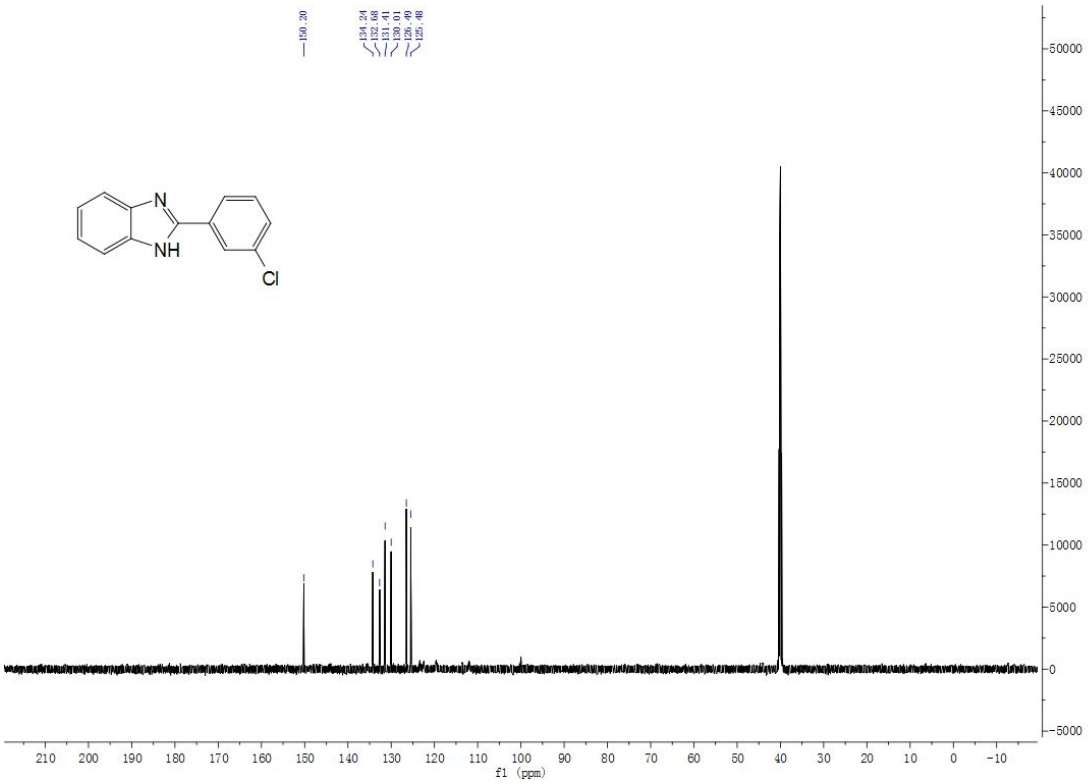
HRMS Spectrum of 2-(4-chlorophenyl)-1*H*-benzimidazole

2-(3-Chlorophenyl)-1*H*-benzimidazole characterization spectrum



^{1}H NMR Spectrum of 2-(3-chlorophenyl)-1*H*-benzimidazole



^{13}C NMR Spectrum of 2-(3-chlorophenyl)-1*H*-benzimidazole

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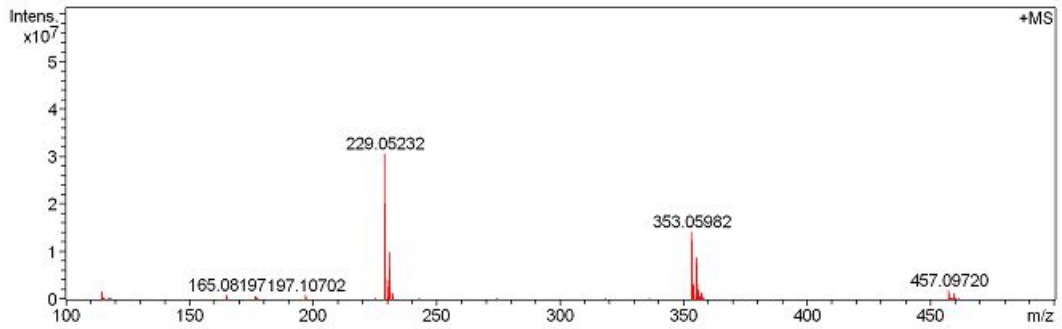

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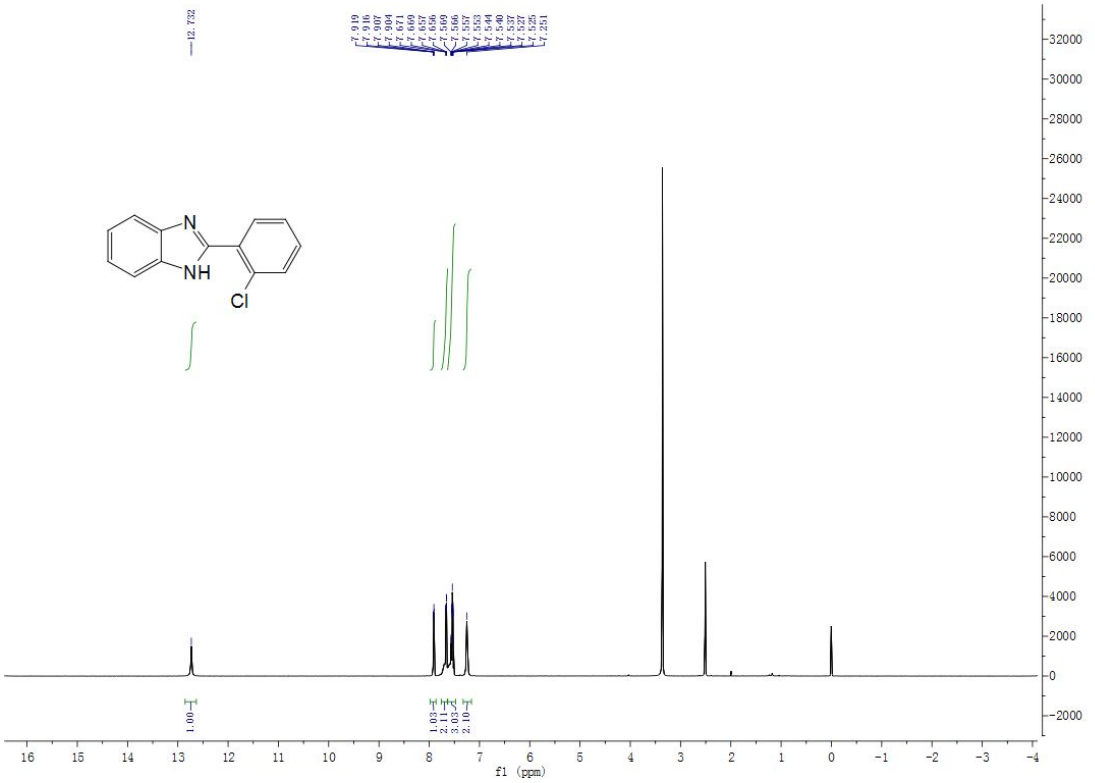
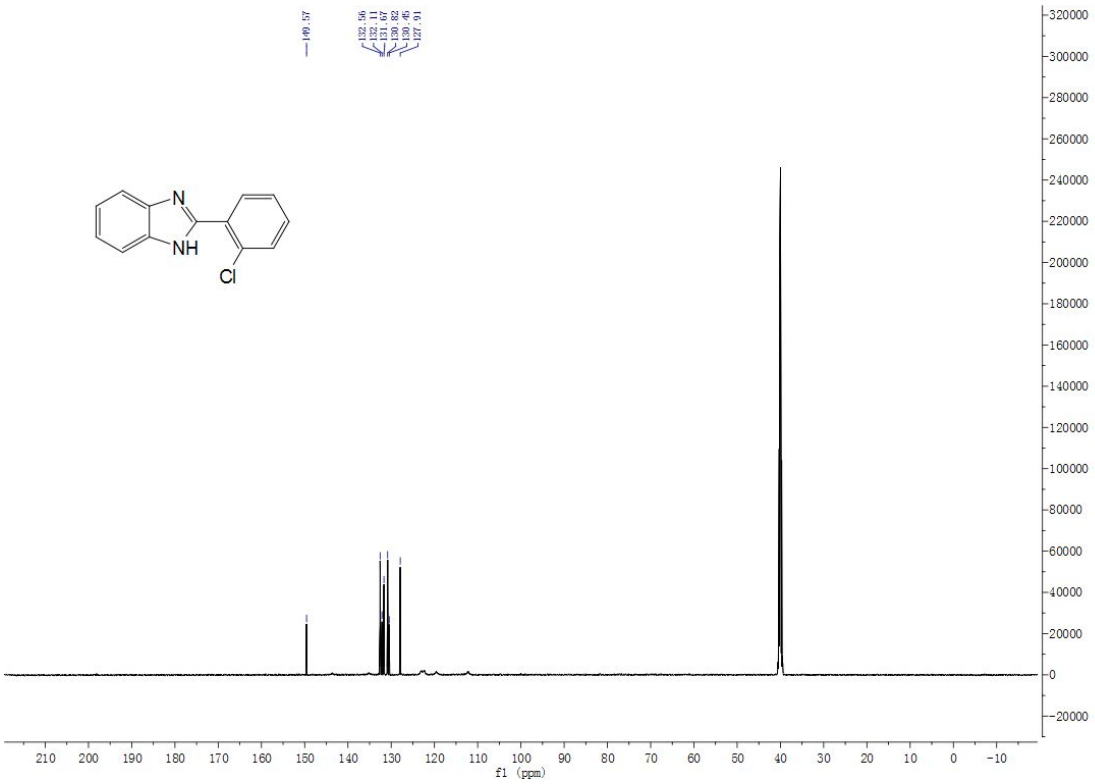
HRMS Spectrum of 2-(3-chlorophenyl)-1*H*-benzimidazole

2-(2-Chlorophenyl)-1*H*-benzimidazole characterization spectrum



^{1}H NMR Spectrum of 2-(2-chlorophenyl)-1*H*-benzimidazole



^{13}C NMR Spectrum of 2-(2-chlorophenyl)-1*H*-benzimidazole

Acquisition Parameter

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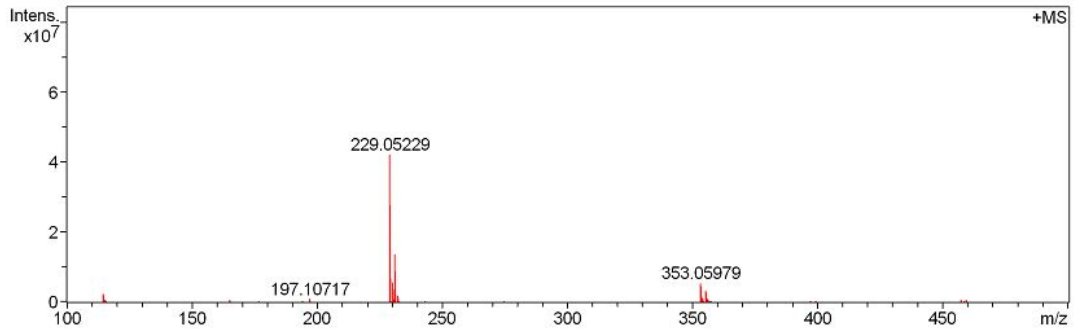
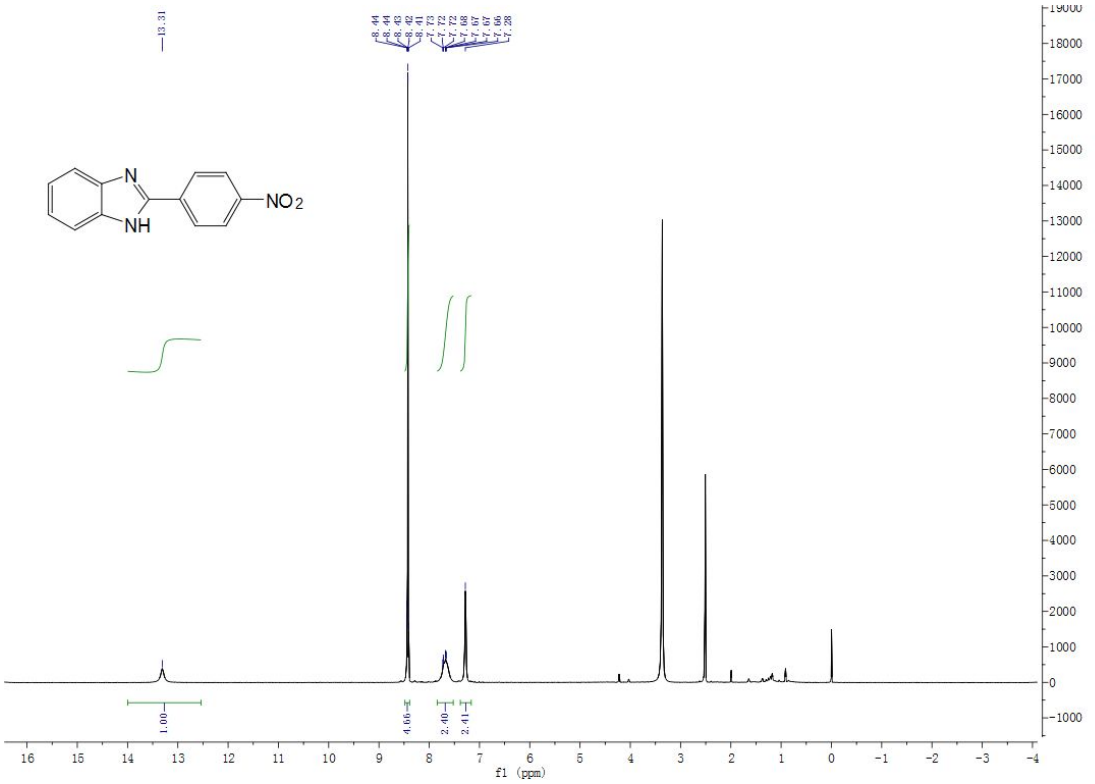
Broadband High Mass	1000.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Sat Apr 6 08:55:38 2013
Source Accumulation	0.0 sec	Drying Gas Flow Rate	4.0 L/min	Data Acquisition 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				



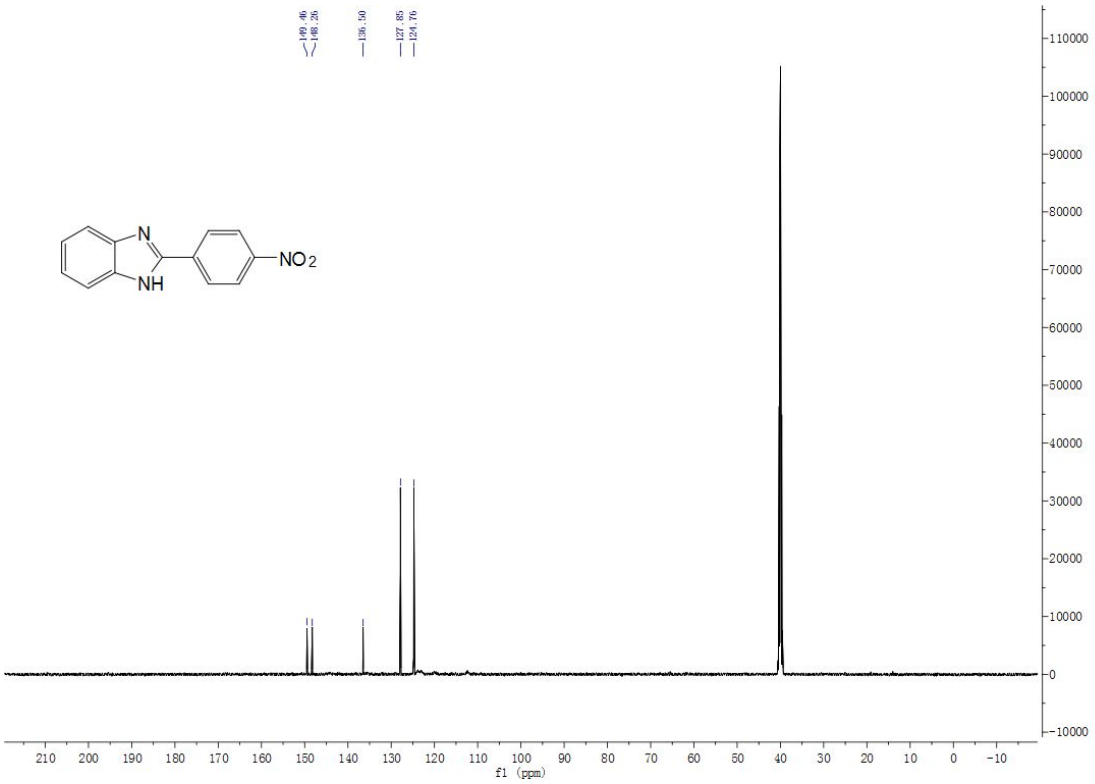
Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e¡¥Conf	N-Rule
229.05229	1	C 13 H 10 Cl N 2	100.00	229.05270	0.4	1.8	6.0	9.5	even	ok

HRMS Spectrum of 2-(2-chlorophenyl)-1*H*-benzimidazole

2-(4-Nitrophenyl)-1*H*-benzimidazole characterization spectrum



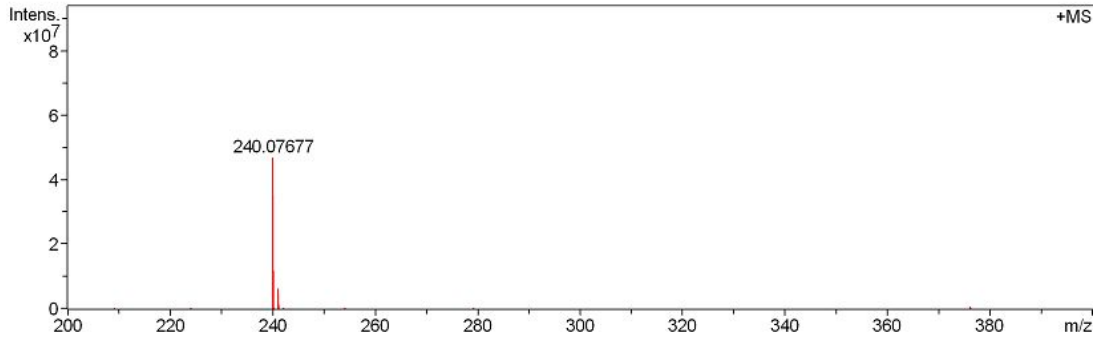
^{1}H NMR Spectrum of 2-(4-nitrophenyl)-1*H*-benzimidazole



^{13}C NMR Spectrum of 2-(4-nitrophenyl)-1*H*-benzimidazole

Acquisition Parameter

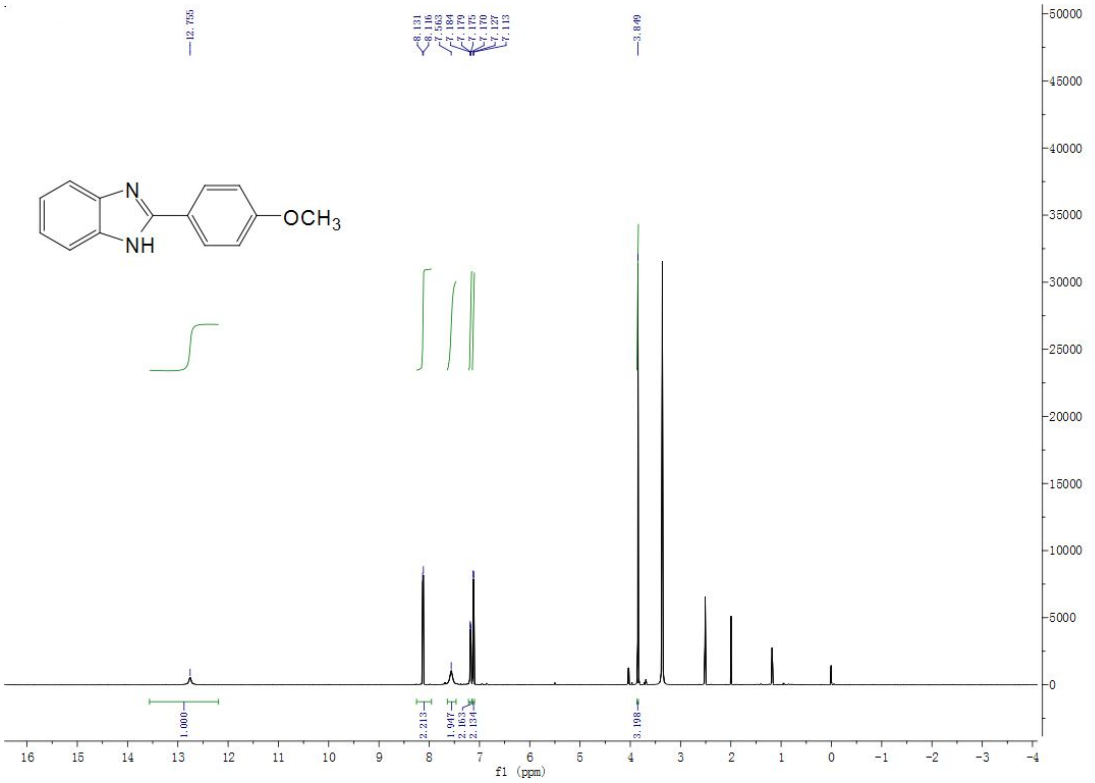
Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	2	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	1000.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Sat Apr 6 08:55:38 2013
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				



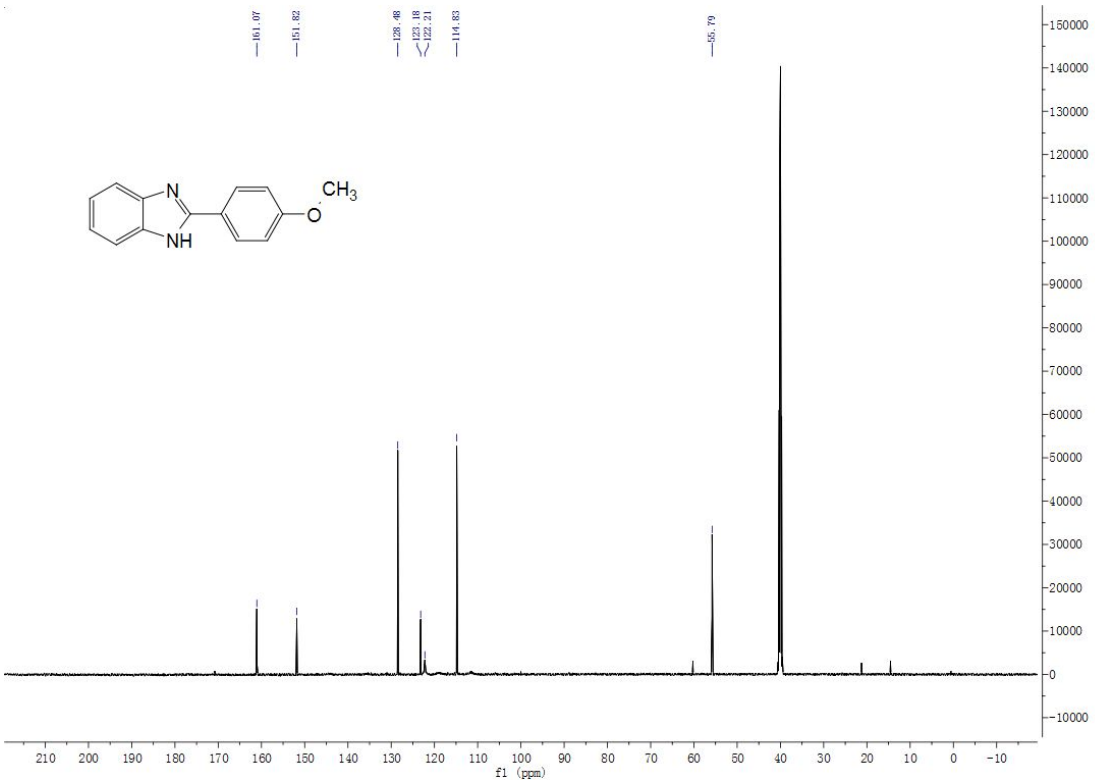
Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e¡¥Conf	N-Rule
240.07677	1	C 13 H 10 N 3 O 2	100.00	240.07675	-0.0	-0.1	8.1	10.5	even	ok

HRMS Spectrum of 2-(4-nitrophenyl)-1*H*-benzimidazole

2-(4-Methoxyphenyl)-1*H*-benzimidazole characterization spectrum



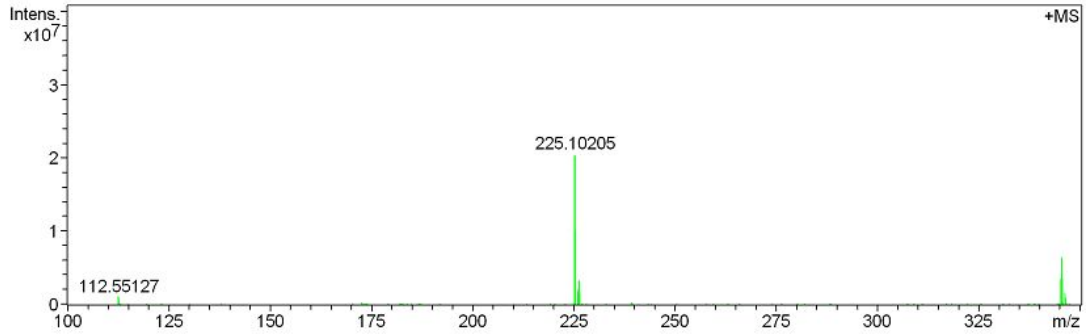
^{1}H NMR Spectrum of 2-(4-methoxyphenyl)-1*H*-benzimidazole



^{13}C NMR Spectrum of 2-(4-methoxyphenyl)-1*H*-benzimidazole

Acquisition Parameter

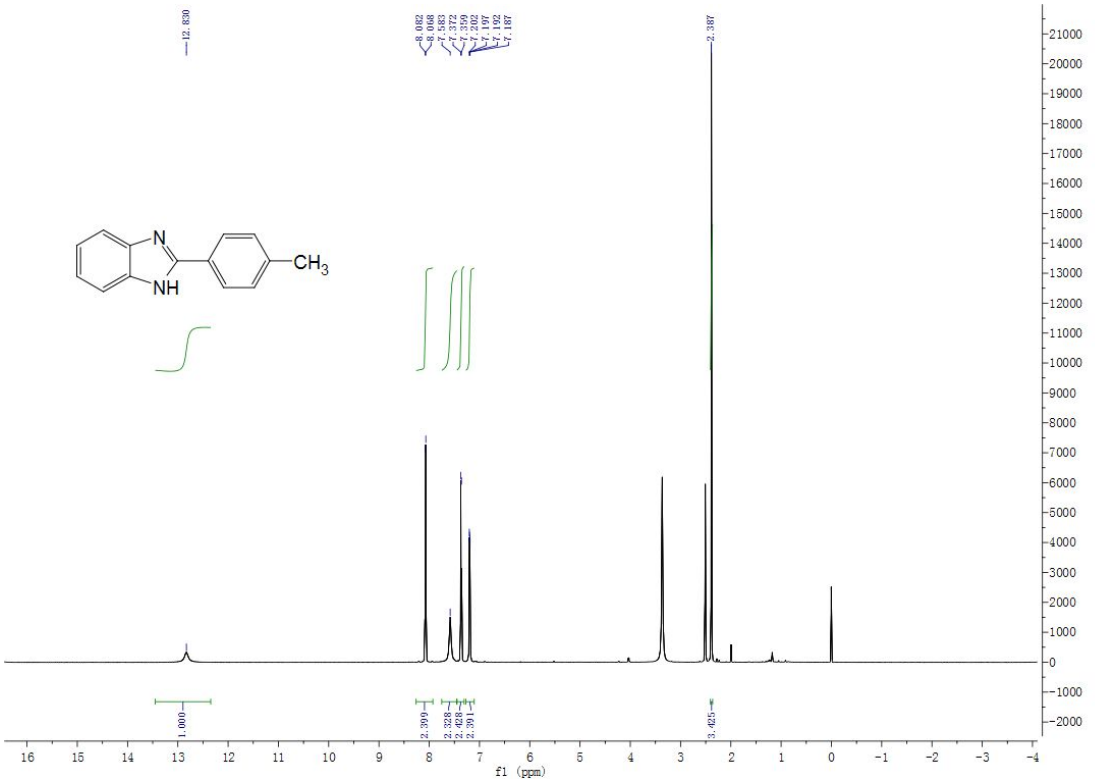
Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	2	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	1000.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Sat Apr 6 08:55:38 2013
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				



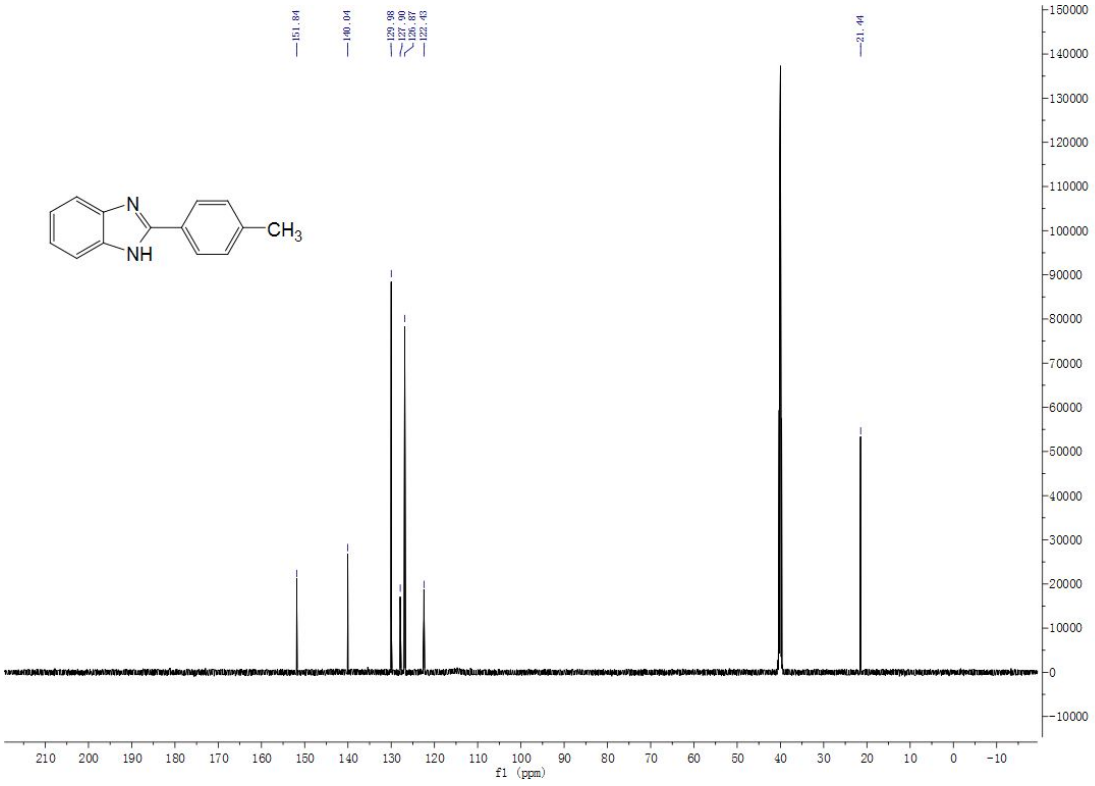
Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e¡¥Conf	N-Rule
225.10205	1	C 14 H 13 N 2 O	100.00	225.10224	0.2	0.8	2.1	9.5	even	ok

HRMS Spectrum of 2-(4-methoxyphenyl)-1*H*-benzimidazole

2-(4-Methylphenyl)-1*H*-benzimidazole characterization spectrum



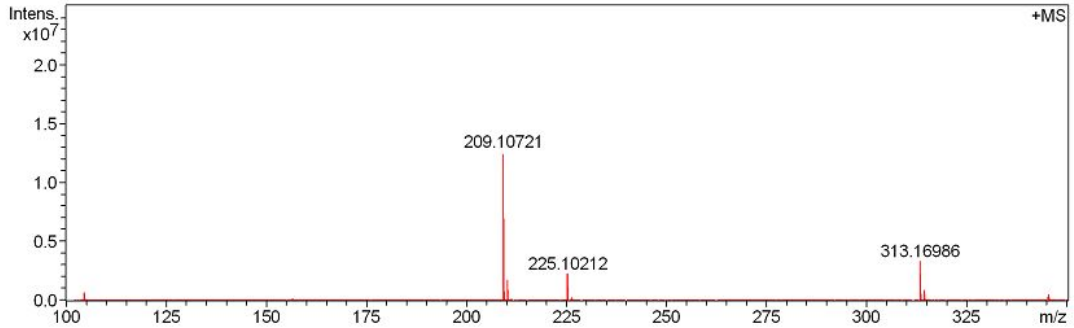
^{1}H NMR Spectrum of 2-(4-methylphenyl)-1*H*-benzimidazole



^{13}C NMR Spectrum of 2-(4-methylphenyl)-1*H*-benzimidazole

Acquisition Parameter

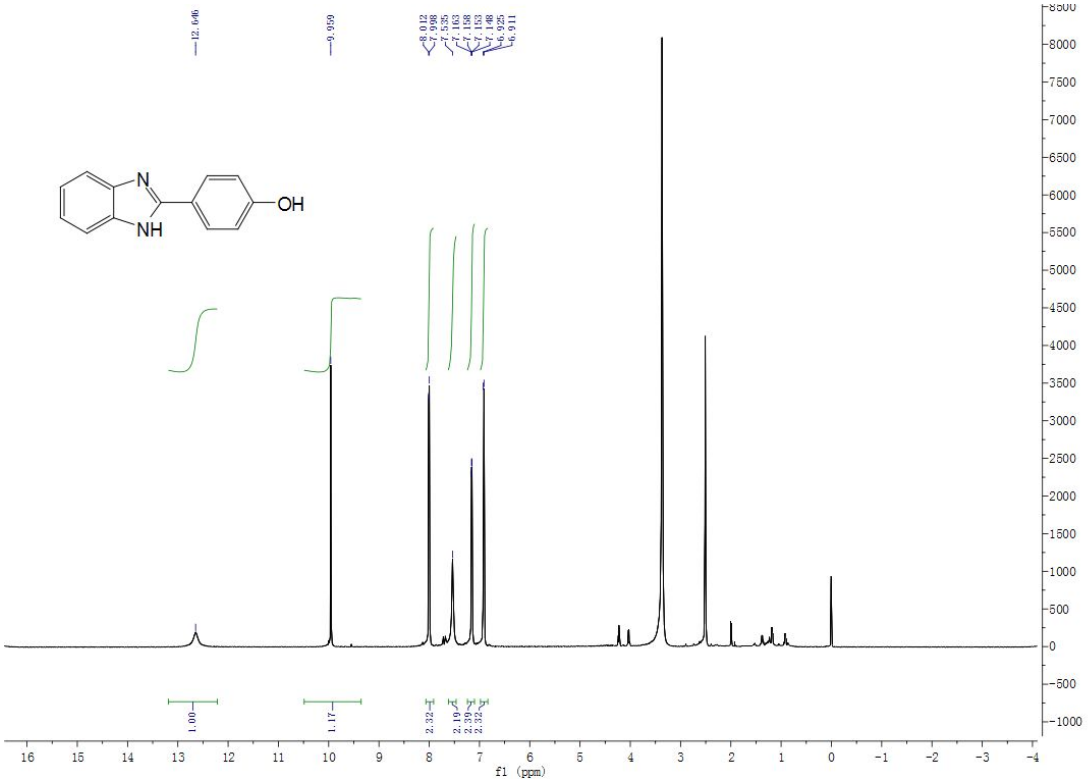
Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	2	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	1000.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Sat Apr 6 08:55:38 2013
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				



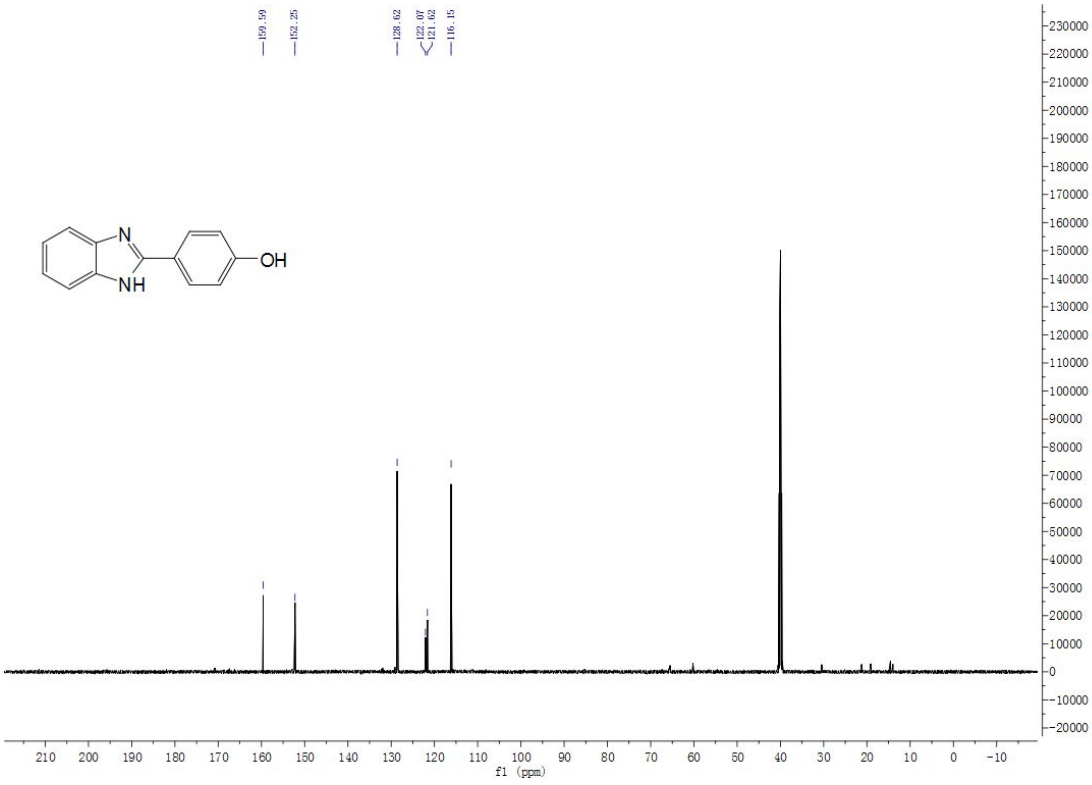
Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e¡¥Conf	N-Rule
209.10721	1	C 14 H 13 N 2	100.00	209.10732	0.1	0.6	11.9	9.5	even	ok

HRMS Spectrum of 2-(4-methylphenyl)-1*H*-benzimidazole

2-(4-Hydroxyphenyl)-1*H*-benzimidazole characterization spectrum



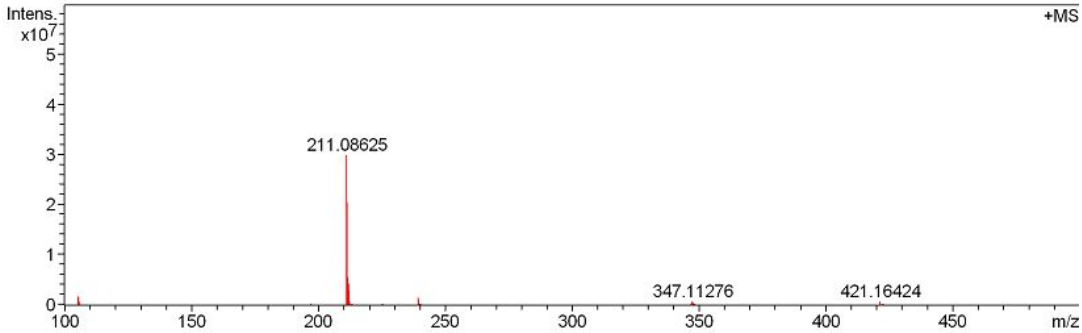
^{1}H NMR Spectrum of 2-(4-hydroxyphenyl)-1*H*-benzimidazole



^{13}C NMR Spectrum of 2-(4-hydroxyphenyl)-1*H*-benzimidazole

Acquisition Parameter

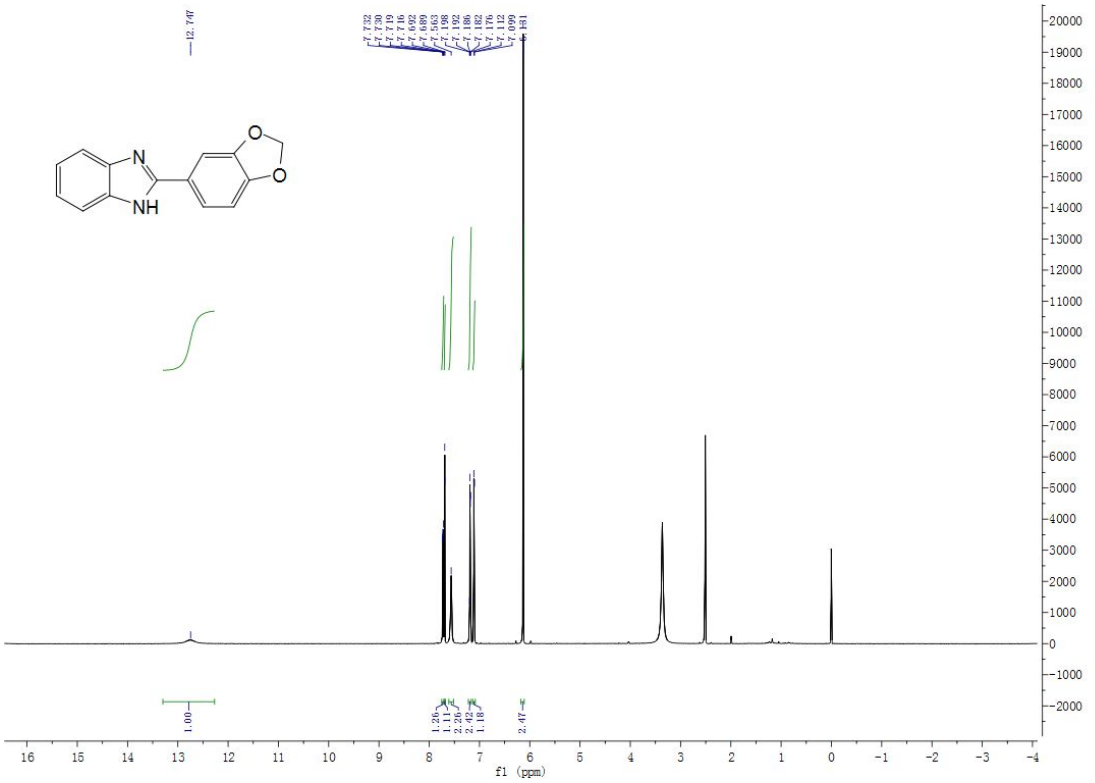
Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	2	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	1000.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Sat Apr 6 08:55:38 2013
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				



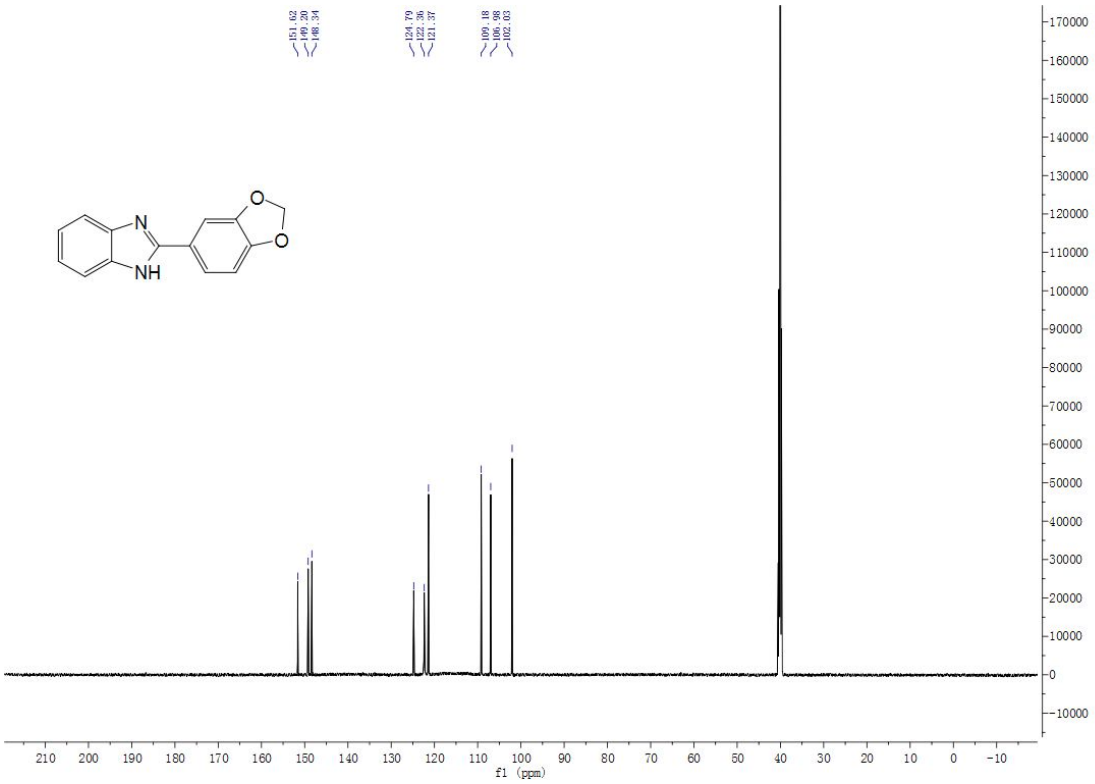
Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
211.08625	1	C 13 H 11 N 2 O	100.00	211.08659	0.3	1.6	2.5	9.5	even	ok

HRMS Spectrum of 2-(4-hydroxyphenyl)-1*H*-benzimidazole

2-(1,3-Benzodioxol-5-yl)-1*H*-benzimidazole characterization spectrum



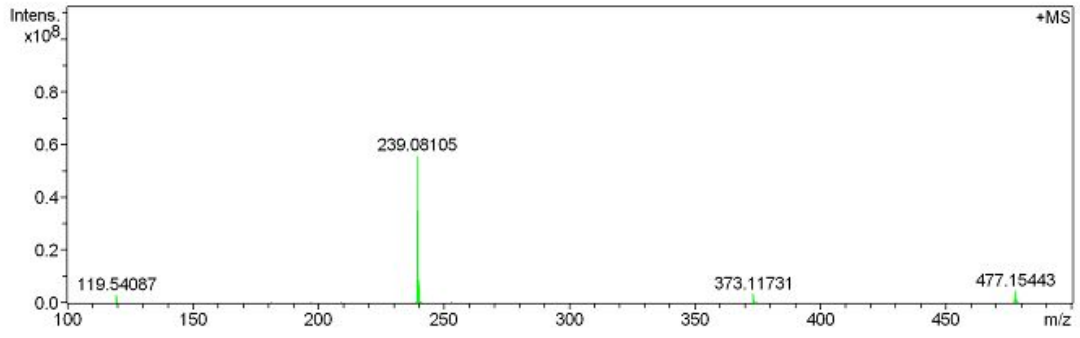
^{1}H NMR Spectrum of 2-(1,3-benzodioxol-5-yl)-1*H*-benzimidazole



^{13}C NMR Spectrum of 2-(1,3-benzodioxol-5-yl)-1*H*-benzimidazole

Acquisition Parameter

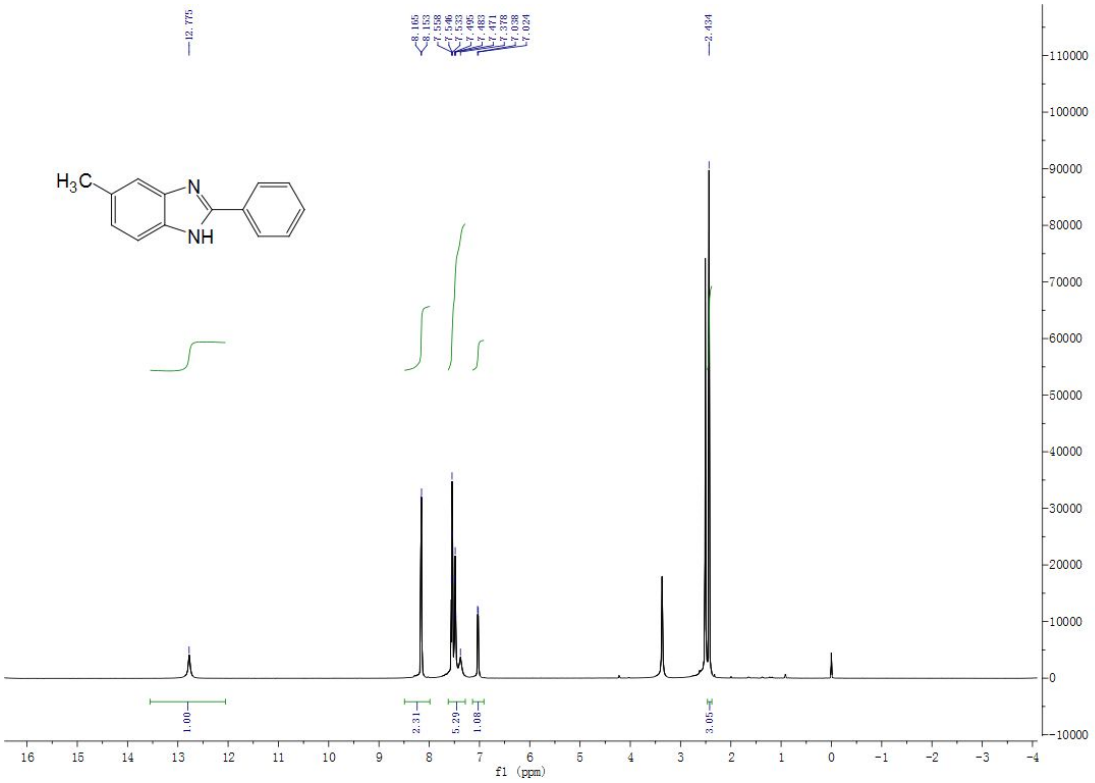
Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	2	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	1000.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Sat Apr 6 08:55:38 2013
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				



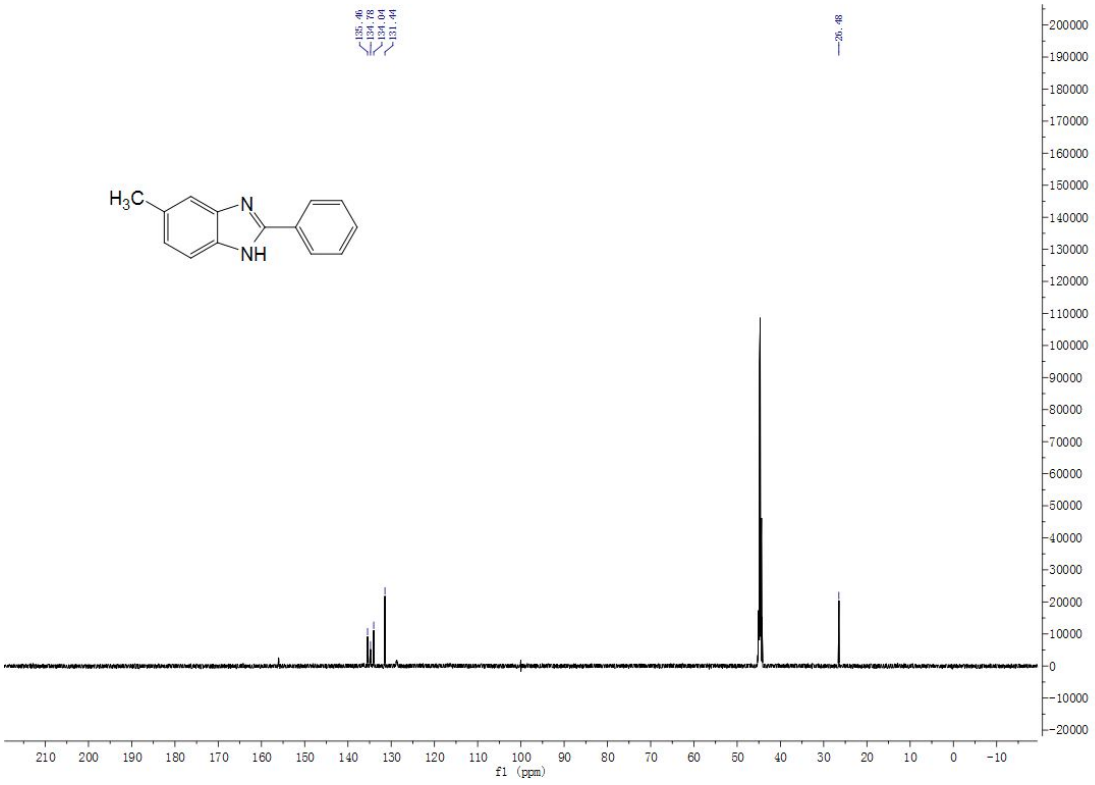
Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e¡¥Conf	N-Rule
239.08105	1	C 14 H 11 N 2 O 2	100.00	239.08150	0.5	1.9	3.5	10.5	even	ok

HRMS Spectrum of 2-(1,3-benzodioxol-5-yl)-1*H*-benzimidazole

5-Methyl-2-phenyl-1*H*-benzimidazole characterization spectrum



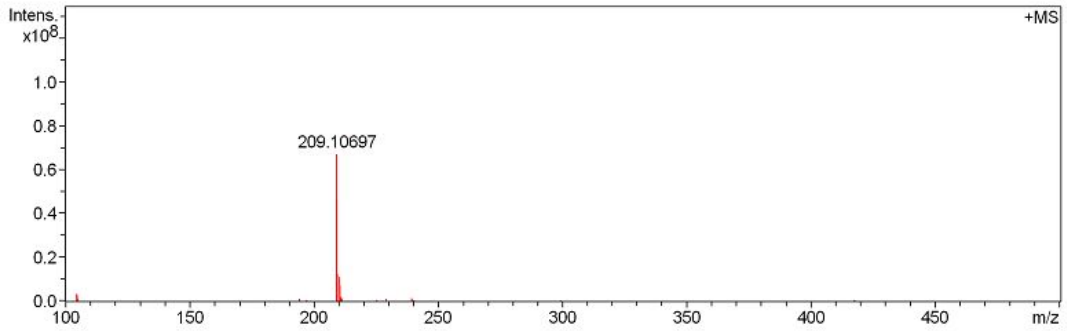
^{1}H NMR Spectrum of 5-methyl-2-phenyl-1*H*-benzimidazole



^{13}C NMR Spectrum of 5-methyl-2-phenyl-1*H*-benzimidazole

Acquisition Parameter

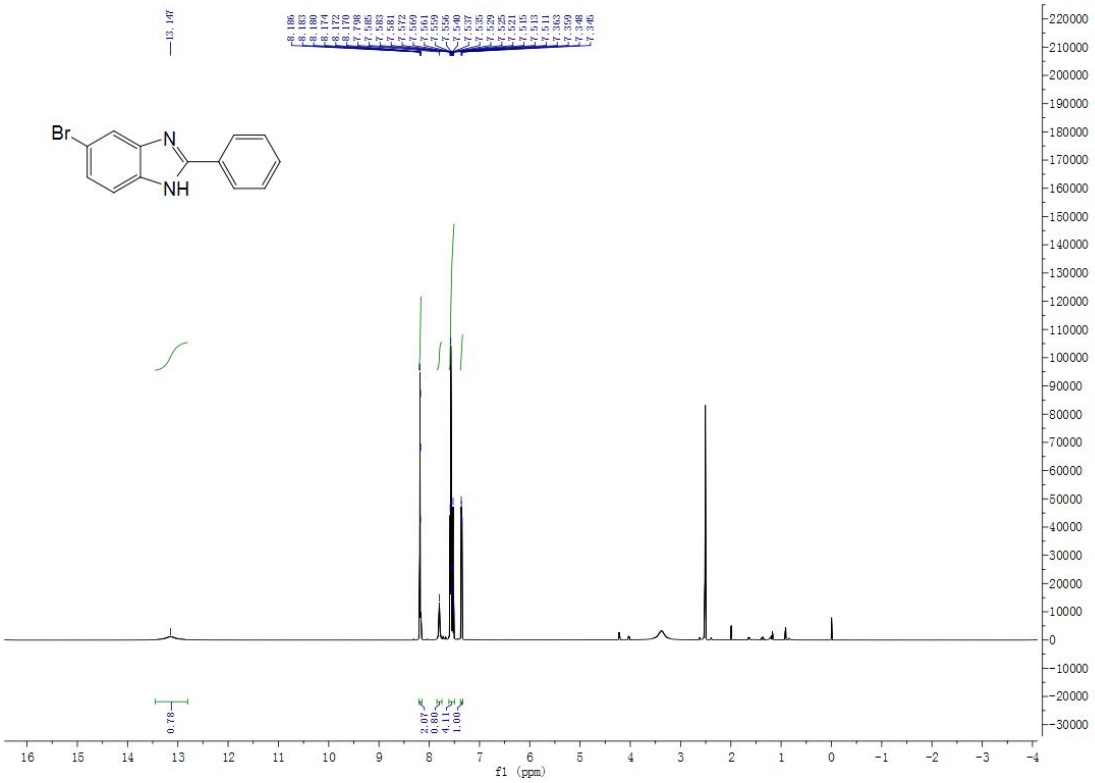
Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	2	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	1000.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Sat Apr 6 08:55:38 2013
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				



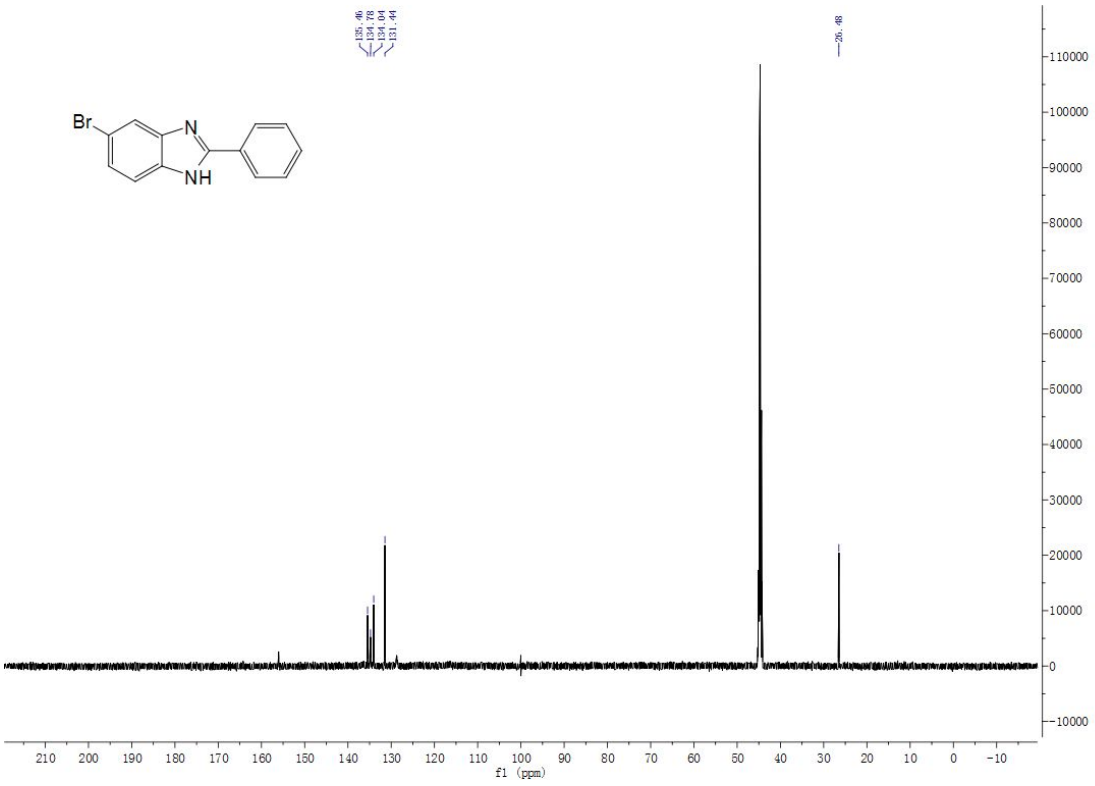
Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e¡¥Conf	N-Rule
209.10697	1	C 14 H 13 N 2	100.00	209.10732	0.4	1.7	1.6	9.5	even	ok

HRMS Spectrum of 5-methyl-2-phenyl-1*H*-benzimidazole

5-Bromo-2-phenyl-1*H*-benzimidazole characterization spectrum



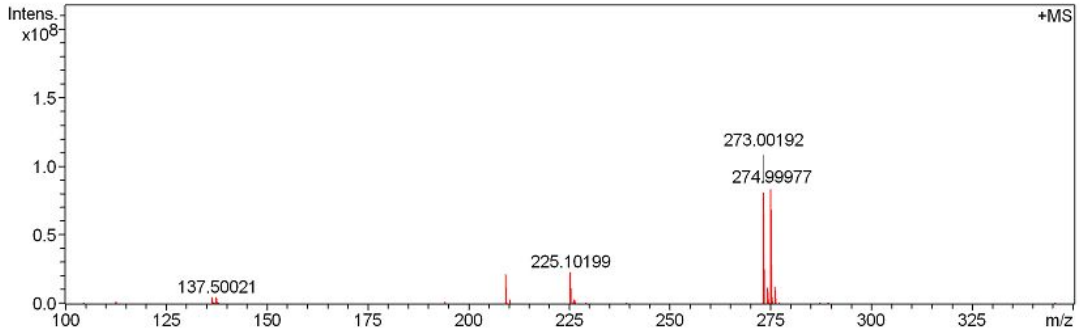
^{1}H NMR Spectrum of 5-bromo-2-phenyl-1*H*-benzimidazole



^{13}C NMR Spectrum of 5-bromo-2-phenyl-1*H*-benzimidazole

Acquisition Parameter

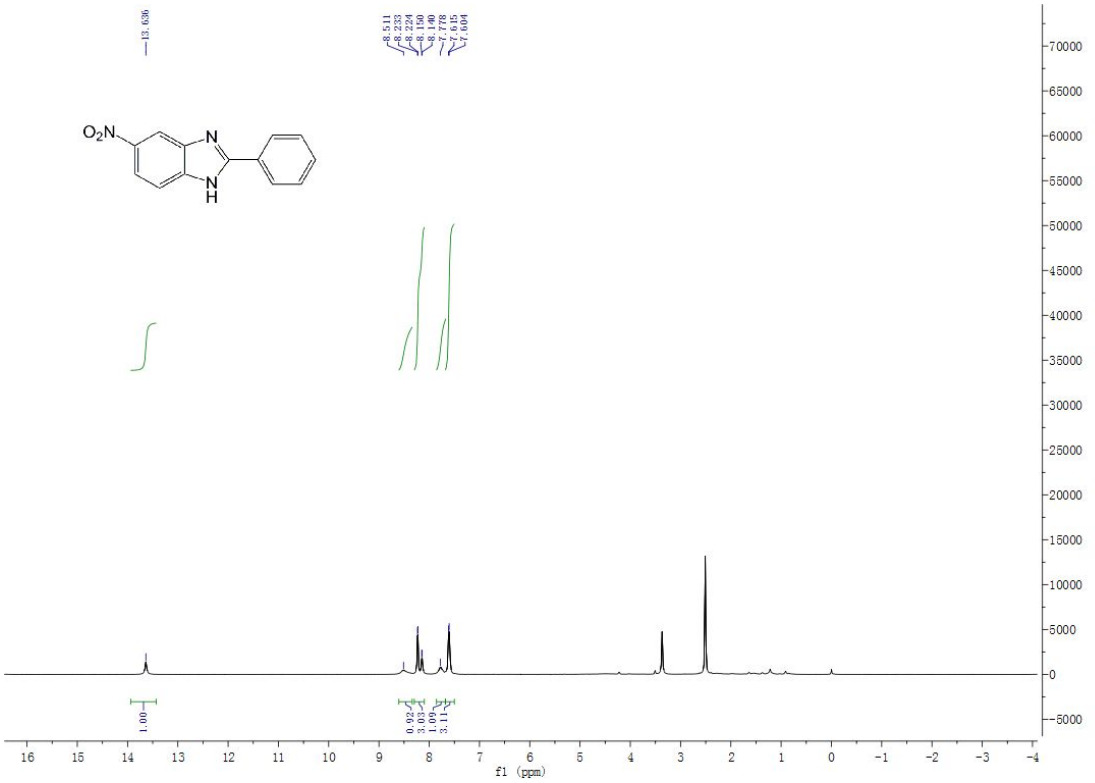
Polarity	Positive	Source	APCI	No. of Laser Shots	20
Averaged Scans	2	No. of Cell Fills	1	Laser Power	51.0 %
Broadband Low Mass	100.3 m/z	End Plate	1500.0 V	MALDI Plate	300.0 V
Broadband High Mass	1000.0 m/z	Capillary Entrance	2000.0 V	Imaging Spot Diameter	2000.0 µm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Sat Apr 6 08:55:38 2013
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				



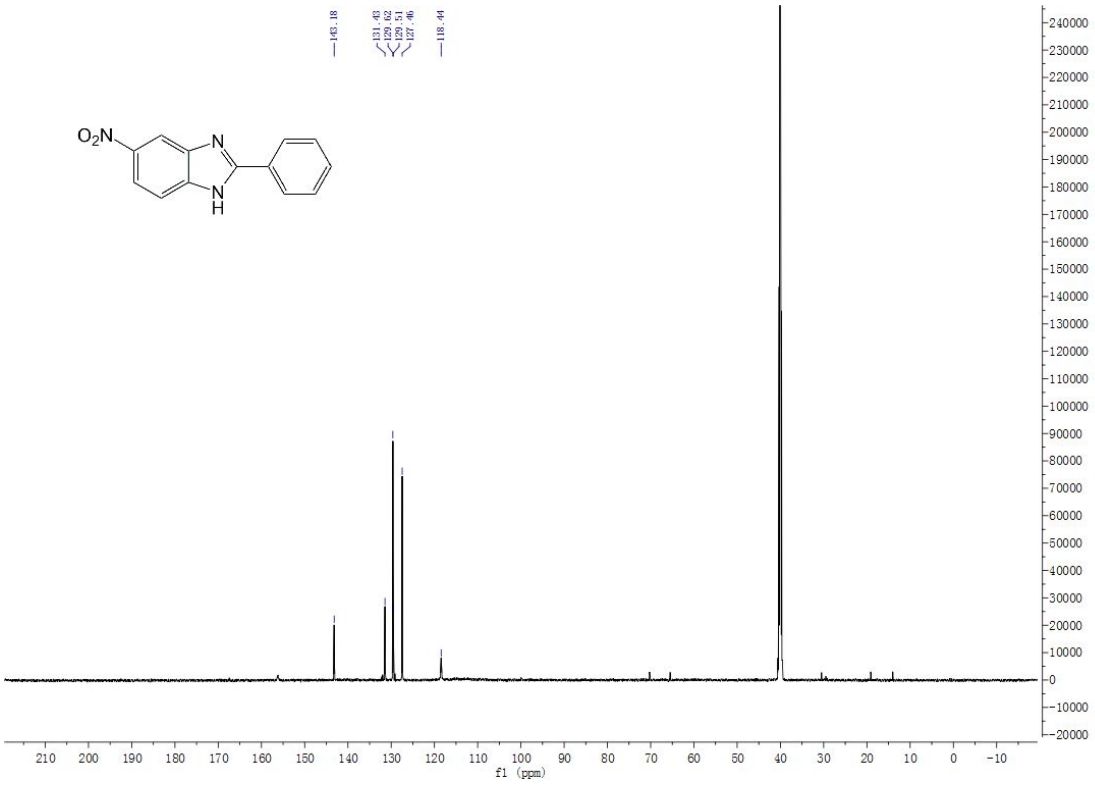
Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e¡¥Conf	N-Rule
273.00192	1	C 13 H 10 Br N 2	100.00	273.00219	0.3	1.0	14.2	9.5	even	ok

HRMS Spectrum of 5-bromo-2-phenyl-1*H*-benzimidazole

5-Nitro-2-phenyl-1*H*-benzimidazole characterization spectrum



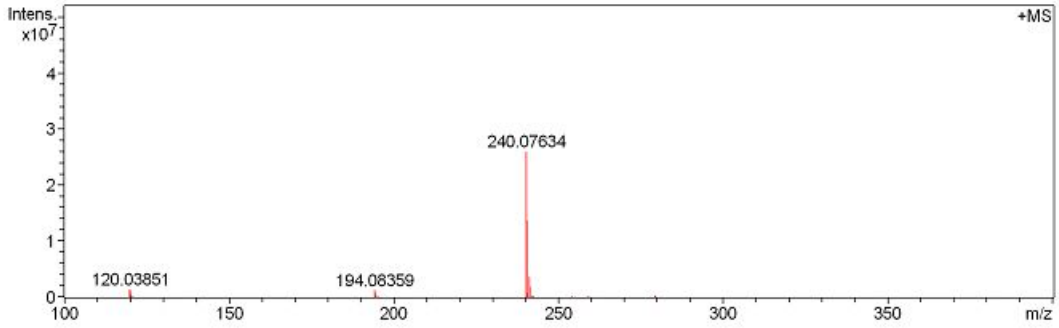
^{1}H NMR Spectrum of 5-nitro-2-phenyl-1*H*-benzimidazole



^{13}C NMR Spectrum of 5-nitro-2-phenyl-1*H*-benzimidazole

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	1000.0 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	Sat Apr 6 08:55:38 2013
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
240.07634	1	C 13 H 10 N 3 O 2	100.00	240.07675	0.4	1.7	6.2	10.5	even	ok

HRMS Spectrum of 5-nitro-2-phenyl-1*H*-benzimidazole