

Document: Supporting Information

Role of graphene oxide as heterogeneous acid catalyst and benign oxidant for synthesis of benzimidazoles and benzothiazoles

Dhopte, Kiran B.,¹ Zambare, Rahul S.,¹ Patwardhan, Anand V.,¹ Nemade, Parag R.^{1,2,*}

¹ Department of Chemical Engineering, Institute of Chemical Technology, Nathalal Parekh Marg, Matunga, Mumbai Maharashtra, 400019, India.

² Department of Oils, Oleochemicals and Surfactant Technology, Institute of Chemical Technology, Nathalal Parekh Marg, Mumbai, Maharashtra, 400 019, India.

Corresponding Author*

Tel.: + 91- 22 3361 2027;

Fax: +91- 22 3361 1020;

E-mail address: pr.nemade@ictmumbai.edu.in.

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General information

All the products are previously reported compounds and were also confirmed by ^1H NMR, ^{13}C NMR, MS, elemental analysis and melting point analysis. Mass spectra of compound were analyzed by both GC-MS and MS. GC-MS analysis were obtained on Shimadzu GCMS-QP 2010 (Rtx-17, 30 m $\times$ 25mm ID, film thickness 0.25 μm df) (column flow- 2 mL/min, 80 $^\circ\text{C}$ to 240 $^\circ\text{C}$ at 10 $^\circ$ /min. rise.) while MS spectra were obtained from Finnigan LCD Advantage Max mass spectrometer (LCQAD 30000, Thermo Electron Corporation, USA) with nitrogen sheath gas (flow rate of 40×10^{-6} m 3 /min) and auxiliary N $_2$ (flow rate 18×10^{-6} m 3 /min) where capillary temperature was maintained at 548 K with voltage of 420 V and ion spray voltage at 5 KV. Synthesized compounds were characterized by ^1H NMR (Varian 200 MHz NMR Spectrometer), ^{13}C NMR spectra (50 MHz). Chemical shifts are reported in parts per million (δ) relative to tetramethylsilane as internal standard. Melting point of synthesized compounds was measured by capillary method and by DSC, Q-100 (TA Instrument USA) with nitrogen flow rate of 50 mL/min.

Images of re-oxidized Graphene oxide

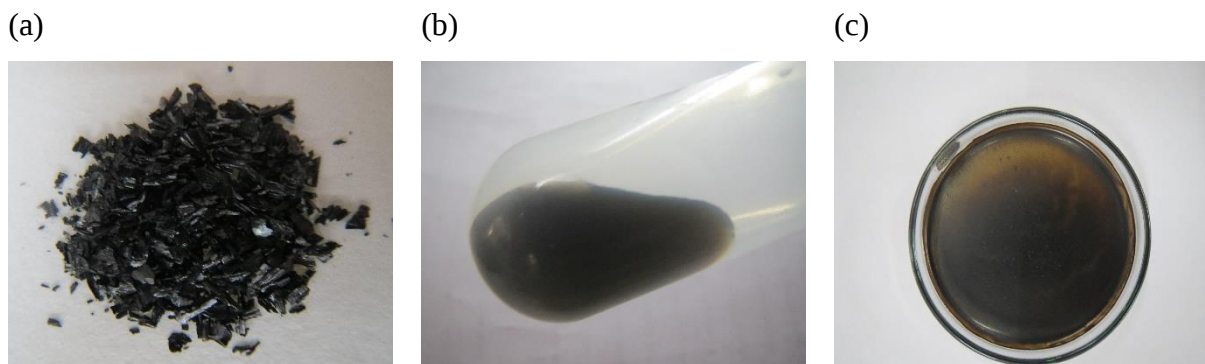


Figure S1. (a) Partially reduced GO after 5th recycle; (b) re-oxidized GO after centrifugation; (c) re-oxidized GO after drying

Table S1: Elemental analysis of pure Graphene oxide and graphene oxide recovered after 5th recycle for synthesis of benimidazoles and benzthiazoles.¹

Sample	Element (% Wt.)				Associated Moisture	O/C ratio (%)
	C	H	O	N		
Graphene oxide	37.3	2.8	33.3	0.1	26.5	
Graphene oxide (moisture-free)	50.8	3.8	45.3	0.1	-	89.3
Graphene oxide recovered after 5 th recycle (GO-R5)	60.0	1.7	13.9	4.5	19.9	
GO-R5 (moisture-free)	74.9	2.1	17.4	5.6	-	23.2

Spectral data of selected compounds

1a. 2-Phenyl-1,3-benzothiazole:² Yellow solid; mp: 110 °C; IR (KBr): $\nu_{\max}(\text{cm}^{-1}) = 3045, 1691, 1507, 1318, 1035, 806, 755$ cm^{-1} ; ^1H NMR (200 MHz, CDCl_3): δ 8.11–8.07 (m, 3H), 7.92–7.90 (d, $J = 8$ Hz, 1H), 7.52–7.48 (m, 4H), 7.41–7.37 (t, $J = 16$ Hz, 1h), ^{13}C NMR (50 MHz, CDCl_3): δ 168.06, 154.17, 135.09, 133.66, 130.96, 129.02, 127.58, 126.31, 125.19, 123.26, 121.62; GC-MS (EI) m/z (%) = 211(100) M^+ , 108(36), 82(11), 69(22); analytically calculated for $\text{C}_{13}\text{H}_9\text{NS}$: C, 73.92; H, 4.29; N, 6.63; S, 15.18 Found C, 73.92; H, 4.21; N, 6.54; S, 15.17

2a. 2-(4-Chlorophenyl)-1,3-benzothiazole: Yellow solid; mp: 117 °C; IR (KBr): $\nu_{\max}(\text{cm}^{-1}) = 3033, 1691, 1556, 1470, 1093, 973, 829, 756$; ^1H NMR (200 MHz, CDCl_3): δ 8.09–7.88 (m, 4H), 7.55–7.37 (m, 4H), ^{13}C NMR (50 MHz, CDCl_3): 129.32, 128.75, 126.53, 125.46, 123.32, 121.70; GC-MS (EI) m/z (%) = 245 (100) M^+ , 210 (10), 108 (37), 69 (28); analytically calculated for $\text{C}_{13}\text{H}_8\text{ClNS}$: C, 63.54; H, 3.28; N, 5.70; S, 13.04 Found C, 63.78; H, 3.16; N, 5.58; S, 13.01

3a. 2-(3-Chlorophenyl)-1,3-benzothiazole: White solid; mp: 116 °C; IR (KBr): $\nu_{\max}(\text{cm}^{-1}) = 3033, 1606, 1508, 1455, 1325, 1052, 756, 701$ cm^{-1} ; ^1H NMR (200 MHz, CDCl_3): δ 7.17–7.12 (m = 4H), 6.72–6.70 (d, $J = 8$ Hz, 2H), 6.61–6.56 (d, $J = 16$ Hz, 2H), ^{13}C NMR (50 MHz, CDCl_3): 148.62, 136.83, 133.72, 131.59, 130.23, 129.83, 128.27, 118.80, 118.27, 115.24; GC-MS (EI) m/z (%) = 245(100) M^+ , 210(15), 108(46), 82(18), 69(22); analytically calculated for $\text{C}_{13}\text{H}_8\text{ClNS}$: C, 63.54; H, 3.28; N, 5.70; S, 13.05 Found C, 63.51; H, 3.19; N, 5.60; S, 13.01

4a. 2-(4-Fluorophenyl)benzothiazole: Yellow solid; mp: 99 °C; IR (KBr): $\nu_{\max}(\text{cm}^{-1}) = 3035, 1691, 1608, 1323, 1050, 816, 756, 701$; ^1H NMR (200 MHz, CDCl_3): δ 8.13–8.04 (m, 3H), 7.93–7.89 (d, $J = 16$ Hz, 1H), 7.54–7.35 (m, 2H), 7.23–7.15 (m, 2H), ^{13}C NMR (50 MHz, CDCl_3): 166.90, 163.36, 154.25, 135.21, 129.73, 126.58, 125.41, 123.35, 121.78, 116.21; GC-MS (EI) m/z (%) =

229 (100) M^+ , 114 (08), 108 (50), 82 (14), 69 (26); analytically calculated for $C_{13}H_8FNS$: C, 68.11; H, 3.51; N, 6.11; S, 13.98 Found C, 68.18; H, 3.41; N, 6.12; S, 13.88

5a. 2-(4-Bromophenyl)-1,3-benzothiazole: Yellow solid; mp: 126 °C; IR (KBr): $\nu_{\max}(\text{cm}^{-1}) = 3310, 1596, 1070, 972, 829, 754$; ^1H NMR (200 MHz, CDCl_3): δ 8.09–7.90 (m, 4H), 7.67–7.37 (m, 4H), ^{13}C NMR (50 MHz, CDCl_3): 154.20, 135.17, 132.38, 129.05, 126.65, 125.59, 123.45, 121.81; GC-MS (EI) m/z (%) = 291 (100) M^+ , 210 (47), 108 (52), 69 (55); analytically calculated for $C_{13}H_8BrNS$: C, 53.81; H, 2.77; N, 4.82; S, 11.05 Found C, 53.51; H, 2.39; N, 4.78; S, 10.96

6a. 2-(4-Nitrophenyl)benzothiazole: Yellow solid; mp: 221 °C; IR (KBr): $\nu_{\max}(\text{cm}^{-1}) = 3330, 1624, 1515, 1352, 1000, 754$; ^1H NMR (200 MHz, CDCl_3): δ 8.14–8.06 (m, 1H), 7.86–7.64 (m, 3H), 7.34–7.12 (m, 3H), 6.71–6.58 (m, 1H), ^{13}C NMR (50 MHz, CDCl_3): 155.64, 148.18, 134.03, 133.75, 132.43, 130.20, 127.89, 127.33, 126.28, 124.59, 117.67, 115.22; GC-MS (EI) m/z (%) = 256 (100) M^+ , 239 (32), 94 (20); analytically calculated for $C_{13}H_8N_2O_2S$: C, 60.93; H, 3.15; N, 10.93; S, 12.51 Found C, 60.86; H, 3.07; N, 10.92; S, 12.64

7a. 4-Benzothiazole-2-phenyl-dimethylamine: White solid; mp: 175 °C; IR (KBr): $\nu_{\max}(\text{cm}^{-1}) = 3050, 1621, 1509, 1228, 1015, 815, 755, 703$; ^1H NMR (200 MHz, CDCl_3): δ 8.00–7.82 (m, 4H), 7.44–7.30 (m, 2H), 6.77–6.73 (d, $J = 16$ Hz, 2H), 3.06 (s, 6H), ^{13}C NMR (50 MHz, CDCl_3): 152.34, 134.61, 129.04, 126.15, 124.36, 122.38, 121.50, 111.84, 40.33; GC-MS (EI) m/z (%) = 254 (100) M^+ , 238 (13), 177 (21), 142 (20), 101 (17); analytically calculated for $C_{15}H_{14}N_2S$: C, 70.83; H, 5.54; N, 11.01; S, 12.60 Found C, 70.25; H, 5.17; N, 11.01; S, 12.17

8a. 2-(4-Methylphenyl)benzothiazole: White solid; mp: 79 °C IR (KBr): $\nu_{\max}(\text{cm}^{-1}) = 3412, 2936, 1610, 1484, 1313, 962, 829$; ^1H NMR (200 MHz, CDCl_3): δ 8.02–7.82 (m, 4H), 7.47–7.20 (m, 4H), 2.37 (s, 3H), ^{13}C NMR (50 MHz, CDCl_3): 129.76, 127.52, 125.04, 123.07, 121.62, 21.58;

GC-MS (EI) m/z (%) = 225 (100) M^+ , 108 (13), 82 (13), 69 (18); analytically calculated for $C_{14}H_{11}NS$: C, 74.63; H, 4.91; N, 6.21; S, 14.23 Found C, 74.31; H, 4.87; N, 6.14; S, 14.32

9a. 2-(4-Methoxyphenyl)benzothiazole: white solid; mp: 113 °C; IR (KBr): $\nu_{\max}(\text{cm}^{-1})$ = 3060, 1691, 1605, 1506, 1037, 758, 702 cm^{-1} ; ^1H NMR (200 MHz, CDCl_3): δ 8.05–8.02 (d, J = 12 Hz, 3H), 7.88–7.86 (d, J = 8 Hz, 1H), 7.49–7.45 (t, J = 16 Hz, 1H), 7.37–7.33 (t, J = 16 Hz, 1H), 7.20–7.14 (m, 1H), 7.01–6.99 (d, J = 8 Hz, 2H), 3.88 (s, 3H), ^{13}C NMR (50 MHz, CDCl_3): 167.85, 161.94, 154.25, 137.06, 134.88, 131.92, 129.12, 128.38, 126.20, 124.79, 122.84, 121.41, 114.38, 55.47; GC-MS (EI) m/z (%) = 241(100) M^+ , 226 (41), 198 (32); analytically calculated for $C_{14}H_{11}NOS$: C, 69.68; H, 4.59; N, 5.80; S, 13.28 Found C, 68.24; H, 4.53; N, 5.66; S, 13.11

10a. 2-(2-Thienyl)-1,3-benzothiazole: Yellow solid; mp: 90 °C; IR (KBr): $\nu_{\max}(\text{cm}^{-1})$ = 3015, 1693, 1606, 1505, 1318, 1052, 757, 701; ^1H NMR (200 MHz, CDCl_3): δ 8.05–8.01 (d, J = 16 Hz, 1H), 7.87–7.83 (d, J = 16 Hz, 1H), 7.66–7.64 (d, J = 8 Hz, 1H), 7.52–7.32 (m, 3H), ^{13}C NMR (50 MHz, CDCl_3): 163.00, 153.87, 137.52, 134.86, 129.42, 128.73, 128.18, 126.57, 125.37, 123.14, 121.59, 115.33; GC-MS (EI) m/z (%) = 217 (100) M^+ , 108 (30), 82 (14), 69 (26); analytically calculated for $C_{11}H_7NS_2$: C, 60.82; H, 3.24; N, 6.44; S, 29.52 Found C, 60.61; H, 3.16; N, 6.48; S, 29.47

11a. 2-(2-Hydroxyphenyl)benzothiazole: Yellow solid; mp: 110 °C; IR (KBr): $\nu_{\max}(\text{cm}^{-1})$ = 3547, 1622, 1521, 1321, 1021, 806, 750, 699; ^1H NMR (200 MHz, CDCl_3): δ 8.62 (s, 1H), 7.68 (s, 1H), 7.44–7.36 (m, 3H), 7.01–6.73 (m, 4H), ^{13}C NMR (50 MHz, CDCl_3): 202.53, 159.91, 153.94, 134.62, 129.36, 126.34, 124.85, 122.52, 121.57, 116.10; MS (EI) m/z (%) = 227 (100) M^+ , 153 (10), 113 (25); analytically calculated for $C_{13}H_9NOS$: C, 68.70; H, 3.99; N, 6.16; S, 14.10 Found C, 68.72; H, 3.63; N, 6.09; S, 13.96

12a. 2-(4-Hydroxyphenyl)benzothiazole: Yellow solid; IR (KBr): $\nu_{\max} (\text{cm}^{-1}) = 3573, 1606, 1508, 1318, 1035, 806, 756, 700$; ^1H NMR (200 MHz, CDCl_3): δ 8.05–7.86 (m, 2H), 7.48–7.30 (m, 3H), 6.97–6.92 (d, $J = 20$ Hz 1H), 6.28–6.02 (m, 1H), 5.80–5.59 (m, 1H), ^{13}C NMR (50 MHz, CDCl_3): 160.49, 129.02, 126.41, 124.89, 122.28, 116.06; analytically calculated for $\text{C}_{13}\text{H}_9\text{NOS}$: C, 68.70; H, 3.99; N, 6.16; S, 14.10 Found C, 68.02; H, 3.91; N, 6.07; S, 13.96

13a. 2-Styrylbenzothiazole:³ Off white solid; mp: 112 °C;⁴ ^1H NMR (200 MHz, CDCl_3): δ 8.03–8.01 (m, 1H), 7.66–7.62 (m, 2H), 7.43–7.39 (m, 3H), 7.33–7.29 (m, 2H), 7.10–7.03 (m, 3H); ^{13}C NMR (50 MHz, CDCl_3): 129.18, 129.01, 128.95, 128.71, 126.44, 110.31; GC-MS (EI) m/z (%) = 236 (100) M^+ , 221 (40), 193 (43), 165 (30), 76 (18)

14a. 2-Propylbenzothiazole: ^1H NMR (200 MHz, CDCl_3): δ 7.99–7.93 (m, 1H), 7.82–7.80 (m, 1H), 7.44–7.40 (m, 1H), 7.34–7.30 (m, 1H), 3.09–3.06 (t, $J = 6$ Hz, 2H), 1.93–1.84 (m, 2H), 1.06–1.01 (t, $J = 10$ Hz, 3H); ^{13}C NMR (50 MHz, CDCl_3): 172.20, 135.09, 125.82, 124.59, 122.45, 121.44, 36.20, 23.09, 13.70; GC-MS (EI) m/z (%) = 177 (25) M^+ , 149 (100), 108 (20), 69 (18), 45 (17)

15a. 2-Phenylbenzimidazole:⁵ Brown solid; mp: 293 °C;⁶ IR (KBr): $\nu_{\max} (\text{cm}^{-1}) = 3456, 1412, 1280, 1124, 972, 745$; ^1H NMR (200 MHz, CDCl_3): δ 8.08–8.06 (d, $J = 8$ Hz, 2H), 7.66–7.64 (m, 2H), 7.52–7.46 (m, 3H), 7.30–7.26 (m, 2H), ^{13}C NMR (50 MHz, CDCl_3): 151.64, 130.40, 129.25, 126.70, 123.20; GC-MS (EI) m/z (%) = 194 (100) M^+ , 166 (8), 97 (13), 63 (11); analytically calculated for $\text{C}_{13}\text{H}_{10}\text{N}_2$: C, 80.38; H, 5.18; N, 14.42; Found C, 80.15; H, 5.51; N, 14.30

16a. 2-(3-Nitrophenyl)-1H-benzimidazole: Yellow solid; mp: 205 °C; IR (KBr): $\nu_{\max} (\text{cm}^{-1}) = 3452, 1522, 1344, 741$; ^1H NMR (200 MHz, CDCl_3): δ 8.84 (s, 1H), 8.52–8.48 (d, $J = 16$ Hz, 1H), 8.33–8.29 (d, $J = 16$ Hz, 1H), 7.75–7.67 (t, $J = 16$ Hz, $J = 16$, 3H), 7.37–7.31 (m, 2H), ^{13}C NMR (50

MHz, CDCl₃): 138.50, 130.95, 125.98, 125.00, 121.93, 111.50; analytically calculated for C₁₃H₉N₃O₂: C, 65.27; H, 3.79; N, 17.56; Found C, 65.15; H, 3.51; N, 17.50

MS (ESI): m/z [M + K]⁺ = 278.04

17a 2-(3-Chlorophenyl)-1H-benzimidazole: Yellow solid; mp: 290 °C; IR (KBr): $\nu_{\max}(\text{cm}^{-1})$ = 3100, 1579, 1438, 1124, 743; ¹H NMR (200 MHz, CDCl₃): δ 8.00 (s, 1H), 7.88–7.82 (m, 1H), 7.39–7.36 (m, 2H), 7.26–7.21 (m, 4H), ¹³C NMR (50 MHz, CDCl₃): 149.04, 148.36, 143.57, 132.48, 130.70, 124.22, 123.26, 120.82, 119.23, 111.68.

MS (ESI): m/z [M + K]⁺ = 267.01

18a. 2-(4-Fluorophenyl)-1H-benzimidazole: Off white; mp: 249 °C; IR (KBr): $\nu_{\max}(\text{cm}^{-1})$ = 3252, 1608, 1442, 1229, 1158, 868, 750; ¹H NMR (200 MHz, CDCl₃): δ 8.03–8.00 (m, 2H), 7.60–7.57 (m, 2H), 7.25–7.22 (m, 2H), 7.12–7.07 (m, 2H), ¹³C NMR (50 MHz, CDCl₃): 165.20, 162.71, 151.18, 131.30, 128.70, 127.77, 126.27, 122.97, 116.30; GC-MS (EI) m/z (%) = 212 (100) M⁺, 106 (12), 64 (11); analytically calculated for C₁₃H₉FN₂: C, 73.57; H, 4.27; N, 13.19; Found C, 73.15; H, 4.16; N, 13.04

19a. 2-(4-Bromophenyl)-1H-benzimidazole: White solid; mp: 296 °C; IR (KBr): $\nu_{\max}(\text{cm}^{-1})$ = 3301, 1595, 1432, 1276, 964, 829, 754; ¹H NMR (200 MHz, CDCl₃): δ 7.93–7.91 (d, J = 8 Hz, 2H), 7.61–7.59 (d, J = 8 Hz, 2H), 7.26–7.23 (m, 4H), ¹³C NMR (50 MHz, CDCl₃): 154.02, 152.05, 150.80, 148.43, 141.65, 132.29, 128.12, 123.12; analytically calculated for C₁₃H₉BrN₂: C, 57.16; H, 3.32; N, 10.25; Found C, 57.18; H, 3.25; N, 10.20

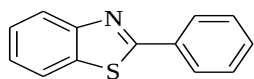
20a. 2-(2-Hydroxyphenyl)-1H-benzimidazole: Brown solid; mp: 241 °C; IR (KBr): $\nu_{\max}(\text{cm}^{-1})$ = 3435, 1597, 1458, 1262, 754; ¹H NMR (200 MHz, CDCl₃): δ 7.64 (br, s, 1H), 7.61–7.59 (d, J = 8 Hz, 1H), 7.39–7.28 (m, 4H), 7.16–7.12 (t, J = 16 Hz, 1H), 6.96–6.78 (m, 2H), ¹³C NMR (50 MHz,

CDCl₃): 136.56, 134.91, 129.55, 126.43, 125.03, 122.92, 121.68, 116.11; GC-MS (EI) *m/z* (%) = 209 (100) M⁺, 180 (22), 105 (92), 77 (88), 51 (37); analytically calculated for C₁₃H₉ON₂: C, 74.63; H, 4.33; N, 13.38; Found C, 74.28; H, 4.24; N, 13.25

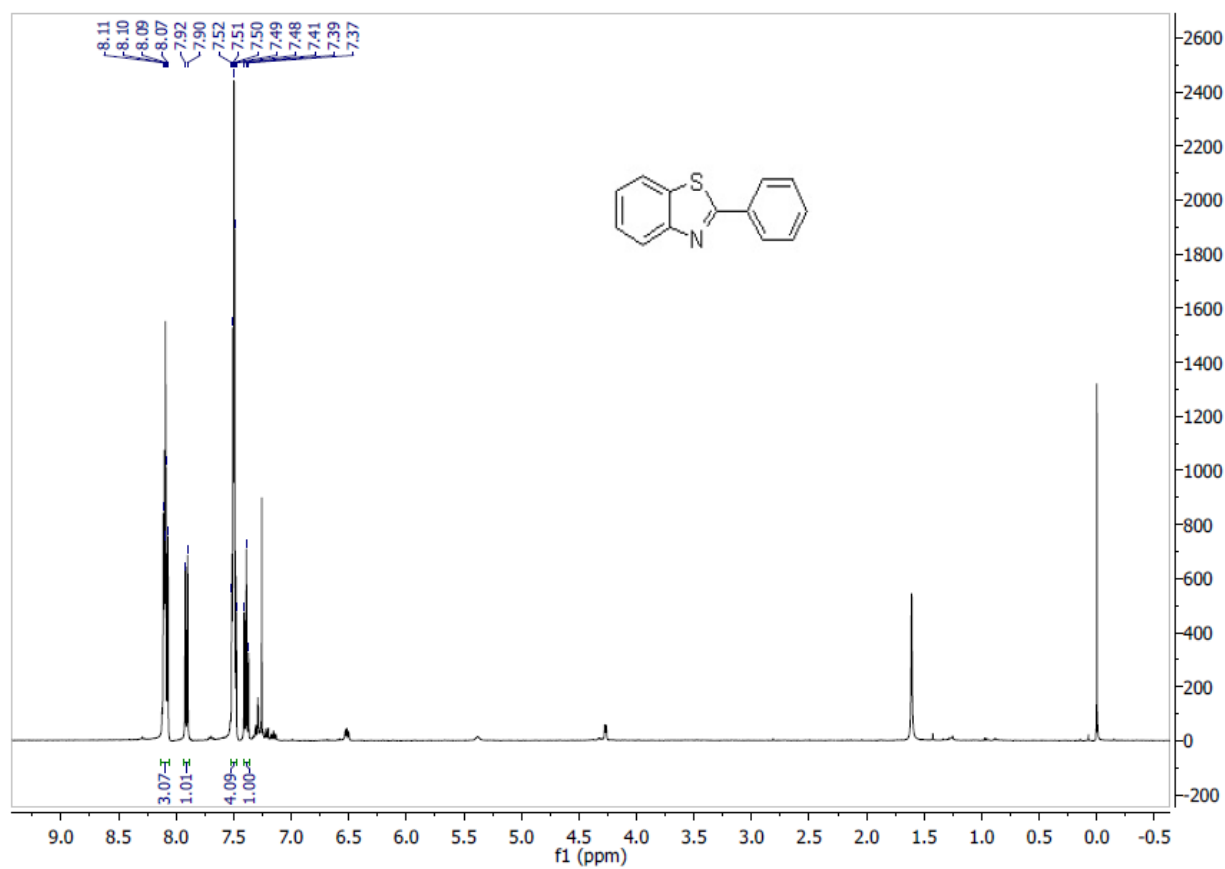
21a. 6-(MethylPhenyl)-1H-benzimidazole:⁷ White solid; mp: 245 °C; ¹H NMR (200 MHz, CDCl₃): δ 7.96 (s, 1H), 7.39–7.34 (d, *J* = 10 Hz, 2H), 7.24–7.19 (m, 3H), 7.08–7.00 (m, 2H), 2.41 (s, 3H); ¹³C NMR (50 MHz, CDCl₃): 166.67, 153.79, 137.65, 130.95, 129.42, 128.93, 127.52, 126.31, 125.35, 123.19, 122.91, 121.60; GC-MS (EI) *m/z* (%) = 209 (43) M⁺, 139 (12), 111 (44), 97 (87), 57 (100), 43 (64)

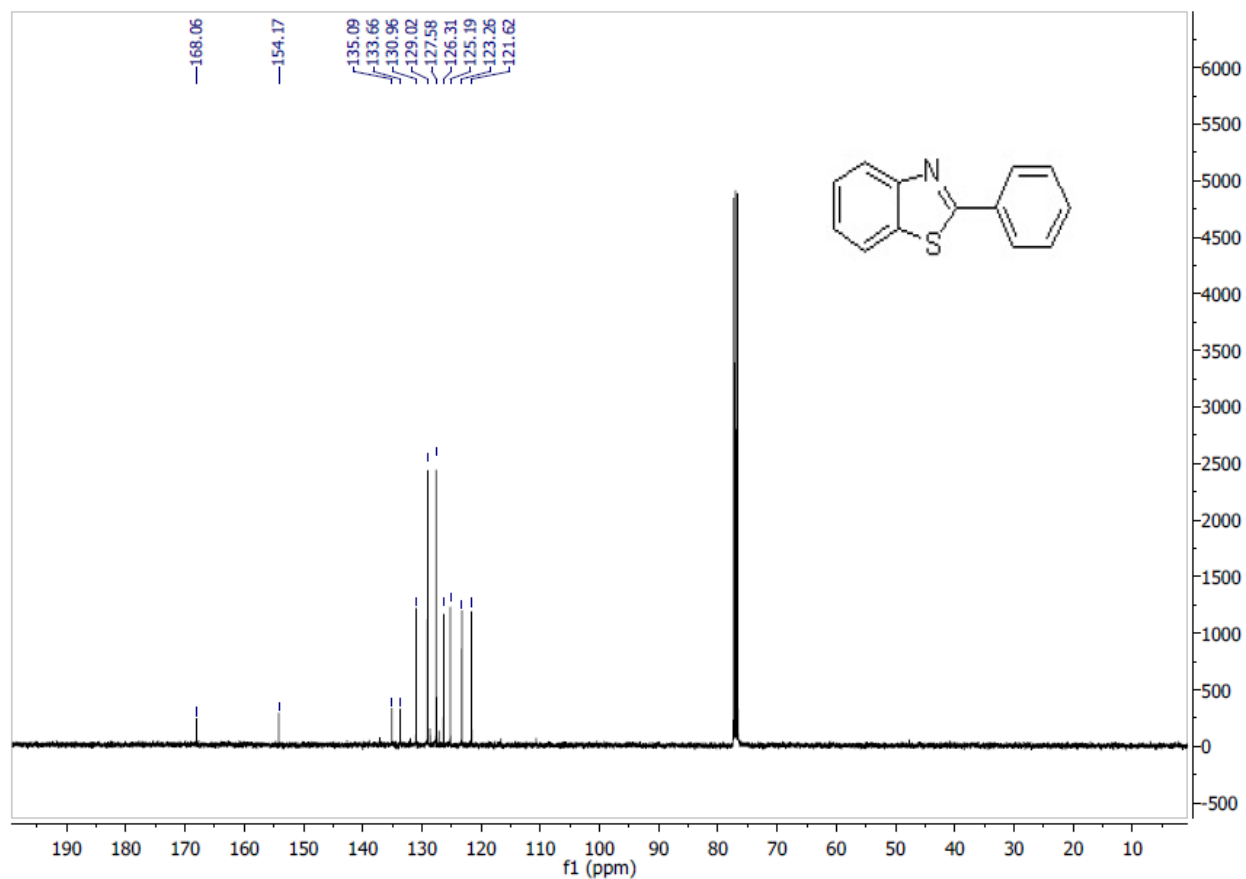
Copies of ^1H , ^{13}C NMR, MS, Elemental analysis spectra of selected products

1a. 2-Phenyl-1,3-benzothiazole



Mol. Wt.: 211.19



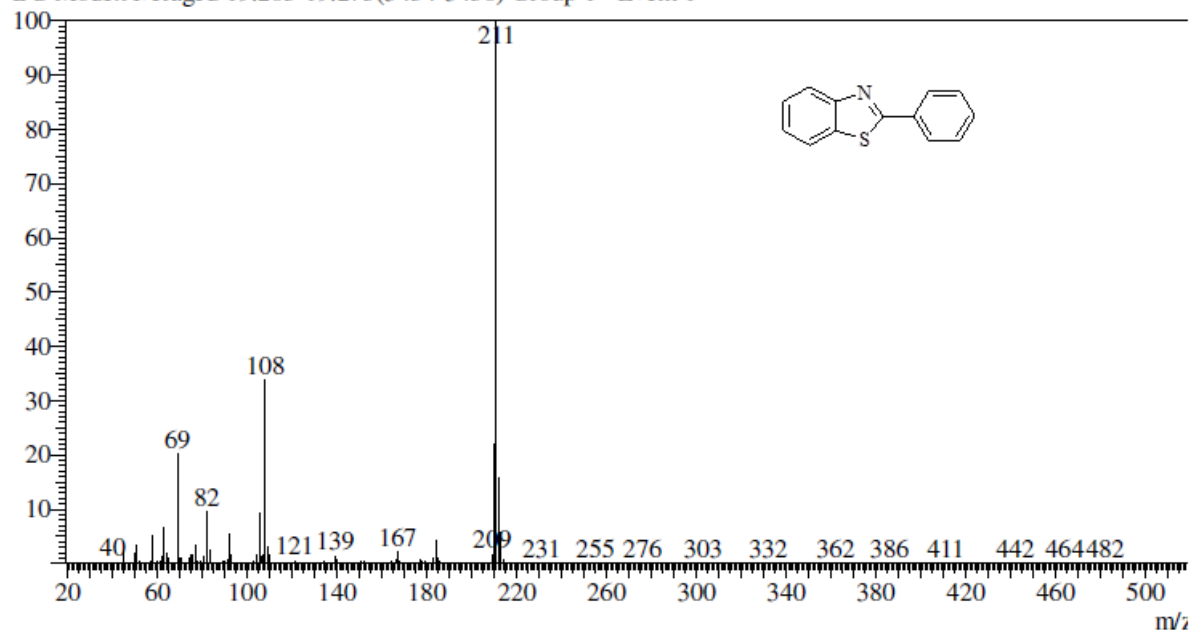


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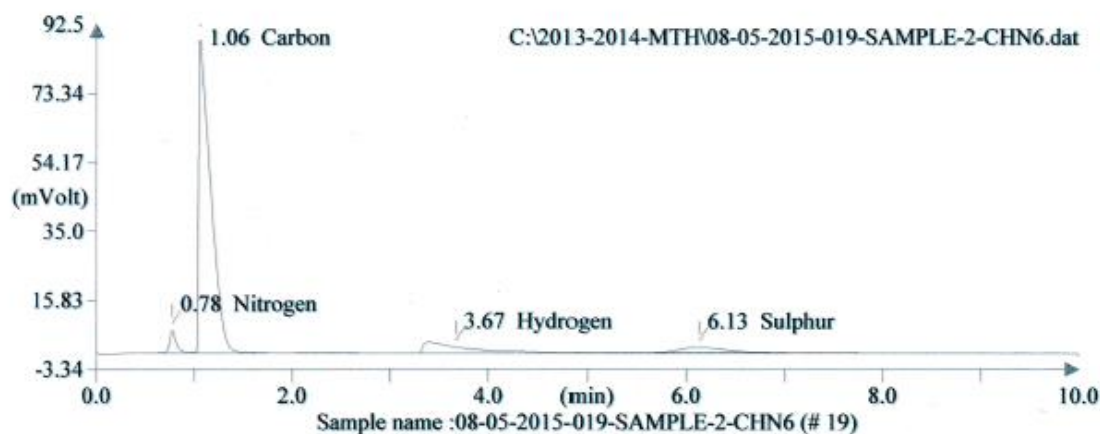
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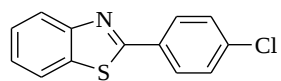
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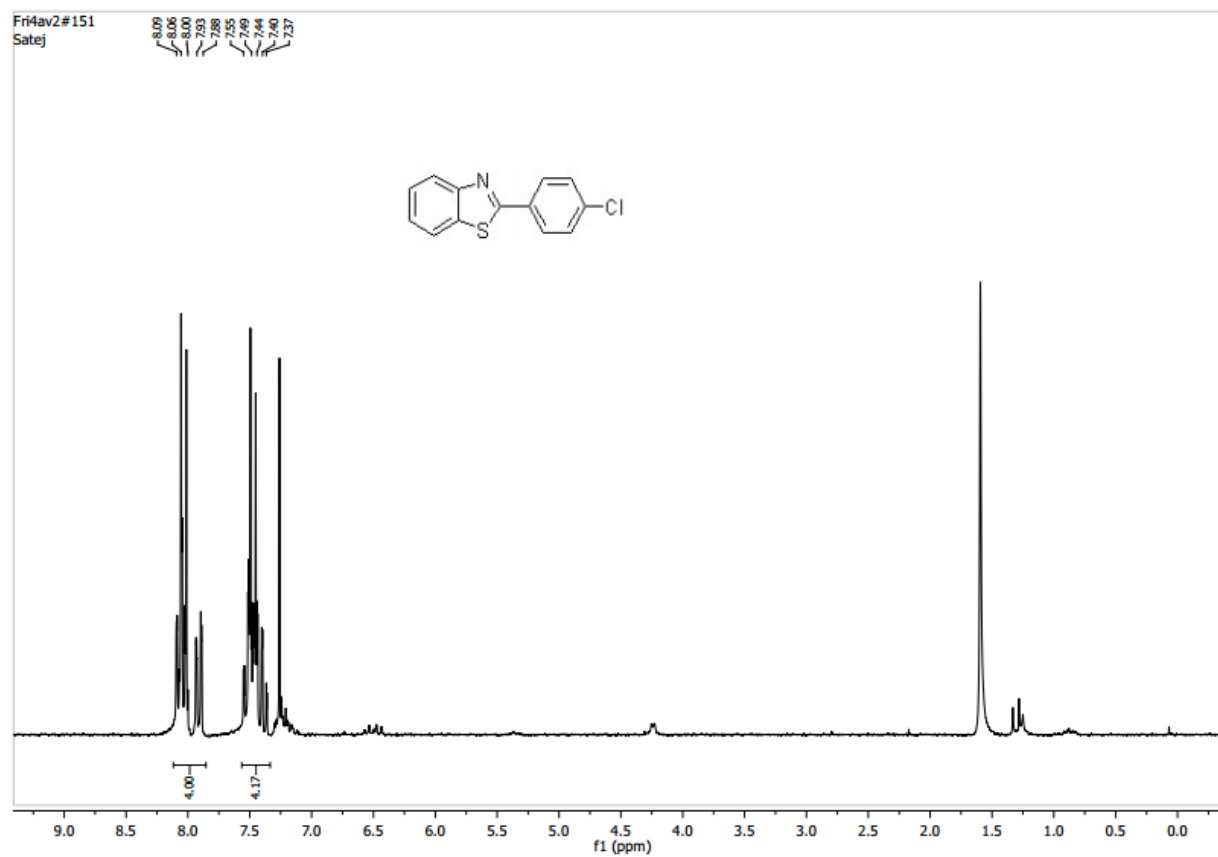


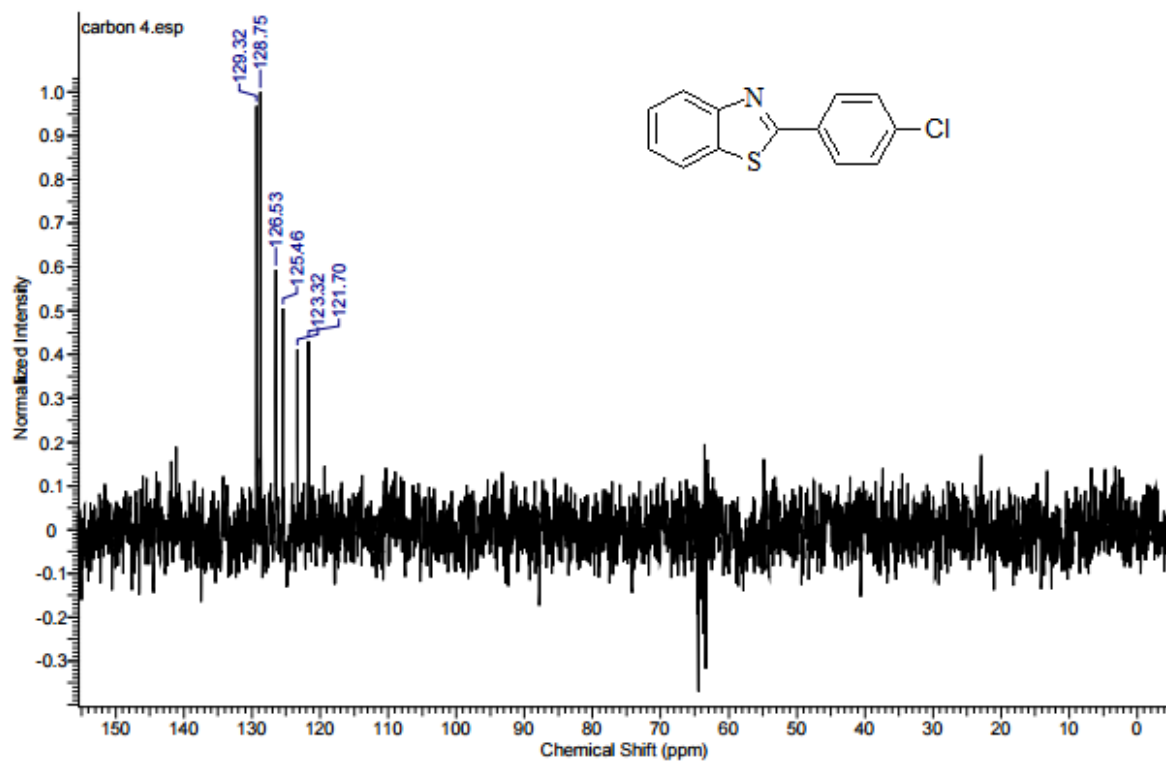
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2a. 2-(4-Chlorophenyl)-1,3-benzothiazole



Mol. Wt.: 245.7



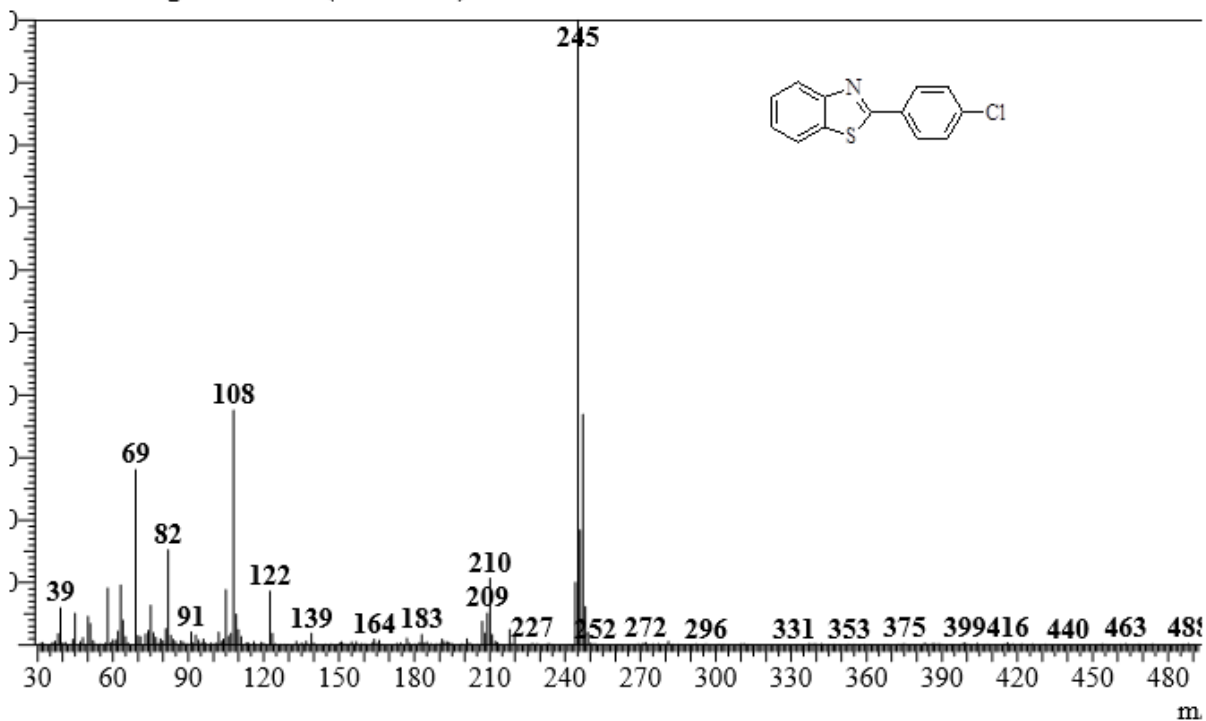


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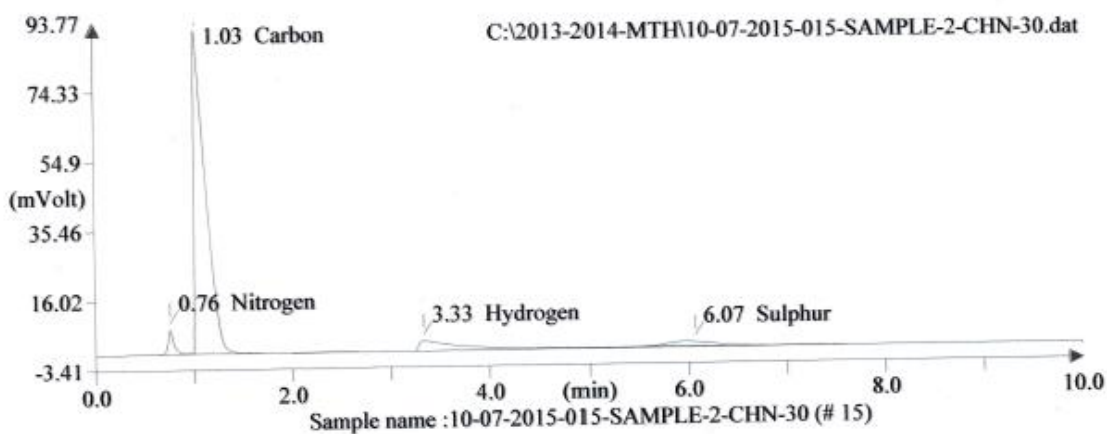
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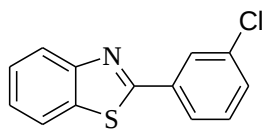
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 Sample weight: 2.743
 Protein factor: 6.25

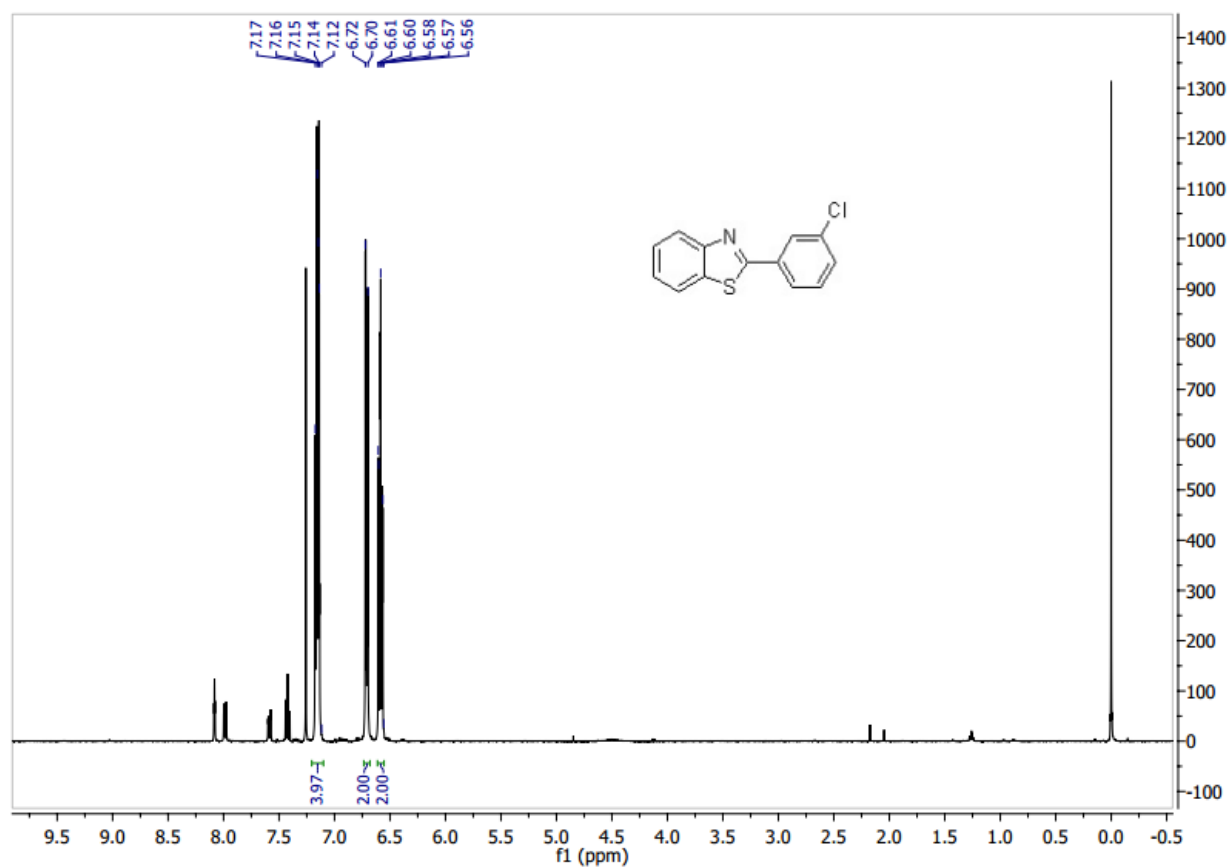


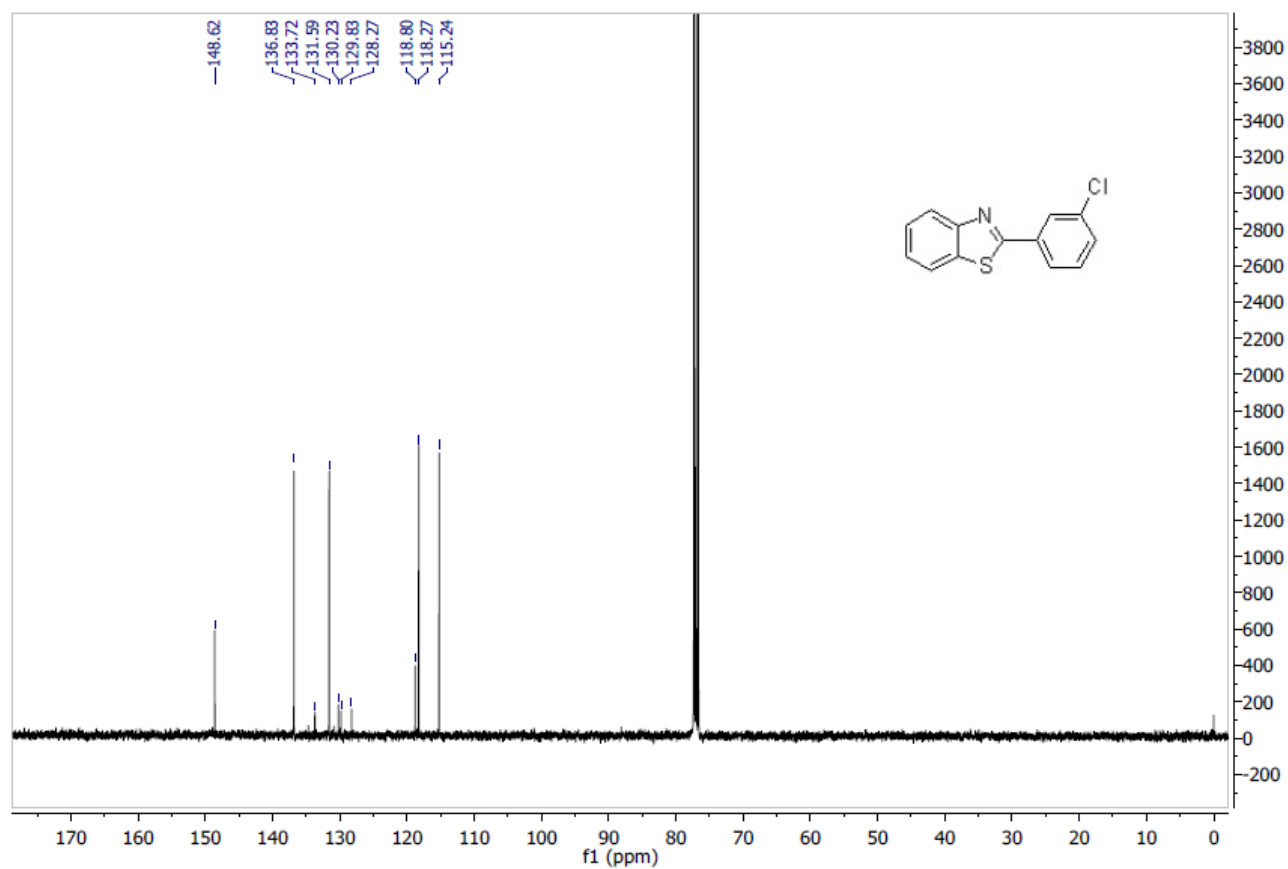
Peak Number (#)	Retention Time (min)	Area (.1* μ V*sec)	Element %	Component
1	0.758	281346	5.586	Nitrogen
2	1.033	7347440	63.783	Carbon
3	3.333	1009268	3.161	Hydrogen
4	6.067	561713	13.010	Sulphur
		9199767	85.540	

3a. 2-(3-Chlorophenyl)-1,3-benzothiazole



Mol. Wt.: 254.7



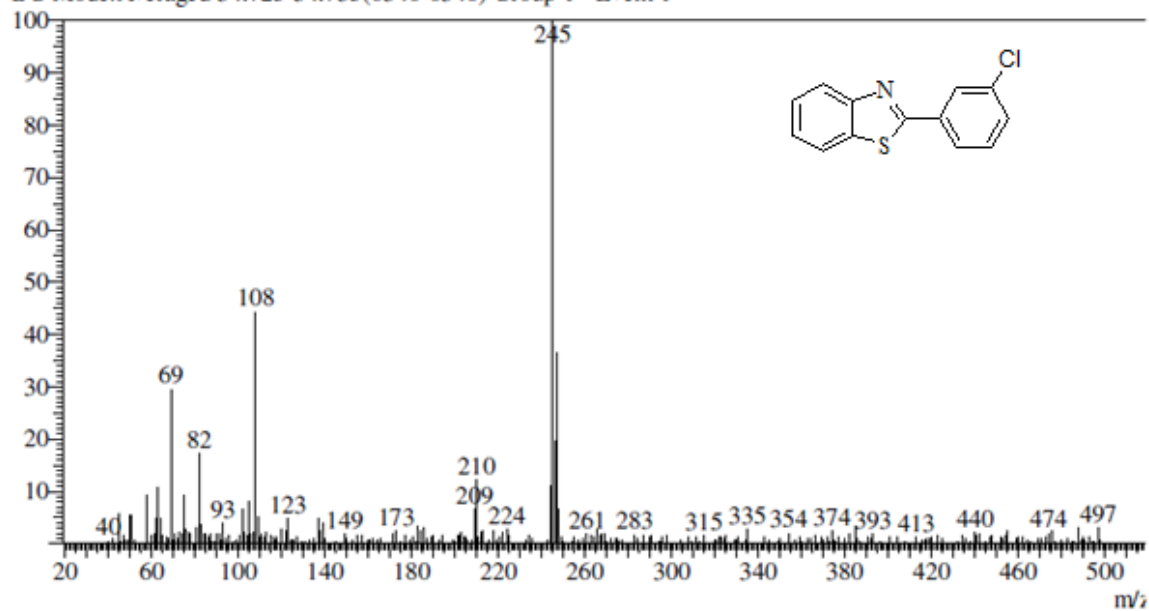


Line#:1 R.Time:34.040(Scan#:6209)

MassPeaks:290

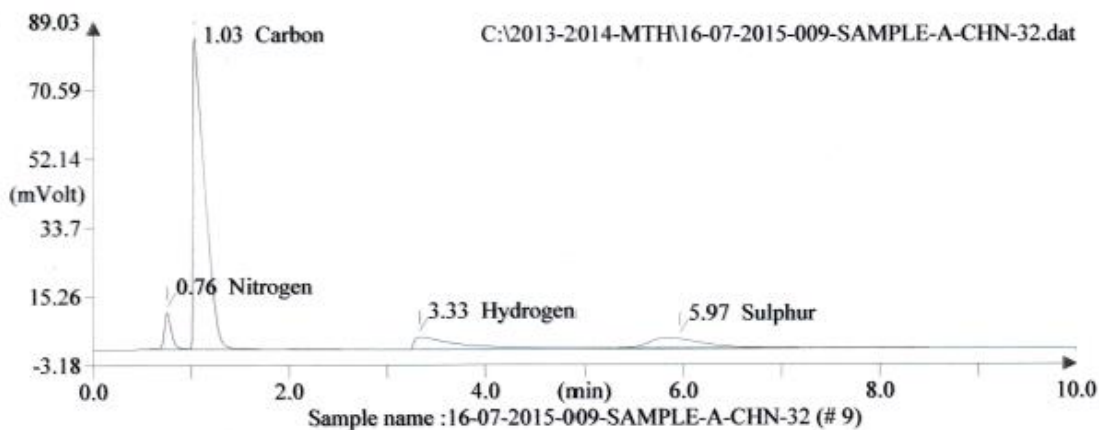
RawMode:Averaged 34.035-34.045(6208-6210) BasePeak:245(1229)

BG Mode:Averaged 34.725-34.735(6346-6348) Group 1 - Event 1



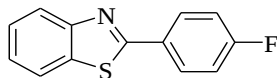
SAIF-IIT BOMBAY

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\16-07-2014-CHNS.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 07/16/2015 17:16
 Printed: 07-17-2015 11:05
 Elemental Analyser method:
 Sampler method:
 Sample ID: 16-07-2015-009-SAMPLE-A-CHN-32 (# 9)
 Analysis type: UnkNown
 Chromatogram filename: 16-07-2015-009-SAMPLE-A-CHN-32.dat
 Calibration method: K Factors
 Sample weight: 2.849
 Protein factor: 6.25

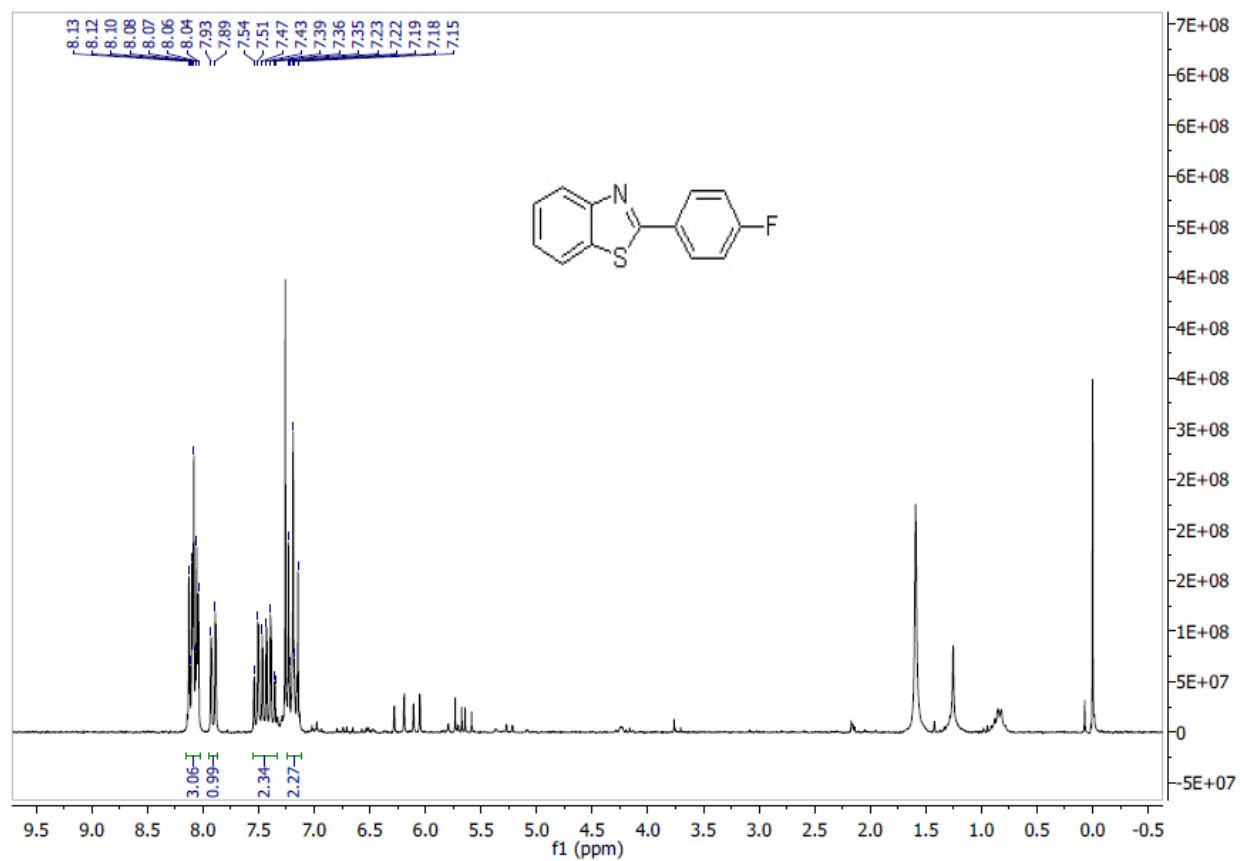


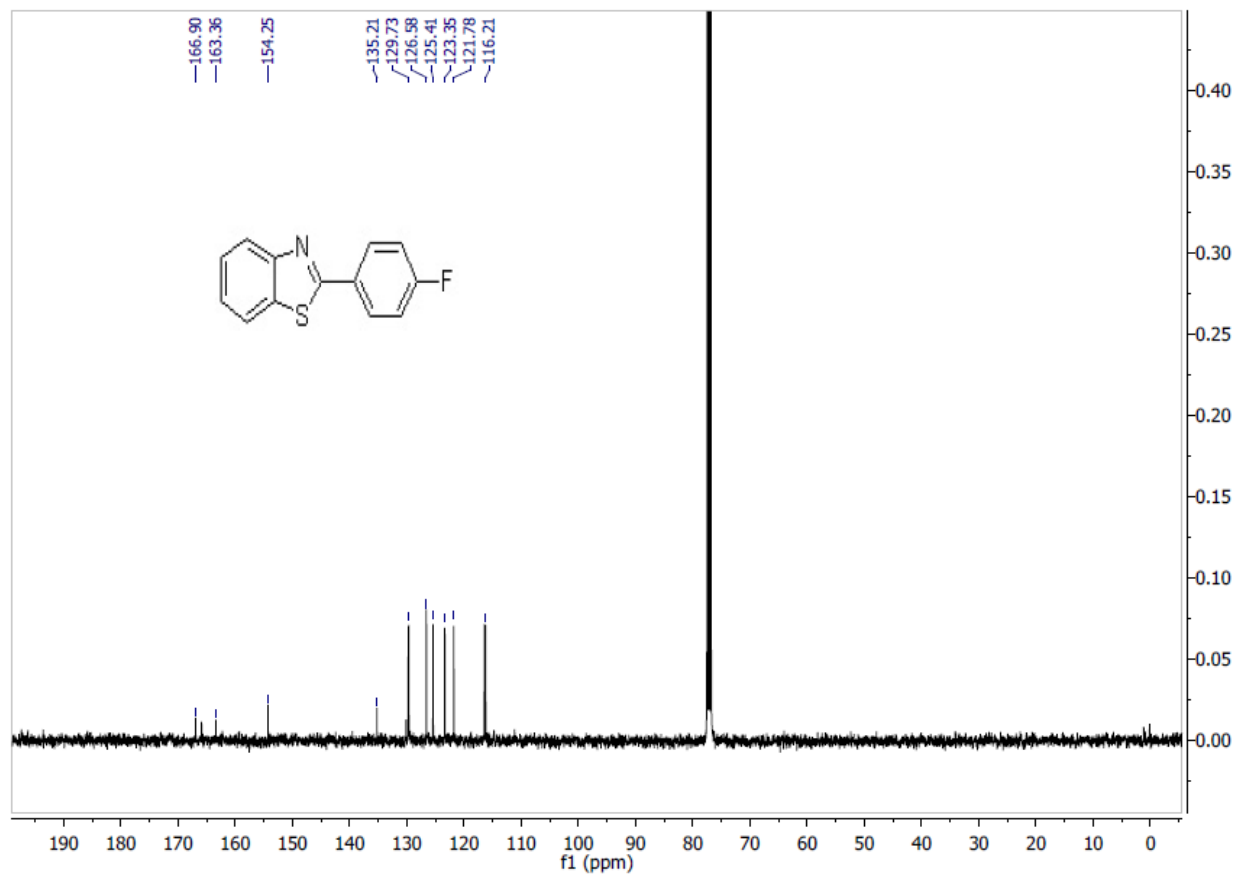
Peak Number (#)	Retention Time (min)	Area (.1 * uV * sec)	Element %	Component
1	0.758	275524	5.601	Nitrogen
2	1.033	7547808	63.511	Carbon
3	3.333	961843	3.193	Hydrogen
4	5.967	593353	13.016	Sulphur
		9378528	85.321	

4a. 2-(4-Fluorophenyl)-1H-benzothiazole

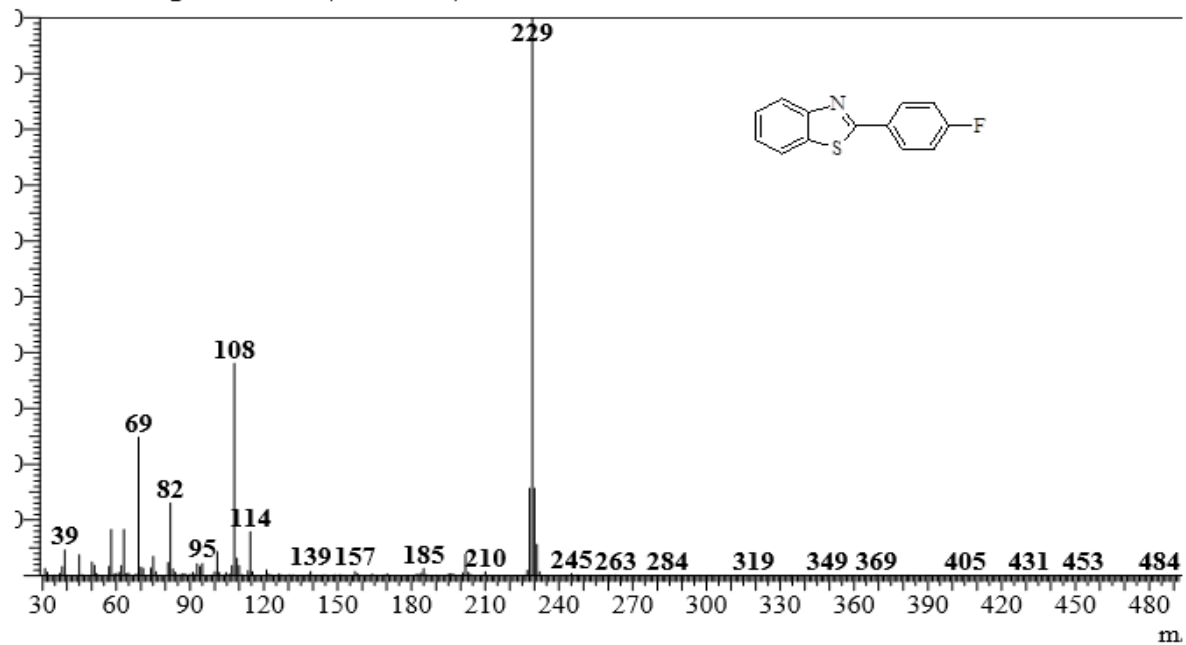


Mol. Wt.: 229.2



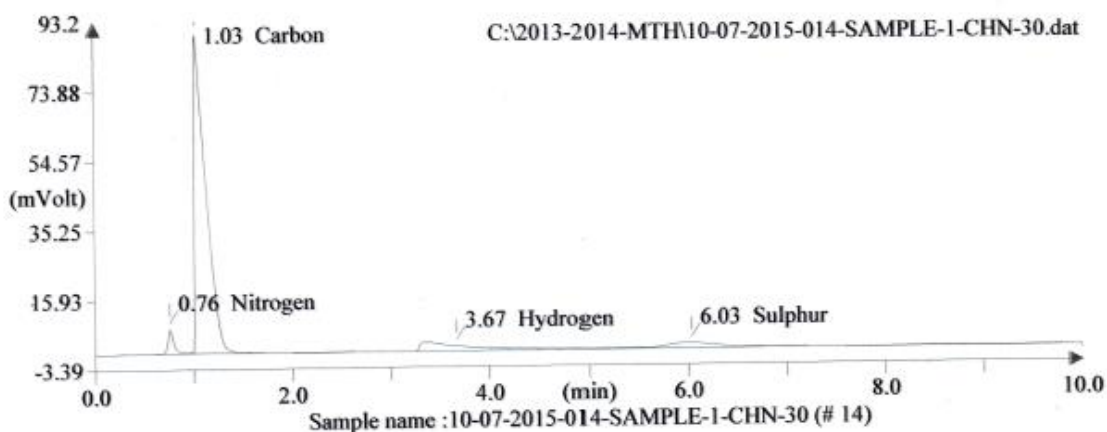


ie#:3 R.Time:12.2(Scan#:1099)
 issPeaks:288
 wMode:Averaged 12.1-12.2(1098-1100) BasePeak:229(44132)
 i Mode:Averaged 15.7-15.7(1524-1526)



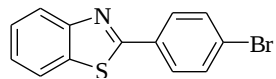
SAIF-IIT BOMBAY

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\10-07-2015-CHNS.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 07/10/2015 16:56
 Printed: 07-13-2015 13:40
 Elemental Analyser method:
 Sampler method:
 Sample ID: 10-07-2015-014-SAMPLE-1-CHN-30 (# 14)
 Analysis type: UnkNown
 Chromatogram filename: 10-07-2015-014-SAMPLE-1-CHN-30.dat
 Calibration method: K Factors
 Sample weight: 2.514
 Protein factor: 6.25

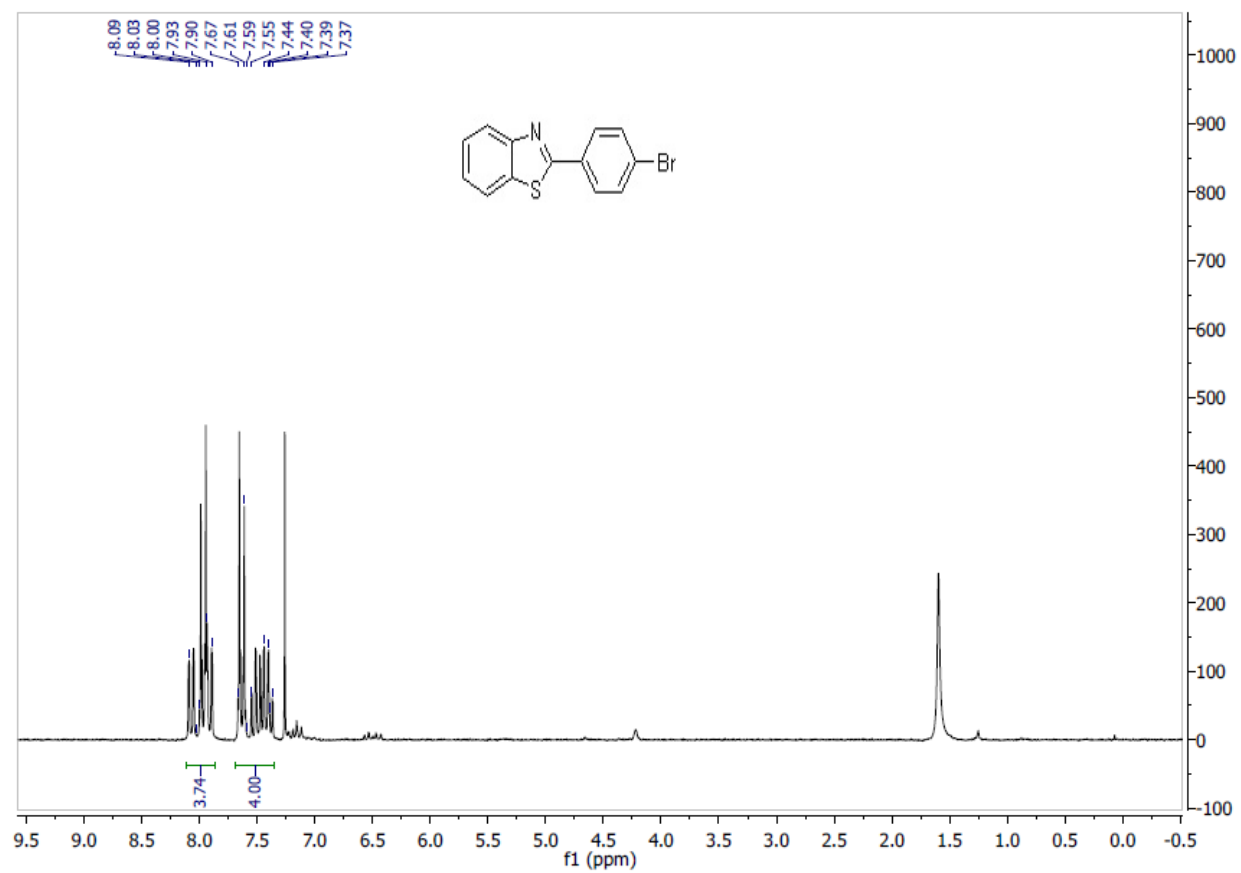


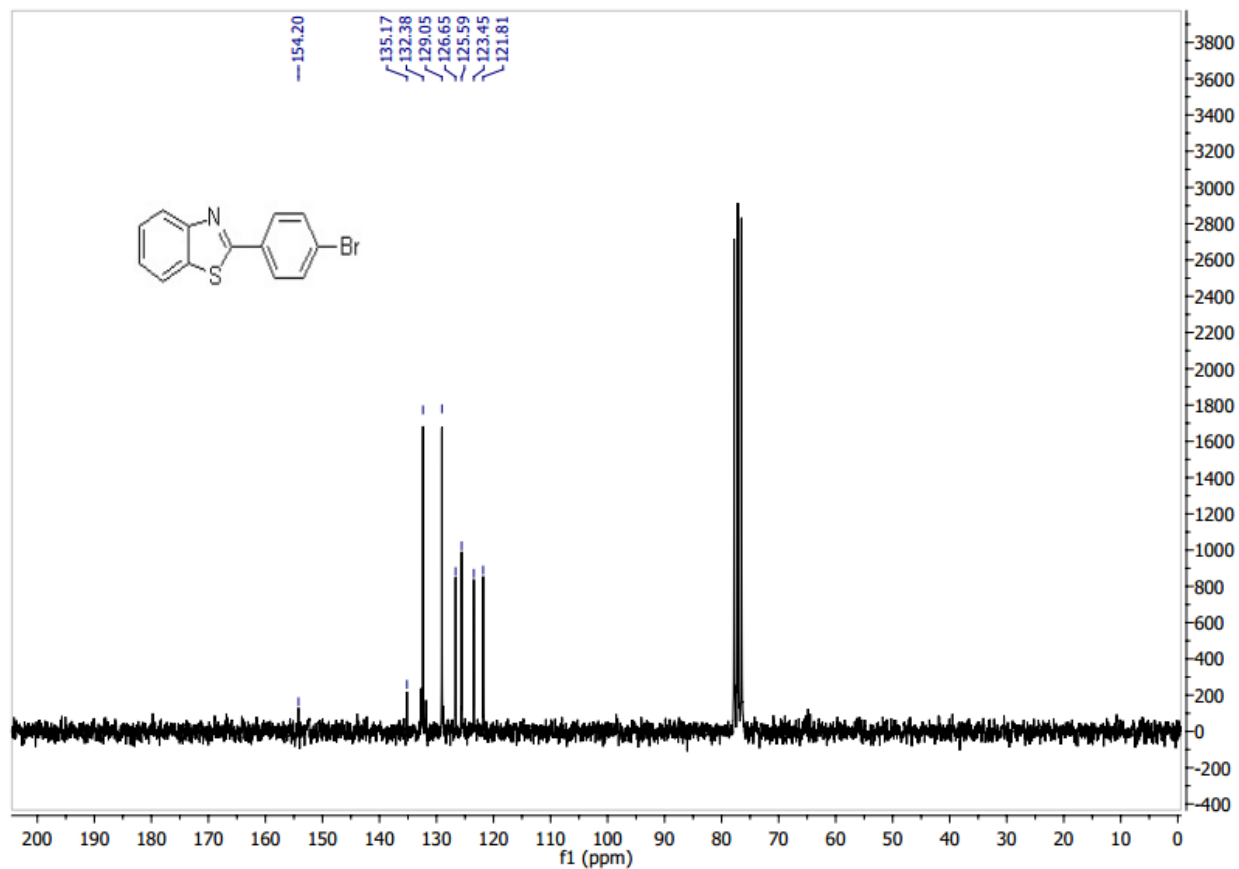
Peak Number (#)	Retention Time (min)	Area (.1*uV*sec)	Element %	Component
1	0.758	282472	6.123	Nitrogen
2	1.033	7199857	68.188	Carbon
3	3.667	999047	3.414	Hydrogen
4	6.033	549280	13.881	Sulphur
		9030656	91.606	

5a. 2-(4-Bromophenyl)-1,3-benzothiazole

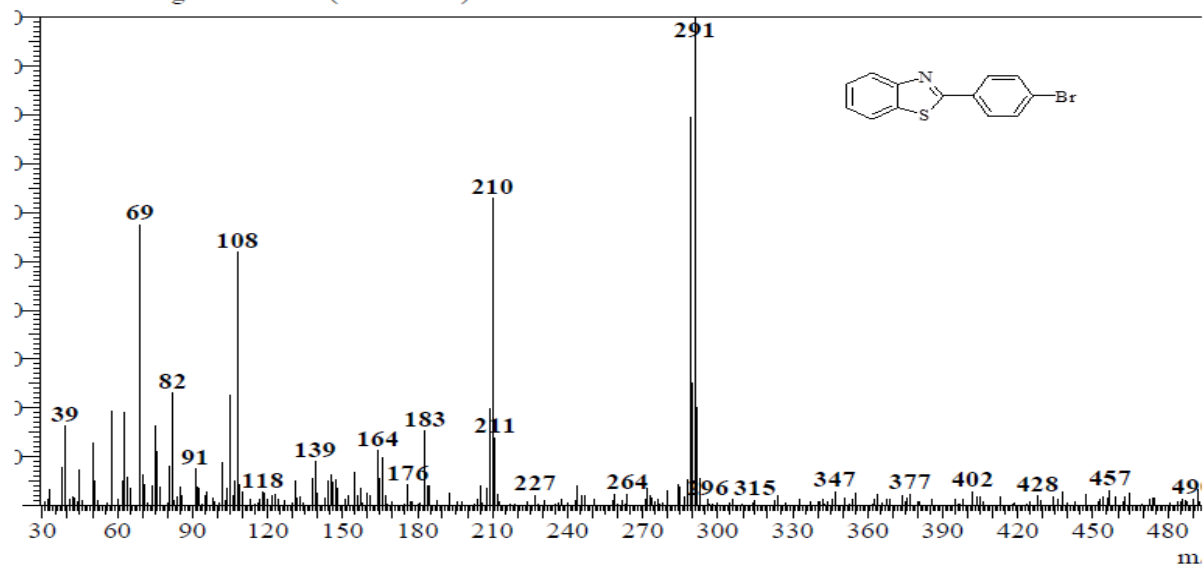


Mol. Wt.: 290.1



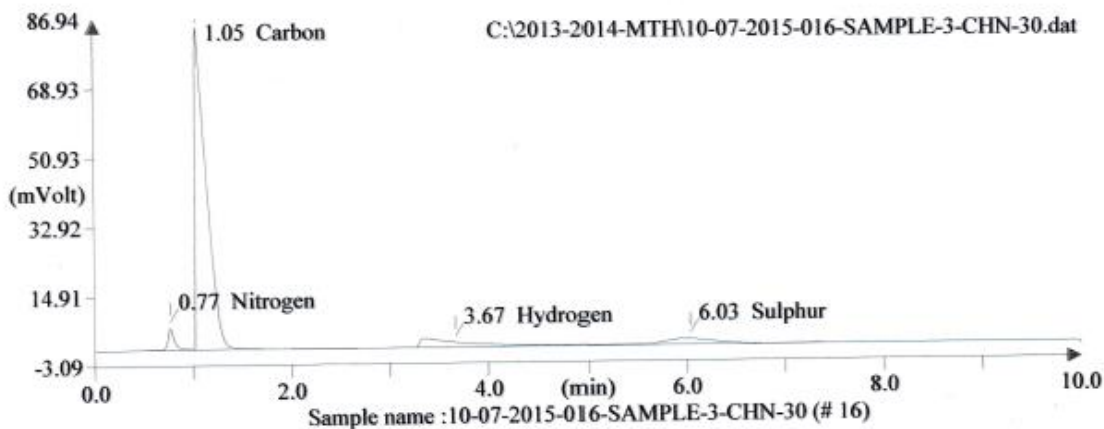


ie#:1 R.Time:16.2(Scan#:1590)
 issPeaks:254
 wMode:Averaged 16.2-16.3(1589-1591) BasePeak:291(2215)
 i Mode:Averaged 16.4-16.4(1604-1606)



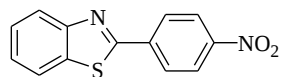
SAIF-IIT BOMBAY

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\10-07-2015-CHNS.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 07/10/2015 17:17
 Printed: 07-13-2015 13:42
 Elemental Analyser method:
 Sampler method:
 Sample ID: 10-07-2015-016-SAMPLE-3-CHN-30 (# 16)
 Analysis type: UnkNown
 Chromatogram filename: 10-07-2015-016-SAMPLE-3-CHN-30.dat
 Calibration method: K Factors
 Sample weight: 2.955
 Protein factor: 6.25

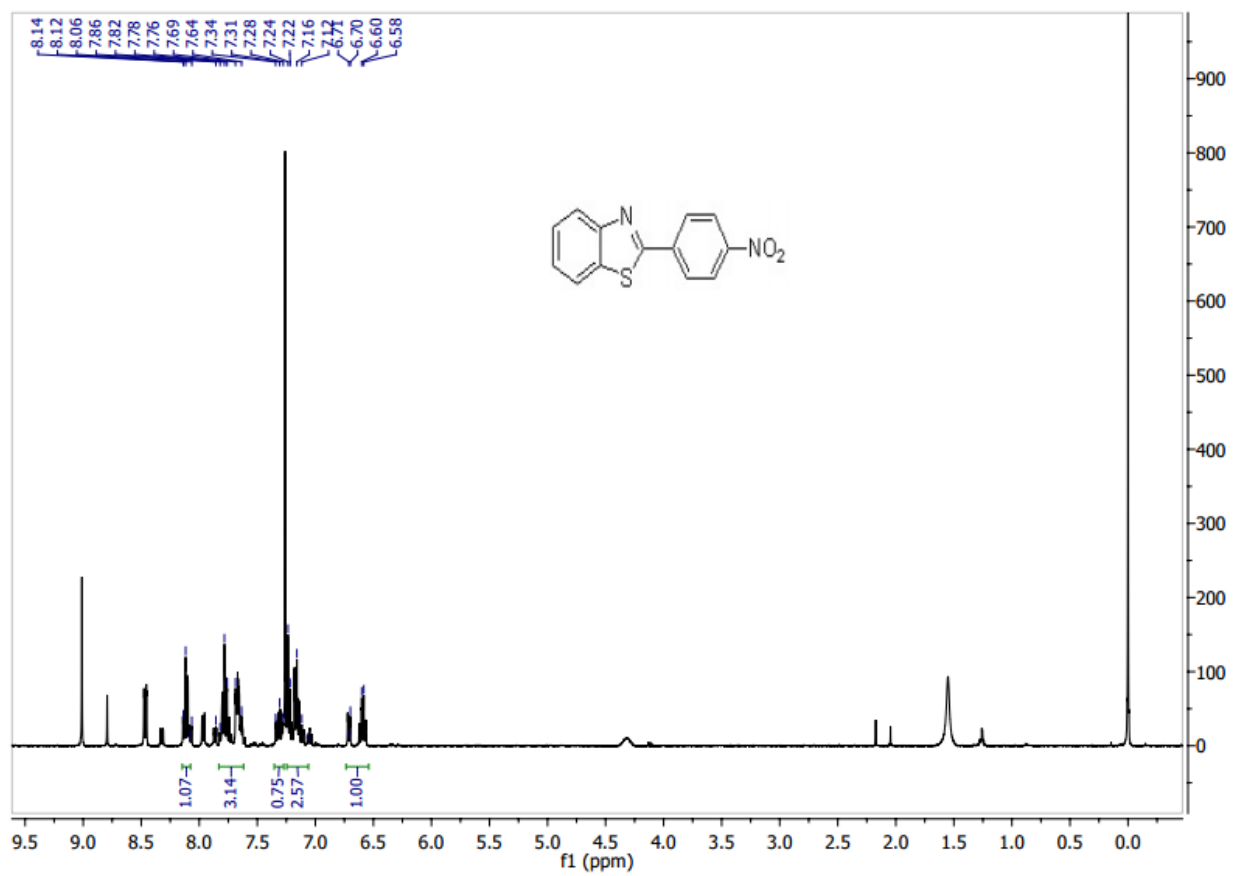


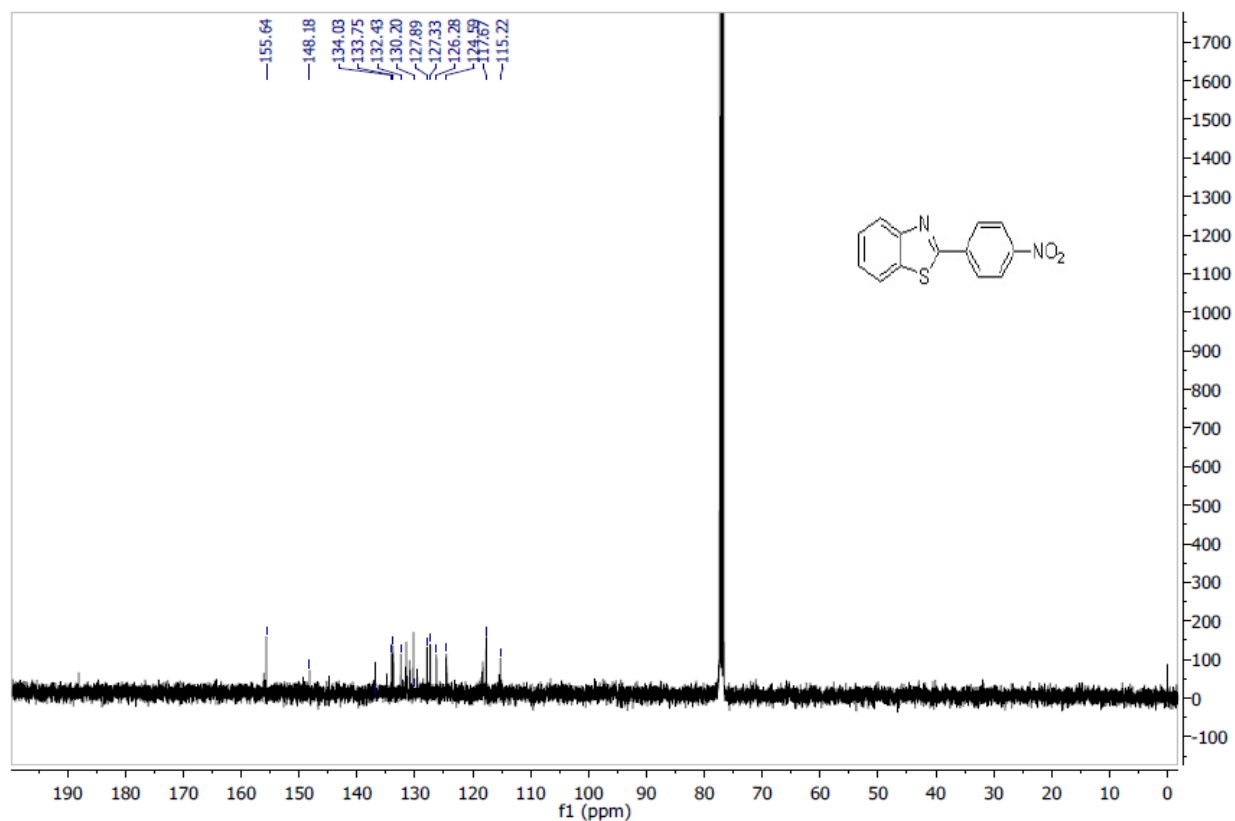
Peak Number (#)	Retention Time (min)	Area (.1*uV*sec)	Element %	Component
1	0.767	262230	4.786	Nitrogen
2	1.050	6644377	53.512	Carbon
3	3.667	823126	2.393	Hydrogen
4	6.033	509880	10.963	Sulphur
		8239613	71.654	

6a. 2-(4-Nitrophenyl)benzothiazole

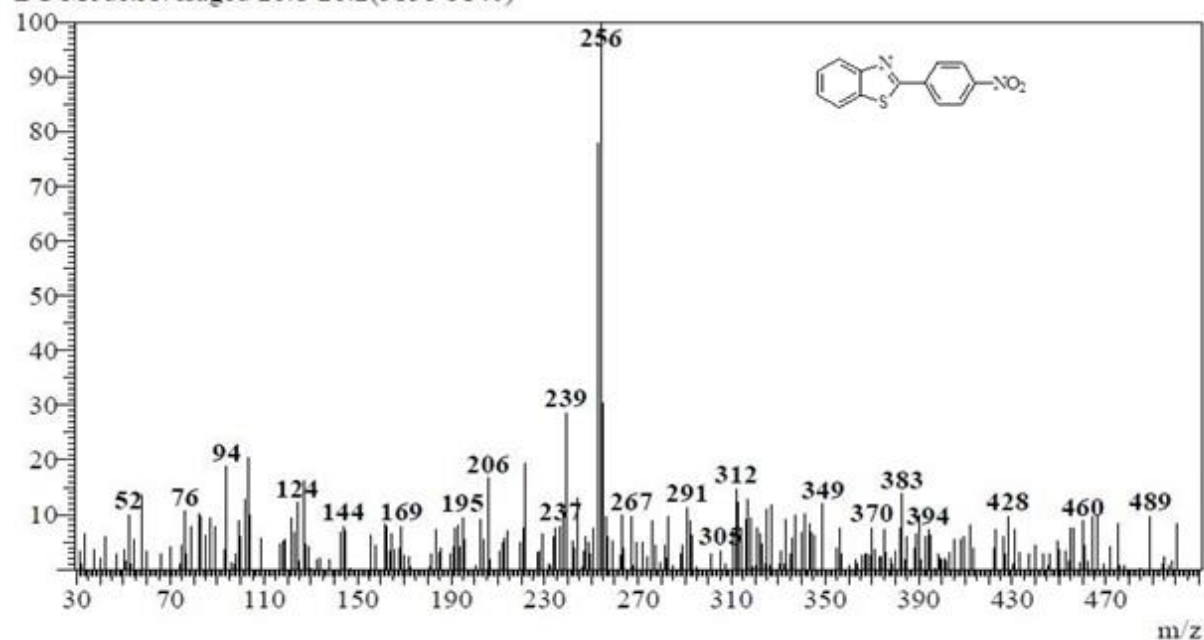


Mol. Wt.: 256



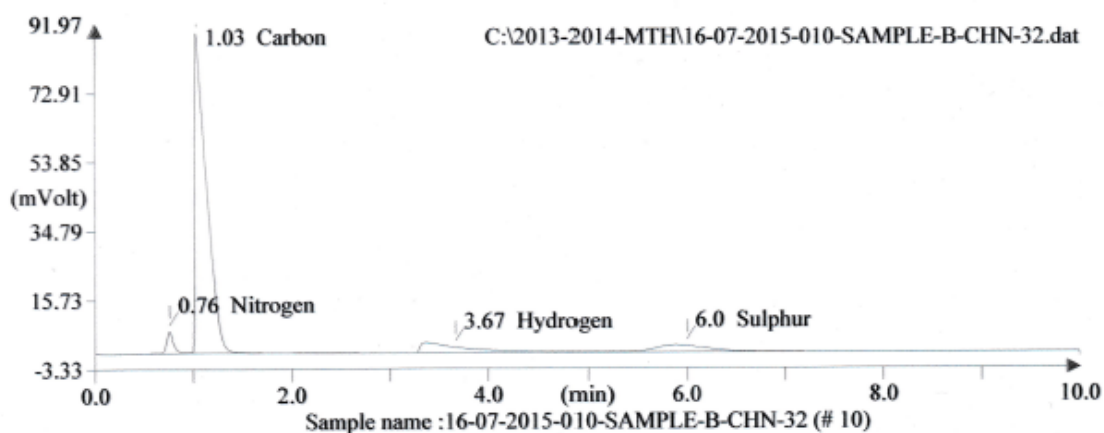


Line#:3 R.Time:27.0(Scan#:2999)
 MassPeaks:248
 RawMode:Averaged 27.0-27.0(2998-3000) BasePeak:254(444)
 BG Mode:Averaged 28.1-28.2(3138-3140)



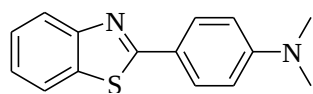
SAIF-IIT BOMBAY

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\16-07-2014-CHNS.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 07/16/2015 17:26
 Printed: 07-17-2015 11:06
 Elemental Analyser method:
 Sampler method:
 Sample ID: 16-07-2015-010-SAMPLE-B-CHN-32 (# 10)
 Analysis type: UnkNown
 Chromatogram filename: 16-07-2015-010-SAMPLE-B-CHN-32.dat
 Calibration method: K Factors
 Sample weight: 2.591
 Protein factor: 6.25

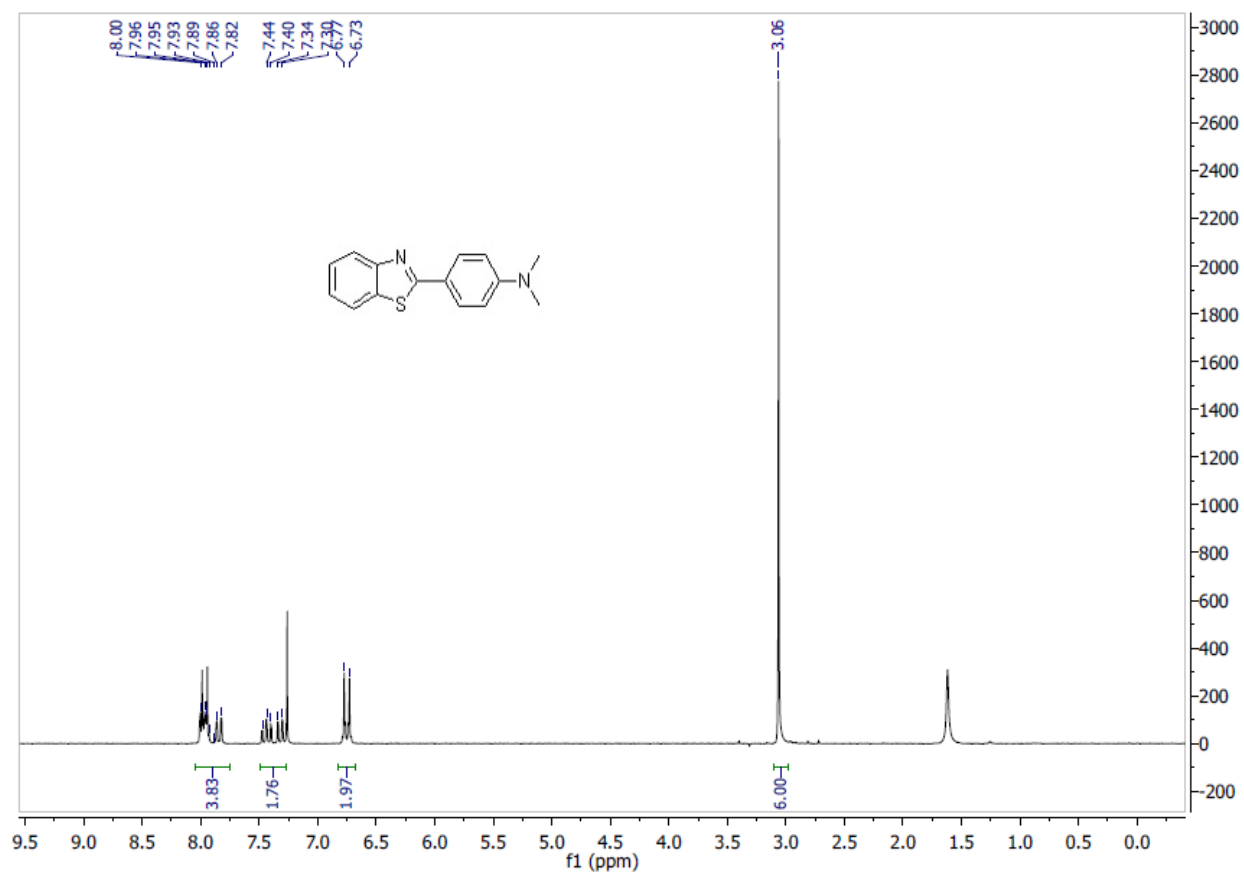


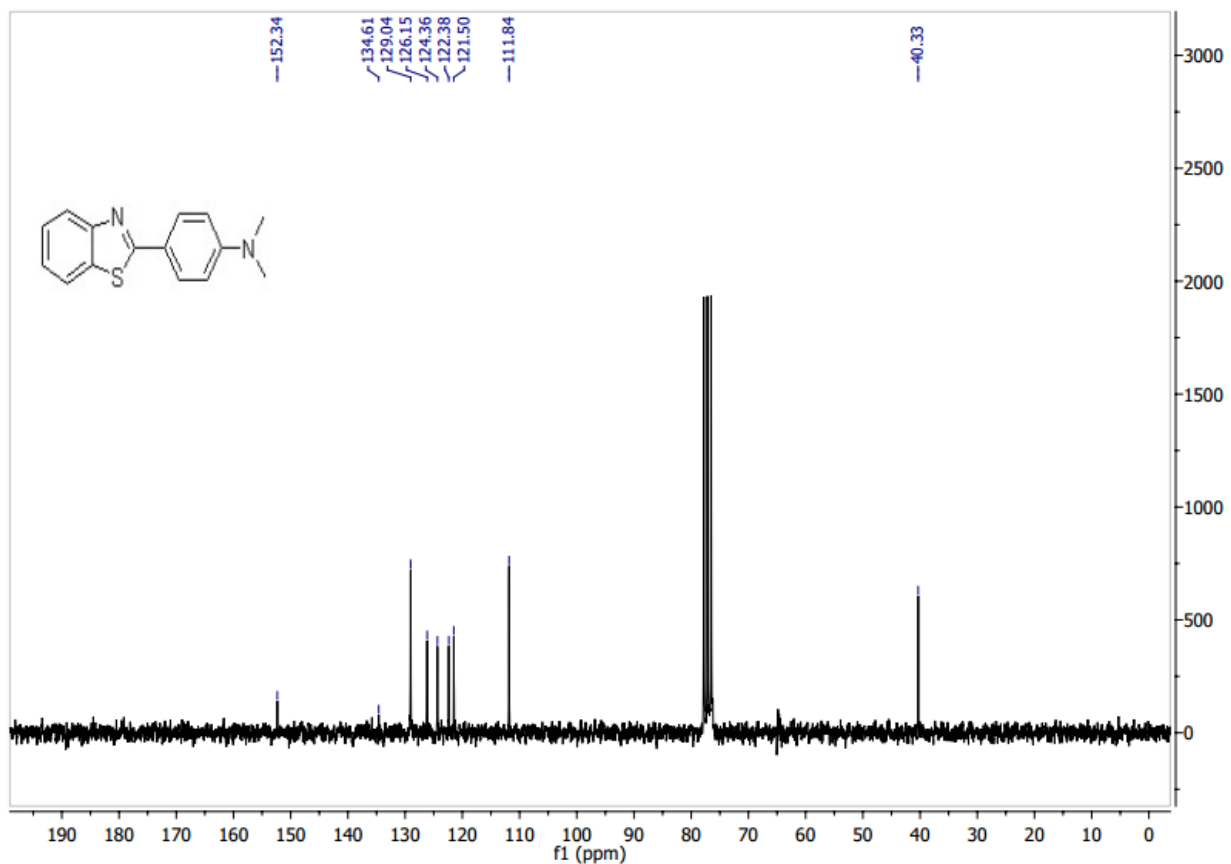
Peak Number (#)	Retention Time (min)	Area (.1* μ V*sec)	Element %	Component
1	0.758	514778	10.922	Nitrogen
2	1.033	6577839	60.861	Carbon
3	3.667	841260	3.073	Hydrogen
4	6.000	525235	12.642	Sulphur
		8459112	87.498	

7a. 4-Benzothiazole-2-phenyl-dimethylamine



Mol. Wt.: 254.3



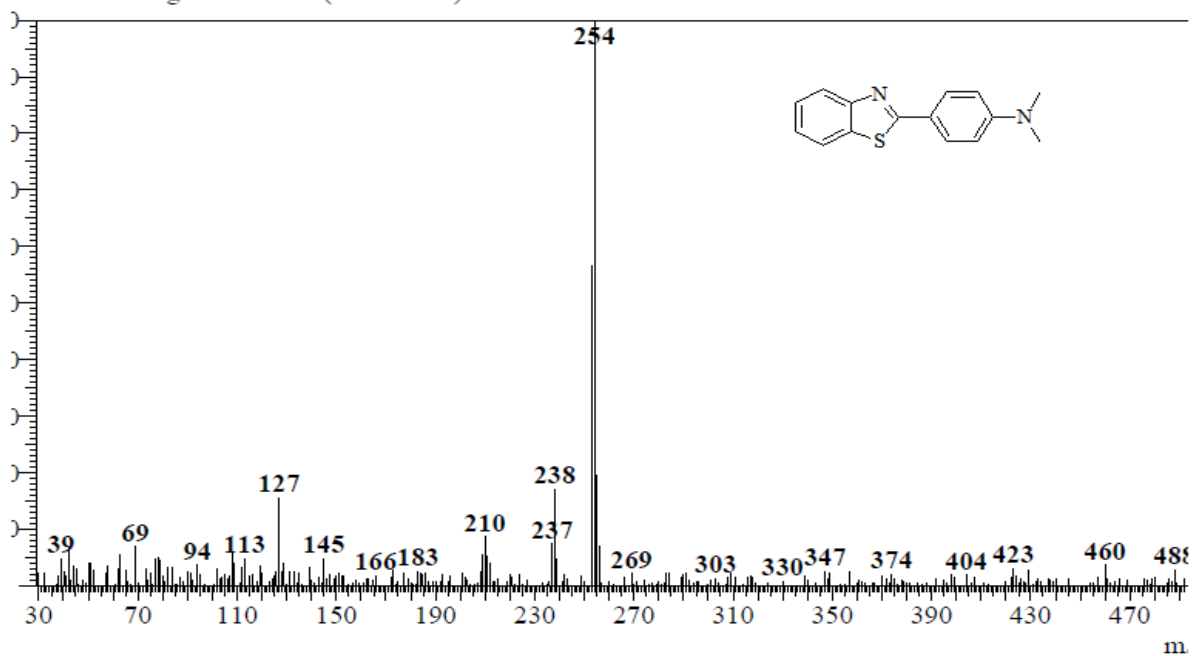


ie#:3 R.Time:21.6(Scan#:2231)

issPeaks:285

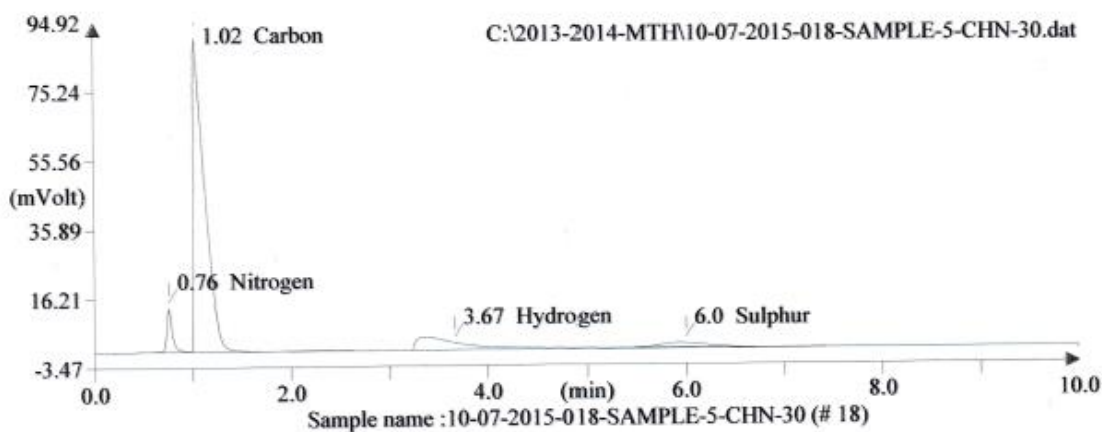
wMode:Averaged 21.6-21.6(2230-2232) BasePeak:254(3396)

i Mode:Averaged 22.8-22.8(2380-2382)



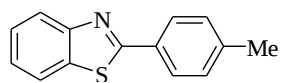
SAIF-IIT BOMBAY

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\10-07-2015-CHNS.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 07/10/2015 17:38
 Printed: 07-13-2015 13:43
 Elemental Analyser method:
 Sampler method:
 Sample ID: 10-07-2015-018-SAMPLE-5-CHN-30 (# 18)
 Analysis type: UnkNown
 Chromatogram filename: 10-07-2015-018-SAMPLE-5-CHN-30.dat
 Calibration method: K Factors
 Sample weight: 2.448
 Protein factor: 6.25

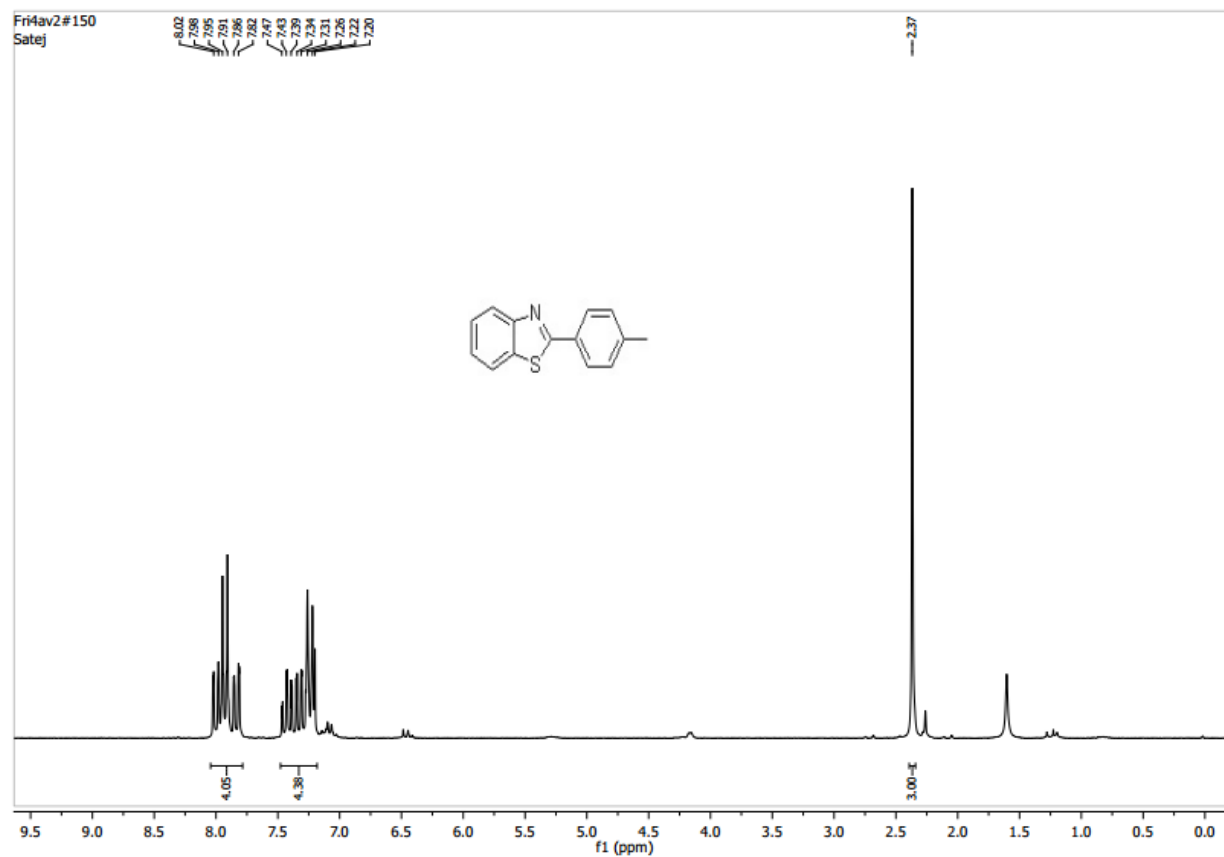


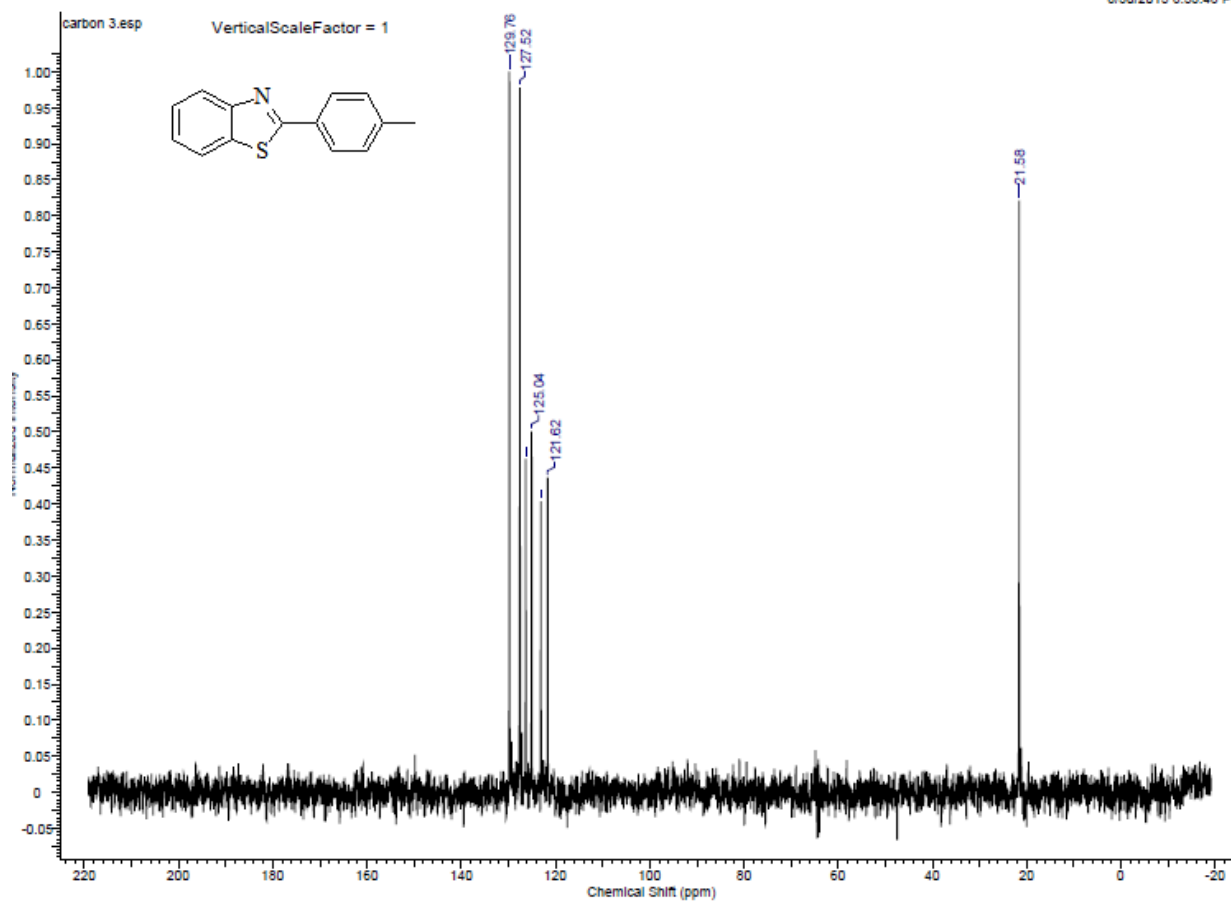
Peak Number (#)	Retention Time (min)	Area (.1*uV*sec)	Element %	Component
1	0.758	469765	11.019	Nitrogen
2	1.025	7223541	70.258	Carbon
3	3.667	1473334	5.171	Hydrogen
4	6.000	469128	12.175	Sulphur
		9635768	98.623	

8a. 2-(4-Methylphenyl)benzothiazole



Mol. Wt.: 225.2



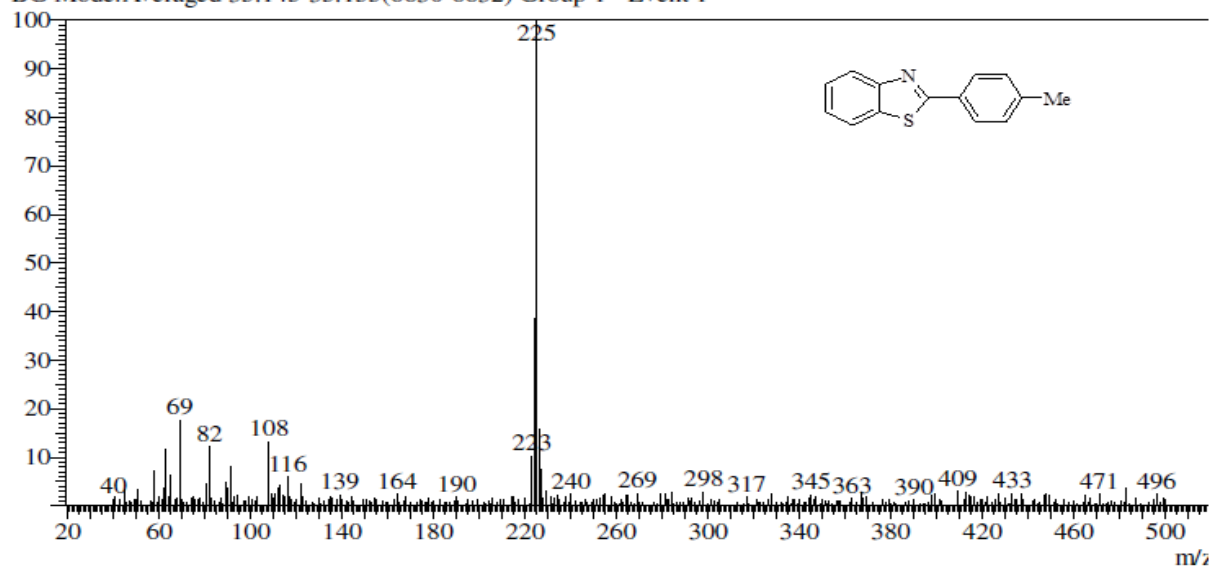


Line#:1 R.Time:33.590(Scan#:6319)

MassPeaks:348

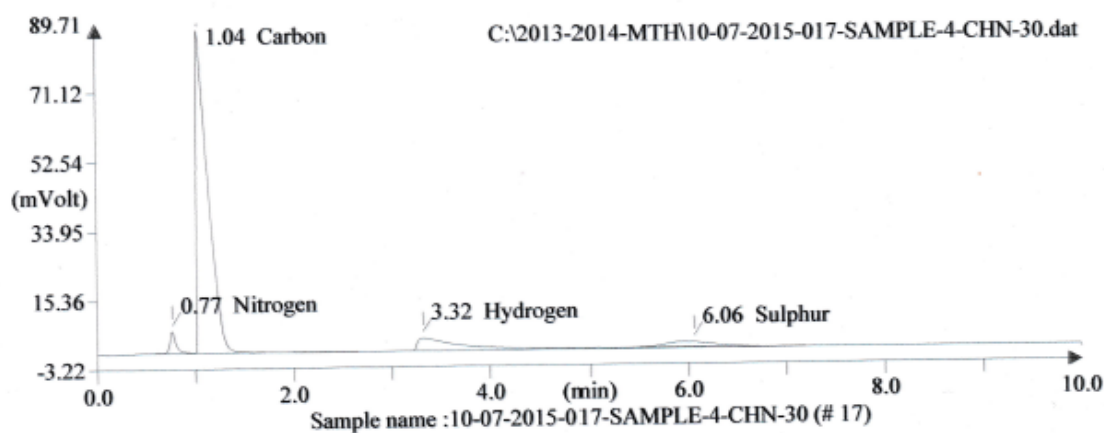
Raw Mode:Averaged 33.585-33.595(6318-6320) BasePeak:225(1333)

BG Mode:Averaged 35.145-35.155(6630-6632) Group 1 - Event 1



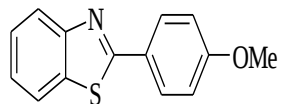
SAIF-IIT BOMBAY

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\10-07-2015-CHNS.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 07/10/2015 17:28
 Printed: 07-13-2015 13:43
 Elemental Analyser method:
 Sampler method:
 Sample ID: 10-07-2015-017-SAMPLE-4-CHN-30 (# 17)
 Analysis type: UnkNown
 Chromatogram filename: 10-07-2015-017-SAMPLE-4-CHN-30.dat
 Calibration method: K Factors
 Sample weight: 2.26
 Protein factor: 6.25

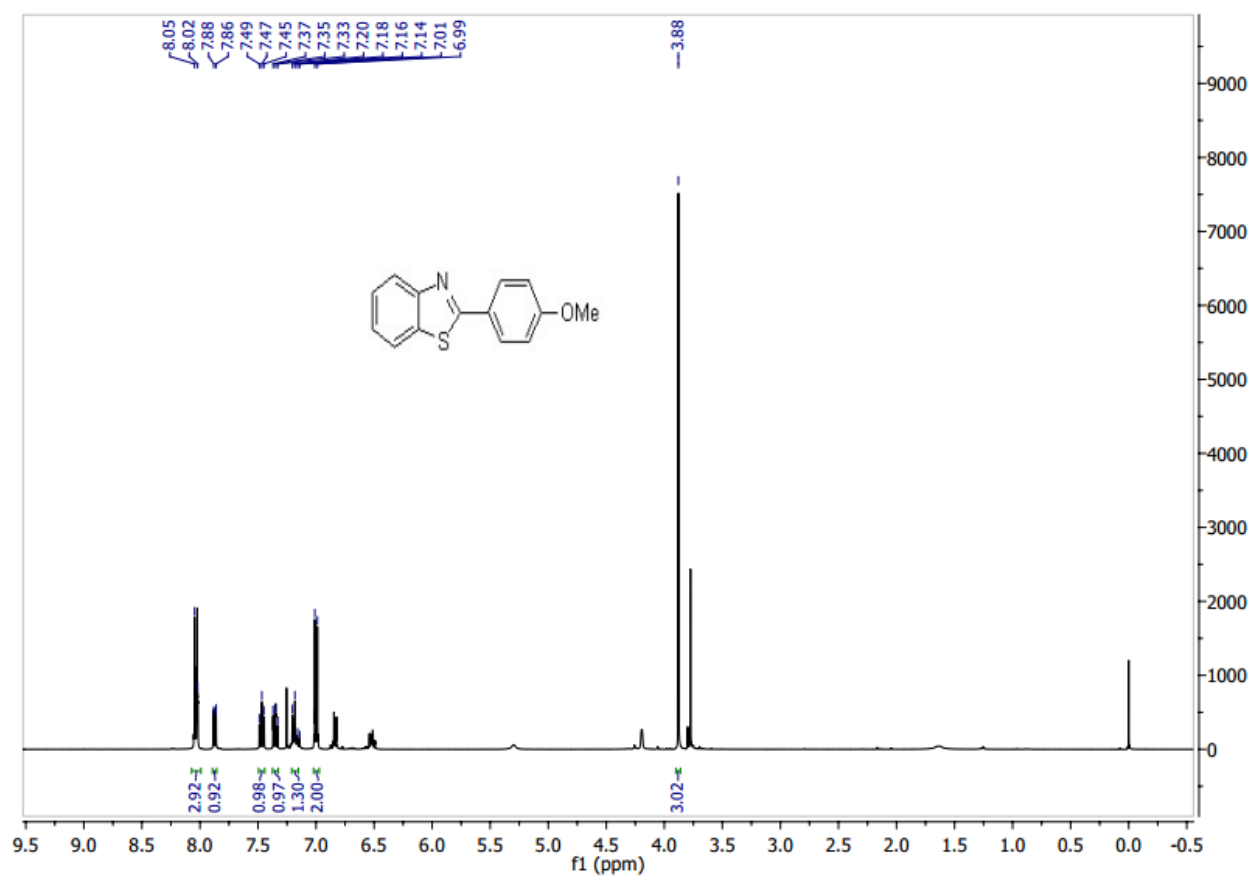


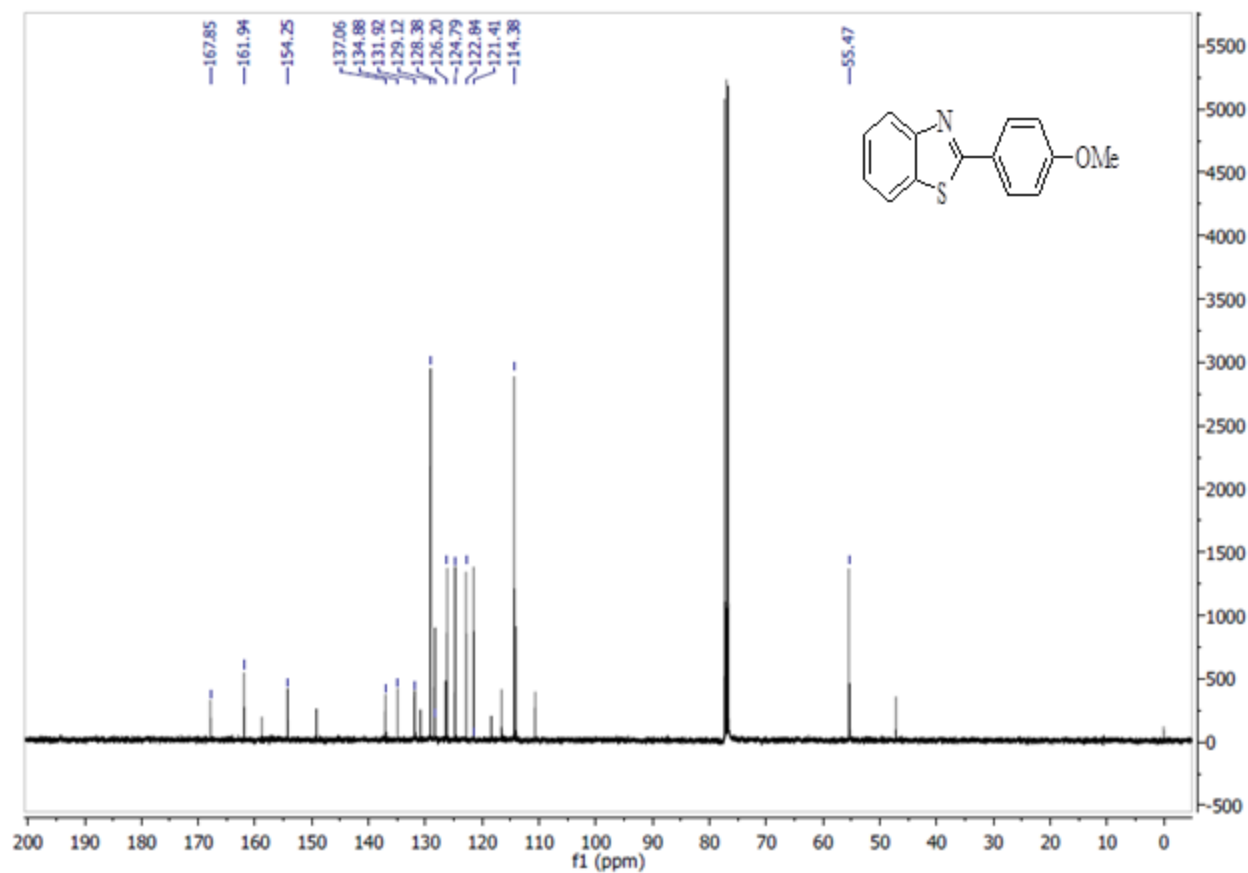
Peak Number (#)	Retention Time (min)	Area (.1*uV*sec)	Element %	Component
1	0.767	258004	6.143	Nitrogen
2	1.042	7054683	74.314	Carbon
3	3.317	1283007	4.878	Hydrogen
4	6.058	509631	14.327	Sulphur
		9105324	99.662	

9a. 2-(4-Methoxyphenyl)benzothiazole



Mol. Wt.: 241.28



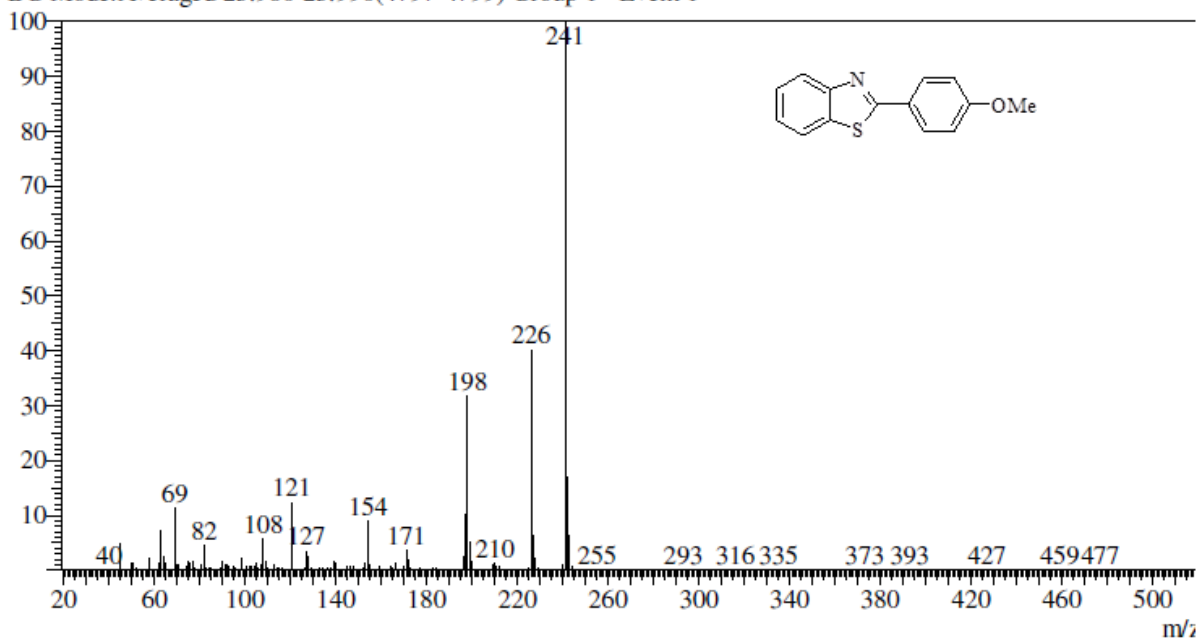


Line#:1 R.Time:25.245(Scan#:4650)

MassPeaks:343

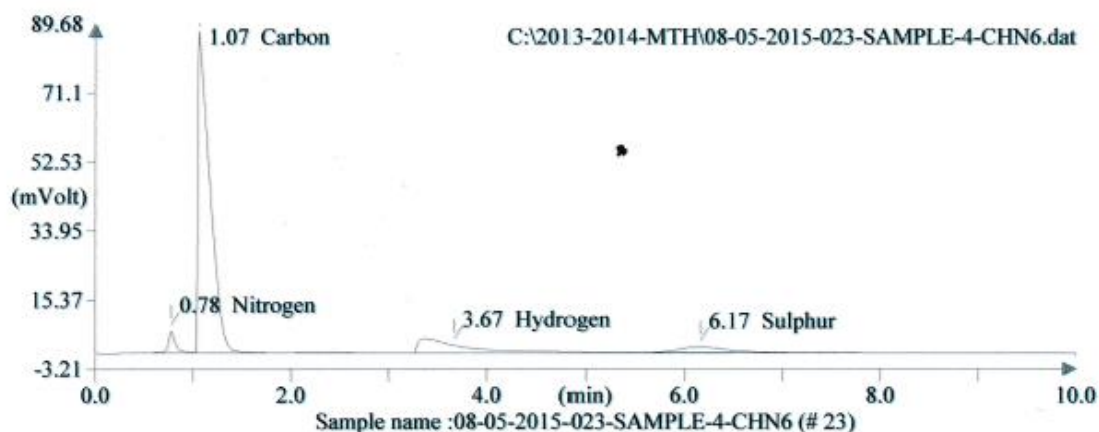
RawMode:Averaged 25.240-25.250(4649-4651) BasePeak:241(142547)

BG Mode:Averaged 25.980-25.990(4797-4799) Group 1 - Event 1



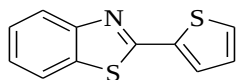
SAIF-IIT BOMBAY

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\08-05-2015-CHNS.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 05/08/2015 18:21
 Printed: 05-08-2015 18:31
 Elemental Analyser method:
 Sampler method:
 Sample ID: 08-05-2015-023-SAMPLE-4-CHN6 (# 23)
 Analysis type: UnkNown
 Chromatogram filename: 08-05-2015-023-SAMPLE-4-CHN6.dat
 Calibration method: K Factors
 Sample weight: 2.421
 Protein factor: 6.25

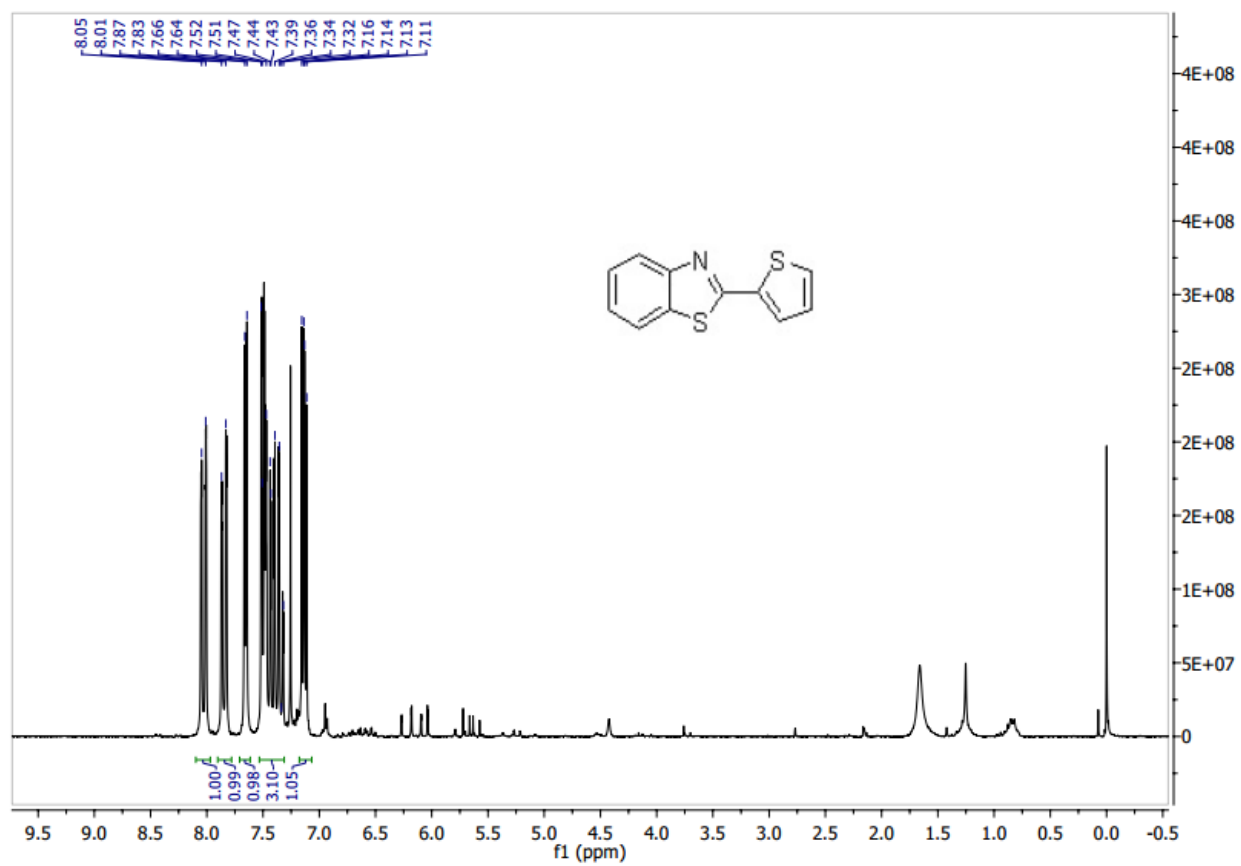


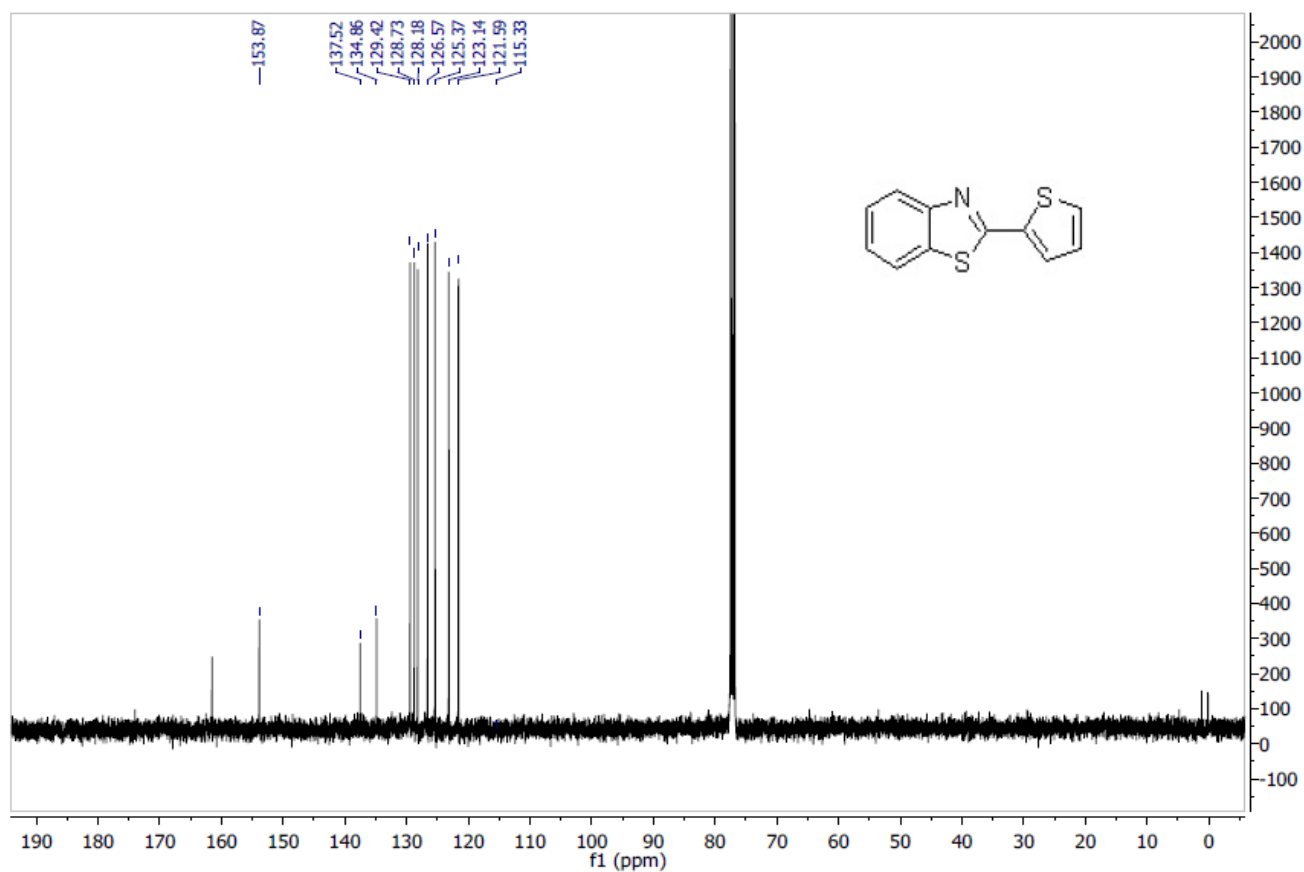
Peak Number (#)	Retention Time (min)	Area (.1* μ V*sec)	Element %	Component
1	0.775	284988	5.660	Nitrogen
2	1.067	7245252	68.242	Carbon
3	3.667	1315157	4.537	Hydrogen
4	6.167	521625	13.116	Sulphur
		9367022	91.555	

10a. 2-Thienyl-1,3-benzothiazole



Mol. Wt.: 217.2



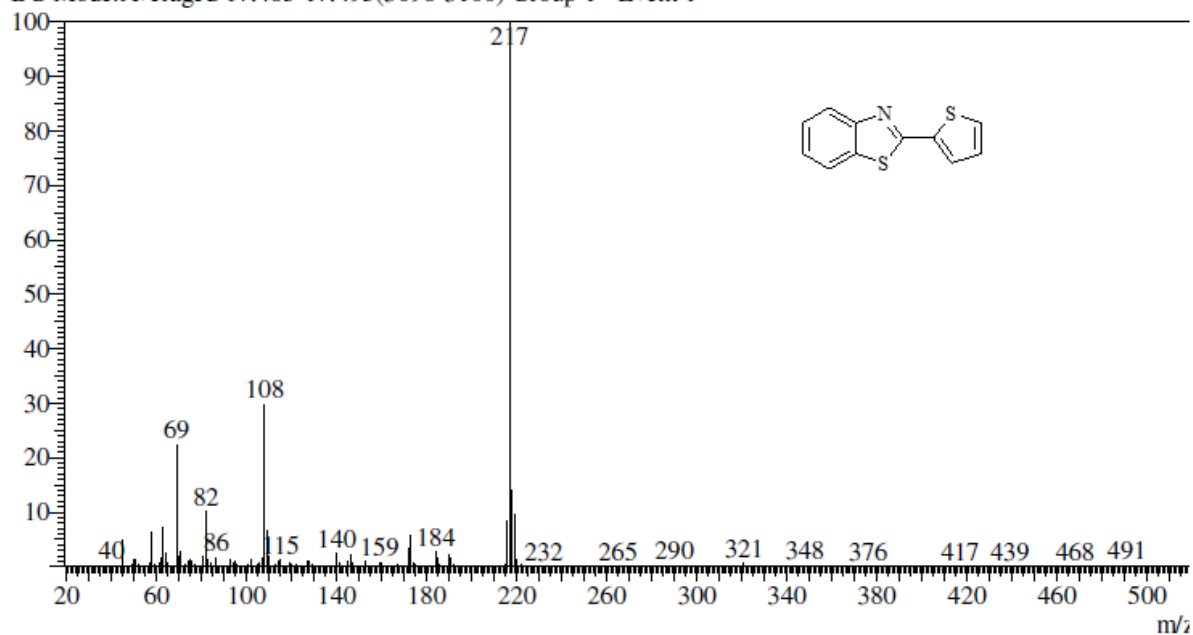


Line#:1 R.Time:16.930(Scan#:2987)

MassPeaks:298

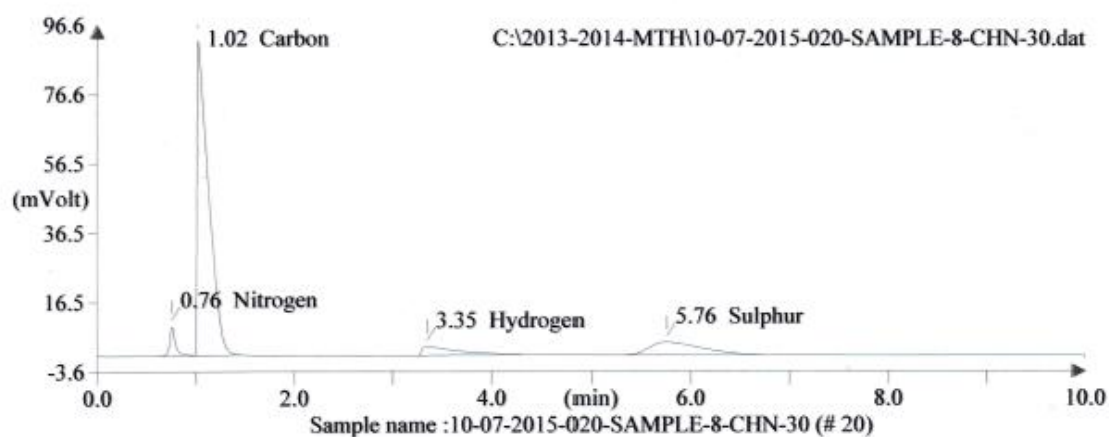
RawMode:Averaged 16.925-16.935(2986-2988) BasePeak:217(23853)

BG Mode:Averaged 17.485-17.495(3098-3100) Group 1 - Event 1



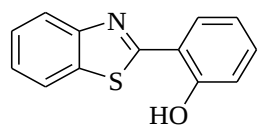
SAIF-IIT BOMBAY

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\10-07-2015-CHNS.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 07/10/2015 17:59
 Printed: 07-13-2015 13:49
 Elemental Analyser method:
 Sampler method:
 Sample ID: 10-07-2015-020-SAMPLE-8-CHN-30 (# 20)
 Analysis type: UnkNown
 Chromatogram filename: 10-07-2015-020-SAMPLE-8-CHN-30.dat
 Calibration method: K Factors
 Sample weight: 2.872
 Protein factor: 6.25

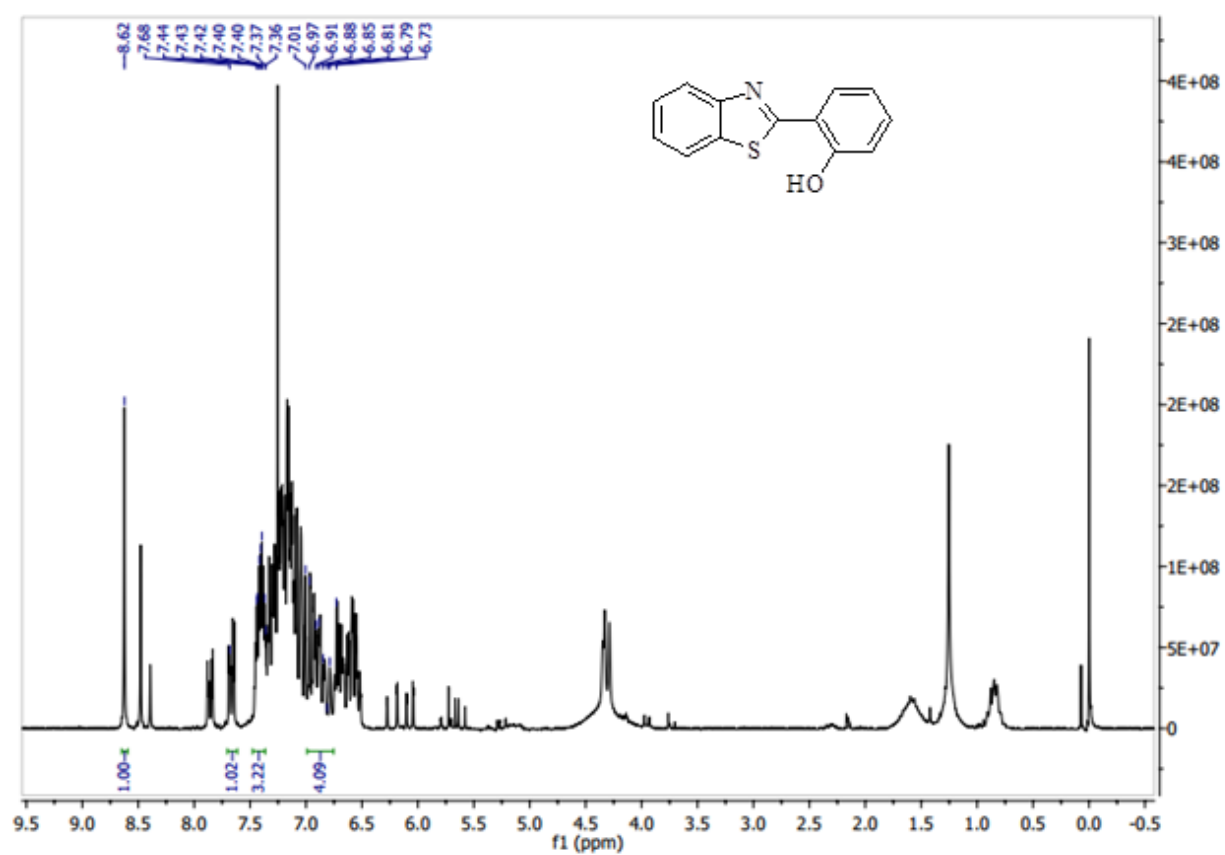


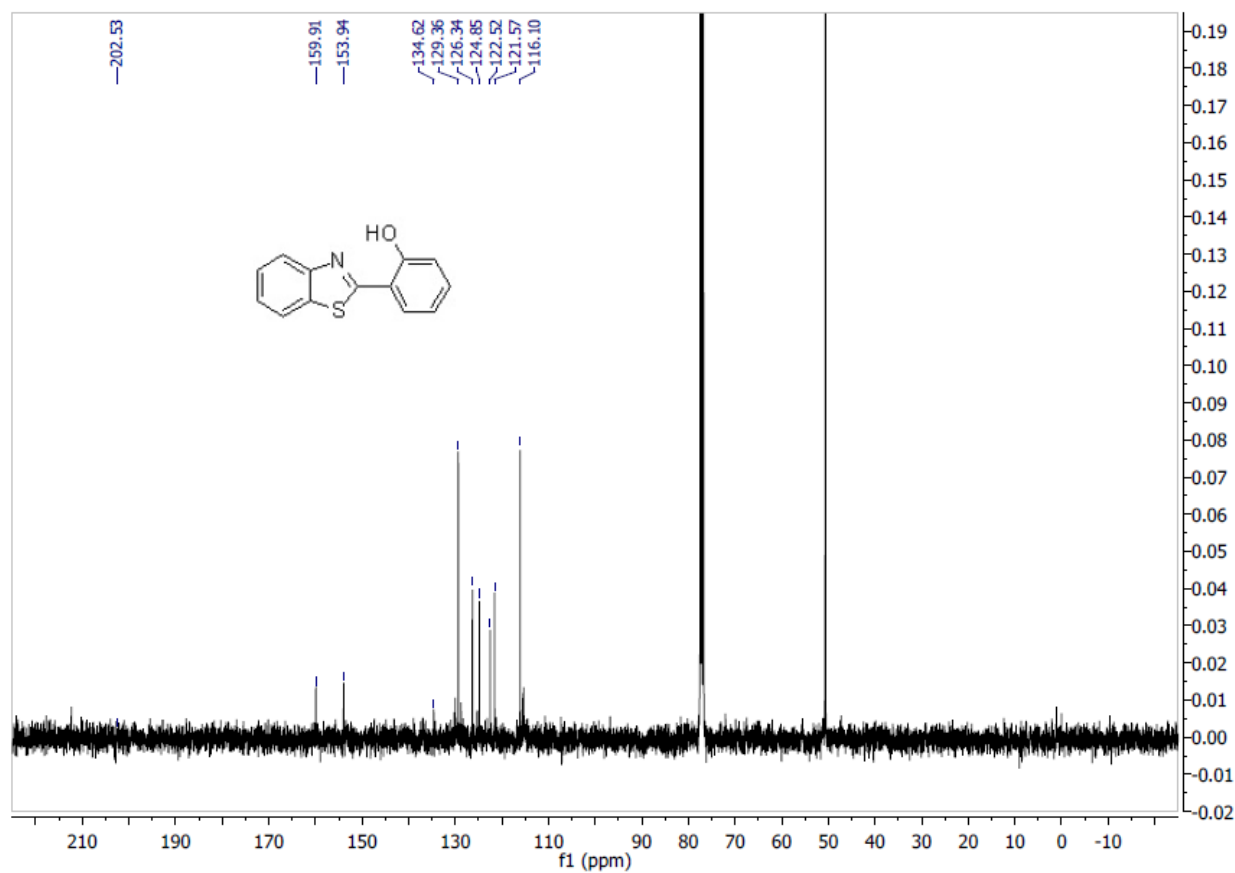
Peak Number (#)	Retention Time (min)	Area (.1* μ V*sec)	Element %	Component
1	0.758	334917	6.485	Nitrogen
2	1.025	7311114	60.616	Carbon
3	3.350	658011	3.160	Hydrogen
4	5.758	1056026	29.474	Sulphur
		9360068	99.735	

11a. 2-(2-Hydroxyphenyl)benzothiazole



Mol. Wt. 227.1



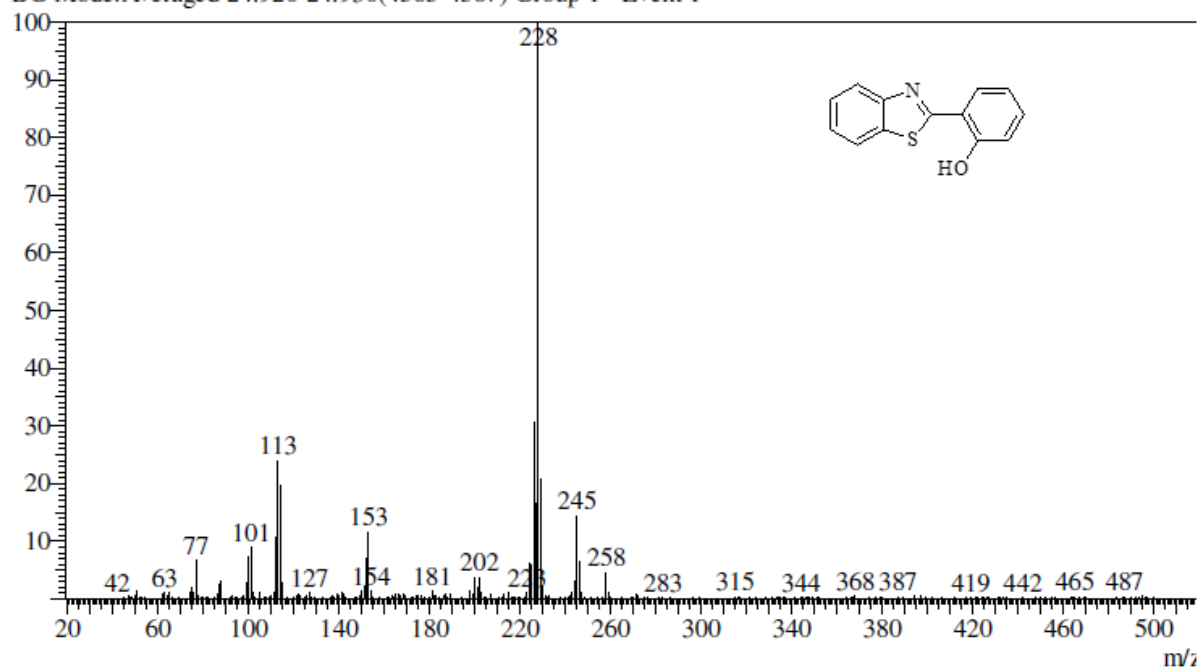


Line#:5 R.Time:24.275(Scan#:4456)

MassPeaks:338

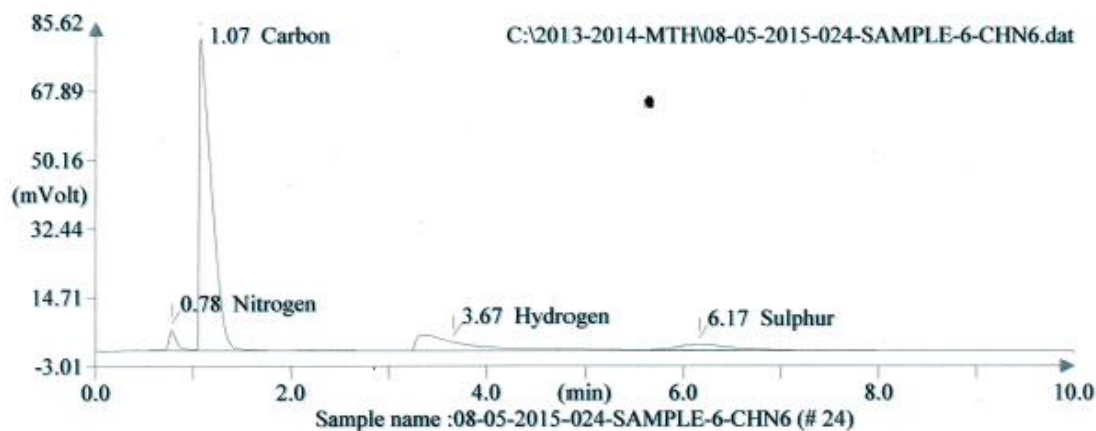
RawMode:Averaged 24.270-24.280(4455-4457) BasePeak:228(8620)

BG Mode:Averaged 24.920-24.930(4585-4587) Group 1 - Event 1



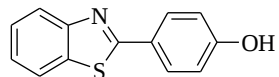
SAIF-IIT BOMBAY

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\08-05-2015-CHNS.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 05/08/2015 18:32
 Printed: 05-08-2015 18:42
 Elemental Analyser method:
 Sampler method:
 Sample ID: 08-05-2015-024-SAMPLE-6-CHN6 (# 24)
 Analysis type: UnkNown
 Chromatogram filename: 08-05-2015-024-SAMPLE-6-CHN6.dat
 Calibration method: K Factors
 Sample weight: 2.374
 Protein factor: 6.25

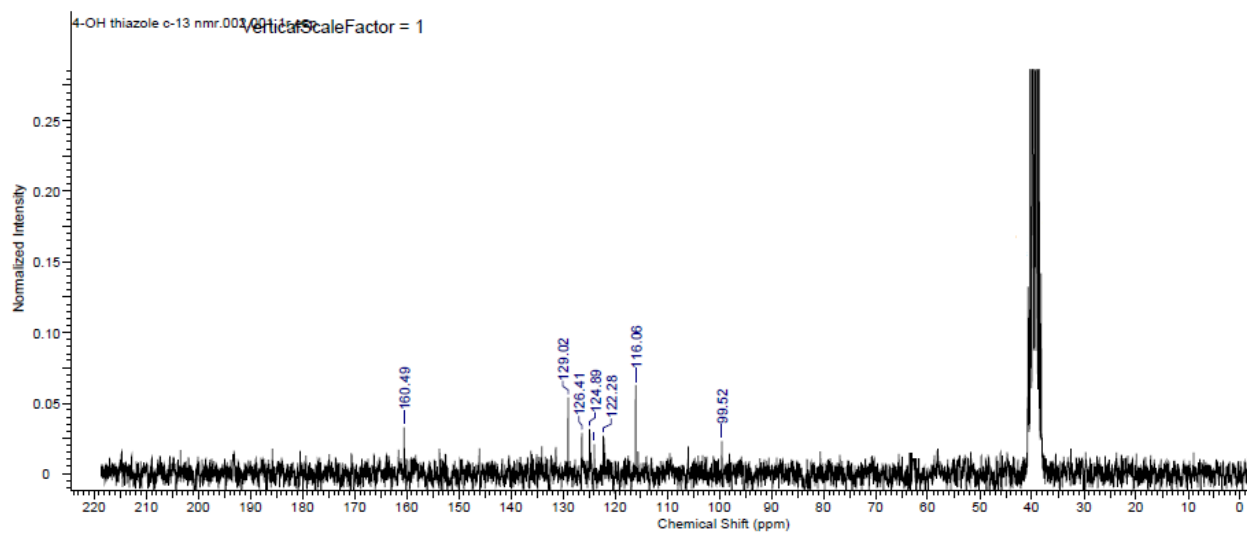
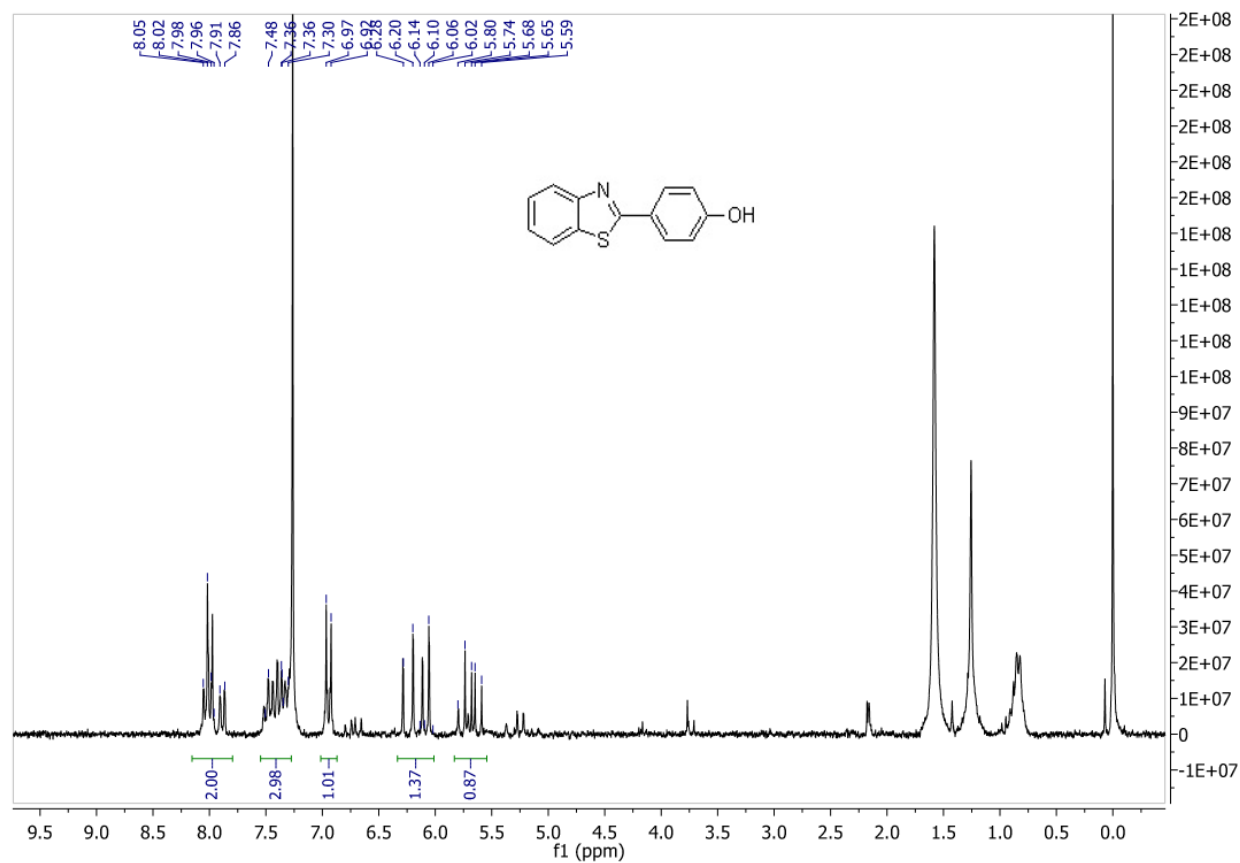


Peak Number (#)	Retention Time (min)	Area (.1* μ V*sec)	Element %	Component
1	0.783	300586	6.091	Nitrogen
2	1.075	7154475	68.721	Carbon
3	3.667	1031974	3.631	Hydrogen
4	6.167	534138	13.969	Sulphur
		9021173	92.412	

12a. 2-(4-Hydroxyphenyl)benzothiazole

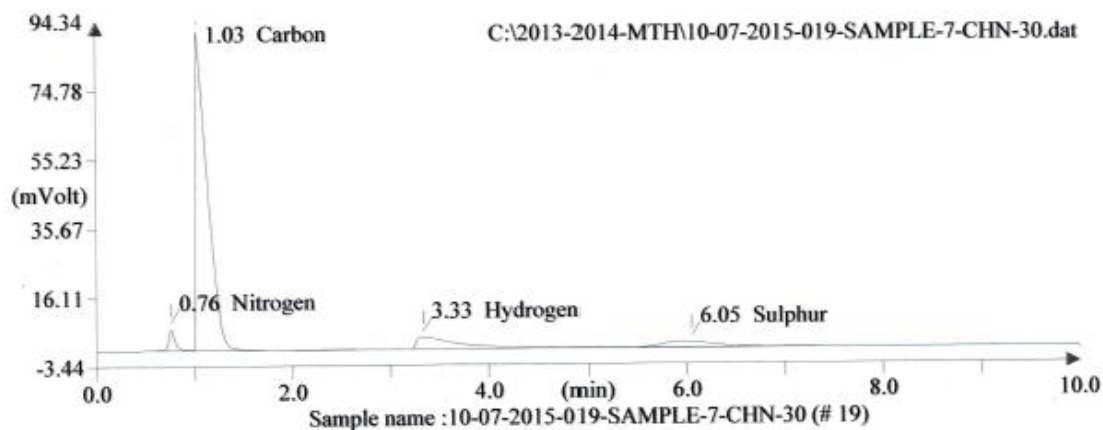


Mol. Wt.: 227.1



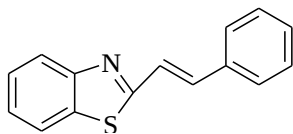
SAIF-IIT BOMBAY

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\10-07-2015-CHNS.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 07/10/2015 17:49
 Printed: 07-13-2015 13:43
 Elemental Analyser method:
 Sampler method:
 Sample ID: 10-07-2015-019-SAMPLE-7-CHN-30 (# 19)
 Analysis type: UnkNown
 Chromatogram filename: 10-07-2015-019-SAMPLE-7-CHN-30.dat
 Calibration method: K Factors
 Sample weight: 2.66
 Protein factor: 6.25

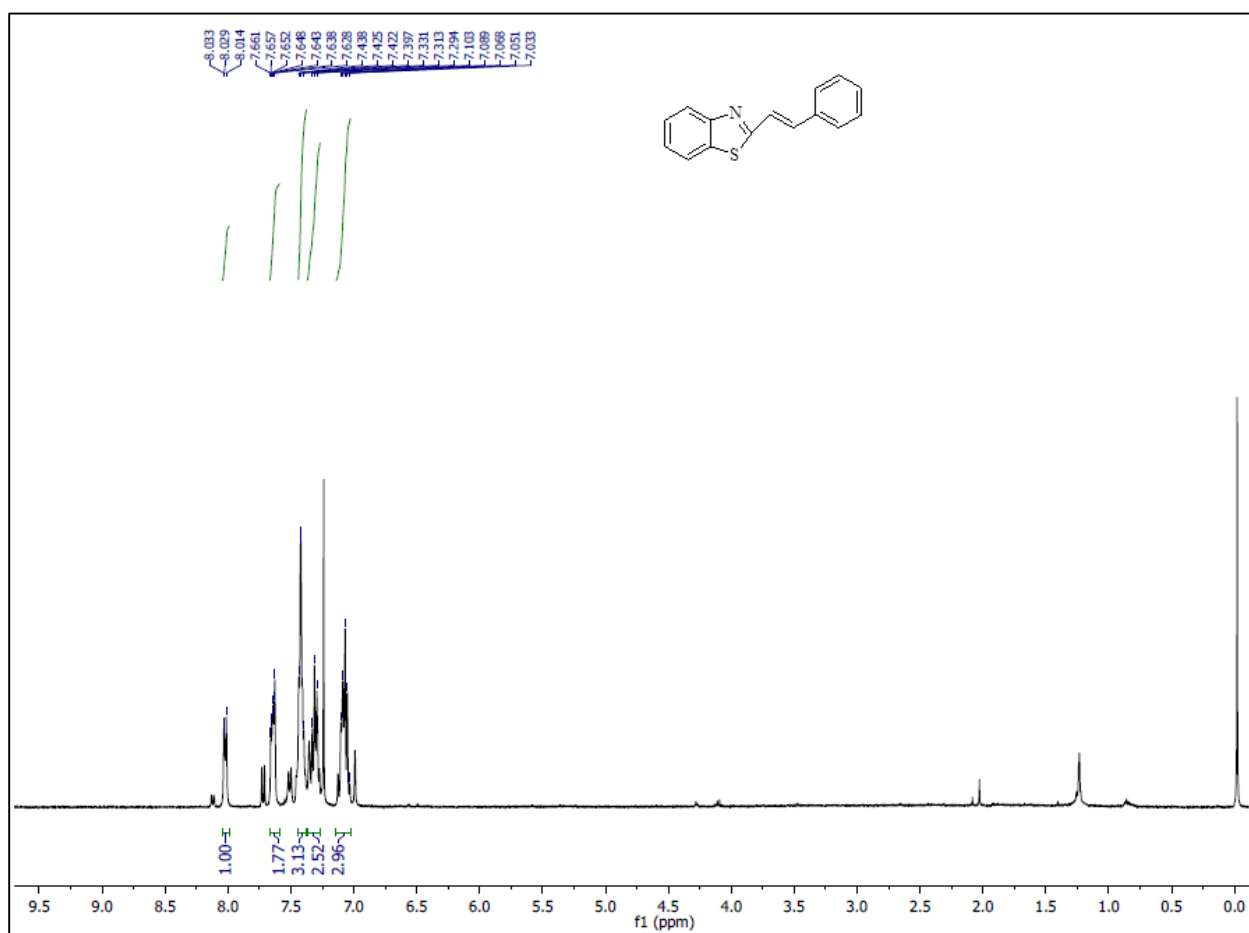


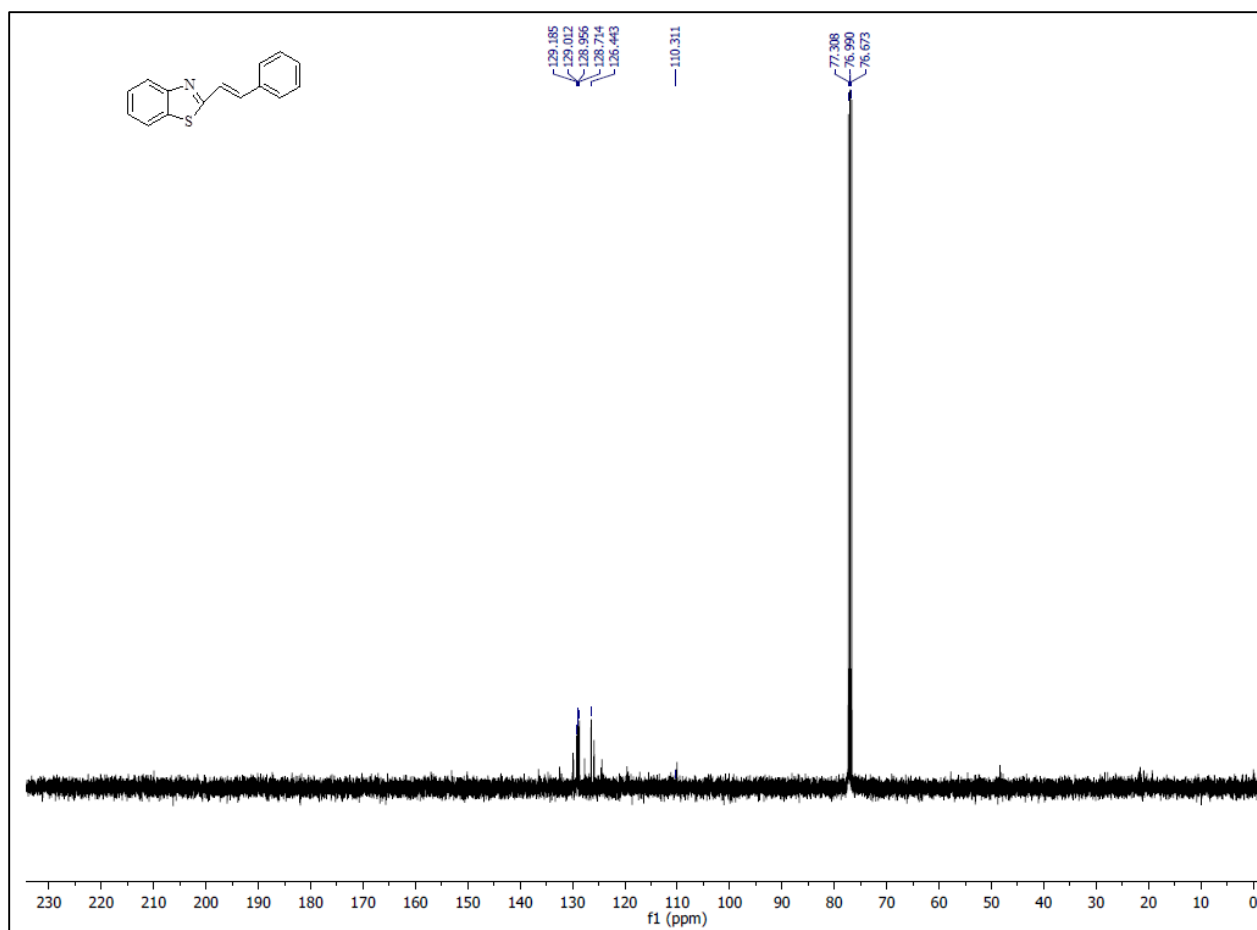
Peak Number (#)	Retention Time (min)	Area (.1*uV*sec)	Element %	Component
1	0.758	300368	6.074	Nitrogen
2	1.033	7597899	68.028	Carbon
3	3.333	1211749	3.913	Hydrogen
4	6.050	584512	13.961	Sulphur
		9694528	91.976	

13a. 2-Styryl-benzothiazole:



Mol. Wt.: 236



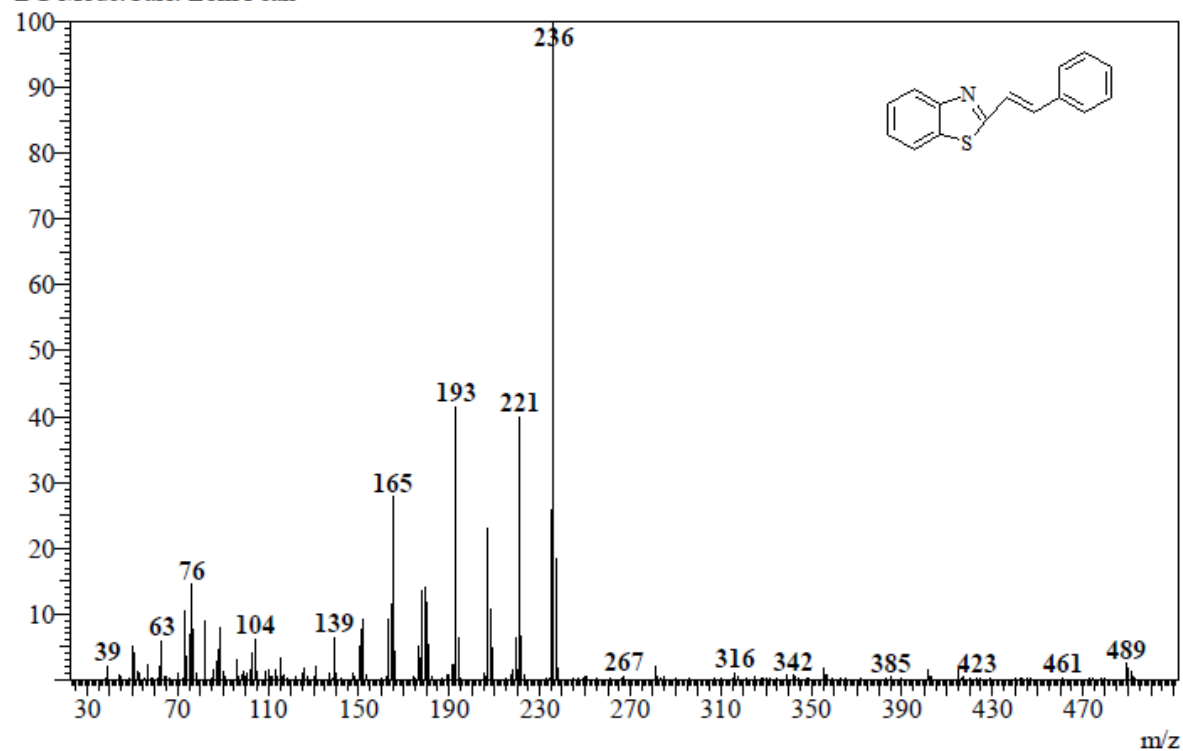


Line#:1 R.Time:18.6(Scan#:3123)

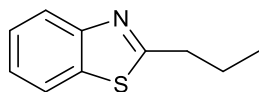
MassPeaks:302

RawMode:Averaged 18.6-18.6(3122-3124) BasePeak:236(25948)

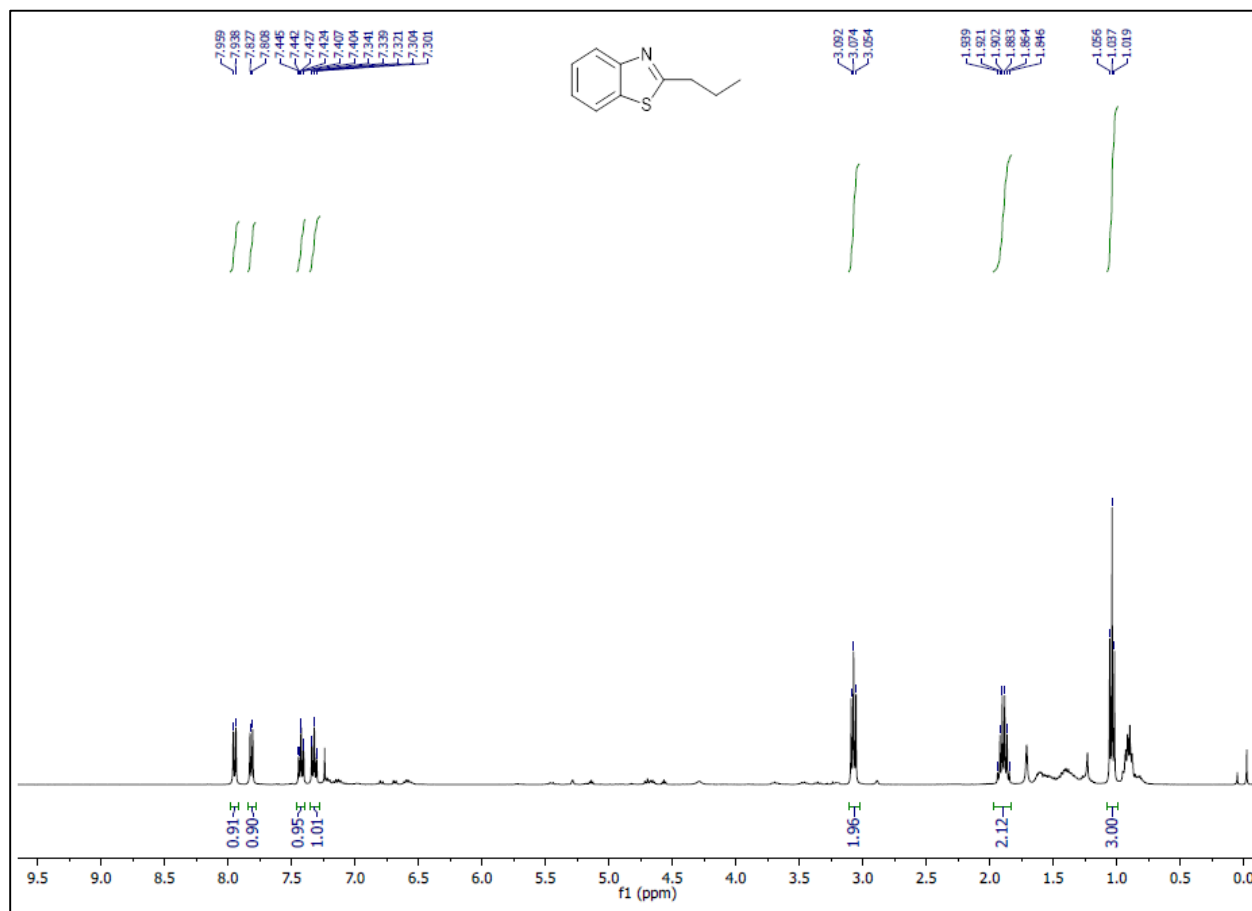
BG Mode:Calc. from Peak

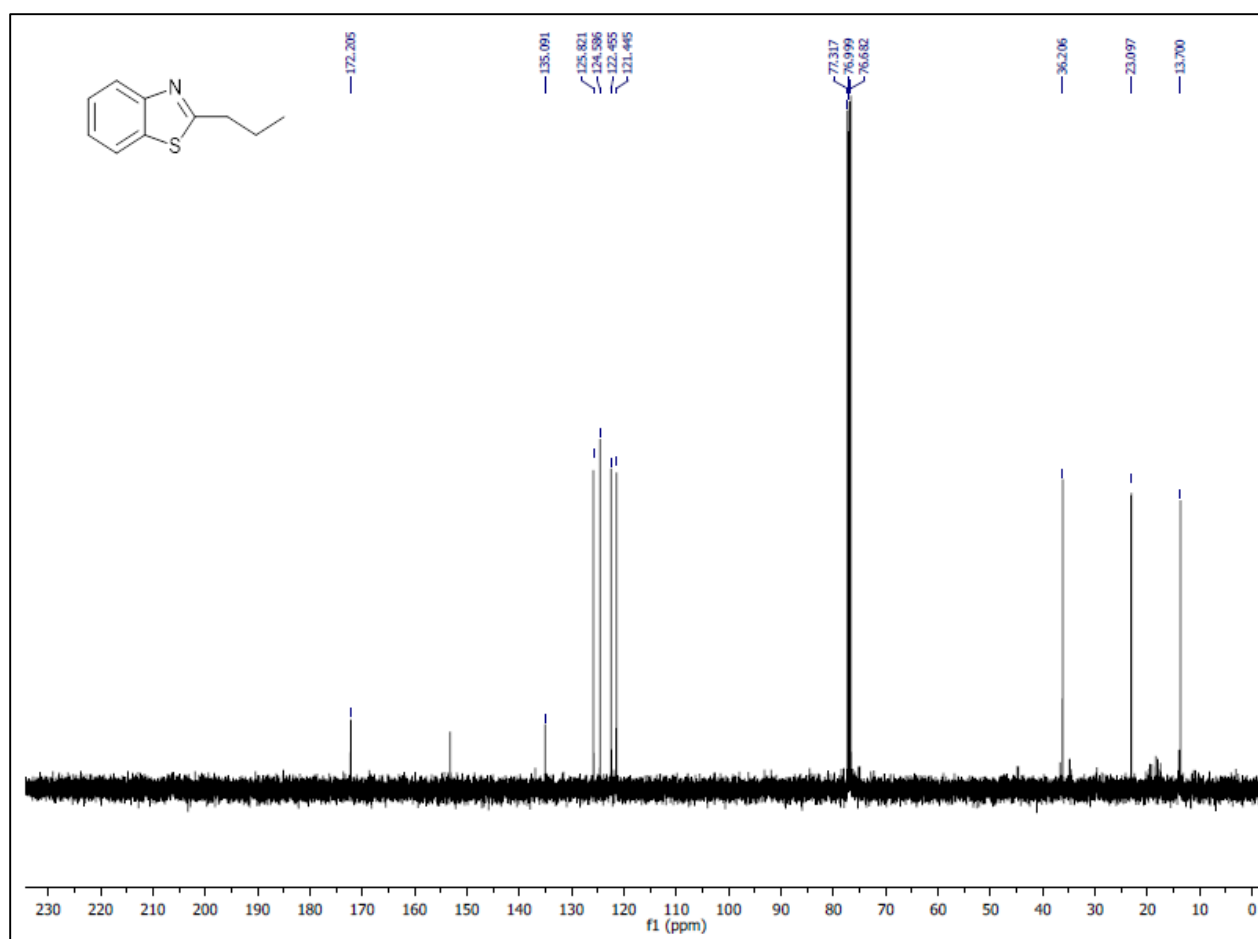


14a. 2-Butyl-benzothiazole:



Mol. Wt.: 177



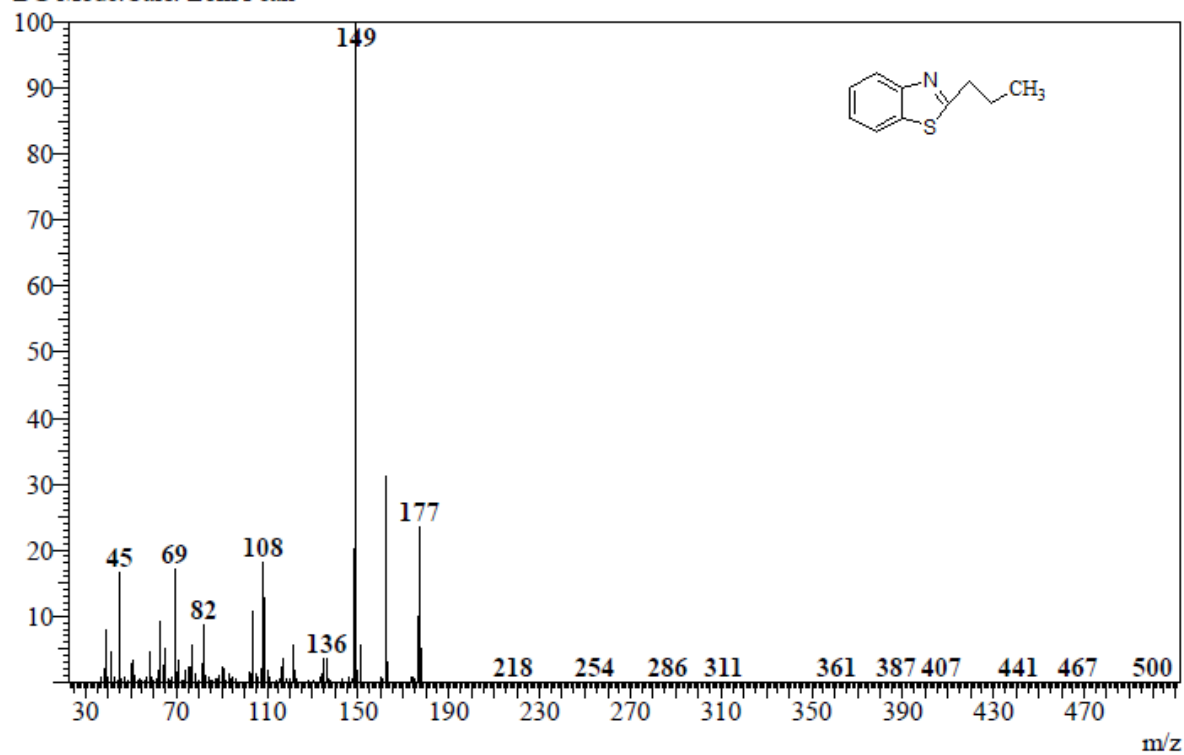


Line#:1 R.Time:10.4(Scan#:1489)

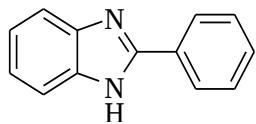
MassPeaks:297

RawMode:Averaged 10.4-10.4(1488-1490) BasePeak:149(7991066)

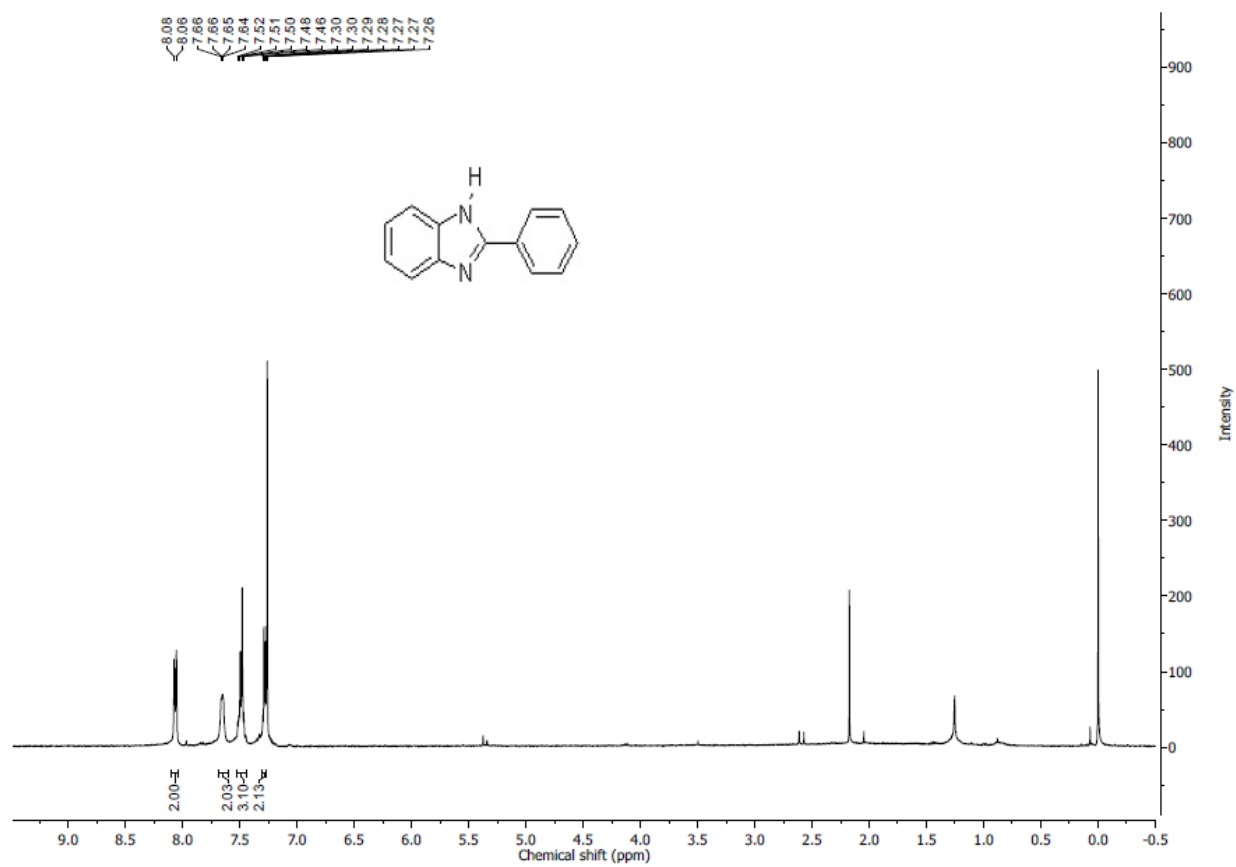
BG Mode:Calc. from Peak

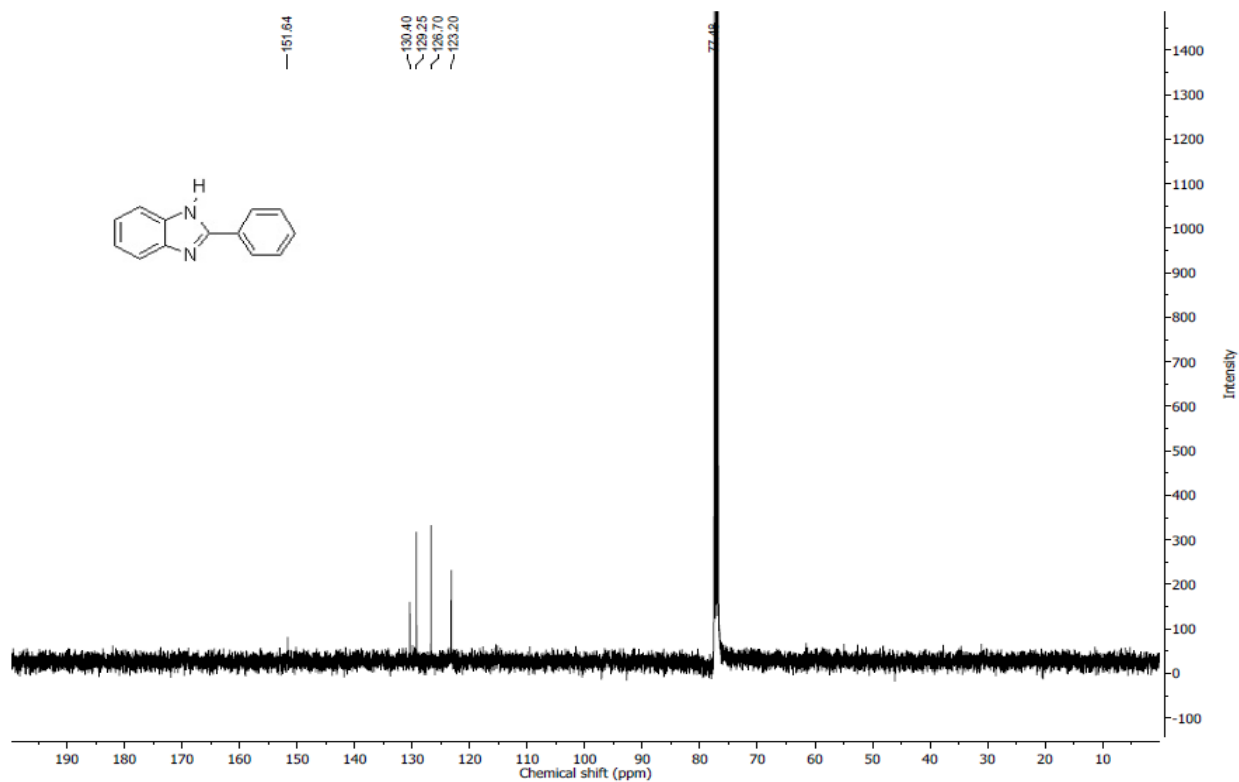


15a 2-Phenylbenzimidazole



Mol. Wt.: 194.2



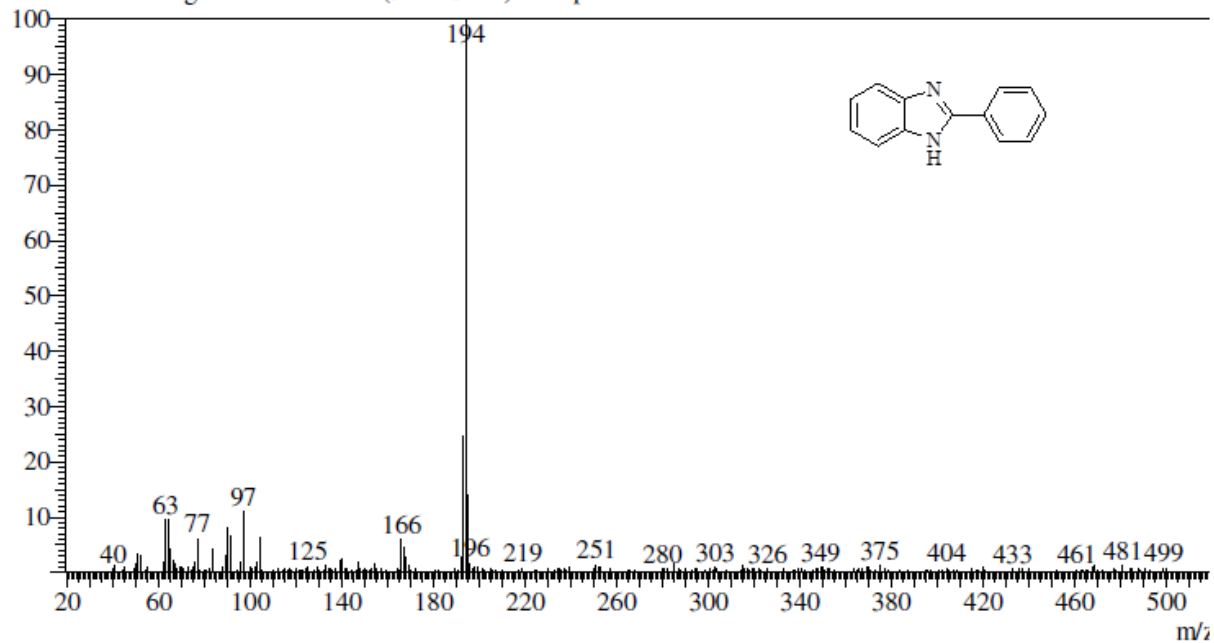


Line#:1 R.Time:17.100(Scan#:3021)

MassPeaks:303

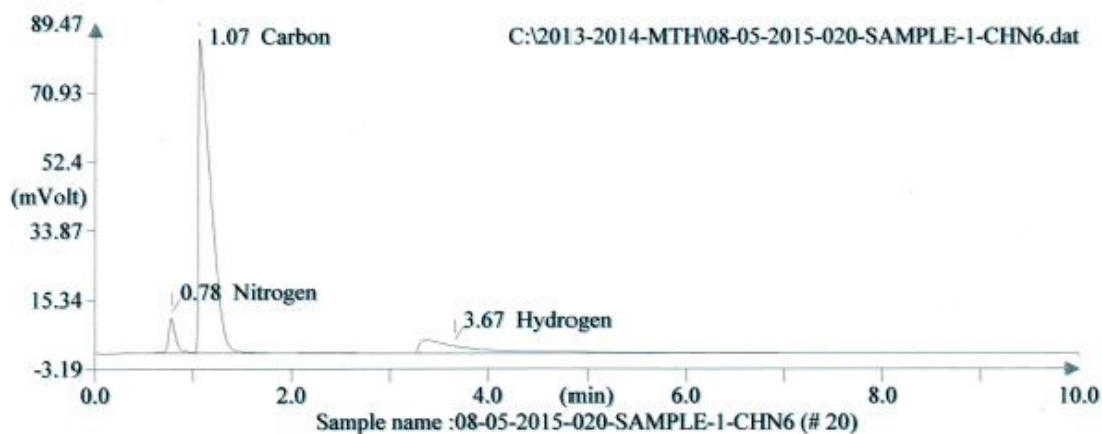
RawMode:Averaged 17.095-17.105(3020-3022) BasePeak:194(2927)

BG Mode:Averaged 18.070-18.080(3215-3217) Group 1 - Event 1



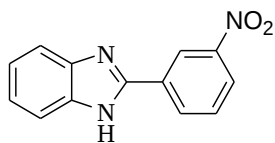
SAIF-IIT BOMBAY

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\08-05-2015-CHNS.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 05/08/2015 17:50
 Printed: 05-08-2015 18:00
 Elemental Analyser method:
 Sampler method:
 Sample ID: 08-05-2015-020-SAMPLE-1-CHN6 (# 20)
 Analysis type: UnkNown
 Chromatogram filename: 08-05-2015-020-SAMPLE-1-CHN6.dat
 Calibration method: K Factors
 Sample weight: 2.04
 Protein factor: 6.25

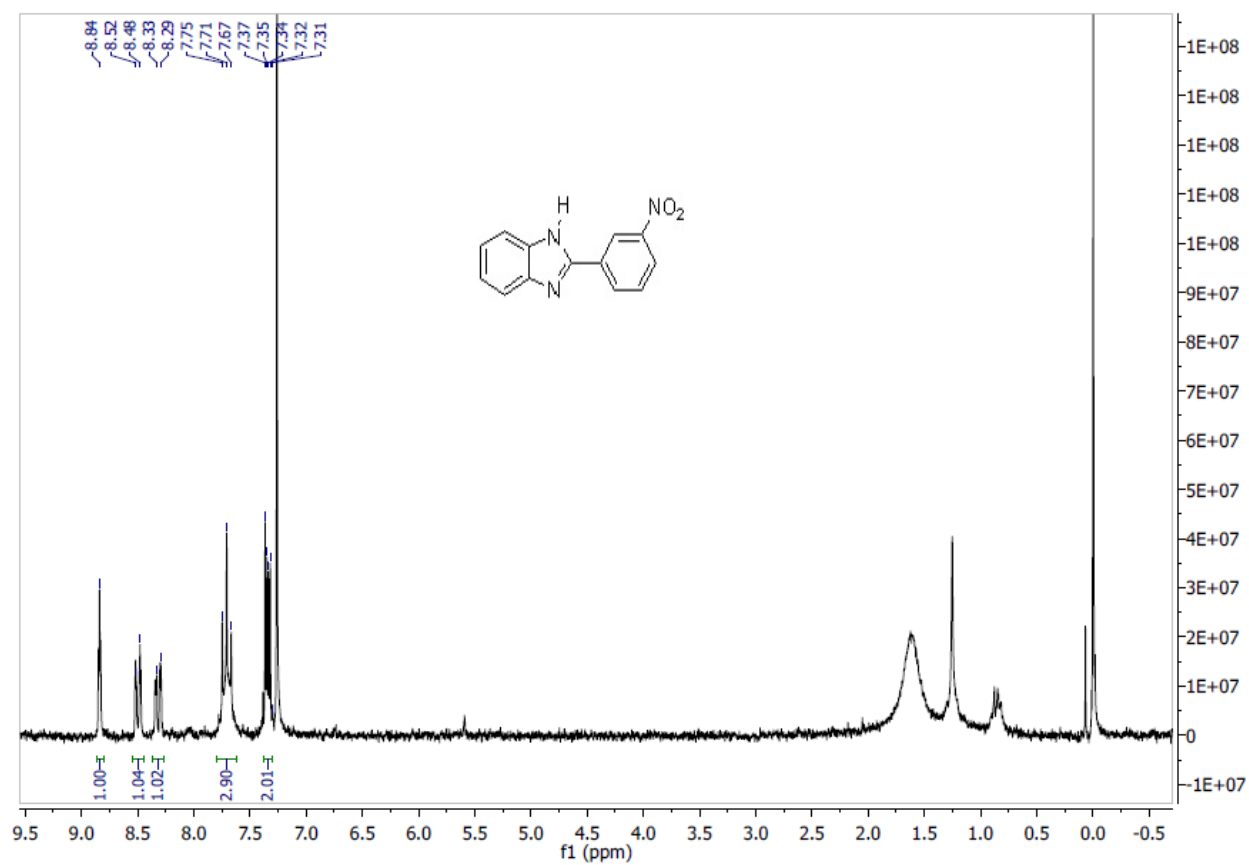


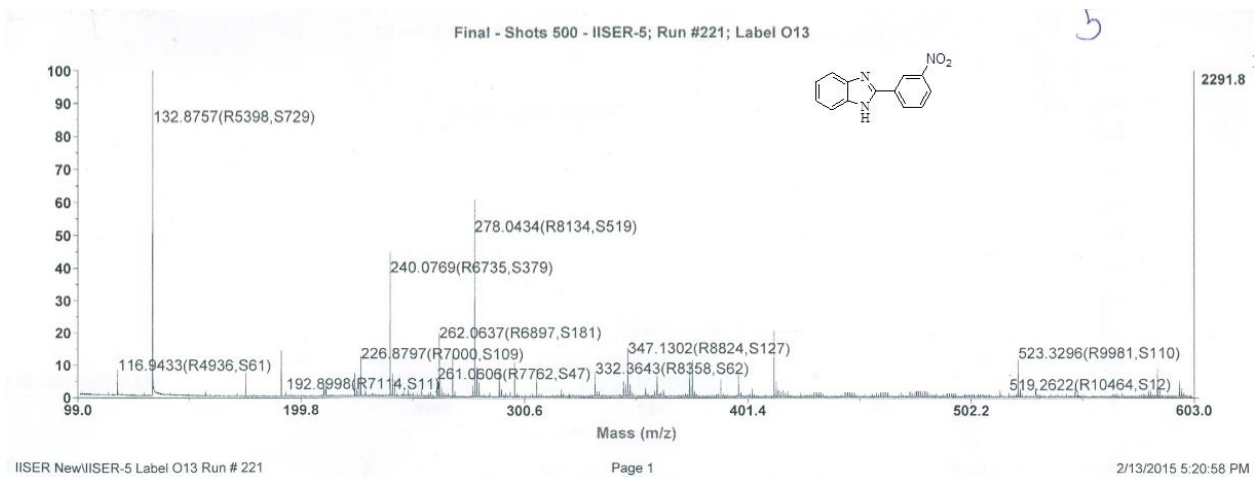
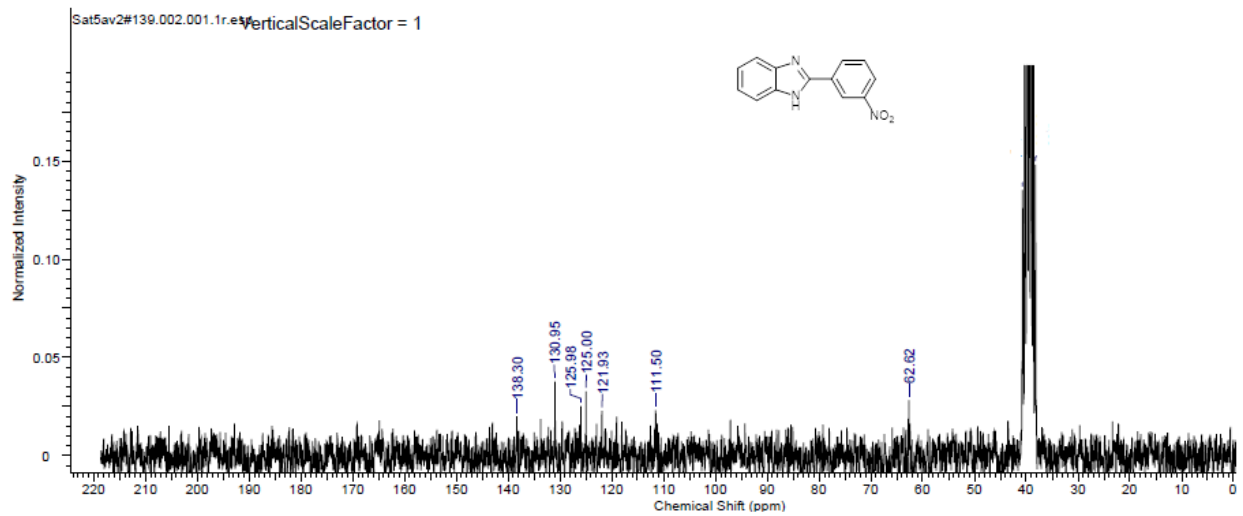
Peak Number (#)	Retention Time (min)	Area (.1*uV*sec)	Element %	Component
1	0.783	573613	14.306	Nitrogen
2	1.067	7170320	80.150	Carbon
3	3.667	1346343	5.512	Hydrogen
		9090276	99.968	

16a. 2-(3-Nitrophenyl)-1H-benzimidazole



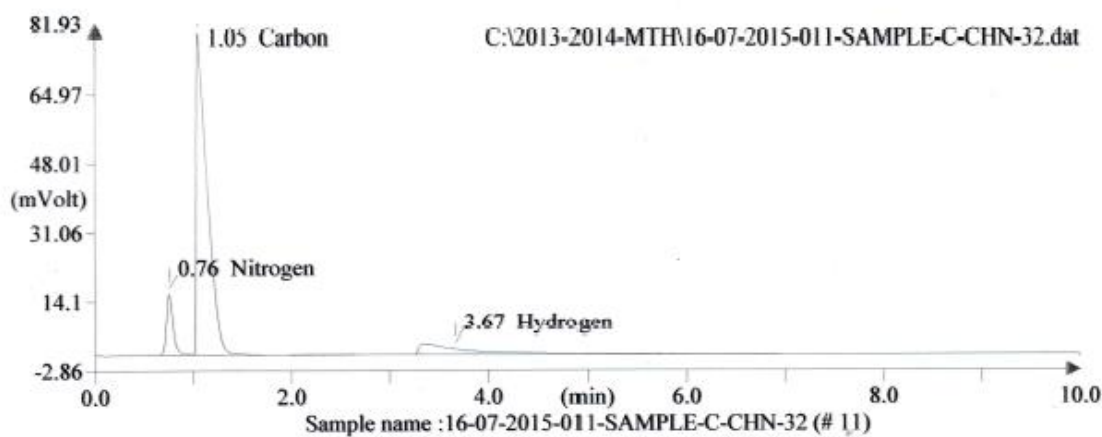
Mol. Wt.: 239.2





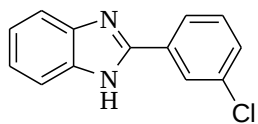
SAIF-IIT BOMBAY

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\16-07-2014-CHNS.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 07/16/2015 17:37
 Printed: 07-17-2015 11:06
 Elemental Analyser method:
 Sampler method:
 Sample ID: 16-07-2015-011-SAMPLE-C-CHN-32 (# 11)
 Analysis type: UnkNown
 Chromatogram filename: 16-07-2015-011-SAMPLE-C-CHN-32.dat
 Calibration method: K Factors
 Sample weight: 2.401
 Protein factor: 6.25

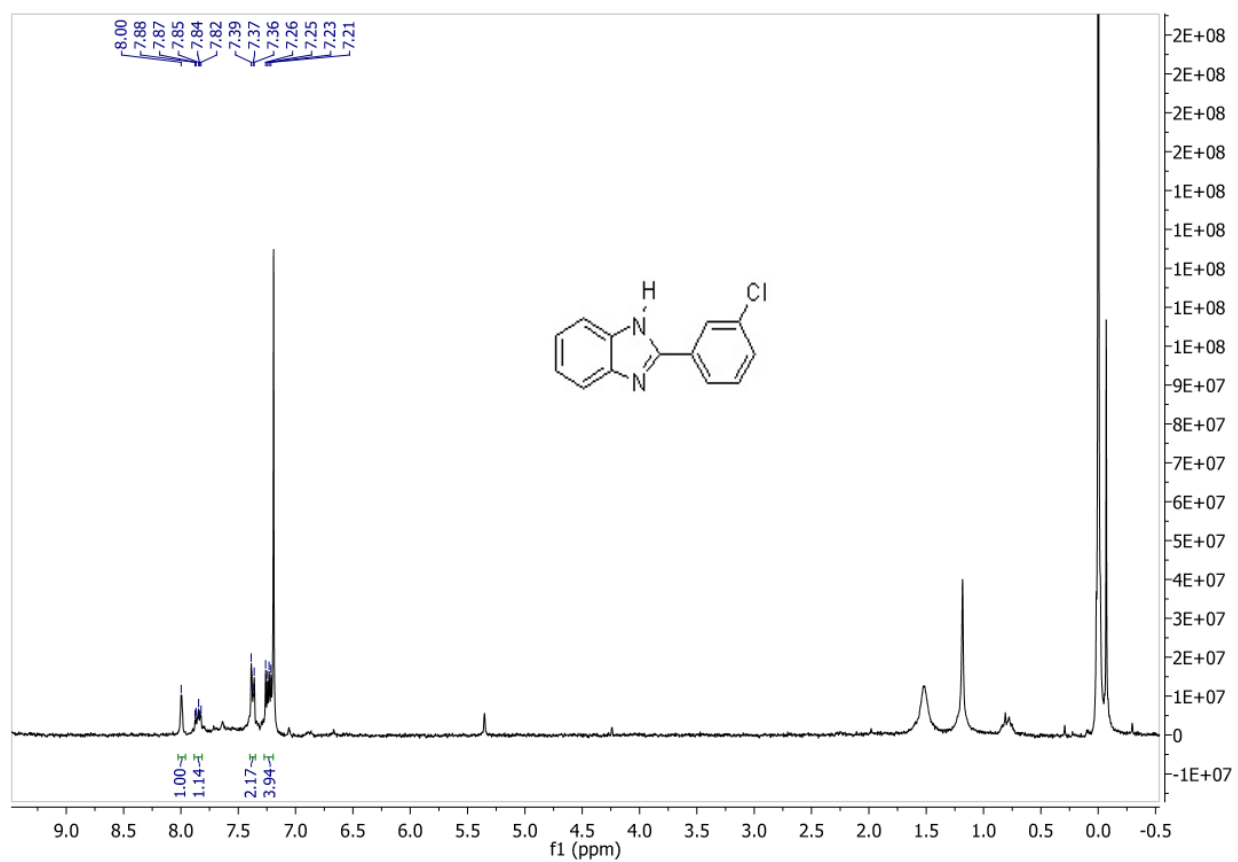


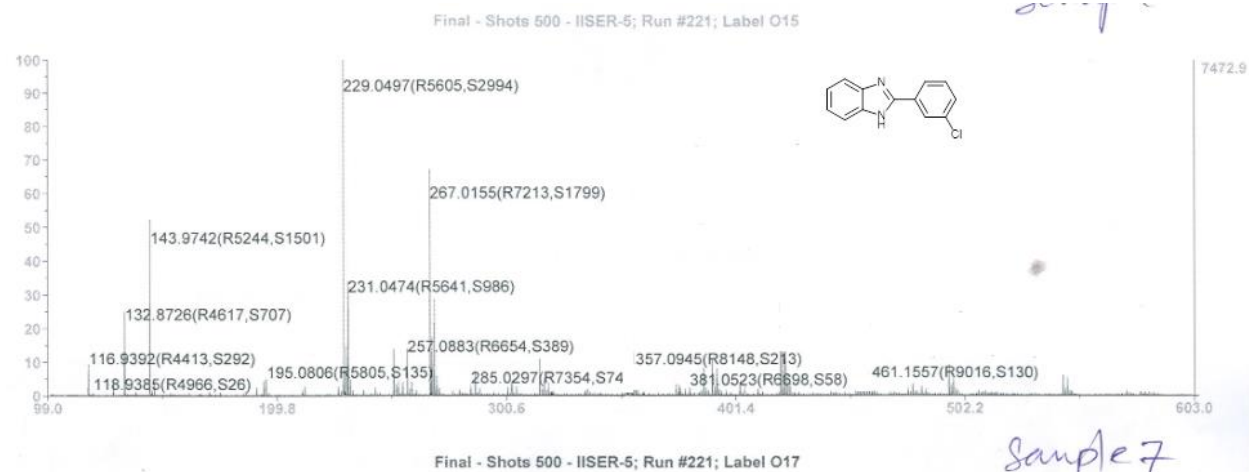
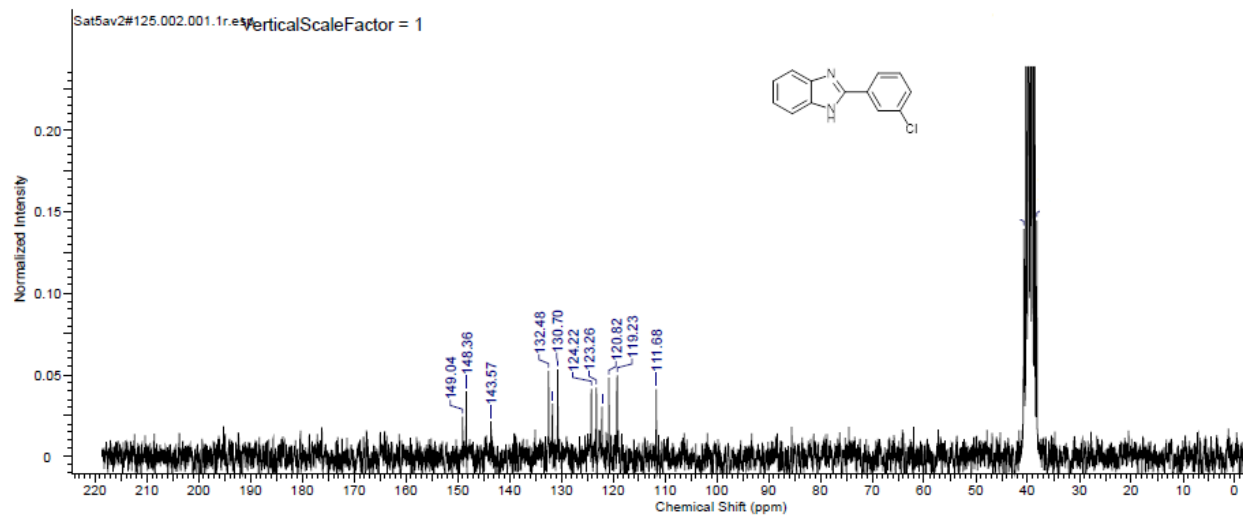
Peak Number (#)	Retention Time (min)	Area (.1*uV*sec)	Element %	Component
1	0.758	710007	17.502	Nitrogen
2	1.050	6525116	65.150	Carbon
3	3.667	890713	3.511	Hydrogen
		8125836	86.163	

17a. 2-(3-Chlorophenyl)-1H-benzimidazole

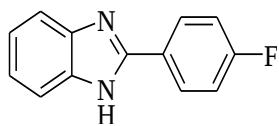


Mol. Wt.: 239.6

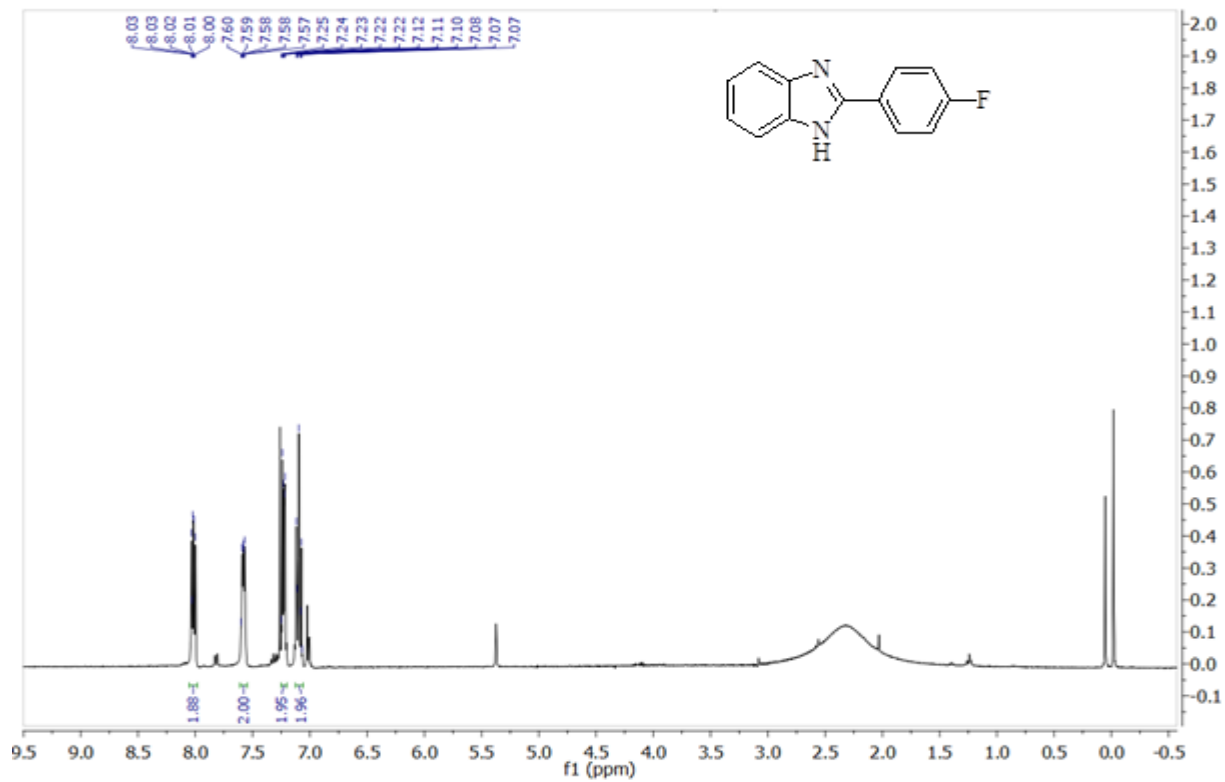


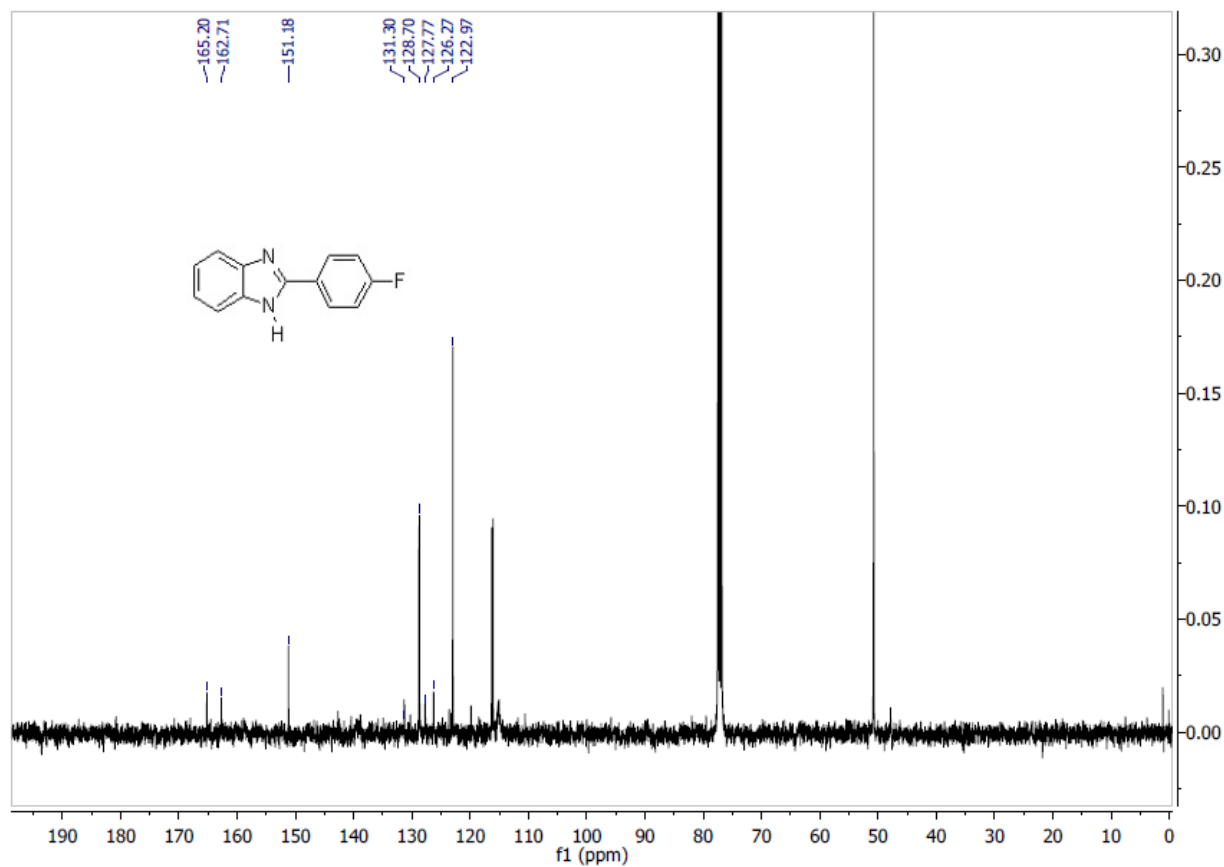


18a. 2-(4-Fluorophenyl)-1H-benzimidazole



Mol. Wt.: 212.2



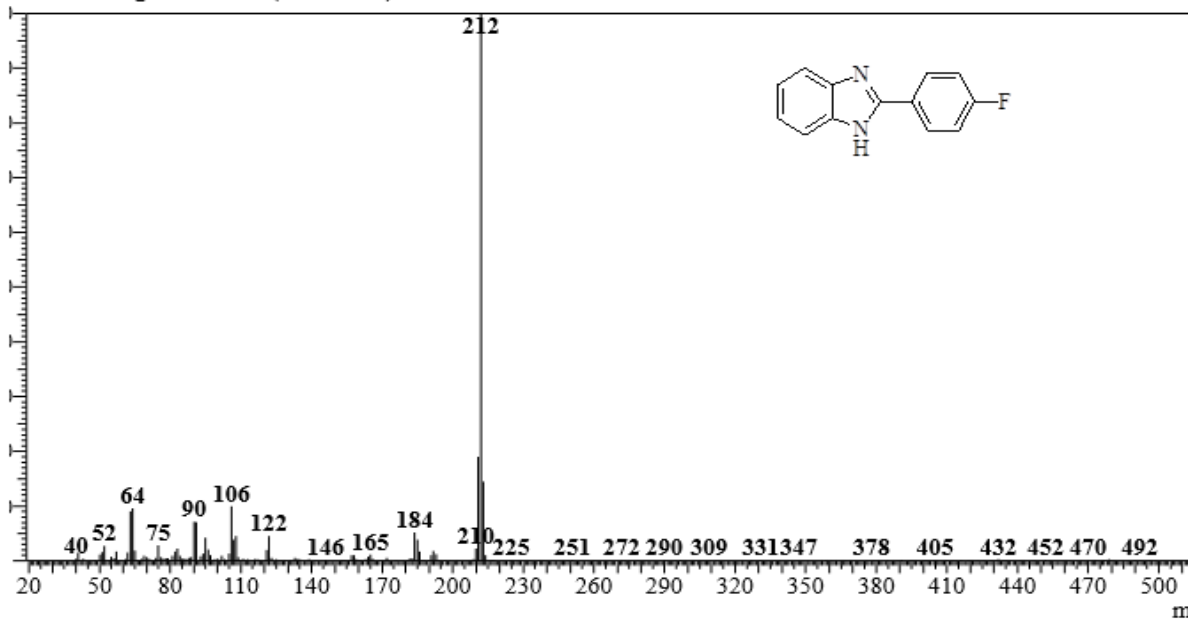


e#:1 R.Time:28.5(Scan#:3060)

ssPeaks:311

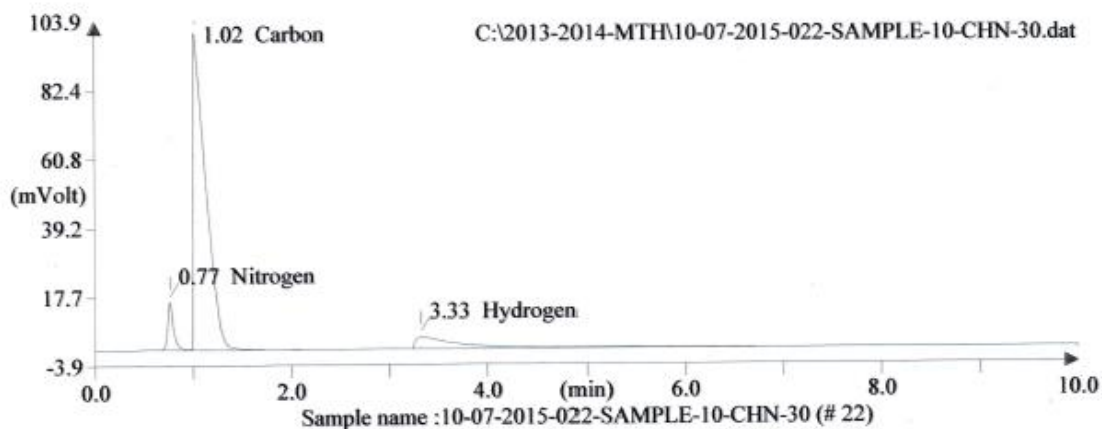
vMode:Averaged 28.5-28.5(3059-3061) BasePeak:212(11401)

Mode:Averaged 30.7-30.8(3330-3332)



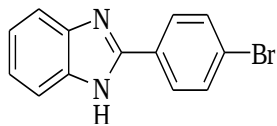
SAIF-IIT BOMBAY

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\10-07-2015-CHNS.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 07/10/2015 18:20
 Printed: 07-13-2015 13:45
 Elemental Analyser method:
 Sampler method:
 Sample ID: 10-07-2015-022-SAMPLE-10-CHN-30 (# 22)
 Analysis type: UnkNown
 Chromatogram filename: 10-07-2015-022-SAMPLE-10-CHN-30.dat
 Calibration method: K Factors
 Sample weight: 2.819
 Protein factor: 6.25

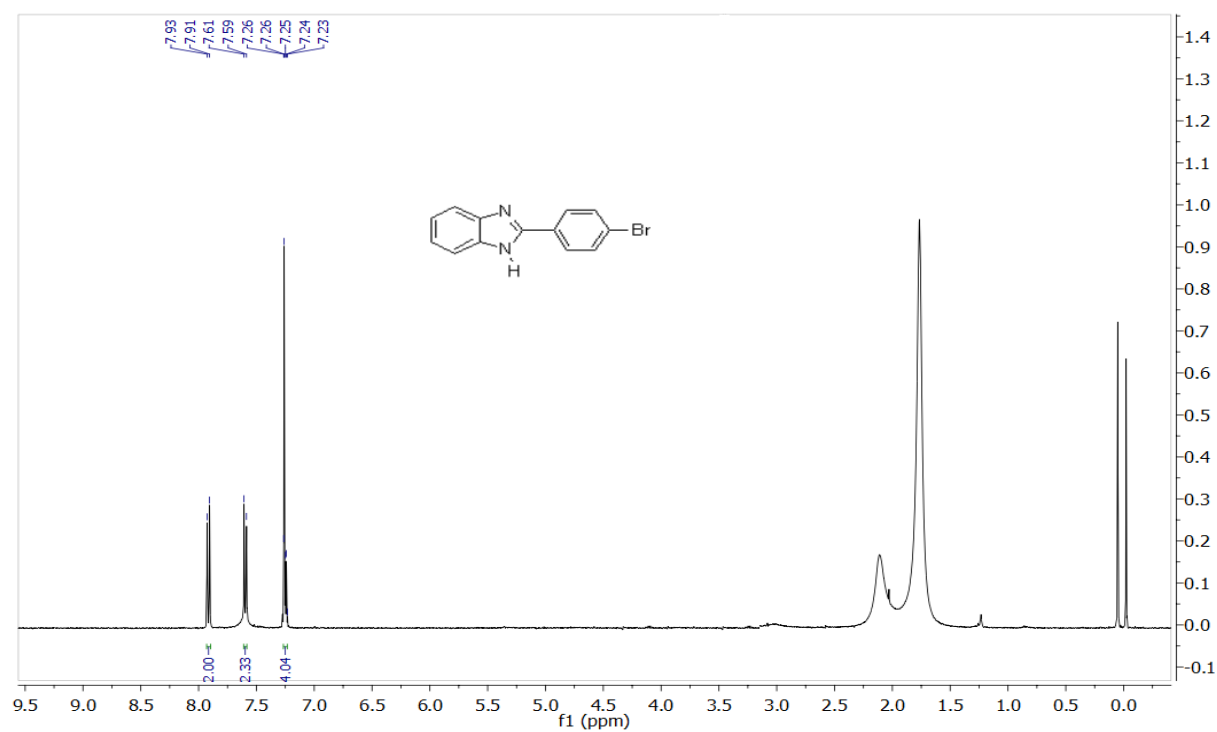


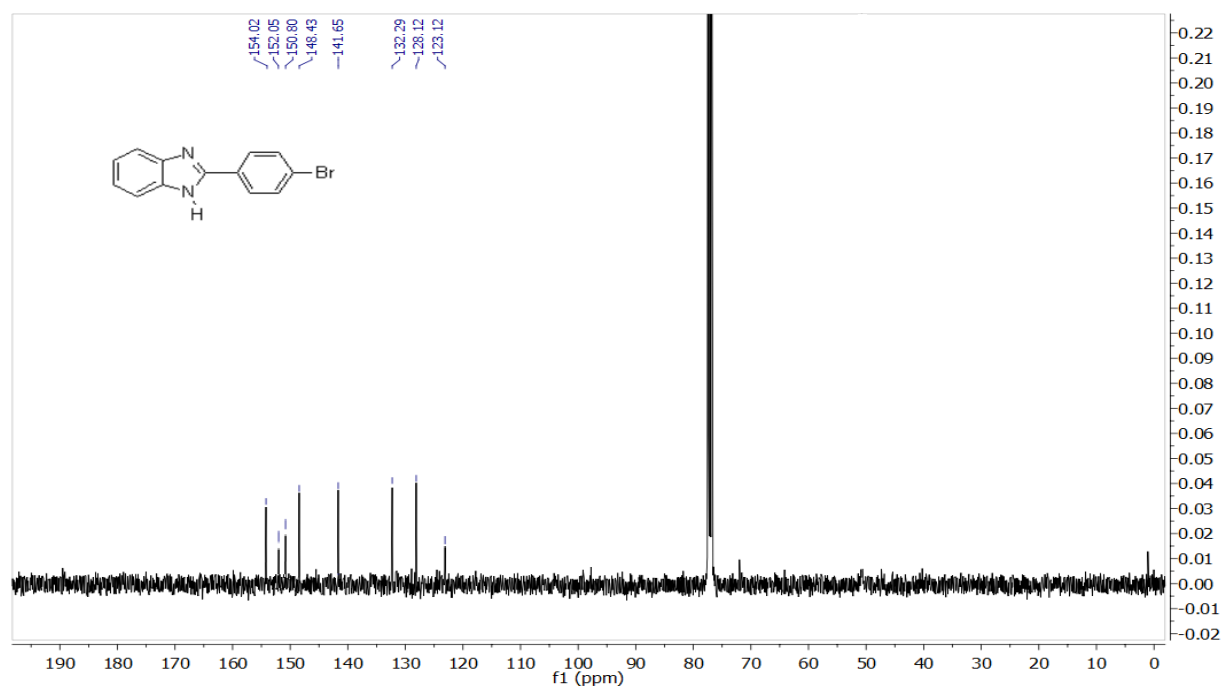
Peak Number (#)	Retention Time (min)	Area (.1*uV*sec)	Element %	Component
1	0.767	629914	13.043	Nitrogen
2	1.025	8653016	73.150	Carbon
3	3.325	1364987	4.160	Hydrogen
		10647917	89.981	

19a. 2-(4-Bromophenyl)-1H-benzimidazole



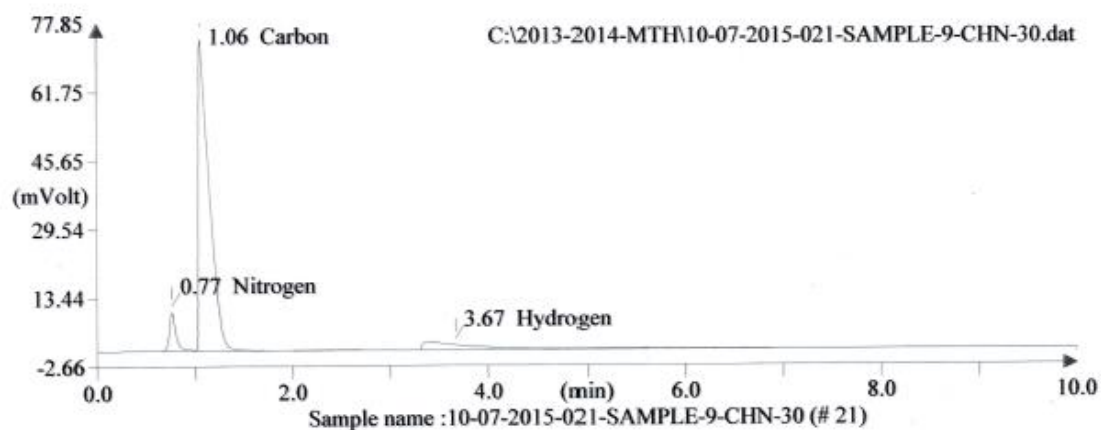
Mol. Wt.: 290.1





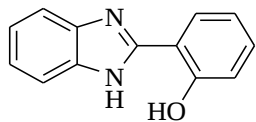
SAIF-IIT BOMBAY

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\10-07-2015-CHNS.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 07/10/2015 18:10
 Printed: 07-13-2015 13:44
 Elemental Analyser method:
 Sampler method:
 Sample ID: 10-07-2015-021-SAMPLE-9-CHN-30 (# 21)
 Analysis type: UnkNown
 Chromatogram filename: 10-07-2015-021-SAMPLE-9-CHN-30.dat
 Calibration method: K Factors
 Sample weight: 2.279
 Protein factor: 6.25

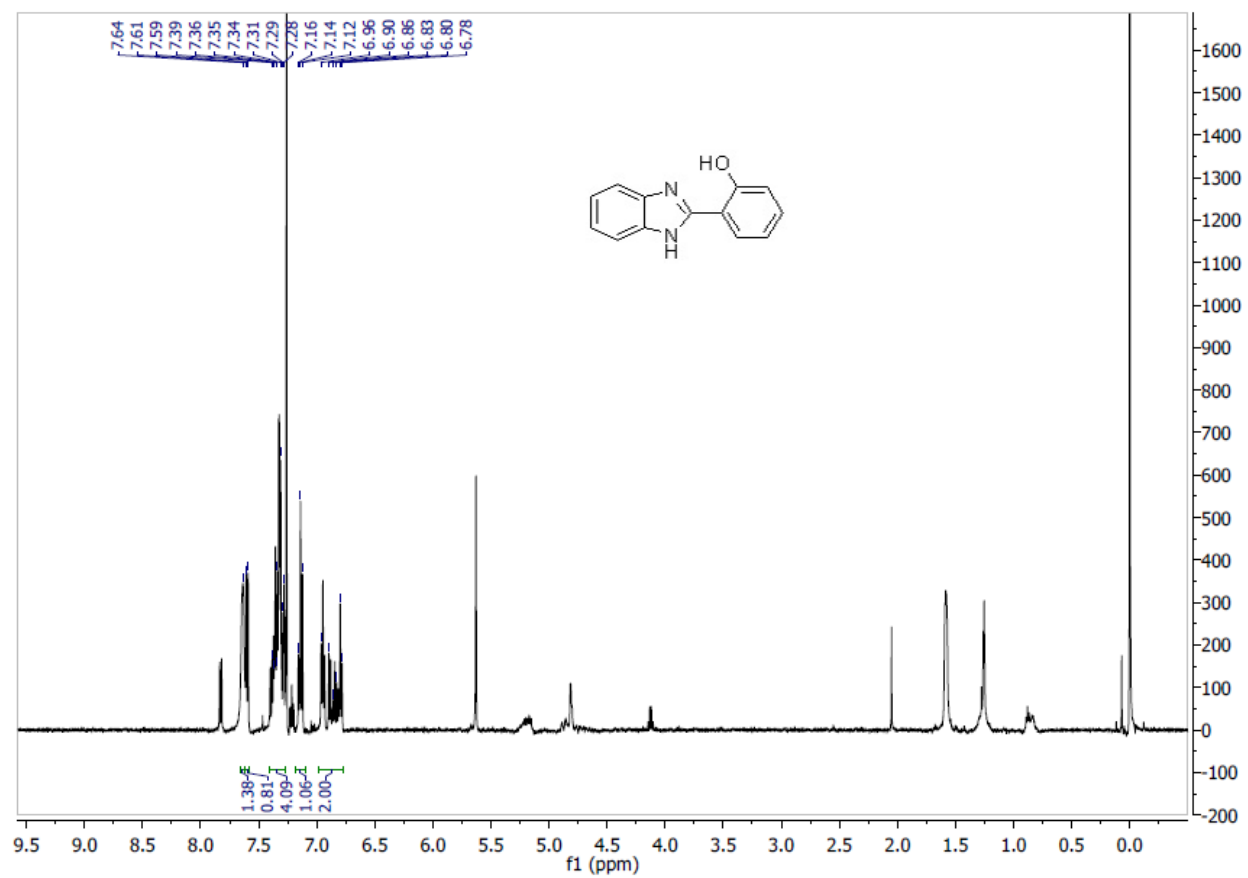


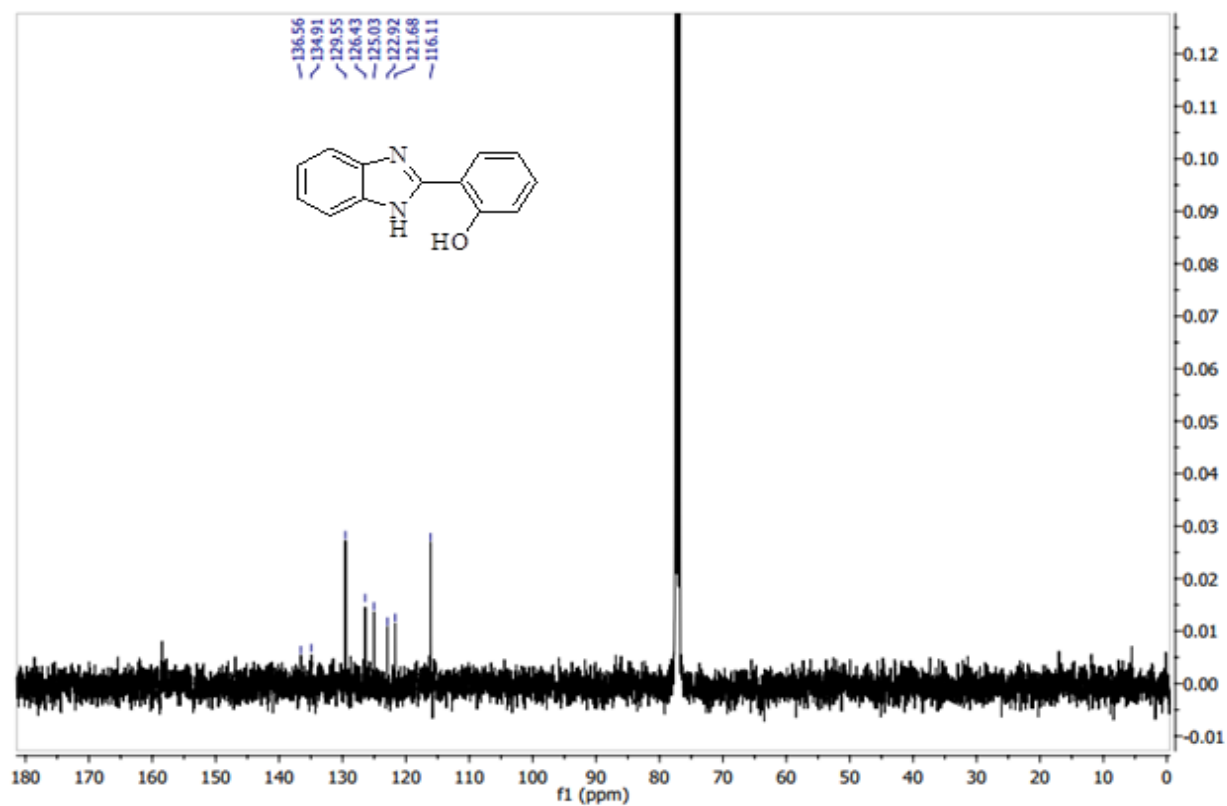
Peak Number (#)	Retention Time (min)	Area (.1*uV*sec)	Element %	Component
1	0.767	409480	10.202	Nitrogen
2	1.058	5483198	57.188	Carbon
3	3.667	863737	3.256	Hydrogen
		6756415	70.646	

20a. 2-(2-Hydroxyphenyl)-1H-benzimidazole



Mol. Wt.: 209.2



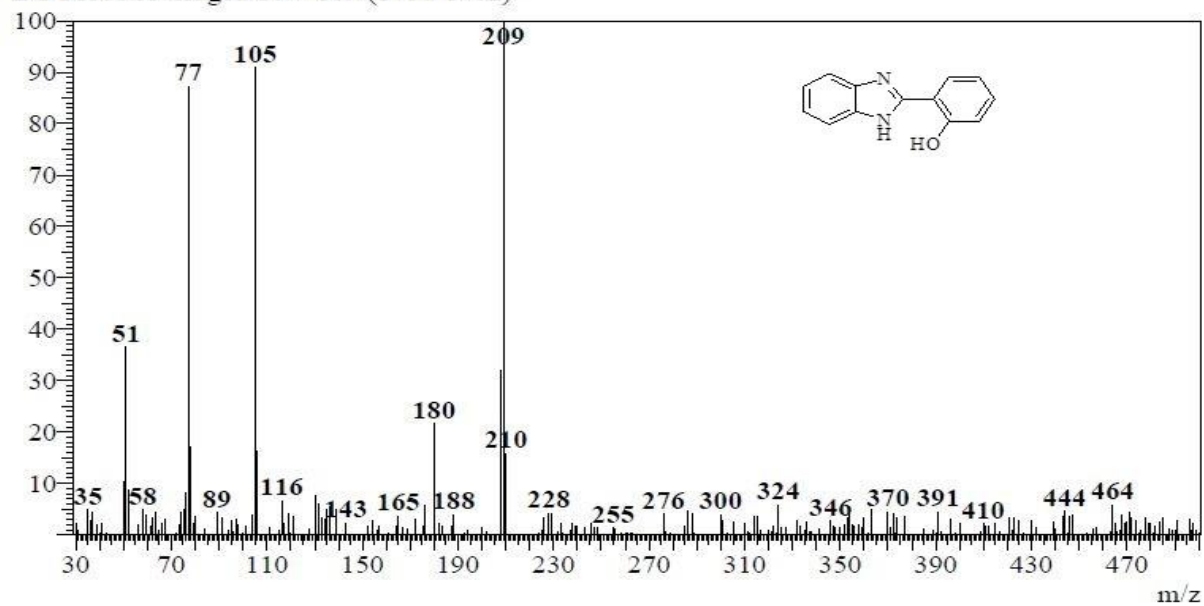


Line#:2 R.Time:16.0(Scan#:1677)

MassPeaks:222

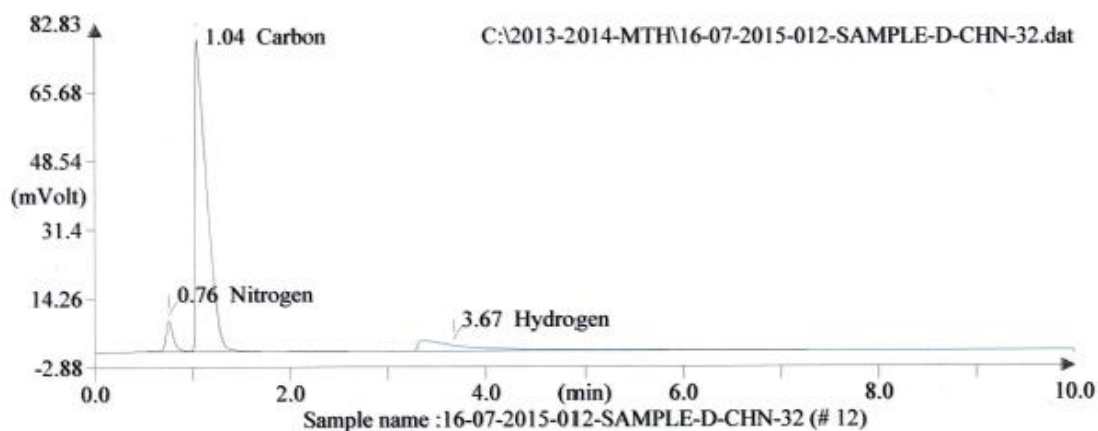
RawMode:Averaged 16.0-16.0(1676-1678) BasePeak:209(1062)

BG Mode:Averaged 16.7-16.7(1760-1762)



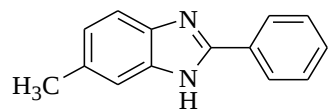
SAIF-IIT BOMBAY

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\16-07-2014-CHNS.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 07/16/2015 17:51
 Printed: 07-17-2015 11:06
 Elemental Analyser method:
 Sampler method:
 Sample ID: 16-07-2015-012-SAMPLE-D-CHN-32 (# 12)
 Analysis type: UnkNown
 Chromatogram filename: 16-07-2015-012-SAMPLE-D-CHN-32.dat
 Calibration method: K Factors
 Sample weight: 2.054
 Protein factor: 6.25

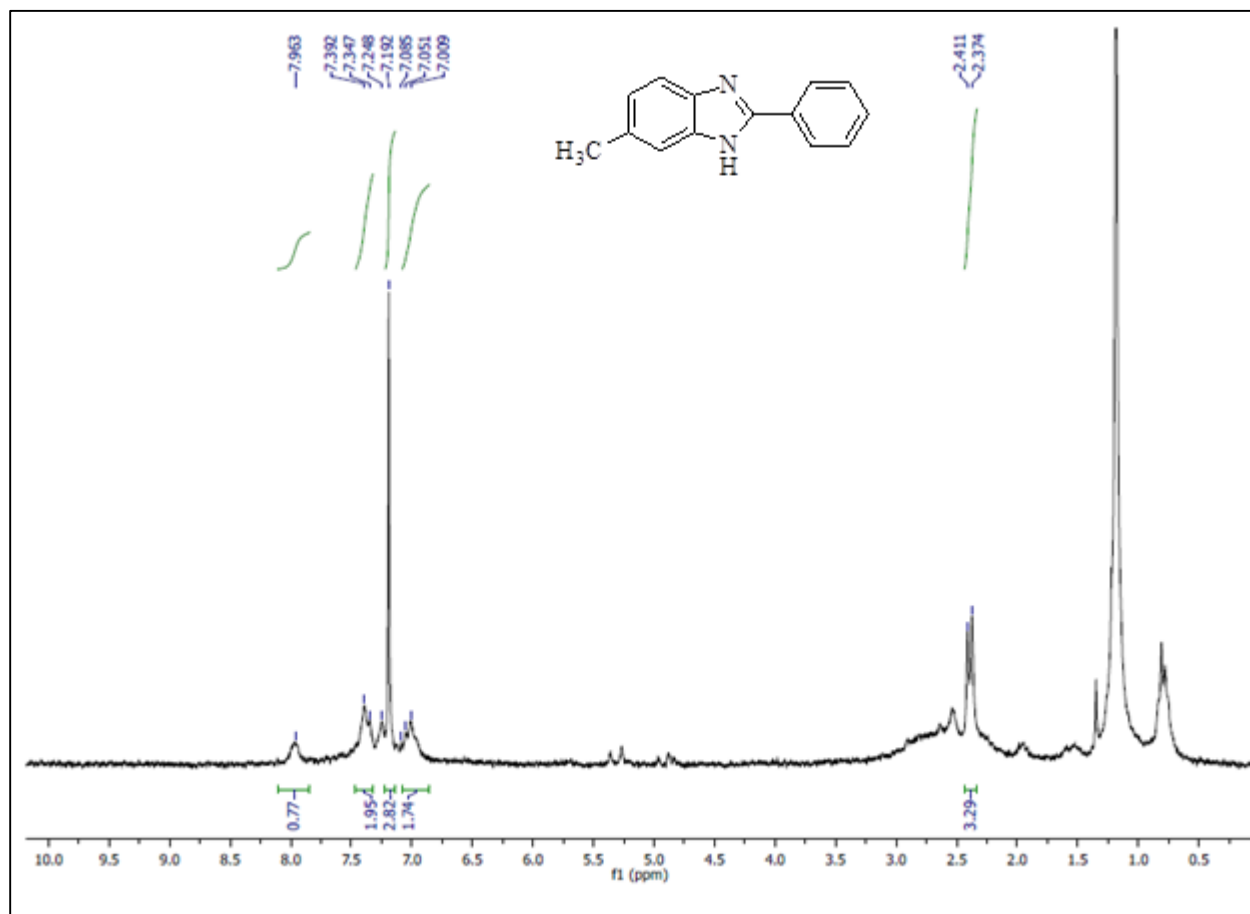


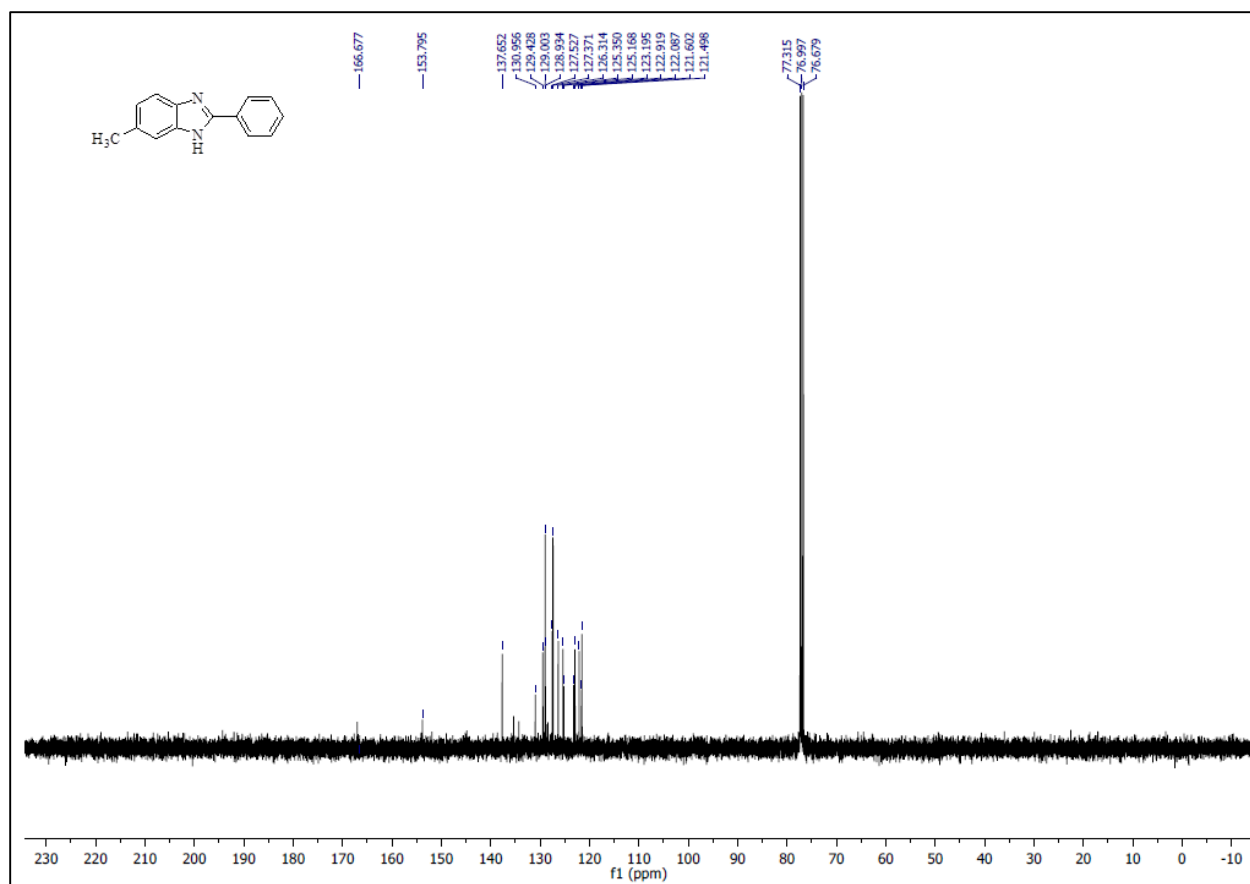
Peak Number (#)	Retention Time (min)	Area (.1*uV*sec)	Element %	Component
1	0.758	479690	13.259	Nitrogen
2	1.042	6365052	74.289	Carbon
3	3.667	920197	4.240	Hydrogen
		7764939	91.788	

21a. 6-Methyl-2-phenyl-1H-benzimidazole



Mol. Wt.: 209



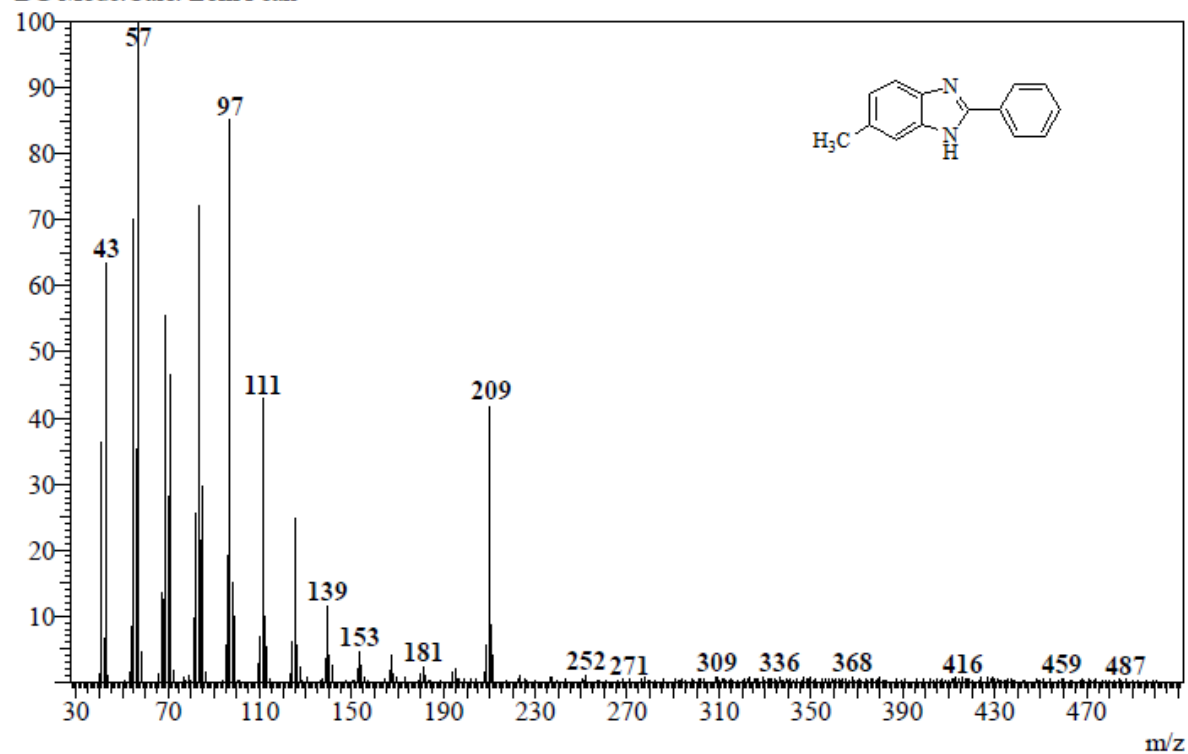


Line#:1 R.Time:21.7(Scan#:2368)

MassPeaks:312

RawMode:Averaged 21.7-21.7(2367-2369) BasePeak:57(4504)

BG Mode:Calc. from Peak



References

- 1 T. Bhattacharya, B. Majumdar, D. Dey and T. K. Sarma, *RSC Adv.*, 2014, **4**, 45831–45837
- 2 K. S. Sadek, R. A. Mekheimer, A. A. Hamid, F. Elnahas and M. H. Elnagdi, *Molecules*, 2012, **17**, 6011–6019
- 3 X. Shi, J. Guo, J. Liu, M. Ye, and Q. Xu, *Chem. Eur. J.*, 2015, **21**, 9988 – 9993
- 4 C. Mukhopadhyay and A. Datta, *J. Heterocycl. Chem.*, 2009, **46**, 91–95.
- 5 M. R. Marri, S. Peraka, A. K. Macharla, N. Mamede, S. Kodumuri, N. Nama, *Tetrahedron. Lett.*, 2014, **55**, 6520–6525
- 6 V. S. Padalkar, V. D. Gupta, K. R. Phatangare, V. S. Patil, P. G. Umape, N. Sekar, *Green Chem. Lett. and Reviews*, 2012, **5**, 139–145
- 7 T. B. Nguyen, L. Ermolenko, W. A. Dean and A. Al-Mourabit, *Org. Lett.*, 2012, **23**, 5948–5951