

Supporting Information

Synthesis of benzothiazoles using iron-anchored polysulfonamide modified layered double oxide/sodium alginate nanocomposite

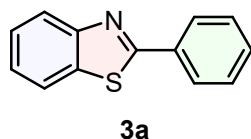
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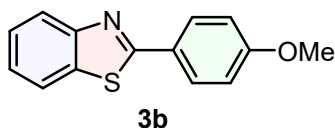
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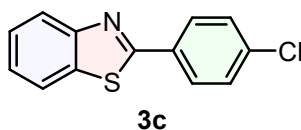
Corresponding Author: rgvaghei@yahoo.com & ghorbani@basu.ac.ir



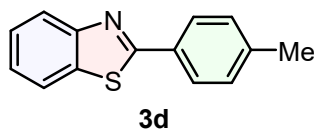
2-Phenylbenzo[d]thiazole (3a): Mp: 112-114 °C; IR (KBr): ν (cm⁻¹) 3004, 2958, 2836, 1673, 1497, 1367, 1169, 112, 1003, 775, 576; ¹H NMR (300 MHz, DMSO-*d*₆): δ_{H} (ppm) 7.77 (m, CH aromatic, 5H), 8.62 (d, *J*=3 Hz, CH aromatic, 2H), 9.29 (d, *J*=5 Hz, CH aromatic, 2H); ¹³C NMR (75 MHz, DMSO-*d*₆): δ_{C} (ppm) 112.33, 116.30, 122.48, 128.92, 129.86, 132.24, 134.72, 136.74, 144.46, 146.45, 150.39; MS *m/z* (%): 211.1.



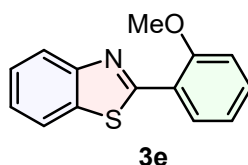
2-(4-Methoxy phenyl)benzo[d]thiazole (3b): Mp: 120-122 °C; IR (KBr): ν (cm⁻¹) 3059, 2957, 2836, 1679, 1611, 1579, 1513, 1434, 1346, 1284, 1255, 1170, 1100, 1030, 815, 790, 677, 612, 537; ¹H NMR (300 MHz, DMSO-*d*₆): δ_{H} (ppm) 3.76 (s, OCH₃, 3H), 6.86 (d, *J*=4 Hz, CH aromatic, 2H), 6.94 (d, *J*=5 Hz, CH aromatic, 2H), 7.13 (t, CH aromatic, 2H), 7.43 (d, *J*=4 Hz, CH aromatic, 2H); ¹³C NMR (75 MHz, DMSO-*d*₆): δ_{C} (ppm) 55.61, 112.31, 114.66, 116.32, 122.44, 128.83, 129.34, 132.10, 137.78, 141.17, 147.63, 150.34; MS *m/z* (%): 245.13.



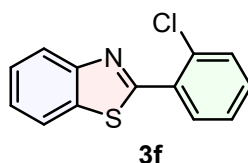
2-(4-Chlorophenyl)benzo[d]thiazole (3c): Mp: 108-110 °C; IR (KBr): ν (cm⁻¹) 3070, 2961, 1906, 1801, 1629, 1619, 1474, 1399, 1288, 1090, 972, 829, 731, 483; ¹H NMR (300 MHz, DMSO-*d*₆): δ_{H} (ppm) 7.48 (d, *J*=4 Hz, CH aromatic, 2H), 7.52 (d, *J*=5 Hz, CH aromatic, 2H), 7.60 - 8.18 (m, CH aromatic, 4H); ¹³C NMR (75 MHz, DMSO-*d*₆): δ_{C} (ppm) 112.03, 115.85, 117.31, 119.35, 129.80, 131.61, 134.34, 135.29, 139.83, 147.22, 149.41; MS *m/z* (%): 245.65.



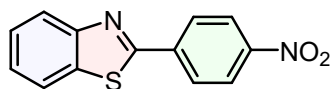
2-(4-Methyl Phenyl)benzo[d]thiazole (3d): Mp: 80-82 °C; IR (KBr): ν (cm⁻¹) 3058, 2900, 1674, 1646, 1509, 1403, 1202, 1102, 1034, 781, 694, 614, 550; ¹H NMR (300 MHz, DMSO-*d*₆): δ_{H} (ppm) 2.47 (s, CH₃, 3H), 7.34 - 7.36 (m, CH aromatic, 2H) 7.60 (dd, *J*=5, 3.8 Hz, CH aromatic, 2H), 7.70 (dd, *J*=6 Hz, CH aromatic, 2H), 7.82 (dd, *J*=4, 2.8 Hz, CH aromatic, 2H), ¹³C NMR (75 MHz, DMSO-*d*₆): δ_{C} (ppm) 123.42, 126.21, 127.68, 128.93, 130.40, 130.67, 131.13, 147.73, 153.41, 162.52, 167.94; MS *m/z* (%): 225.63.



2-(2-Methoxy phenyl)benzo[d]thiazole (3e): Mp: 118-120 °C; IR (KBr): ν (cm⁻¹) 3056, 3020, 1673, 1498, 1402, 1365, 1221, 1207, 1166, 1099, 802, 695, 613, 550; ¹H NMR (300 MHz, DMSO-*d*₆): δ_{H} (ppm) 3.65 (s, OCH₃, 3H), 7.17 (d, *J*=3 Hz, CH aromatic, 2H), 7.33 (t, *J*=5 Hz, CH aromatic, 2H), 7.97-8.42 (m, CH aromatic, 4H); ¹³C NMR (75 MHz, DMSO-*d*₆): δ_{C} (ppm) 67.86, 123.15, 123.83, 125.05, 126.31, 127.36, 129.75, 130.91, 131.83, 135.60, 138.46, 154.01, 165.31, 167.23; MS *m/z* (%): 244.1.

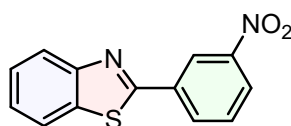


2-(2-Chlorophenyl)benzo[d]thiazole (3f): Mp: 80-82 °C; IR (KBr): ν (cm⁻¹) 3405, 2998, 2834, 1607, 1582, 1513, 1248, 1177, 1028; ¹H NMR (300 MHz, DMSO-*d*₆): δ_{H} (ppm) 7.12 (t, *J*=5 Hz, CH aromatic, 2H), 7.39 (d, *J*=4 Hz, CH aromatic, 2H), 7.54 (d, *J*=5 Hz, CH aromatic, 2H), 7.64 - 8.08 (m, CH aromatic, 2H); ¹³C NMR (75 MHz, DMSO-*d*₆): δ_{C} (ppm) 118.70, 120.89, 122.47, 127.73, 128.80, 129.37, 130.34, 130.82, 132.25, 135.52, 140.26, 143.67, 148.57; MS *m/z* (%): 245.65.



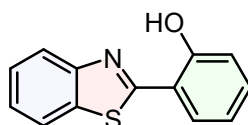
3g

2-(4-Nitrophenyl)benzo[d]thiazole (3g): Mp: 230-232 °C; IR (KBr): ν (cm⁻¹) 3409, 2961, 1725, 1669, 1522, 1344, 1250, 1158, 1046, 973, 904, 852, 730, 690; ¹H NMR (300 MHz, DMSO-*d*₆): δ_{H} (ppm) 7.63 (d, *J*=5 Hz, CH aromatic, 2H), 7.75 (d, *J*=6 Hz, CH aromatic, 2H), 7.35 (t, *J*=5 Hz, CH aromatic, 2H), 9.03 (d, *J*=4 Hz, CH aromatic, 2H); ¹³C NMR (75 MHz, DMSO-*d*₆): δ_{C} (ppm) 122.33, 124.14, 128.85, 129.25, 130.79, 132.14, 133.42, 135.03, 137.47, 148.50, 150.25; MS *m/z* (%): 255.86.



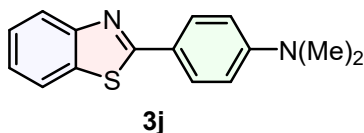
3h

2-(3-Nitrophenyl)benzo[d]thiazole (3h): Mp: 180-182 °C; IR (KBr): ν (cm⁻¹) 3369, 3100, 2889, 1666, 1606, 1522, 1345, 1221, 1171, 1112, 1035, 973, 852, 767, 687, 620; ¹H NMR (300 MHz, DMSO-*d*₆): δ_{H} (ppm) 7.54 (t, *J*=7 Hz, CH aromatic, 2H), 8.15 (d, *J*=5 Hz, CH aromatic, 2H), 8.24 (d, *J*=5 Hz, CH aromatic, 2H), 8.50 (d, *J*=5 Hz, CH aromatic, 1H), 8.84 (s, CH aromatic, 1H); ¹³C NMR (75 MHz, DMSO-*d*₆): δ_{C} (ppm) 112.11, 113.12, 117.94, 122.29, 129.25, 130.92, 132.13, 140.44, 143.88, 150.33, 157.06; MS *m/z* (%): 256.10.

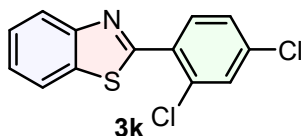


3i

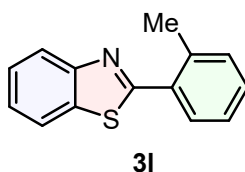
2-(2-Hydroxy phenyl)benzo[d]thiazole (3i): Mp: 125-127 °C; IR (KBr): ν (cm⁻¹) 3063, 1791, 1619, 1589, 1487, 1438, 1315, 1272, 1220, 1036, 973, 817, 742, 703, 465; ¹H NMR (300 MHz, DMSO-*d*₆): δ_{H} (ppm) 7.43 (d, *J*=5 Hz, CH aromatic, 1H), 7.52 (t, *J*=7 Hz, CH aromatic, 2H), 7.55 (d, *J*=5 Hz, CH aromatic, 1H), 8.05 - 8.13 (m, CH aromatic, 4H), 10.56 (s, OH, 1H); ¹³C NMR (75 MHz, DMSO-*d*₆): δ_{C} (ppm) 116.51, 120.66, 123.35, 126.36, 129.45, 132.96, 134.75, 139.49, 143.90, 147.03, 154.09, 160.13, 164.79; MS *m/z* (%): 227.1.



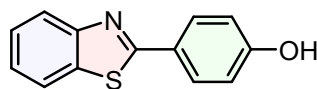
4-(Benzo[d]thiazol-2-yl)-*N,N*-dimethylaniline (3j): Mp: 260-262 °C; IR (KBr): ν (cm⁻¹) 3409, 2925, 1662, 1608, 1522, 1448, 1385, 1211, 1173, 1035, 817, 755, 692, 613; ¹H NMR (300 MHz, DMSO-*d*₆): δ_{H} (ppm) 3.04 (s, CH₃, 6H), 6.90 (d, *J*=5 Hz, CH aromatic, 2H), 7.33 (t, *J*=5 Hz, CH aromatic, 2H), 7.97 (d, *J*=5 Hz, CH aromatic, 2H); 8.05 (d, *J*=5.8 Hz, CH aromatic, 2H) ¹³C NMR (75 MHz, DMSO-*d*₆): δ_{C} (ppm) 52.27, 112.27, 116.21, 120.87, 122.45, 124.86, 128.89, 132.20, 136.50, 140.66, 144.75, 150.82; MS *m/z* (%): 255.



2-(2,4-Dichlorophenyl)benzo[d]thiazole (3k): Mp: 140-142 °C; IR (KBr): ν (cm⁻¹) 3389, 3063, 2786, 1673, 1584, 1549, 1482, 1425, 1377, 1316, 1259, 1106, 1061, 965, 859, 726, 692, 448; ¹H NMR (300 MHz, DMSO-*d*₆): δ_{H} (ppm) 7.51 (d, *J*=5 Hz, CH aromatic, 1H), 7.61 (d, *J*=4 Hz, CH aromatic, 1H), 7.81 (s, CH aromatic, 1H), 8.10-8.22 (m, CH aromatic, 4H); ¹³C NMR (75 MHz, DMSO-*d*₆): δ_{C} (ppm) 112.18, 116.15, 121.72, 124.14, 128.51, 128.85, 130.79, 132.14, 135.03, 137.47, 148.50, 150.25, 157.38; MS *m/z* (%): 280.03.

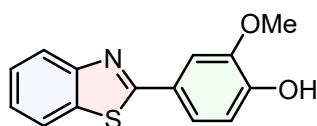


2-(*o*-tolyl)benzo[d]thiazole (3l): Mp: 60-62 °C; IR (KBr): ν (cm⁻¹) 3063, 1791, 1619, 1589, 1487, 1371, 1438, 1315, 1272, 1220, 1036, 973, 817, 742, 703, 465; ¹H NMR (300 MHz, DMSO-*d*₆): δ_{H} (ppm) 2.51 (s, CH₃, 3H), 7.34 (d, *J*=5 Hz, CH aromatic, 1H), 7.42 (t, *J*=6 Hz, CH aromatic, 2H), 7.54 (d, *J*=5 Hz, CH aromatic, 1H), 7.72-8.08 (m, CH aromatic, 4H); ¹³C NMR (75 MHz, DMSO-*d*₆): δ_{C} (ppm) 30.20, 112.27, 116.21, 120.87, 122.45, 124.86, 128.10, 128.89, 129.31, 132.20, 136.50, 140.66, 144.75, 147.80; MS *m/z* (%): 225.66.



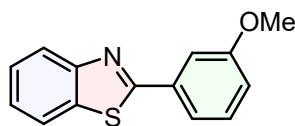
3m

2-(4-Hydroxyphenyl)benzo[d]thiazole (3m): Mp: 126-127 °C; IR (KBr): ν (cm⁻¹) 3389, 3063, 2786, 1673, 1584, 1482, 1315, 1316, 1259, 1106, 1061, 859, 826, 754, 726, 692, 480, 448; ¹H NMR (300 MHz, DMSO-*d*₆): δ_{H} (ppm) 7.36 (d, *J*=5 Hz, CH aromatic, 2H), 7.41 (d, *J*=5 Hz, CH aromatic, 2H), 7.57 (t, *J*=7 Hz, CH aromatic, 2H), 7.82 (d, *J*=6 Hz, CH aromatic, 2H), 8.95 (d, *J*=5 Hz, CH aromatic, 1H), 9.67 (s, OH, 1H); ¹³C NMR (75 MHz, DMSO-*d*₆): δ_{C} (ppm) 116.17, 122.46, 128.87, 129.29, 129.95, 132.18, 134.54, 138.65, 142.53, 145.50, 150.38; MS *m/z* (%): 227.5.



3n

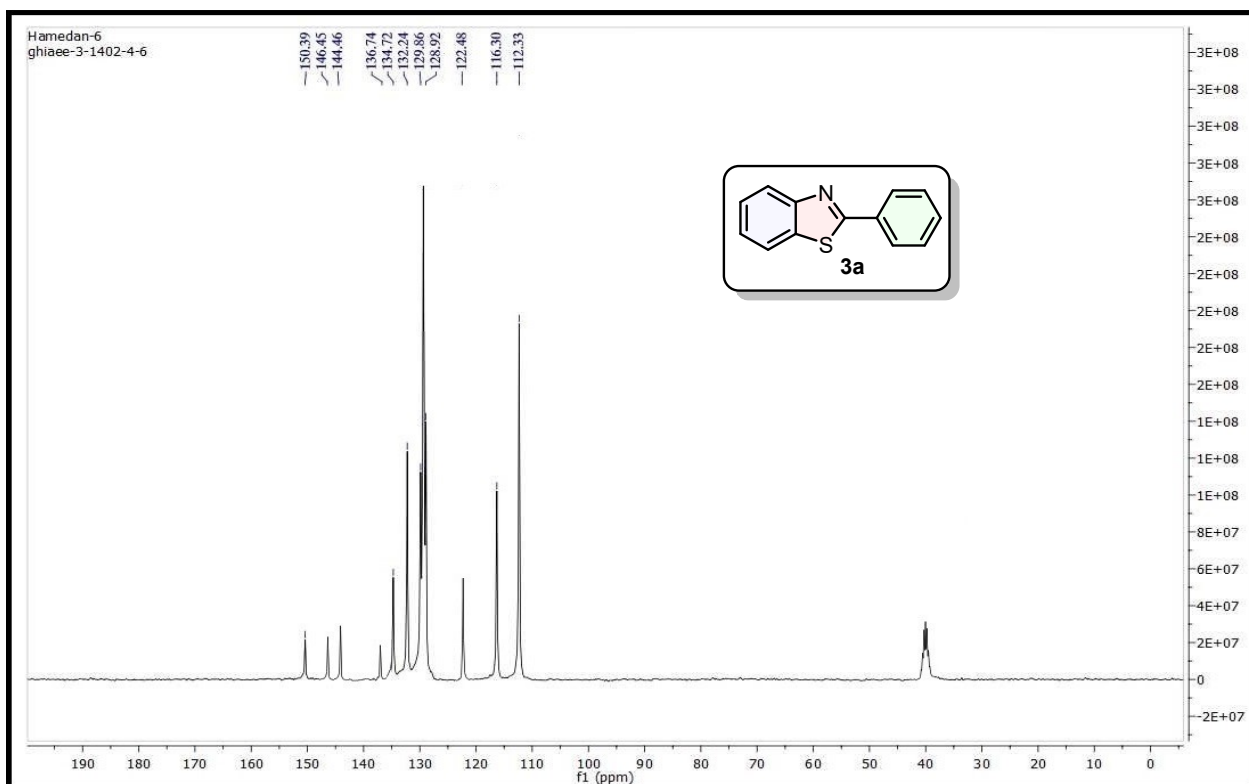
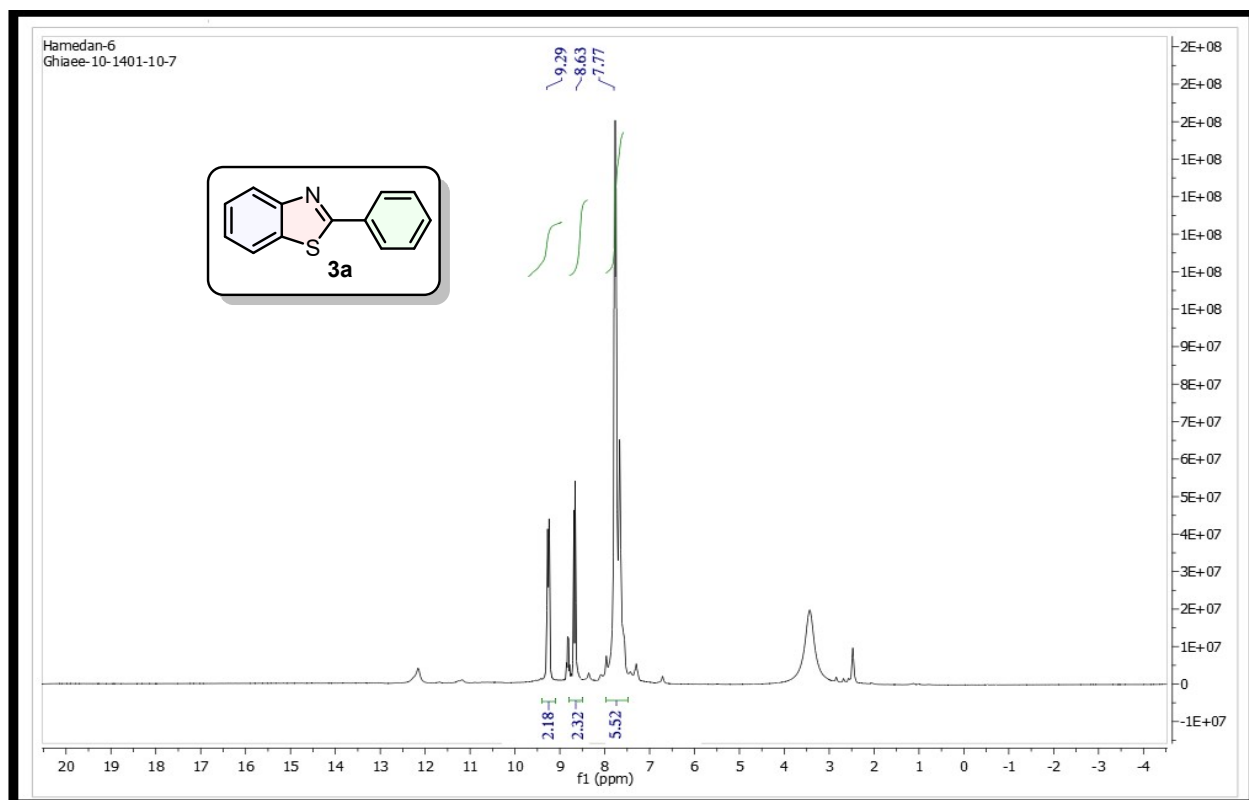
4-(Benzo[d]thiazol-2-yl)-2-methoxyphenol (3n): Mp: 164-165 °C; IR (KBr): ν (cm⁻¹) 3109, 2935, 1684, 1589, 1605, 1524, 1478, 1428, 1365, 1194, 1124, 756, 726, 617; ¹H NMR (300 MHz, DMSO-*d*₆): δ_{H} (ppm) 3.89 (s, OCH₃, 3H), 7.41 (d, *J*=6 Hz, CH aromatic, 1H), 7.50 (s, CH aromatic, 1H), 7.64 (d, *J*=6 Hz, CH aromatic, 1H), 7.71 (t, *J*=6 Hz, CH aromatic, 2H), 8.00 (d, *J*=5 Hz, CH aromatic, 1H), 8.06 (d, *J*=6 Hz, CH aromatic, 1H), 9.54 (s, OH, 1H); ¹³C NMR (75 MHz, DMSO-*d*₆): δ_{C} (ppm) 55.85, 110.54, 116.40, 121.25, 122.38, 124.12, 125.56, 126.90, 134.45, 148.00, 140.29, 153.79, 167.83; MS *m/z* (%): 257.31.



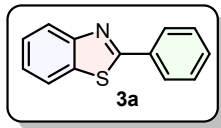
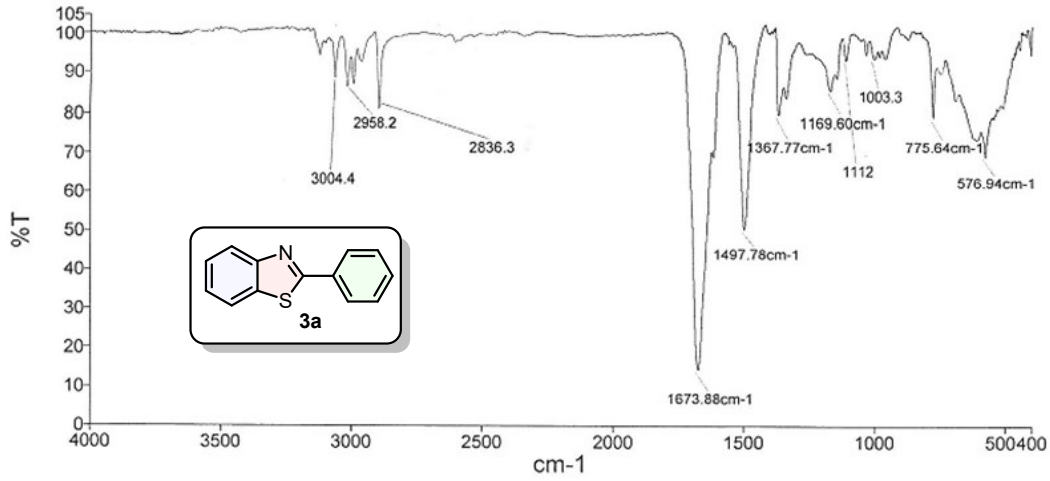
3o

2-(3-Methoxyphenyl)benzo[d]thiazole (3o): Mp: 164-165 °C; IR (KBr): ν (cm⁻¹) 3405, 2998, 2834, 1607, 1582, 1513, 1248, 1177, 1365, 1028; ¹H NMR (300 MHz, DMSO-*d*₆): δ_{H} (ppm) 3.76 (s, OCH₃, 3H), 7.58 (d, *J*=5 Hz, CH aromatic, 1H), 7.63 (t, *J*=6 Hz, CH aromatic, 1H), 7.89 (t, *J*=7 Hz, CH aromatic, 2H), 8.18 (d, *J*=5 Hz, CH aromatic, 1H), 8.24 (d, *J*=6 Hz, CH aromatic, 1H), 8.43 (d, *J*=5 Hz, CH aromatic, 1H), 8.64 (s, CH aromatic, 1H); ¹³C NMR (75 MHz, DMSO-*d*₆): δ_{C} (ppm) 35.18, 110.03, 112.19, 114.34, 116.17, 118.82, 122.39, 125.18, 128.80, 129.18, 132.08, 137.04, 143.38, 150.28; MS *m/z* (%): 245.132.

¹H and ¹³C NMR, IR and Mass Spectra of the Benzothiazoles 2-Phenylbenzo[d]thiazole (3a)

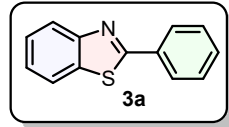
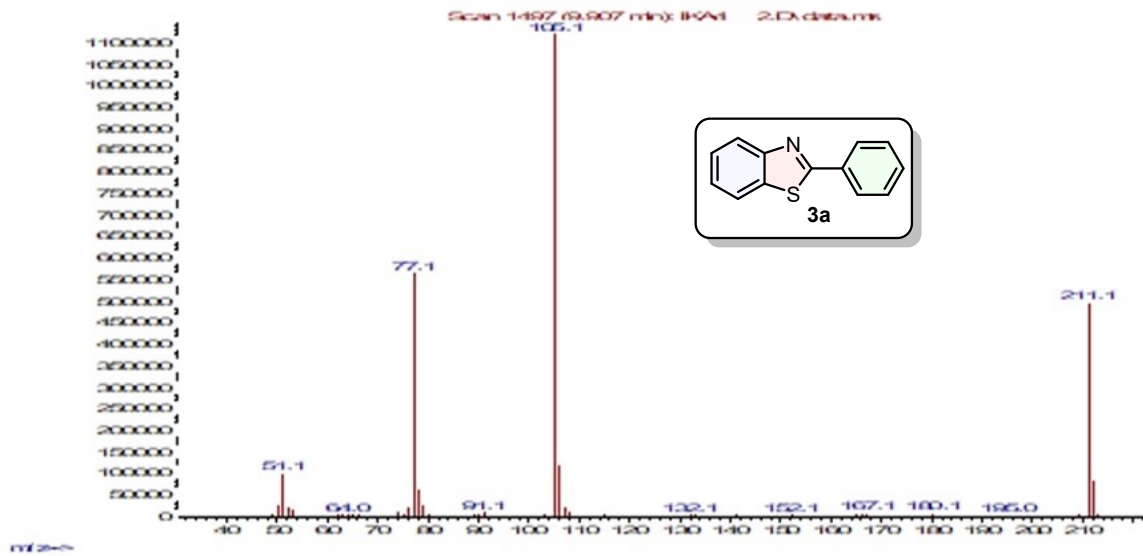


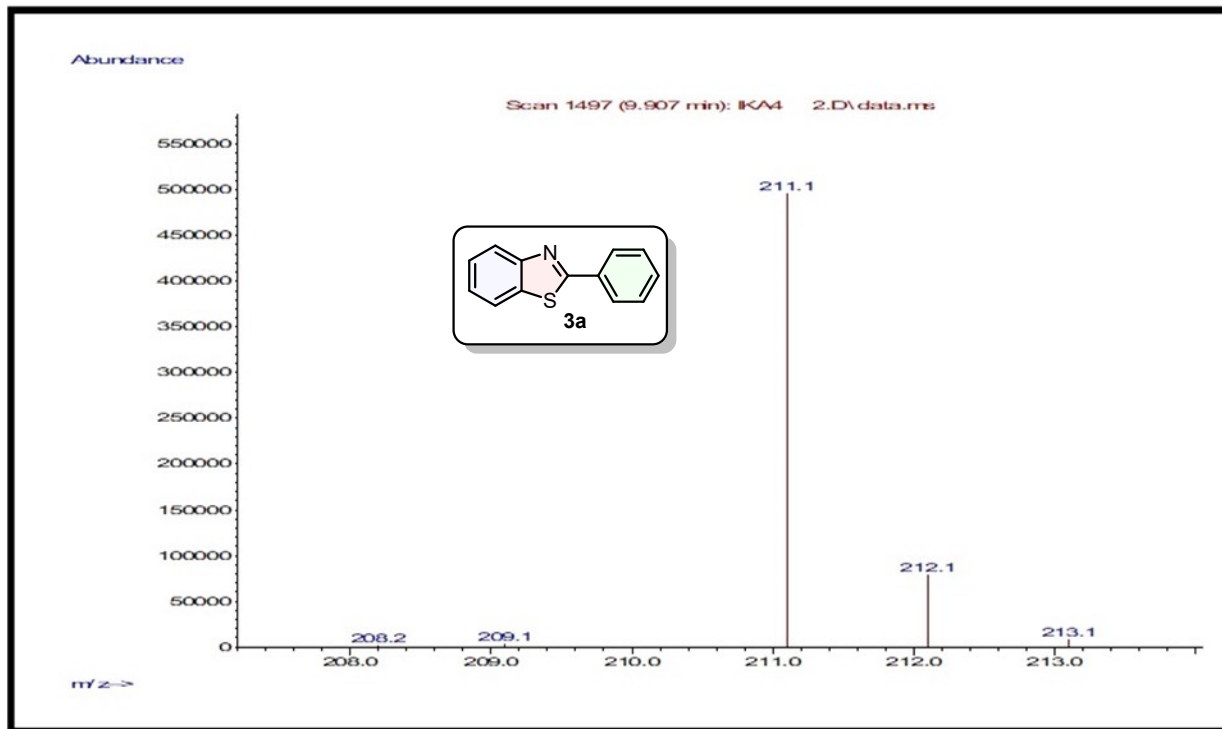
Analyst M-Khorshidi
Date Tuesday, September 13, 2022 11:57 AM



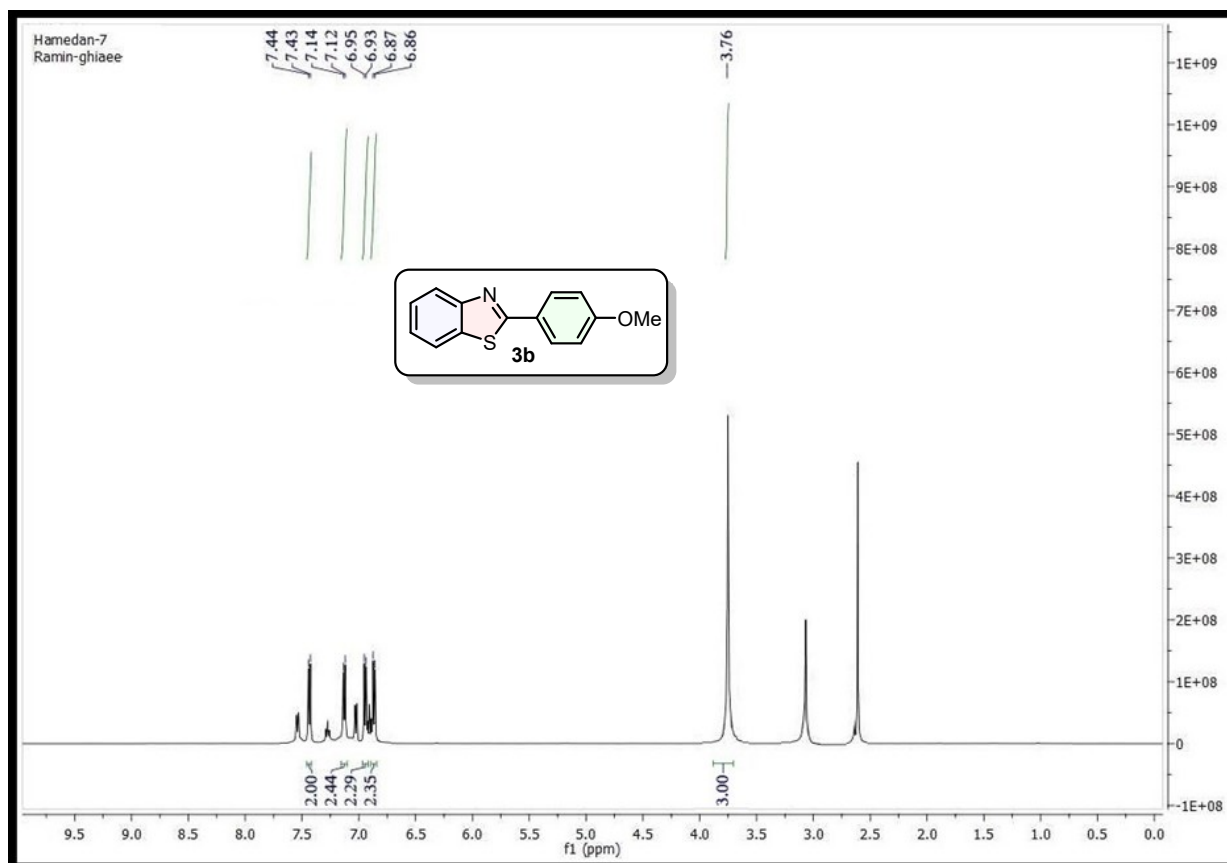
4.2 Sample 049 By 10 Date Tuesday, September 13 2022

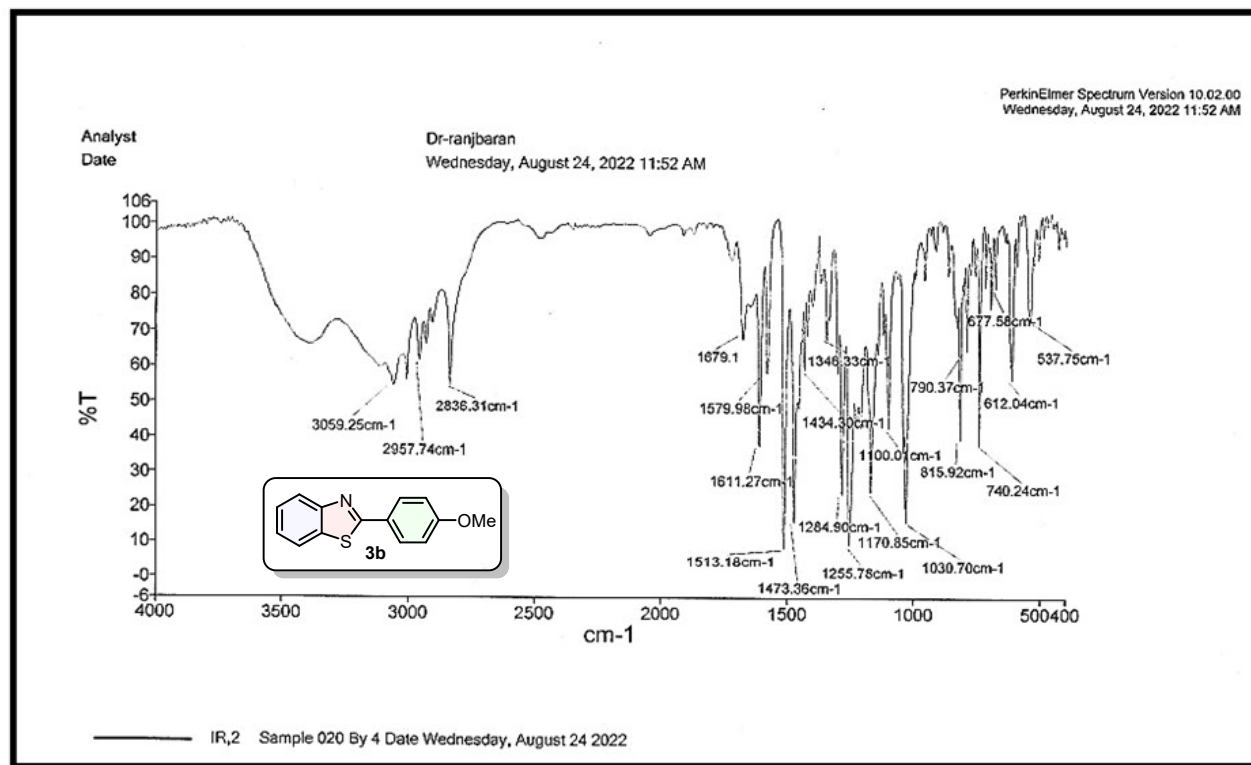
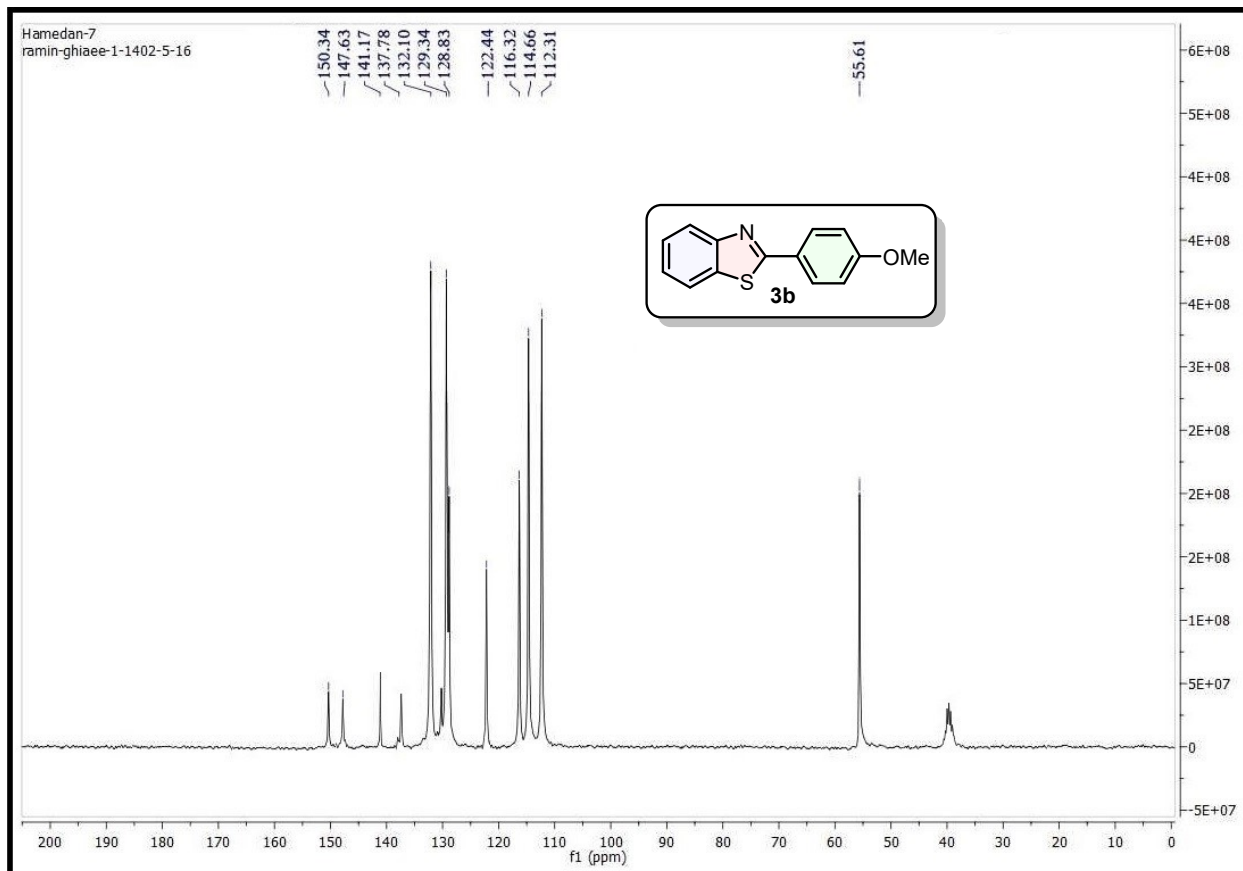
Abundance

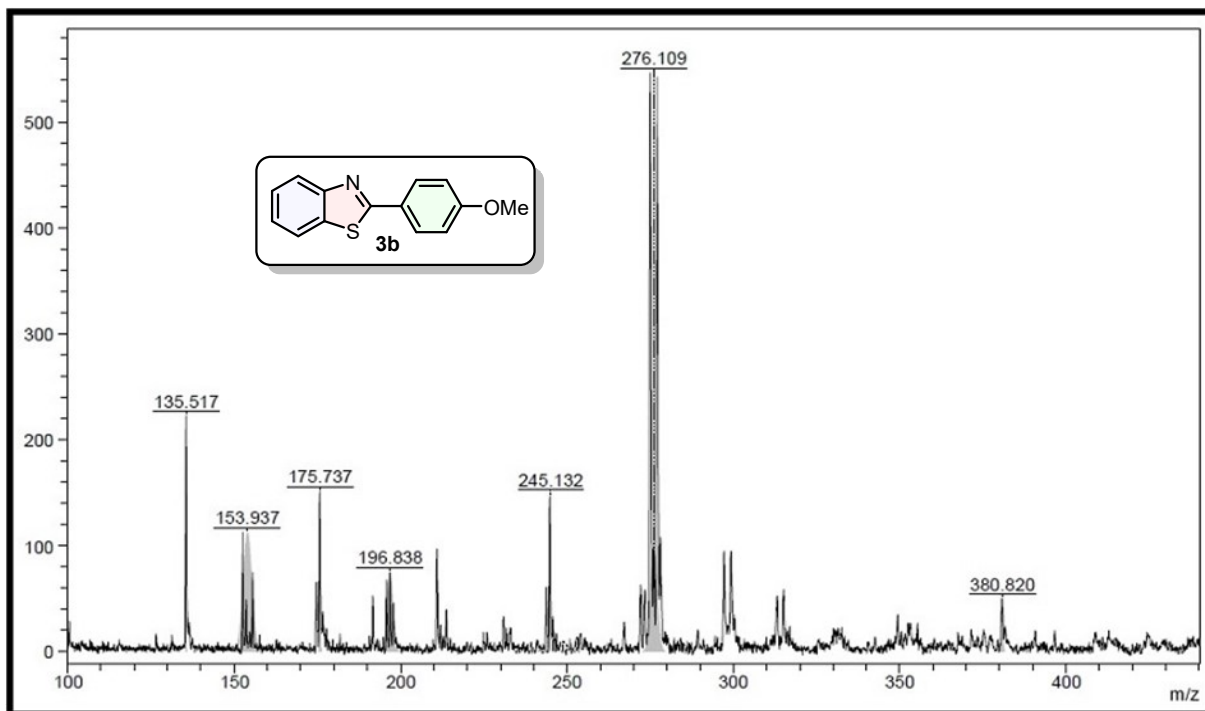




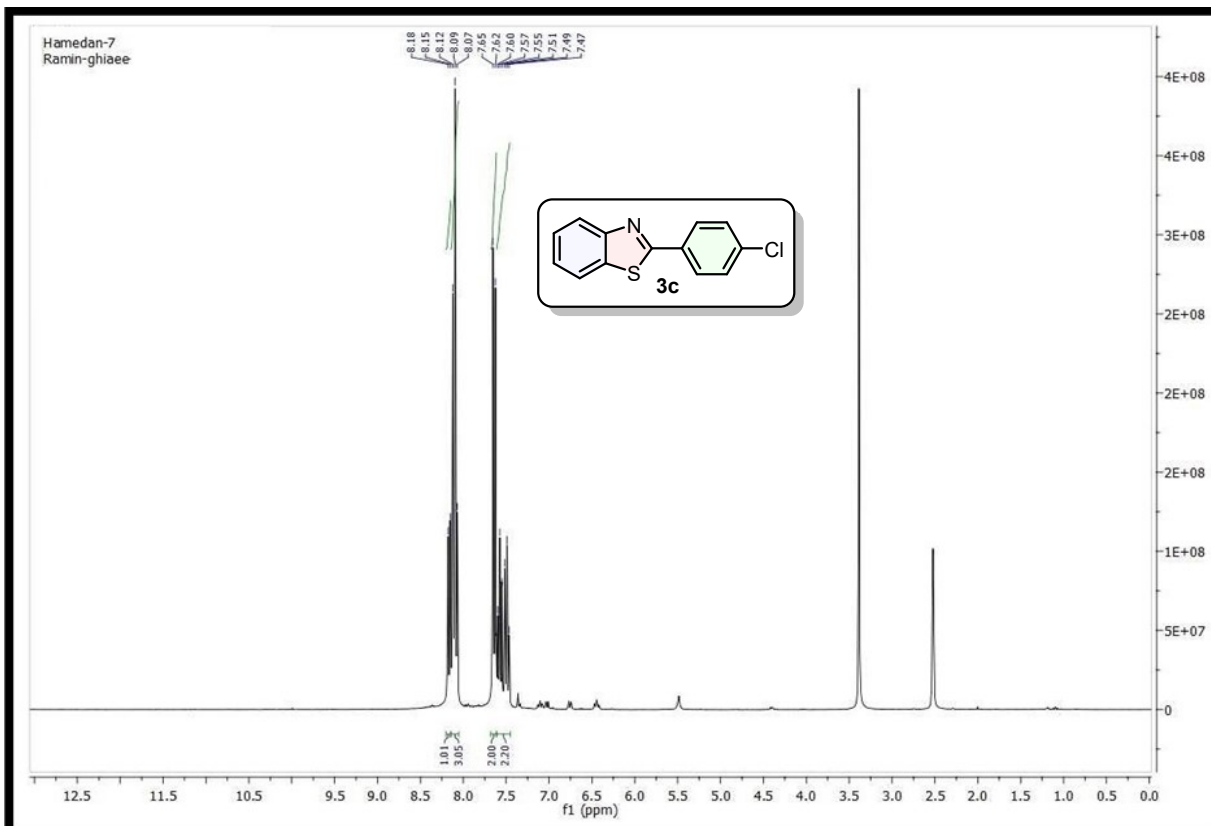
2-(4-Methoxy phenyl)benzo[d]thiazole (3b)

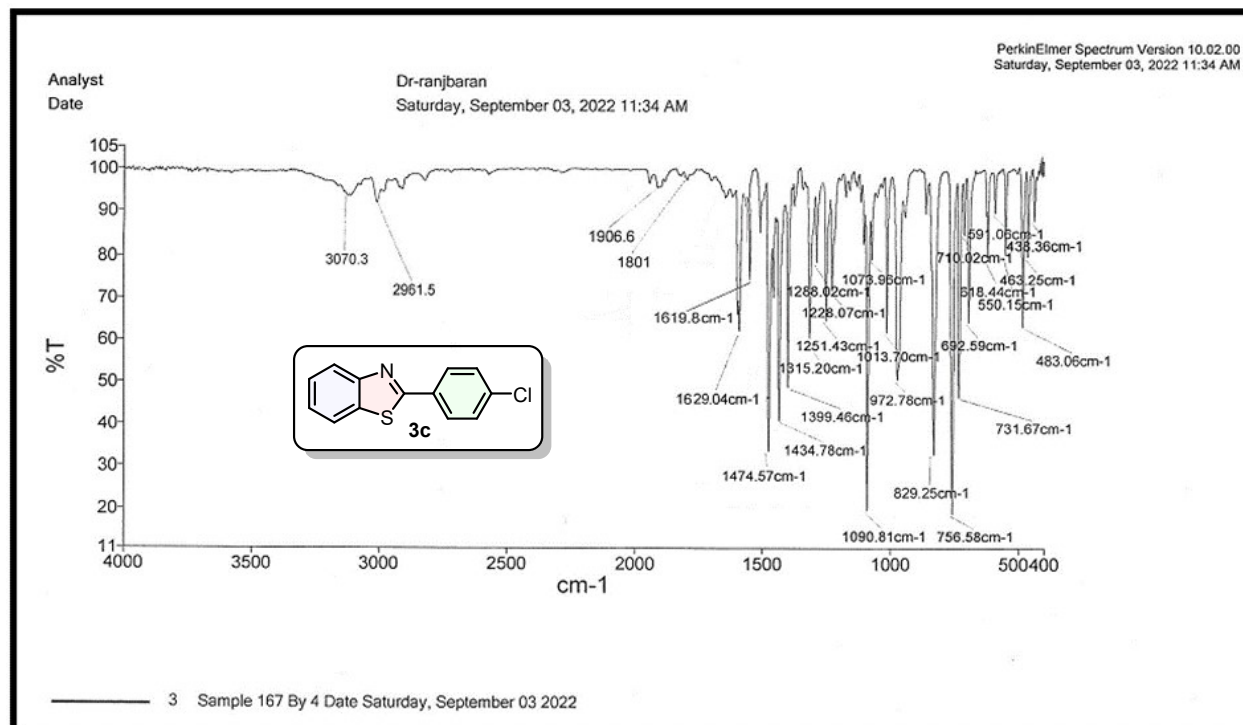
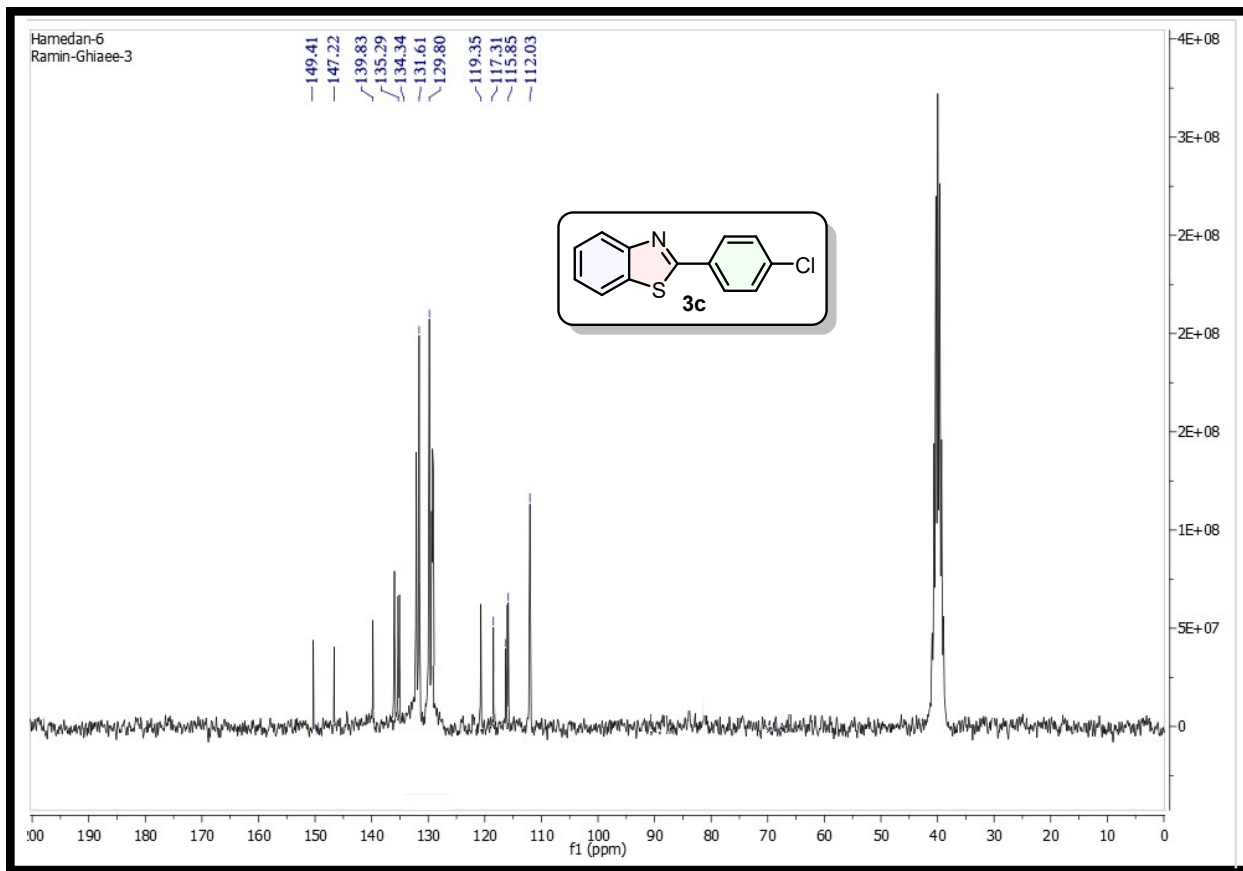


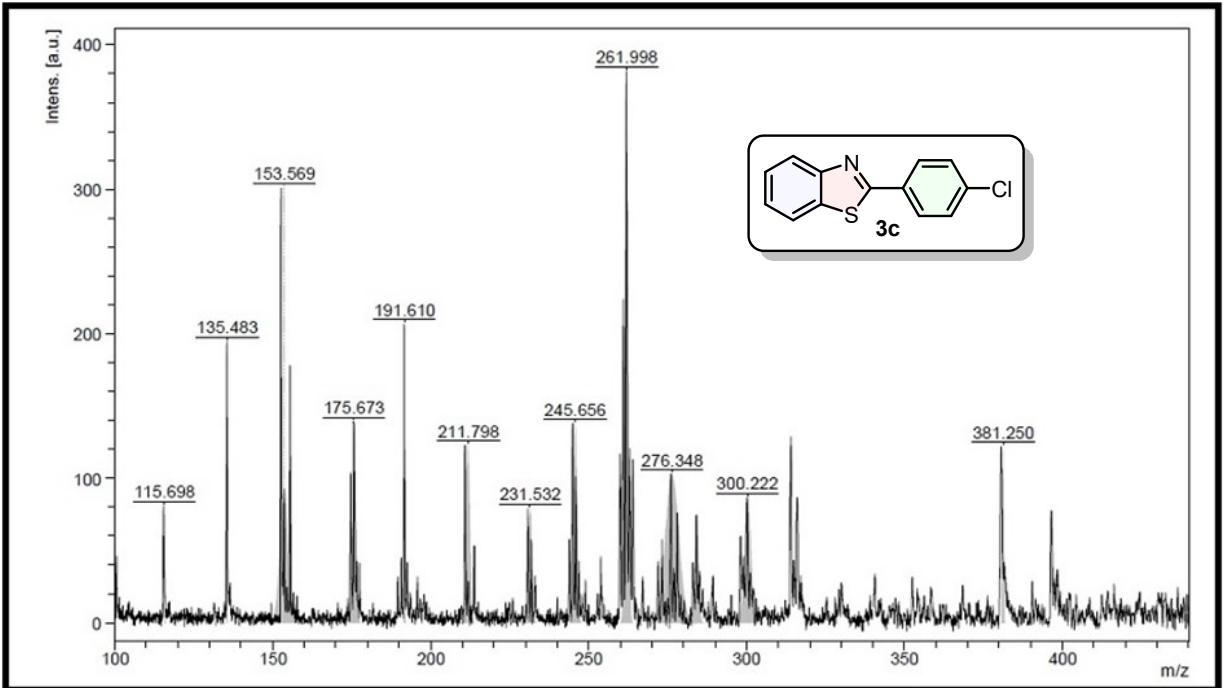




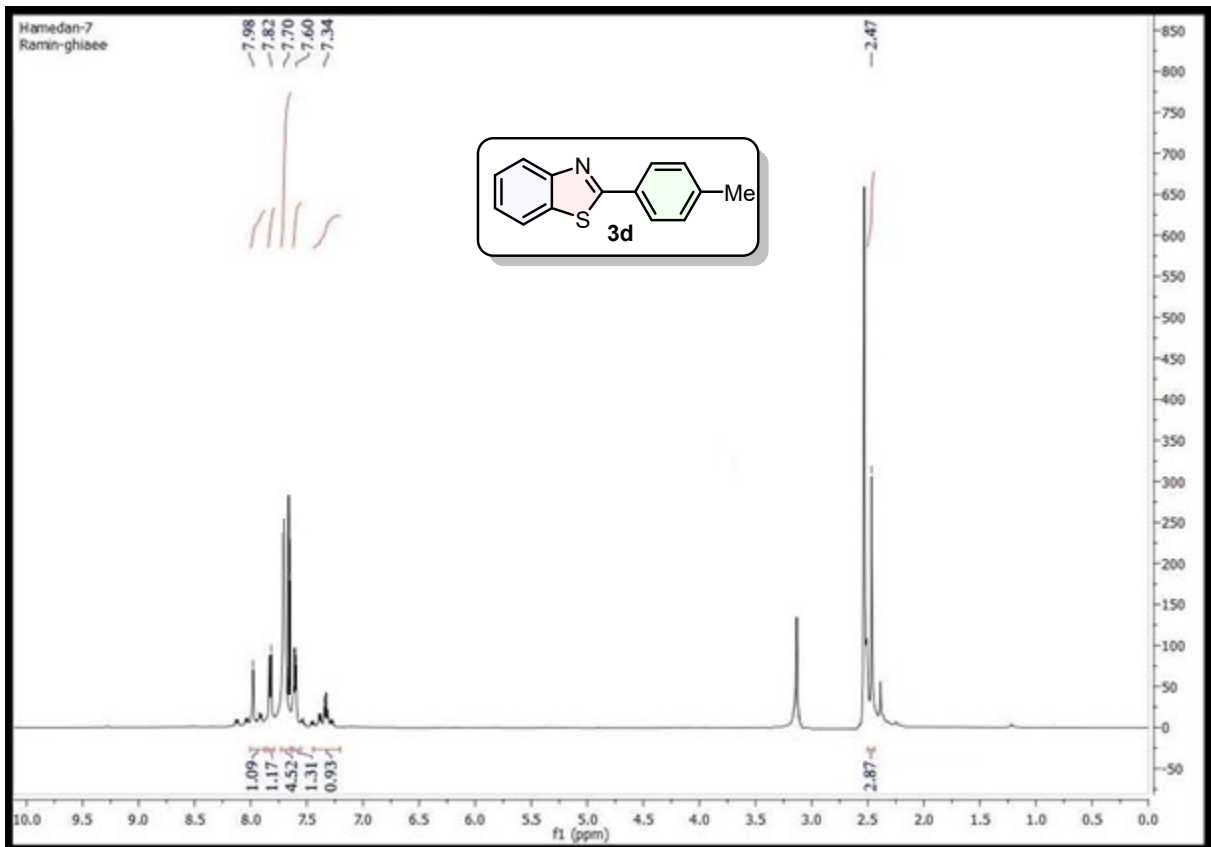
2-(4-Chlorophenyl)benzo[d]thiazole (3c)

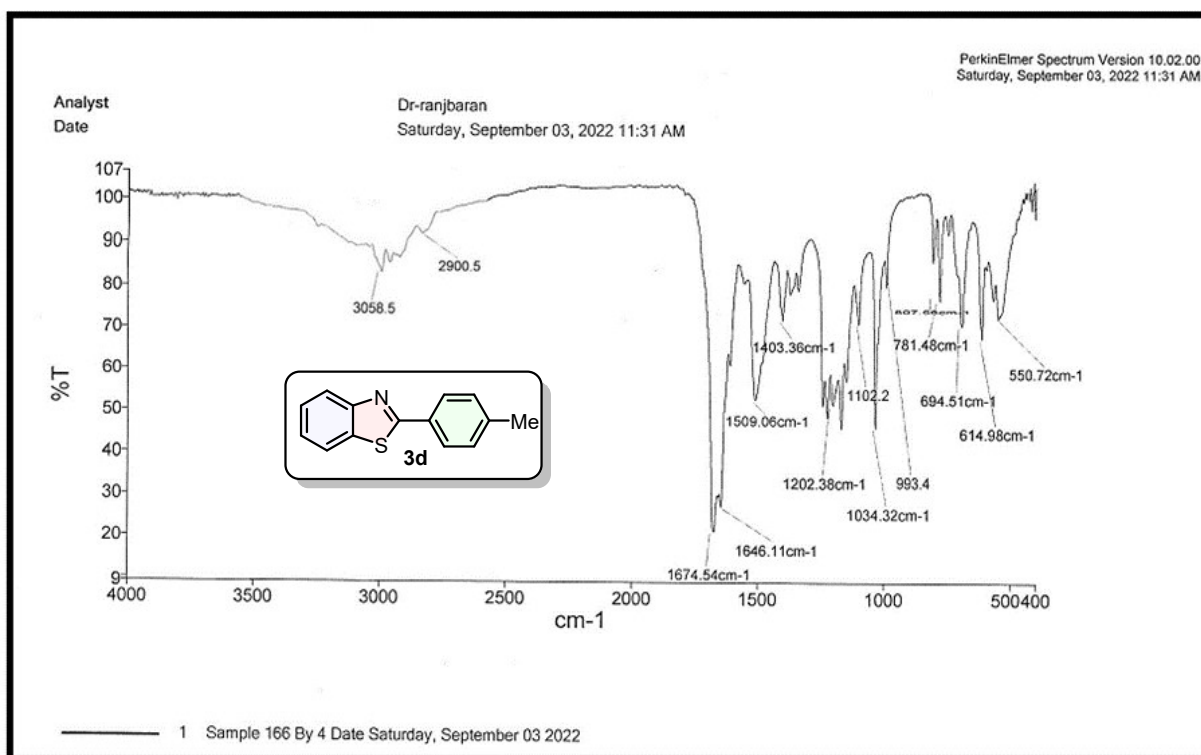
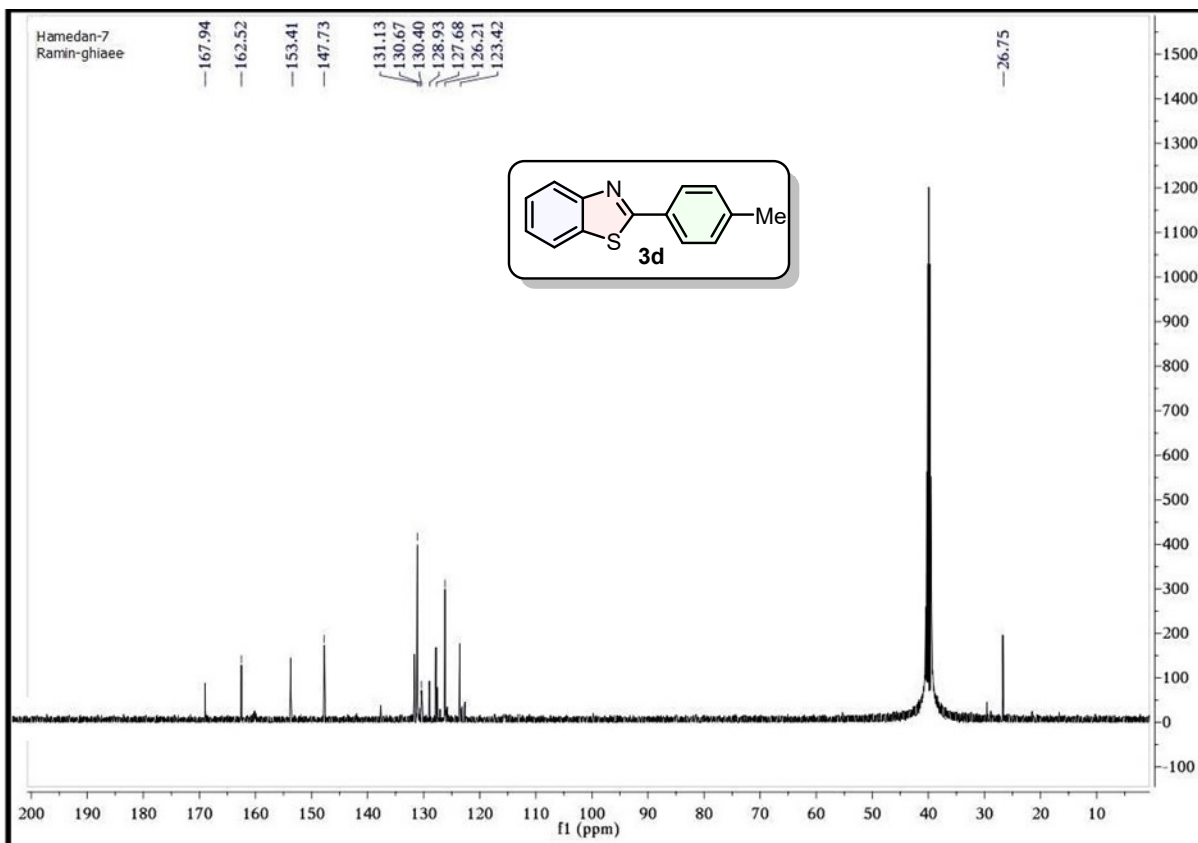


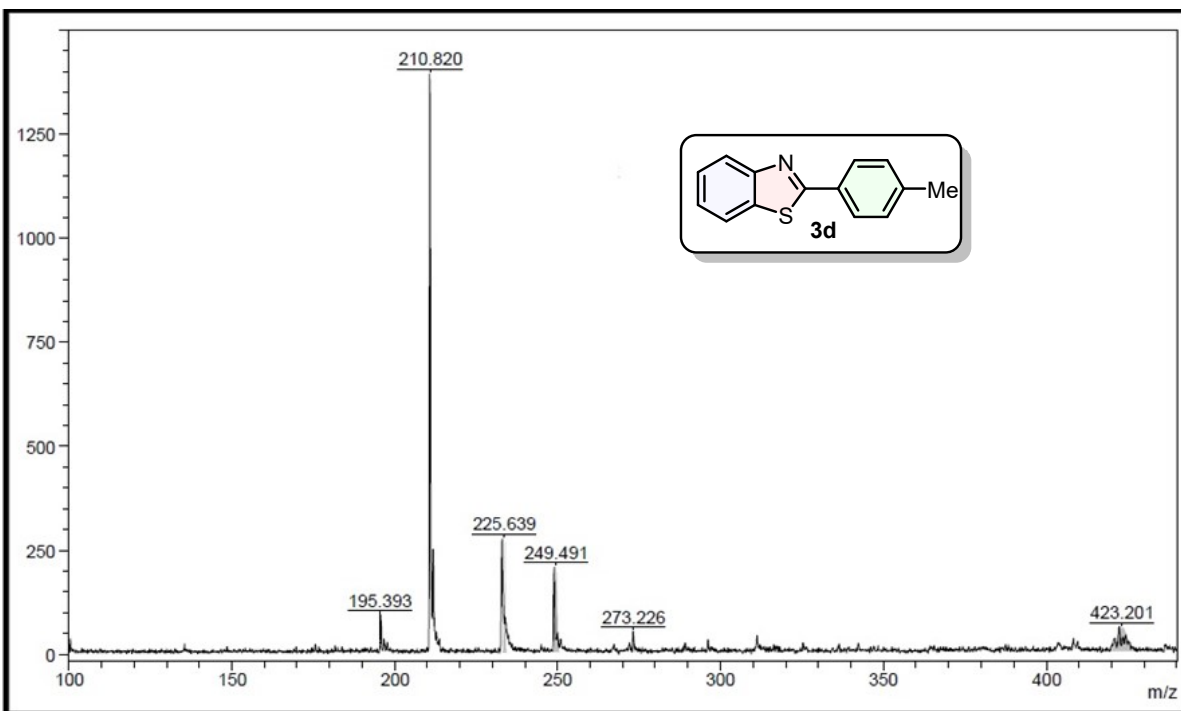




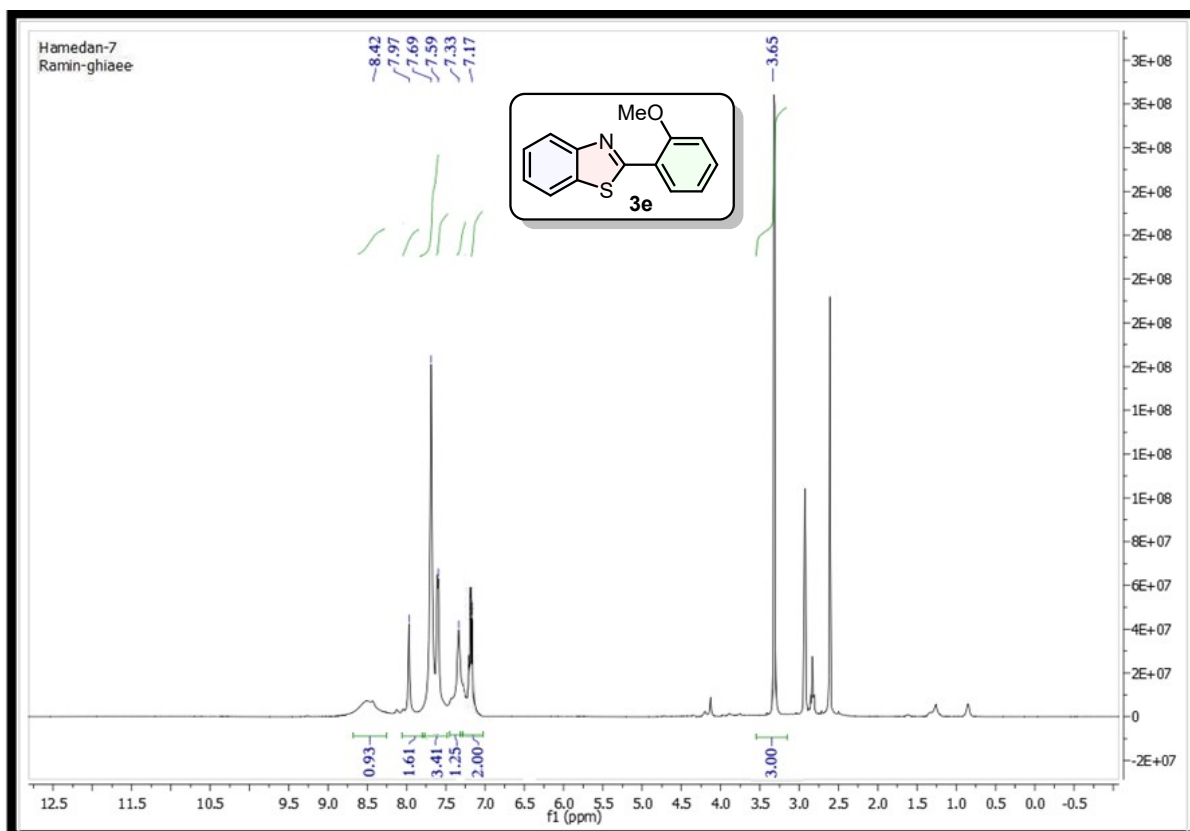
2-(4-Methyl Phenyl)benzo[d]thiazole (3d)

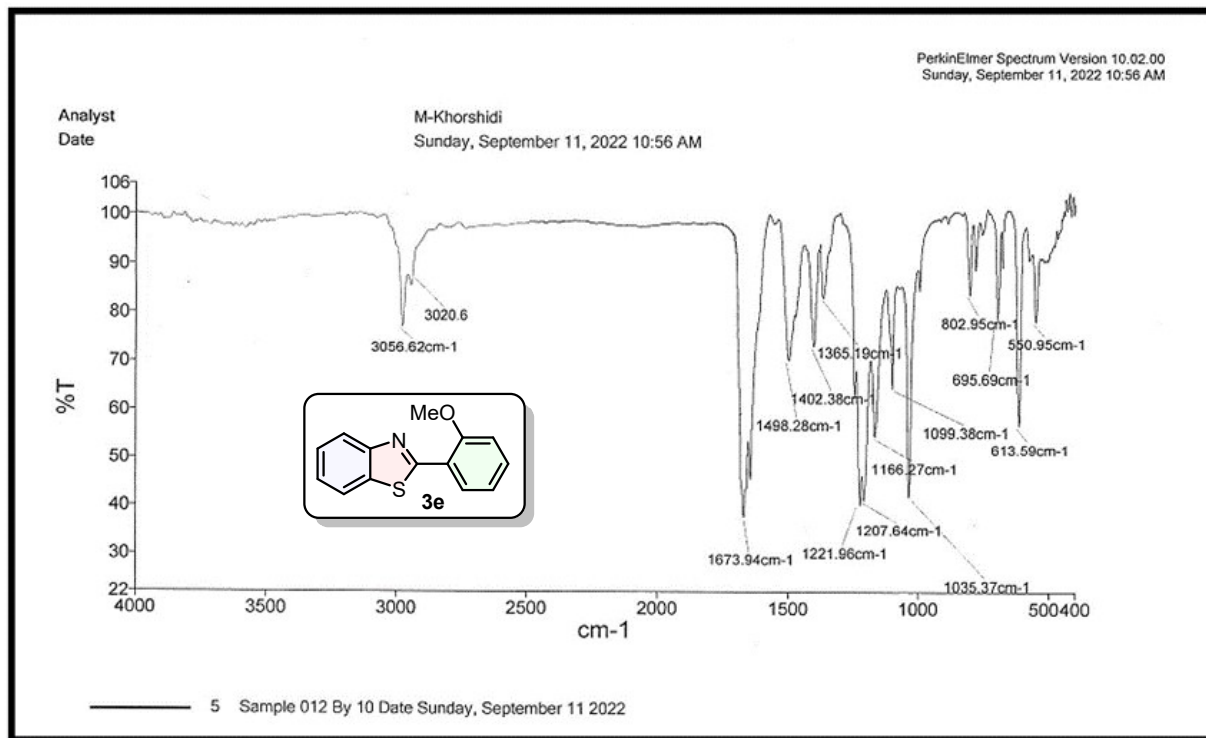
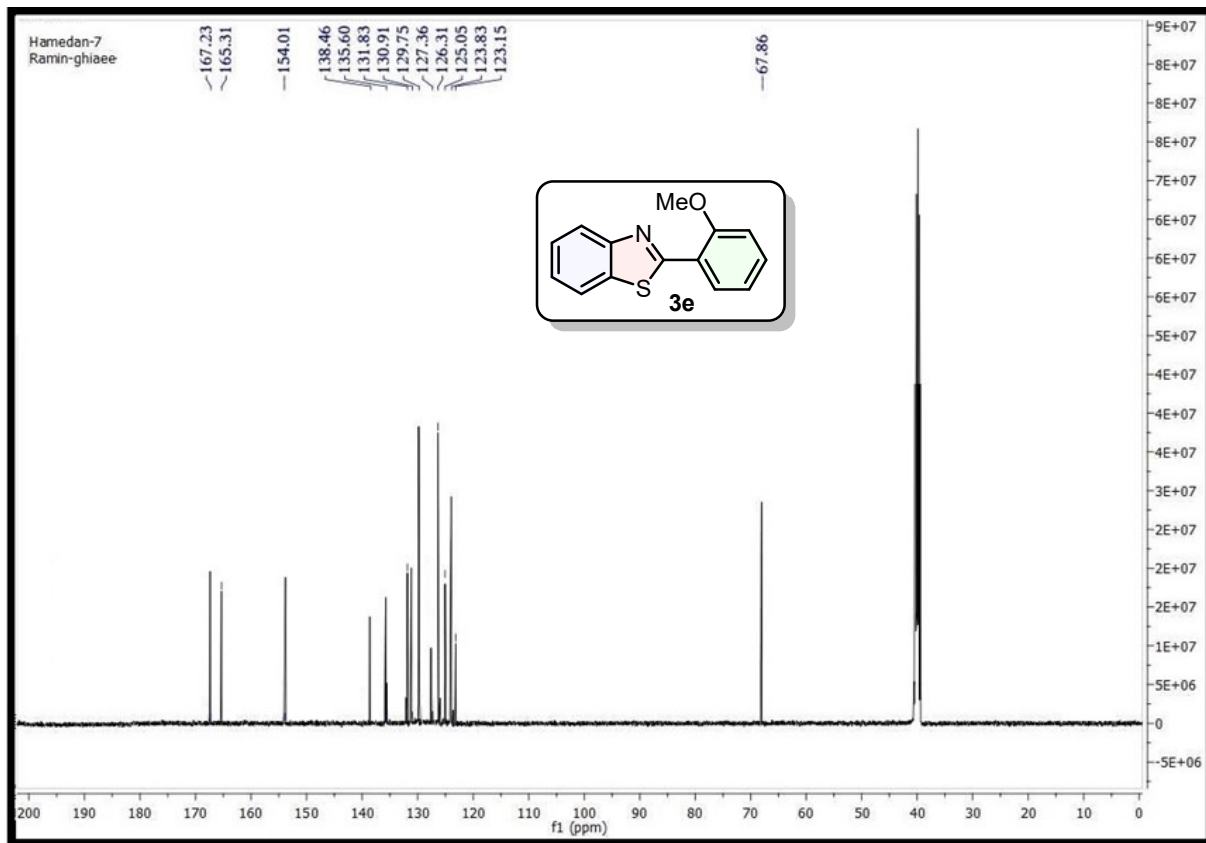


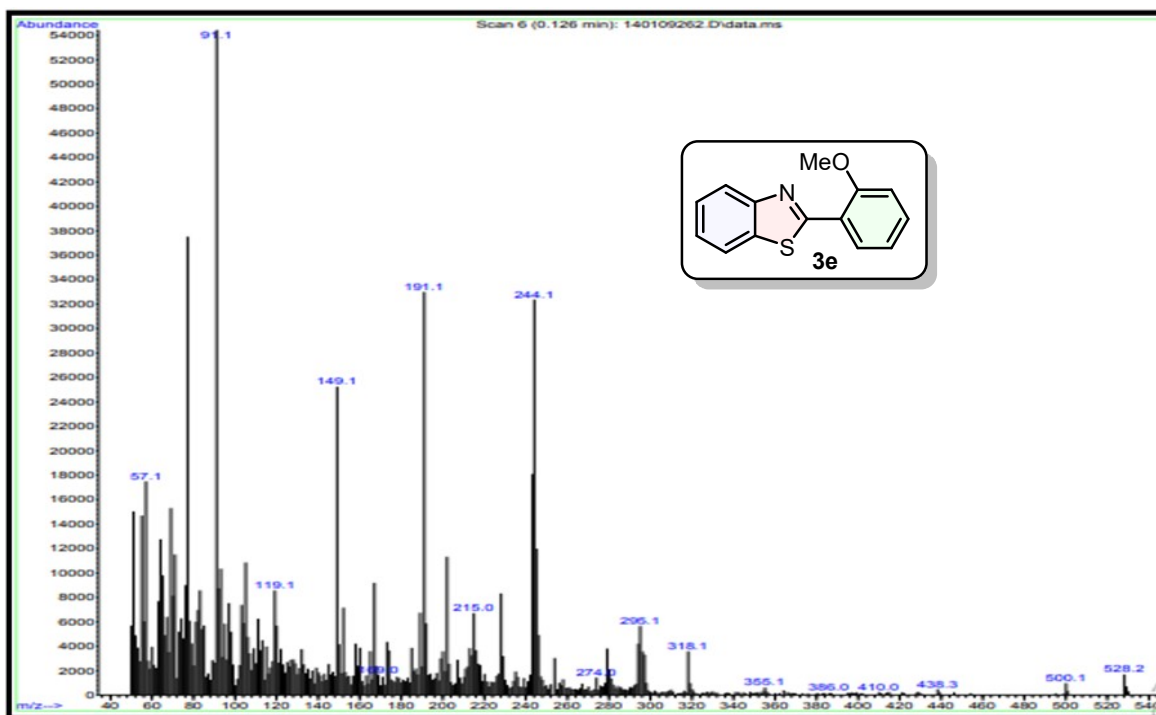




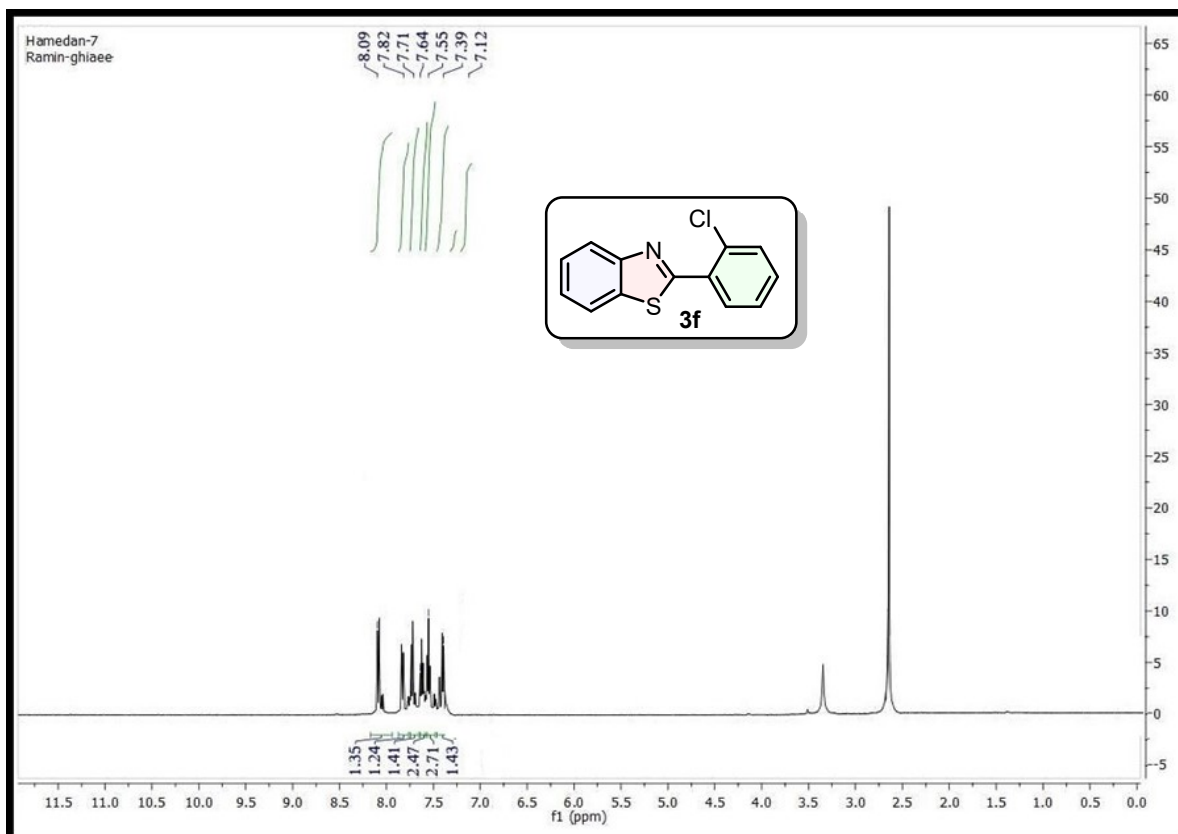
2-(2-Methoxy phenyl)benzo[d]thiazole (3e)

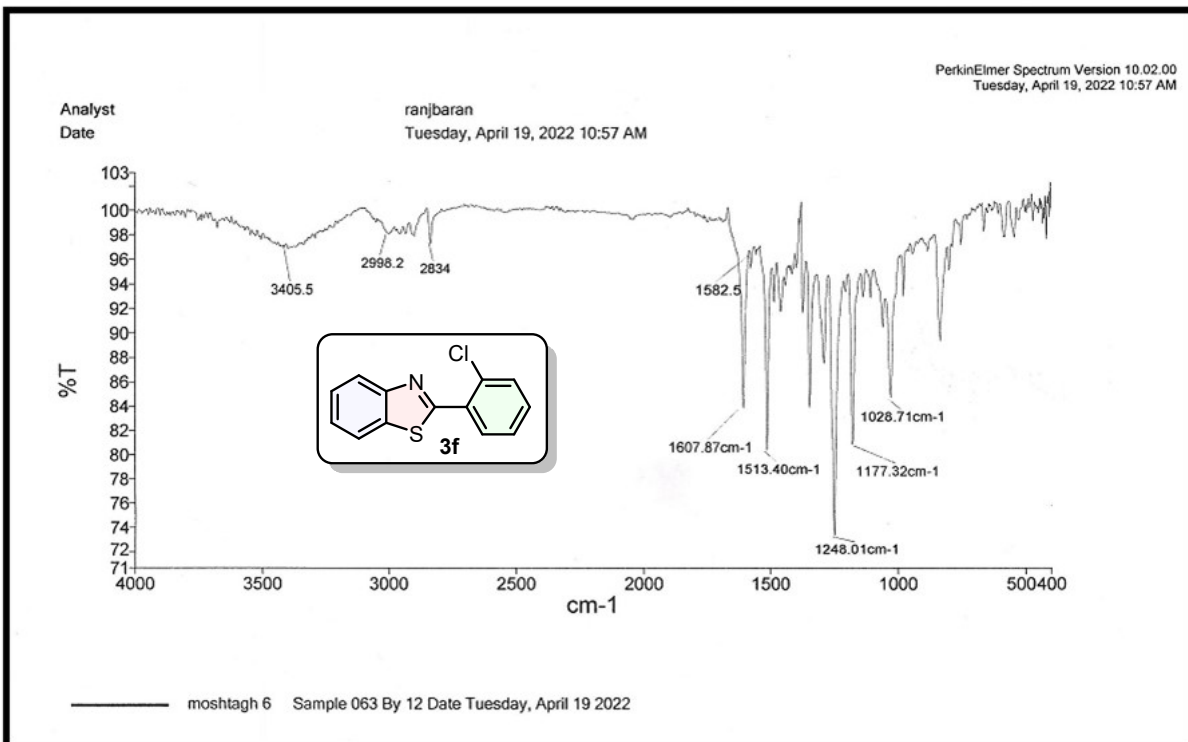
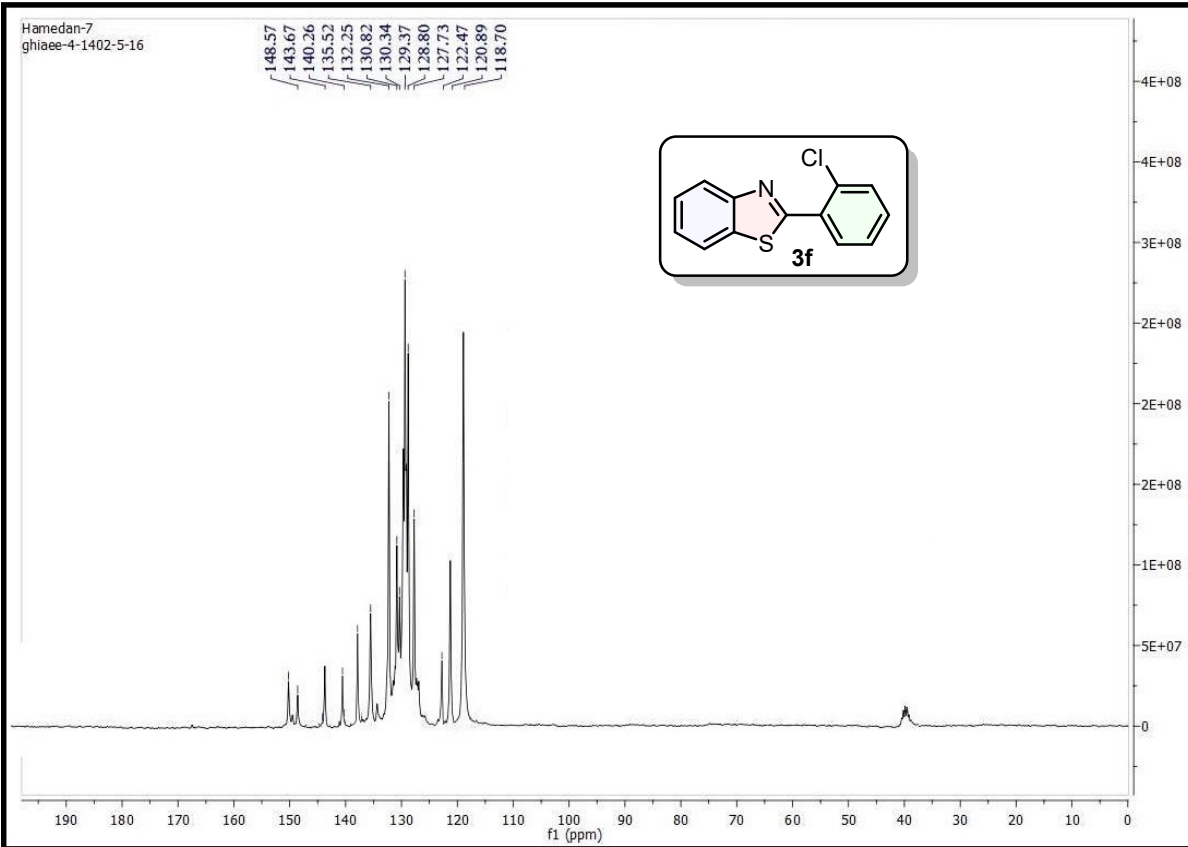


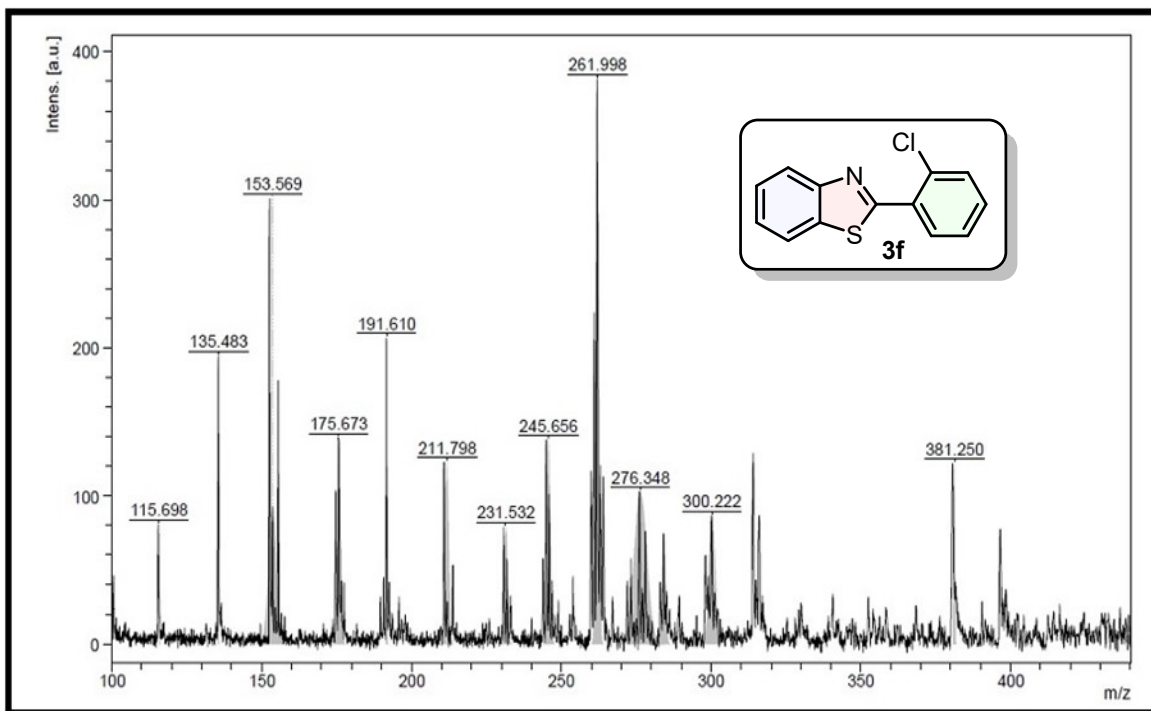




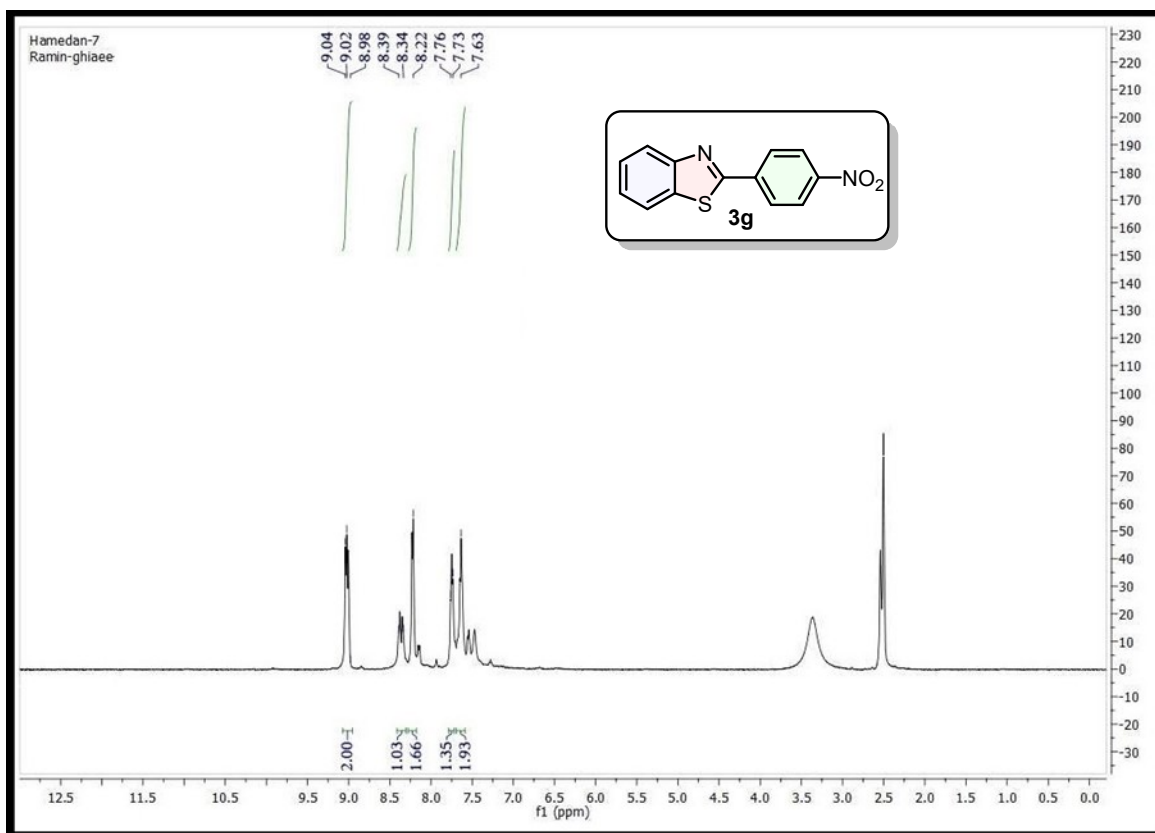
2-(2-Chlorophenyl)benzo[d]thiazole (**3f**)

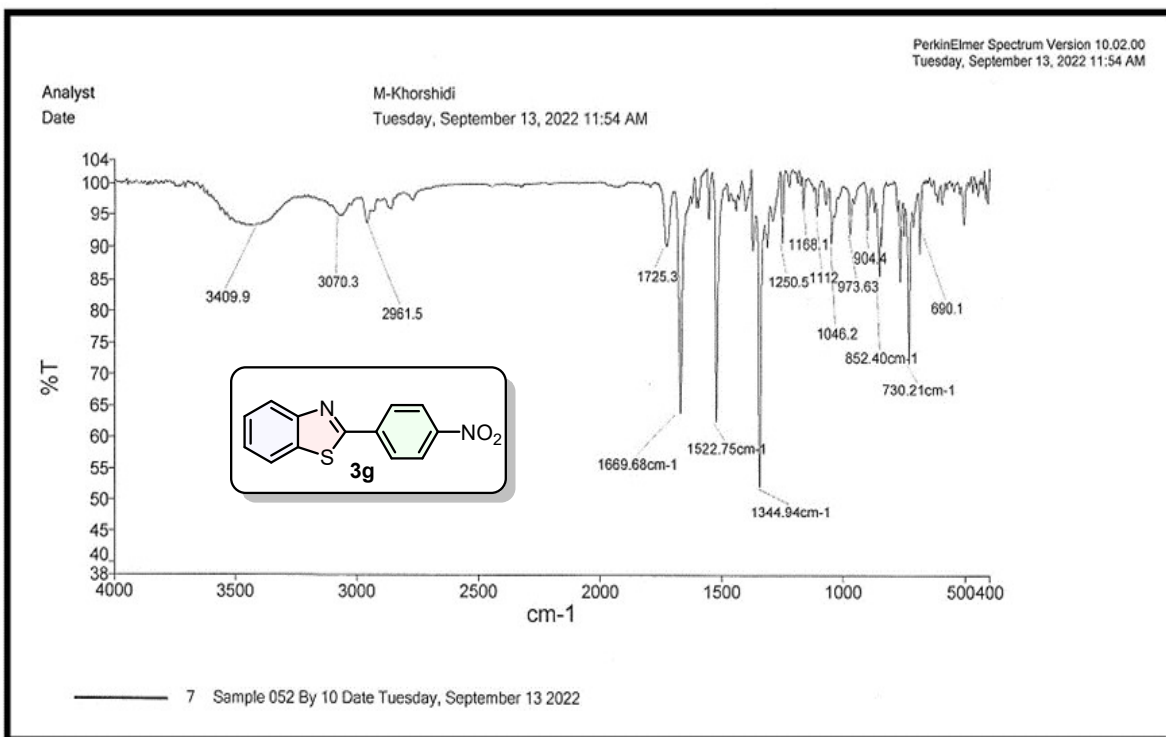
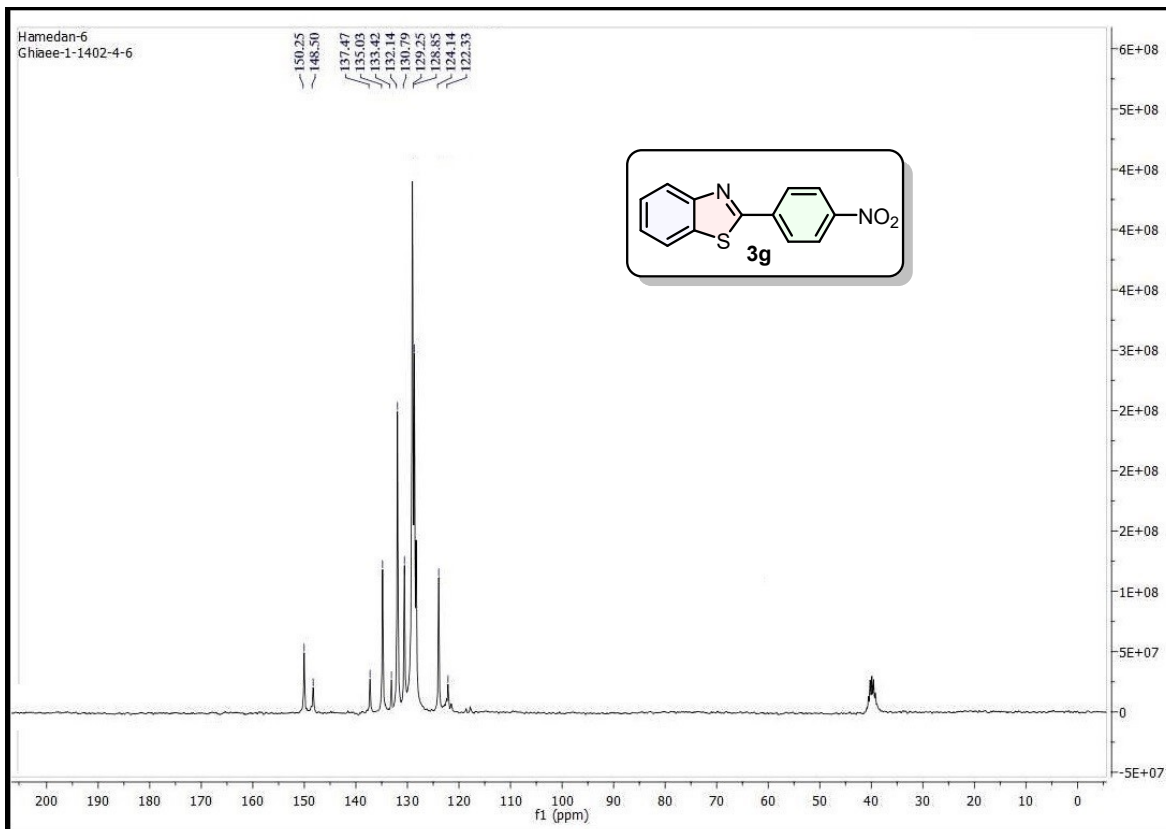


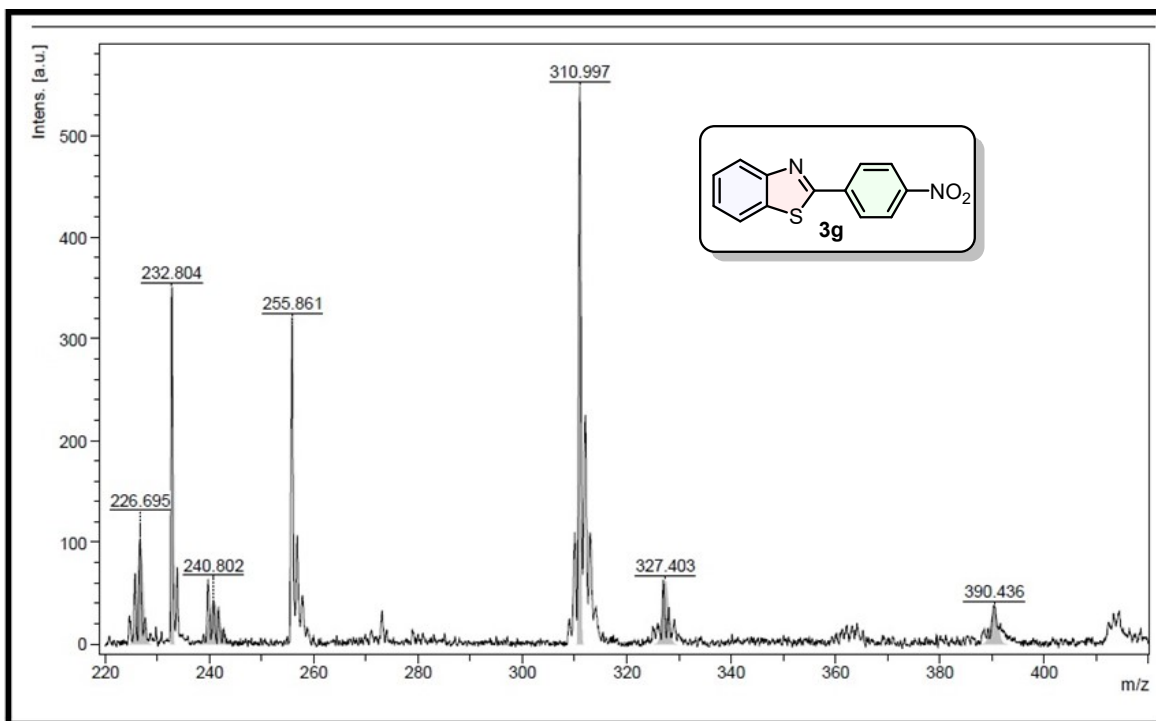




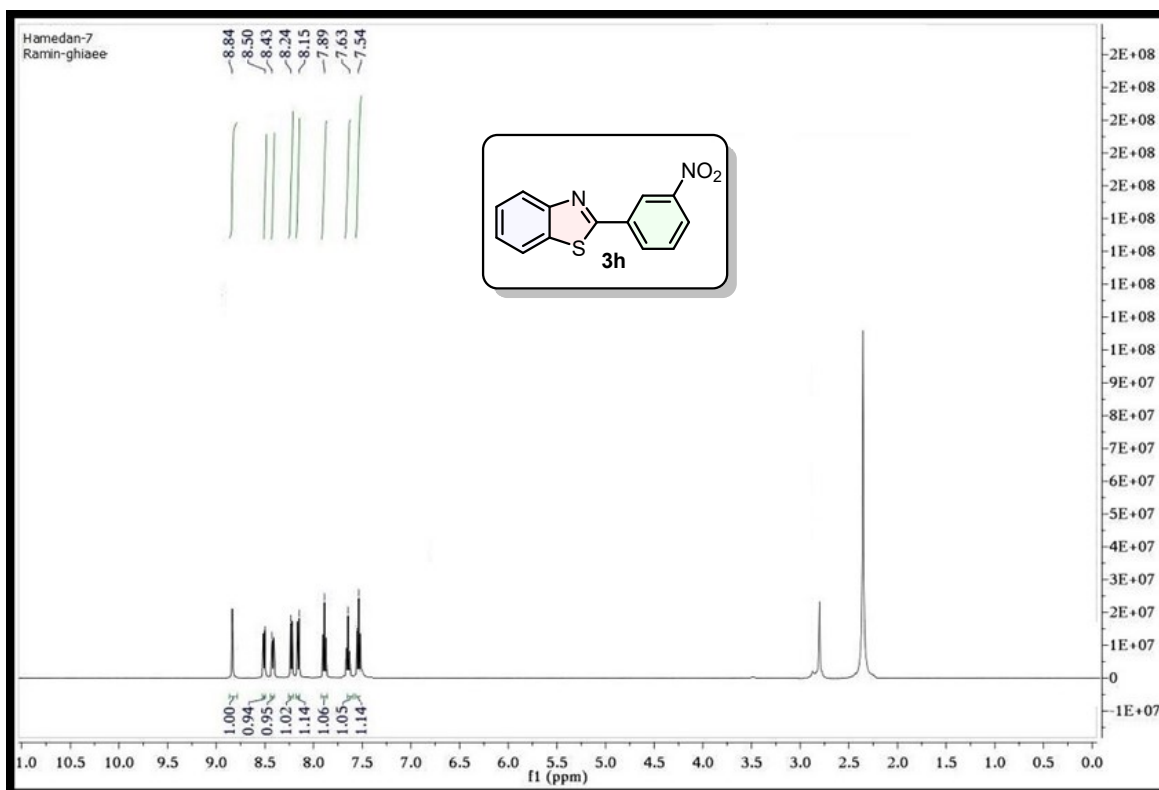
2-(4-Nitrophenyl)benzo[d]thiazole (3g)

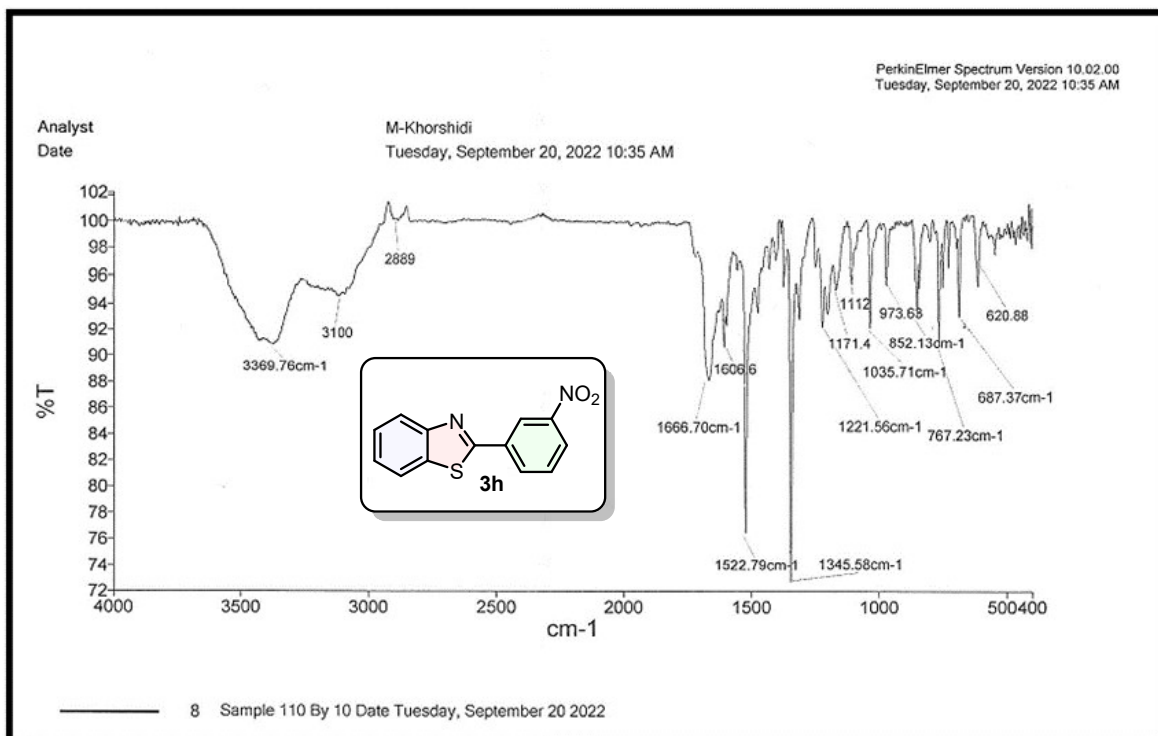
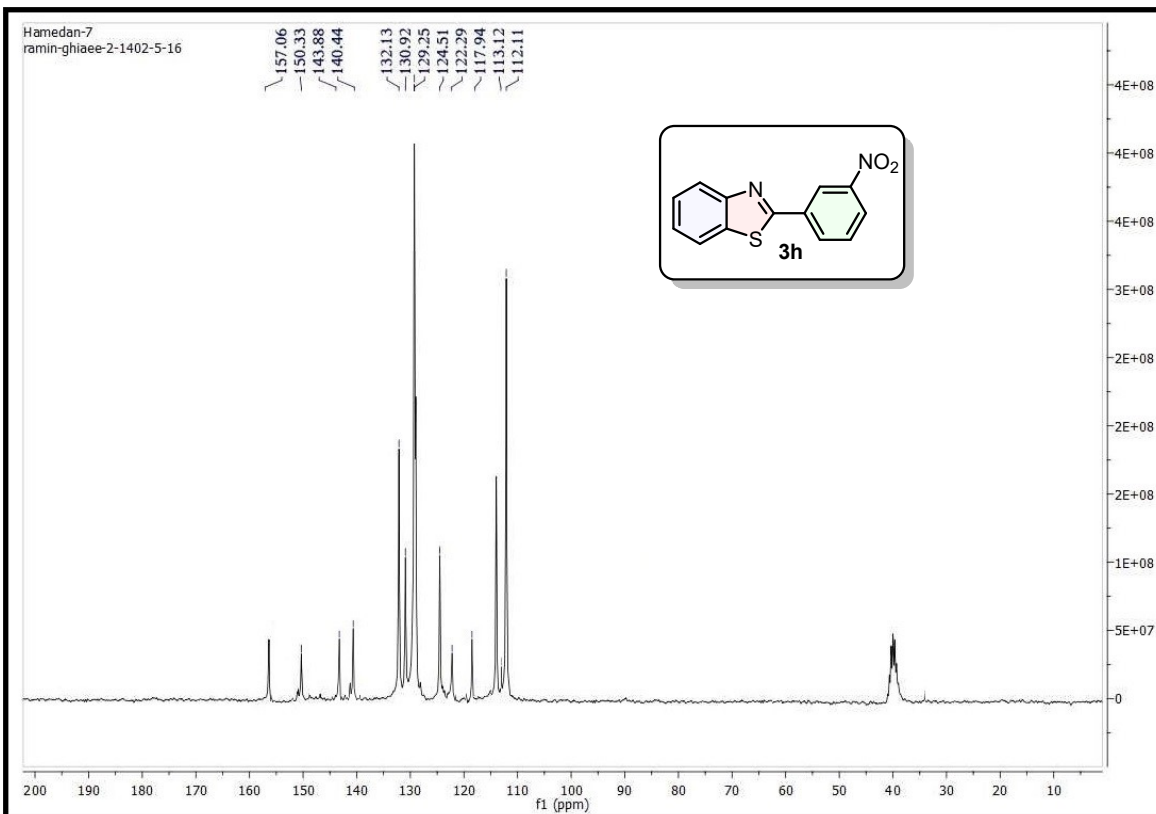


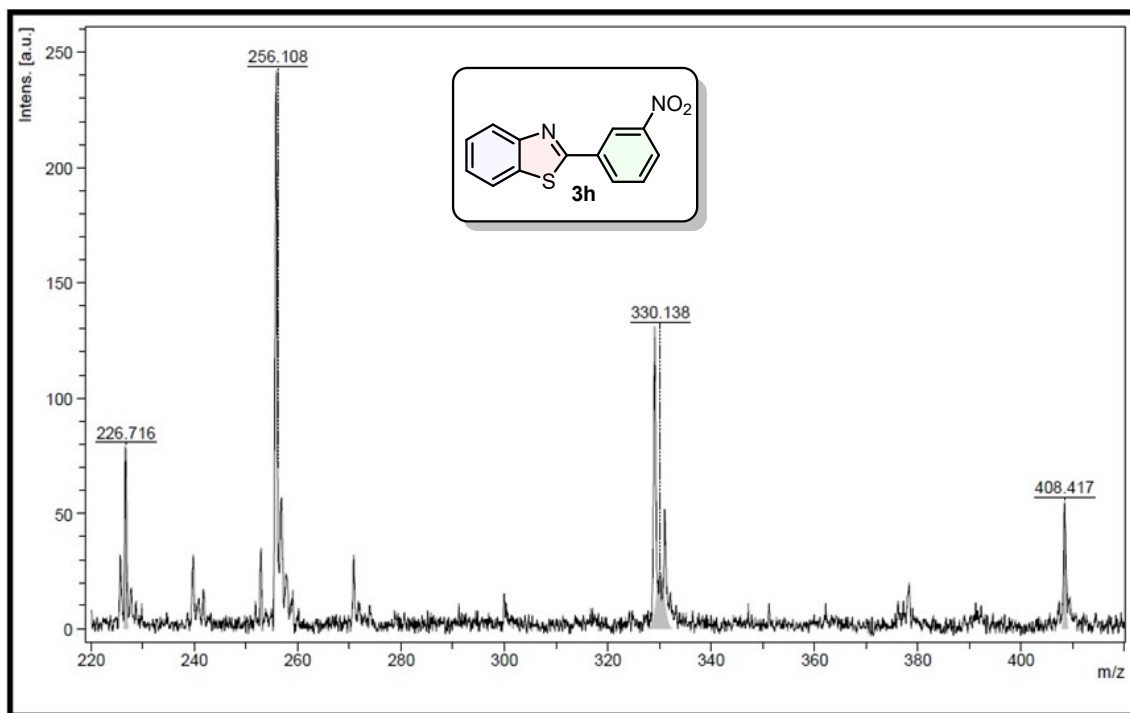




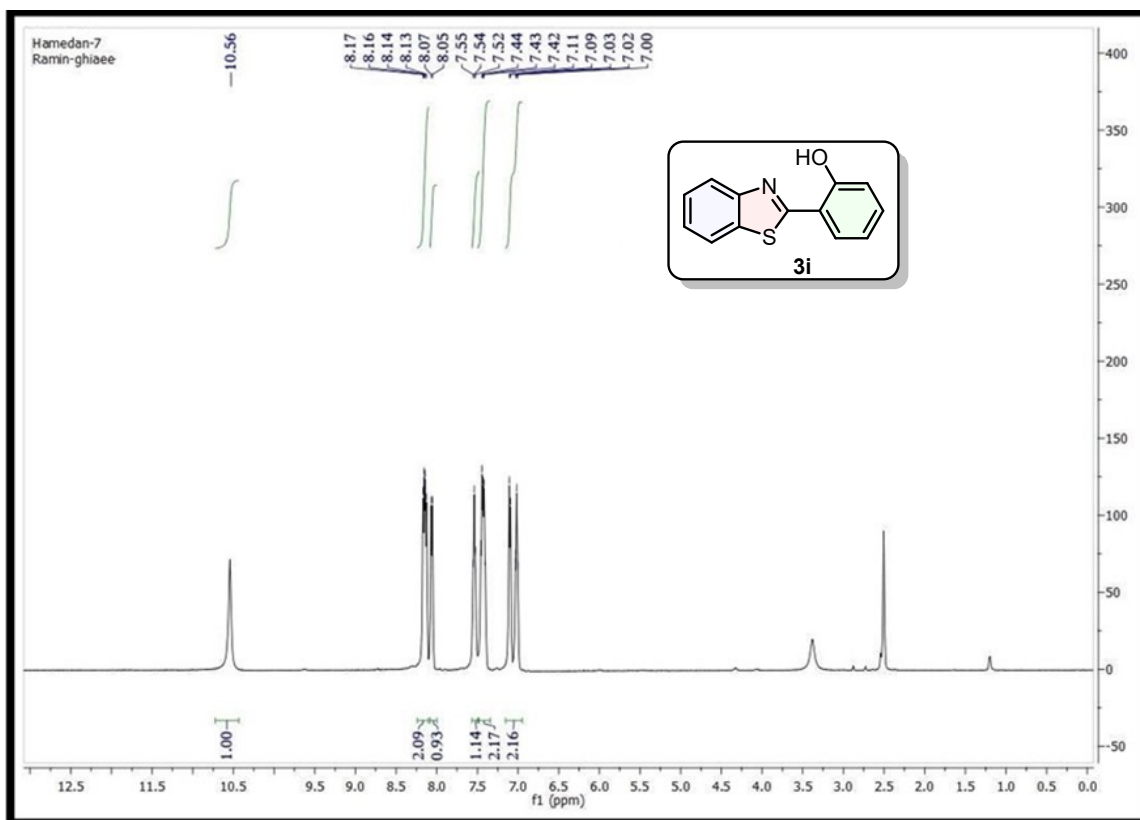
2-(3-Nitrophenyl)benzo[d]thiazole (**3h**)

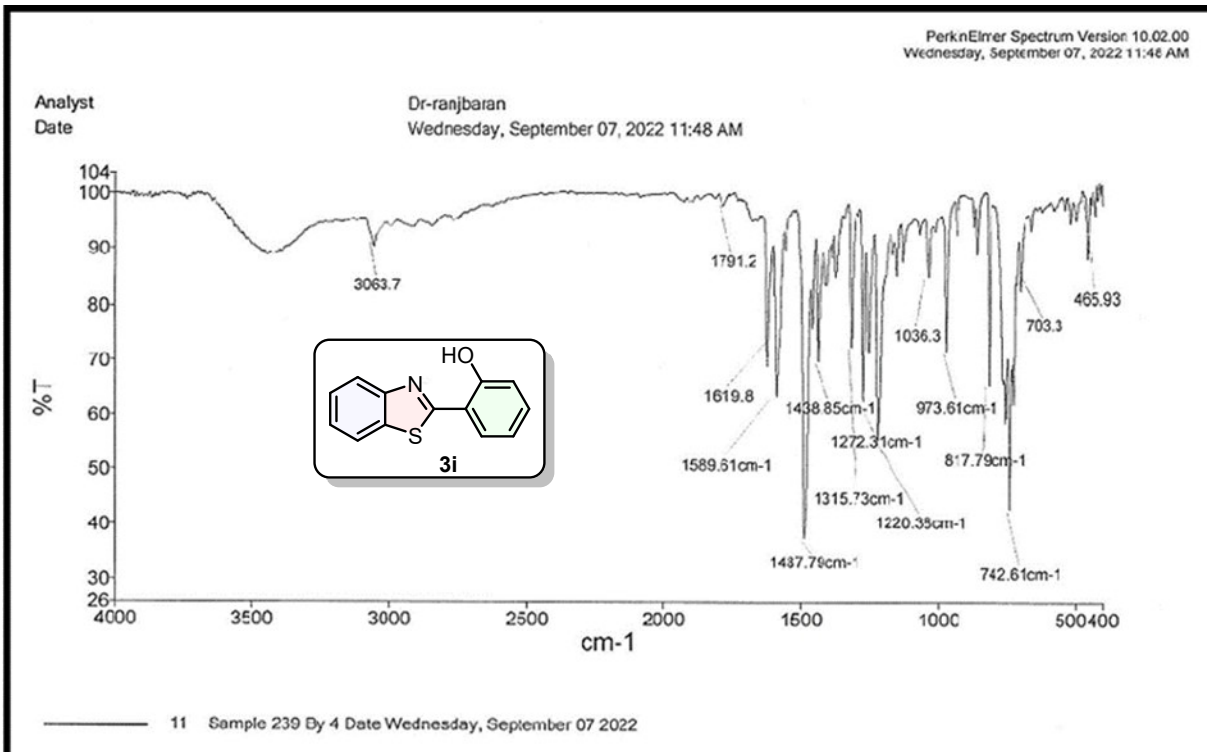
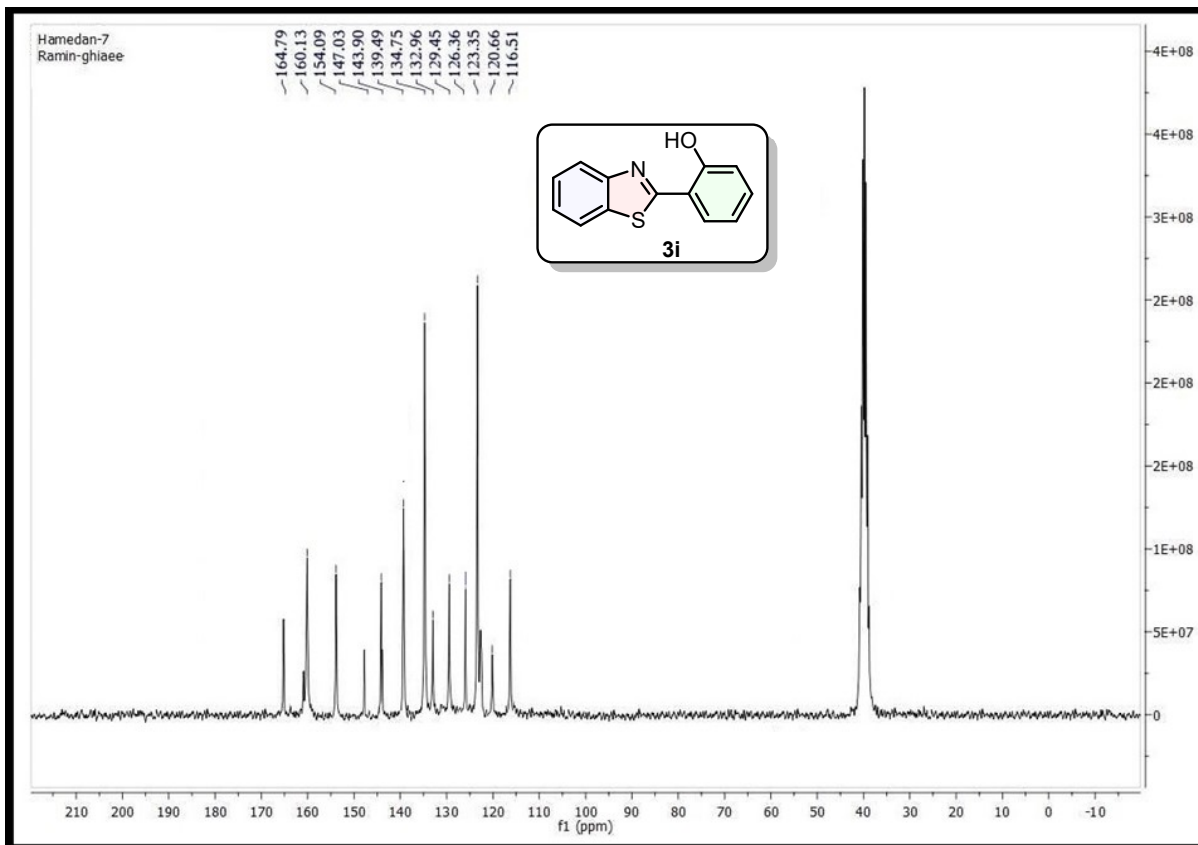


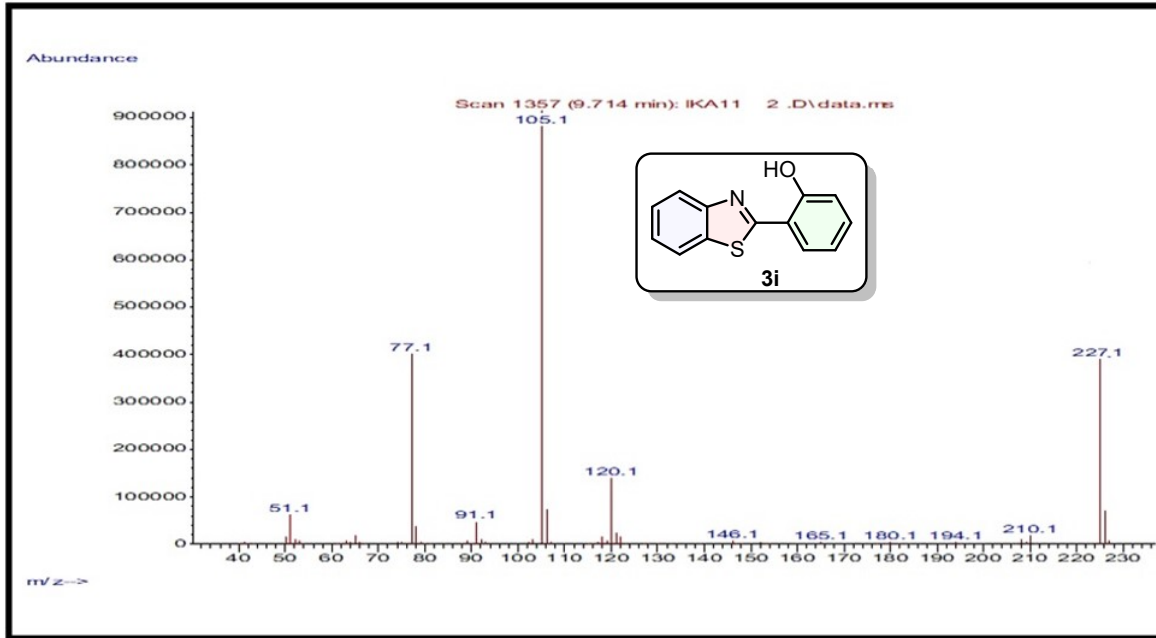




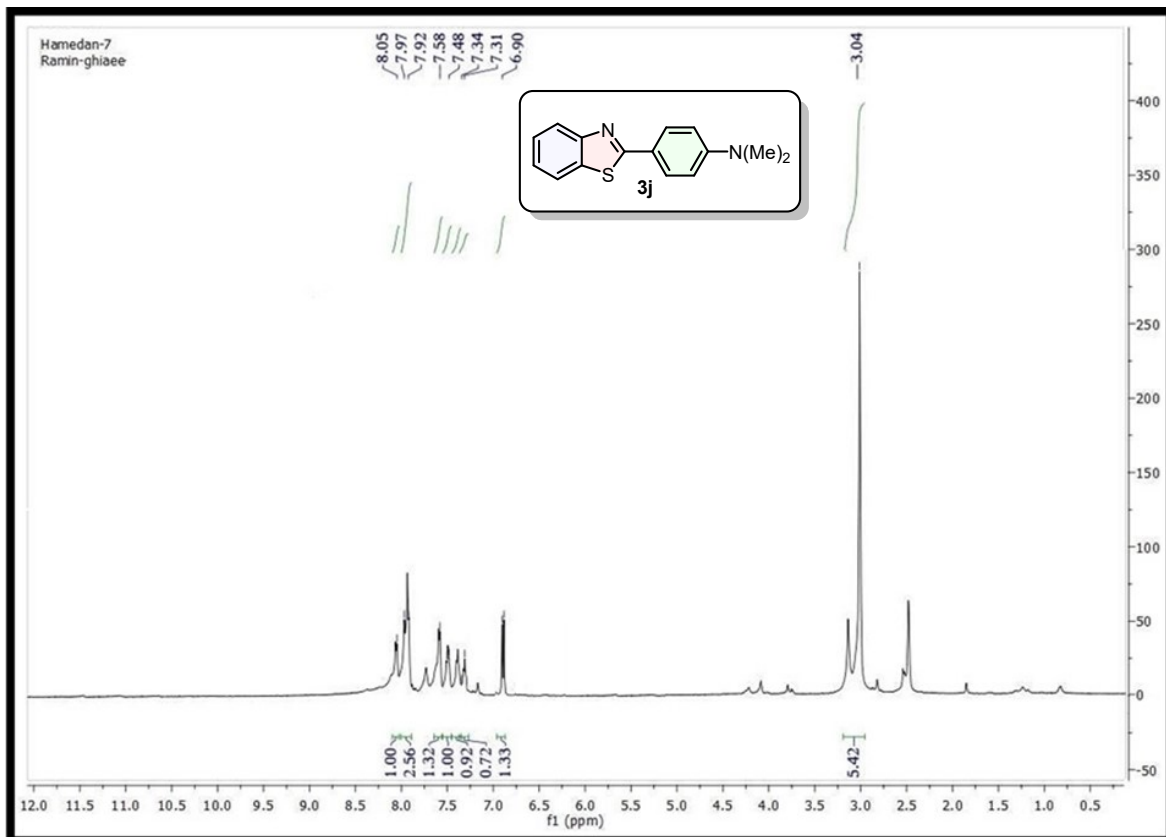
2-(2-Hydroxy phenyl)benzo[d]thiazole (3i)

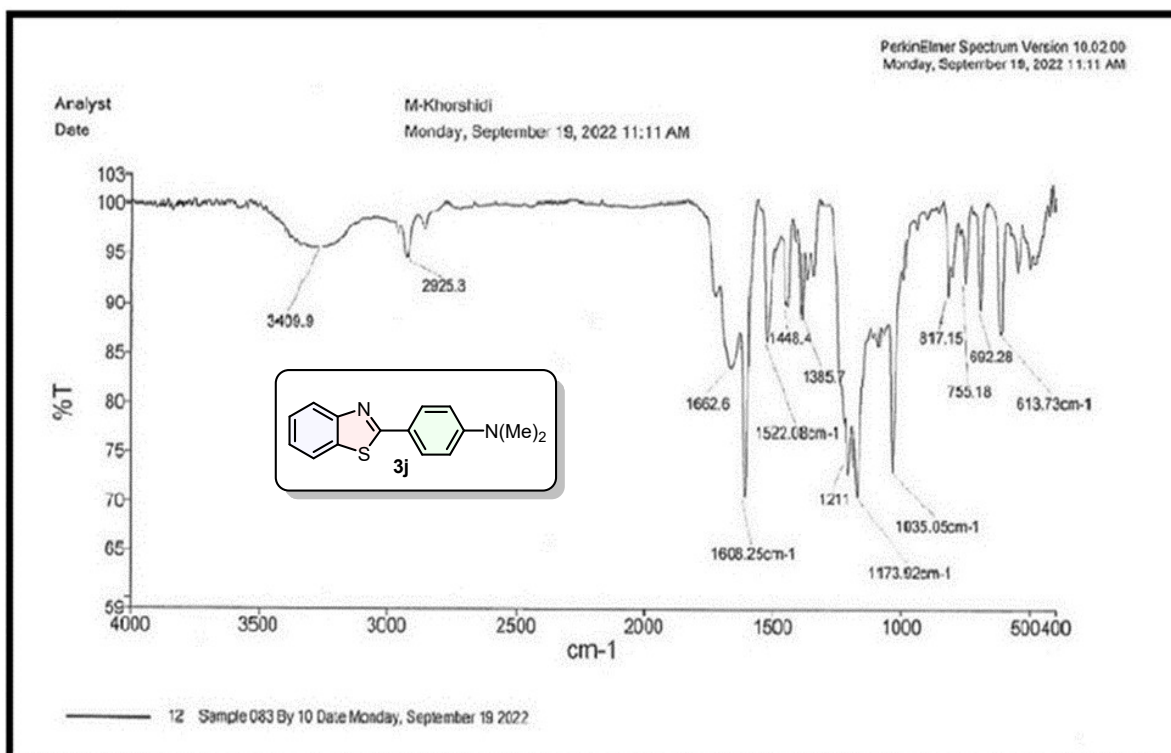
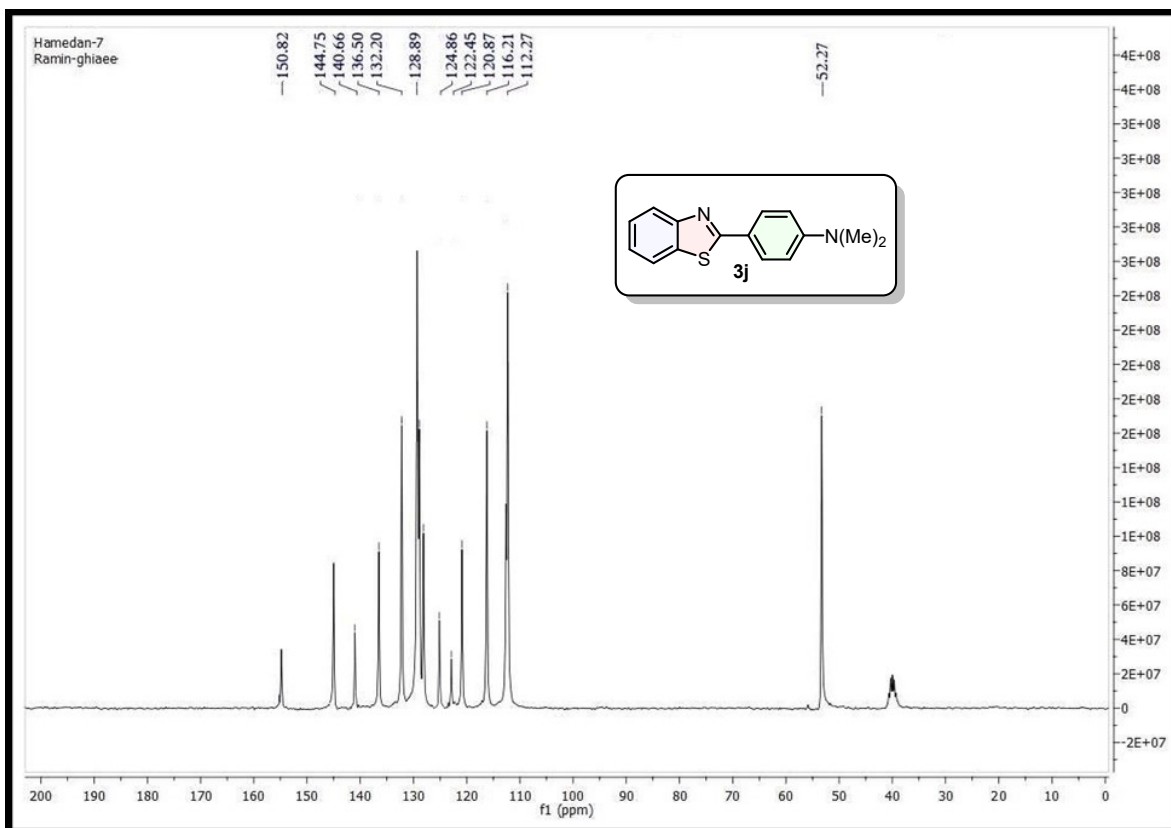


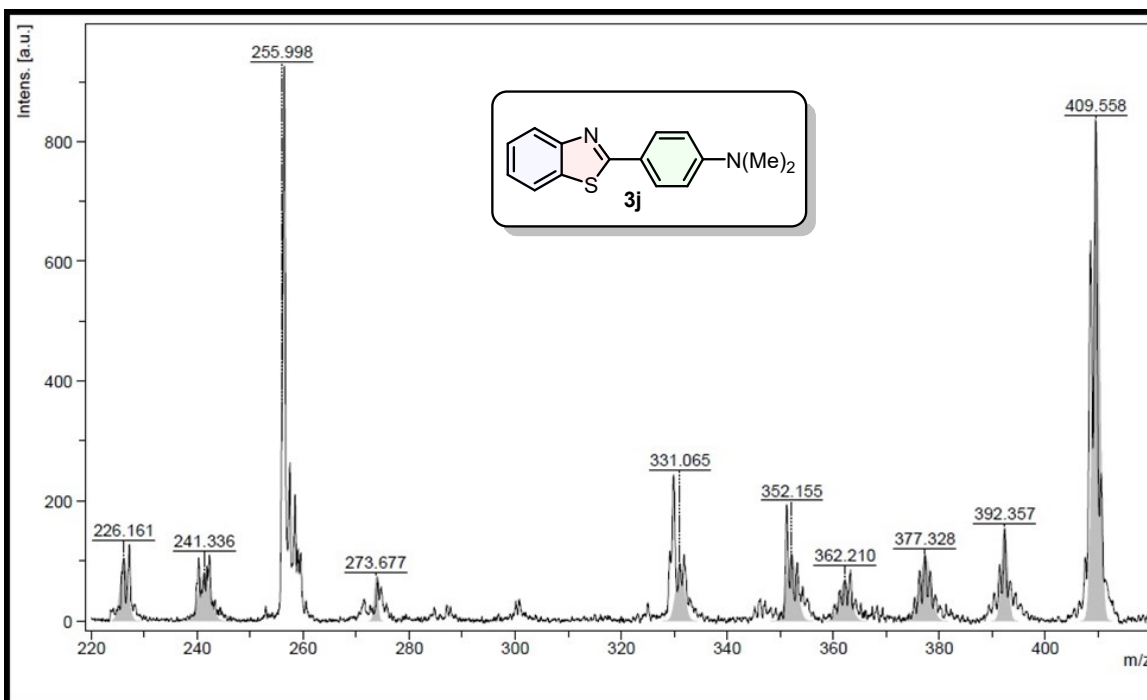




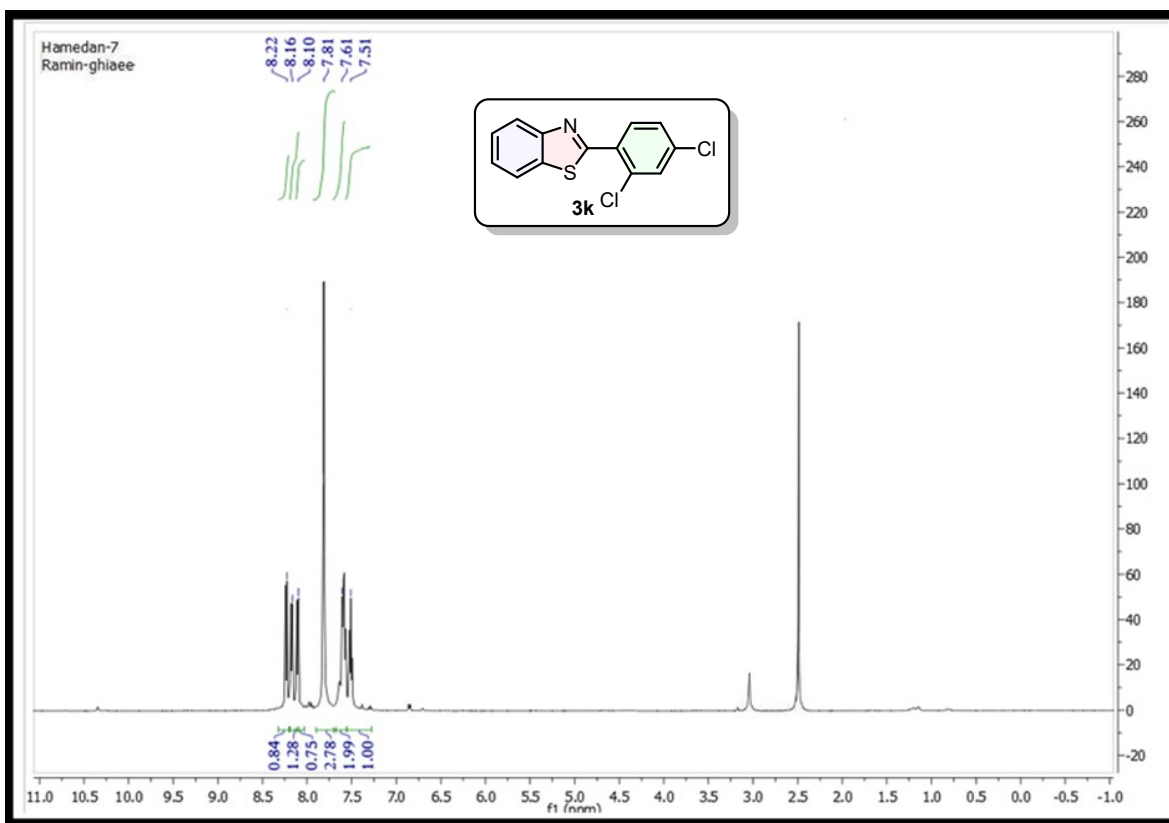
4-(Benzo[d]thiazol-2-yl)-N,N-dimethylaniline (3j)

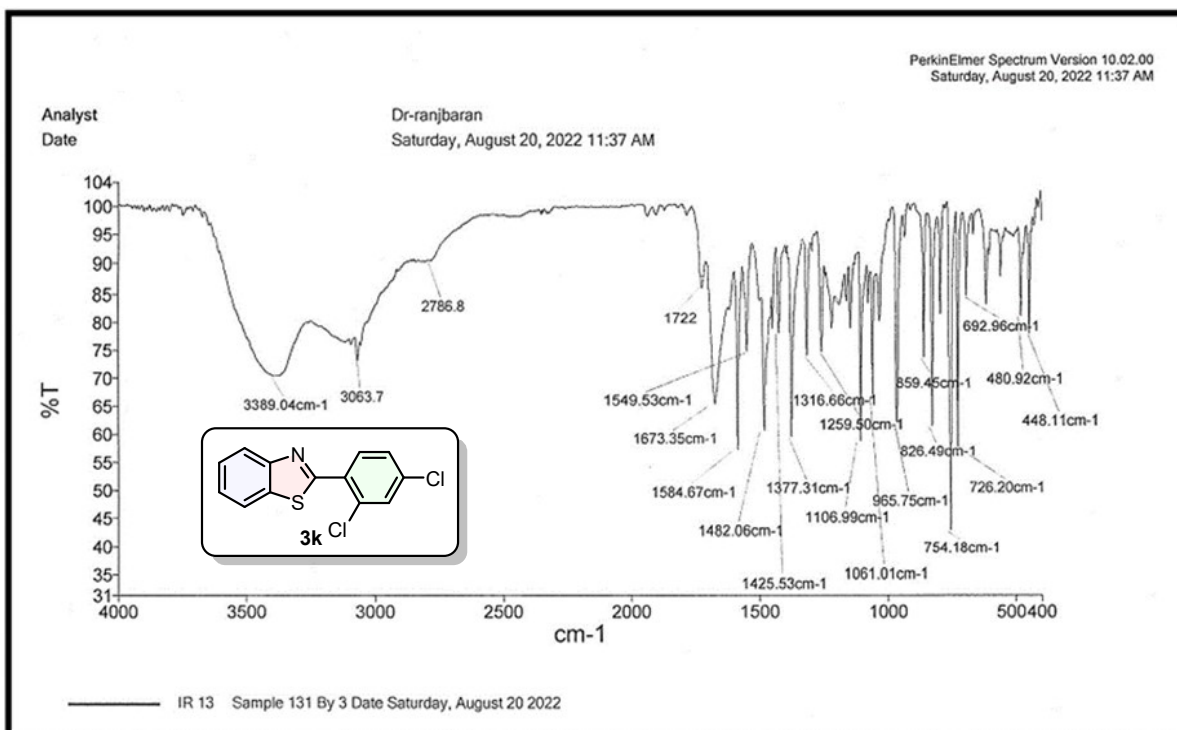
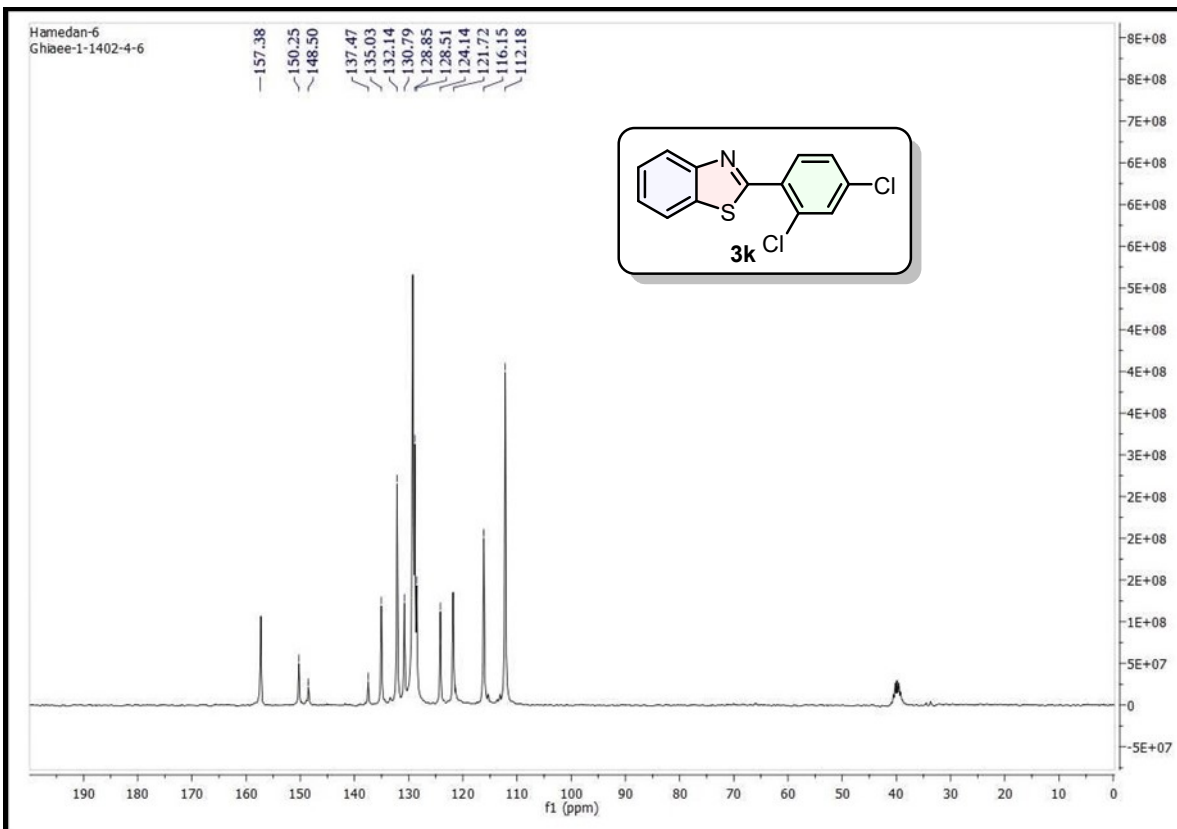


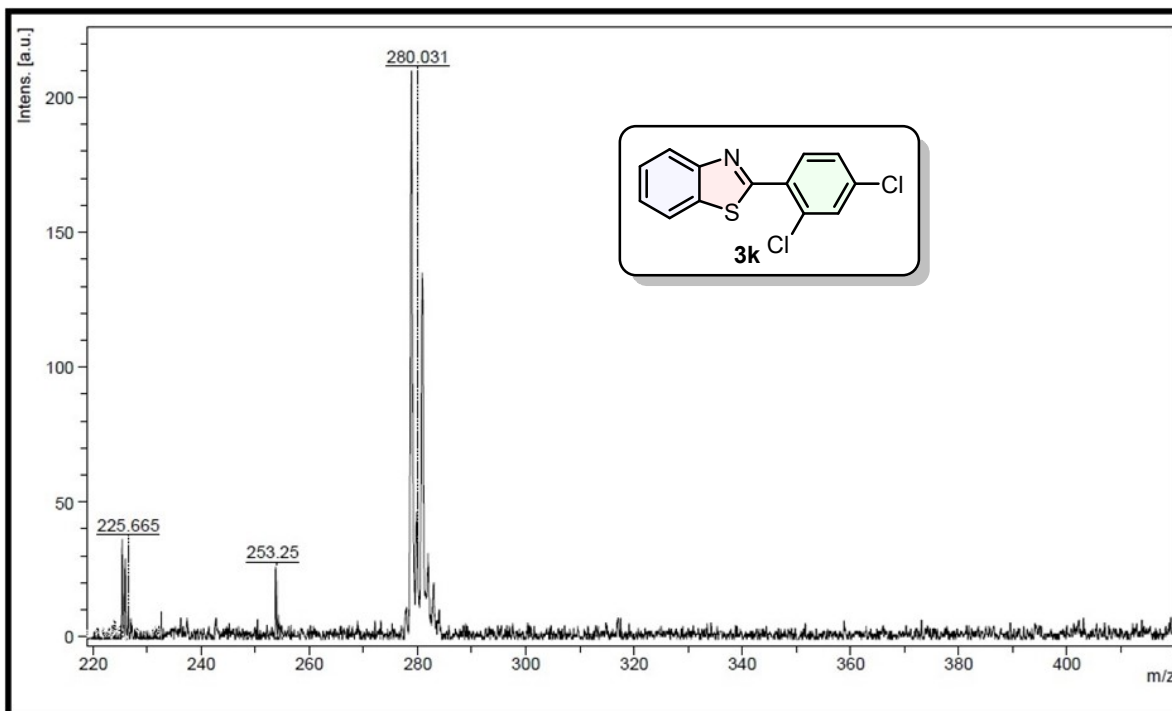




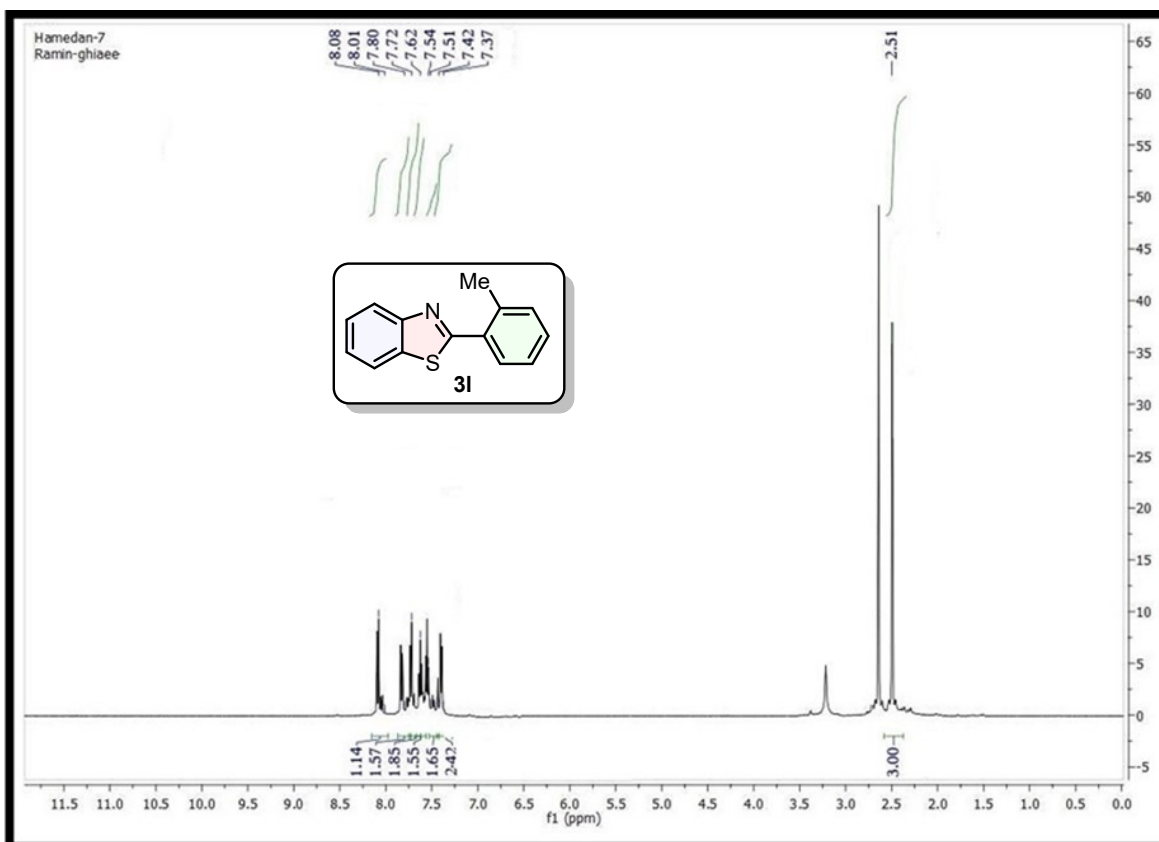
2-(2,4-Dichlorophenyl)benzo[d]thiazole (**3k**)

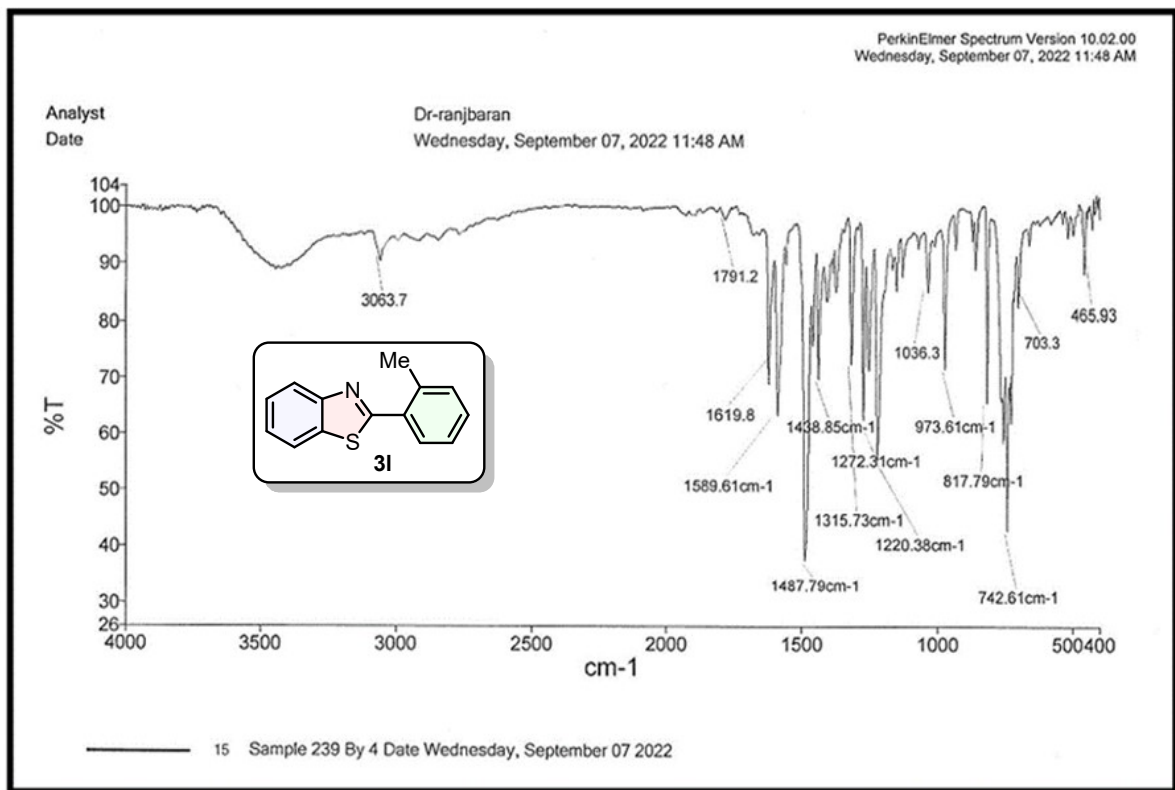
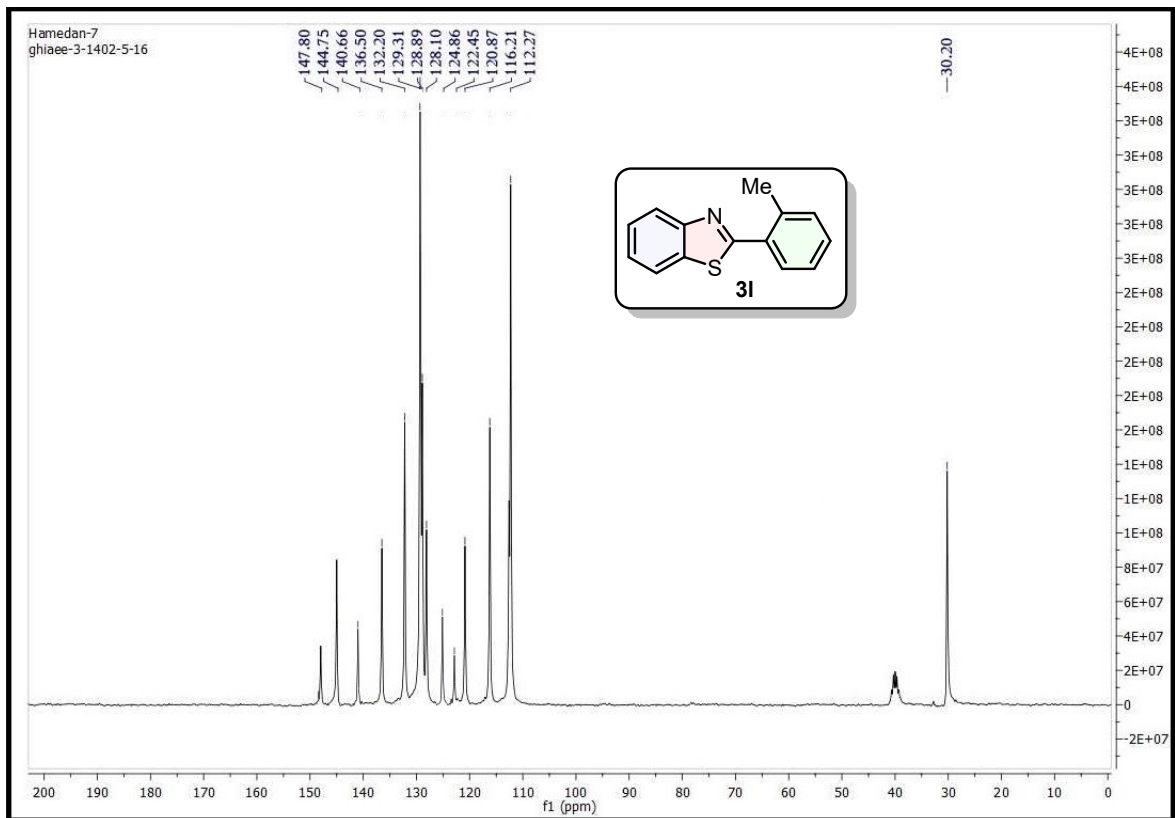


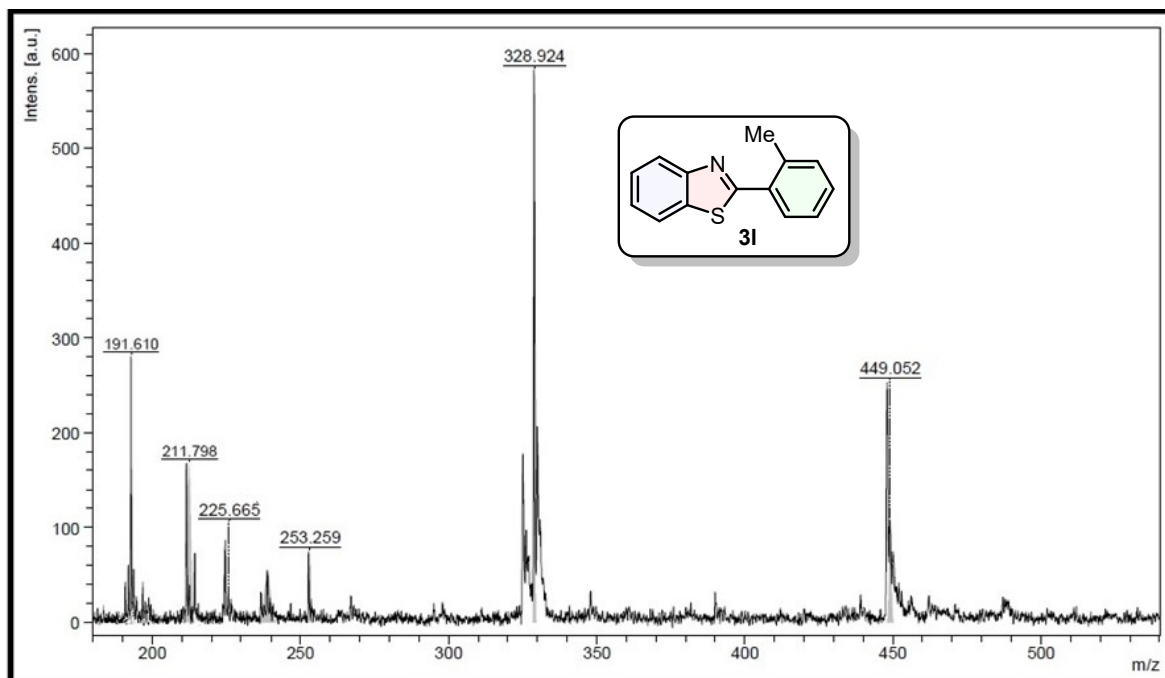




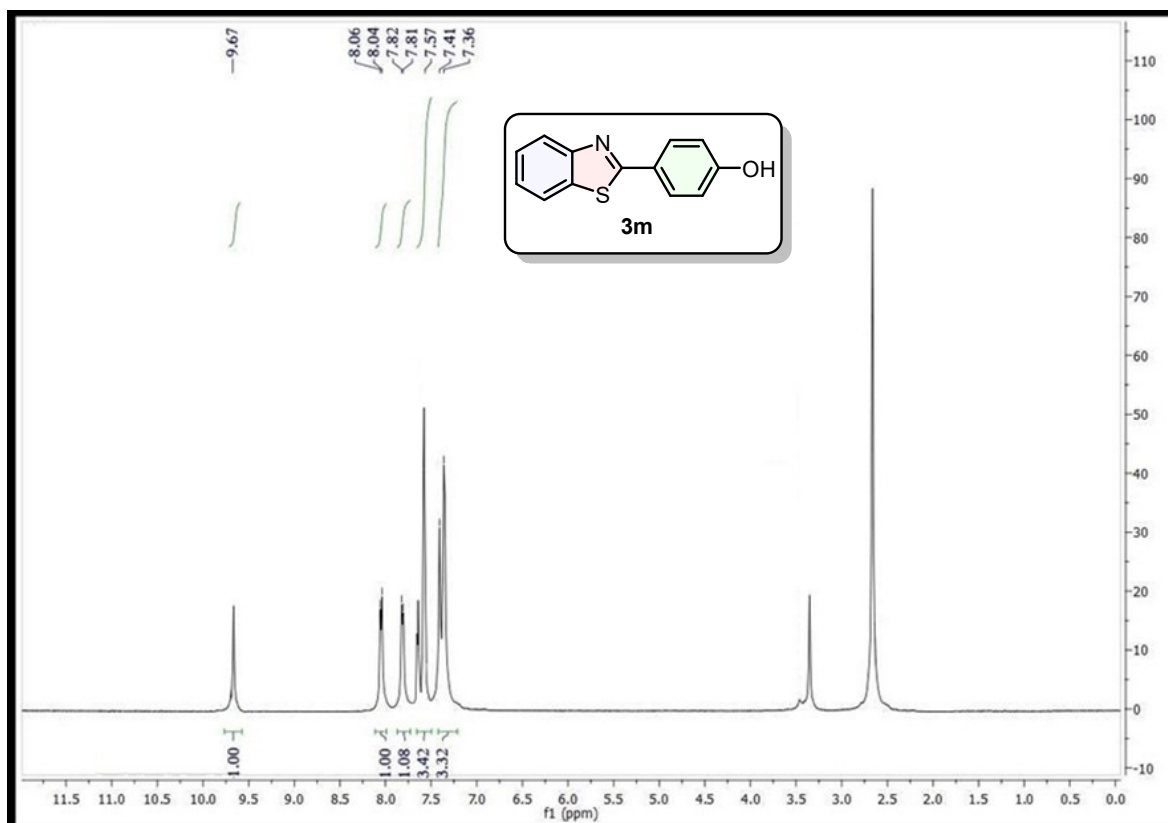
2-(*o*-tolyl)benzo[d]thiazole (**3l**)

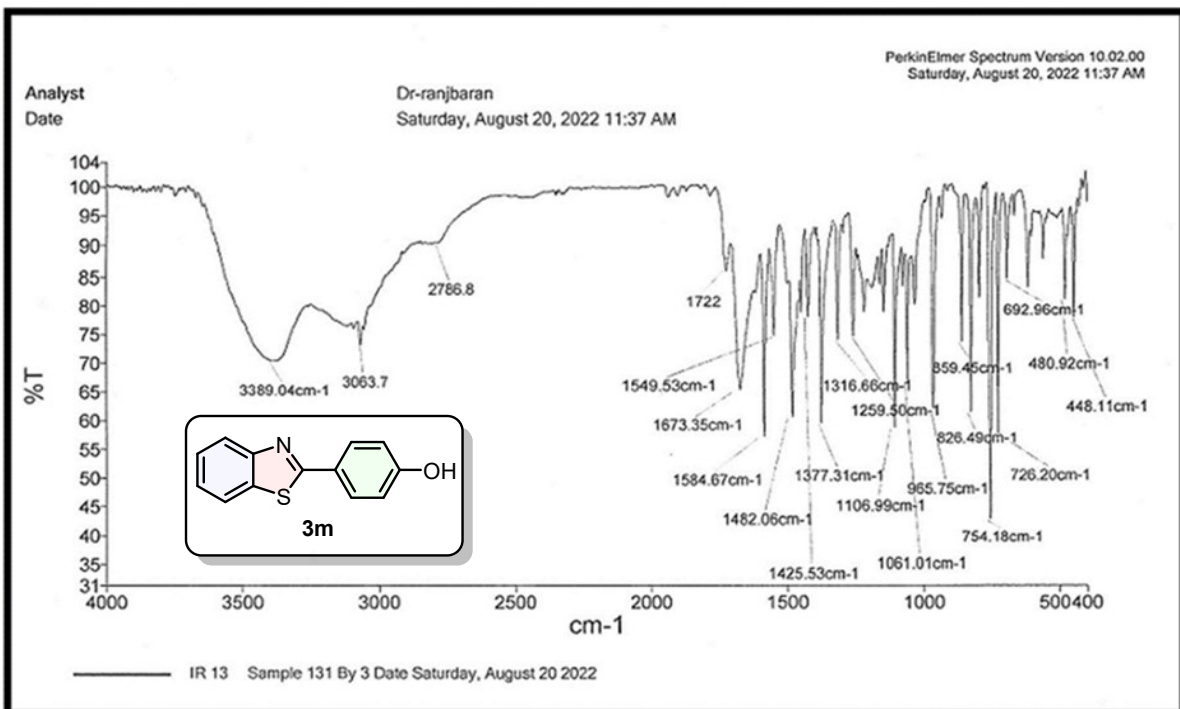
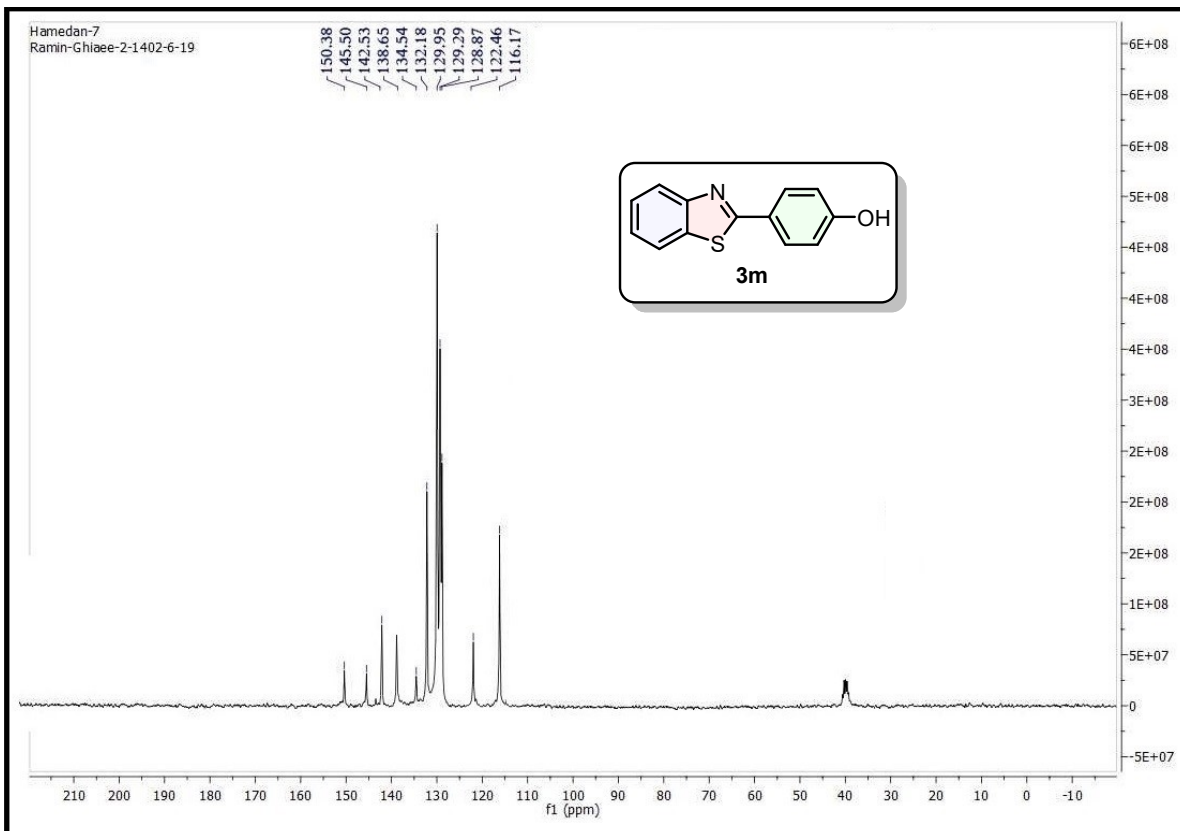


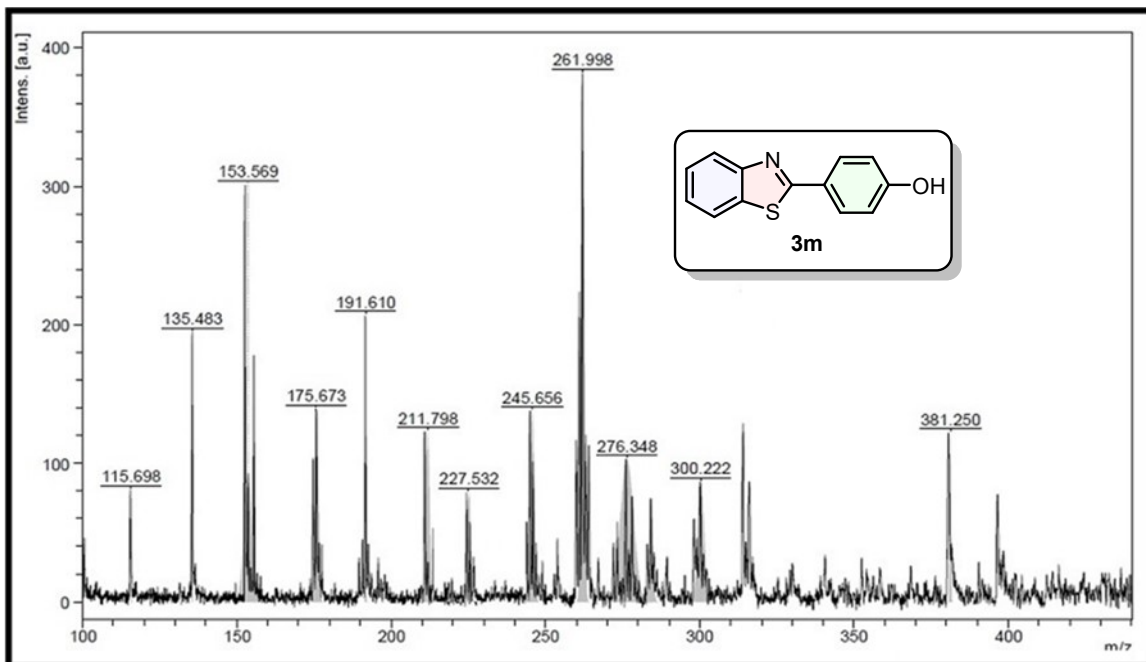




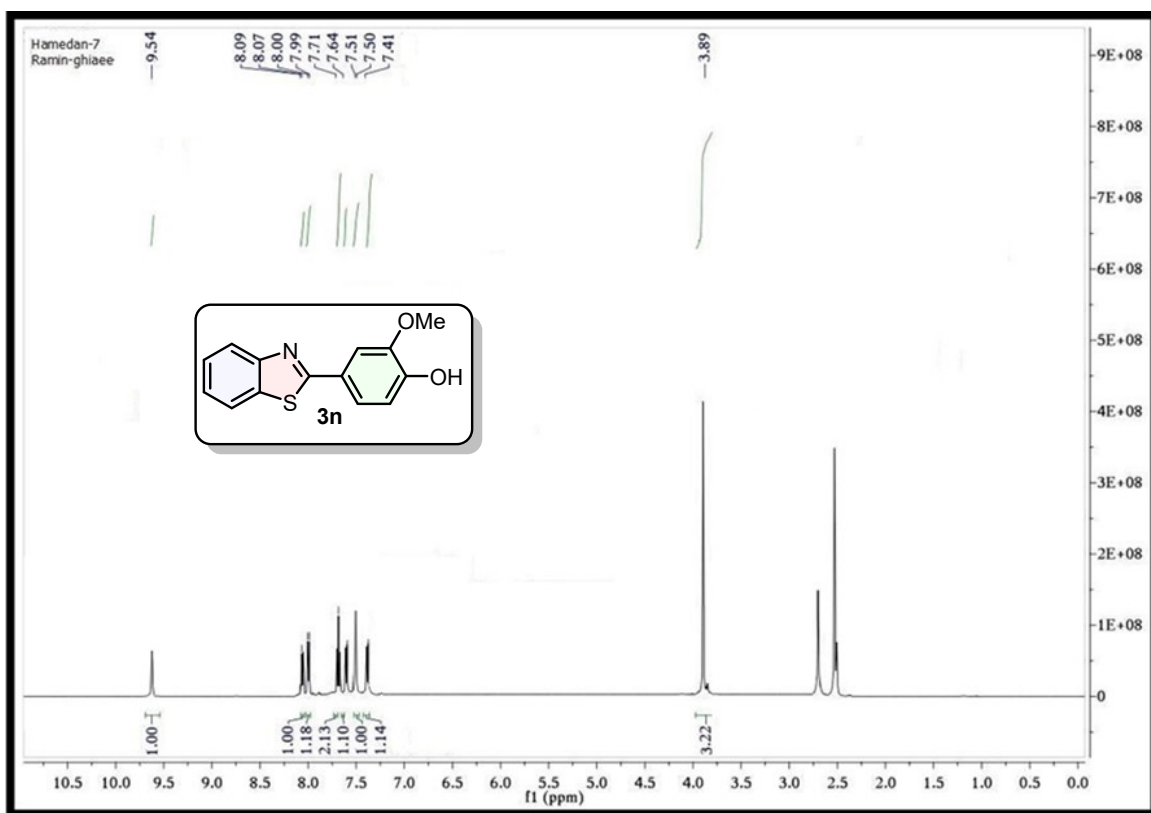
2-(4-Hydroxy phenyl)benzo[d]thiazole (3m)

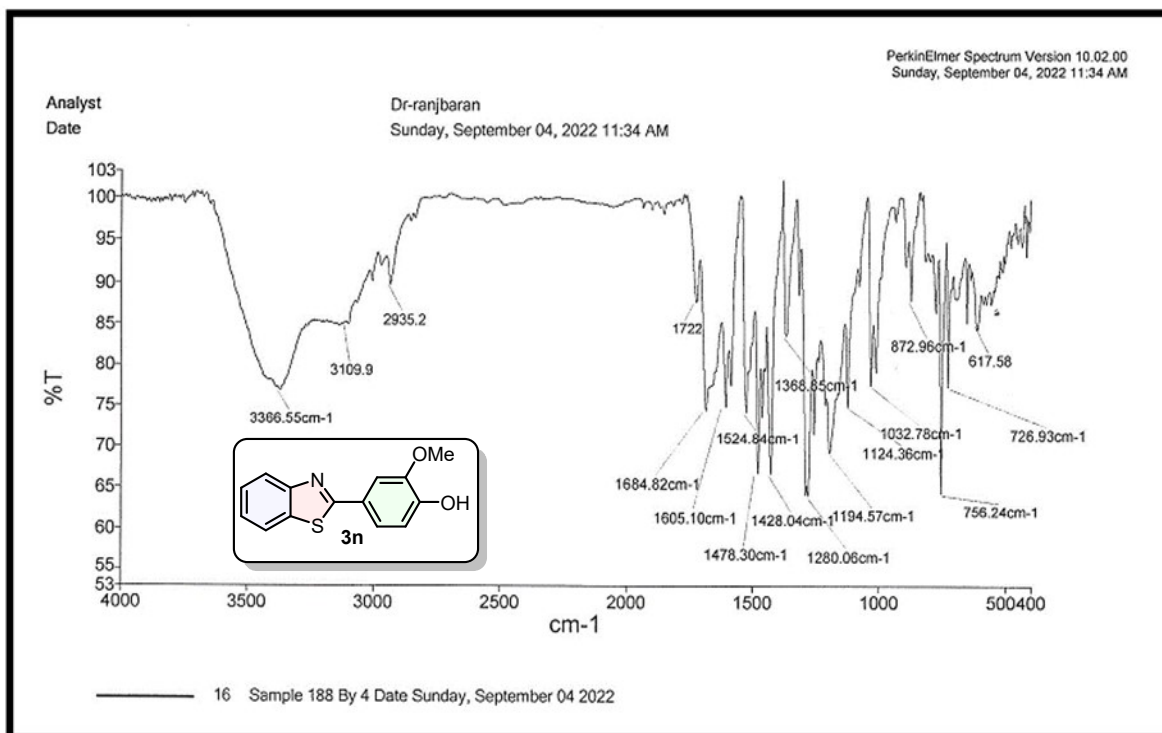
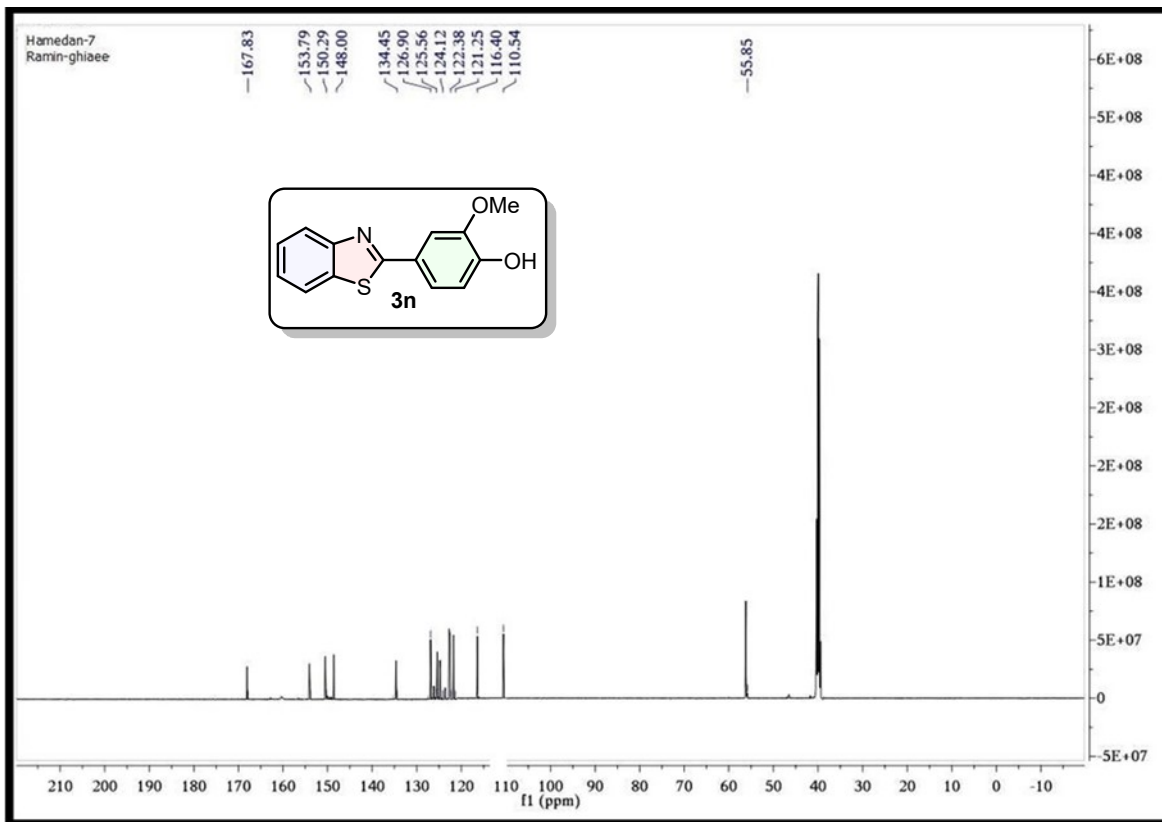


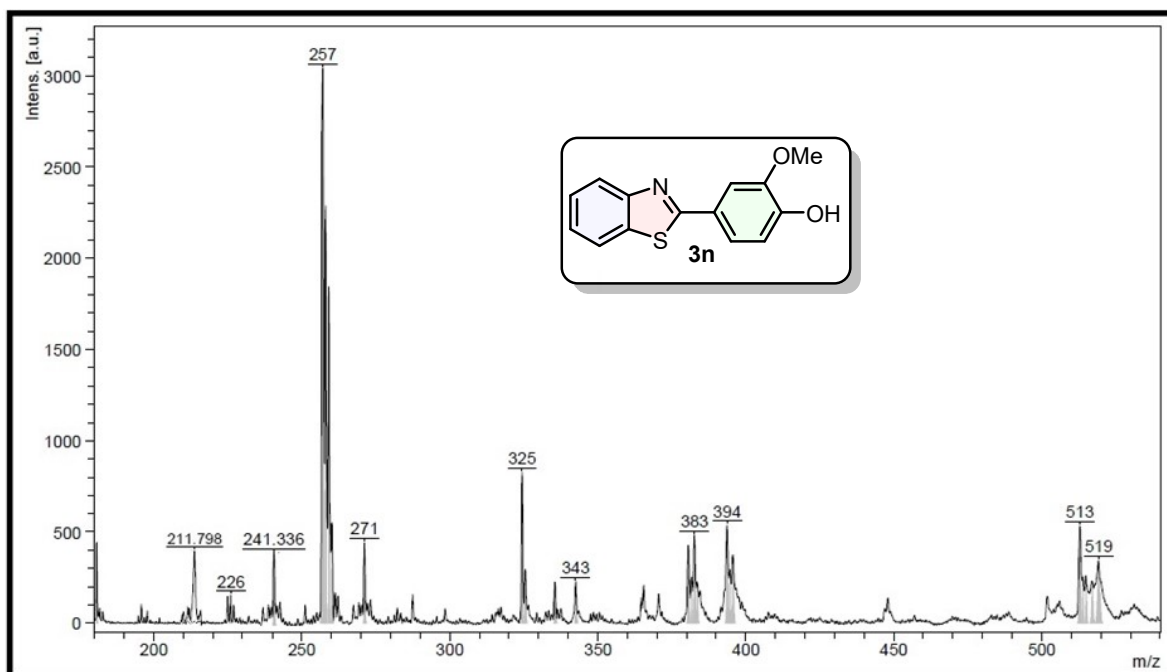




4-(Benzo[d]thiazol-2-yl)-2-methoxyphenol (3n)







2-(3-Methoxyphenyl)benzo[d]thiazole (**3o**)

