Electronic Supplementary Material (ESI) for RSC Advances.

Supplementary Information

Magnetically separable Fe_3O_4@chitin as an eco-friendly nanocatalyst with high efficiency for green synthesis of 5-substituted-1*H*-tetrazoles under solvent-free conditions

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Experimental

General

The purity determinations of the products were accomplished by TLC on silica gel polygram STL G/UV 254 plates. The melting points of products were determined with an Electrothermal Type 9100 melting point apparatus. The FTIR spectra were provided on pressed KBr pellets using an AVATAR 370 FT-IR spectrometer (Therma Nicolet spectrometer, USA) at room temperature in the range between 4000 and 400 cm^{-1}. The NMR spectra were obtained in Brucker Avance 100, 300 and 400 MHz instruments in $CDCl_3$, DMSO-d_6 and CD_3CN. Elemental analysis was performed using a Thermo Finnigan Flash EA 1112 Series instrument. Mass spectra were recorded with a CH7A Varianmat Bremem instrument at 70 eV electron impact ionization, in *m/z* (rel %). All of the products were known compounds and they were characterized by the FT-IR spectroscopy, ^{1}H NMR, ^{13}C NMR spectroscopy, and mass spectrometry and comparison of their melting points with known compounds.

5-Phenyl-1*H*-tetrazole (Table 2, Entry 1) (0.138 g, 95%); White solid; mp 214-216 ºC (Lit.[1] 214-216 ºC); FT-IR (KBr): ν_{max}/cm^{-1} 3125, 3043, 2982, 2913, 2835, 2765, 2692, 2606, 2557, 2488, 1613, 1563, 1485, 1465, 1409, 1254 (N–N=N–), 1163 (C–N), 1056, 726, 703, 687; ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ 7.61 (s, 3H, Ar-H), 8.05 (s, 2 H, Ph); ^{13}C NMR (100 MHz, DMSO-d_6, ppm) δ 124.6, 127.4, 129.9, 131.7, 155.7.

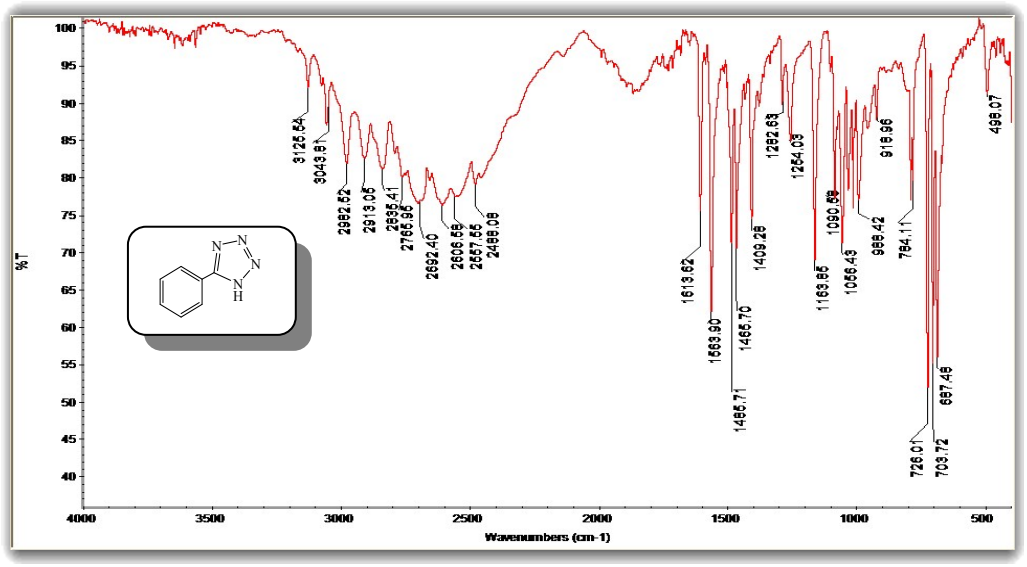

Figure 1: FT-IR (KBr) of 5-Phenyl-1*H*-tetrazole.

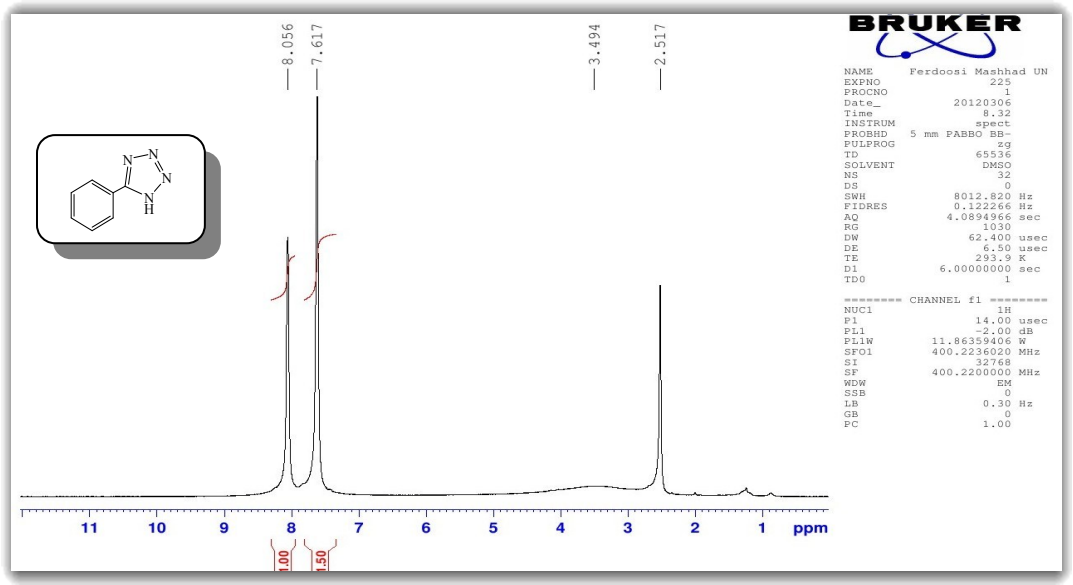

Figure 2: [1]H NMR (400 MHz, DMSO-d_6) of 5-Phenyl-1*H*-tetrazole.

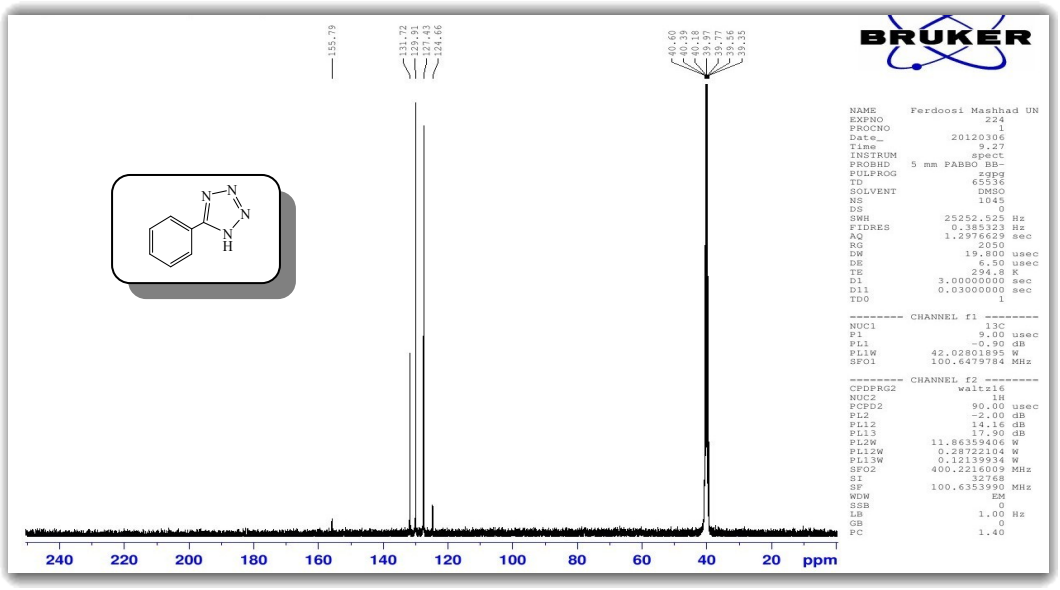

Figure 3: [13]C NMR (100 MHz, DMSO-d_6) of 5-Phenyl-1*H*-tetrazole.

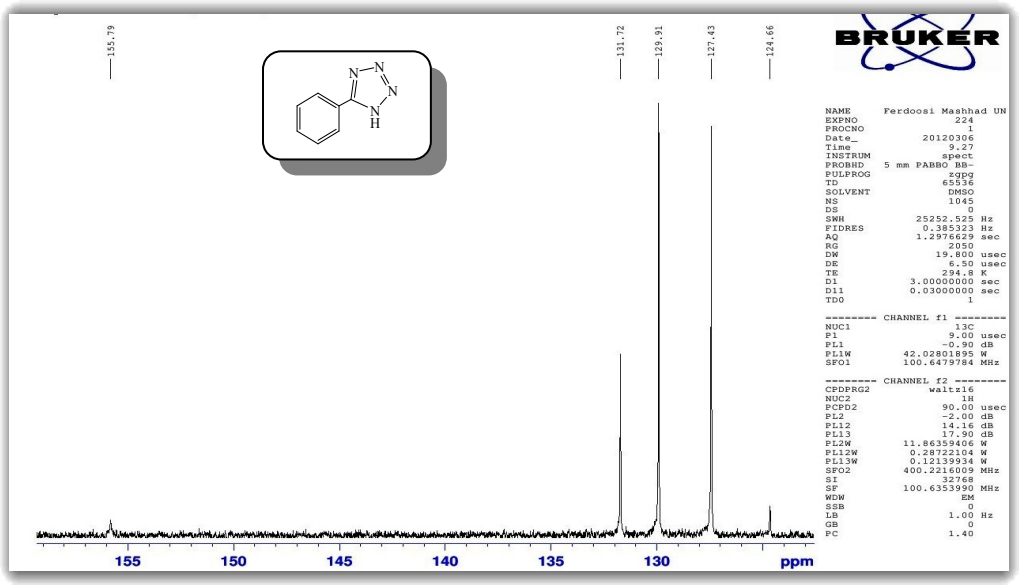

Figure 4: [13]C NMR (100 MHz, DMSO-d_6) of 5-Phenyl-1*H*-tetrazole expanded.

5-(4-Boromophenyl)-1*H*-tetrazole (Table 2, Entry 2) (0.20 g, 90 %); White solid; mp 264-265 ºC (Lit.[2] 265 ºC); FT-IR (KBr): ν_{max}/cm^{-1} 3089, 3063, 2996, 2900, 2844, 2761, 2729, 2633, 1652, 1604, 1560, 1482, 1431, 1405, 1278 (N–N=N–), 1157 (C-N), 1076, 1054, 1018, 829, 744, 502; ^{1}H NMR (300 MHz, DMSO-d_6, ppm) δ 7.78 (d, J = 8.4 Hz, 2H, Ar-H), 7.95 (d, J = 7.8 Hz, 2H, Ar-H); ^{13}C NMR (75 MHz, DMSO-d_6. ppm) δ 123.9, 125.1, 129.2, 132.8, 155.3.

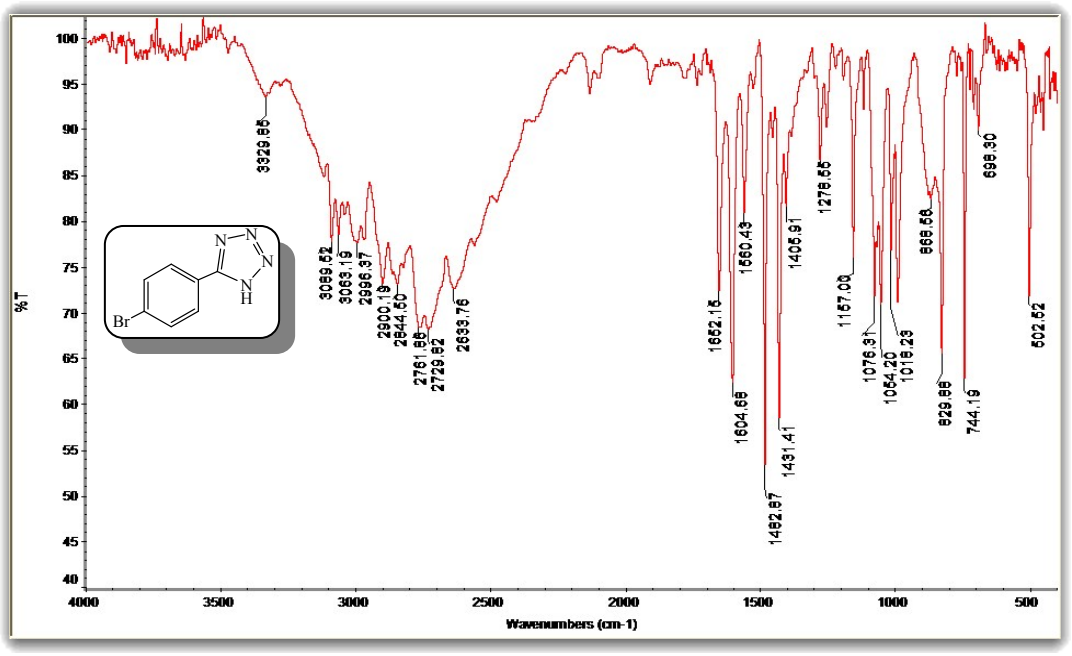

Figure 5: FT-IR (KBr) of 5-(4-Boromophenyl)-1*H*-tetrazole.

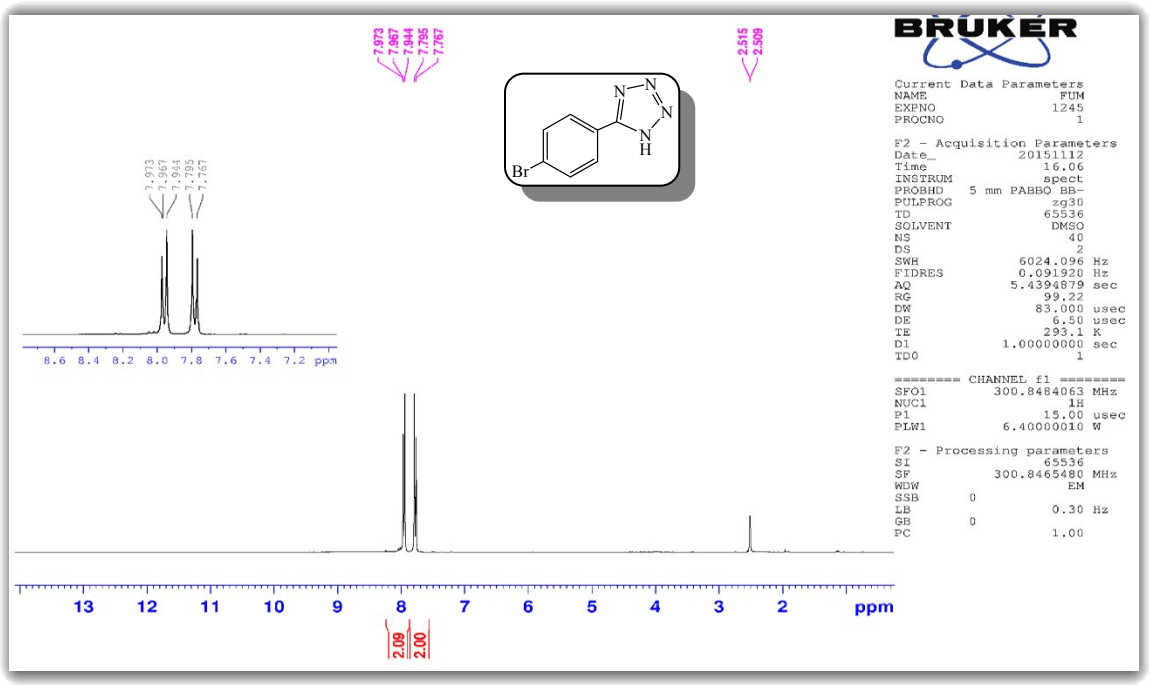

Figure 6: [1]H NMR (300 MHz, DMSO-d_6) of 5-(4-Boromophenyl)-1*H*-tetrazole.

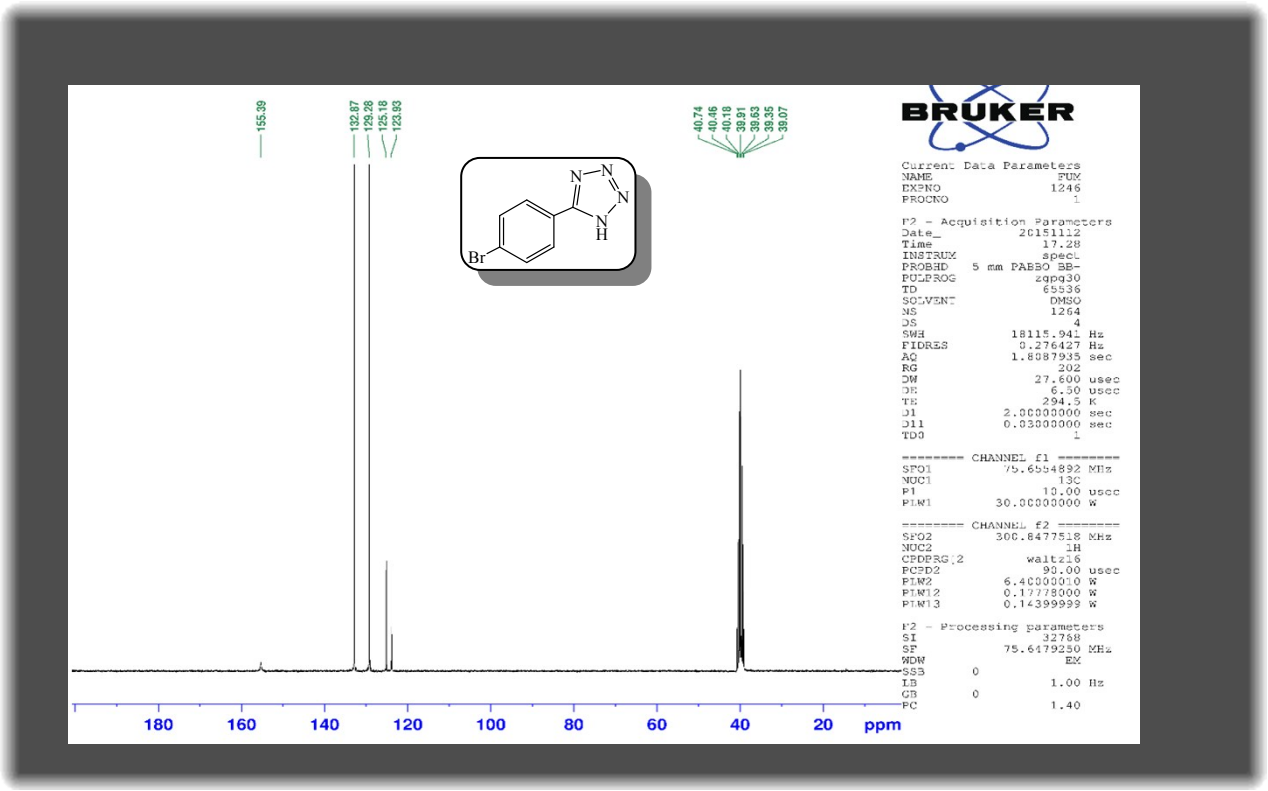

Figure 7: [13]C NMR (75 MHz, DMSO-d_6) of 5-(4-Boromophenyl)-1*H*-tetrazole

5-(4-Chlorophenyl)-1*H*-tetrazole (Table 2, Entry 3) (0.171, 95 %); White solid; mp 261-262 ºC (Lit.[3] 261-263 ºC). FT-IR (KBr): ν_{max}/cm^{-1} 3092, 3060, 3007, 2978, 2907, 2851, 2725, 2622, 2537, 2471, 1609, 1564, 1486, 1435, 1274 (N–N=N–), 1160 (C-N), 1096, 1053, 1020, 990, 833, 745, 508; ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ 7.68 (d, J = 8.4 Hz, 2H, Ar-H), 8.05 (d, J = 8.8 Hz, 2H, Ar-H); ^{13}C NMR (100 MHz, DMSO-d_6, ppm) δ 123.5, 129.2, 130.0, 136.4, 155.3.

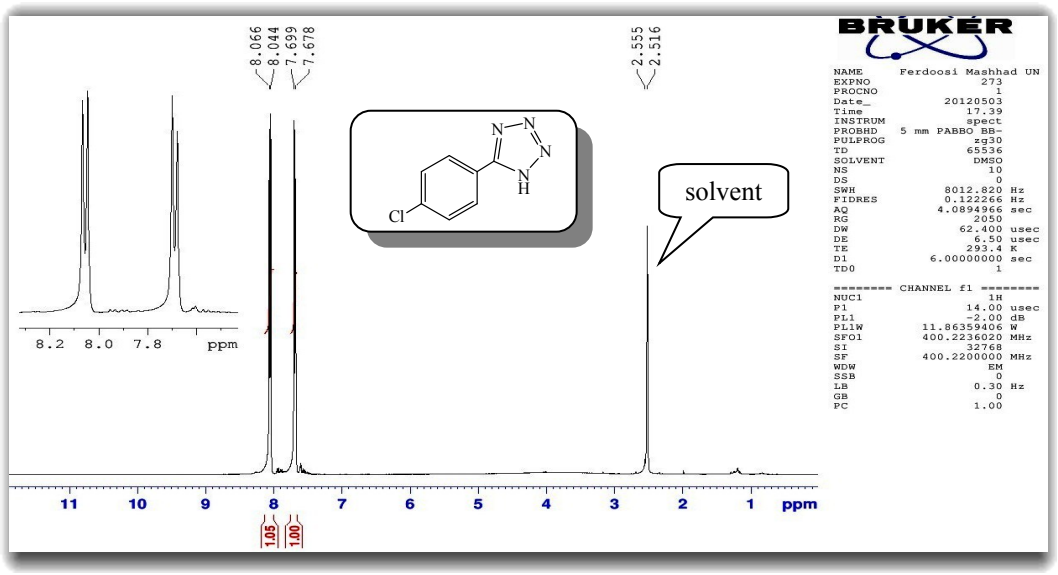

Figure 8: ^{1}H NMR (400 MHz, DMSO-d_6) of 5-(4-Chlorophenyl)-1*H*-tetrazole.

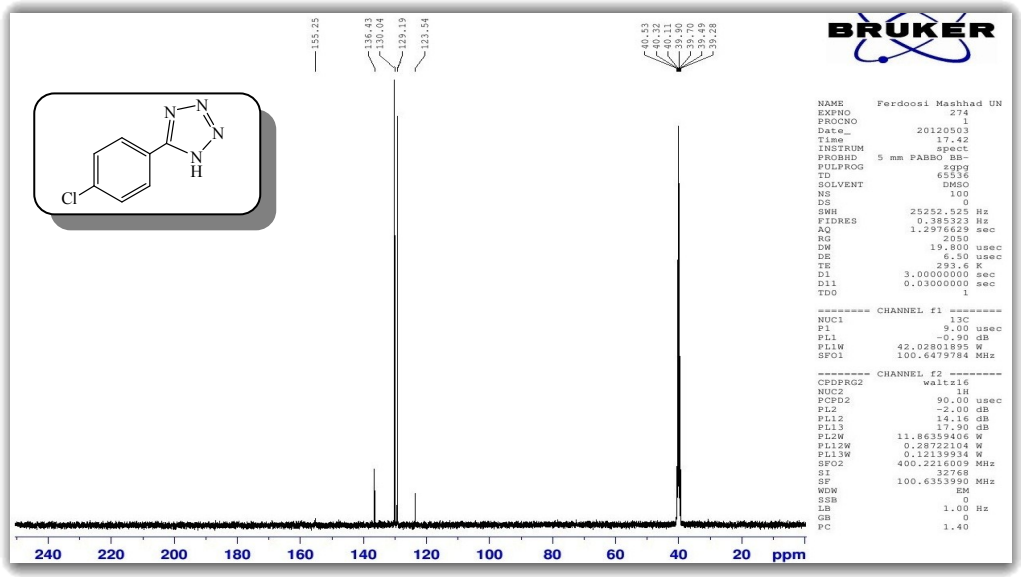

Figure 9: ^{13}C NMR (100 MHz, DMSO-d_6) of 5-(4-Chlorophenyl)-1*H*-tetrazole.

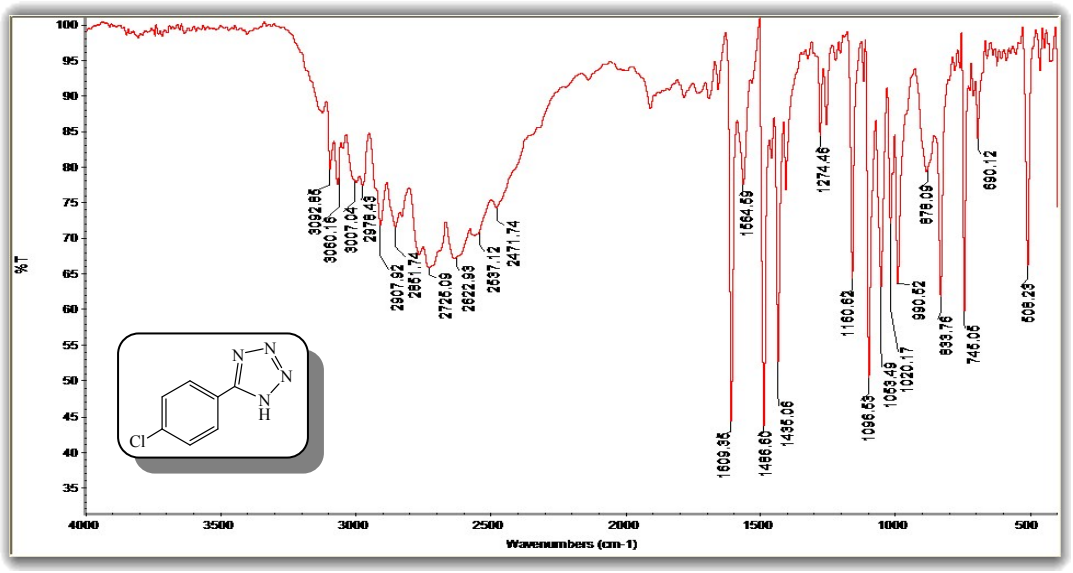

Figure 10: FT-IR (KBr) of 5-(4-Chlorohenyl)-1*H*-tetrazole.

5-(4-Nitrophenyl)-1*H*-tetrazole (Table 2, Entry 4) (0.188 g, 98 %); Yellow solid; mp 218-219 ºC (Lit.[4] 219-220 ºC). FT-IR (KBr): ν_{max}/cm^{-1} 3448, 3334, 3235, 3109, 3080, 2974, 2900, 2819, 2659, 1562, 1532, 1488, 1357, 1340, 1315, 1278 (N–N=N–), 1143 (C-N), 1106, 995, 867, 853, 730, 710; ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ 8.31 (d, J = 8.4 Hz, 2H, Ar-H), 8.46 (d, J = 8.8 Hz, 2H, Ar-H); ^{13}C NMR (100 MHz, DMSO-d_6. ppm) δ 125.1, 128.6, 131.0, 149.2, 155.9.

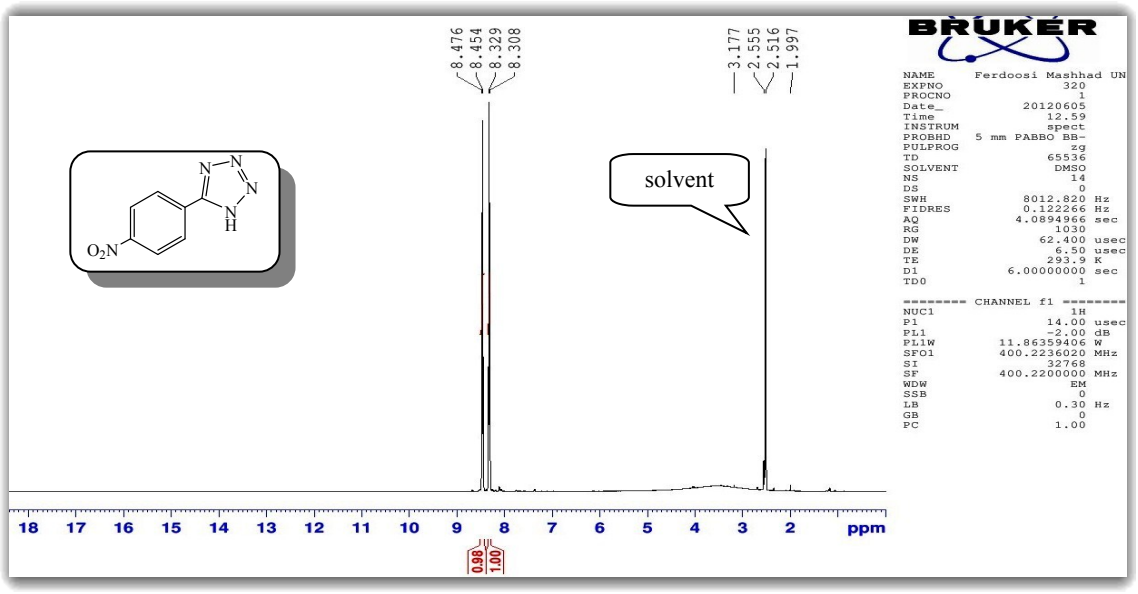

Figure 11: ^{1}H NMR (400 MHz, DMSO-d_6) of 5-(4-Nitrophenyl)-1*H*-tetrazole.

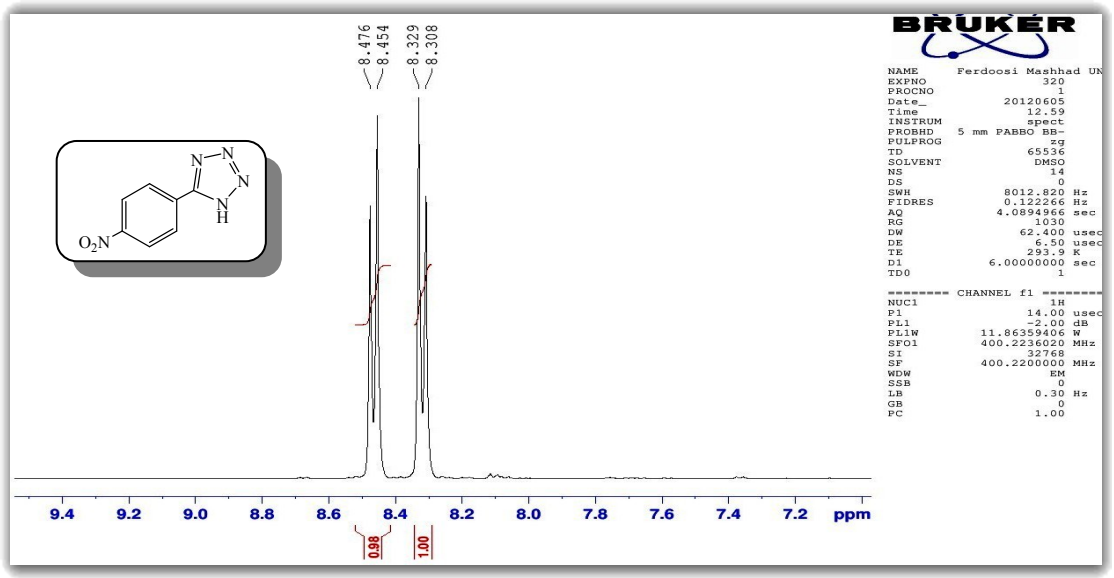

Figure 12: ^{1}H NMR (400 MHz, DMSO-d_6) of 5-(4-Nitrophenyl)-1*H*-tetrazole expanded.

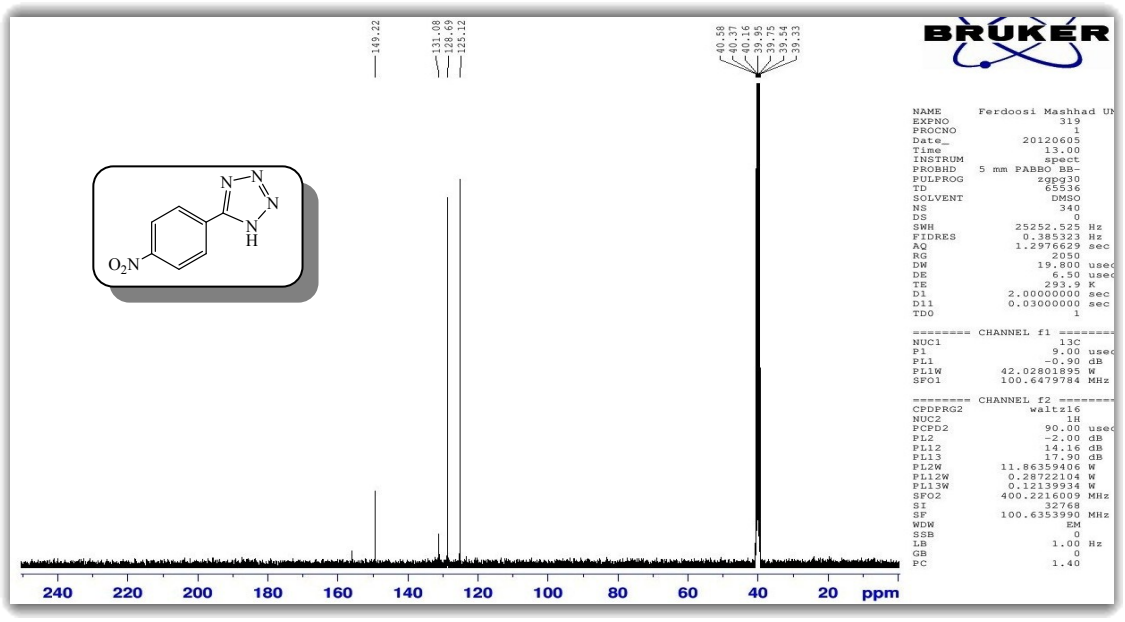

Figure 13: ^{13}C NMR (100 MHz, DMSO-d_6) of 5-(4-Nitrophenyl)-1*H*-tetrazole.

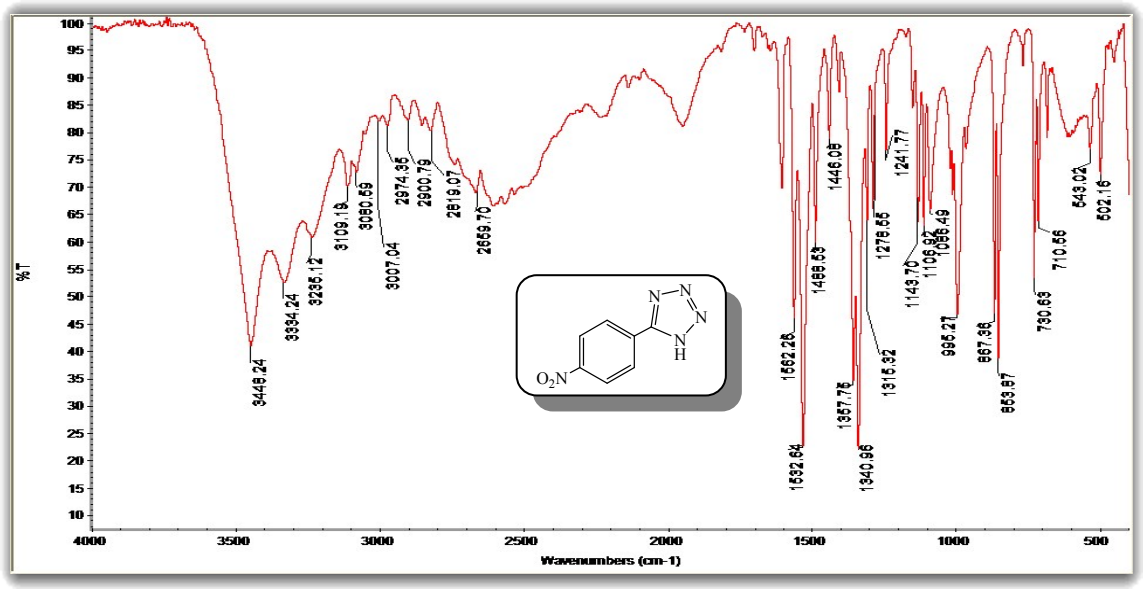

Figure 14: FT-IR (KBr) of 5-(4-Nitrophenyl)-1*H*-tetrazole.

4-(1*H*-tetrazol-5-yl)benzonitrile (Table 2, Entry 5) (0.162 g, 95 %); White solid; mp 190-191 ºC (Lit.[4] 192 ºC); FT-IR (KBr): ν_{max}/cm^{-1} 3150, 3092, 3013, 2928, 2861, 2758, 2610, 2231, 1585, 1560, 1494, 1433, 1279 (N–N=N–), 1153 (C-N), 1014, 976, 944, 850, 749, 554; ^{1}H NMR (100 MHz, CD_3CN, ppm) δ 7.90 (d, *J* = 7.5 Hz, 2H, Ar-H), 8.20 (d, *J* = 7.5 Hz, 2H, Ar-H); ^{13}C NMR (100 MHz, DMSO-d_6, ppm) δ 113.8, 118.6, 128.0, 129.1, 133.7, 155.6; MS, *m/z* (%): 171 [M^+], 142 (100) [$M^+ - N_2$], 114 [$M^+ - 2N_2$].

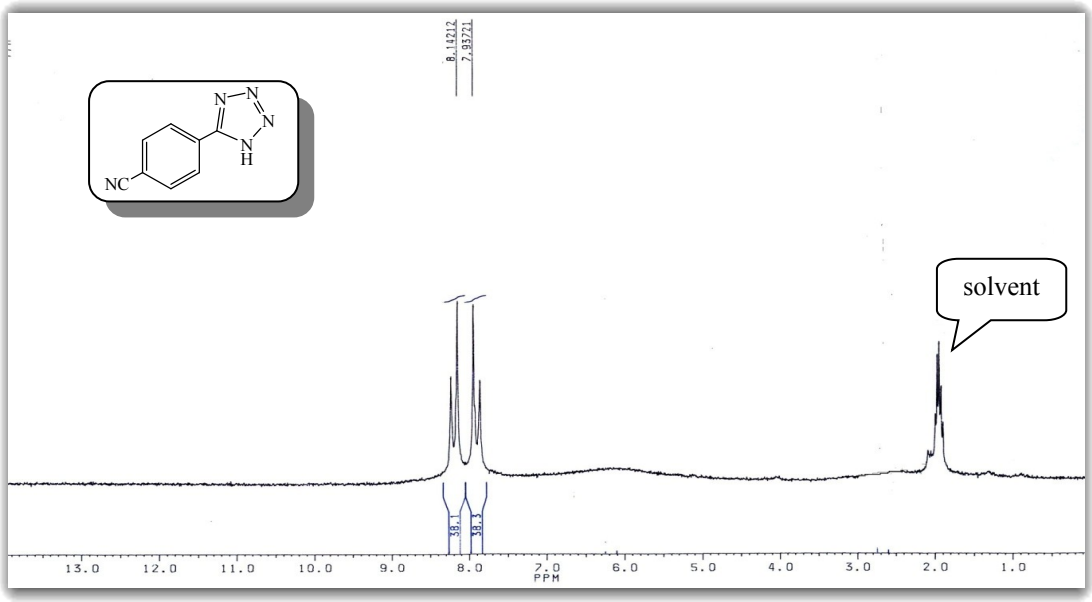

Figure 15: ^{1}H NMR (100 MHz, CD_3CN) of 4-(1*H*-tetrazol-5-yl)benzonitrile.

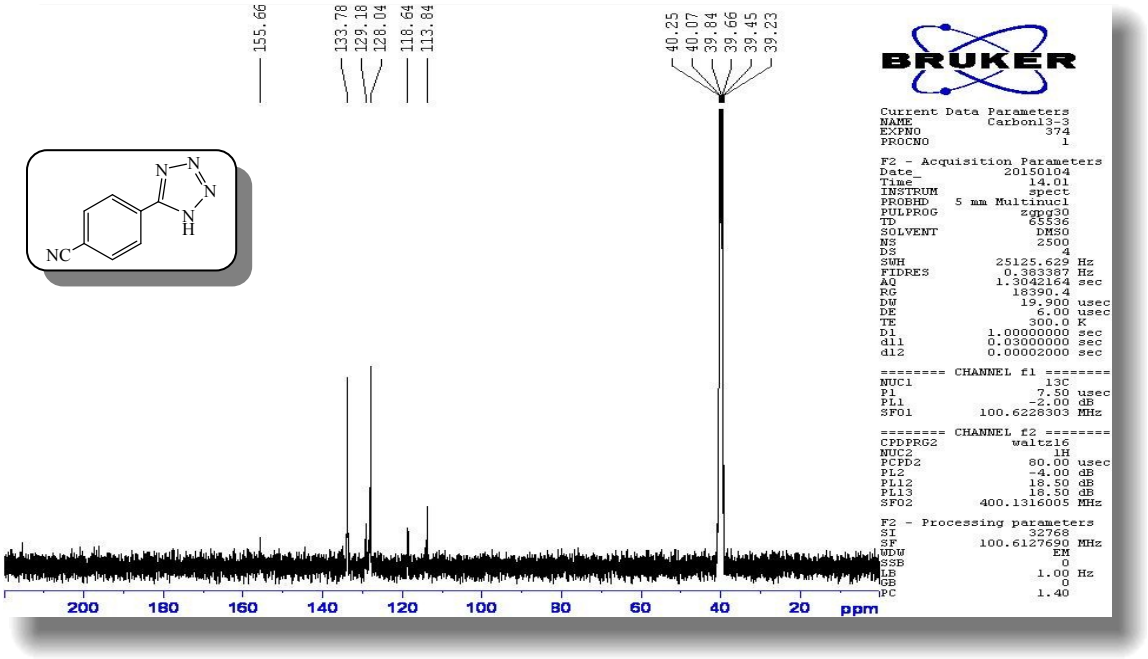

Figure 16: ^{13}C NMR (100 MHz, DMSO-d_6) of 4-(1*H*-tetrazol-5-yl)benzonitrile.

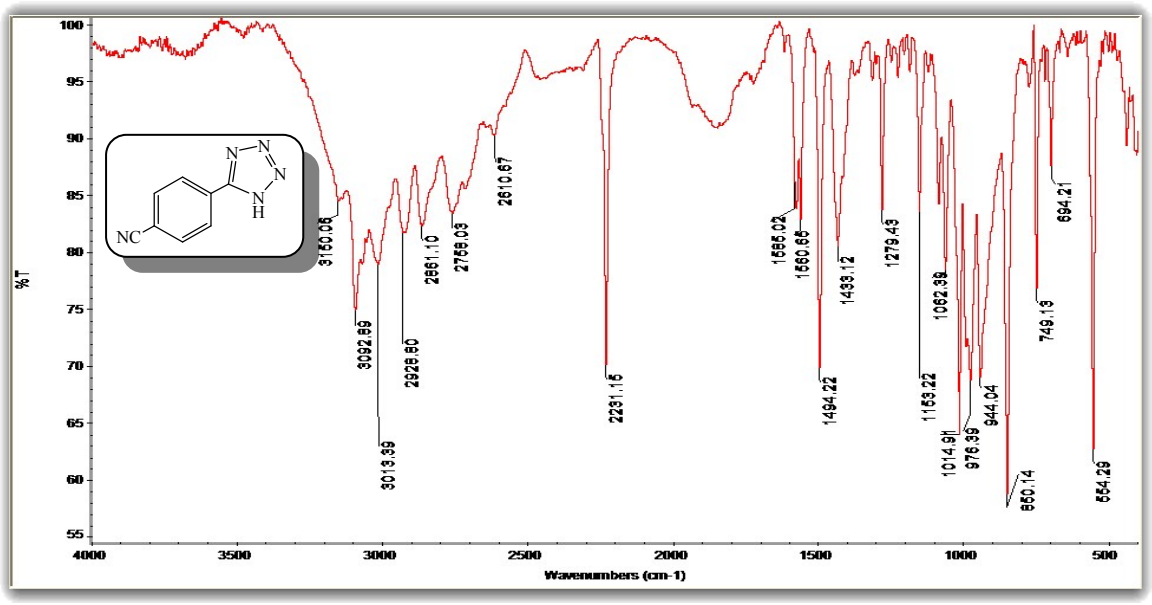

Figure 17: FT-IR (KBr) of 4-(1*H*-tetrazol-5-yl)benzonitrile.

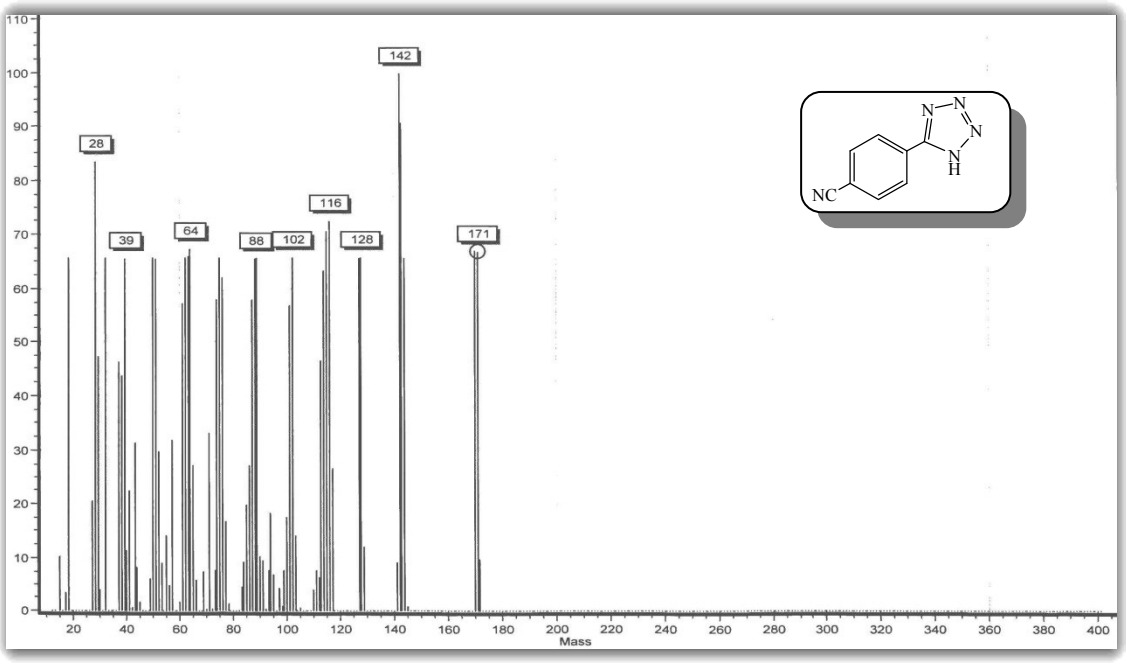

Figure 18: Mass spectrum of 4-(1*H*-tetrazol-5-yl)benzonitrile.

4-Nitro-2-(1*H*-tetrazol-5-yl)benzenamine (Table 2, Entry 6) (0.198 g, 94 %); White solid; mp 268-270 ºC (Lit.[5] 270-271 ºC); FT-IR (KBr): ν_{max}/cm^{-1} 3411, 3321, 3199, 3084, 2937, 1645, 1616, 1572, 1477, 1325, 1278 (N–N=N–), 1141(C-N), 1041, 910, 831, 751, 722; ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ 7.00 (d, J = 9.2 Hz, 1H, Ar-H), 7.94 (br s, 1H, NH), 8.10 (dd, J = 9.2, J = 2,4 Hz, 1H, Ar-H), 8.81 (d, J = 2.4 Hz, 1H, Ph); ^{13}C NMR (100 MHz, DMSO-d_6, ppm) δ 104.4, 116.3, 126.1, 127.6, 136.3, 153.0, 154.4.

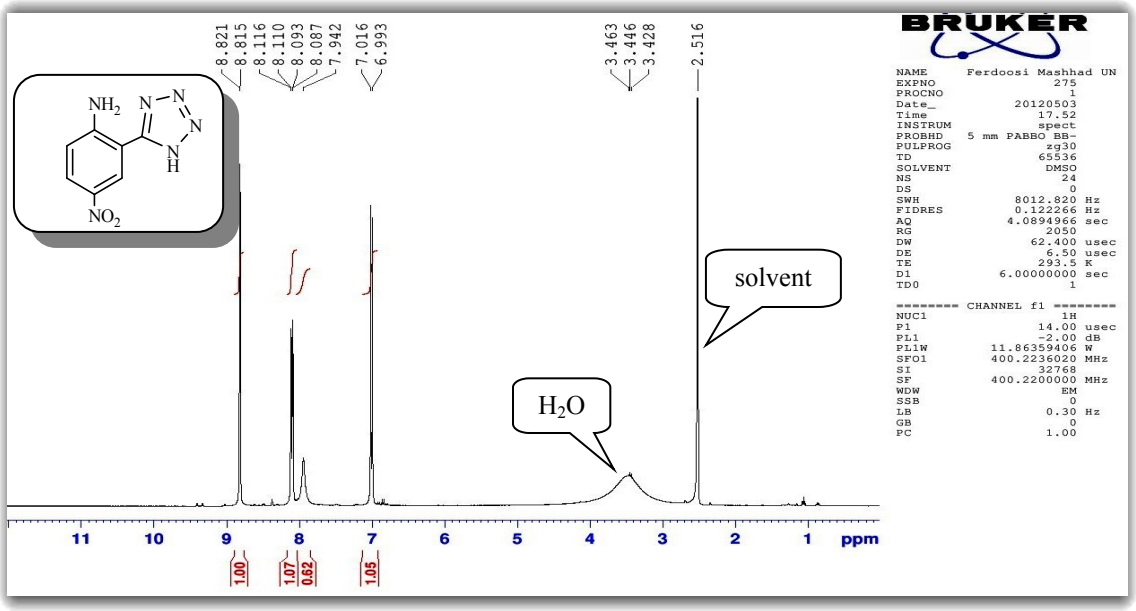

Figure 19: [1]H NMR (400 MHz, DMSO-d_6) of 4-Nitro-2-(1*H*-tetrazol-5-yl)benzenamine.

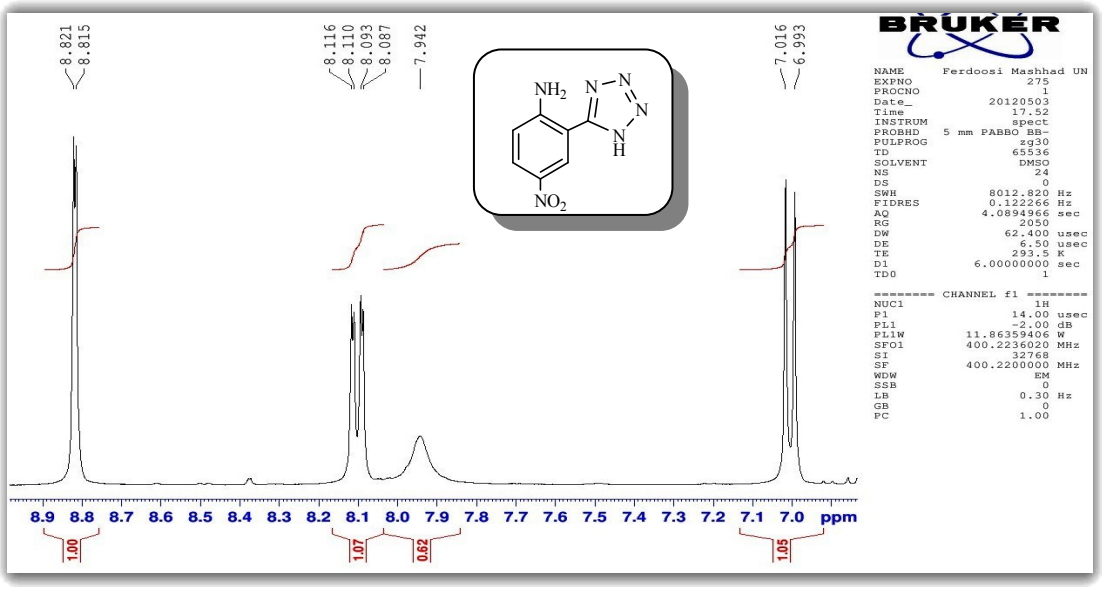

Figure 20: [1]H NMR (400 MHz, DMSO-d_6) of 4-Nitro-2-(1*H*-tetrazol-5-yl)benzenamine expanded.

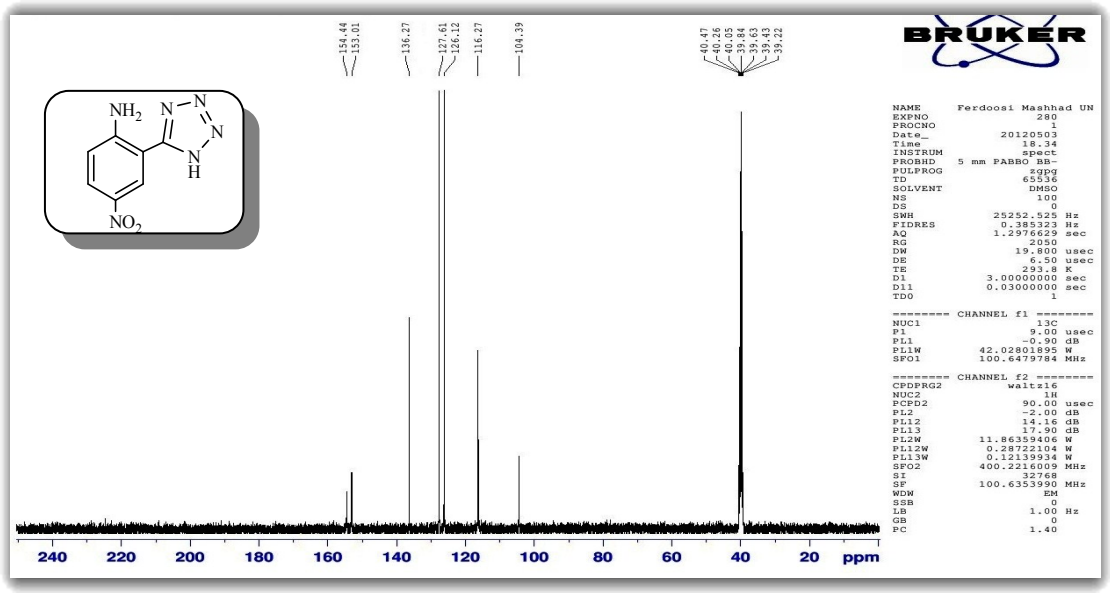

Figure 21: ^{13}C NMR (100 MHz, DMSO-d_6) of 4-Nitro-2-(1*H*-tetrazol-5-yl)benzenamine.

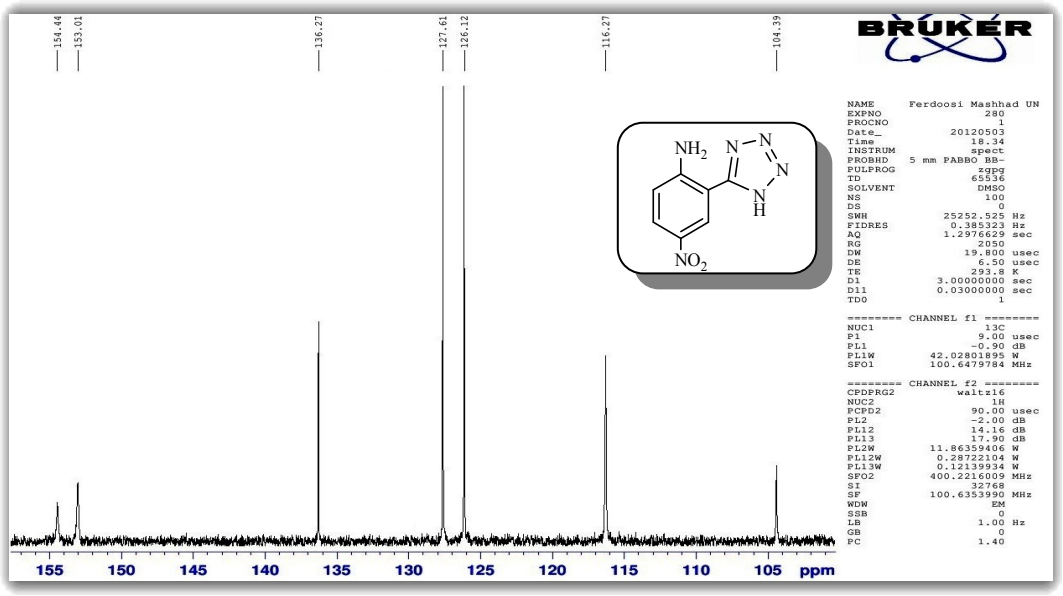

Figure 22: ^{13}C NMR (100 MHz, DMSO-d_6) of 4-Nitro-2-(1*H*-tetrazol-5-yl)benzenamine expanded.

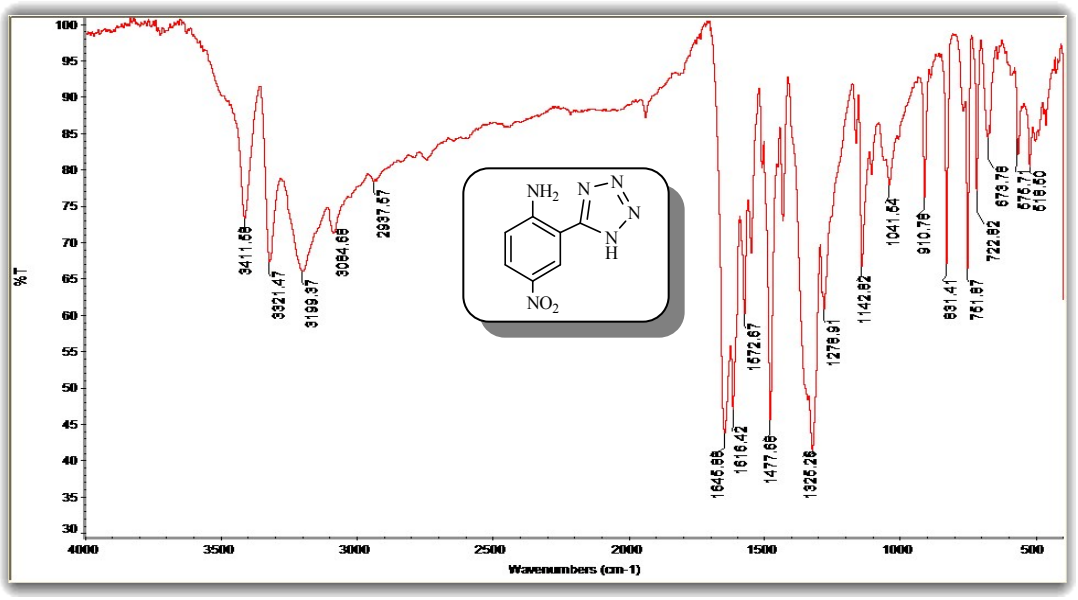

Figure 23: FT-IR (KBr) of 4-Nitro-2-(1*H*-tetrazol-5-yl)benzenamine.

5-(3,5-Dimethoxyphenyl)-1*H*-tetrazole (Table 2, Entry 7) (0.185 g, 90 %); White solid; mp 204-205 ºC (Lit.[6] 204-206 ºC); FT-IR (KBr): ν_{max}/cm^{-1} 3129, 3064, 3011, 2975, 2941, 2843, 2757, 2712, 2634, 1605, 1562, 1480, 1430, 1287 (N–N=N–), 1208, 1167, 1162 (C-N), 1054, 827, 747; ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ 3.84 (s, 6H, -OMe), 6.73 (t, J = 2 Hz, 1H, Ar-H), 7.21 (d, J = 2 Hz, 2H, Ar-H), 16.91 (br s, 1H, NH); ^{13}C NMR (100 MHz, DMSO-d_6, ppm) δ 56.0, 103.4, 105.3, 125.7, 156.3, 161.5; MS, *m/z* (%): 206 [M^+].

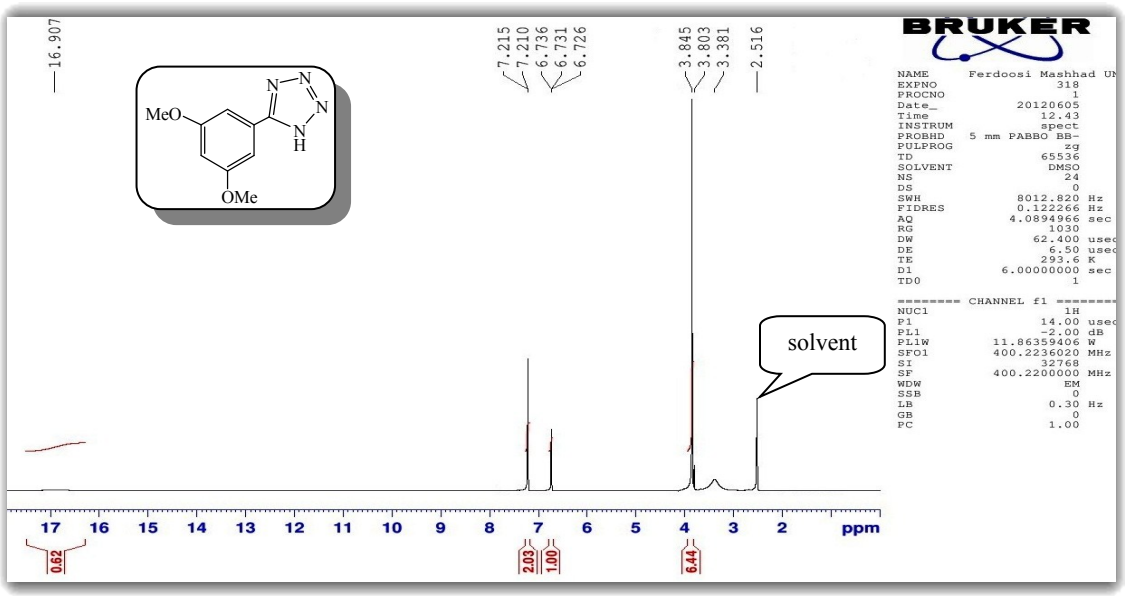

Figure 24: ^{1}H NMR (400 MHz, DMSO-d_6) of 5-(3, 5-Dimethoxyphenyl)-1*H*-tetrazole.

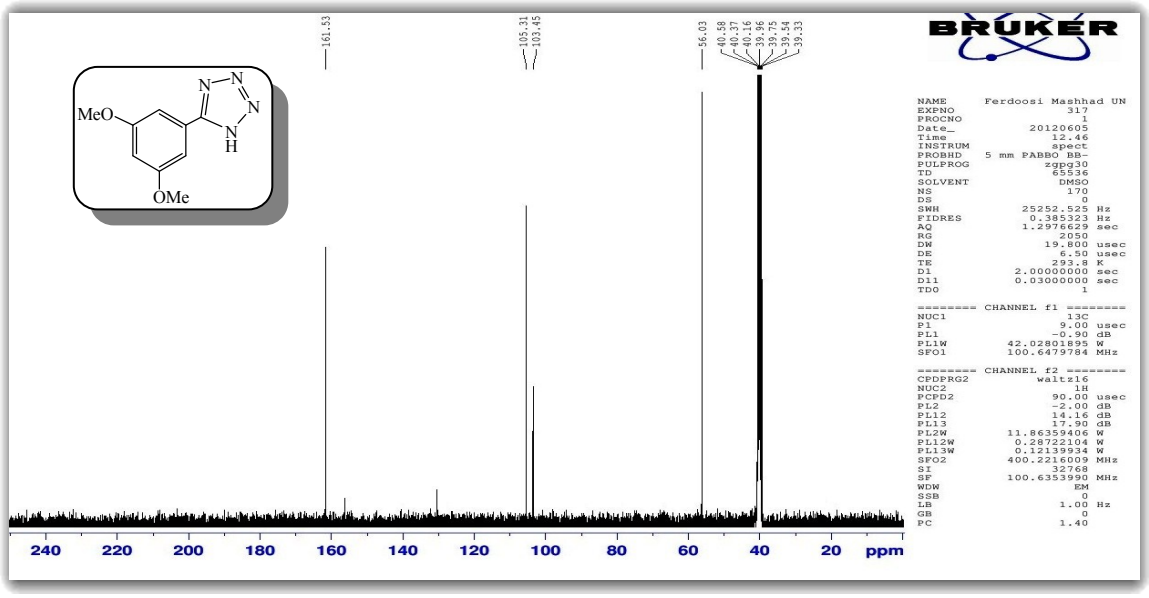

Figure 25: ^{13}C NMR (100 MHz, DMSO-d_6) of 5-(3,5-Dimethoxyphenyl)-1*H*-tetrazole.

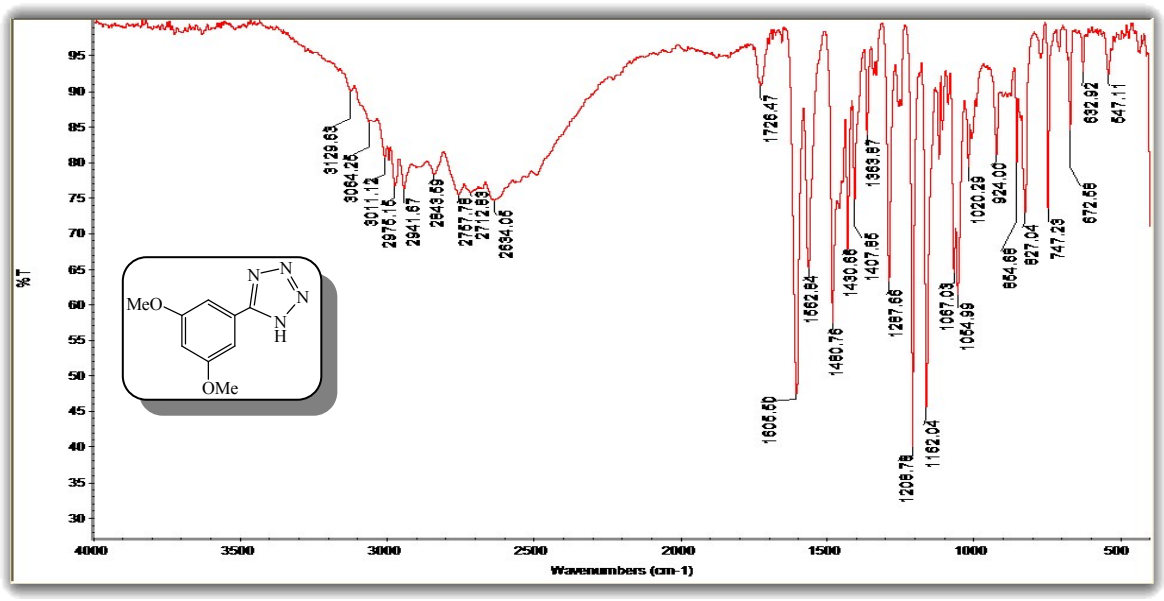

Figure 26: FT-IR (KBr) of 5-(3,5-Dimethoxyphenyl)-1*H*-tetrazole.

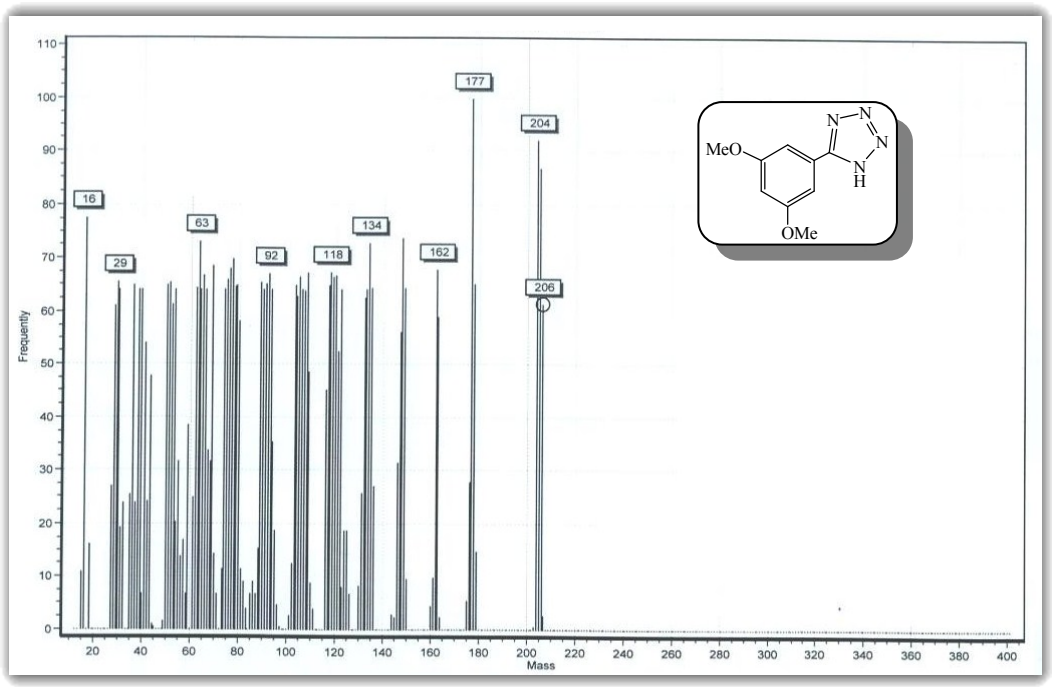

Figure 27: Mass spectrum of 5-(3, 5-Dimethoxyphenyl)-1*H*-tetrazole.

5-*m*-Tolyl-1*H*-tetrazole (Table 2, Entry 8) (0.147 g, 92 %); White solid; mp 149 -150 ºC (Lit.[7] 151-152 ºC); FT-IR (KBr): ν_{max}/cm^{-1} 3120, 3061, 2979, 2917, 2871, 2746, 2611, 2490, 1728, 1605, 1565, 1486, 1463, 1270 (N–N=N–), 1150 (C-N), 1060, 1038, 802, 741, 705, 687; ^{1}H NMR (300 MHz, DMSO-d_6, ppm) δ 2.41 (s, 3H, CH_3), 7.41 (d, J = 7.5 Hz, 1H, Ar-H), 7.50 (t, J = 7.5 Hz, 1H, Ar-H), 7.82-7.88 (m, 2H, Ar-H); ^{13}C NMR (75 MHz, DMSO-d_6, ppm) δ 21.3, 124.4, 124.5, 127.8, 129.8, 132.3, 139.3, 155.6.

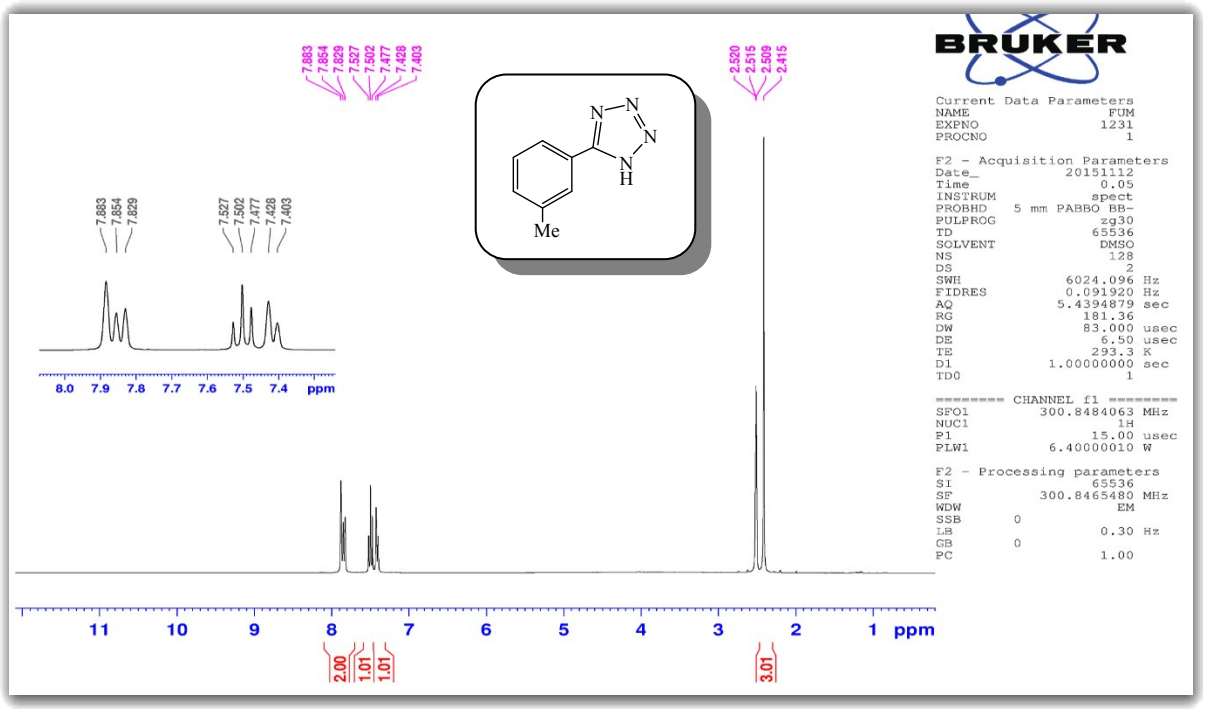

Figure 28: ^{1}H NMR (300 MHz, DMSO-d_6) of 5-*m*-Tolyl-1*H*-tetrazole.

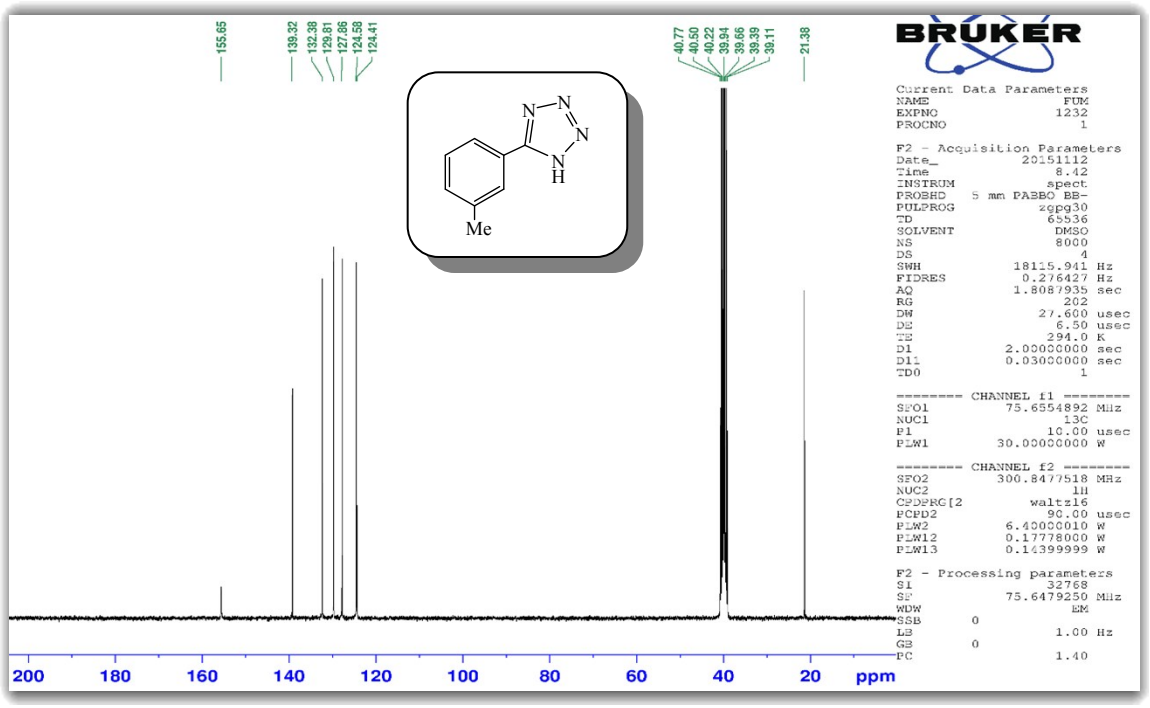

Figure 29: ^{13}C NMR (75 MHz, DMSO-d_6) of 5-*m*-Tolyl-1*H*-tetrazole.

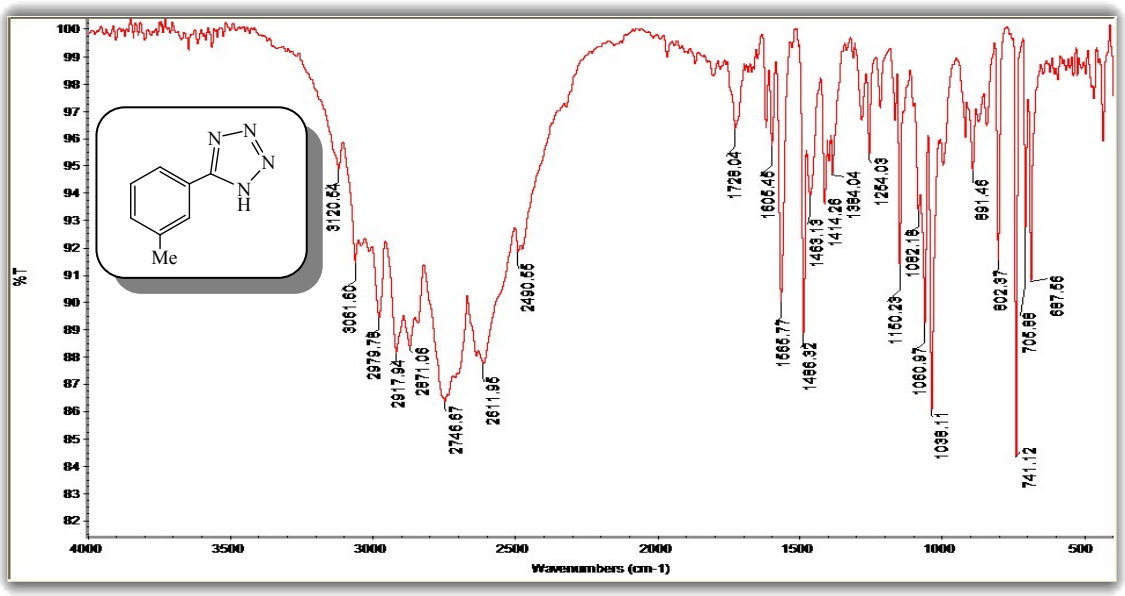

Figure 30: FT-IR (KBr) of 5-*m*-Tolyl-1*H*-tetrazole.

4-(1*H*-tetrazol-5-yl)phenol (Table 2, Entry 9) (0.138 g, 85 %); White solid; mp 218-219 ºC (Lit.[8] 219 ºC); FT-IR (KBr): ν_{max}/cm^{-1} 3252, 3101, 3066, 3019, 3000-2200, 1615, 1599, 1511, 1466, 1413, 1282 (N–N=N–), 1179 (C-N), 832, 752, 514; ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ 6.97 (d, J = 8.4 Hz, 2H, Ar-H), 7.87 (d, J = 8.8 Hz, 2H, Ar-H), 10.20 (br s, 1H, OH); ^{13}C NMR (100 MHz, DMSO-d_6, ppm) δ 115.0, 116.6, 129.2, 155.2, 160.5.

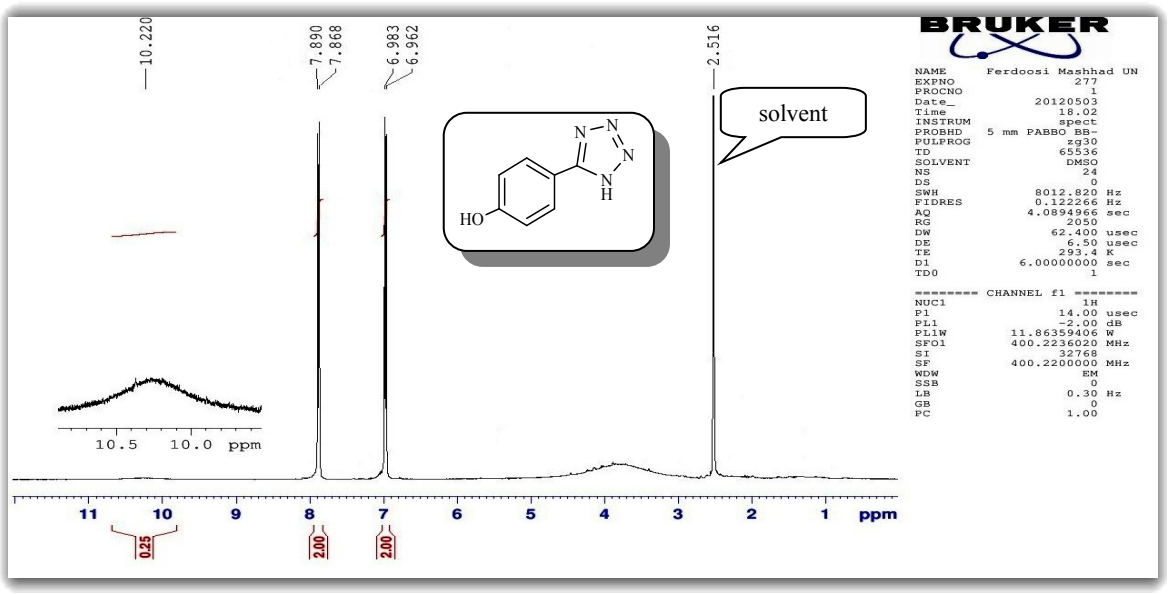

Figure 31: ^{1}H NMR (400 MHz, DMSO-d_6) of 4-(1*H*-tetrazol-5-yl) phenol.

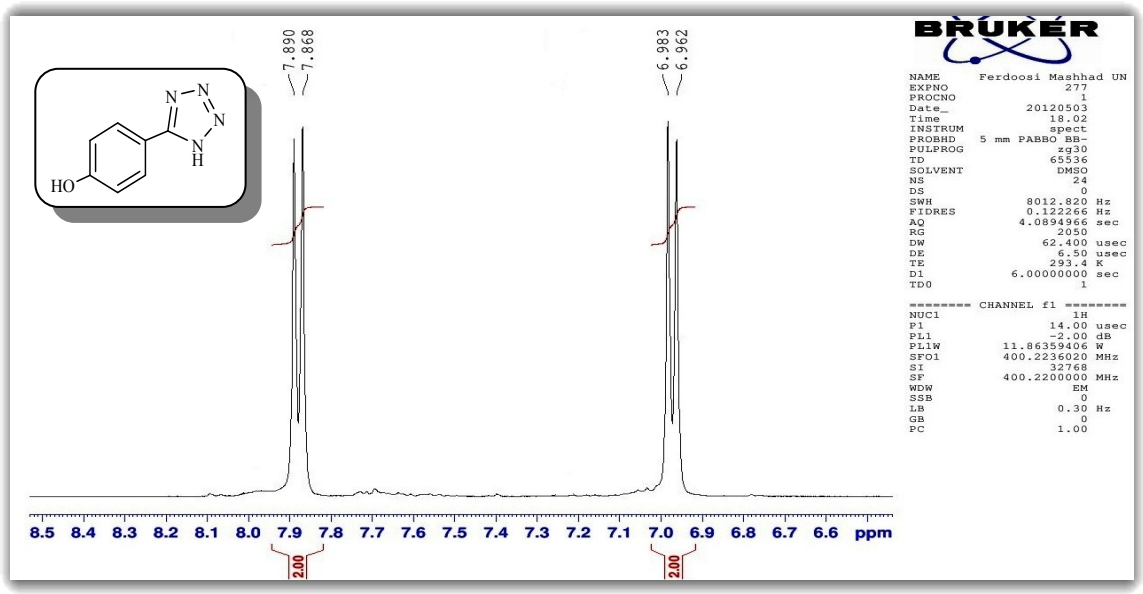

Figure 32: ^{1}H NMR (400 MHz, DMSO-d_6) of 4-(1*H*-tetrazol-5-yl)phenol expanded.

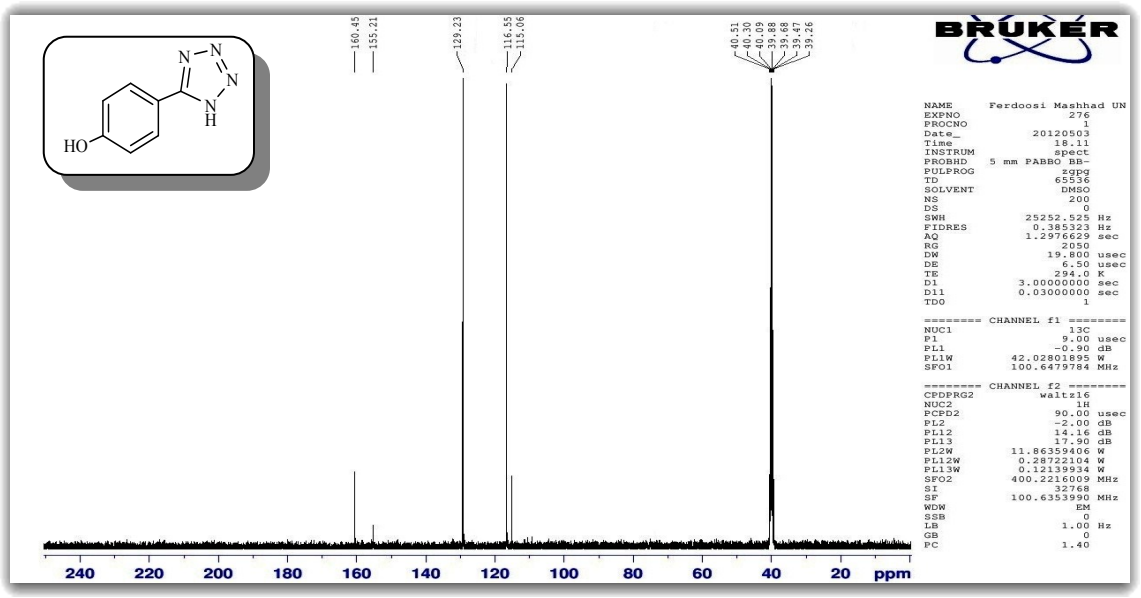

Figure 33: ^{13}C NMR (100 MHz, DMSO-d_6) of 4-(1*H*-tetrazol-5-yl) phenol.

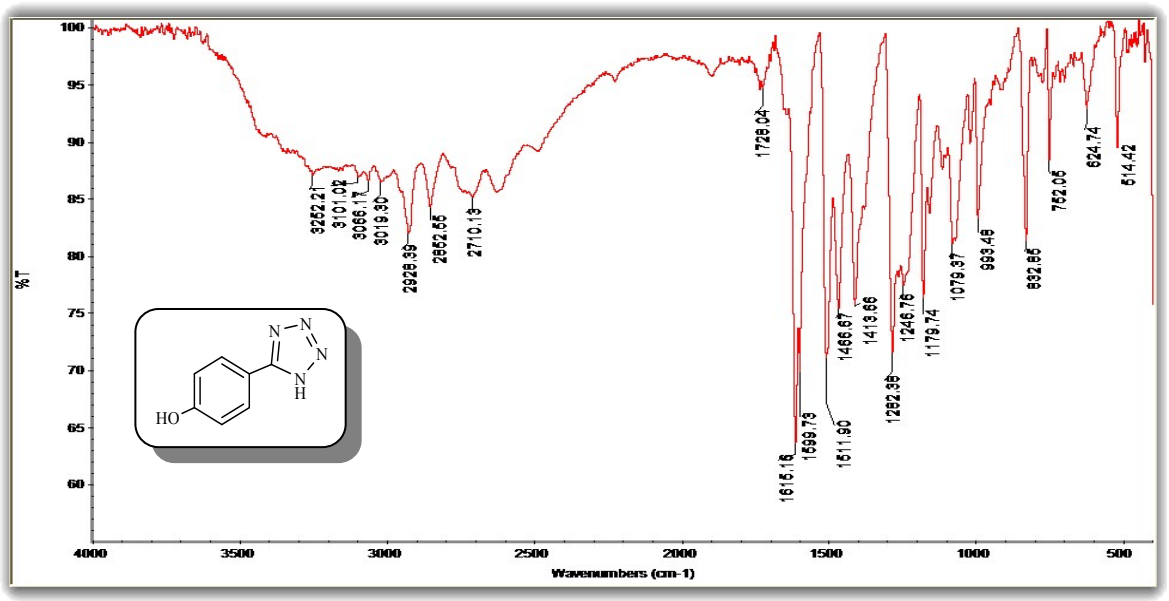

Figure 34: FT-IR (KBr) of 4-(1*H*-tetrazol-5-yl) phenol.

2-(1*H*-tetrazol-5-yl)phenol (Table 2, Entry 10) (0.149 g, 92 %); White solid; mp 218-219 ºC (Lit.[8] 219 ºC): FT-IR (KBr): ν_{max}/cm^{-1} 3175, 2970, 2855, 2721, 2561, 1616, 1545, 1490, 1467, 1392, 1365, 1298, 1266 (N–N=N–), 1155 (C-N), 1071, 999, 837, 745, 465; ^{1}H NMR (300 MHz, DMSO-d_6, ppm) δ 6.99-7.10 (m, 2H, Ar-H), 7.42 (t, J = 7.5 Hz, 1H, Ar-H), 8.00 (d, J = 7.5 Hz, 1H, Ar-H); ^{13}C NMR (75 MHz, DMSO-d_6, ppm) δ 110.9 , 116.7, 120.2, 129.4, 133.0, 152.0, 155.6; MS, *m/z* (%): 162 [M^+], 161 [M^+ - H], 133 (100) [M^+ - N_2], 105 [M^+ - $2N_2$].

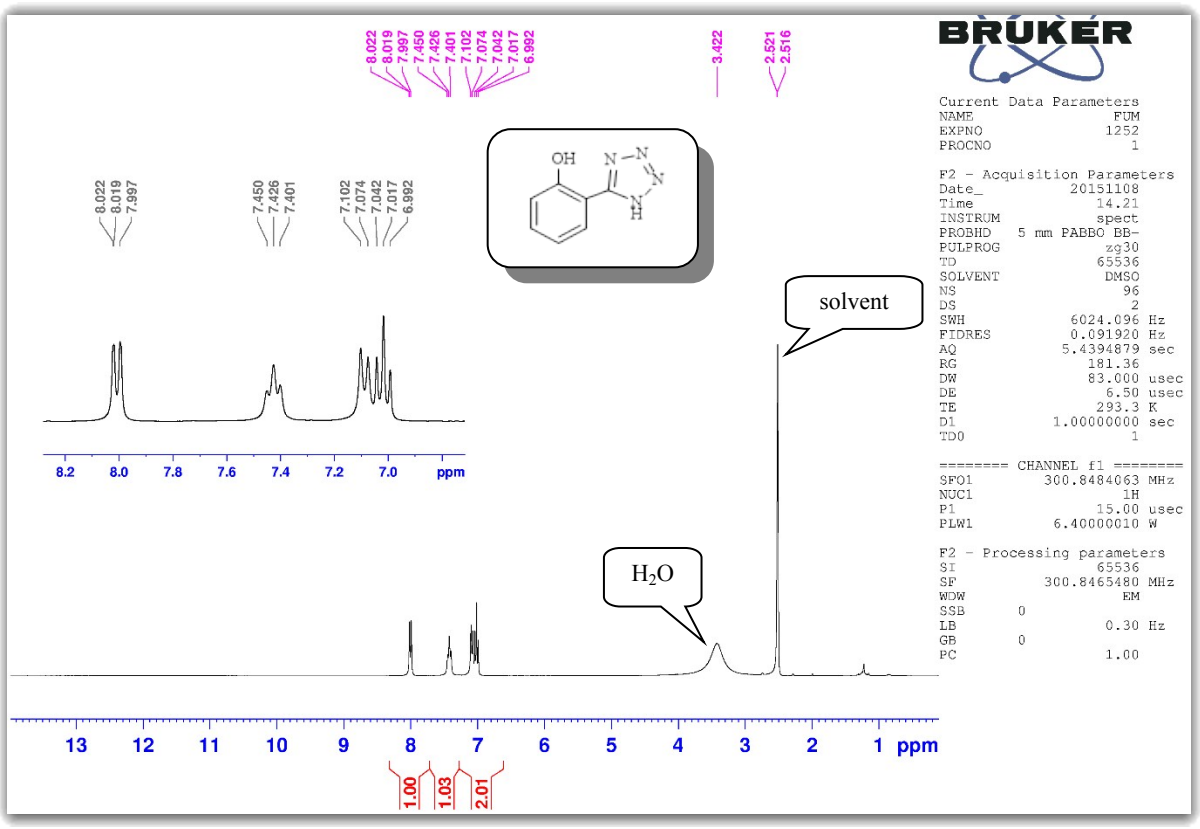

Figure 35: ^{1}H NMR (300 MHz, DMSO-d_6) of 2-(1*H*-tetrazol-5-yl)phenol.

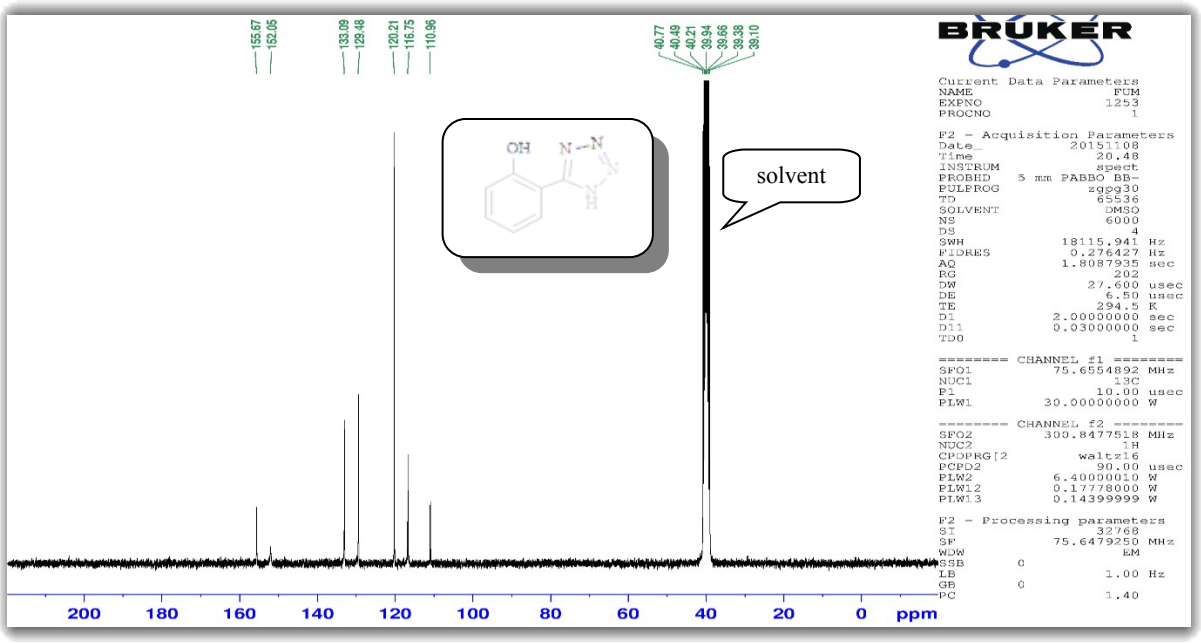

Figure 36: ^{13}C NMR (75 MHz, DMSO-d_6) of 2-(1*H*-tetrazol-5-yl)phenol.

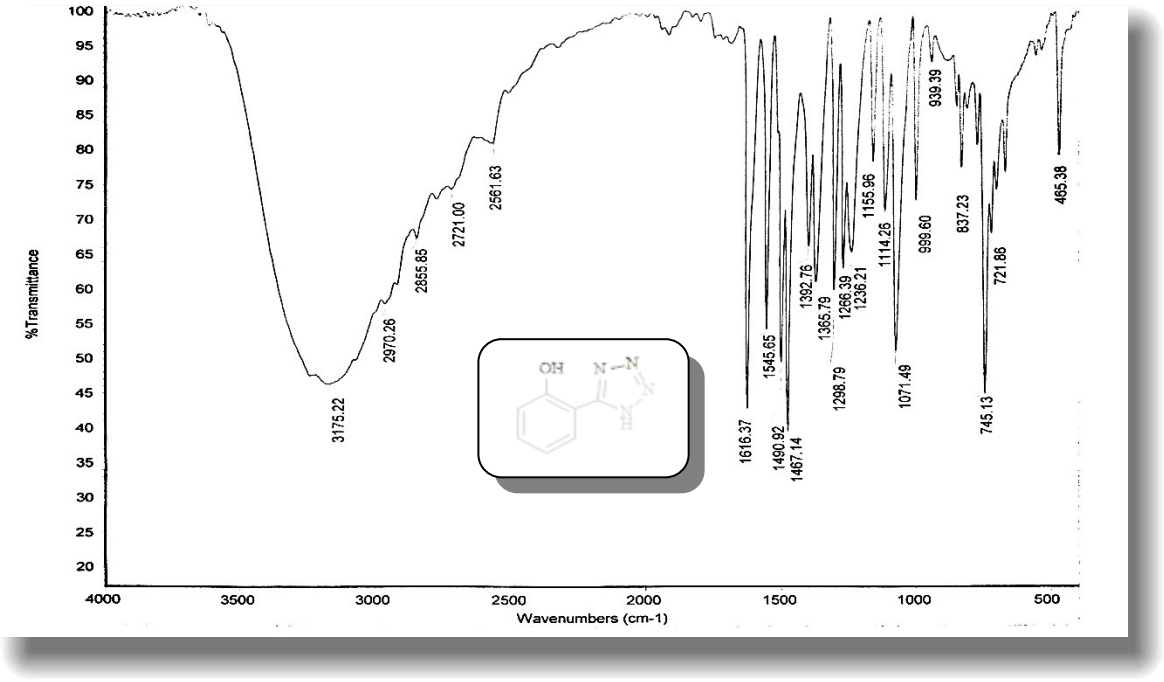

Figure 37: FT-IR (KBr) of 2-(1*H*-tetrazol-5-yl)phenol.

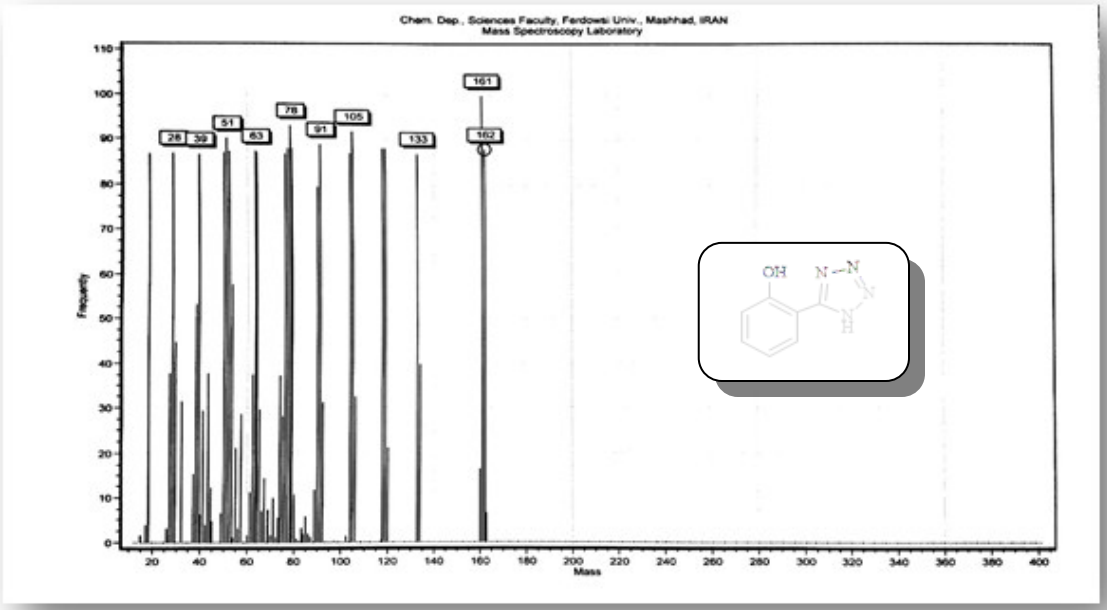

Figure 38: Mass spectrum of 2-(1*H*-tetrazol-5-yl)phenol.

5-(4-Ethoxyphenyl)-1*H*-tetrazole (Table 2, Entry 11) (0.168 g, 88 %); White solid; mp 234-235 ºC (Lit.[9] 234-235 ºC); FT-IR (KBr): ν_{max}/cm^{-1} 3145, 3101, 3060, 2986, 2921, 2868, 2737, 2647, 1613, 1505, 1470, 1394, 1293, 1262 (N–N=N–), 1189 (C-N), 1056, 1041, 923, 827, 751, 653, 522; ^{1}H NMR (300 MHz, DMSO-d_6, ppm) δ 1.36 (t, J = 6.9 Hz, 3H, -OEt), 4.12 (q, J = 6.9 Hz, 2H, -OEt), 7.15 (d, J = 7.2 Hz, 2H, Ar-H), 7.97 (d, J = 6.9 Hz, 2H, Ar-H); ^{13}C NMR (75 MHz, DMSO-d_6, ppm) δ 14.9, 63.8, 115.6, 116.4, 129.1, 155.1, 161.2.

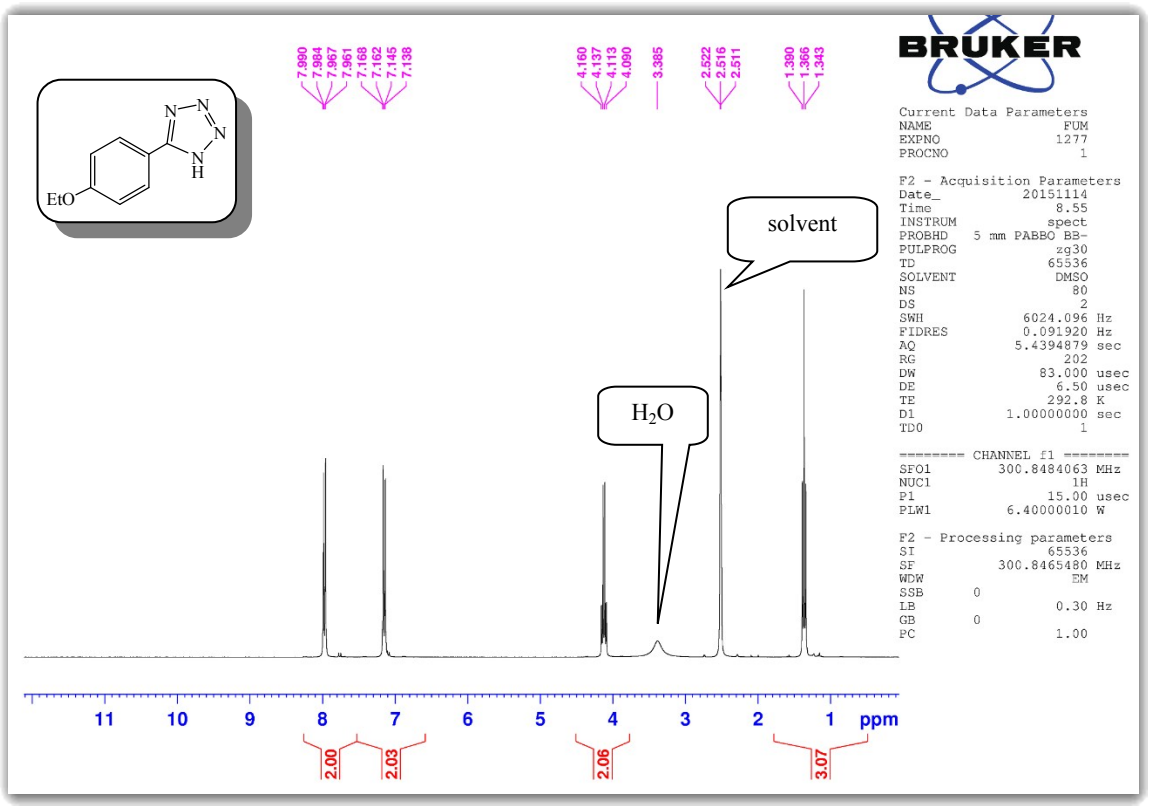

Figure 39: ^{1}H NMR (300 MHz, DMSO-d_6) of 5-(4-Ethoxyphenyl)-1*H*-tetrazole.

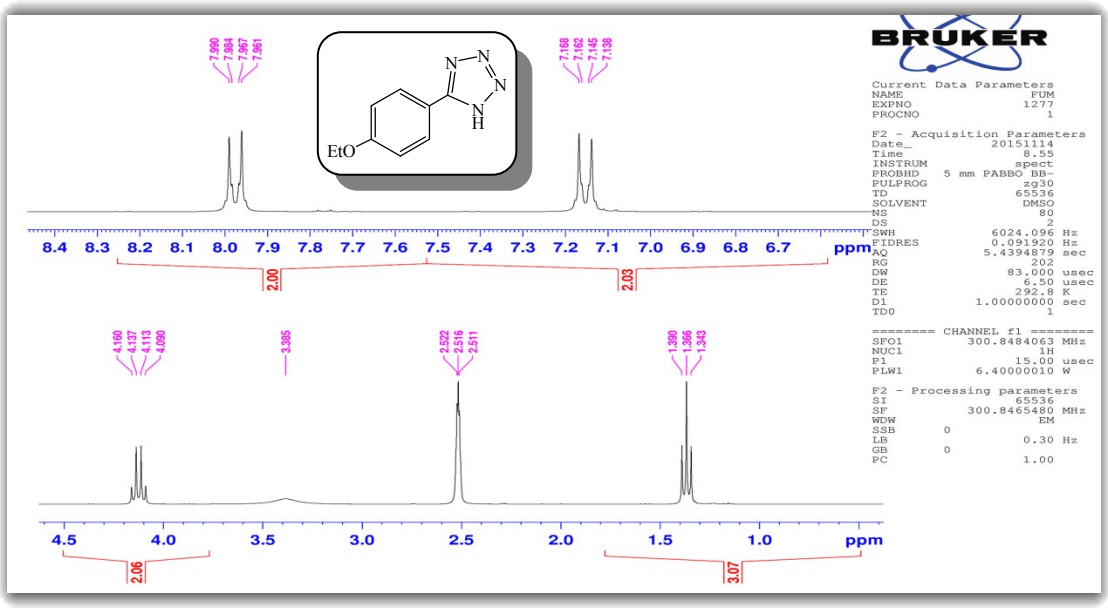

Figure 40: ^{1}H NMR (300 MHz, DMSO-d_6) of 5-(4-Ethoxyphenyl)-1*H*-tetrazole expanded.

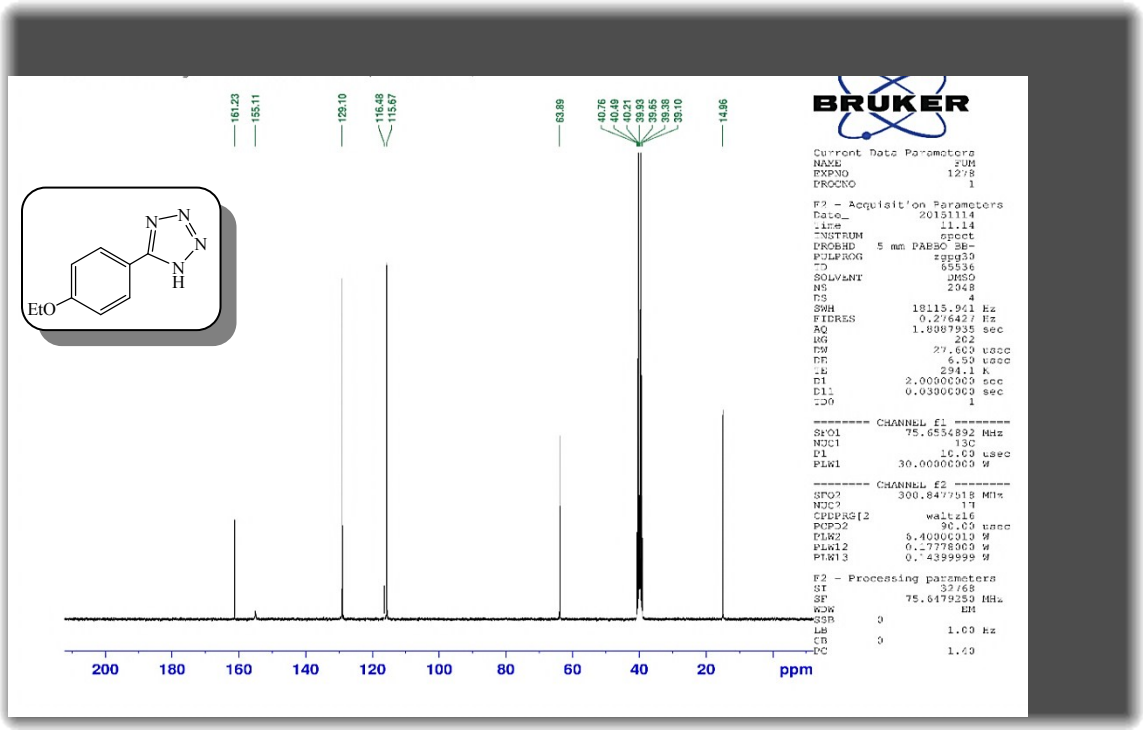

Figure 41: ^{13}C NMR (75 MHz, DMSO-d_6) of 5-(4-Ethoxyphenyl)-1*H*-tetrazole.

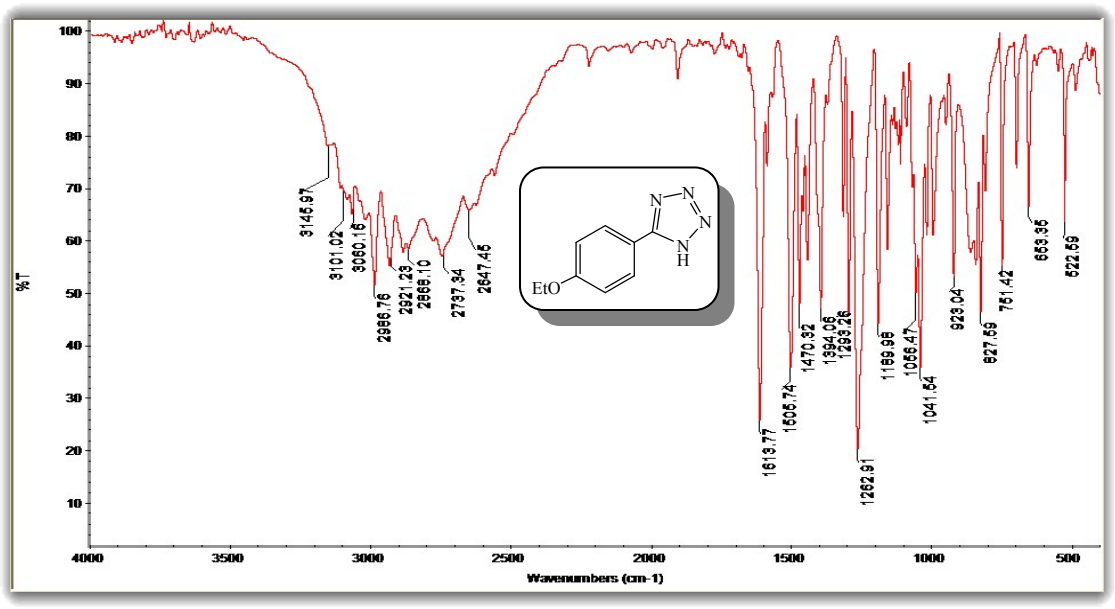

Figure 42: FT-IR (KBr) of 5-(4-Ethoxyphenyl)-1*H*-tetrazole.

5-(Phenanthren-9-yl)-1*H*-tetrazole (Table 2, Entry 12) (0.172 g, 70 %); White solid; mp 241-242 ºC (Lit.[10] 243-244 ºC); FT-IR (KBr): ν_{max}/cm^{-1} 3105, 3076, 3016, 2978, 2878, 2830, 2724, 2686, 2622, 2590, 2520, 2478, 1612, 1565, 1450, 1399, 1278 (N–N=N–), 1246, 1112 (C-N), 1053, 1038, 992, 934, 771, 737, 721, 424; ^{1}H NMR (300 MHz, DMSO-d_6. ppm) δ 7.66-7.94 (m, 4H, Ar-H), 8.16-8.36 (m, 4H, Ar-H), 8.72 (s, 1H, Ar-H); ^{13}C NMR (75 MHz, DMSO-d_6, ppm) δ 123.7, 124.4, 124.5, 125.6, 126.7, 128.4, 129.0, 129.1, 129.9, 130.0, 130.2, 130.9, 131.6, 136.8, 163.4; *m/z* (%): 246 [M^+].

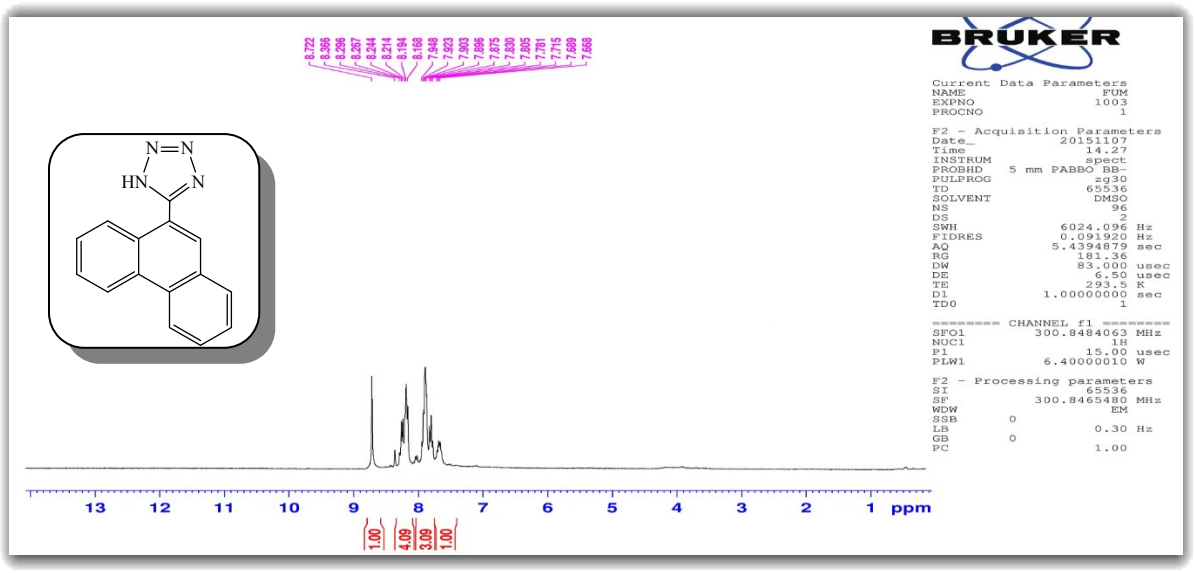

Figure 43: ^{1}H NMR (300 MHz, DMSO-d_6) of 5-(Phenanthren-9-yl)-1*H*-tetrazole.

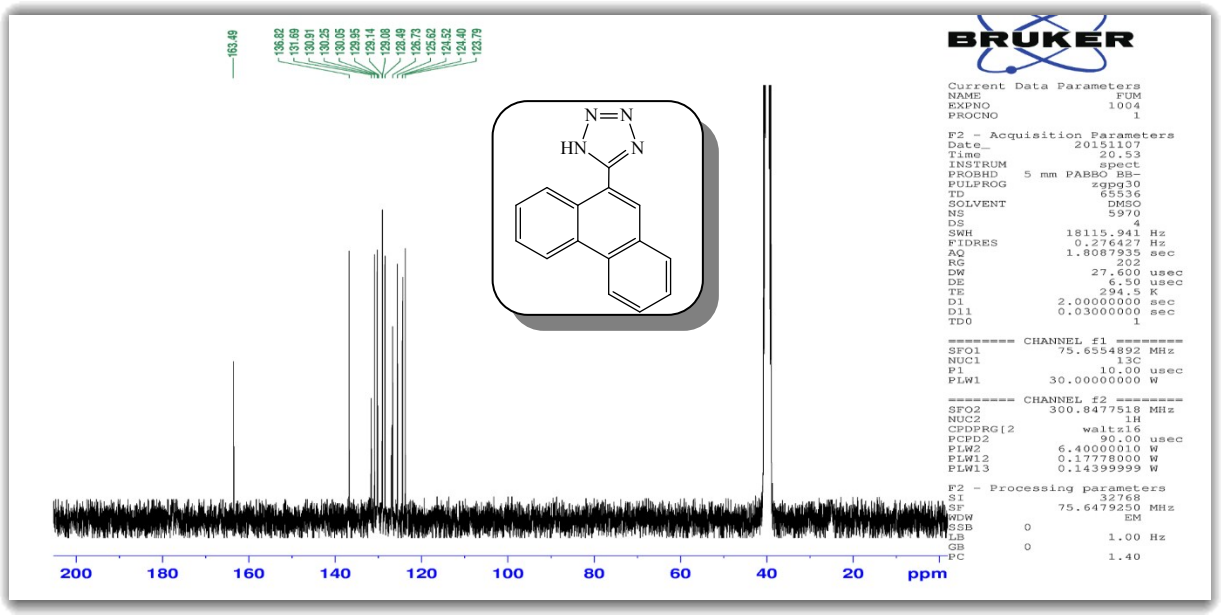

Figure 44: ^{13}C NMR (75 MHz, DMSO-d_6) of 5-(Phenanthren-9-yl)-1*H*-tetrazole.

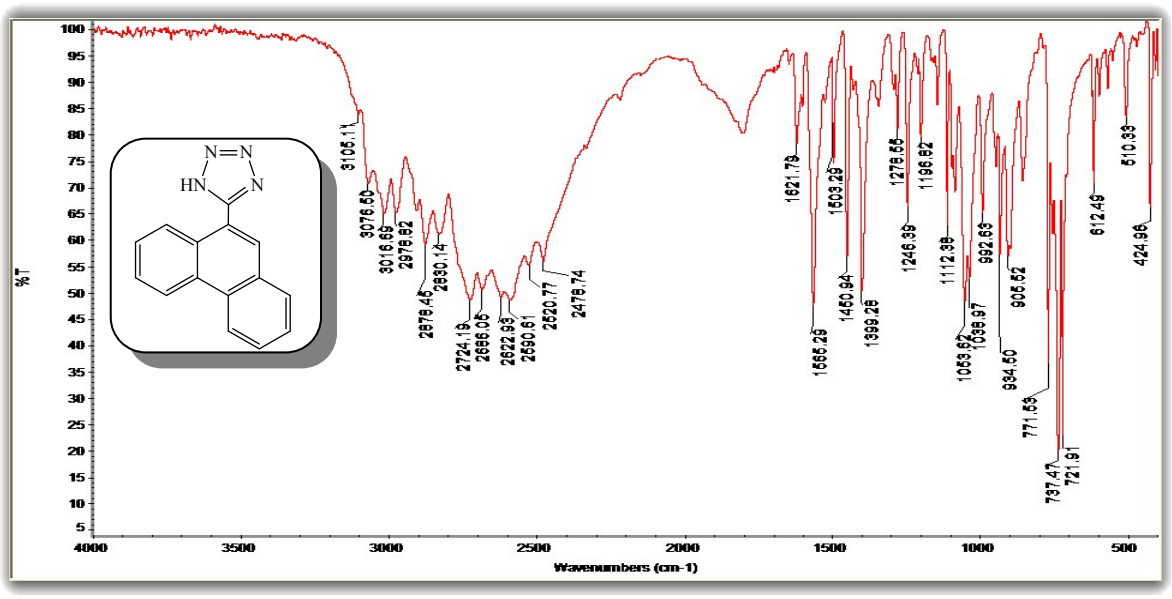

Figure 45: FT-IR (KBr) of 5-(Phenanthren-9-yl)-1*H*-tetrazole.

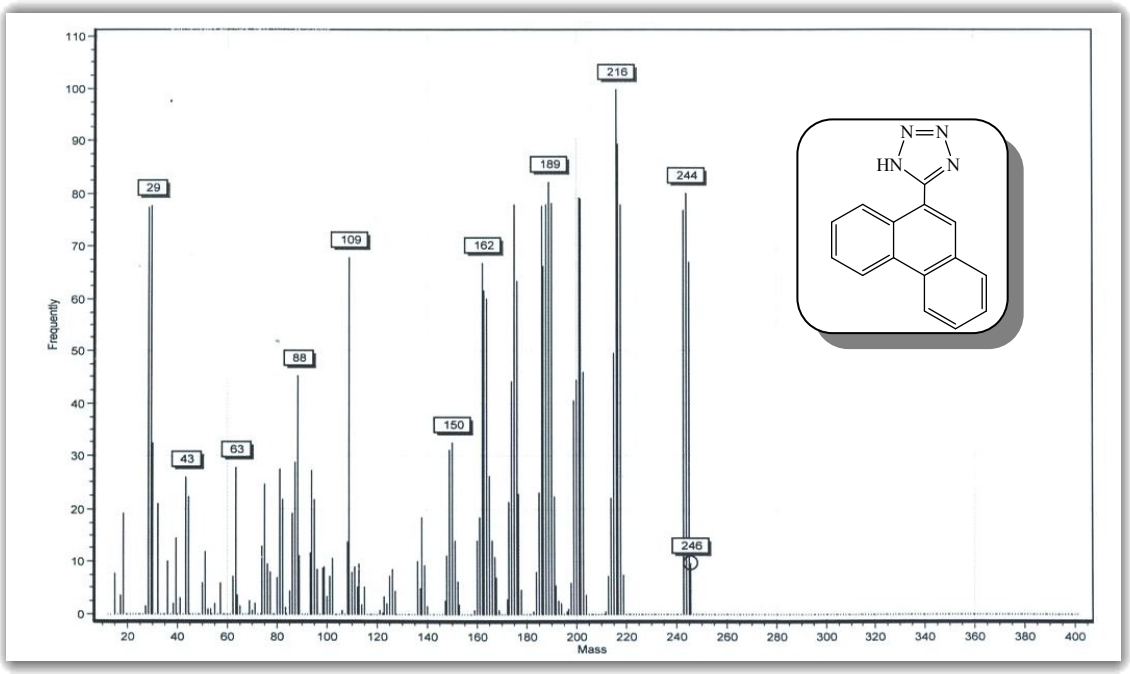

Figure 46: Mass spectrum of 5-(Phenanthren-9-yl)-1*H*-tetrazole.

4-(1*H*-tetrazol-5-yl)pyridine (Table 2, Entry 13) (0.140 g, 95 %); White solid; mp 255-258 ºC (Lit.[11] 255-258 ºC); FT-IR (KBr) ν_{max}/cm^{-1} 3485, 3264, 3099, 3035, 2966, 1624, 1529, 1435, 1388,1292 (N–N=N–),1123 (C-N), 1096, 1042, 1022, 845, 730, 674, 593, 465; ^{1}H NMR (300 MHz, DMSO-d_6, ppm) δ 7.79 (d, J = 5.1 Hz, 1H, Py), 8.35-8.82 (m, 2H, Py); ^{13}C NMR (75 MHz, DMSO-d_6, ppm) δ 122.6, 137.5, 147.6, 155.8.

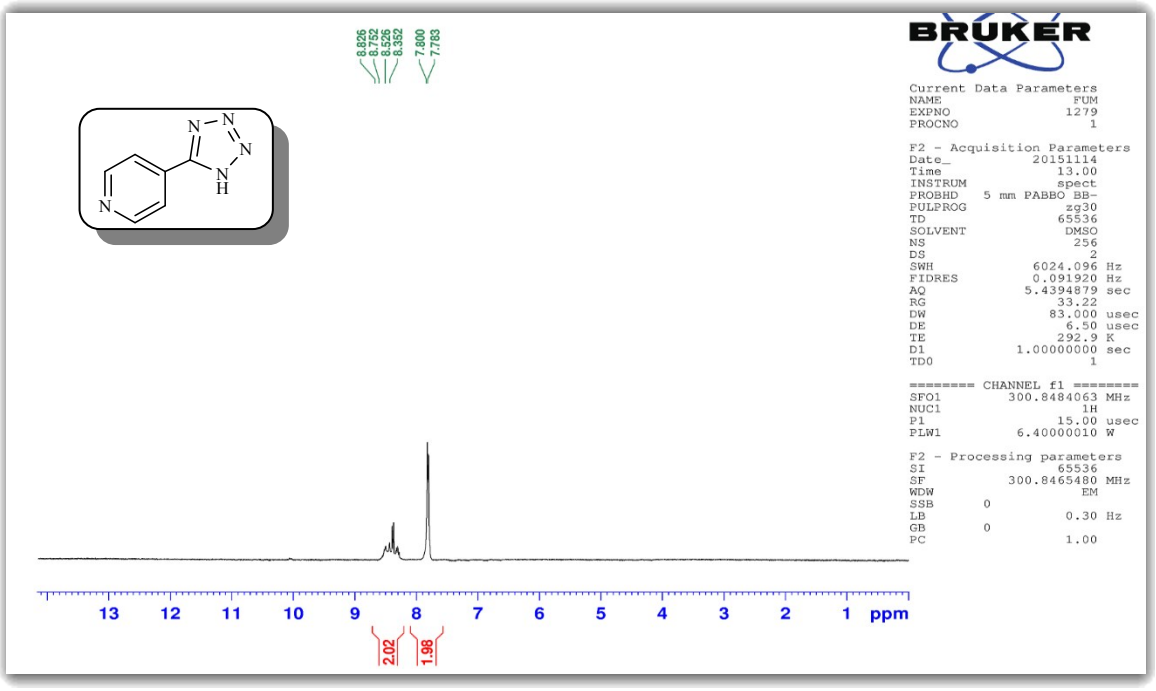

Figure 47: ^{1}H NMR (300 MHz, DMSO-d_6) of 4-(1*H*-tetrazol-5-yl) pyridine.

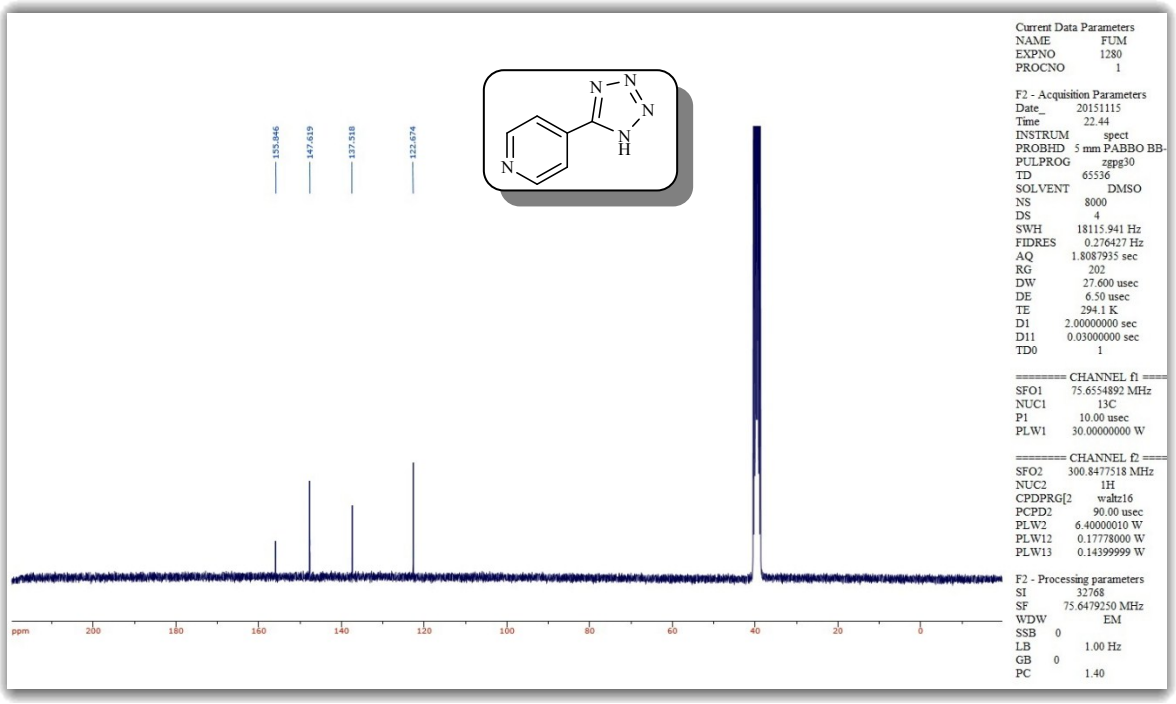

Figure 48: ^{13}C NMR (75 MHz, DMSO-d_6) of 4-(1*H*-tetrazol-5-yl) pyridine.

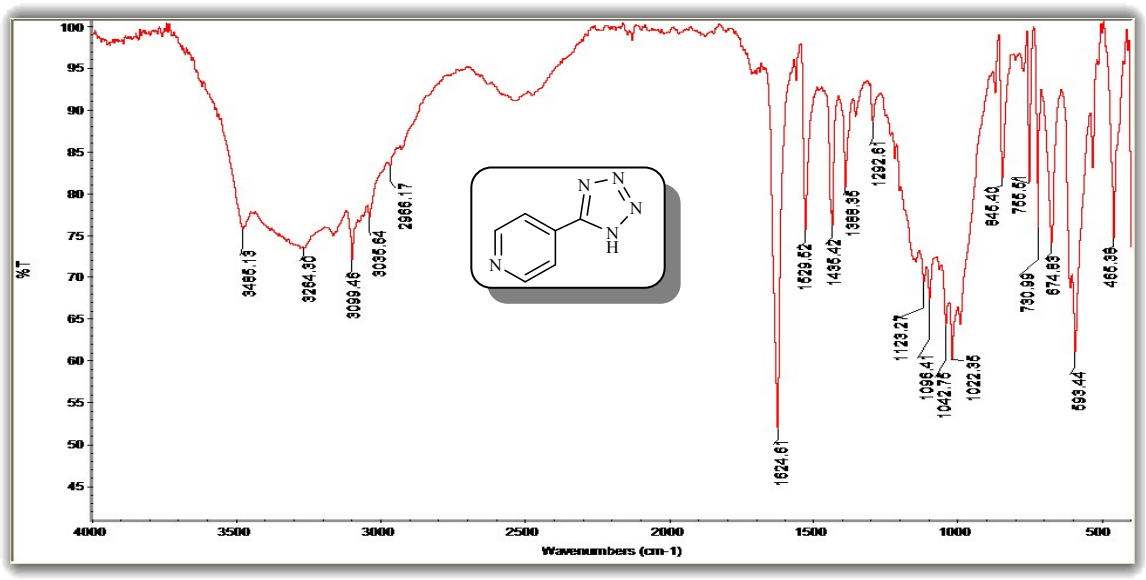

Figure 49: FT-IR (KBr) of 4-(1*H*-tetrazol-5-yl) pyridine.

2-(1*H*-tetrazol-5-yl)pyridine (Table 2, Entry 14) (0.128 g, 87 %); White solid; mp 211-213 ºC (Lit.[3] 210-213 ºC); FT-IR (KBr): ν_{max}/cm^{-1} 3088, 3060, 2959, 2929, 2864, 2737, 2692, 2622, 2582, 1728, 1602, 1557, 1483, 1449, 1405, 1284 (N–N=N–), 1158 (C-N), 1068, 1024, 955, 795, 743, 726, 703, 637, 496; ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ 7.65 (s, 1H, Py), 8.10 (s, 1H, Py), 8.24 (d, J = 6.4 Hz, 1H, Py), 8.81 (s, 1H, Py); ^{13}C NMR (100 MHz, DMSO-d_6, ppm) δ 123.1, 126.7, 138.7, 144.0, 150.6, 155.3.

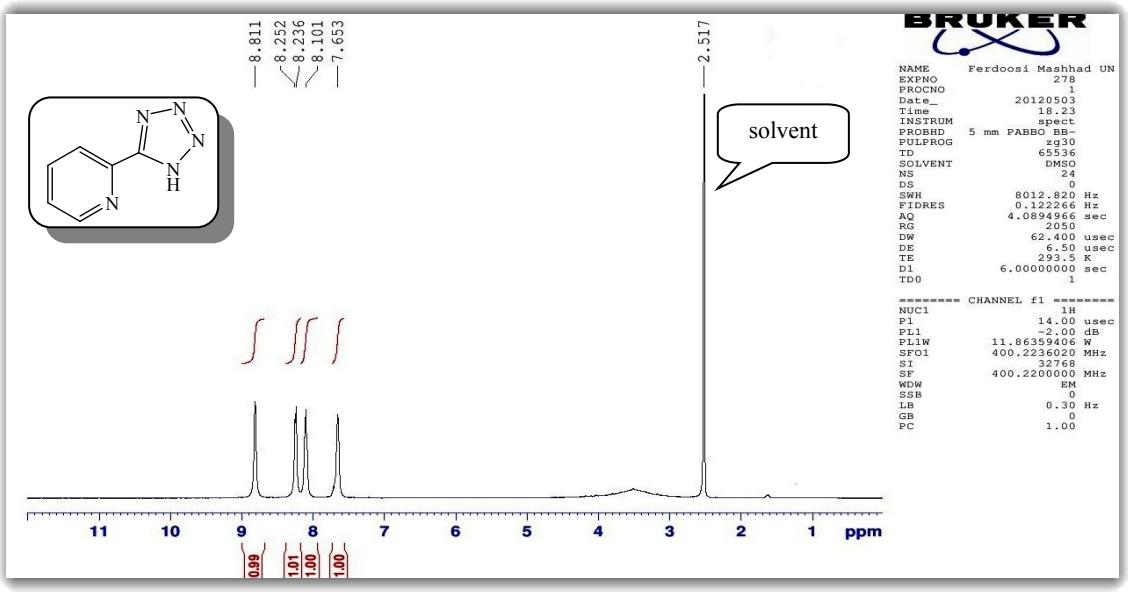

Figure 50: ^{1}H NMR (400 MHz, DMSO-d_6) of 2-(1*H*-tetrazol-5-yl)pyridine.

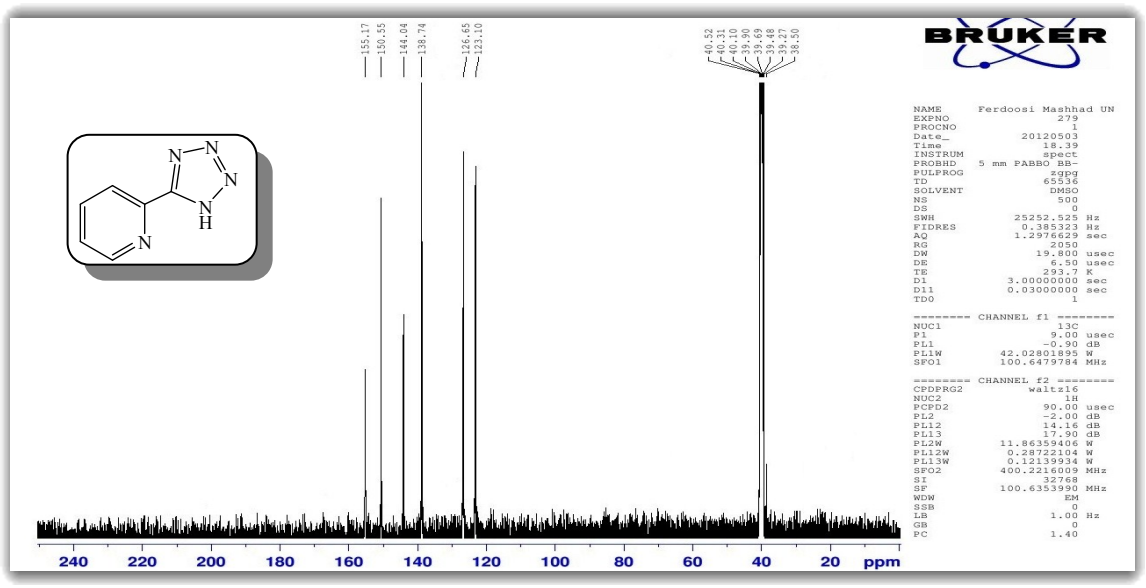

Figure 51: ^{13}C NMR (100 MHz, DMSO-d_6) of 2-(1*H*-tetrazol-5-yl) pyridine.

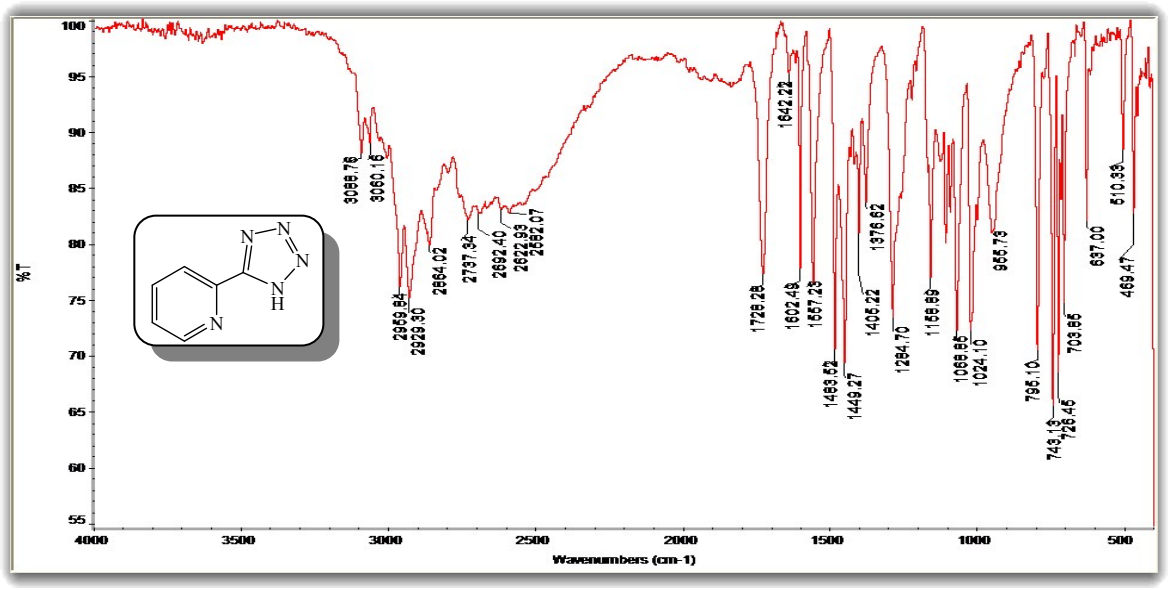

Figure 52: FT-IR of 2-(1*H*-tetrazol-5-yl) pyridine.

5-(Thiophen-2-yl)-1*H*-tetrazole (Table 2, Entry 15) (0.145 g, 95 %); White solid; mp 205-207 ºC (Lit.[12] 205-207 ºC); FT-IR (KBr): ν_{max}/cm^{-1} 3109, 3074, 2974, 2891, 2780, 2722, 2628, 2569, 2500, 2456, 1830, 1595, 1503, 1411, 1233 (N–N=N–), 1139 (C-N), 1046, 962, 853, 740, 719; ^{1}H NMR (300 MHz, DMSO-d_6, ppm) δ 7.29 (t, 1H, J = 4.5 Hz, Tiophen), 7.81 (d, J = 3.3 Hz, 1H, Tiophen), 7.87 (d, J = 4.5 Hz 1H, Tiophen); ^{13}C NMR (75 MHz, DMSO-d_6, ppm) δ 125.7, 129.0, 129.7, 130.8, 151.7; Ms, *m/z* (%): 152 [M^+], 124 (100) [$M^+ - N_2$], 97 [$M^+ - 2N_2$].

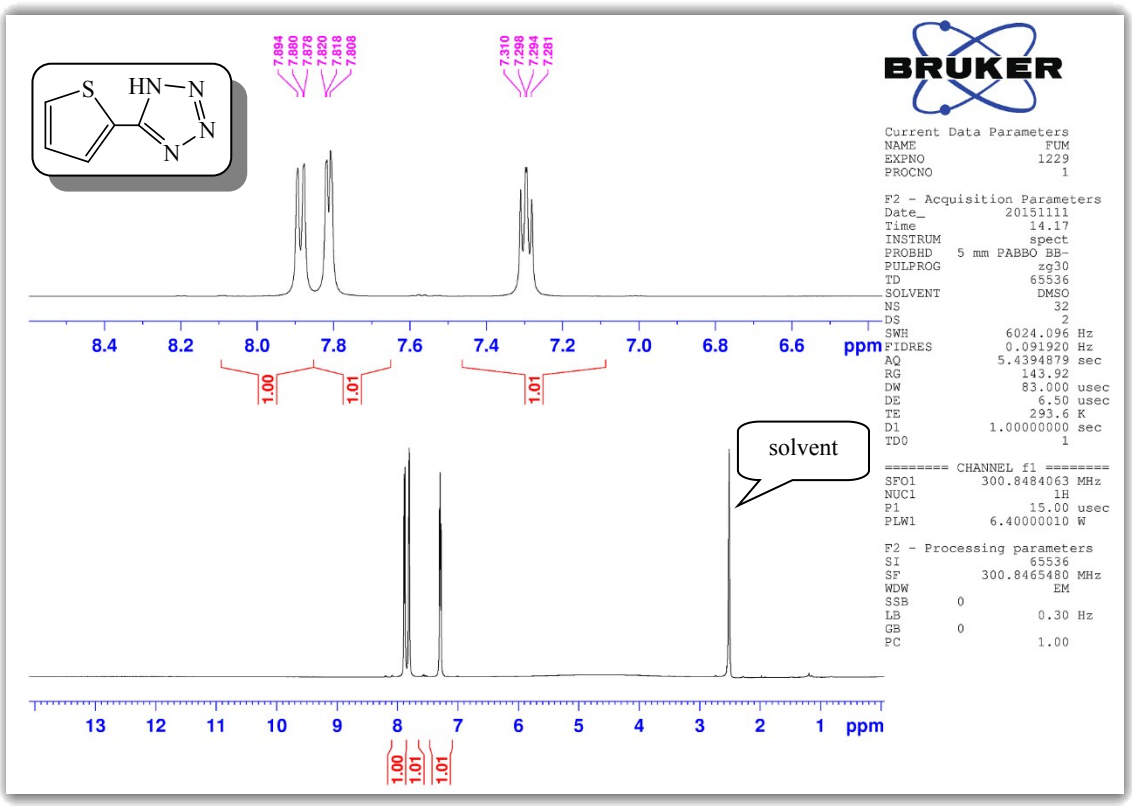

Figure 53: ^{1}H NMR (300 MHz, DMSO-d_6) of 5-(Thiophen-2-yl)-1*H*-tetrazole (**3p**).

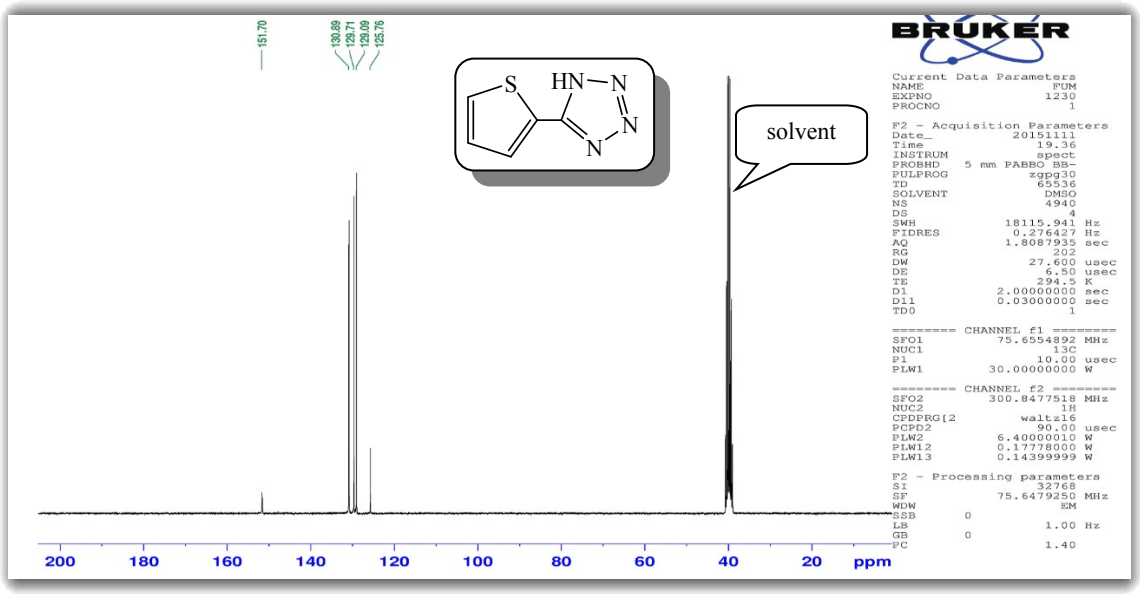

Figure 54: ^{13}C NMR (75 MHz, DMSO-d_6) of 5-(Thiophen-2-yl)-1*H*-tetrazole.

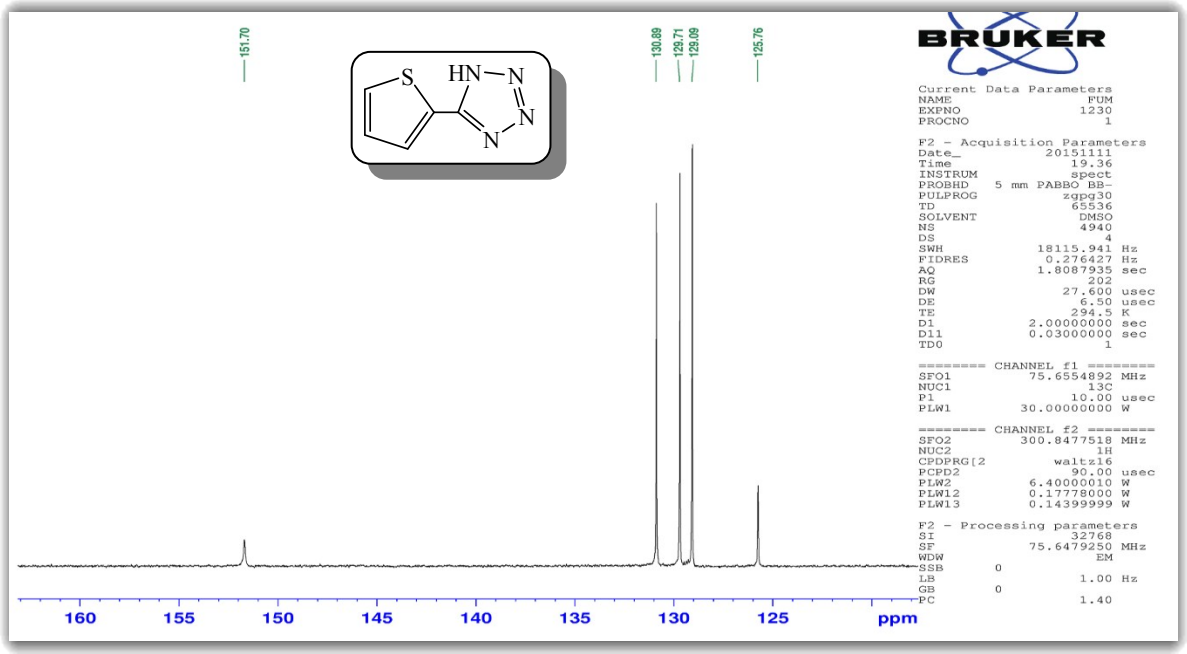

Figure 55: ^{13}C NMR (75 MHz, DMSO-d_6) of 5-(Thiophen-2-yl)-1*H*-tetrazole expanded.

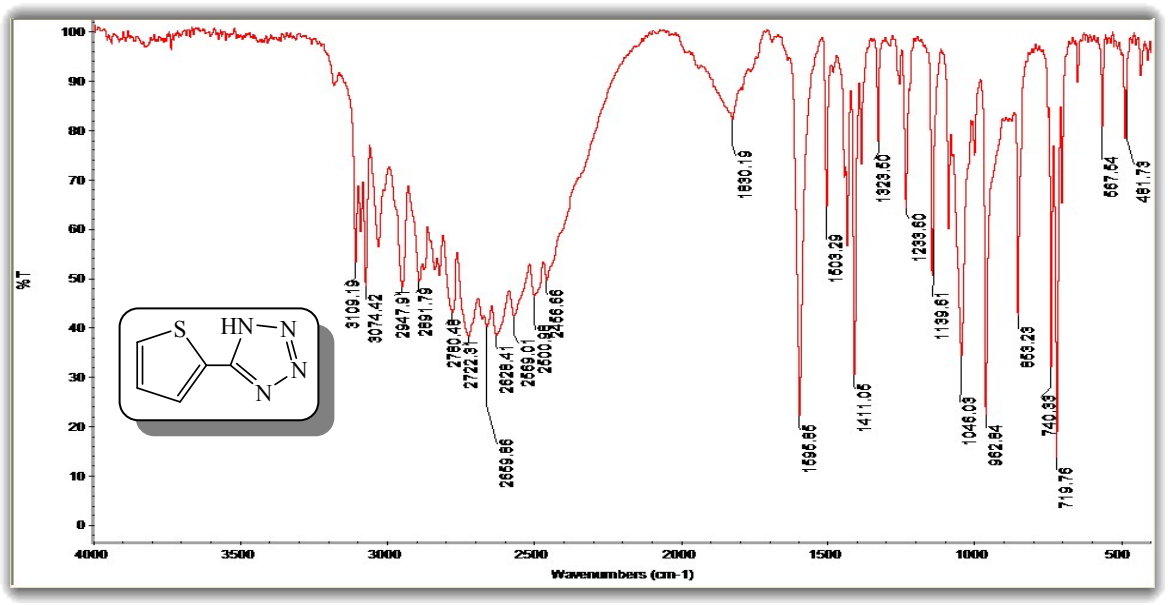

Figure 56: FT-IR (KBr) of 5-(Thiophen-2-yl)-1*H*-tetrazole.

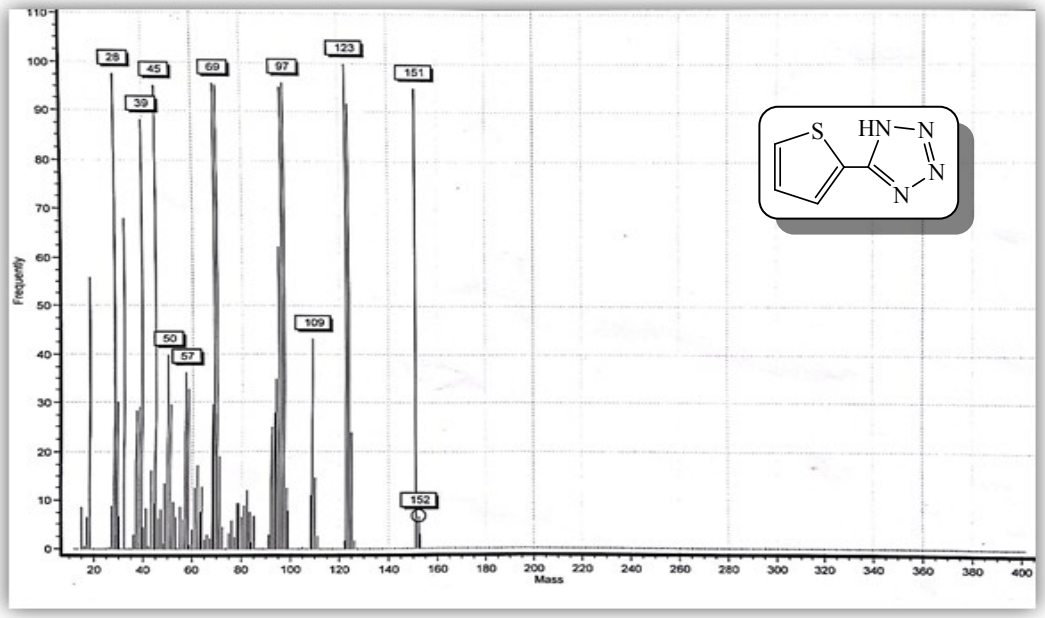

Figure 57: Mass spectrum of 5-(Thiophen-2-yl)-1*H*-tetrazole.

5-Benzyl-1*H*-tetrazole (Table 2, Entry 16) (0.136 g, 85 %); White solid; mp 117-119 ºC (Lit.[13] 118-120 ºC); FT-IR (KBr): ν_{max}/cm^{-1} 3109, 3031, 2984, 2945, 2863, 2778, 2704, 2594, 1768, 1707, 1638, 1549, 1533, 1494, 1457, 1241(N–N=N–), 1108 (C-N), 1074, 772, 734, 695; ^{1}H NMR (300 MHz, DMSO-d_6, ppm) δ 4.29 (s, 2H, -CH_2-), 7.29-7.34 (m, 5H, Ar-H); ^{13}C NMR (75 MHz, DMSO-d_6, ppm) δ 29.3, 127.4, 129.11, 129.19, 136.3, 155.6.

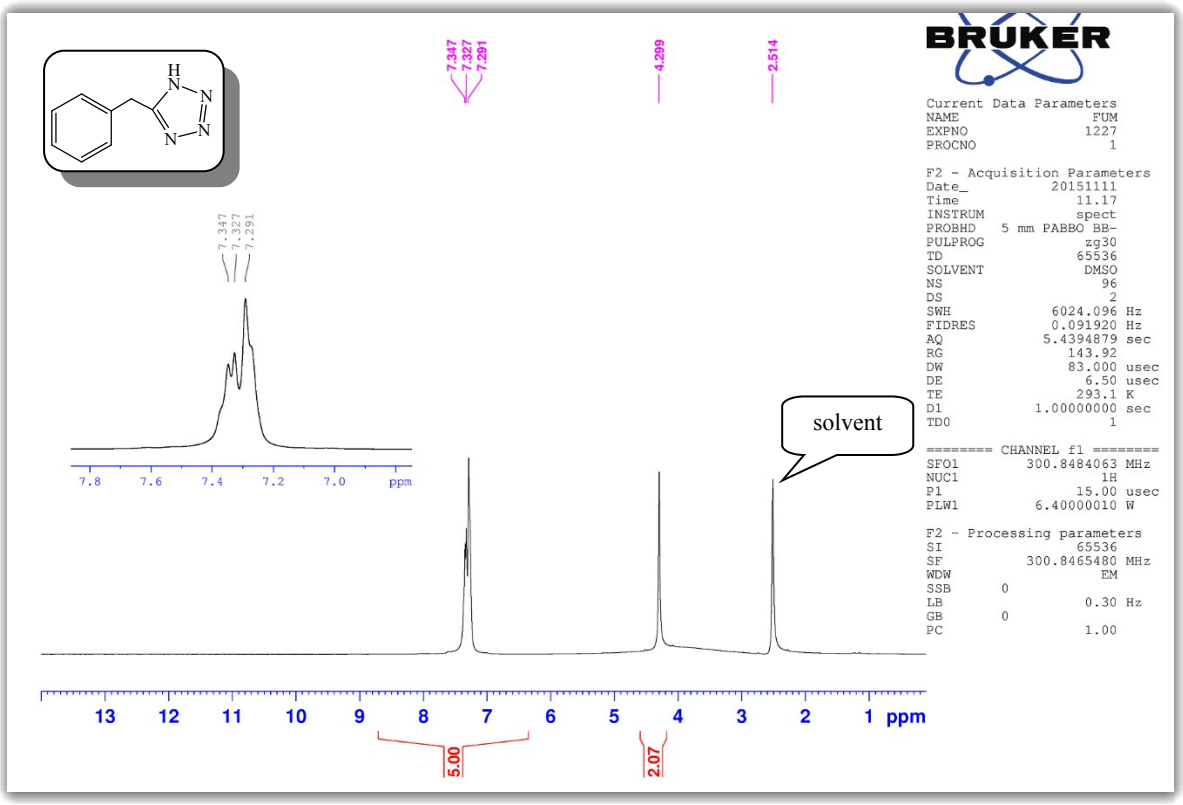

Figure 58: ^{1}H NMR (300 MHz, DMSO-d_6) 5-Benzyl-1*H*-tetrazole.

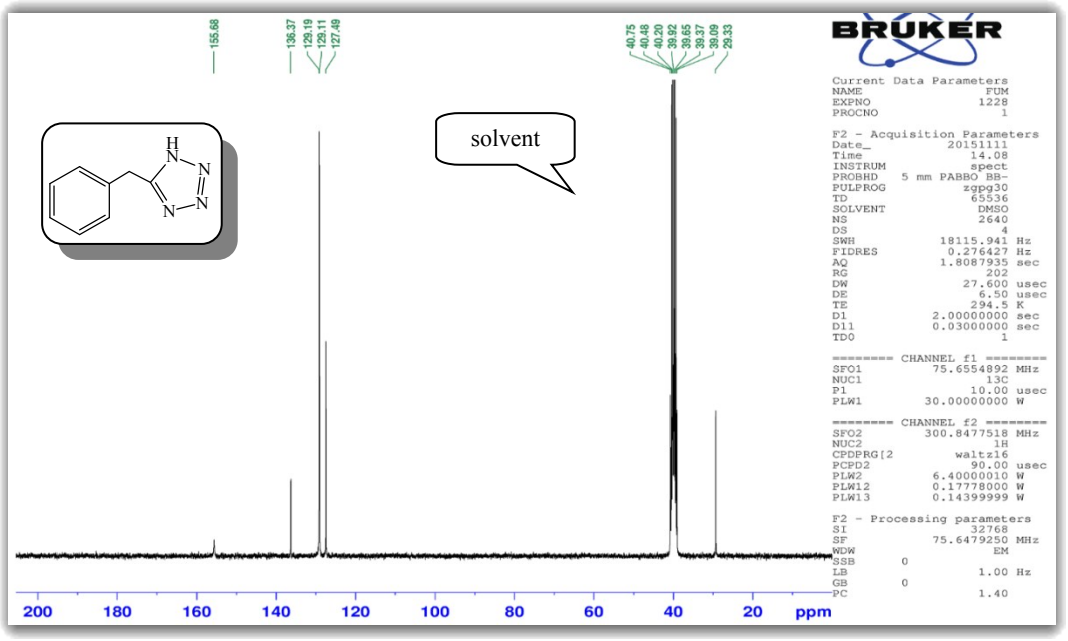

Figure 59: ^{13}C NMR (75 MHz, DMSO-d_6) 5-Benzyl-1*H*-tetrazole.

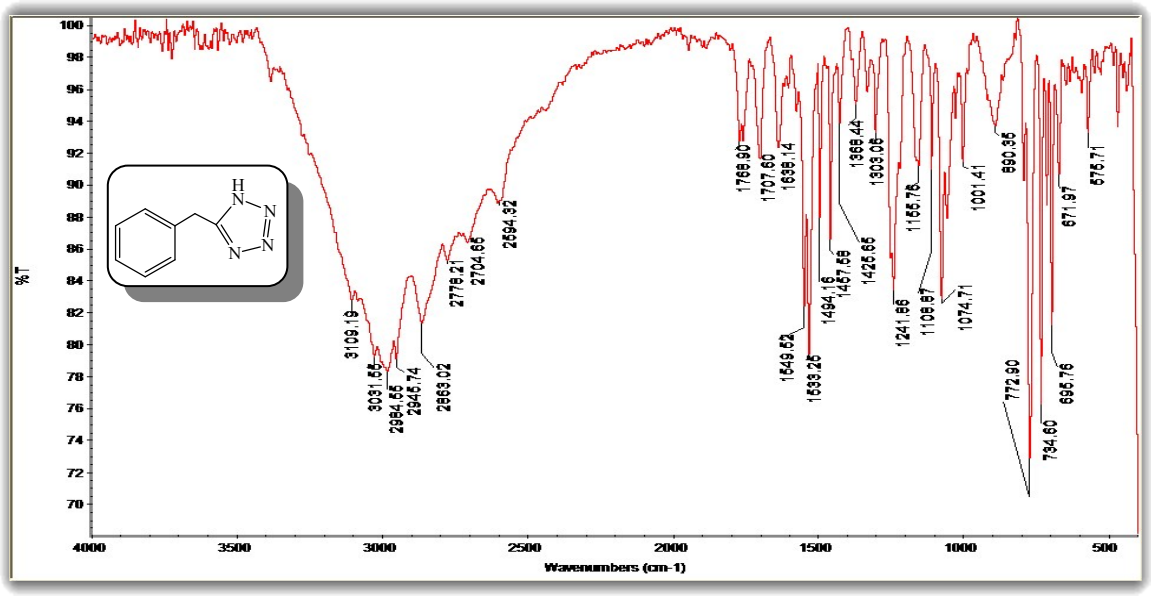

Figure 60: FT-IR of 5-Benzyl-1*H*-tetrazole.

5-Isobutyl-1*H*-tetrazole (Table 2, Entry 17) (0.107 g, 85 %); White solid; mp 52-54 ºC (Lit.[14] 53.5-54 ºC); FT-IR (KBr): ν_{max}/cm^{-1} 3089, 3063, 2971, 2901, 2845, 2765, 2729, 2633, 1605, 1482, 1454, 1430, 1156 (C-N), 1075, 1053, 1017, 990, 829, 772, 743, 502; ^{1}H NMR (300 MHz, $CDCl_3$, ppm) δ 0.87 (d, J = 6.3 Hz, 6H, 2 CH_3), 1.36-1.92 (m, 1H, -CH-), 2.72 (d, J = 7.2 Hz, 2H, -CH_2-), 7.82 (br s, 1H, NH); ^{13}C NMR (75 MHz, DMSO-d_6, ppm) δ 20.9, 22.1, 27.5, 40.2, 164.2.

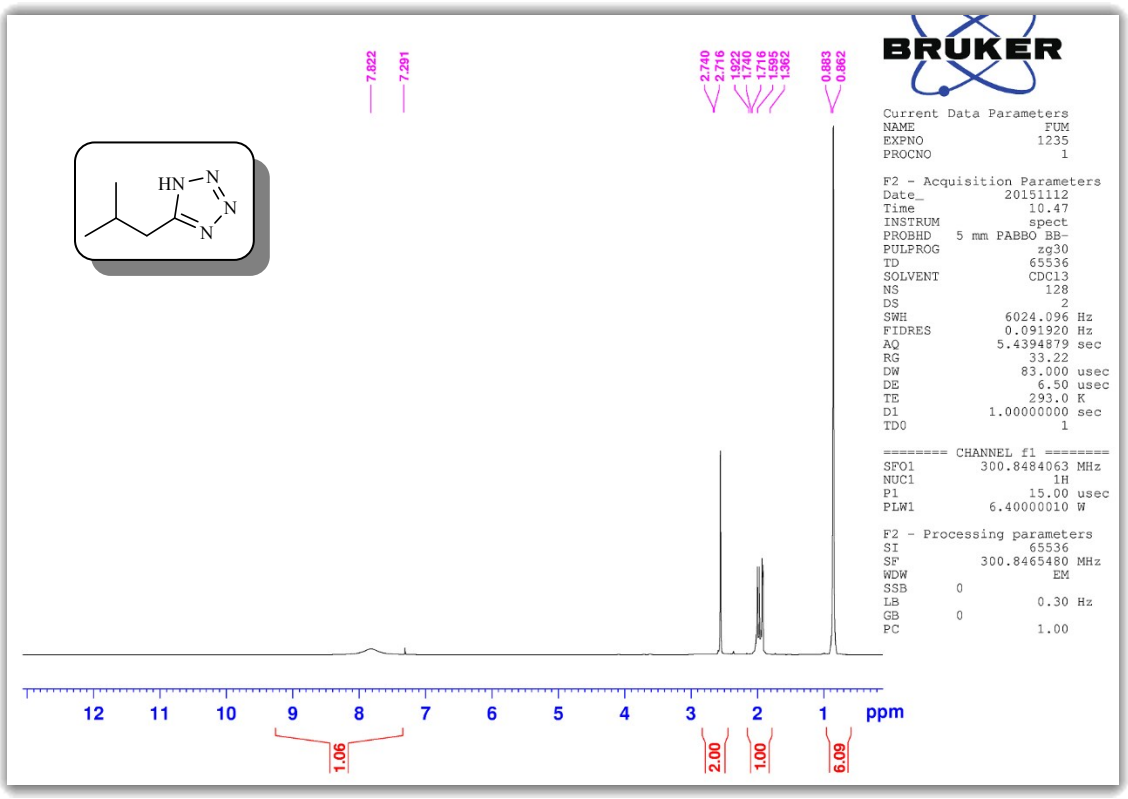

Figure 61: ^{1}H NMR (300 MHz, $CDCl_3$) of 5-Isobutyl-1*H*-tetrazole.

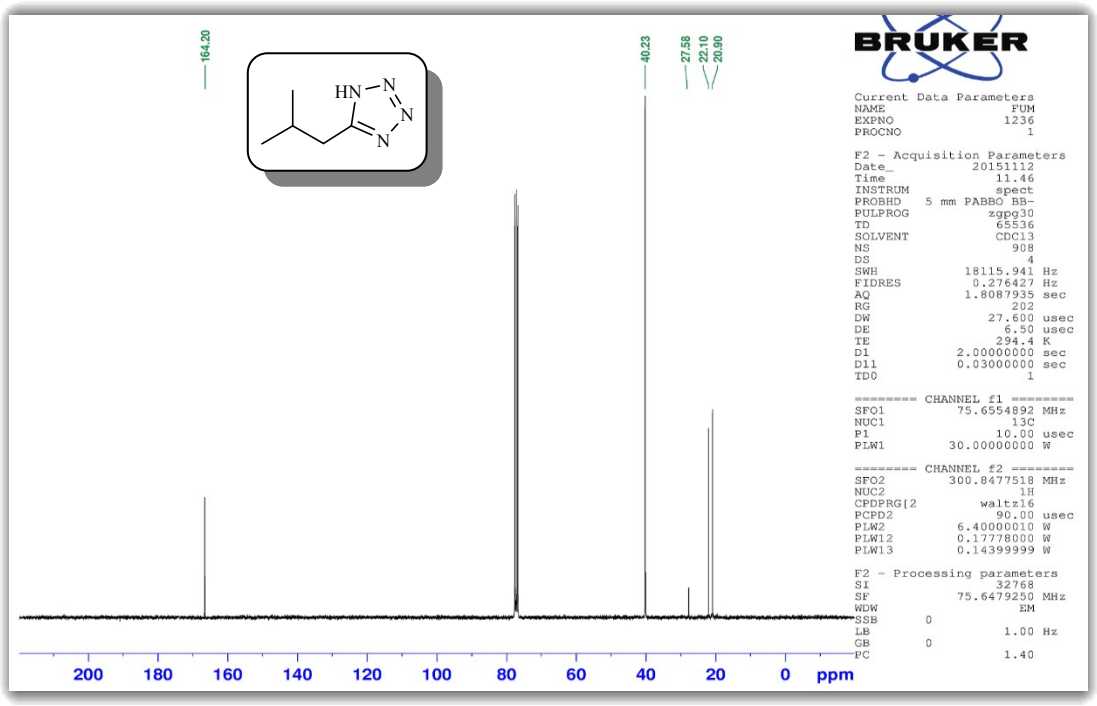

Figure 62: ^{13}C NMR (75 MHz, $CDCl_3$) of 5-Isobutyl-1*H*-tetrazole**.**

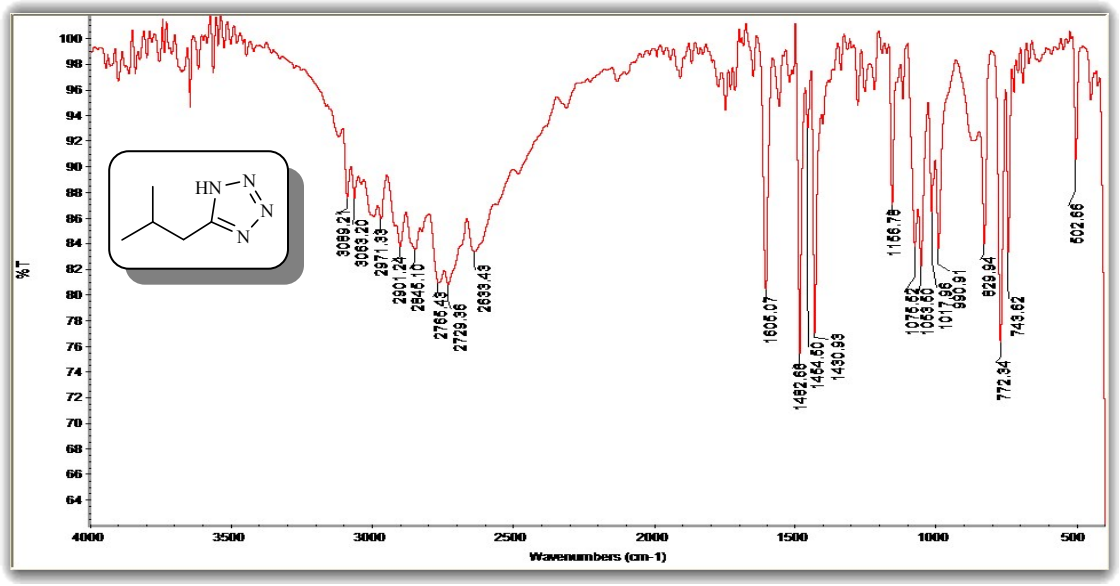

Figure 63: FT-IR of 5-Isobutyl-1*H*-tetrazole.

5-Isopentyl-1*H*-tetrazole (Table 2, Entry 18) (0.131 g, 94 %); White solid; mp 94 ºC (Lit.[14] 95-96 ºC); FT-IR (KBr): ν_{max}/cm^{-1} 2962, 2931, 2874, 2709, 2618, 2482, 1867, 1583, 1553, 1469, 1404, 1110 (C-N), 1048, 772; ^{1}H NMR (300 MHz, $CDCl_3$, ppm) δ 0.93 (d, J = 6 Hz, 6H, 2 CH_3), 1.59-1.72 (m, 3H, -CH-, -CH_2-), 3.00 (t, J = 7.5 Hz, 2H, -CH_2-), 10.98 (br s, 1H, NH); ^{13}C NMR (75 MHz, DMSO-d_6, ppm) δ 21.3, 22.1, 25.1, 27.5, 36.2, 156.6.

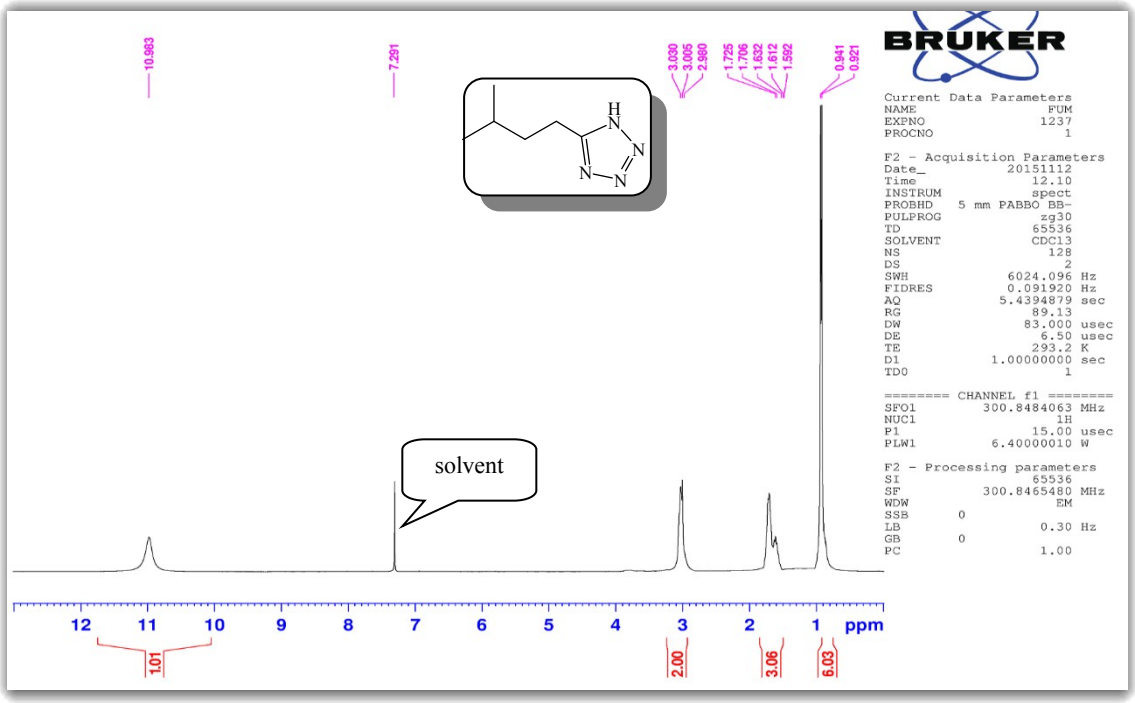

Figure 64: ^{1}H NMR (100 MHz, $CDCl_3$) of 5-Isopentyl-1*H*-tetrazole.

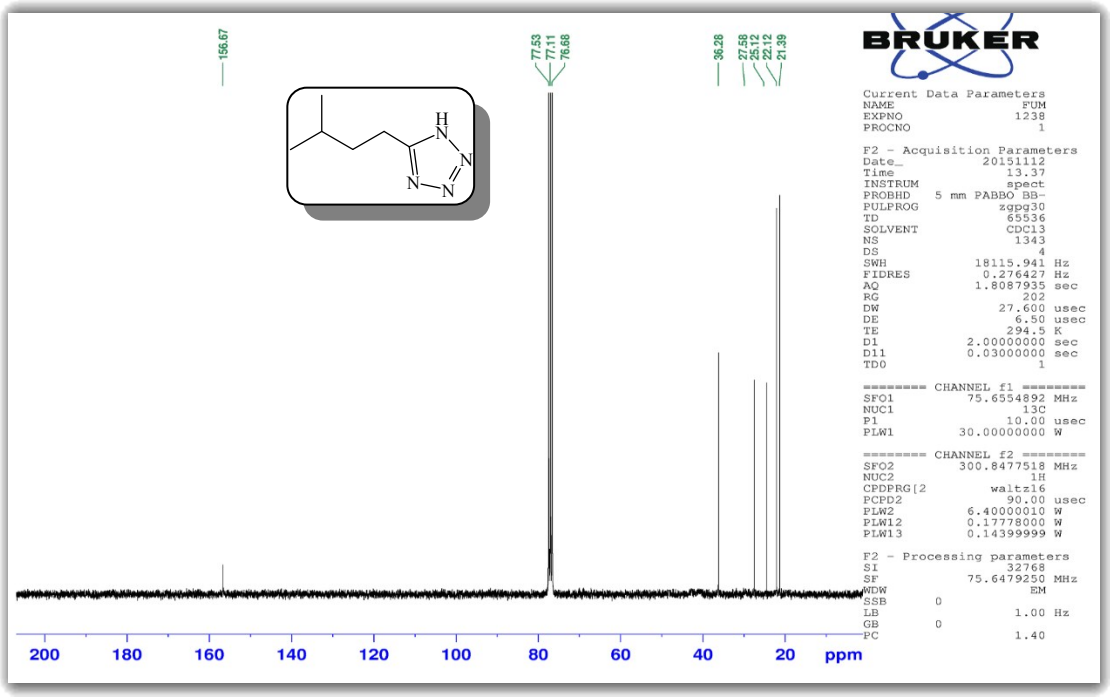

Figure 65: ^{13}C NMR (75 MHz, $CDCl_3$) of 5-Isopentyl-1*H*-tetrazole.

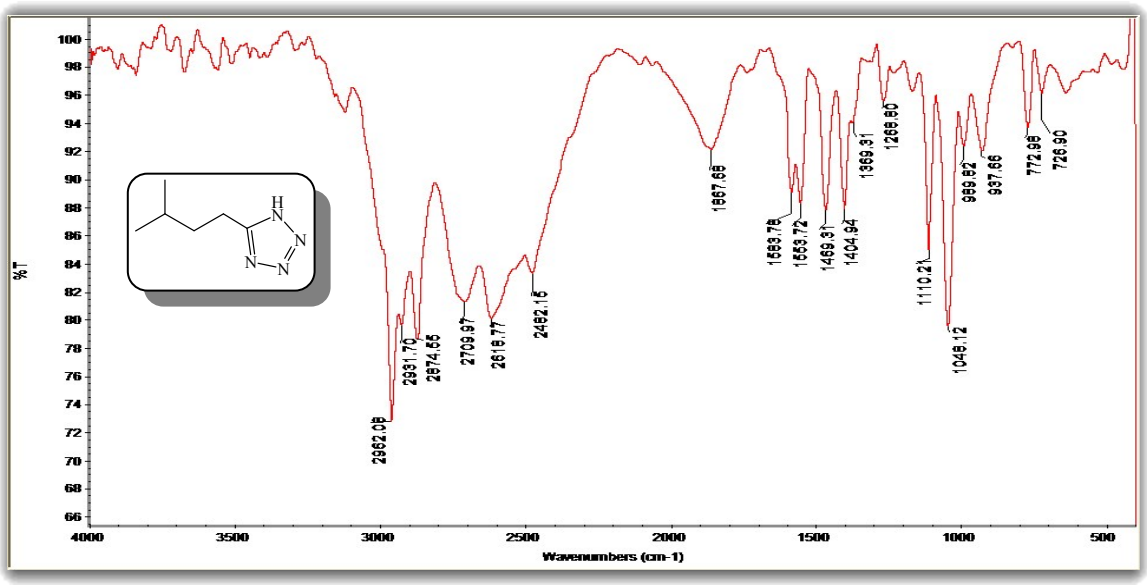

Figure 66: FT-IR of 5-Isopentyl-1*H*-tetrazole.

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