

Supporting Information

Sustainable Generation of Ni(OH)₂ Nanoparticles for the Green Synthesis of 5-Substituted 1*H*-Tetrazoles: A Competent Turn on Fluorescence Sensing of H₂O₂

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¹H and ¹³C NMR data of all products

5-phenyl-1H-tetrazole (Table 2, 2a): White solid; *mp*: 215-216 °C, [lit.¹ 217-219 °C]; ¹H NMR (400 MHz, DMSO-D₆) δ 8.04-8.03 (m, 2H), 7.45-7.44 (m, 3H). ¹³C NMR (75 MHz, DMSO-D₆) δ 156.5, 132.6, 129.7, 127.8, 124.4; Elemental analysis for C₇H₆N₄; Calculated: C, 57.53; H, 4.14; N, 38.34. Found: C, 57.98; H, 4.02; N, 38.08.

5-(p-tolyl)-1H-tetrazole (Table 2, 2b): Colourless crystal; *mp*: 244-245 °C, [lit.² 246-248 °C]; ¹H NMR (400 MHz, CDCl₃) δ 7.65-7.63 (m, 2H), 7.19-7.17 (m, 2H), 5.96 (bs, 1H), 2.33 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 169.4, 142.5, 130.4, 129.2, 127.3, 21.4; Elemental analysis for C₈H₈N₄; Calculated: C, 59.99; H, 5.03; N, 34.98. Found: C, 59.02; H, 4.89; N, 34.68.

5-(2-nitrophenyl)-1H-tetrazole (Table 2, 2c): Pale yellow solid; *mp*: 157-158 °C, [lit.² 158-161 °C]; ¹H NMR (400 MHz, CDCl₃) δ 7.66-7.65 (m, 1H), 7.47-7.45 (m, 1H), 7.28 (m, 1H), 7.07-7.05 (m, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 164.1, 151.5, 134.0, 133.6, 131.3, 126.4, 120.4; Elemental analysis for C₇H₅N₅O₂; Calculated: C, 43.98; H, 2.64; N, 36.64. Found C, 44.20; H, 2.22; N, 36.05.

5-(3-nitrophenyl)-1H-tetrazole (Table 2, 2d): Pale yellow solid; *mp*: 154-155 °C, [lit.¹ 155-157 °C]; ¹H NMR (400 MHz, DMSO-D₆) δ 8.75 (bs, 1H), 8.28-8.23 (m, 2H), 7.95 (bs, 1H), 7.60-7.55 (m 1H); ¹³C NMR (100 MHz, DMSO-D₆) δ 166.9, 147.9, 135.4, 133.9, 129.4, 125.9, 122.7; Elemental analysis for C₇H₅N₅O₂; Calculated: C, 43.98; H, 2.64; N, 36.64. Found: C, 43.11; H, 2.40; N, 36.60.

5-(4-nitrophenyl)-1H-tetrazole (Table 2, 2e): Yellow solid; *mp*: 218-219 °C, [lit.² 218-220 °C]; ¹H NMR (400 MHz, CDCl₃) δ 8.26-8.24 (m, 2H), 7.93-7.91 (m, 2H), 6.09. (bs, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 161.9, 148.1, 139.7, 128.5, 123.9; Elemental analysis for C₇H₅N₅O₂; Calculated: C, 43.98; H, 2.64; N, 36.64. Found: C, 43.75; H, 2.30; N, 36.12.

5-(4-chlorophenyl)-1H-tetrazole (Table 2, 2f): Pale brown solid; *mp*: 248-249 °C, [lit.³ 249-250 °C]; ¹H NMR (400 MHz, DMSO-D₆) δ 7.79-7.78 (m, 2H), 7.34-7.33 (m, 2H), 7.27 (bs, 1H); ¹³C NMR (100 MHz, DMSO-D₆) δ 168.3, 137.5, 132.1, 129.1, 128.4; Elemental analysis for C₇H₅ClN₄; Calculated: C, 46.55; H, 2.79; N, 31.02. Found: C, 46.02; H, 2.85; N, 30.88.

5-(4-bromophenyl)-1H-tetrazole (Table 2, 2h): Light yellow solid; *mp*: 263-264 °C, [lit.⁴ 267-268 °C]; ¹H NMR (400 MHz, DMSO-D₆) δ 7.74-7.72 (m, 2H), 7.54 (bs, 1H), 7.50-7.45 (m, 2H); ¹³C NMR (100 MHz, DMSO-D₆) δ 168.3, 132.6, 131.2, 129.3, 125.9; Elemental analysis for C₇H₅BrN₄; Calculated: C, 37.36; H, 2.24; N, 24.90. Found: C, 37.28; H, 2.11; N, 24.80.

5-(3,4-dimethoxyphenyl)-1H-tetrazole (Table 2, 2i): Off-white solid; *mp*: 198-199 °C, [lit.⁵ 198-200 °C]; ¹H NMR (400 MHz, CDCl₃) δ 7.39 (m, 1H), 7.28-7.25 (m, 1H), 6.81-6.79 (m, 1H), 5.97 (bs, 1H), 3.86 (s, 3H), 3.84 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 169.1, 152.1, 149.0, 125.8, 120.1, 110.8, 110.2, 56.0; ; Elemental analysis for C₉H₁₀N₄O₂; Calculated: C, 52.42; H, 4.89; N, 27.17. Found: C, 52.12; H, 4.67; N, 26.99.

5-(4-methoxyphenyl)-1H-tetrazole (Table 2, 2j): White solid; *mp*: 230-231 °C, [lit.¹ 233-234 °C]; ¹H NMR (400 MHz, DMSO-D₆) δ 7.77-7.74 (m, 2H), 6.86-6.83 (m, 2H), 5.96 (bs, 1H), 3.78 (s, 3H); ¹³C NMR (100 MHz, DMSO-D₆) δ 168.9, 162.3, 129.3, 125.8, 113.5, 55.3; Elemental analysis for C₈H₈N₄O; Calculated: C, 54.54; H, 4.58; N, 31.80. Found: C, 54.48; H, 4.62; N, 31.63.

2-(1H-tetrazol-5-yl)phenol (Table 2, 2k): Whitish solid; *mp*: 217-218 °C, [lit.¹ 219-221 °C]; ¹H NMR (400 MHz, CDCl₃) δ 7.48-7.45 (m, 1H), 7.10-7.08 (m, 2H), 7.06-7.03 (m, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 164.0, 153.5, 132.9, 131.0, 119.6, 117.4, 114.1; Elemental analysis for C₇H₆N₄O; Calculated: C, 51.85; H, 3.73; N, 34.55. Found: C, 51.08; H, 3.92; N, 34.11.

4-(1H-tetrazol-5-yl)phenol (Table 2, 2l): White solid; *mp*: 235-236 °C, [lit.² 238-240 °C]; ¹H NMR (400 MHz, CDCl₃) δ 7.47-7.45 (m, 2H), 6.86-6.84 (m, 2H), 6.05 (bs, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 163.1, 160.3, 134.2, 132.6, 116.4; Elemental analysis for C₇H₆N₄O; Calculated: C, 51.85; H, 3.73; N, 34.55. Found: C, 51.17; H, 3.52; N, 34.42.

5-methyl-2-(1H-tetrazol-5-yl)phenol (Table 2, 2m): Off-white solid; ¹H NMR (400 MHz, CDCl₃) δ 7.31-7.29 (m, 1H), 6.73-6.69 (m, 2H), 2.27 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 158.6, 146.1, 132.5, 128.8, 121.9, 117.0, 116.9, 21.8; Elemental analysis for C₈H₈N₄O; Calculated: C, 54.54; H, 4.58; N, 31.80. Found: C, 54.04; H, 4.60; N, 31.42.

5-(furan-2-yl)-1H-tetrazole (Table 3, 3a): Colourless solid; *mp*: 204-205 °C, [lit.⁶ 206-208 °C]; ¹H NMR (400 MHz, DMSO-D₆) δ 7.42 (t, *J* = 0.8 Hz, 1H), 7.07-7.06 (m, 1H), 6.66 (bs, 1H), 6.44 (dd, *J* = 3.2, 1.6 Hz, 1H); ¹³C NMR (100 MHz, DMSO-D₆) δ 160.0, 148.2, 145.5, 114.2, 112.3; Elemental analysis for C₅H₄N₄O; Calculated: C, 44.12; H, 2.96; N, 41.16. Found: C, 43.95; H, 2.90; N, 41.10.

5-(thiophen-2-yl)-1H-tetrazole (Table 3, 3b): Off-white solid; *mp*: 203-204 °C, [lit.⁷ 205-206 °C]; ¹H NMR (400 MHz, DMSO-D₆) δ 7.59-7.58 (m, 1H), 7.44-7.43 (m, 1H), 7.01 (dd, *J* = 4.8, 4.0 Hz, 1H), 6.23 (bs, 1H); ¹³C NMR (100 MHz, DMSO-D₆) δ 163.9, 138.7, 130.5, 129.1, 127.6; Elemental analysis for C₅H₄N₄S; Calculated: C, 39.46; H, 2.65; N, 36.82. Found: C, 39.16; H, 2.78; N, 36.56.

2-(1H-tetrazol-5-yl)pyridine (Table 3, 3c): Pale brown solid; *mp*: 210-211 °C, [lit.⁴ 211-213 °C]; ¹H NMR (400 MHz, CDCl₃) δ 8.52-8.50 (m, 1H), 8.15-8.13 (m, 1H), 7.82-7.77 (m, 2H), 7.40-7.37 (m, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 166.9, 149.6, 148.3, 137.3, 126.5, 122.4; Elemental analysis for C₆H₅N₅; Calculated: C, 48.98; H, 3.43; N, 47.60. Found: C, 48.37; H, 3.10; N, 47.78.

2-(1H-tetrazol-5-yl)quinoline (Table 3, 3d): Brownish solid; ¹H NMR (400 MHz, CDCl₃) δ 8.22 (m, 2H), 8.03-8.01 (m, 2H), 7.80-7.78 (m, 1H), 7.69-7.66 (m, 1H), 7.55-7.52 (m, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 166.7, 150.3, 148.8, 137.0, 129.6, 129.3, 128.8, 127.6, 127.2, 118.3; Elemental analysis for C₁₀H₇N₅; Calculated: C, 60.91; H, 3.58; N, 35.51. Found: C, 60.52; H, 3.21; N, 35.04.

1H-tetrazole (Table 4, 4a): Colourless solid; 154-155 °C, [lit.⁸ 154 °C]; ¹H NMR (400 MHz, DMSO-D₆) δ 8.81 (s, 1H), 6.82 (bs, 1H); ¹³C NMR (100 MHz, DMSO-D₆) δ 142.9; Elemental analysis for CH₂N₄; Calculated: C, 17.15; H, 2.88; N, 79.98. Found: C, 17.26; H, 2.79; N, 79.03.

5-hexyl-1H-tetrazole (Table 4, 4b): Colourless viscous oil; ¹H NMR (400 MHz, CDCl₃) δ 2.87 (t, *J* = 7.6 Hz, 2H), 1.57-1.52 (m, 2H), 1.30-1.21 (m, 6H), 0.81 (t, *J* = 6.8 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 159.4, 31.5, 29.7, 25.4, 24.8, 22.5, 14.0; Elemental analysis for C₇H₁₄N₄; Calculated: C, 54.52; H, 9.15; N, 36.33. Found: C, 54.36; H, 8.88; N, 36.10.

(E)-5-styryl-1H-tetrazole (Table 4, 4c): Colourless solid; *mp*: 153-154 °C, [lit.⁹ 155-156 °C]; ¹H NMR (400 MHz, CDCl₃) δ 7.47-7.44 (m, 2H), 7.32-7.29 (m, 3H), 6.41-6.37 (d, *J* = 15.6 Hz, 2H), 6.52 (bs, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 155.8, 137.2, 134.2, 128.6, 128.1, 126.6, 110.1; Elemental analysis for C₉H₈N₄; Calculated: C, 62.78; H, 4.68; N, 32.54. Found: C, 62.80; H, 4.32; N, 32.19.

9-(4-iodophenyl)-9H-carbazole (Scheme 4, Compound 5): ¹H NMR (400 MHz, CDCl₃) δ 8.01 (d, *J* = 7.2 Hz, 2H), 7.77 (d, *J* = 7.2 Hz, 2H), 7.30-7.24 (m, 4H), 7.19-7.15 (m, 4H).

9-(4-(5-(quinolin-2-yl)-1H-tetrazol-1-yl)phenyl)-9H-carbazole (Scheme 4, Compound 6): ¹H NMR (400 MHz, CDCl₃) δ: 8.36 (dd, *J* = 16.8, 8.4 Hz, 2H), 8.16 (d, *J* = 8.4 Hz, 1H), 8.08 (d, *J* = 8.0 Hz, 2H), 8.03 (d, *J* = 8.8 Hz, 2H), 7.87 (d, *J* = 8.0 Hz, 1H), 7.79-7.75 (m, 1H), 7.61 (t, *J* = 6.0 Hz, 1H), 7.54 (d, *J* = 8.8 Hz, 2H), 7.36-7.35 (m, 4H), 7.24-7.20 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ: 162.4, 149.4, 146.3, 141.0, 138.1, 137.0, 133.6, 130.5, 129.7, 129.5, 128.3, 127.9, 125.9, 123.3, 121.1, 120.3, 119.9, 118.8, 109.7.

^1H and ^{13}C NMR spectra of all products

Figure S1. ^1H and ^{13}C NMR Spectra of 5-phenyl-1*H*-tetrazole (**2a**)

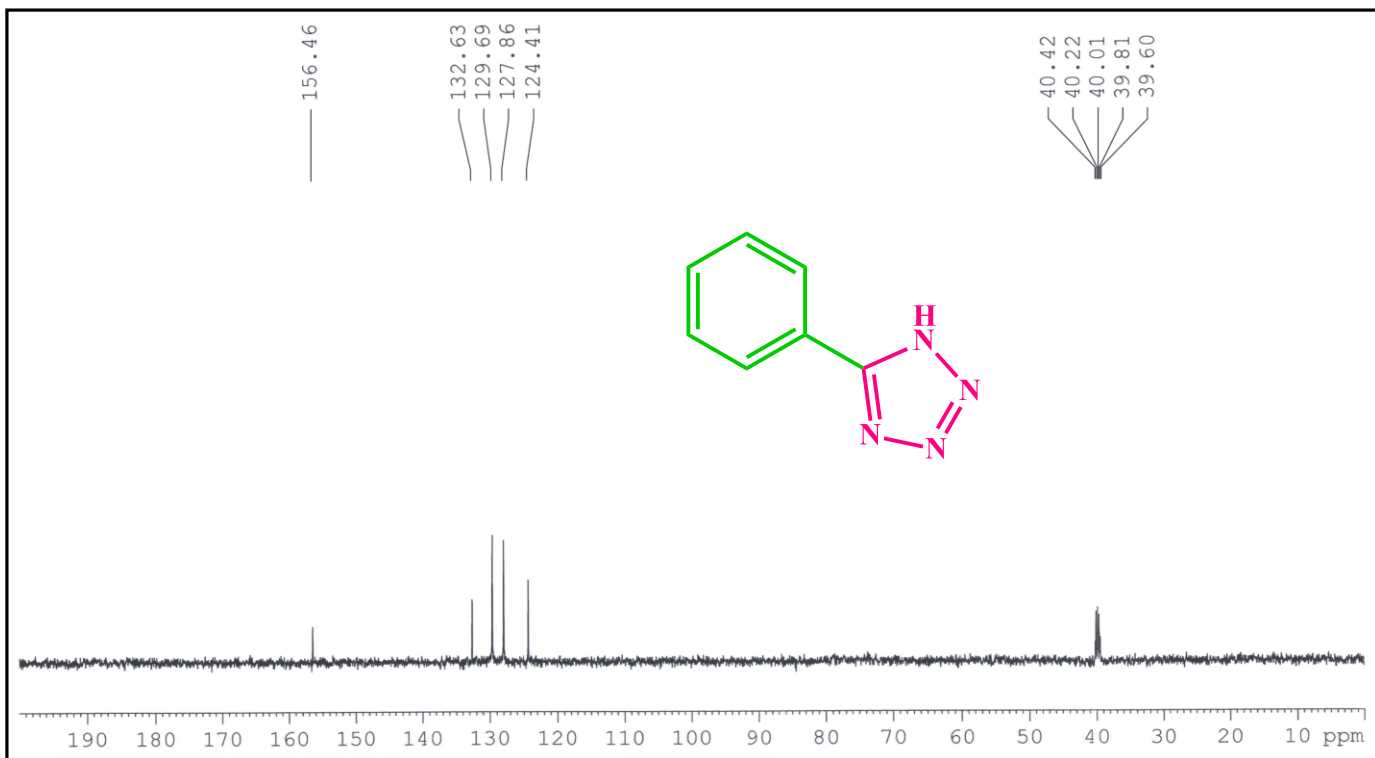
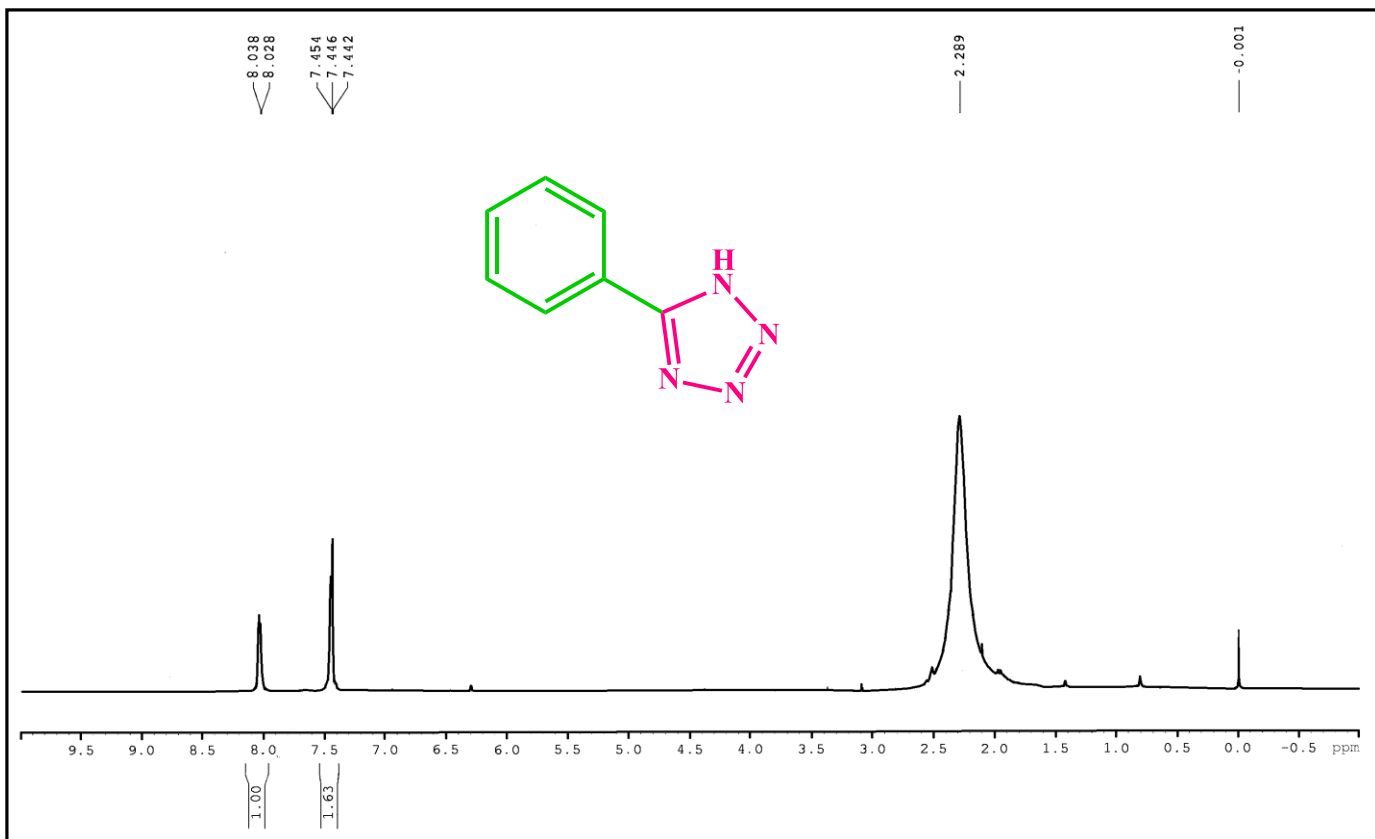


Figure S2. ^1H and ^{13}C NMR Spectra of 5-(p-tolyl)-1*H*-tetrazole (**2b**)

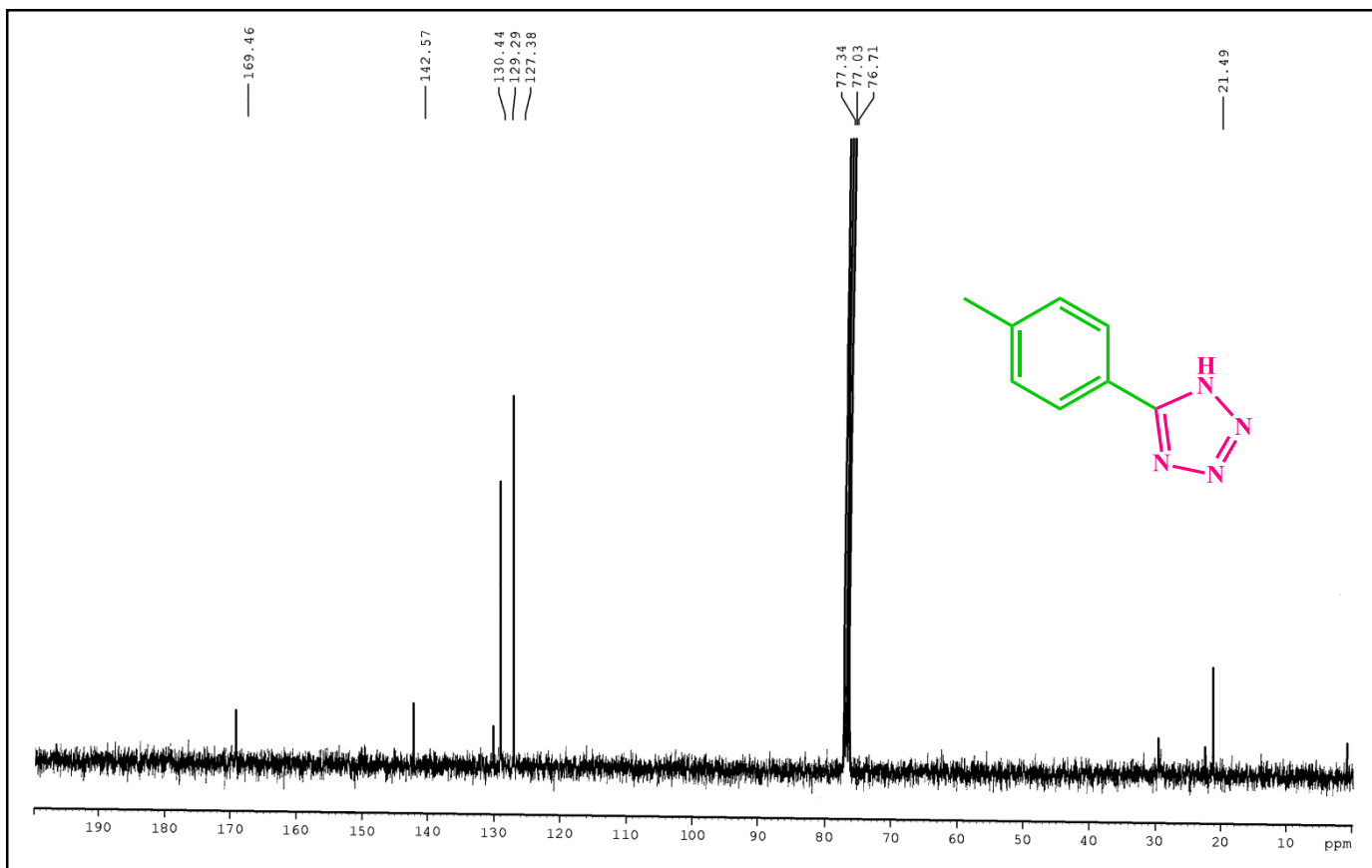
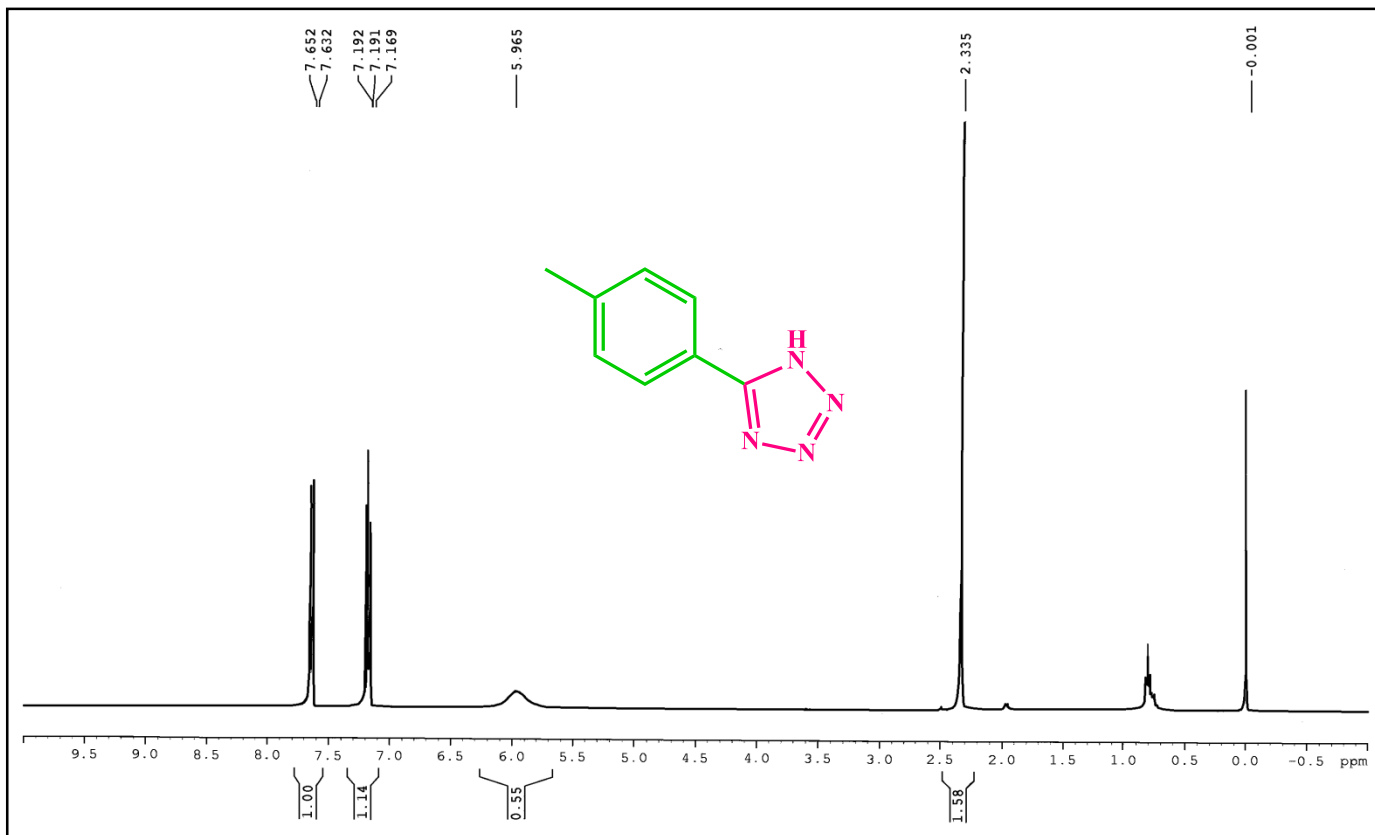


Figure S3. ^1H and ^{13}C NMR Spectra of 5-(2-nitrophenyl)-1*H*-tetrazole (**2c**)

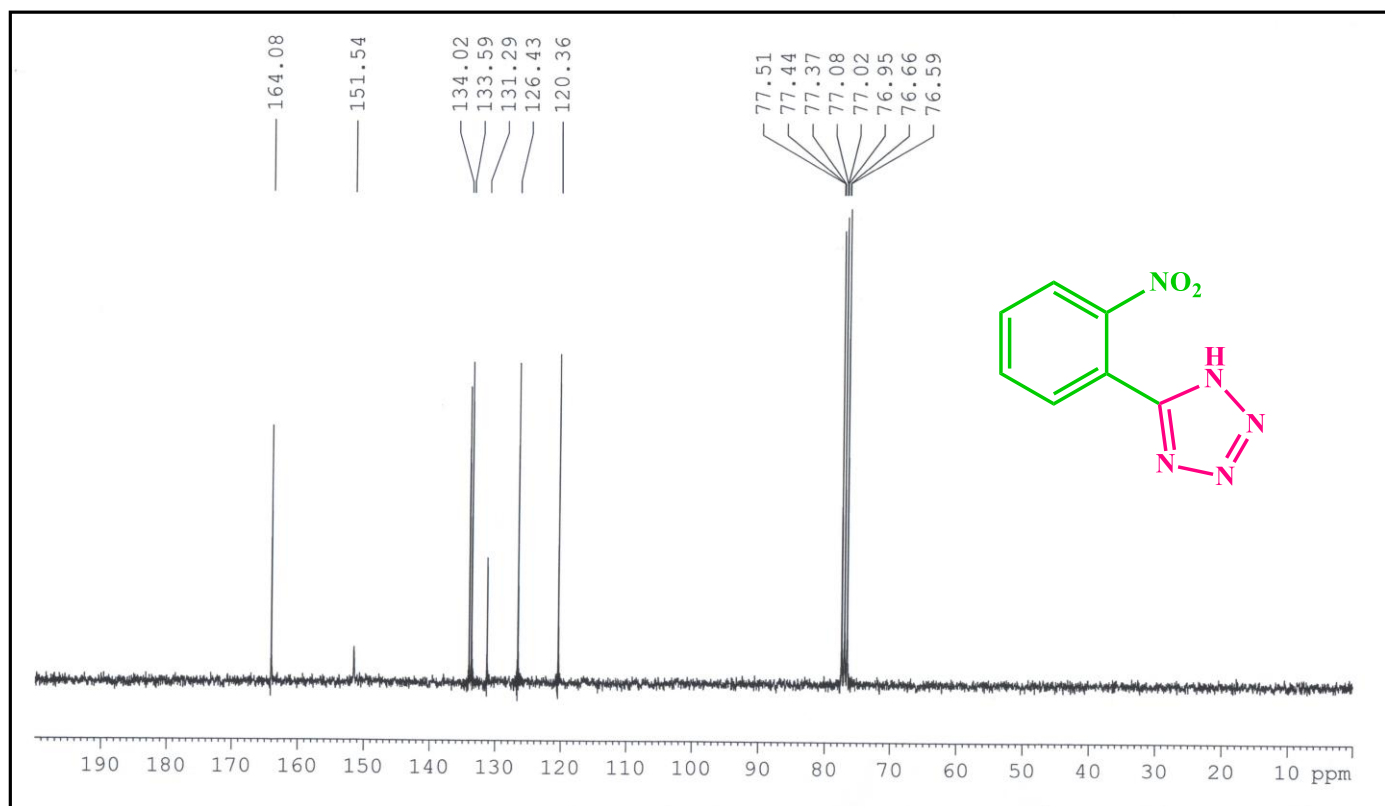
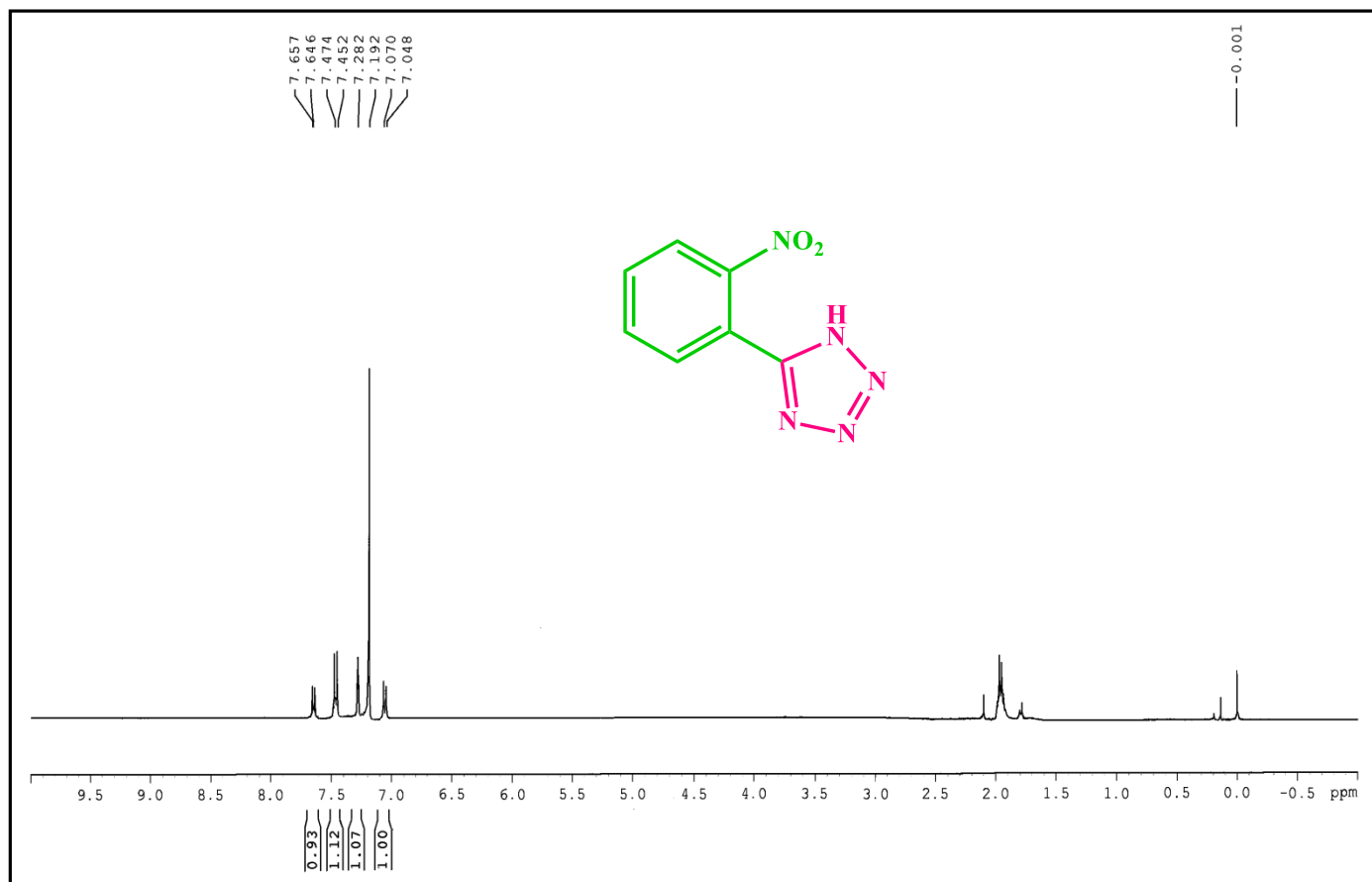


Figure S4. ^1H and ^{13}C NMR Spectra of 5-(3-nitrophenyl)-1*H*-tetrazole (**2d**)

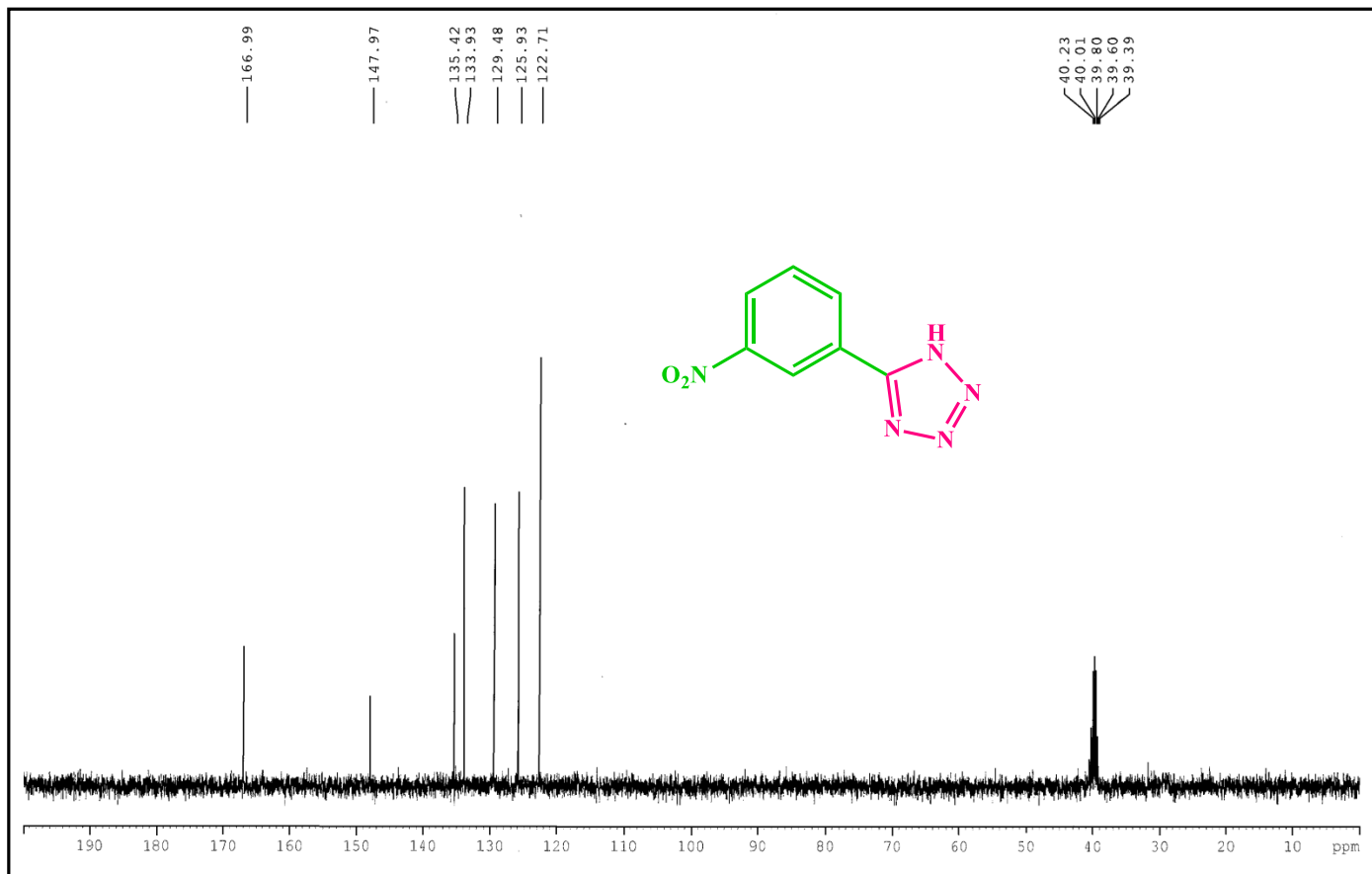
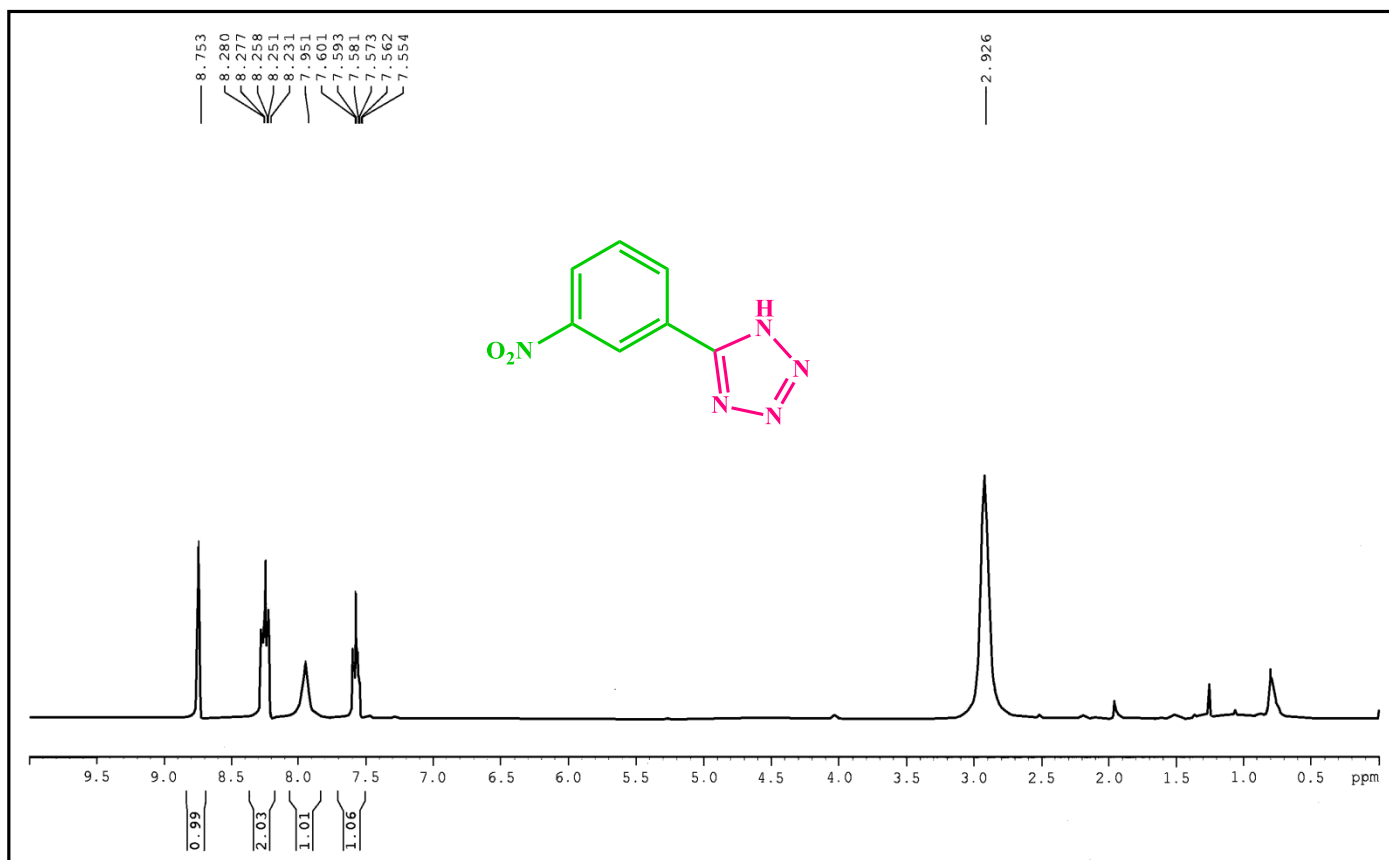


Figure S5. ^1H and ^{13}C NMR Spectra of 5-(4-nitrophenyl)-1*H*-tetrazole (**2e**)

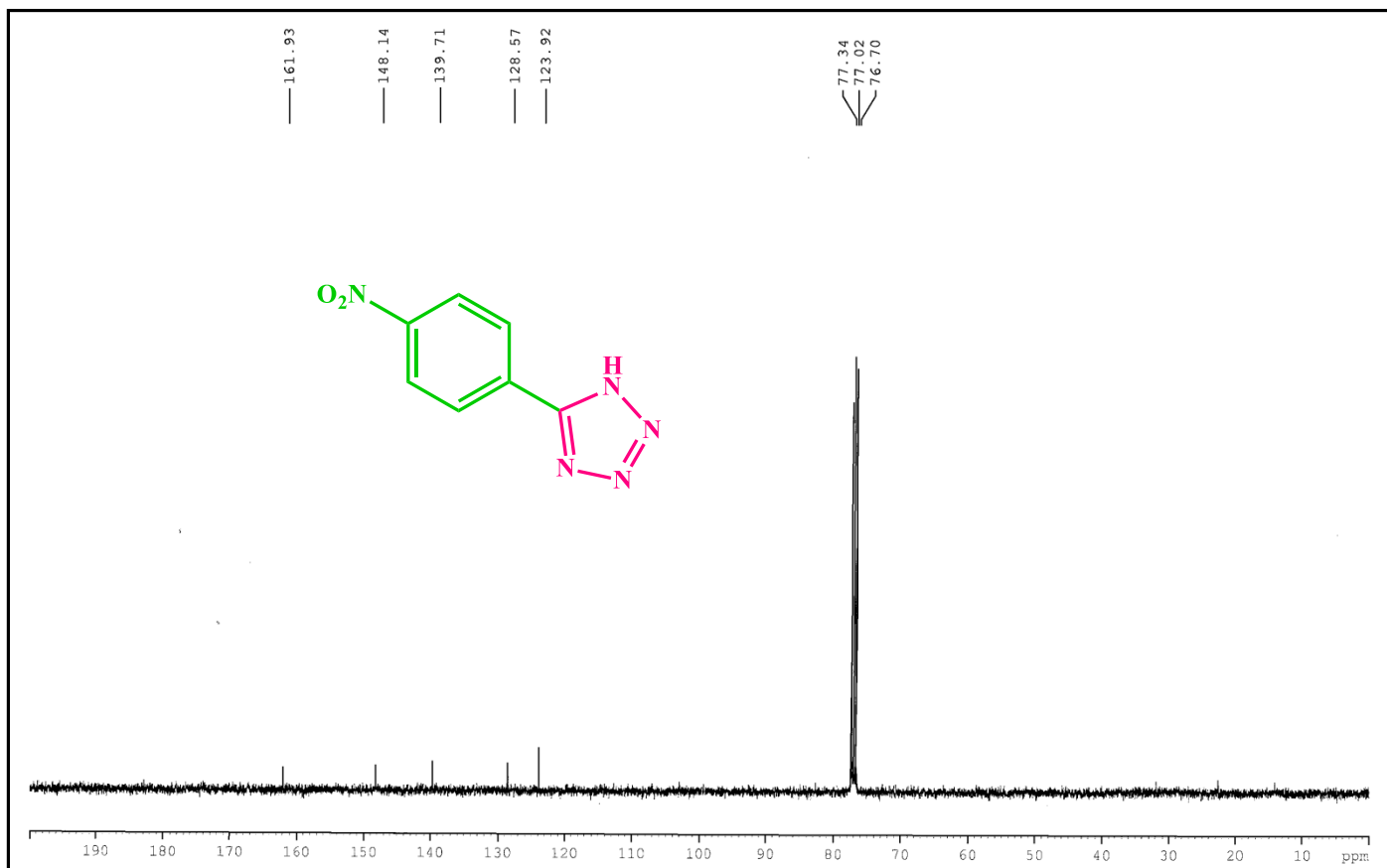
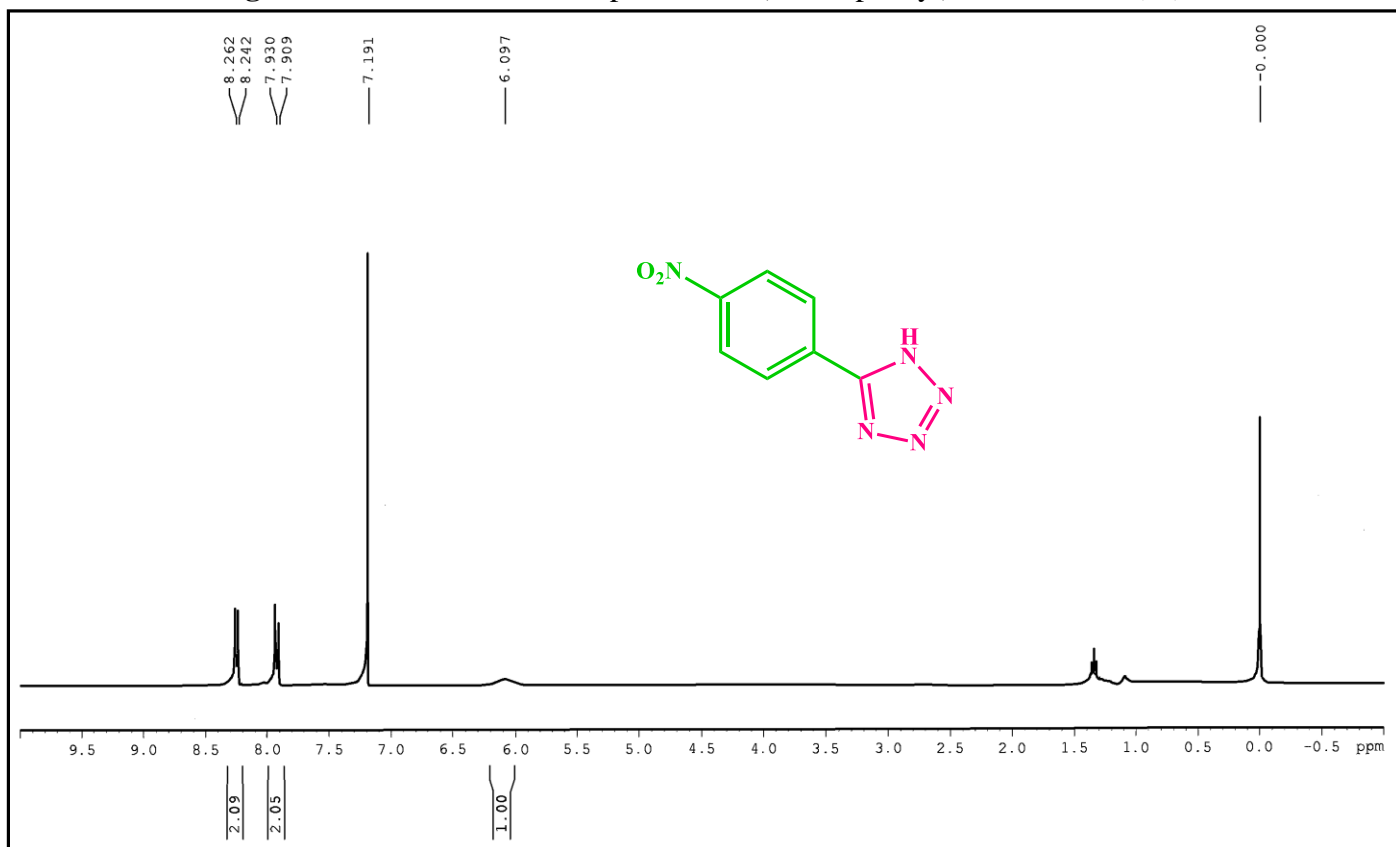


Figure S6. ^1H and ^{13}C NMR Spectra of 5-(4-chlorophenyl)-1H-tetrazole (**2f**)

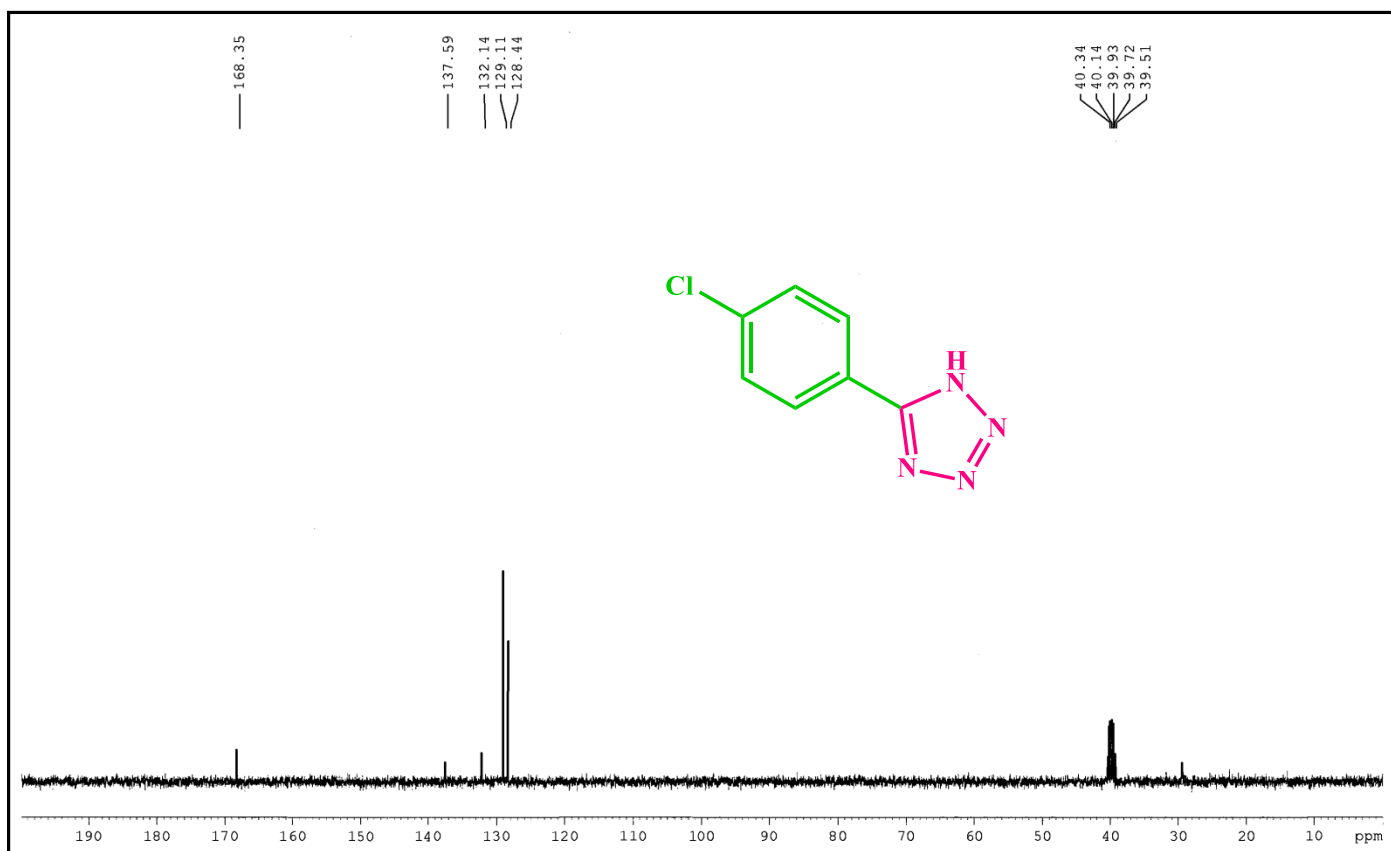
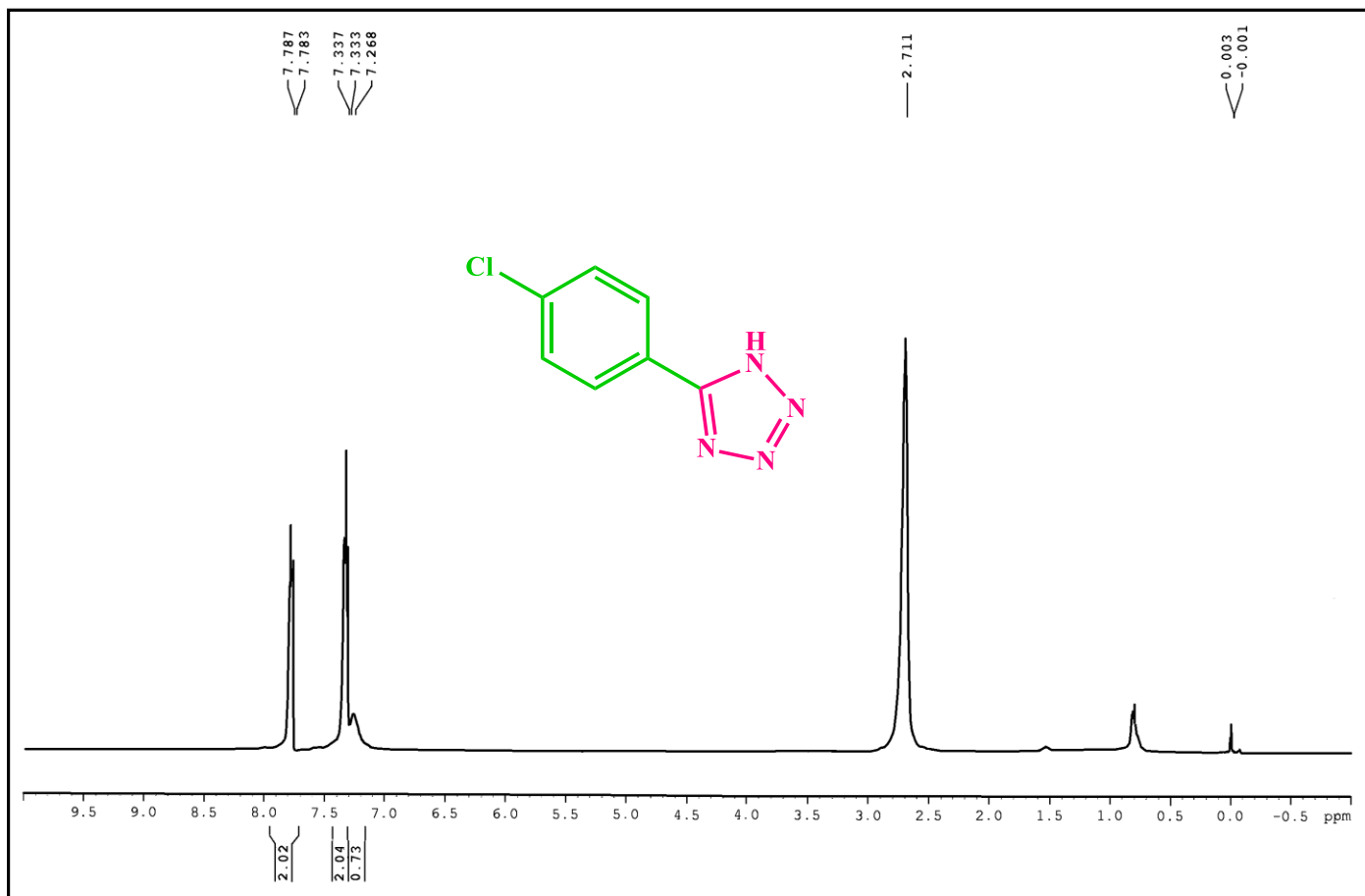


Figure S7. ^1H and ^{13}C NMR Spectra of 5-(4-bromophenyl)-1*H*-tetrazole (**2h**)

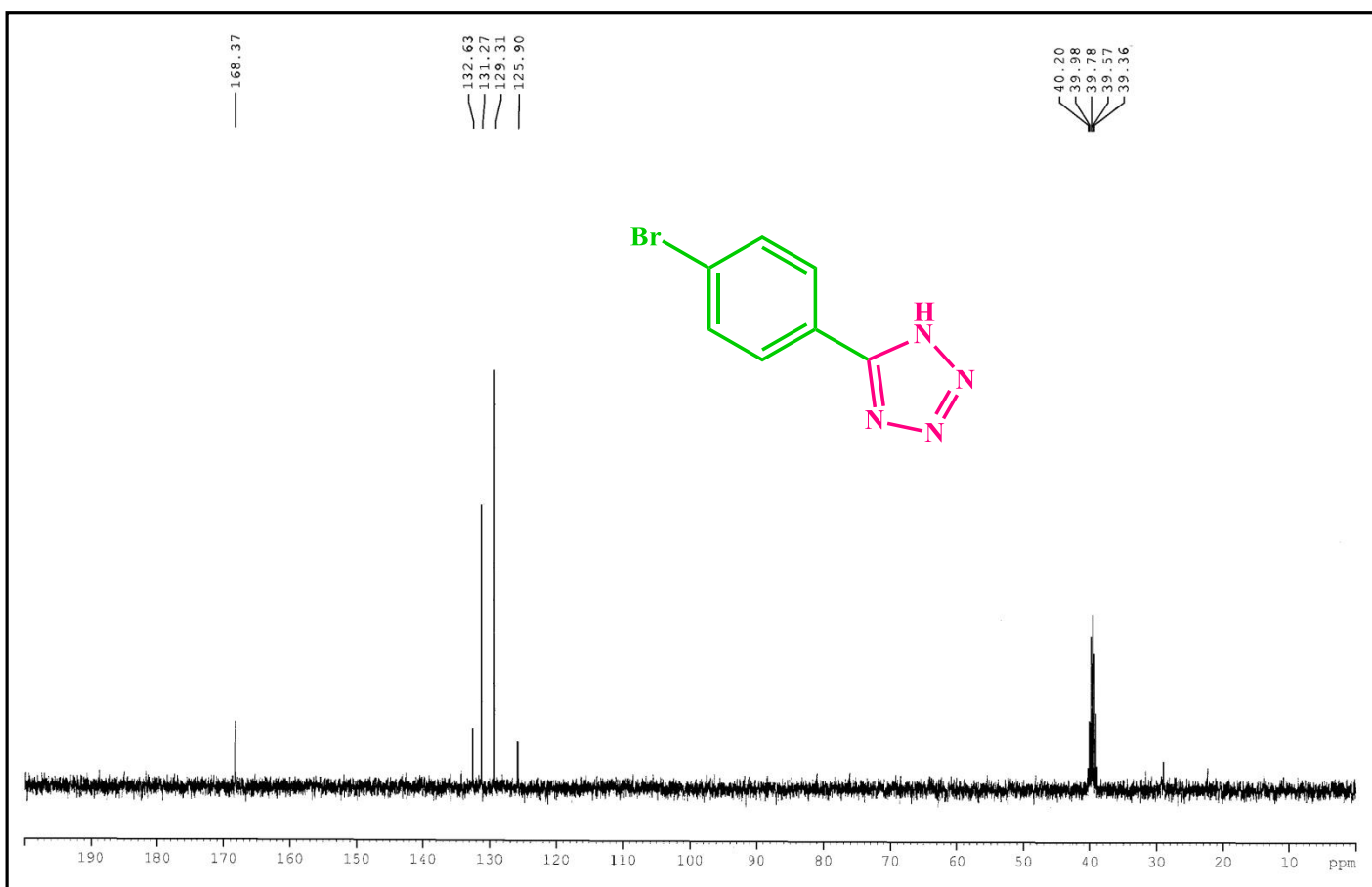
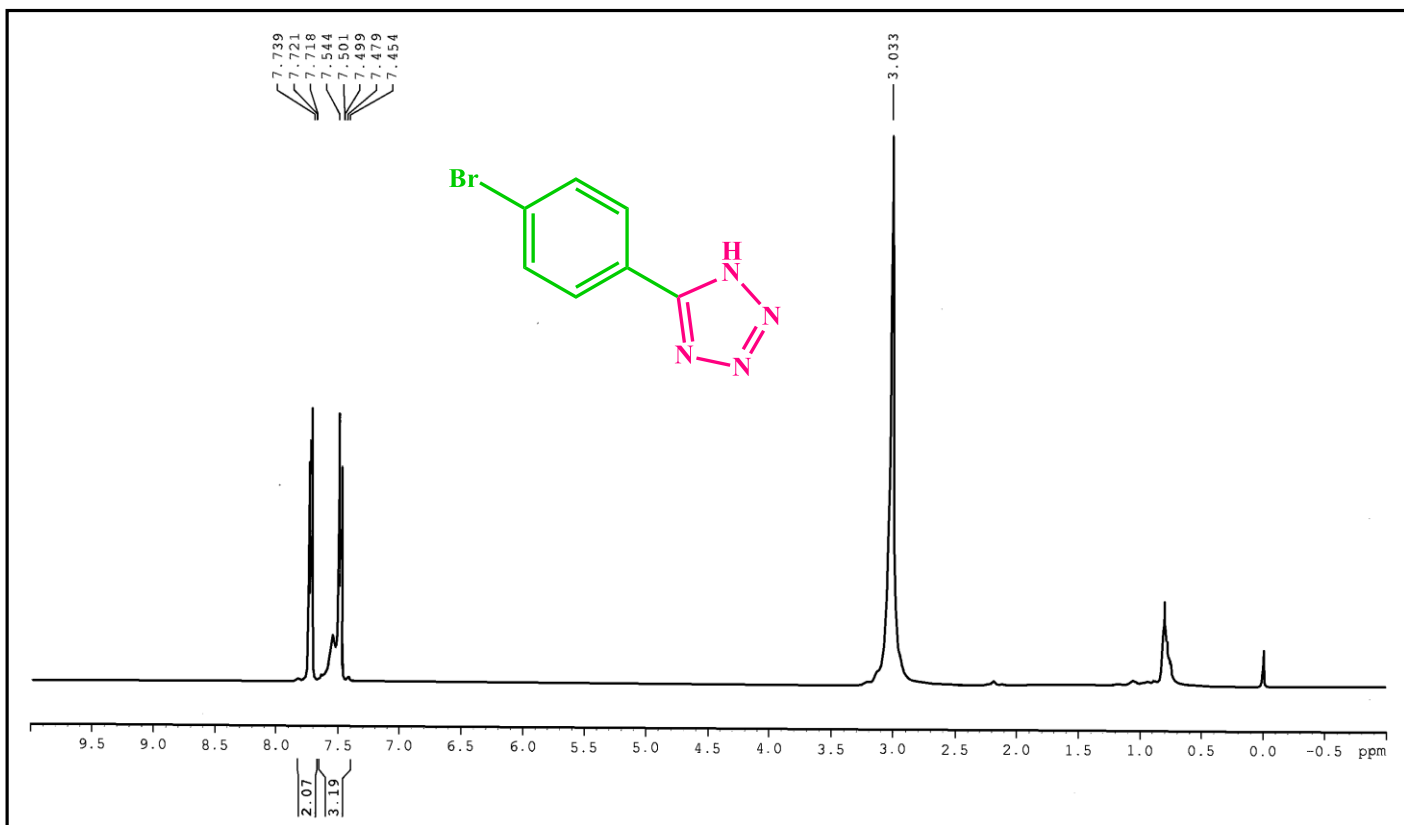


Figure S8. ^1H and ^{13}C NMR Spectra of 5-(3,4-dimethoxyphenyl)-1*H*-tetrazole (**2i**)

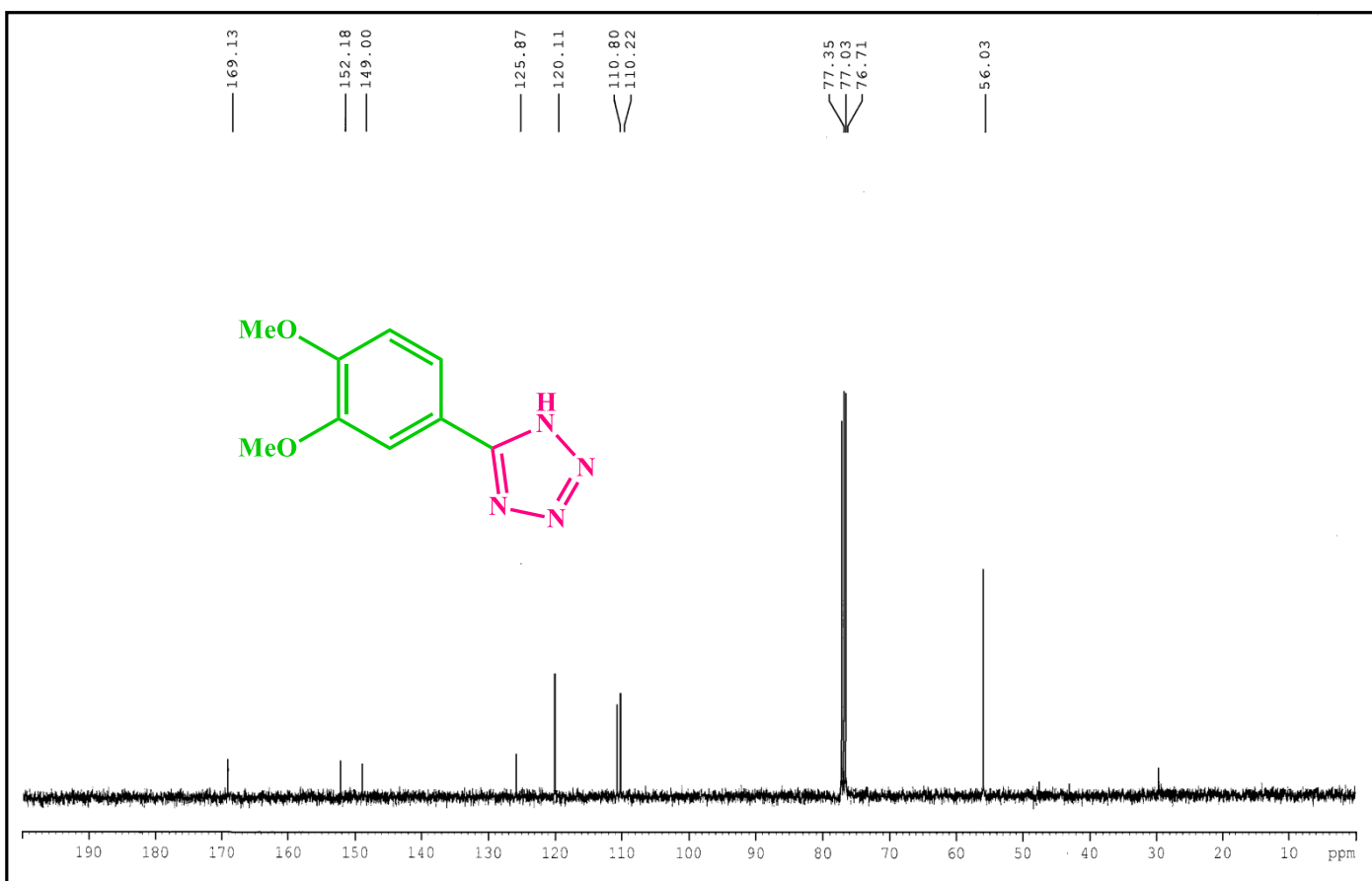
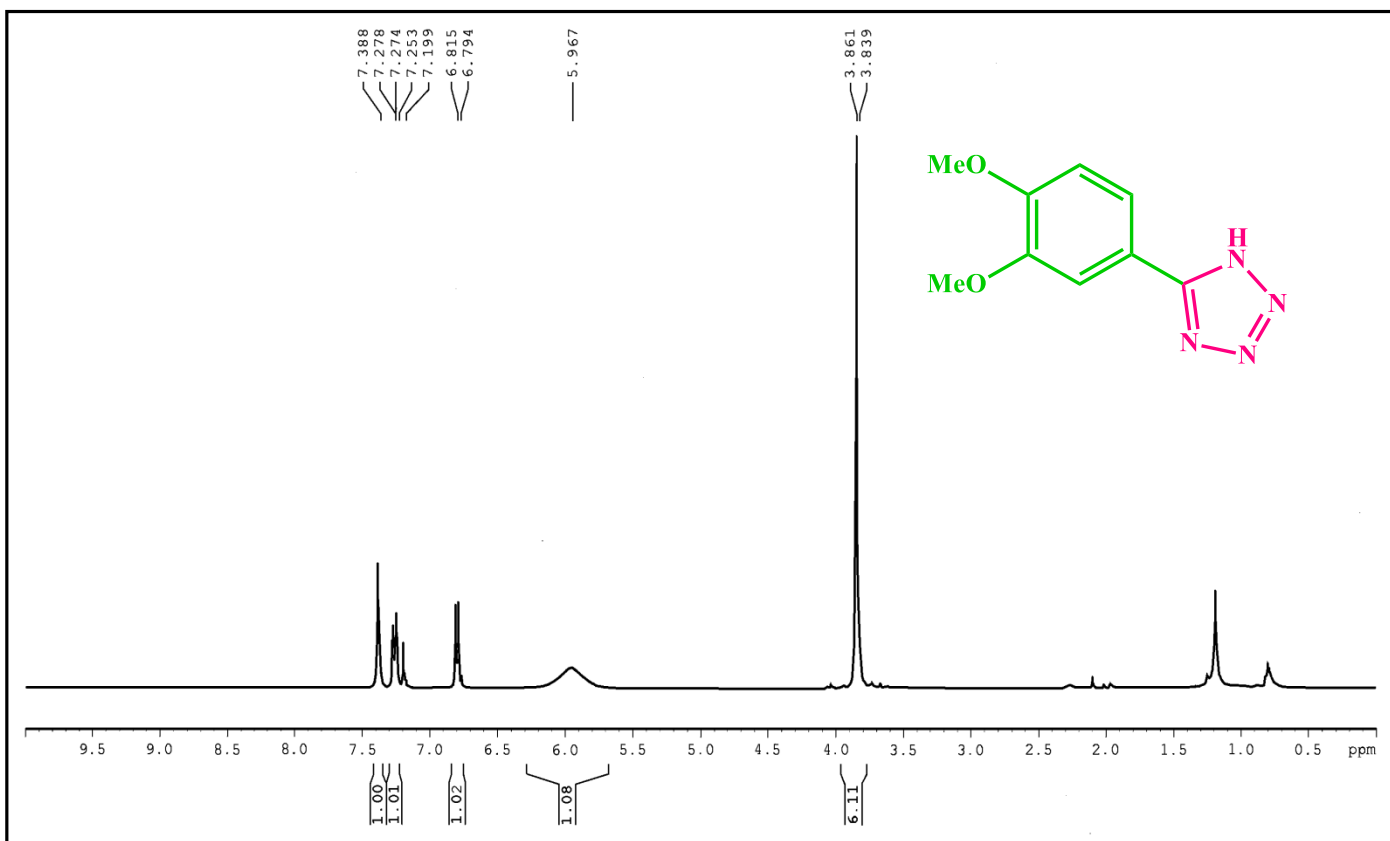


Figure S9. ^1H and ^{13}C NMR Spectra of 5-(4-methoxyphenyl)-1*H*-tetrazole (**2j**)

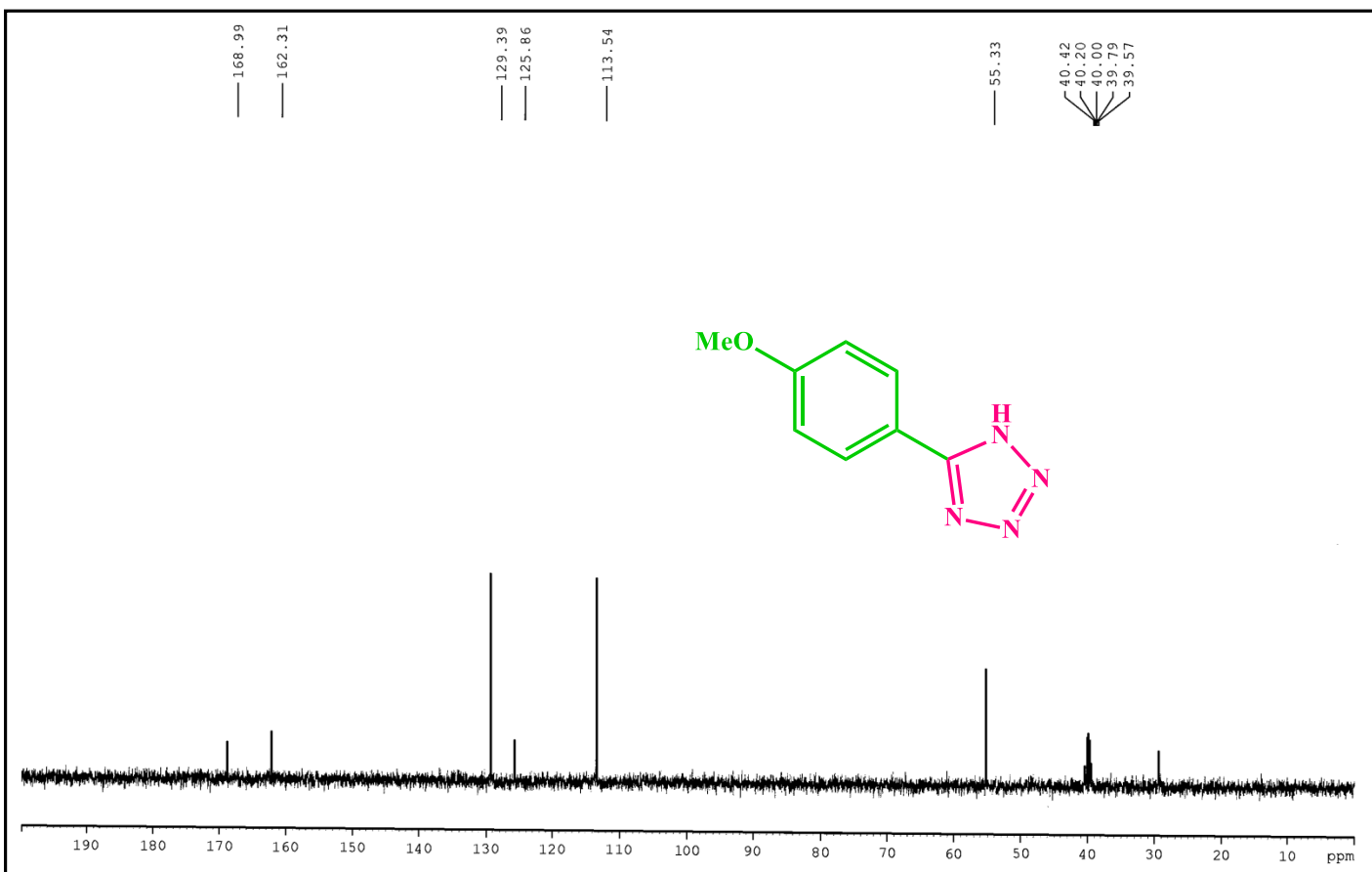
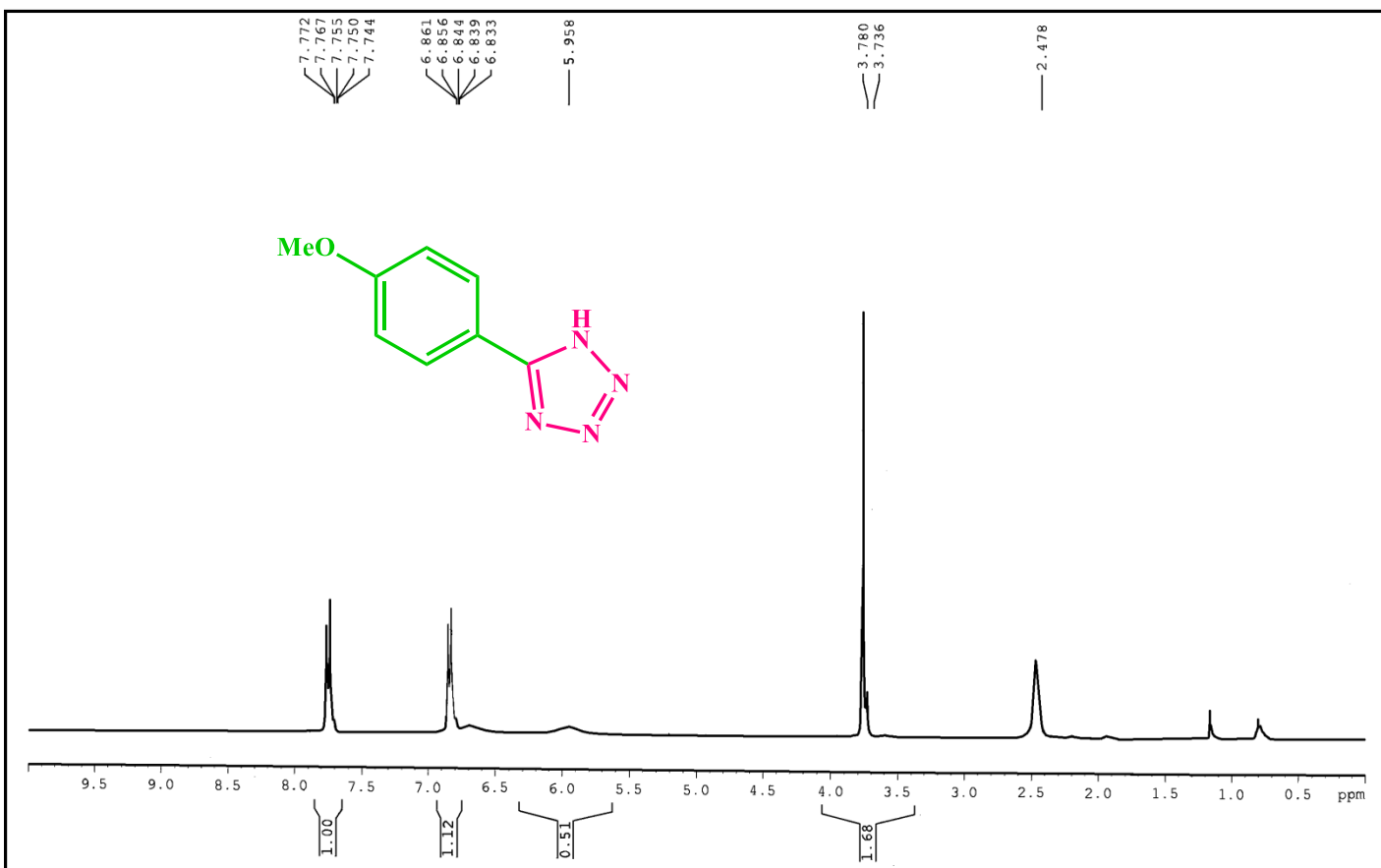


Figure S10. ^1H and ^{13}C NMR Spectra of 2-(1*H*-tetrazol-5-yl)phenol (**2k**)

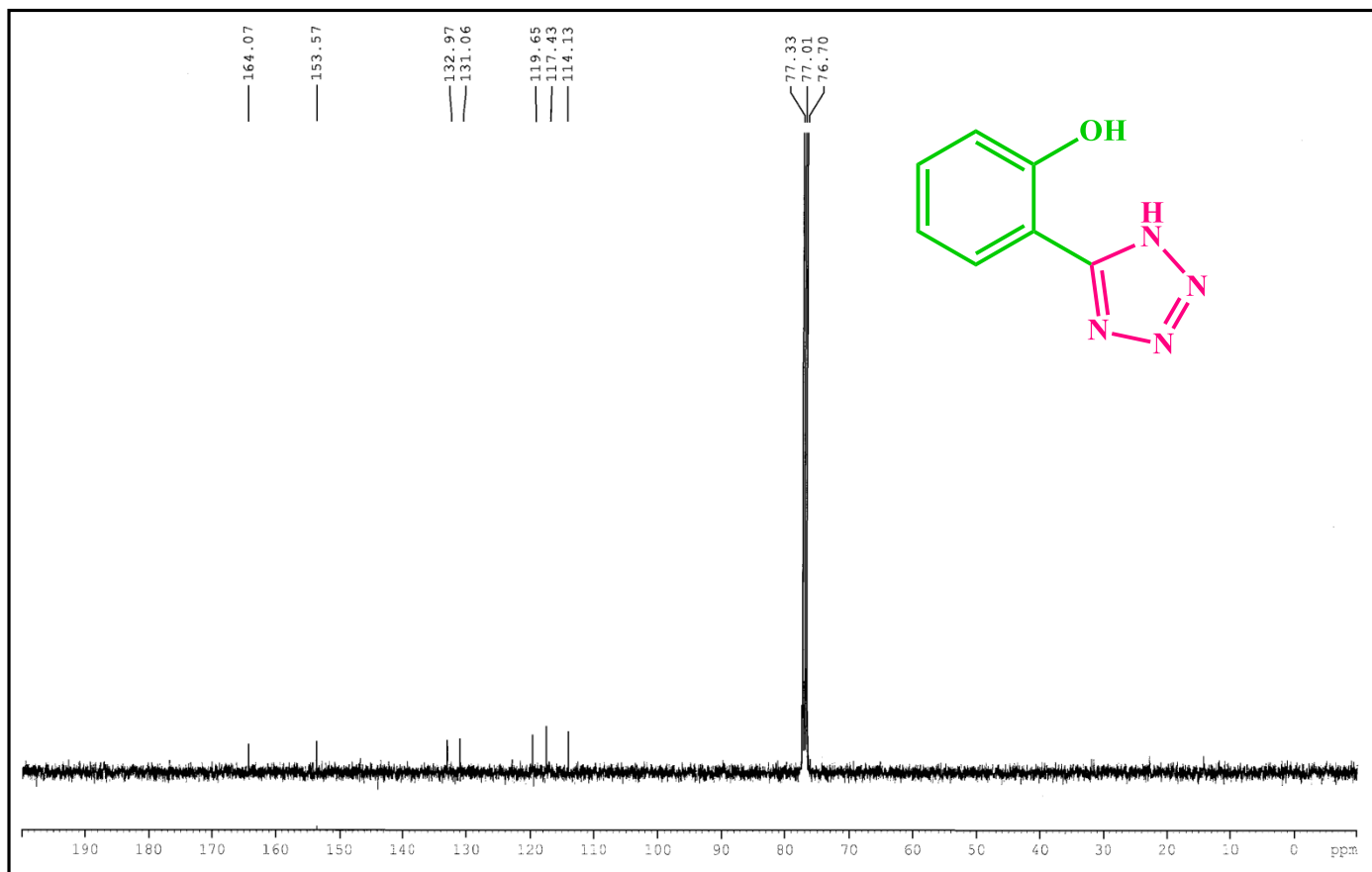
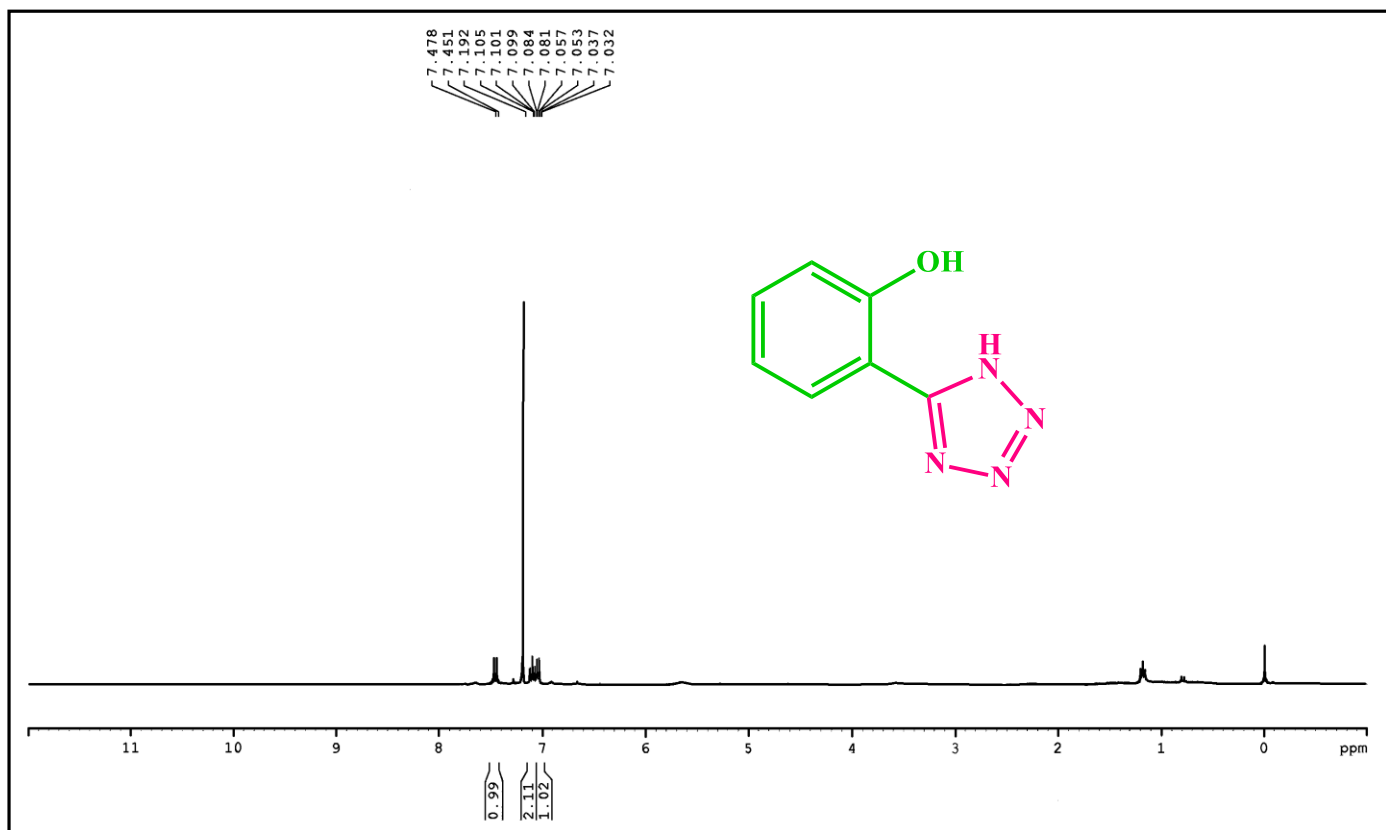


Figure S11. ^1H and ^{13}C NMR Spectra of 4-(1*H*-tetrazol-5-yl)phenol (**21**)

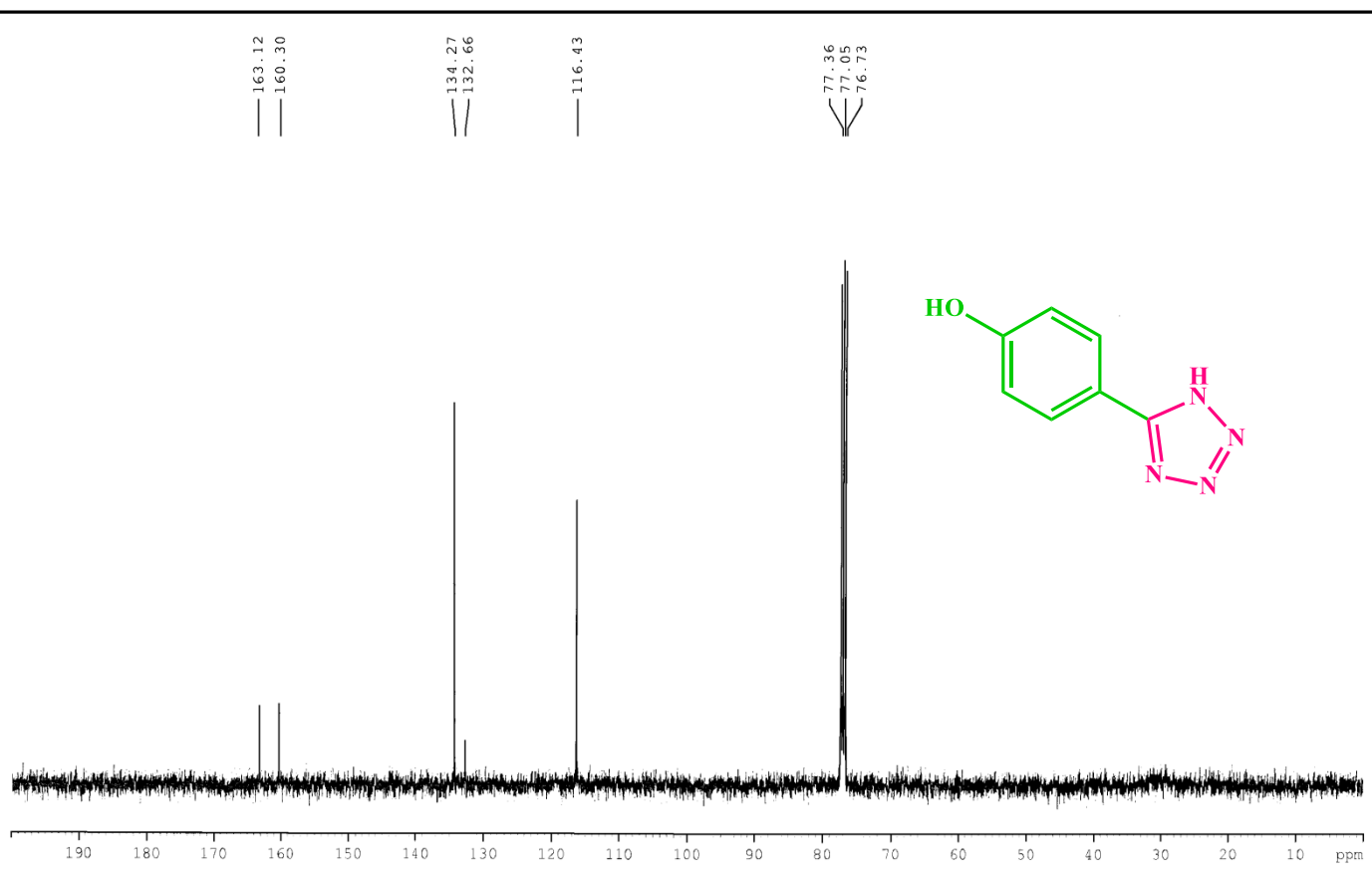
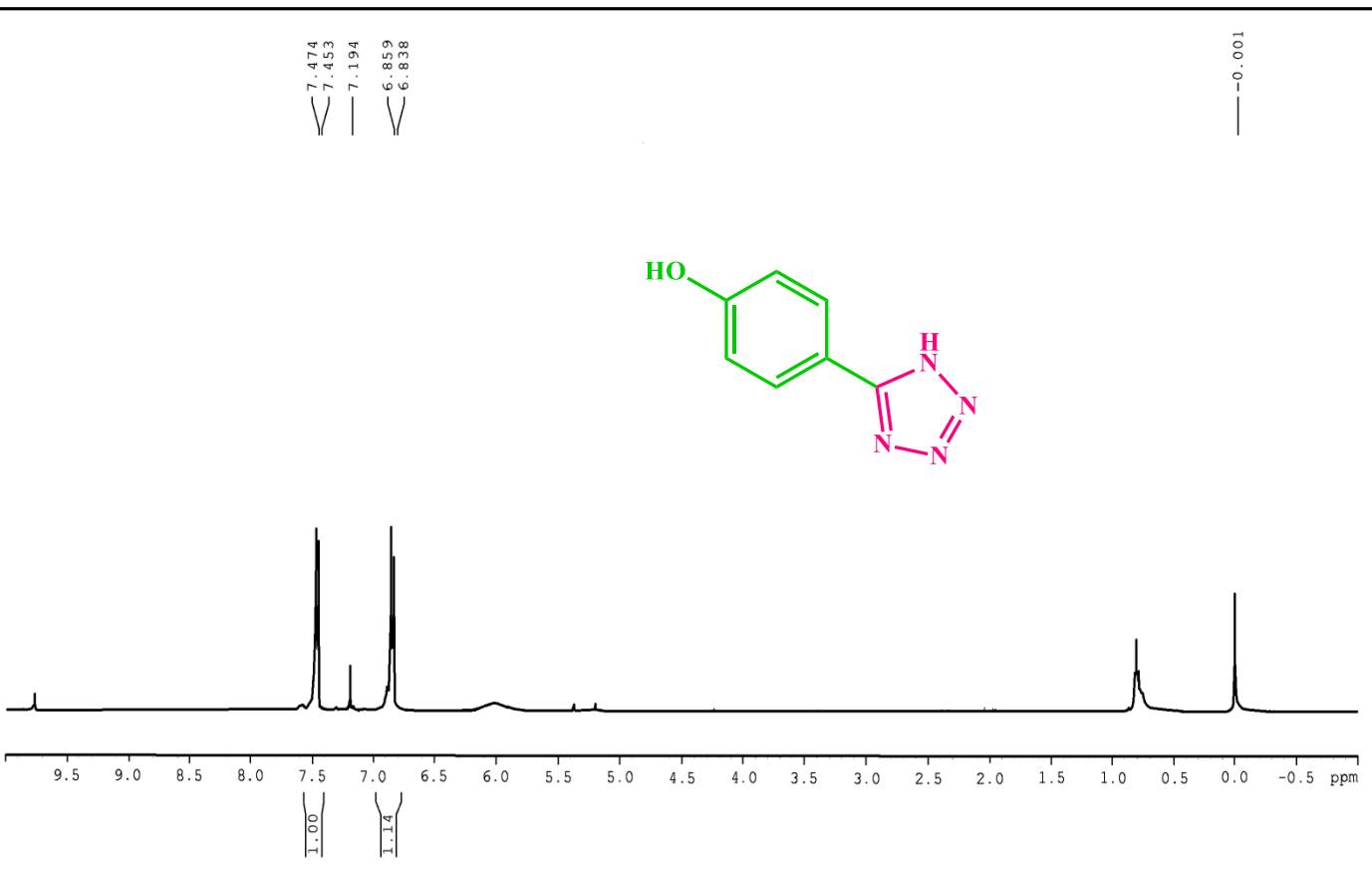


Figure S12. ^1H and ^{13}C NMR Spectra of 5-methyl-2-(1*H*-tetrazol-5-yl)phenol (**2m**)

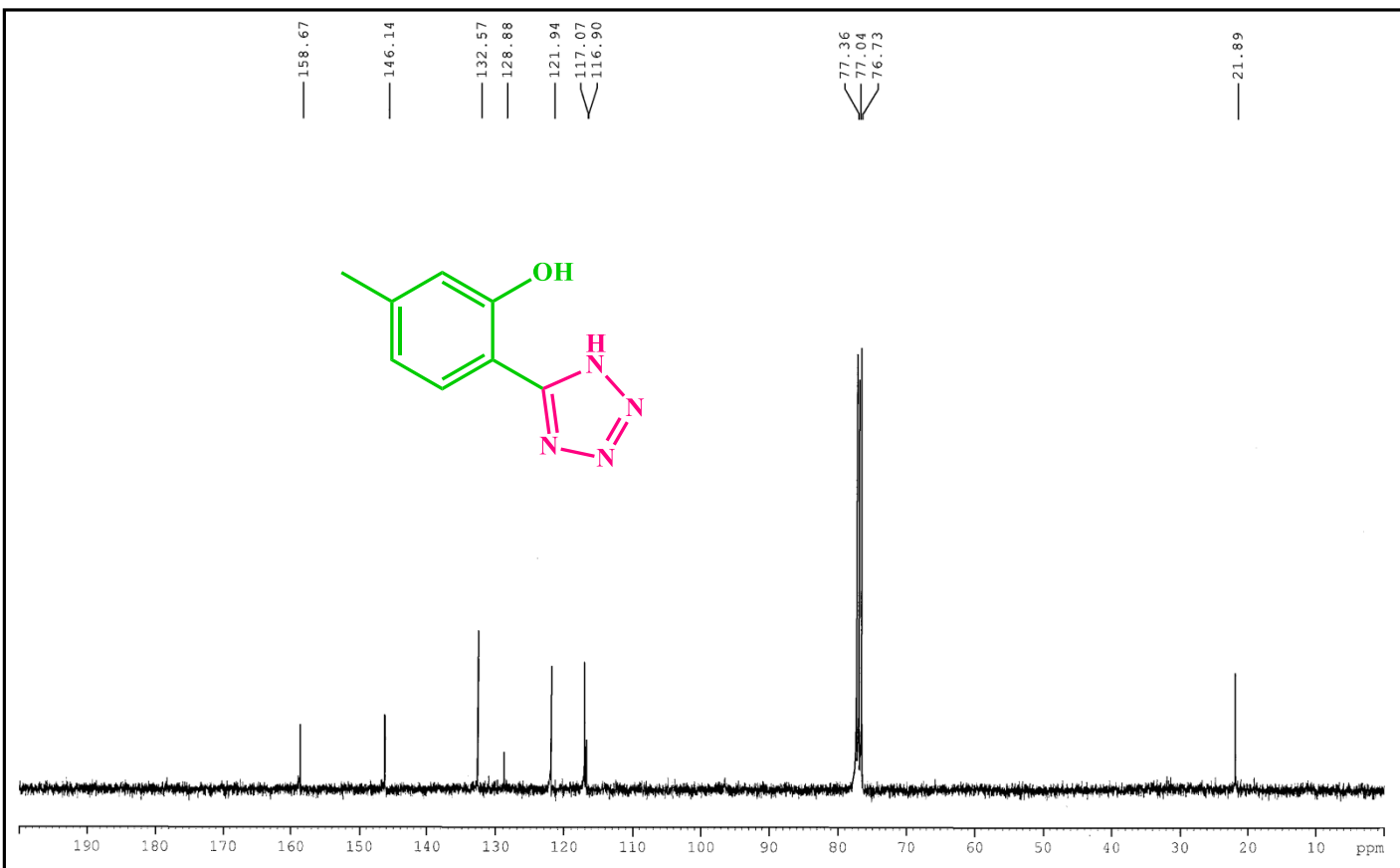
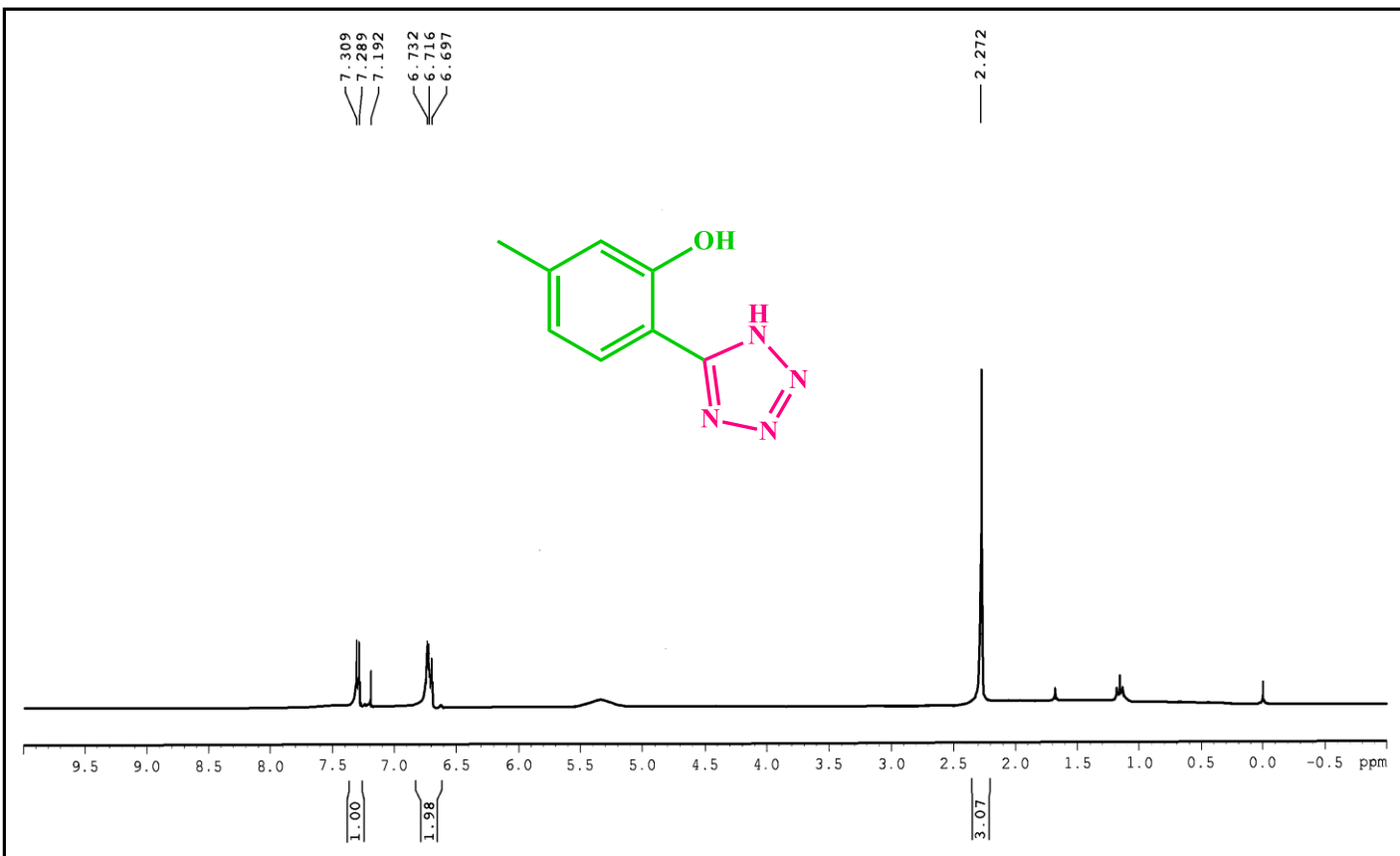


Figure S13. ^1H and ^{13}C NMR Spectra of 5-(furan-2-yl)-1*H*-tetrazole (**3a**)

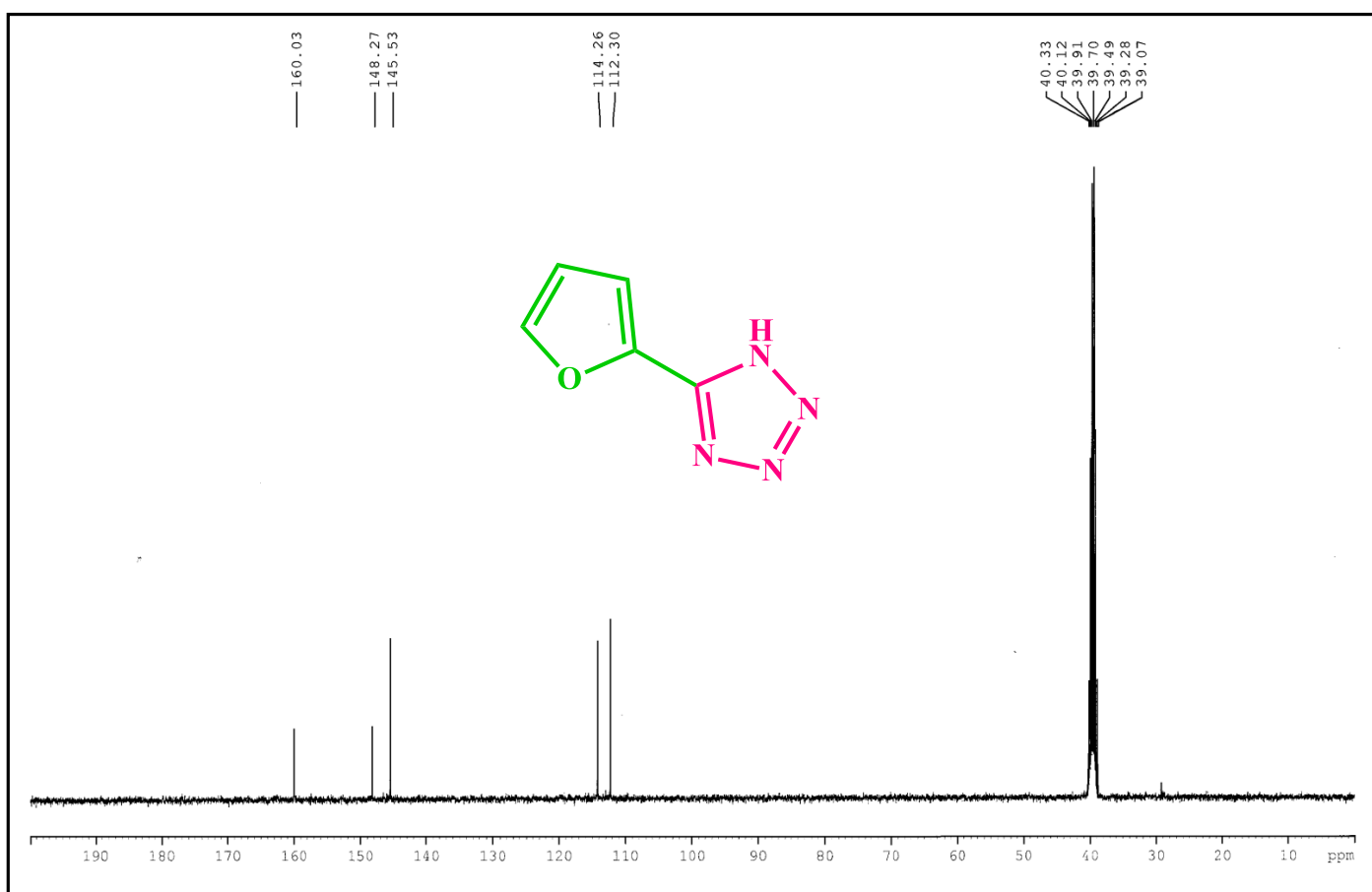
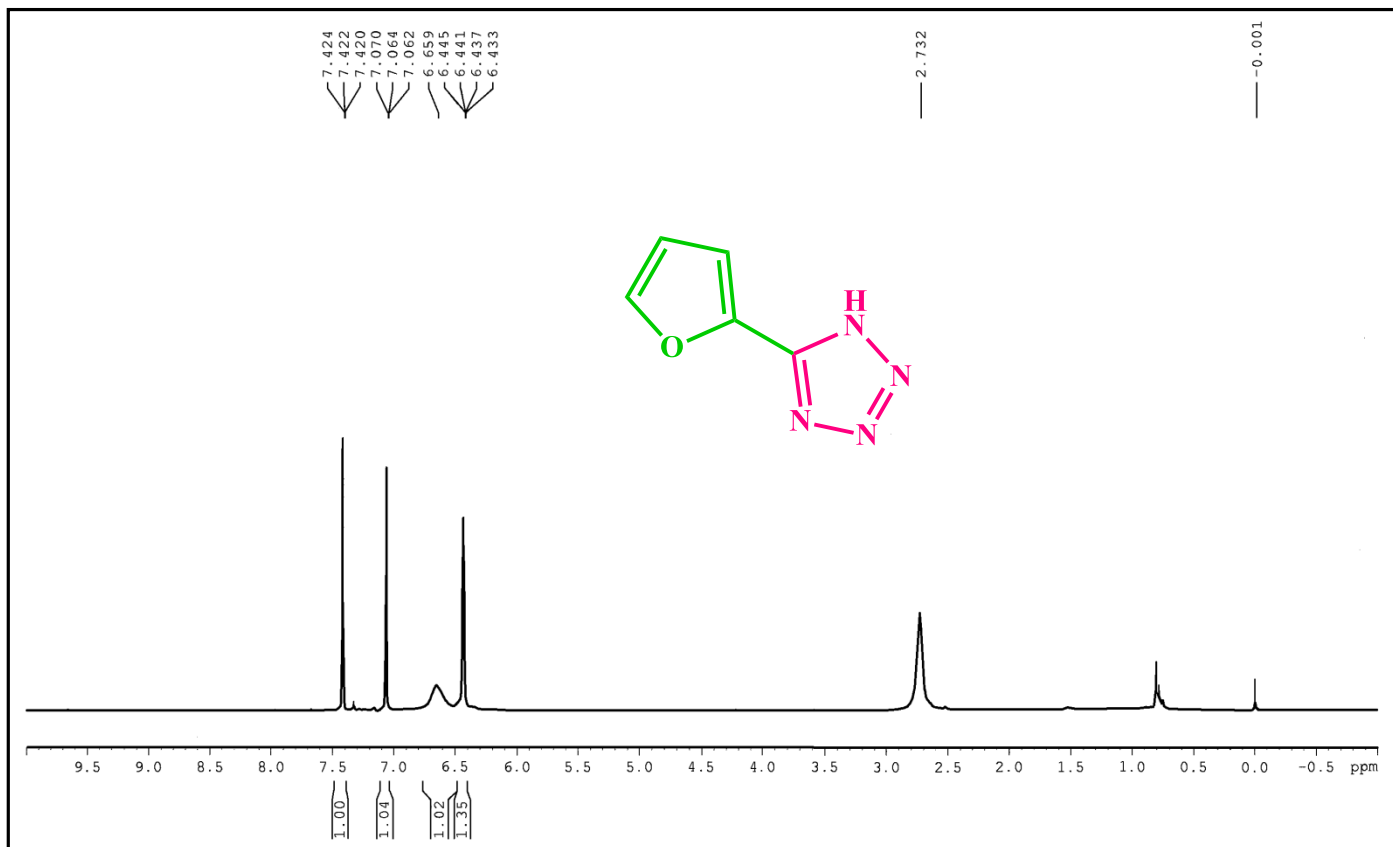


Figure S14. ^1H and ^{13}C NMR Spectra of 5-(thiophen-2-yl)-1H-tetrazole (**3b**)

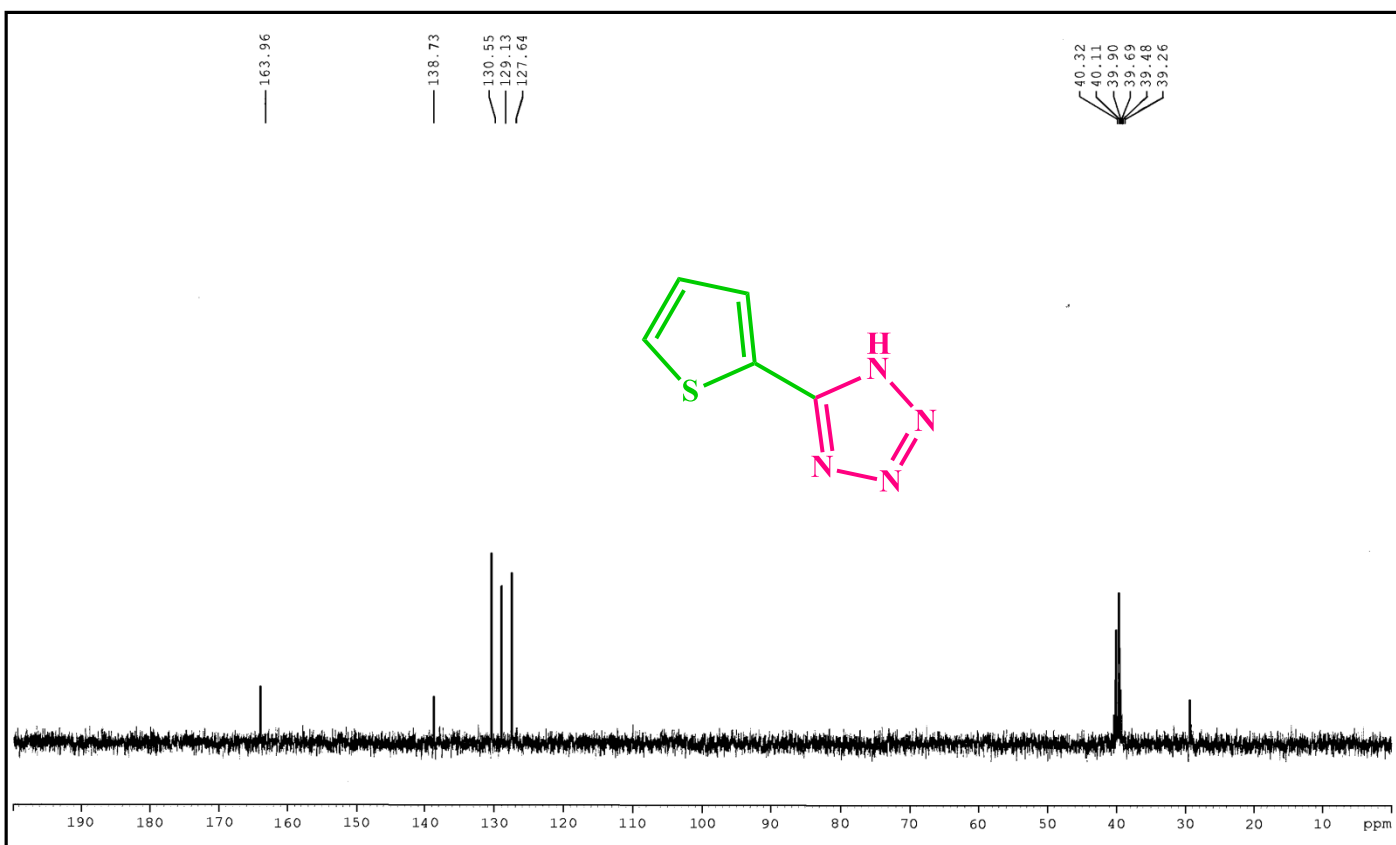
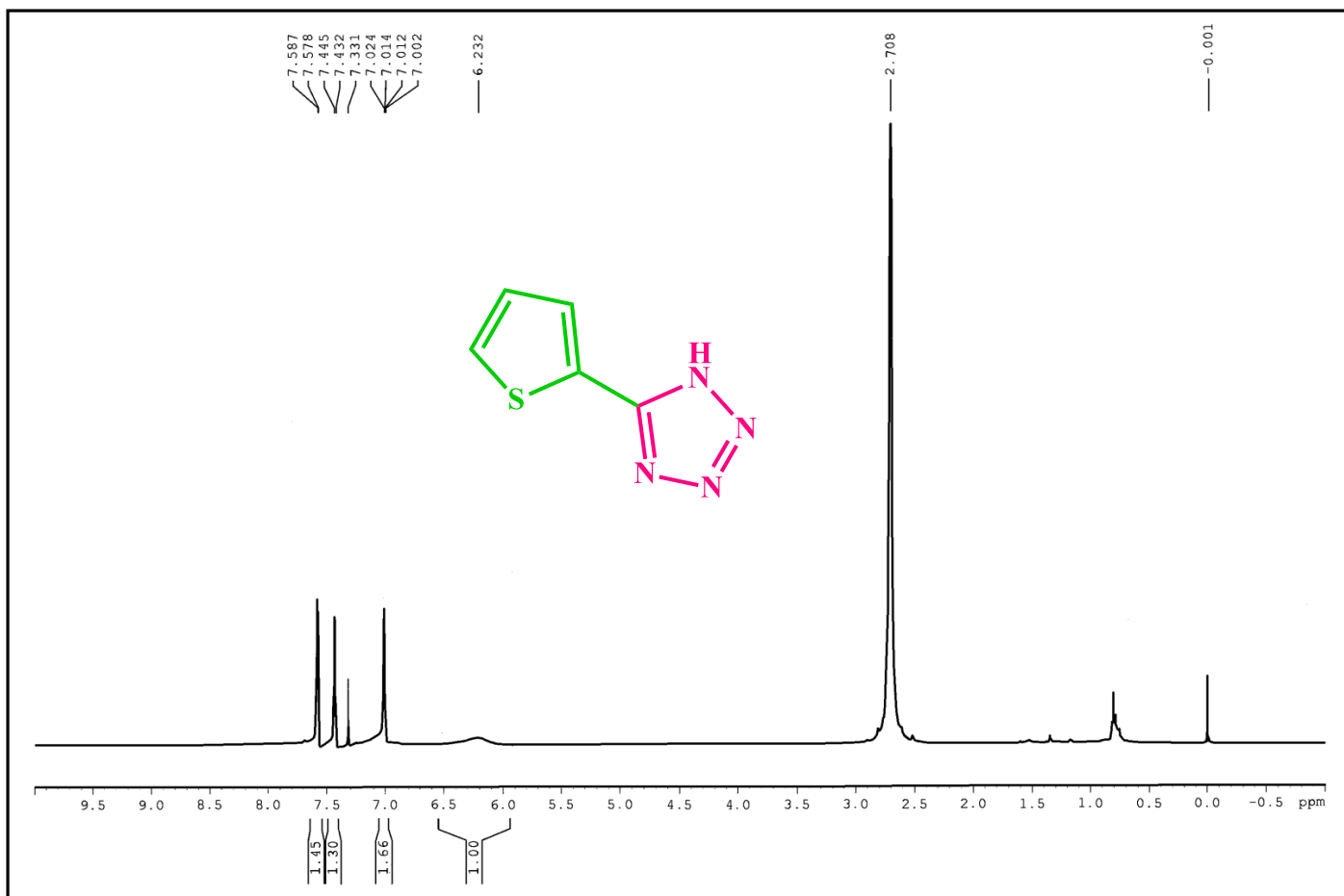


Figure S15. ^1H and ^{13}C NMR Spectra of 2-(1*H*-tetrazol-5-yl)pyridine (**3c**)

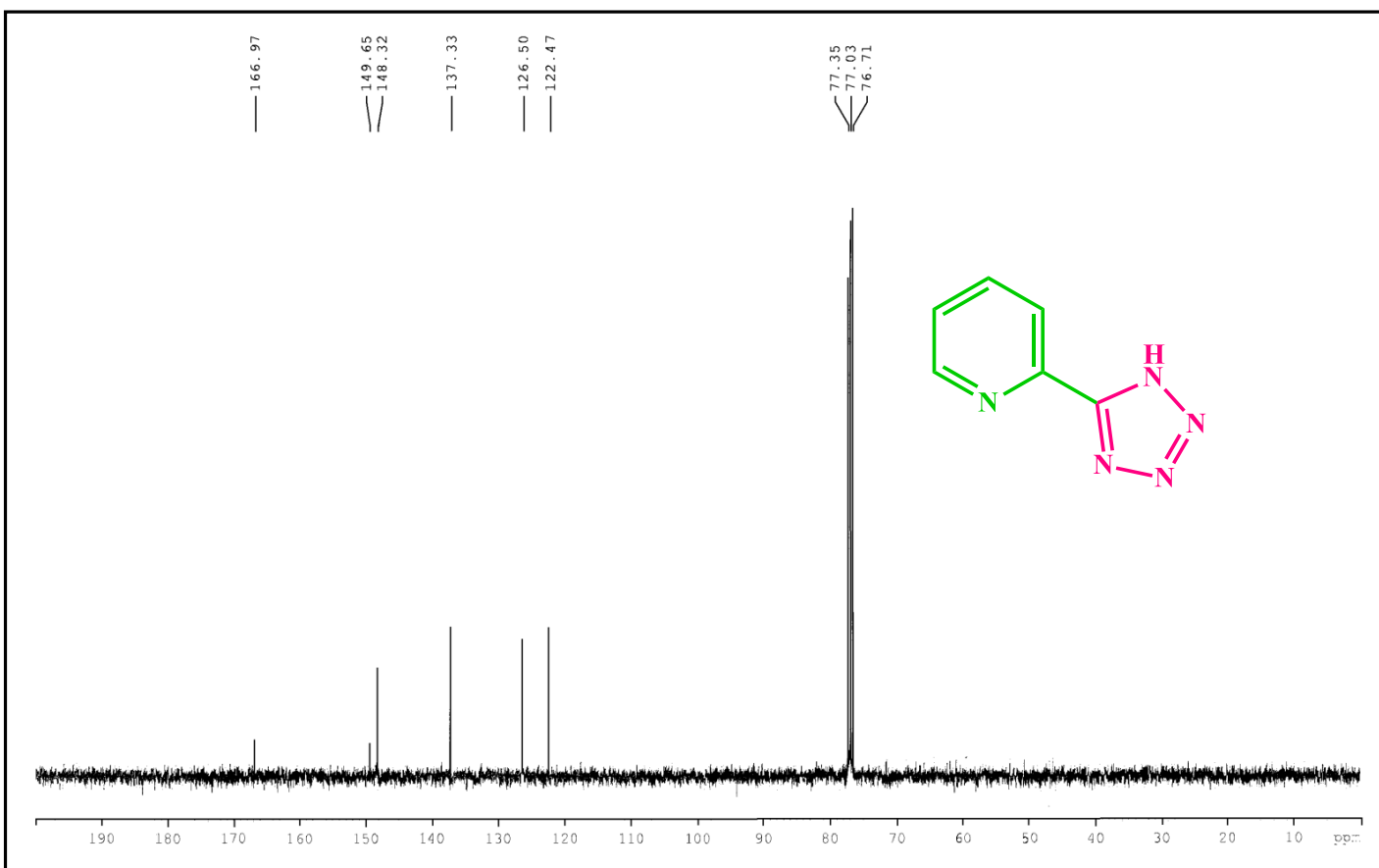
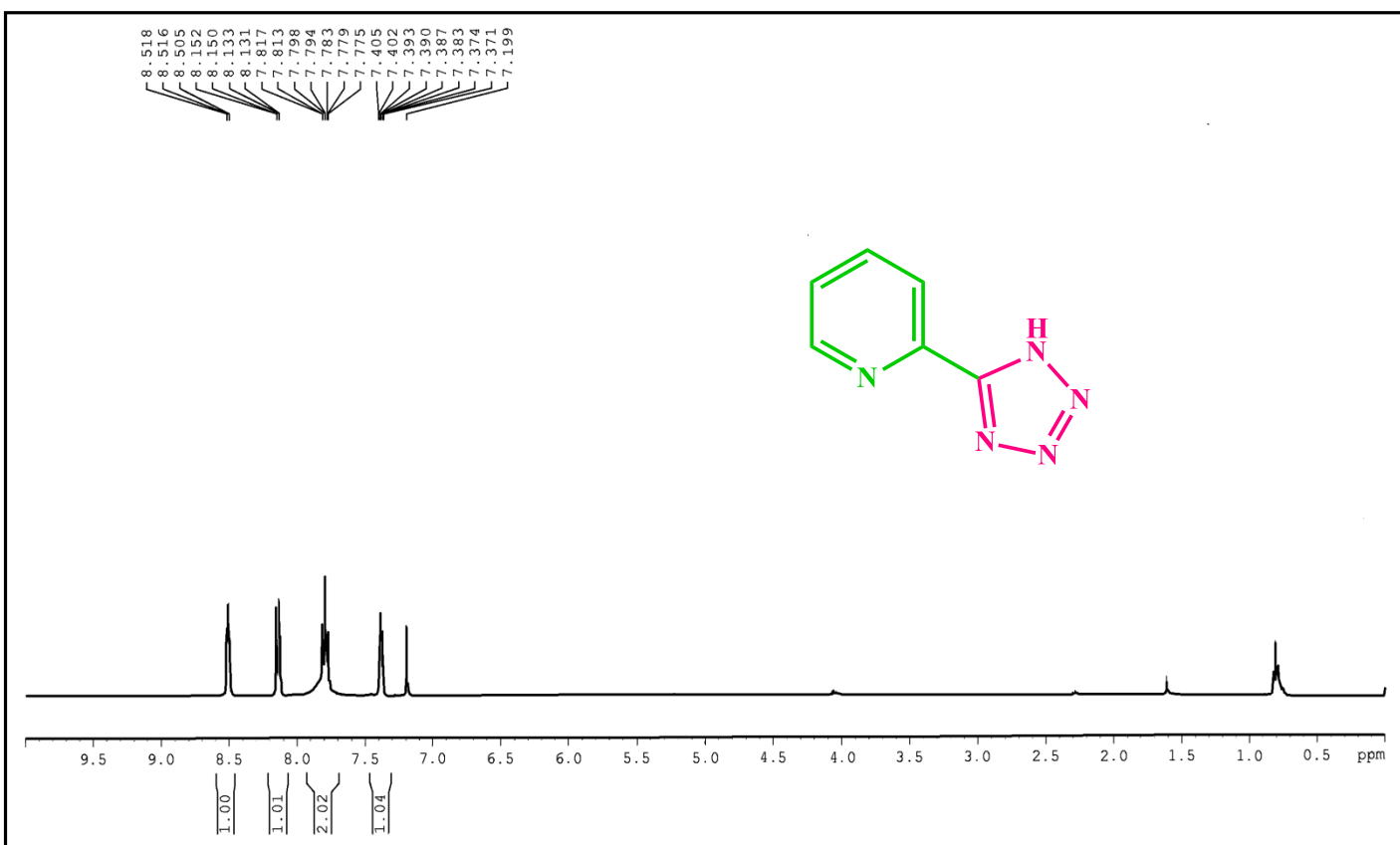


Figure S16. ^1H and ^{13}C NMR Spectra of 2-(1*H*-tetrazol-5-yl)quinoline (**3d**)

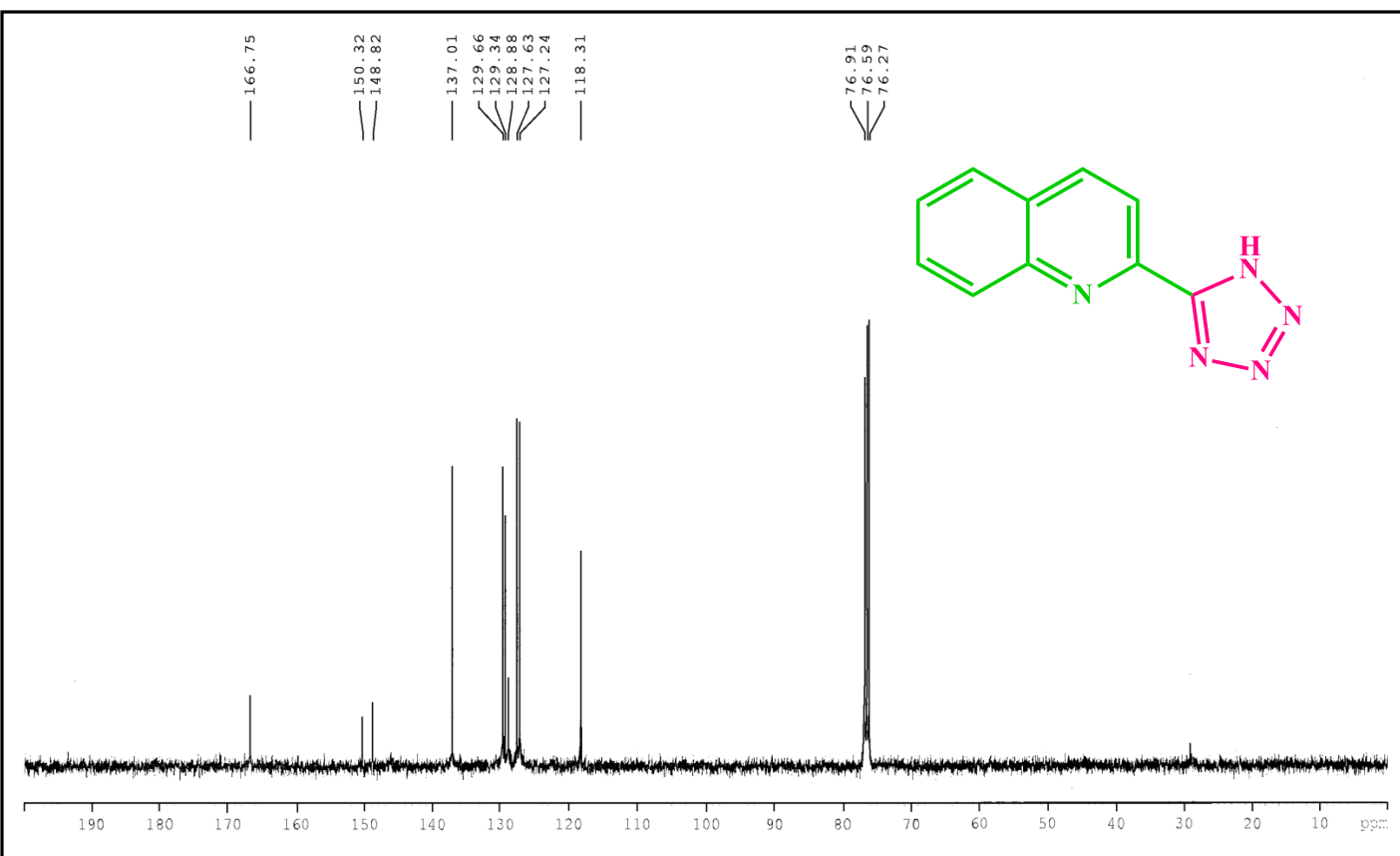
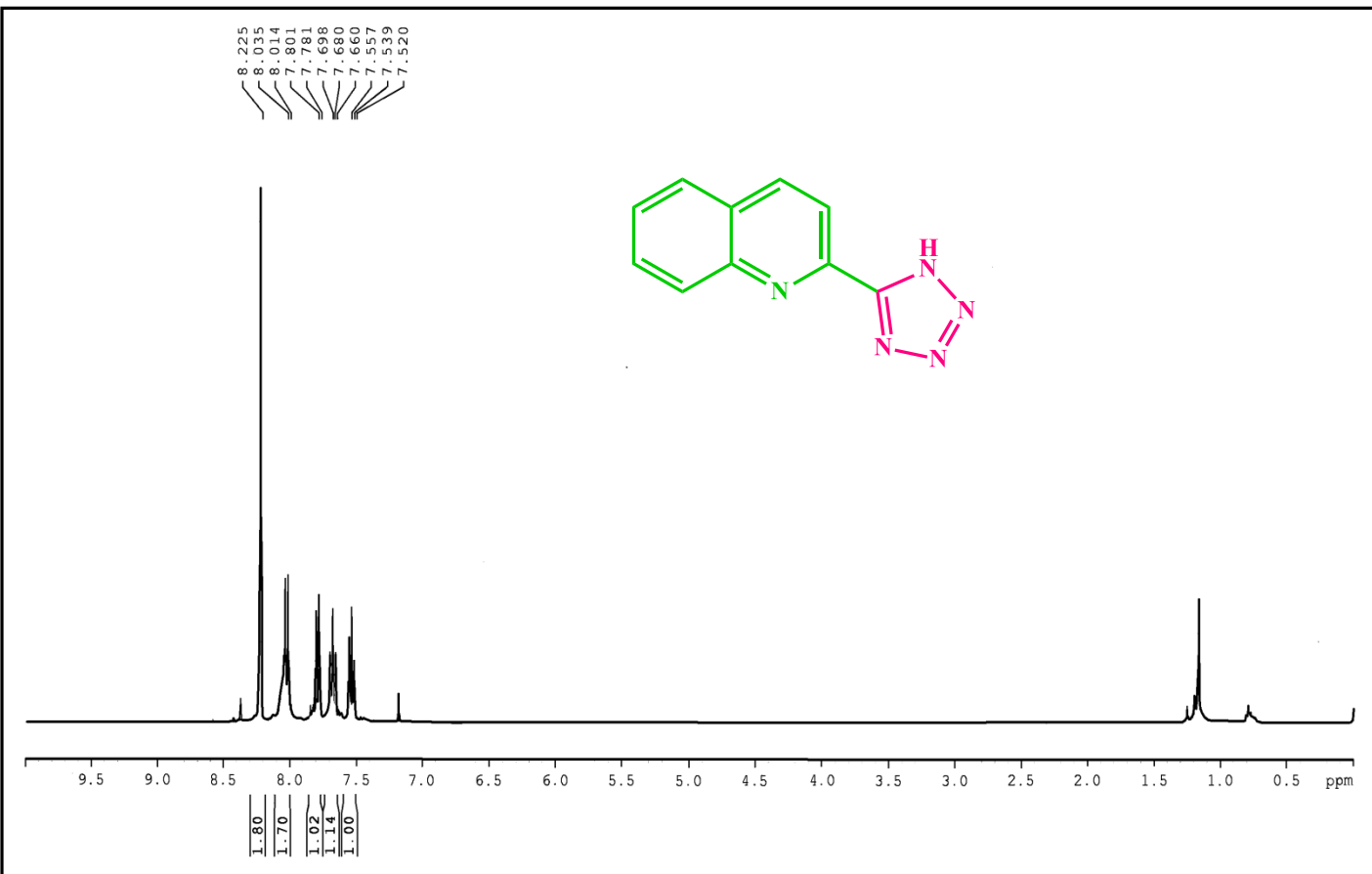


Figure S17. ^1H and ^{13}C NMR Spectra of 1H-tetrazole (4a)

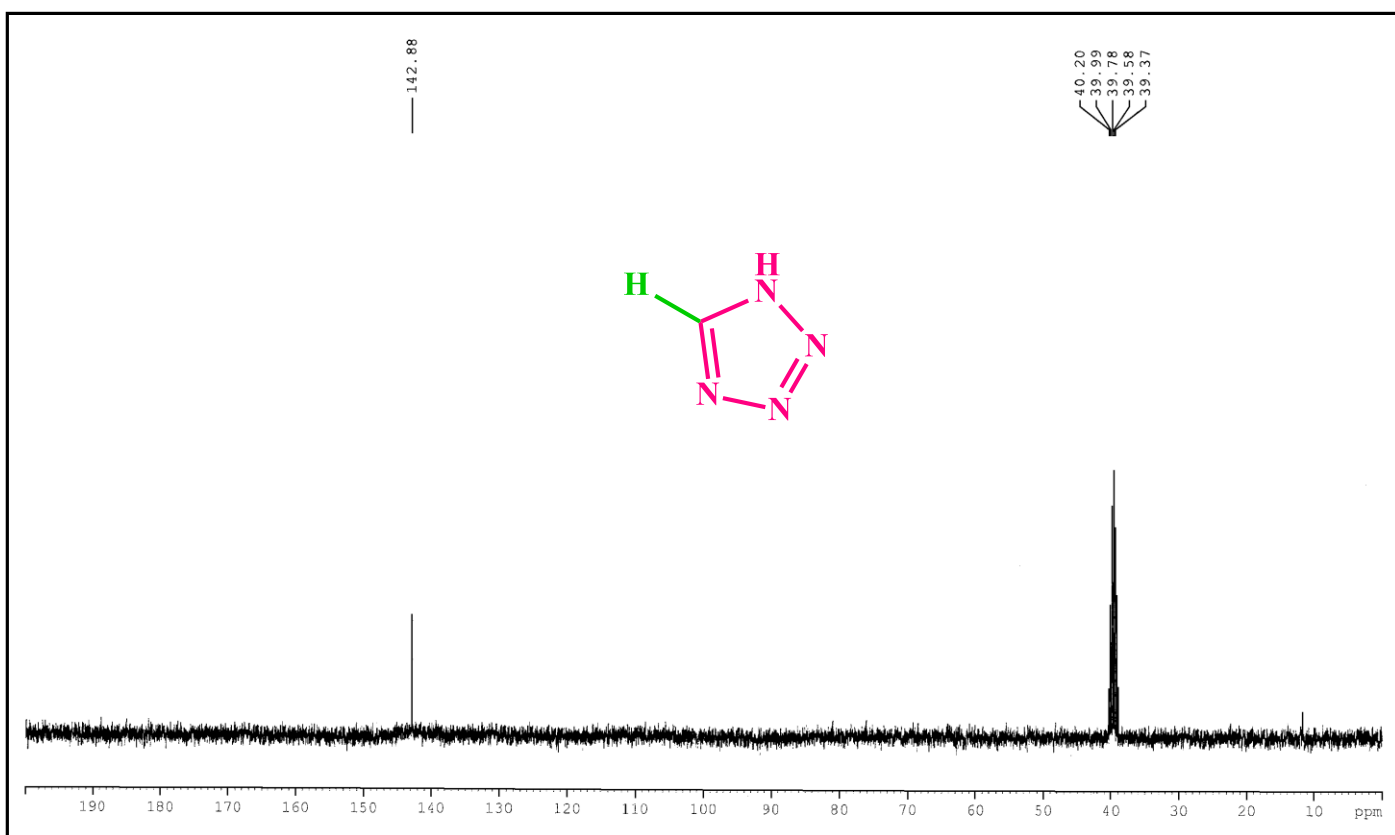
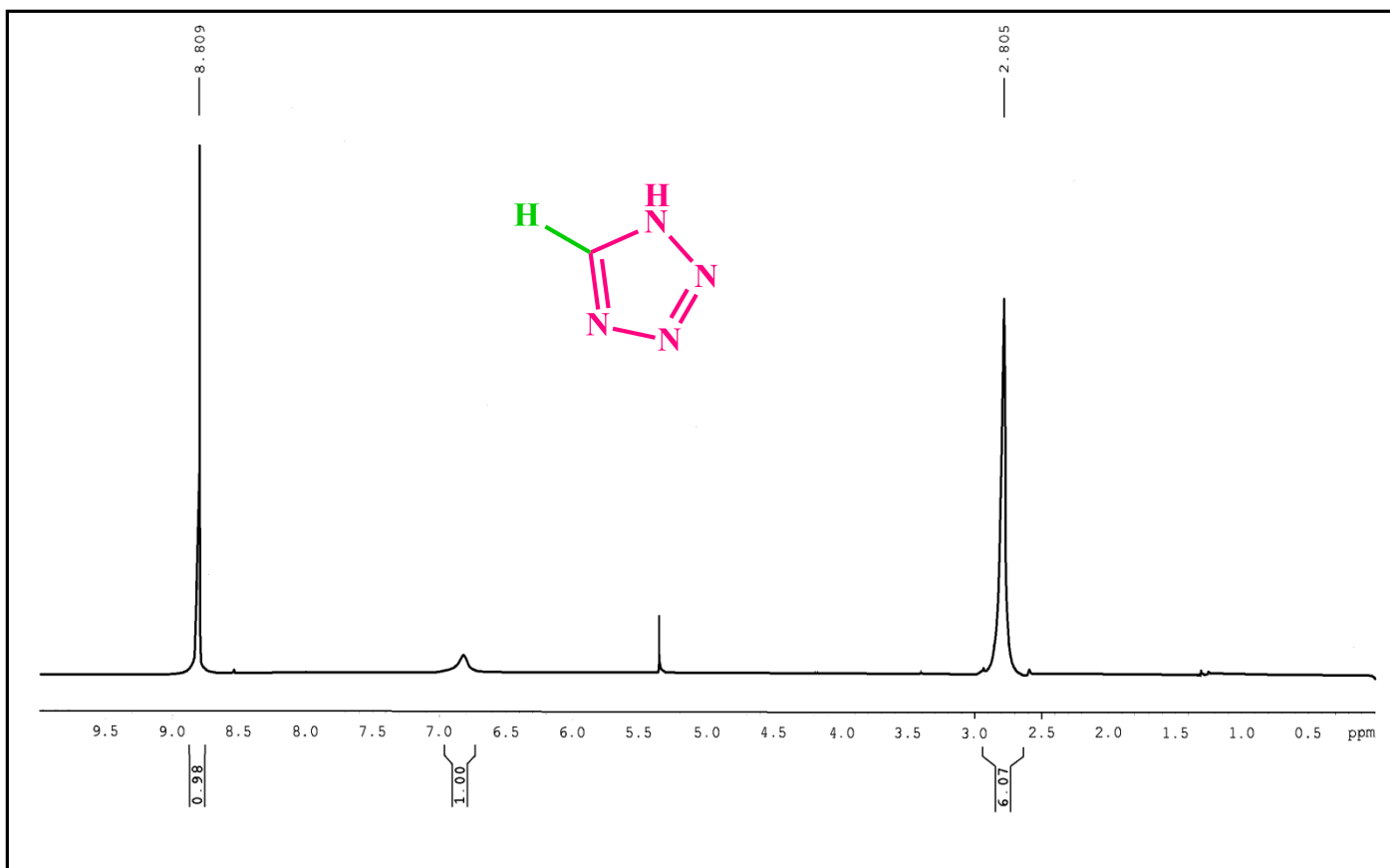


Figure S18. ¹H and ¹³C NMR Spectra of 1*H*-tetrazole (4a)

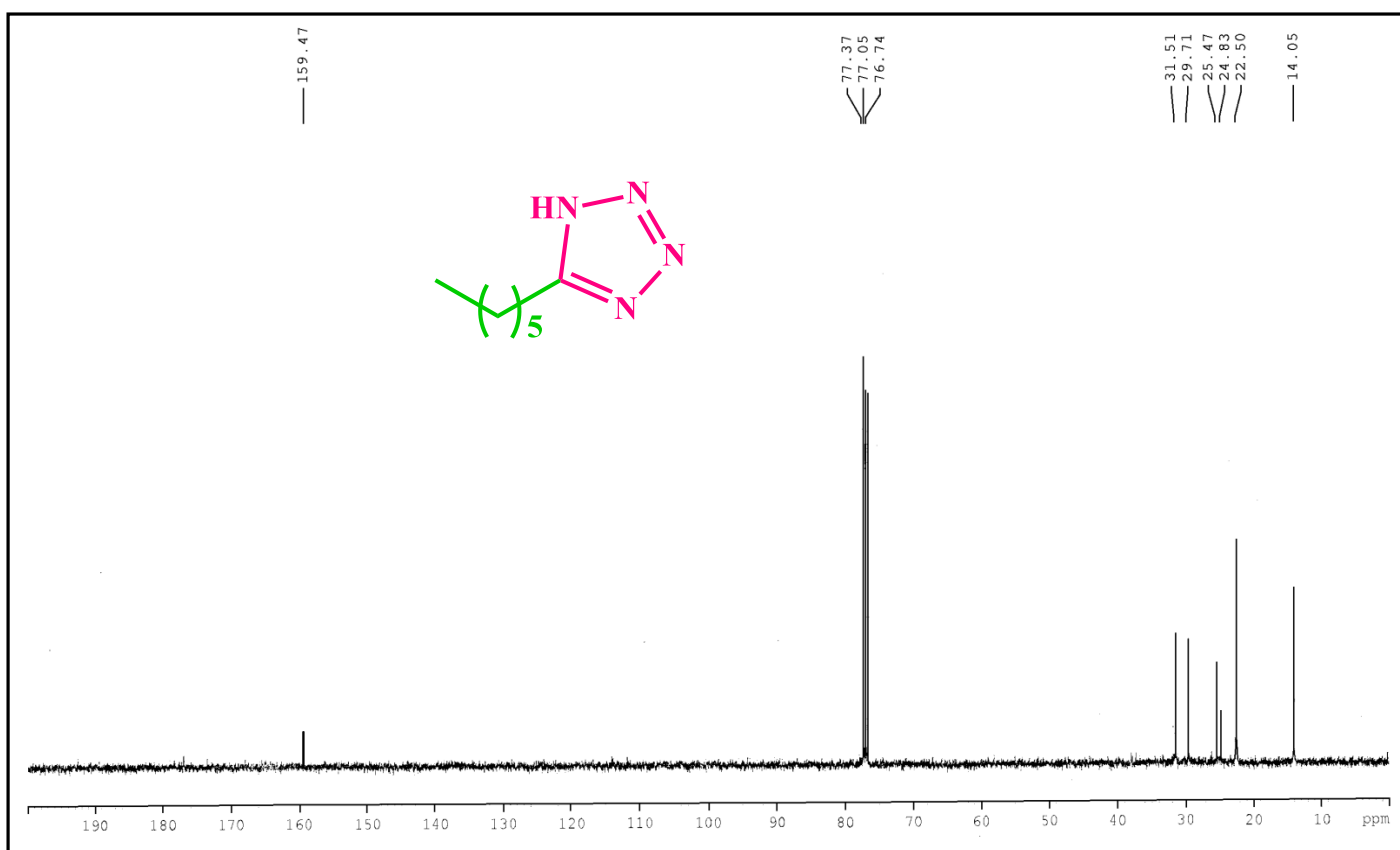
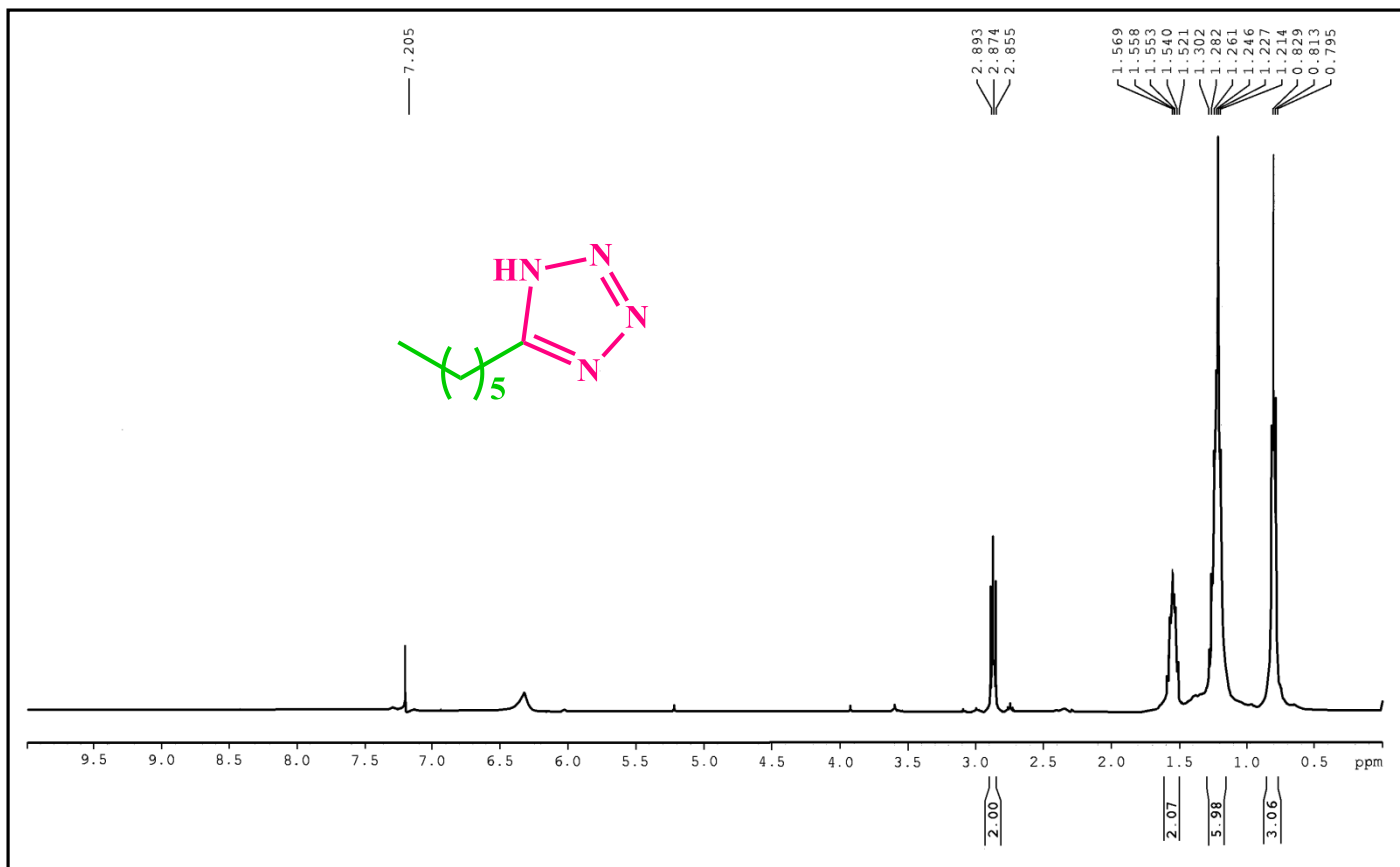


Figure S19. ^1H and ^{13}C NMR Spectra of (E)-5-styryl-1H-tetrazole (**4c**)

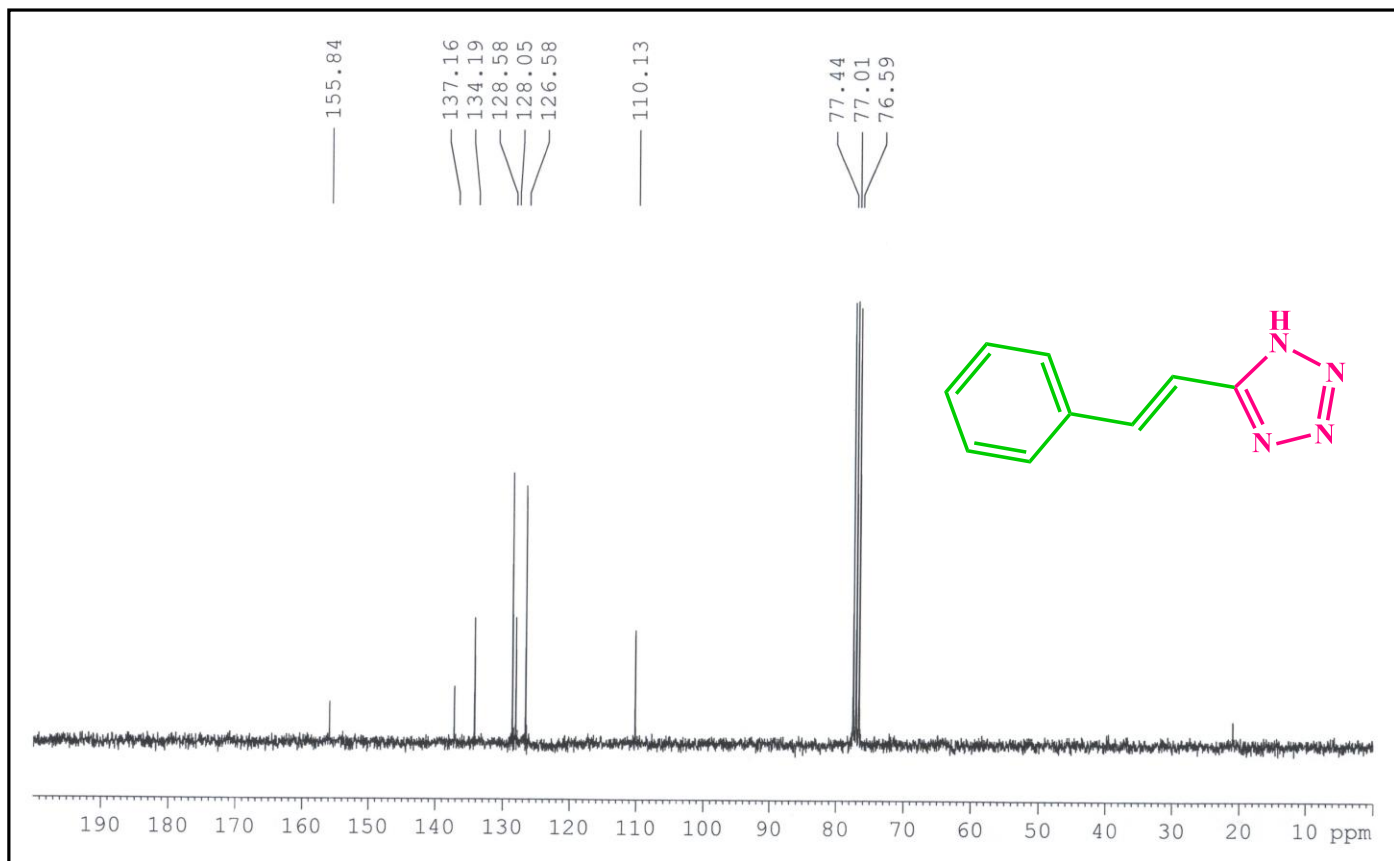
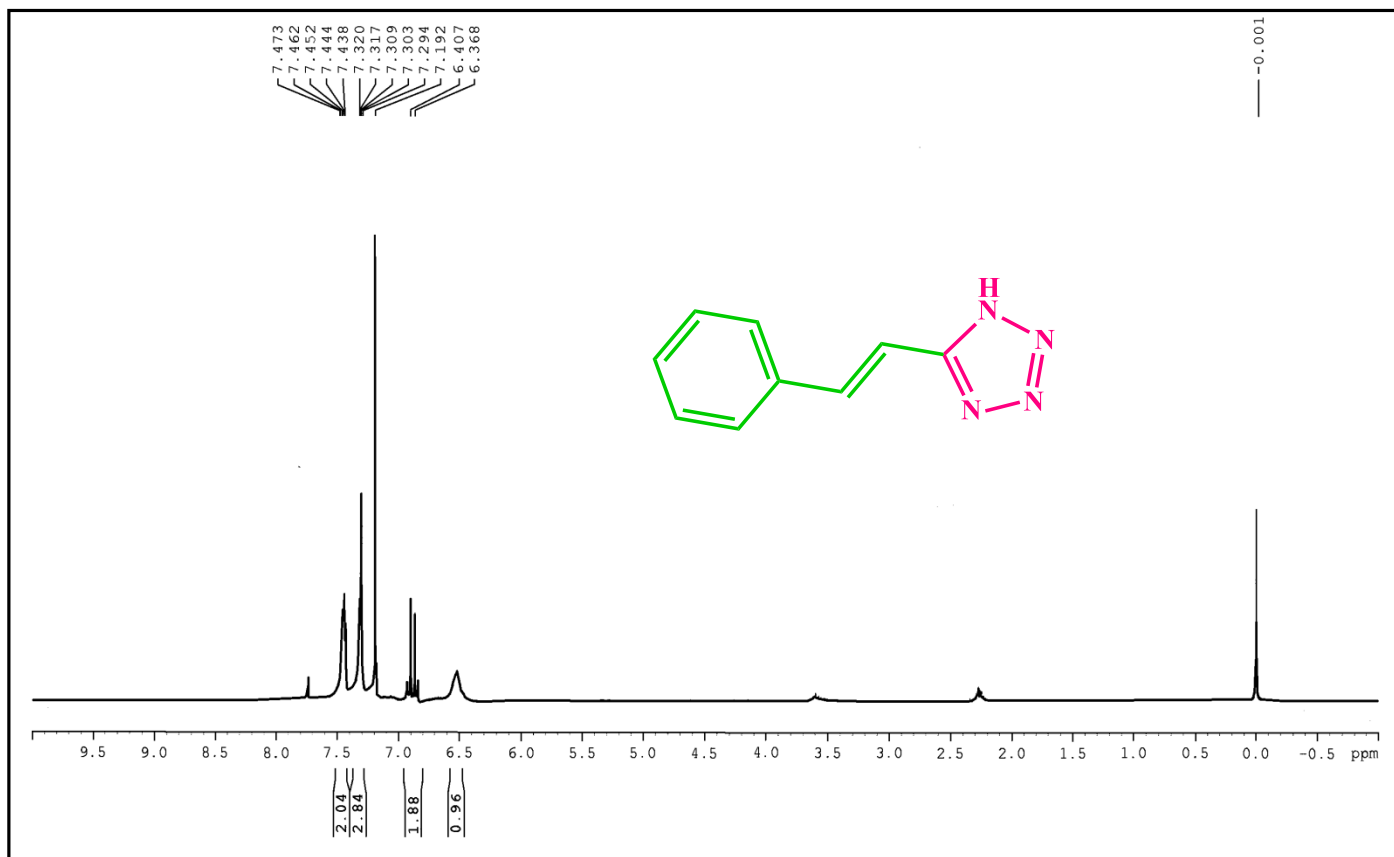


Figure S20. ^1H NMR Spectra of 9-(4-iodophenyl)-9*H*-carbazole (**Compound 5**)

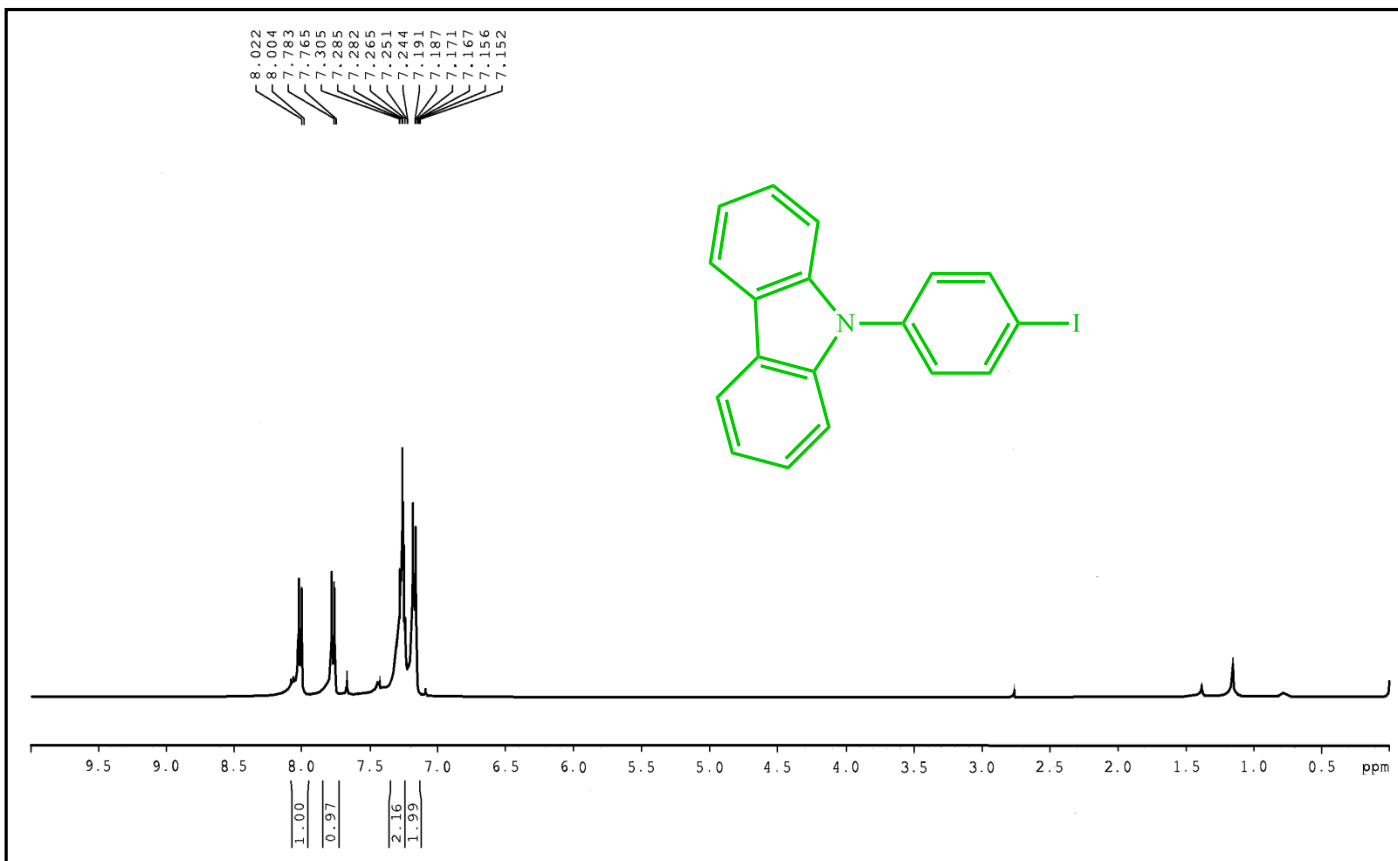
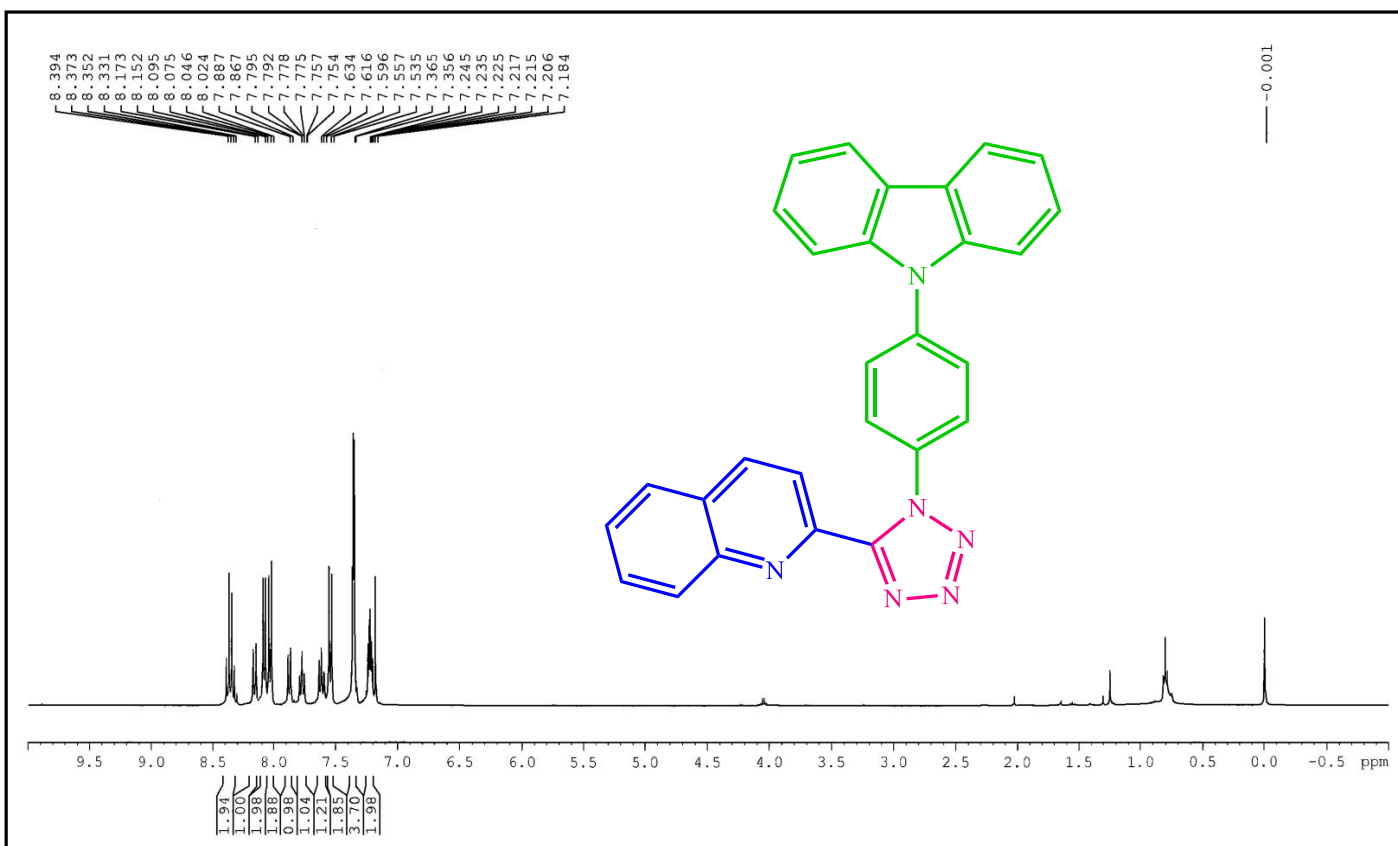


Figure S21. ^1H and ^{13}C NMR Spectra of 9-(4-iodophenyl)-9*H*-carbazole (**Compound 6**)



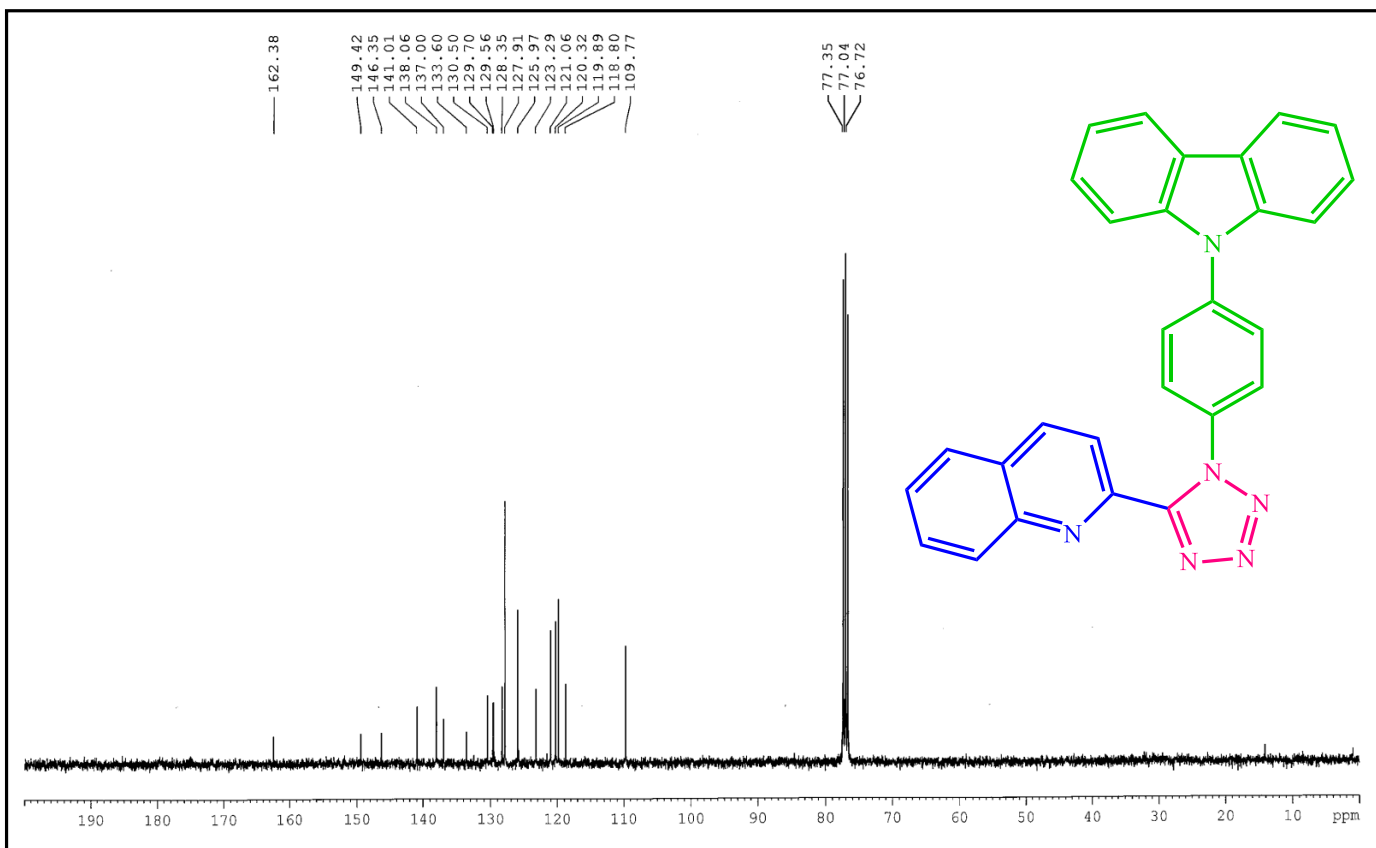


Figure S22. ESI-MS of 9-(4-iodophenyl)-9H-carbazole (**Compound 6**)

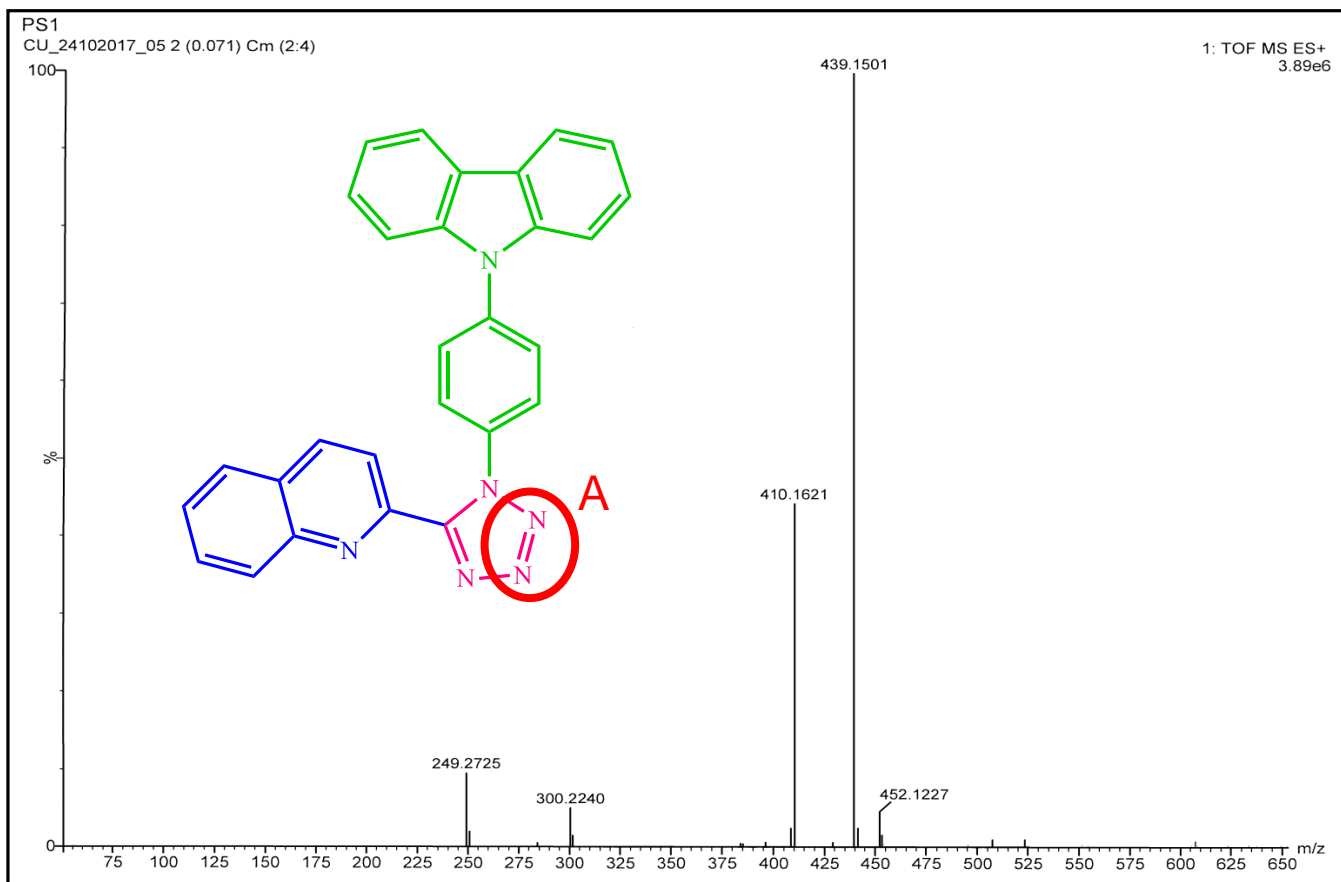
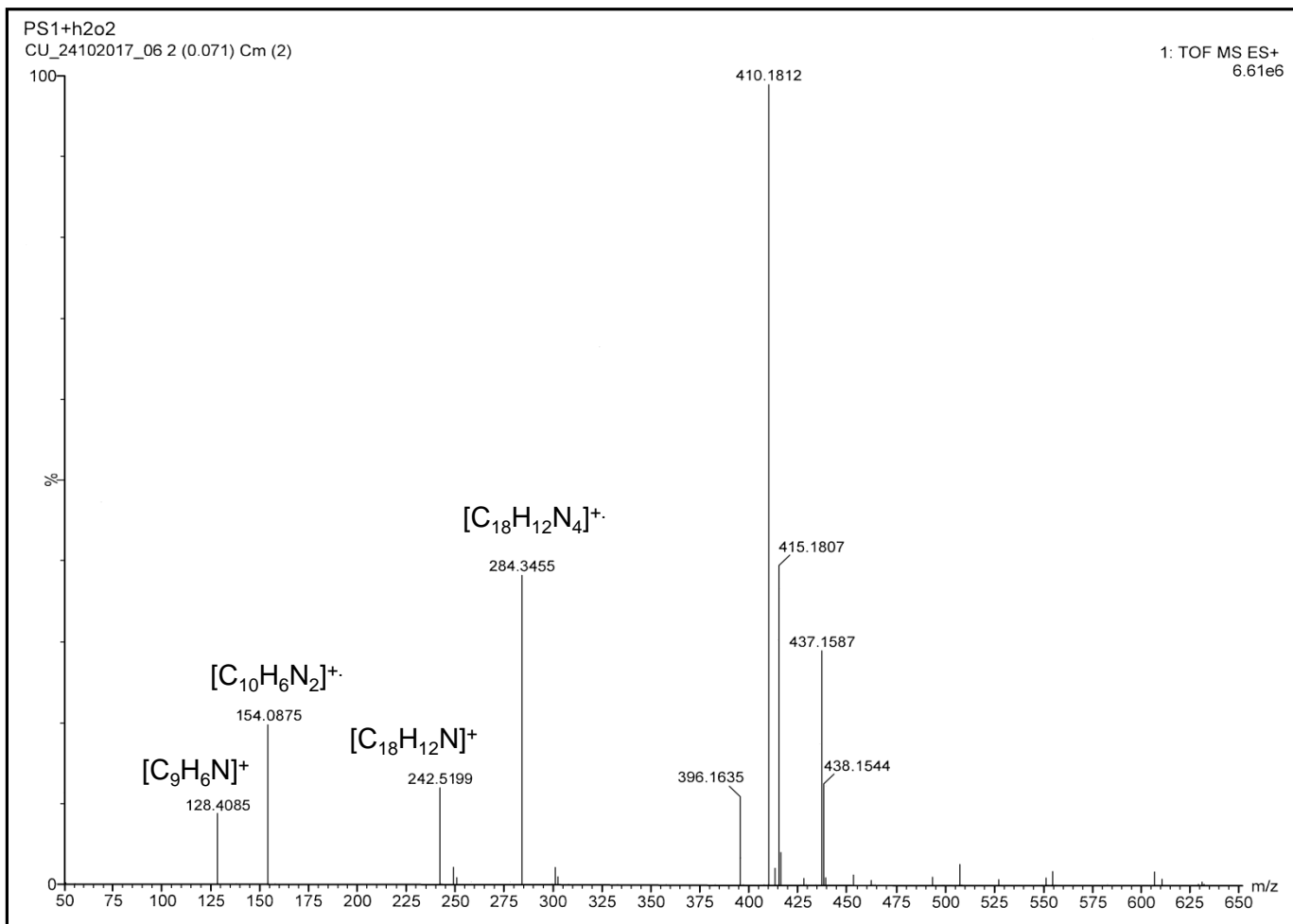


Figure S23. ESI-MS of H₂O₂ treated 9-(4-iodophenyl)-9*H*-carbazole (**Compound 6**)**Table S1.** A comparison for the detection of H₂O₂ sensing

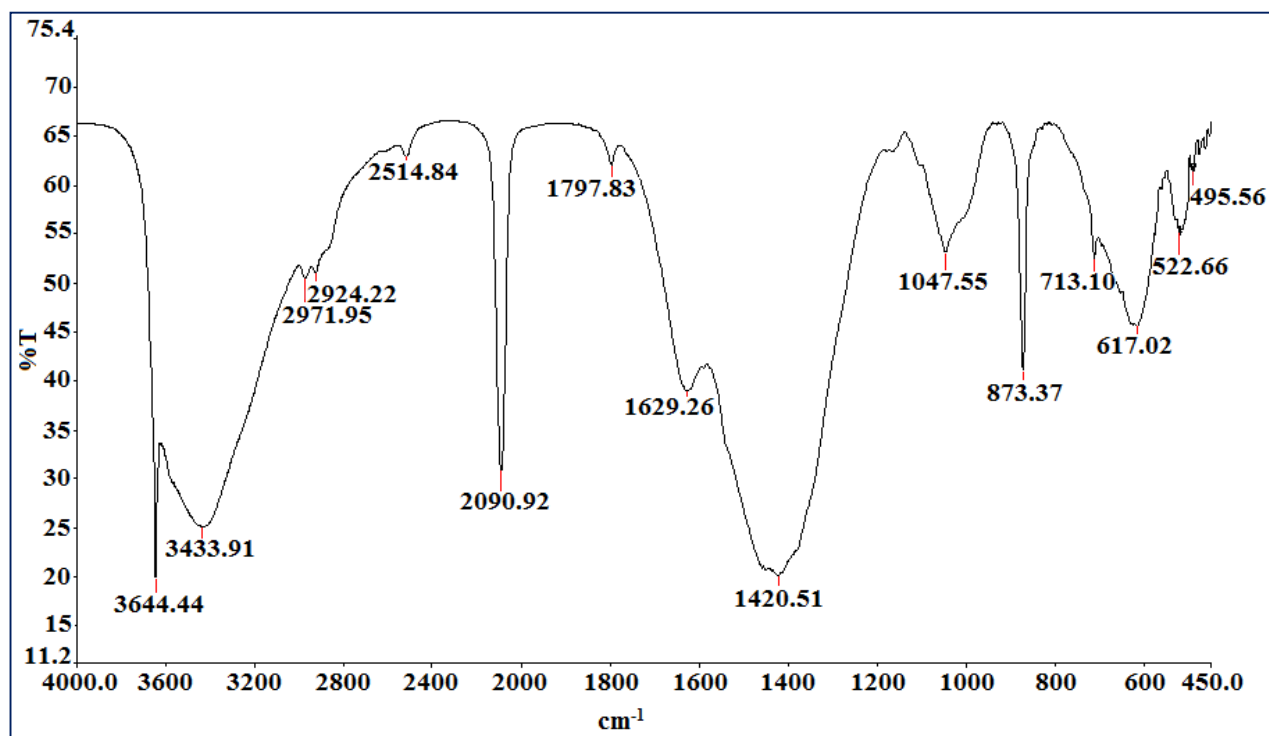
Method	LR	LOD (μ M)	Ref.
Amperometry (Enzymatic Method)	0.2-3.4 mM	40	10
Amperometry (Non Enzymatic Method)	2.0-80 mM	19.6	11
Chemiluminisence (Enzymatic Method)	0.1-3.0 mM	670	12
Fl-Spectrophotometry (Non Enzymatic Method)	100-4500 μ M	80	13
CDs nanoprobe-micro fluorospectrometry (Non Enzymatic Method)	20-20000 μ M	5	14
SI-LOV Spectrophotometry (Enzymatic Method)	1.3-10 mM	500	15
Spectrofluorimetry (Non Enzymatic Method)	2.4-16550 μM	2.22 μM	This work

LR = Linear range, LOD = Limit of detection ($3 \times \sigma/s$)

Table S2. A comparison of the catalytic activity of Ni(OH)₂ NPs for the synthesis of 5-substituted 1*H*-tetrazoles with the previous reports.

Catalyst	Reaction Conditions	T (°C)	t (h)	Yield (%)	Ref.
Cu(OAc) ₂ (25 mol%)	Benzaldoxime, NaN ₃ , DMF	120	12	98	16
InCl ₃ (3 mol%)	Benzaldoxime, NaN ₃ , DMF	120	15	92	17
-	Benzaldoxime, DPPA, DBU, Toluene	110	16	93	3
β-Ni(OH) ₂ NPs (4.32 mol%)	Benzaldoxime NaN ₃ , K ₂ CO ₃ , Water	reflux	10	98	This work

Figure S24. FTIR spectrum of reused catalyst.



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