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Synthesis and Characterization of 5-Substituted 1*H*-tetrazoles in the Presence of Nano-$TiCl_4.SiO_2$

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Here, we describe the IR, ^{13}C and ^{1}H NMR spectra of the novel 5-substituted 1*H*-tetrazolederivatives (Table 2, compounds 9, 11 and 12).

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5-Phenyl-1*H*-tetrazole

Yield: 95%, White crystal.

Figure S1. FT-IR: ῡ (KBr) = 2600-300, 1608, 1563, 1485, 1465, 1409, 1480, 726, 687 cm^{-1}.

Figure S2. ^{1}H NMR (400 MHz, DMSO-d6): 8.01 (brs, 2H), 7.51 (brs, 3H) ppm.

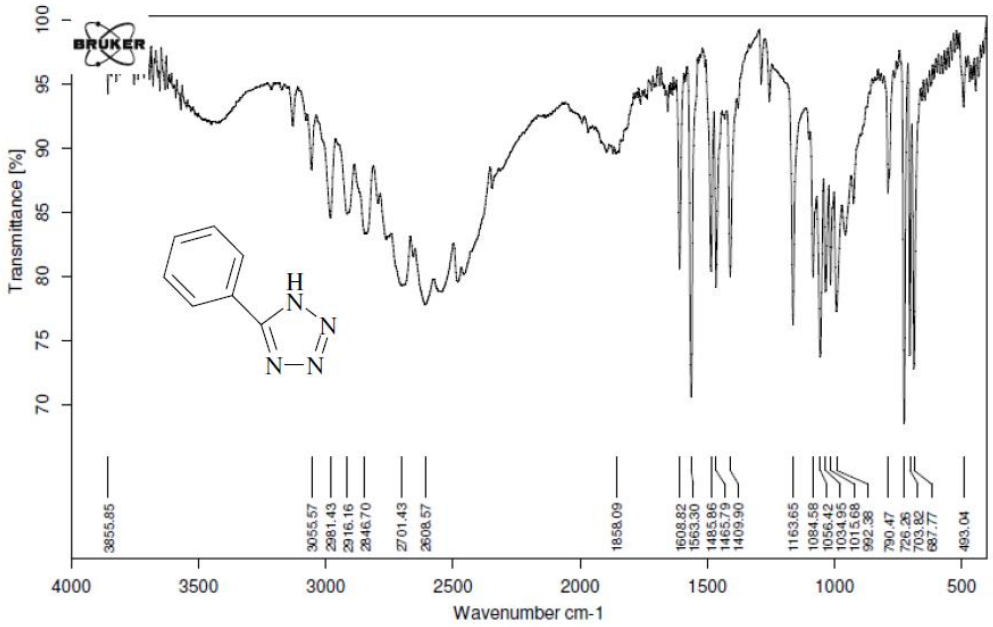

Figure S1. FT-IR (KBr) 5-Phenyl-1*H*-tetrazole

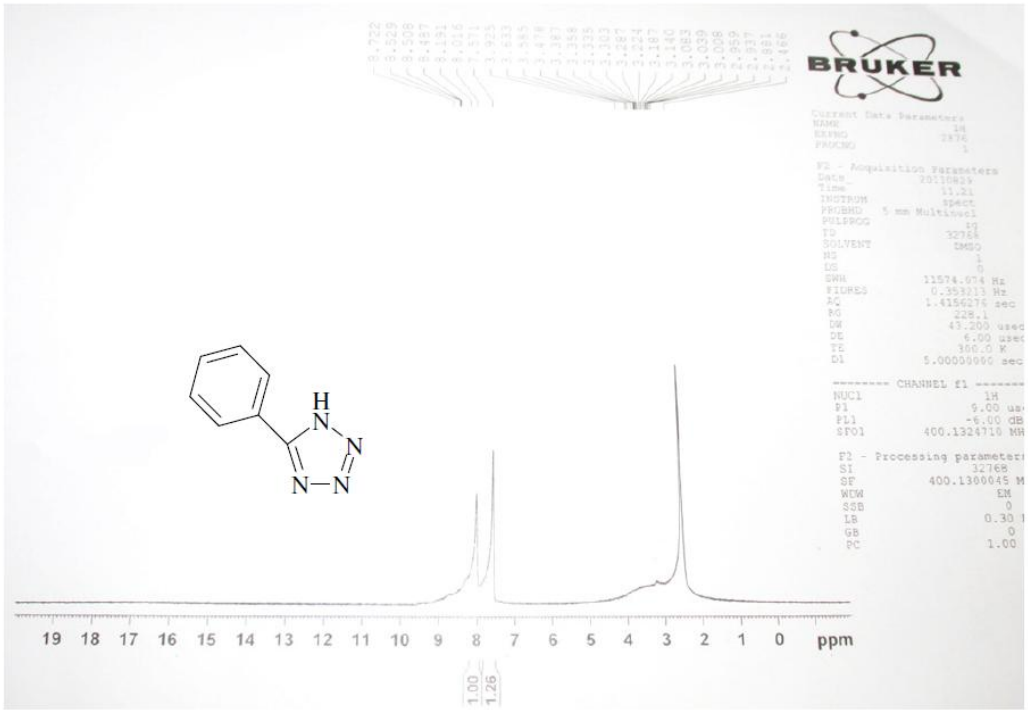

Figure S2. ^{1}H NMR (500 MHz, DMSO-d_6) 5-Phenyl-1*H*-tetrazole

5-(4-Methylphenyl)-1*H*-tetrazole

Yield: 84%, White crystal.

Figure S3. FT-IR: ῡ (KBr) = 2600-300, 1608, 1563, 1485, 1465, 1409, 1480, 726, 687 cm^{-1}.

Figure S4, S5. ^{1}H NMR (500 MHz, $CDCl_3$): 8.81 (d, *J*= 5.8, 2H), 8.09 (d, *J*= 5.8, 2H), 2.5 (s, 3H) ppm.

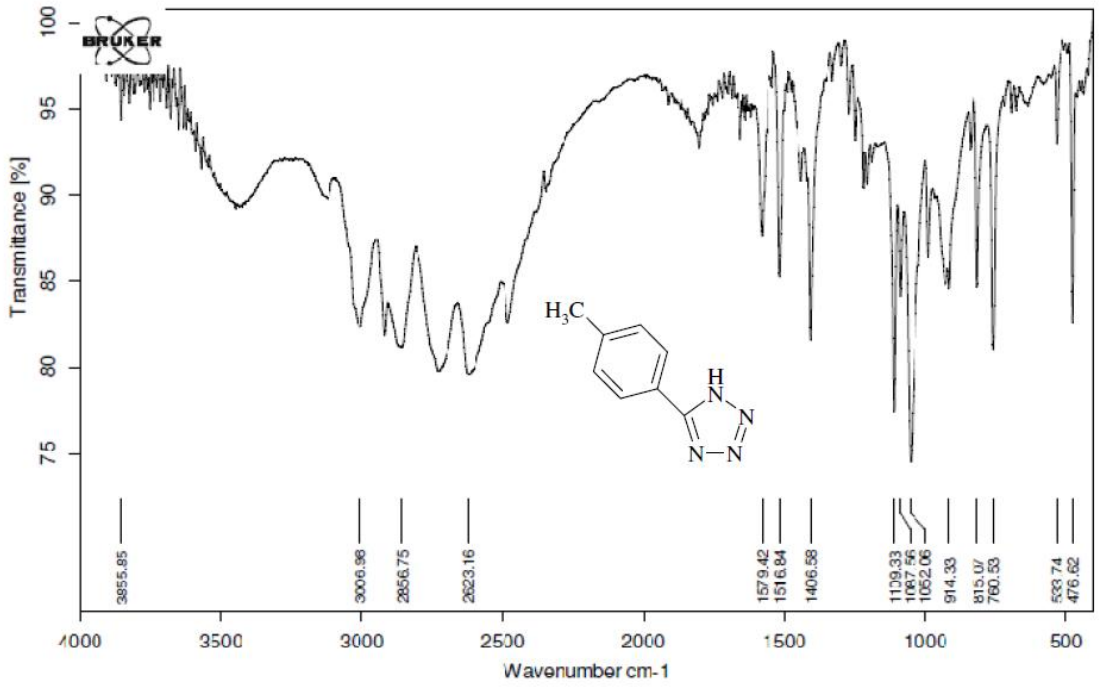

Figure S3. FT-IR (KBr) 5-(4-Methylphenyl)-1*H*-tetrazole

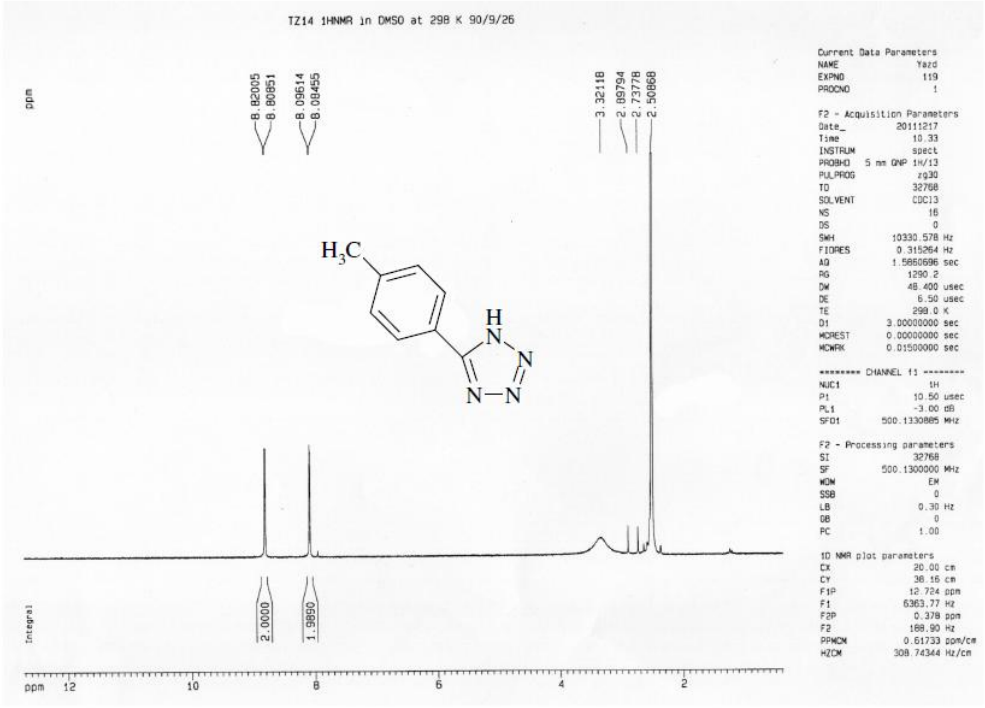

Figure S4: ^{1}H NMR (500 MHz, $CDCl_3$) 5-(4-Methylphenyl)-1*H*-tetrazole

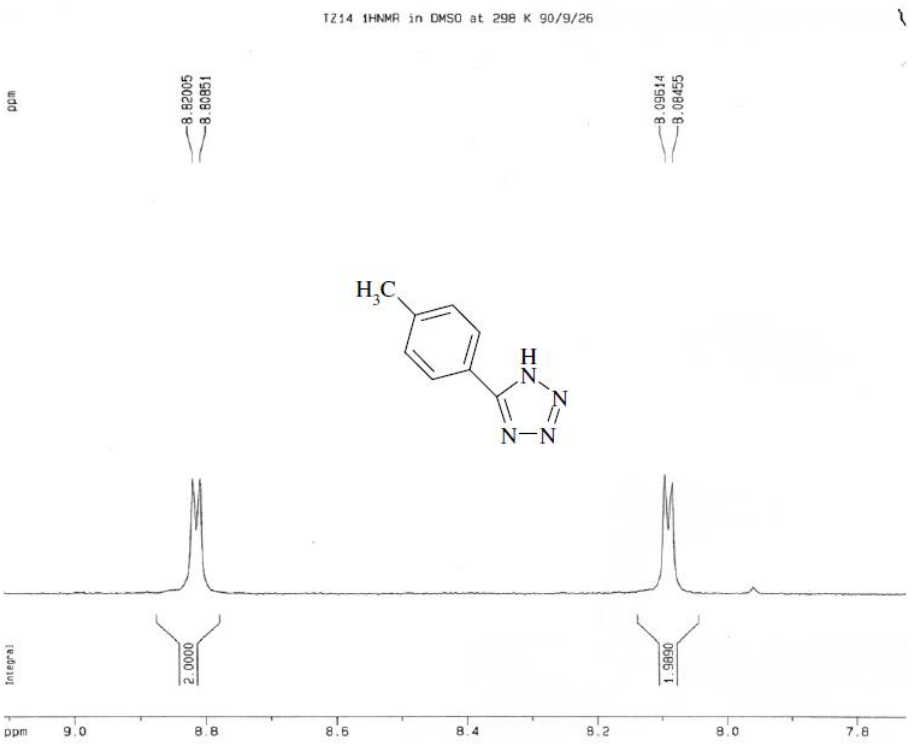

Figure S5: ^{1}H NMR (500 MHz, $CDCl_3$) 5-(4-Methylphenyl)-1*H*-tetrazole (expand)

5-(4-Hydroxyphenyl)-1*H*-tetrazole

Yield: 82%, White crystal.

Figure S6. FT-IR: ῡ (KBr) = 2500-3400, 1648, 1600, 1515, 1470, 1435, 1080, 842 cm^{-1}.

Figure S7, S8. ^{1}H NMR (400 MHz, DMSO-d6): 16.5 (brs, NH), 10.17 (s, 1H), 7.84 (d, *J*= 8, 2H), 6.93 (d, *J*= 7.6, 2H) ppm.

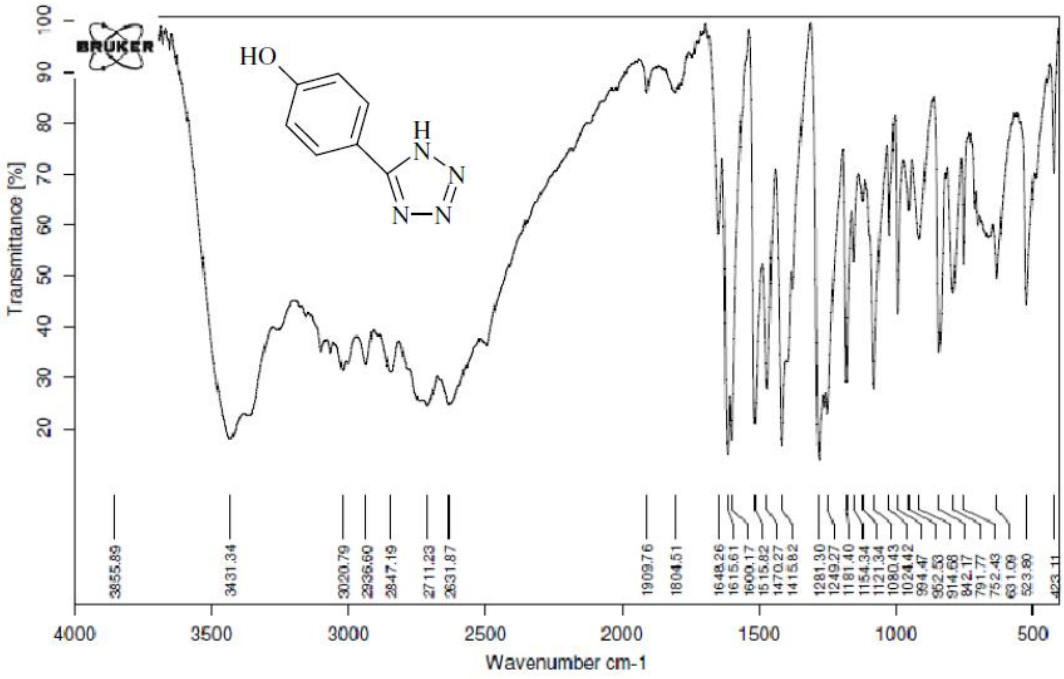

Figure S6: FT-IR (KBr) 5-(4-Hedroxyphenyl)-1*H*-tetrazole

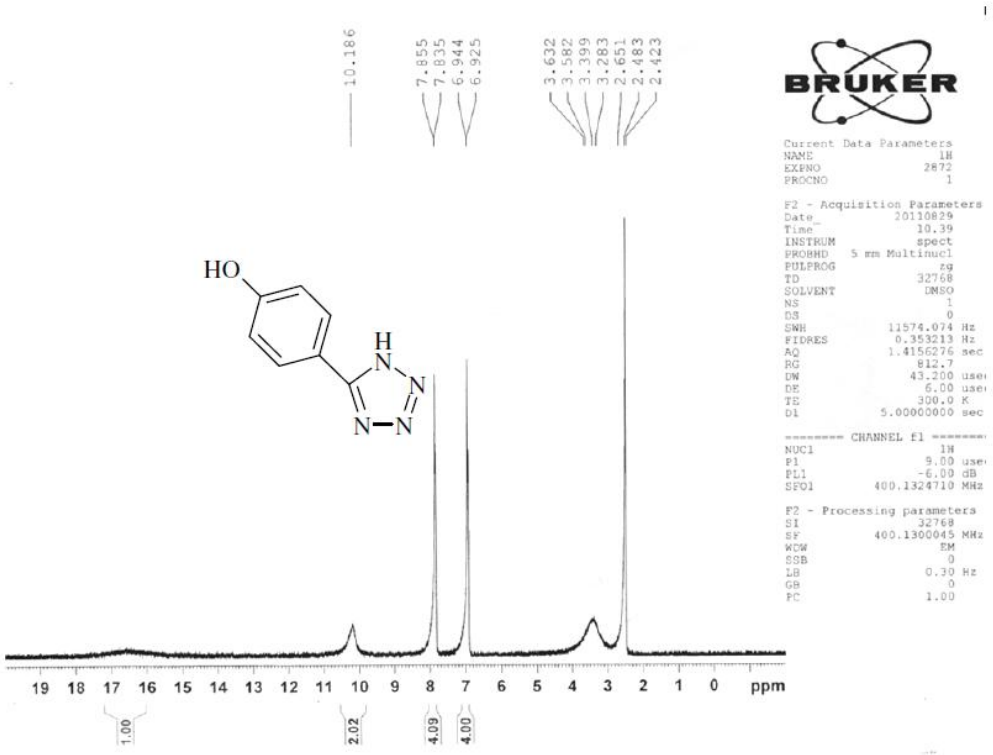

Figure S7. ^{1}H NMR (500 MHz, DMSO-d_6) 5-(4-Hedroxyphenyl)-1*H*-tetrazole

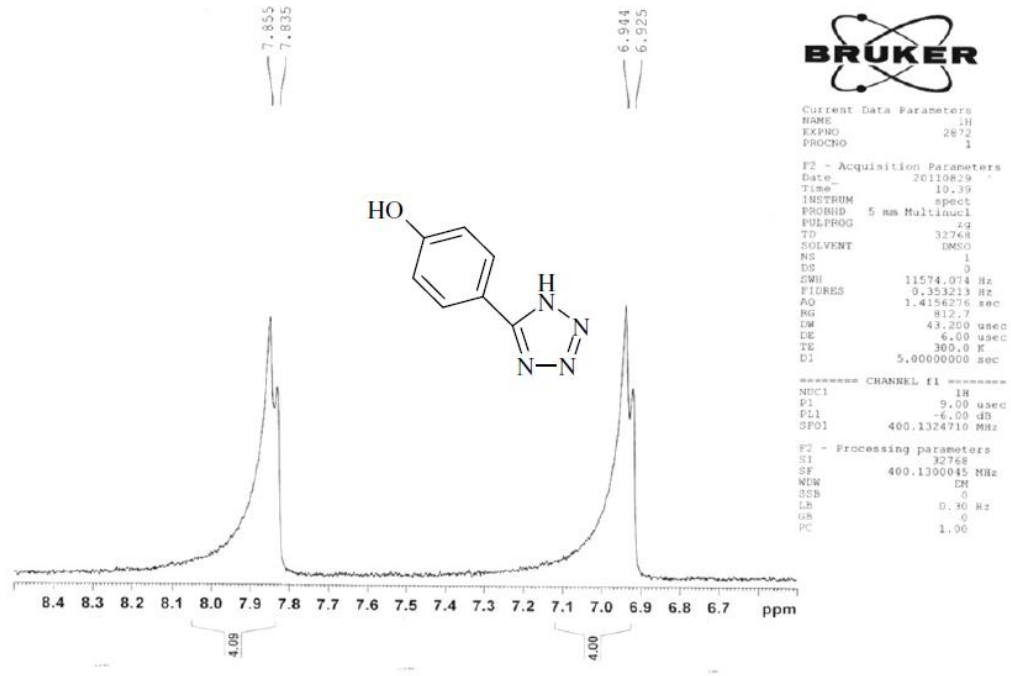

Figure S8. ^{1}H NMR (500 MHz, DMSO-d_6) 5-(4-Hedroxyphenyl)-1*H*-tetrazole (expand)

5-(3-Nitrophenyl)-1*H*-tetrazole

Yield: 92%, White crystal.

Figure S9. FT-IR: ῡ (KBr) = 2600-3300, 1626, 1569, 1528, 1349, 872, 743, 973 cm^{-1}.

Figure S10, S11. ^{1}H NMR (400 MHz, DMSO-d6): 8.82 (s, 1H), 8.43 (dd, *J*= 7.6 and 8 Hz, 2H), 7.89 (t, *J*= 8.4, 1H) ppm.

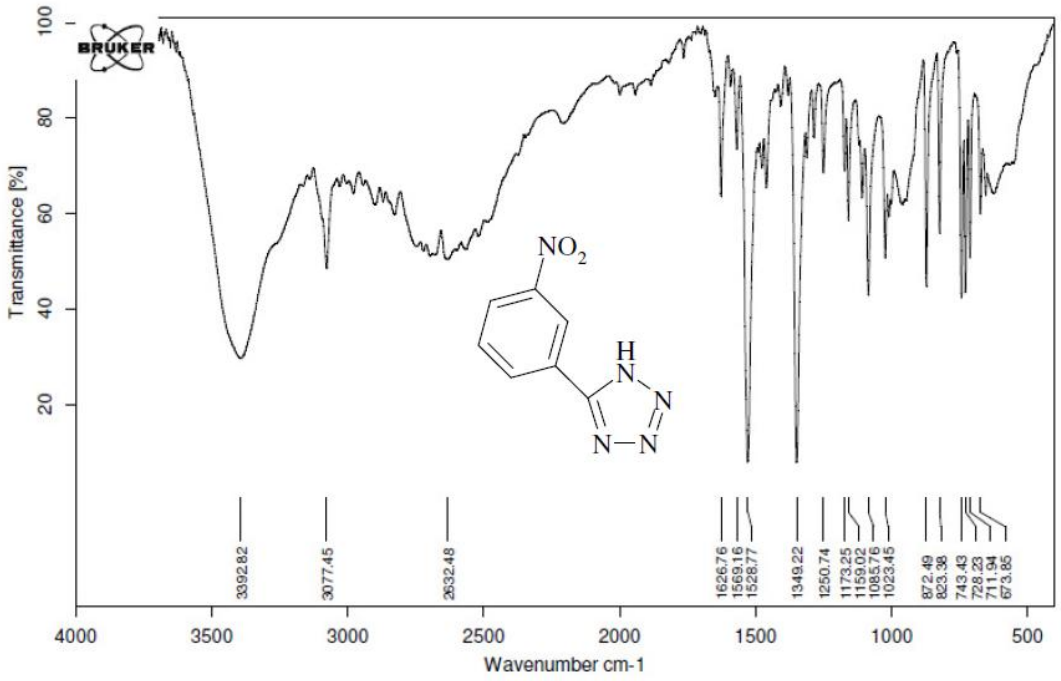

Figure S9: FT-IR (KBr) 5-(3-Nitrophenyl)-1*H*-tetrazole

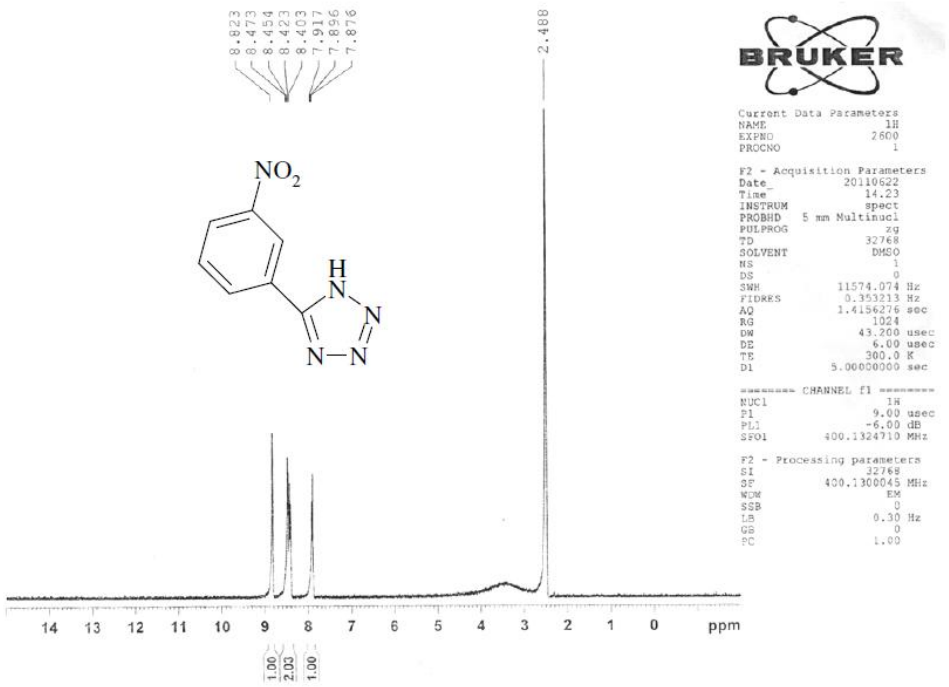

Figure S10. ^{1}H NMR (500 MHz, DMSO-d_6) 5-(3-Nitrophenyl)-1*H*-tetrazole

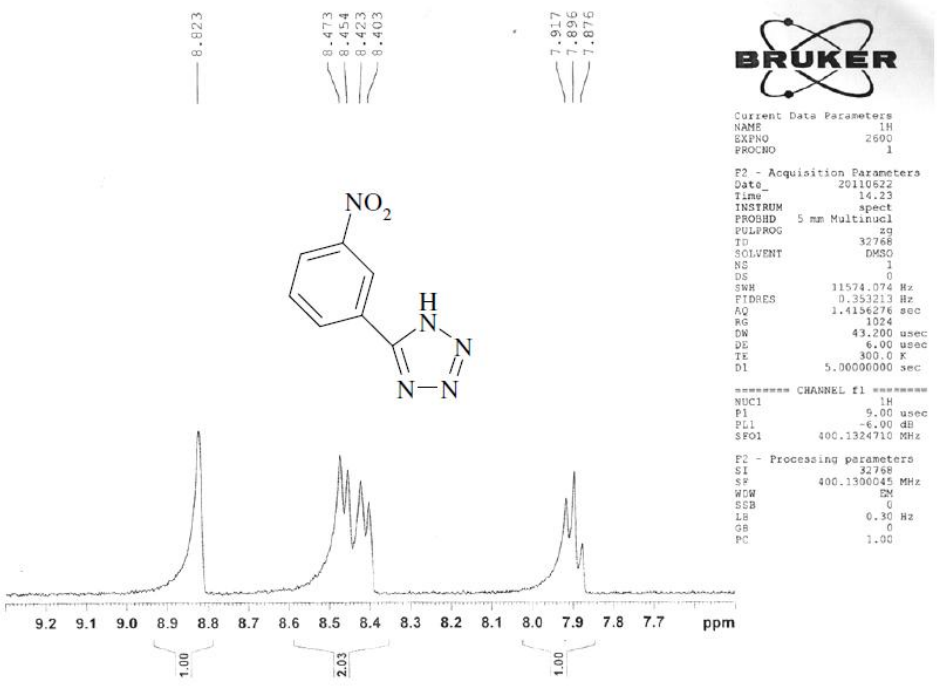

Figure S11. ^{1}H NMR (500 MHz, DMSO-d_6) 5-(3-Nitrophenyl)-1*H*-tetrazole (expand)

5-(4-Chlorophenyl)-1*H*-tetrazole

Yield: 91%, White crystal.

Figure S12. FT-IR: ῡ (KBr) = 2500-3000, 1654, 1610, 1561, 1487, 1434, 831 cm^{-1}.

Figure S13, S14. ^{1}H NMR (500 MHz, DMSO-d6): 8.10 (d, *J*= 10.53, 2H), 7.69 (d, *J*= 8.41, 2H) ppm.

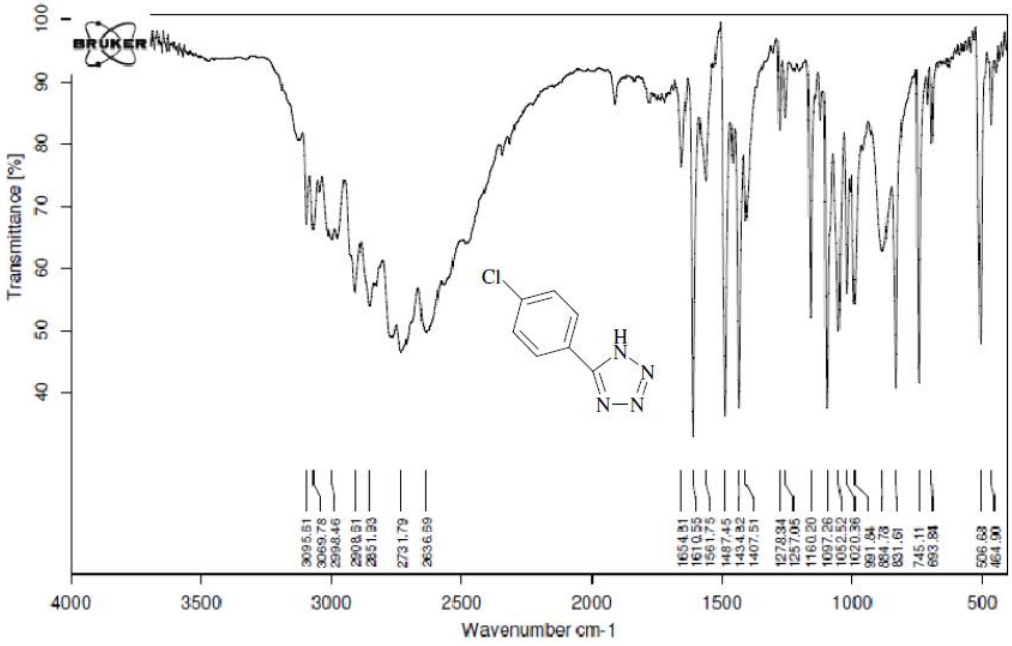

Figure S12: FT-IR (KBr) 5-(4-Chlorophenyl)-1*H*-tetrazole

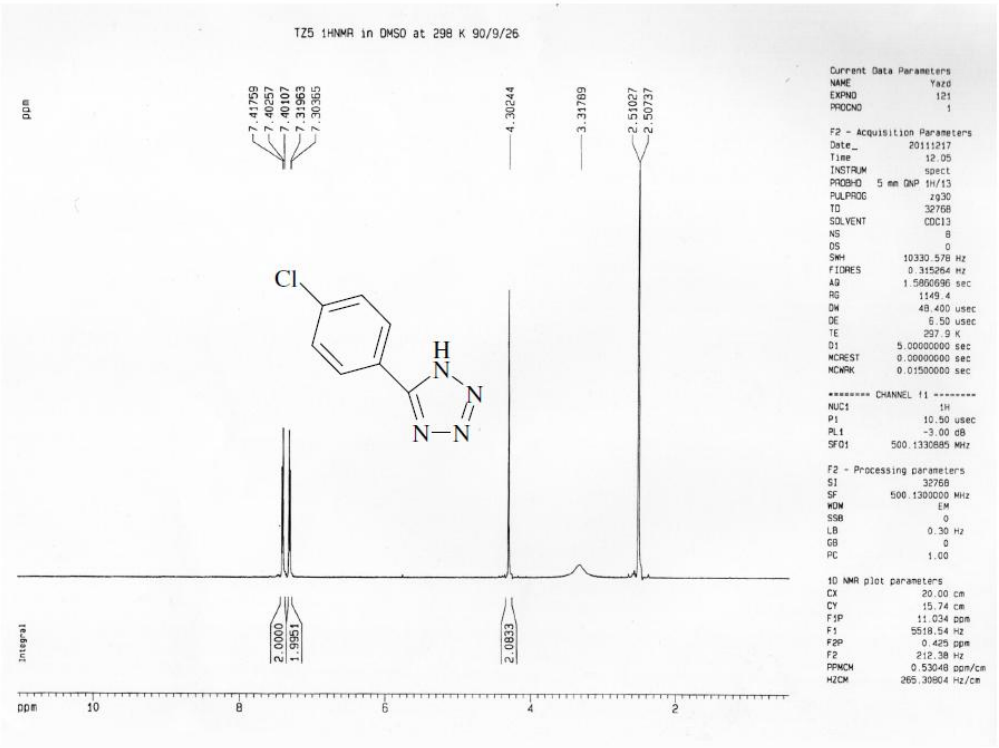

Figure S13. ^{1}H NMR (500 MHz, DMSO-d_6) 5-(4-Chlorophenyl)-1*H*-tetrazole

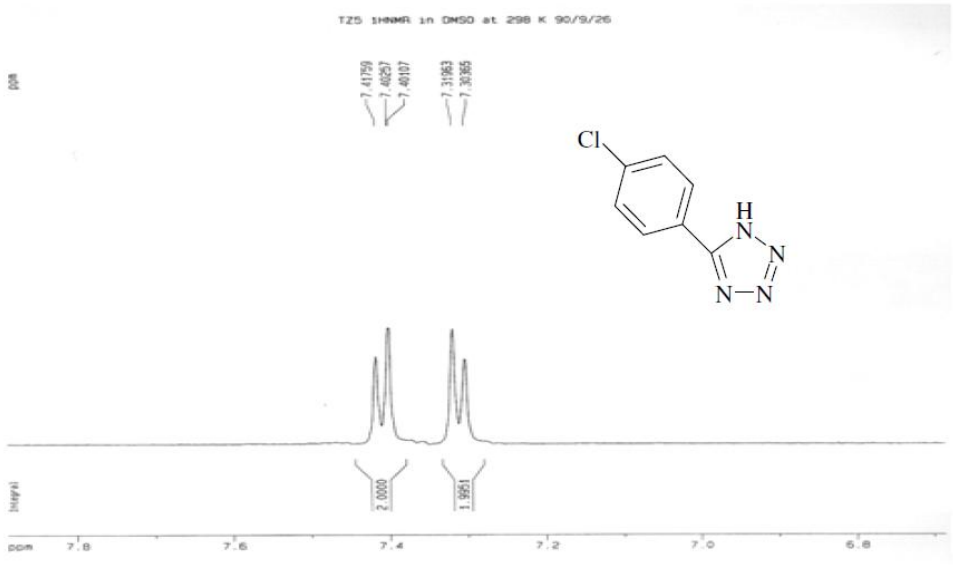

Figure S14. ^{1}H NMR (500 MHz, DMSO-d_6) 5-(4-Chlorophenyl)-1*H*-tetrazole (expand)

5-(4-Bromophenyl)-1*H*-tetrazole

Yield: 90%, White crystal

Figure S15. FT-IR: ῡ (KBr) = 2600-300, 1649, 1604, 1560, 1482, 1431, 1053, 829 cm^{-1}.

Figure S16, S17. ^{1}H NMR (500 MHz, $CDCl_3$): 8.06 (d, *J*= 7.2, 2H), 7.69 (d, *J*= 7.2, 2H) ppm.

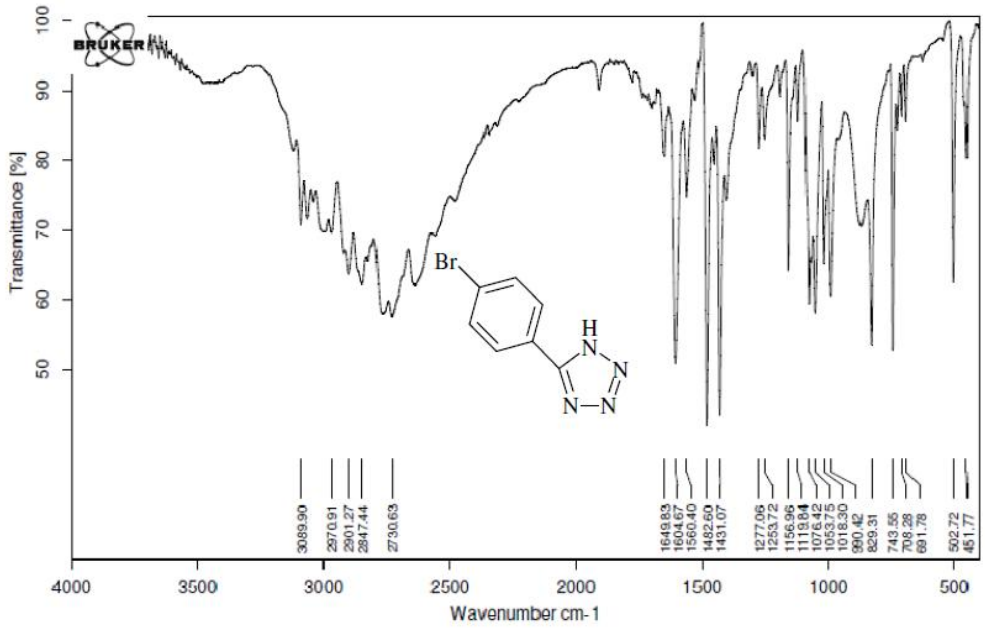

Figure S15: FT-IR (KBr) 5-(4-Bromophenyl)-1*H*-tetrazole

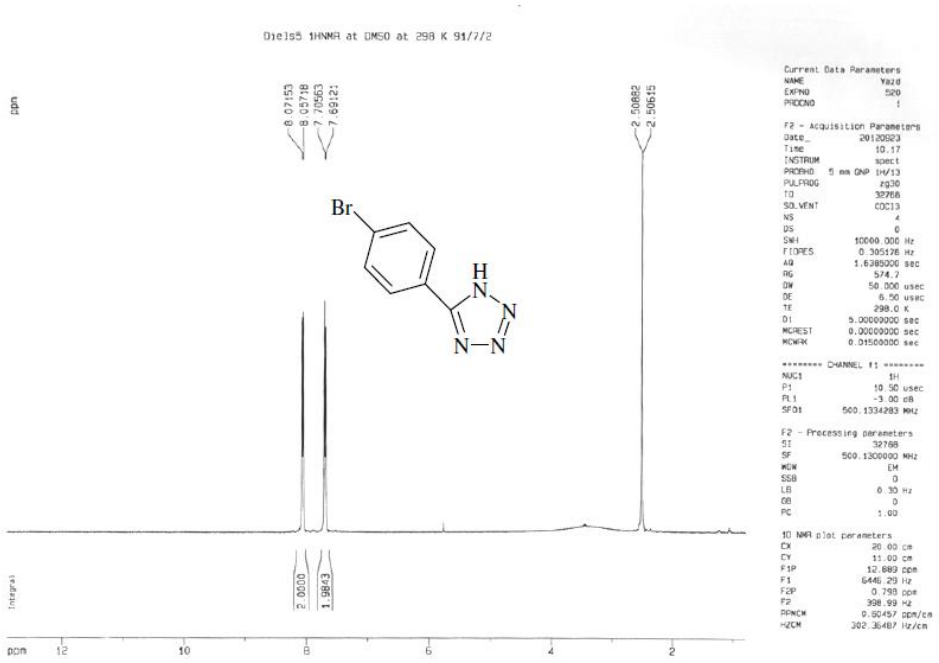

Figure S16. ^{1}H NMR ((500 MHz, $CDCl_3$): 5-(4- Bromophenyl)-1*H*-tetrazole

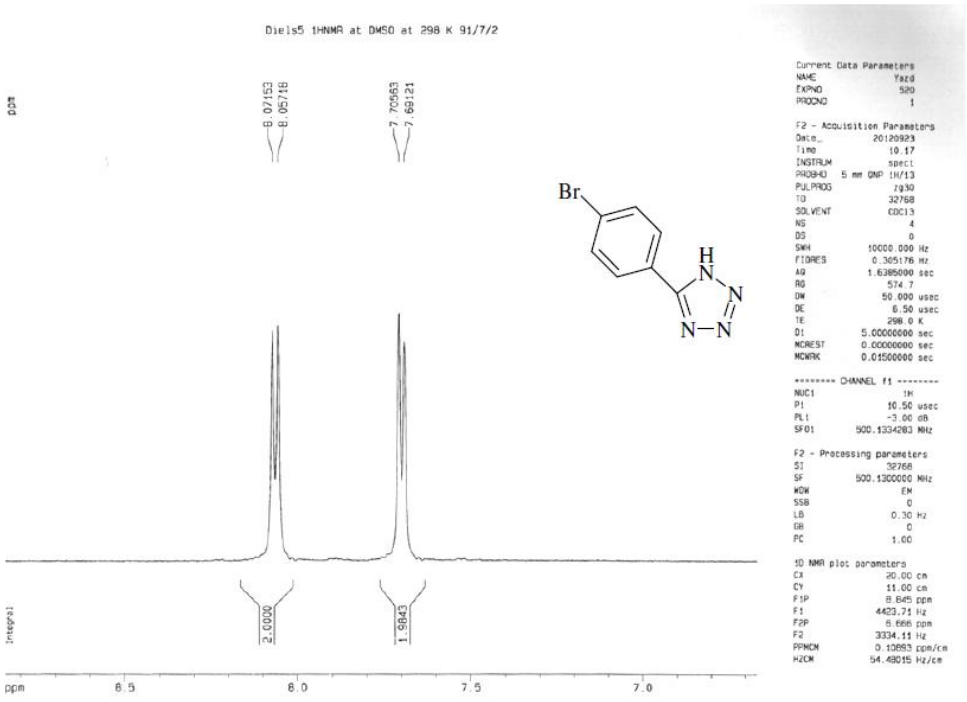

Figure S17. ^{1}H NMR ((500 MHz, $CDCl_3$): 5-(4- Bromophenyl)-1*H*-tetrazole (expand)

5-Benzyltetrazole

Yield: 78%, White crystal.

Figure S18. FT-IR: ῡ (KBr) = 2400-300, 1592, 1549, 1496, 1248, 775 cm^{-1}.

Figure S19, S20. ^{1}H NMR (400 MHz, DMSO-d_6): 4.26 (s, 2H, CH_2), 7.25-1.31 (m, 5H) ppm.

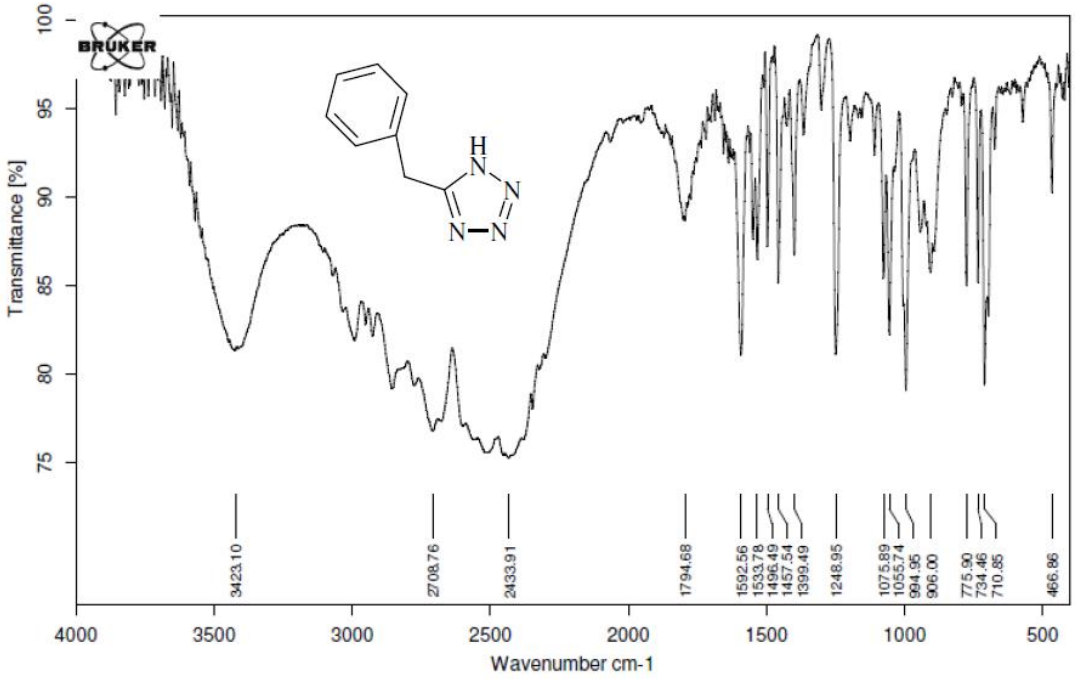

Figure S18: FT-IR (KBr) 5-Benzyltetrazole

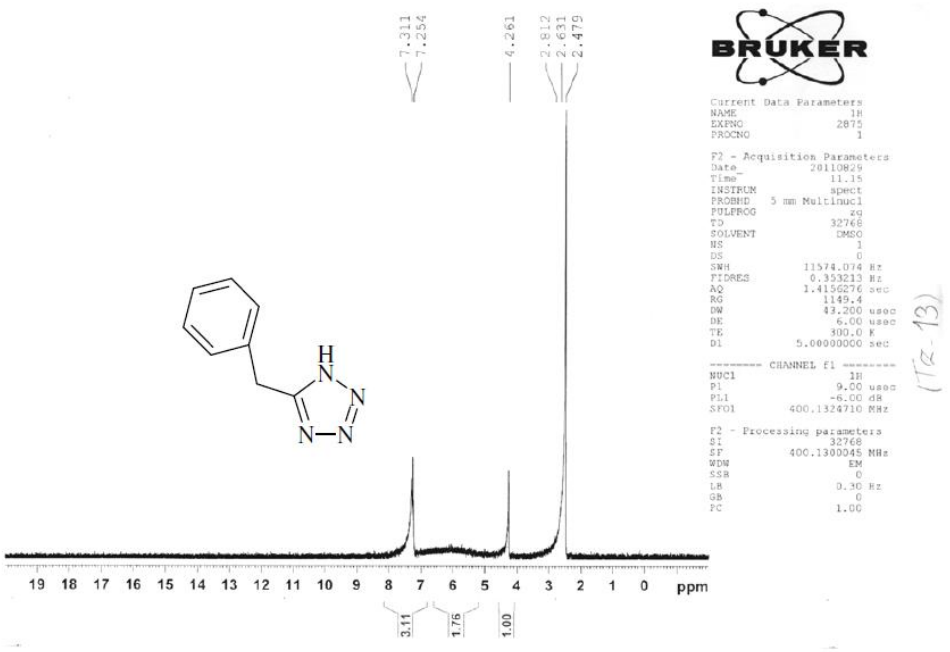

Figure S19. ^{1}H NMR (500 MHz, DMSO-d_6) 5-Benzyltetrazole

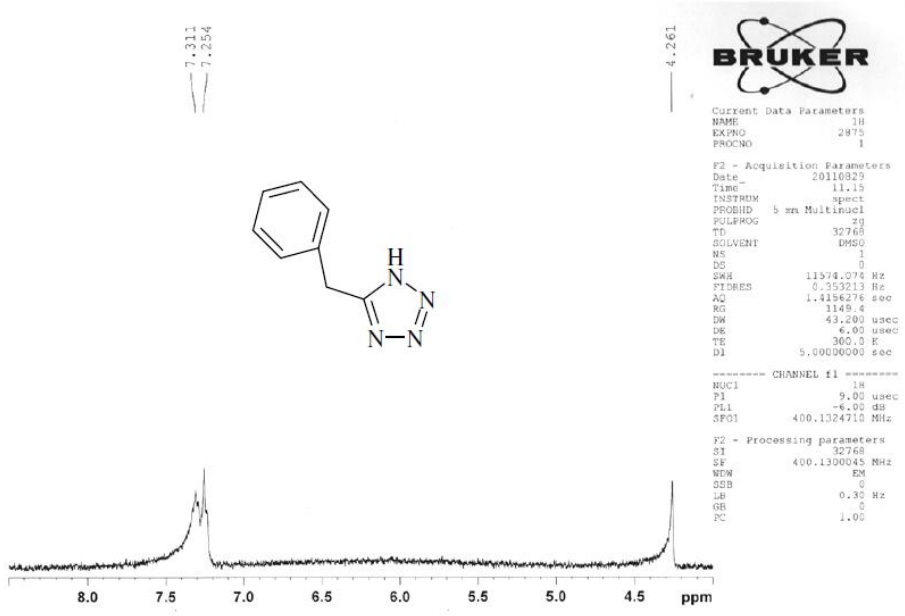

Figure S20. ^{1}H NMR (500 MHz, DMSO-d_6) 5-Benzyltetrazole (expand)

5-((4-Methoxyphenyl)methyl)tetrazole

Yield: 81%, White crystal.

Figure S21. FT-IR: ῡ (KBr) = 2600-3400, 1636, 1514, 1124, 848 cm^{-1}.

Figure S22, S23. ^{1}H NMR (400 MHz, DMSO-d6): 7.19 (brs, 2H), 6.86 (brs, 2H), 4.18 (s, 2H), 3.69 (s, 3H) ppm.

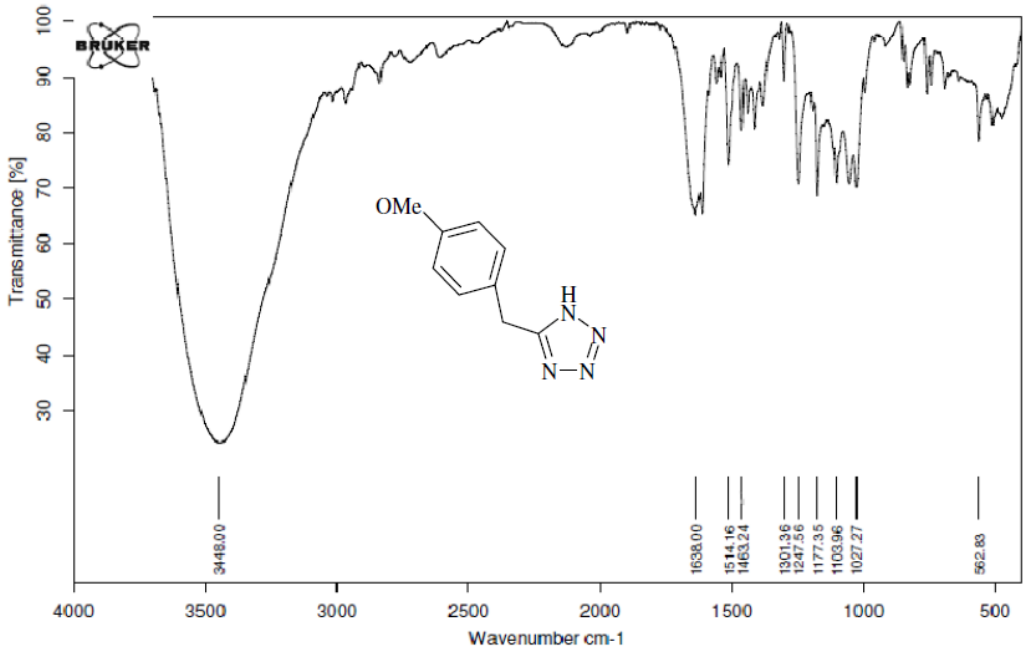

Figure S21: FT-IR (KBr) 5-((4-Methoxyphenyl)methyl)tetrazole

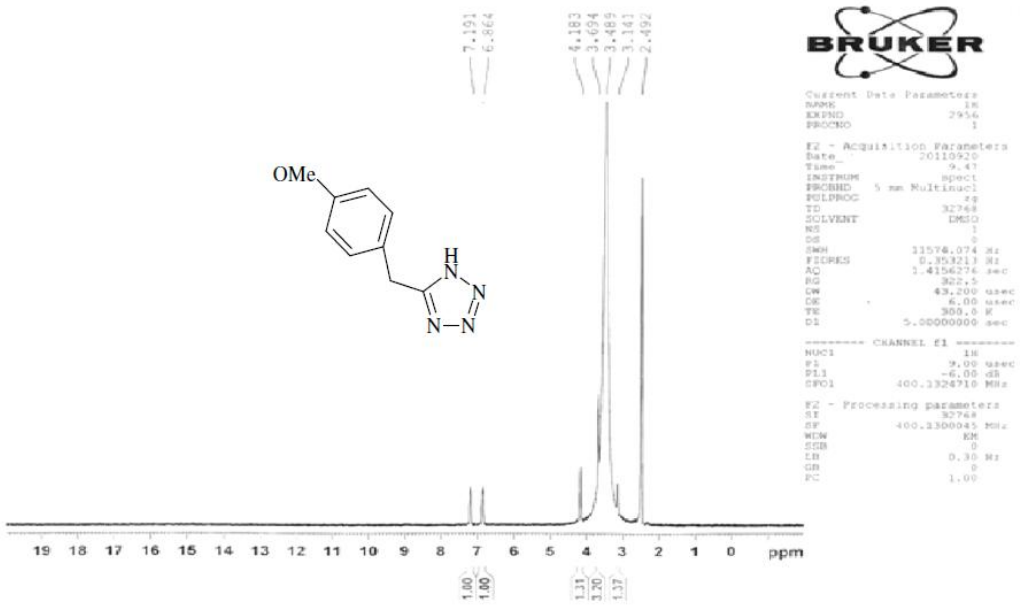

Figure S22. ^{1}H NMR (500 MHz, DMSO-d_6) 5-((4-Methoxyphenyl)methyl)tetrazole

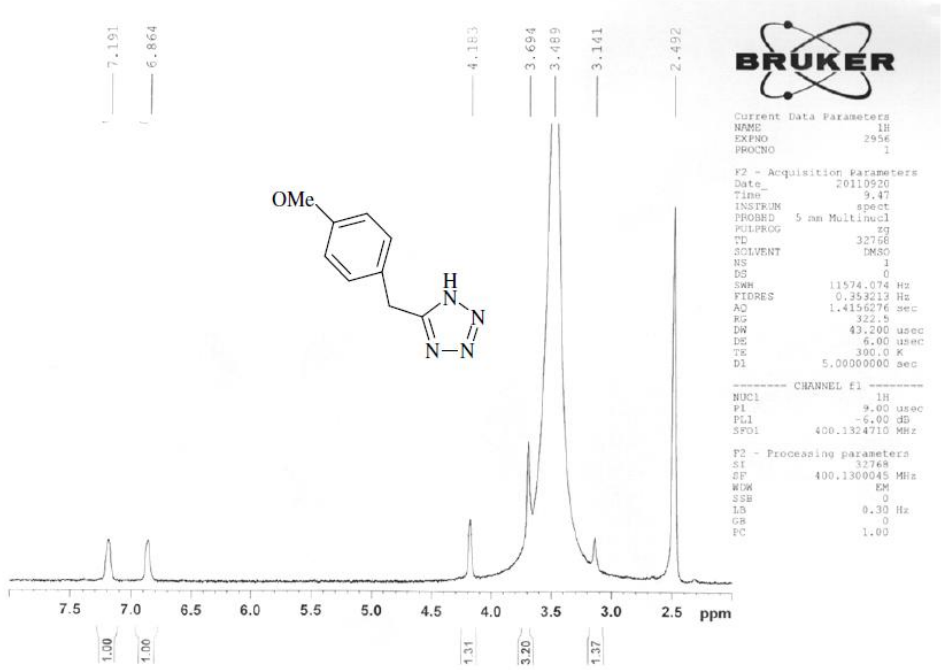

Figure S23. ^{1}H NMR (500 MHz, DMSO-d_6) 5-((4-Methoxyphenyl)methyl)tetrazole (expand)

5-((4-Chlorophenyl)methyl)tetrazole

Yield: 84%, White crystal.

Figure S24. FT-IR: ῡ (KBr) = 2600-300, 1538, 1492, 1407, 1263, 1207, 834 cm^{-1}.

Figure S25, S26. ^{1}H NMR (400 MHz, DMSO-d6): 7.40 (m, 2H), 7.31 (d, *J*= 8, 2H), 4.30 (s, 2H) ppm.

Figure S27. ^{13}C-NMR (125 MHz, DMSO) δ = 155.0, 132.5, 128.9, 124.7, 123.6, 28.8 ppm.

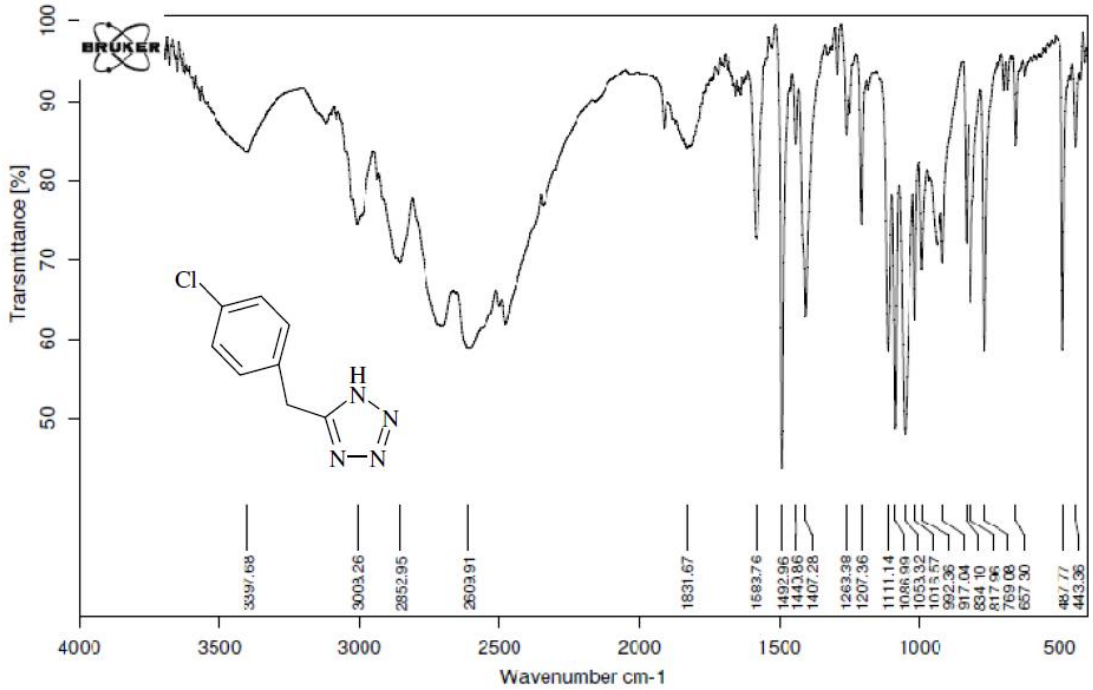

Figure S24: FT-IR (KBr) 5-((4-Chlorophenyl)methyl)tetrazole

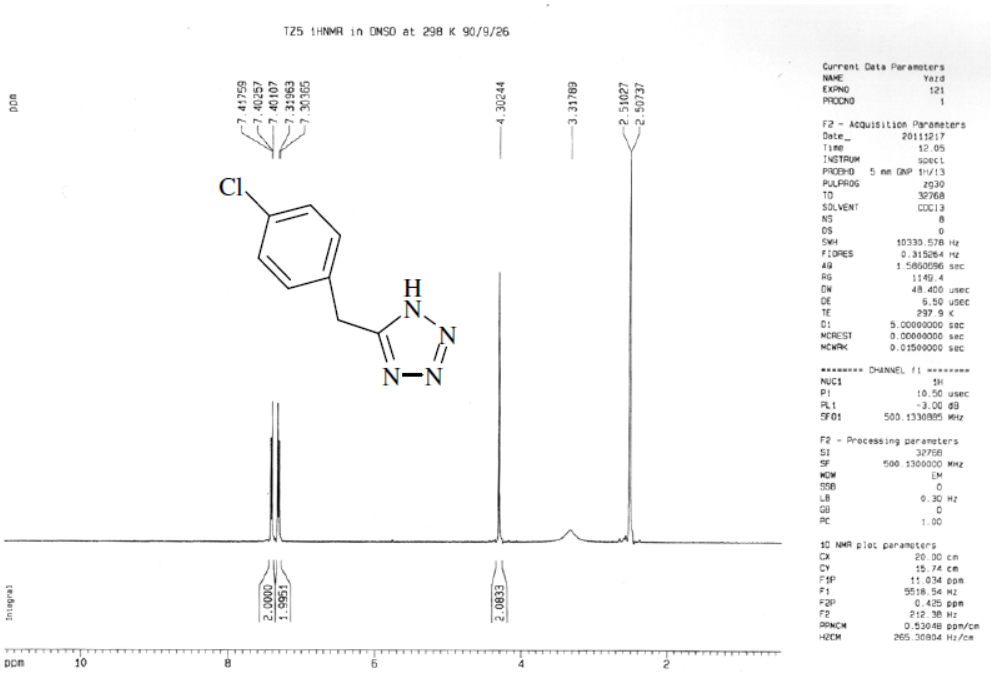

Figure S25. ^{1}H NMR (500 MHz, DMSO-d_6) 5-((4-Chlorophenyl)methyl)tetrazole

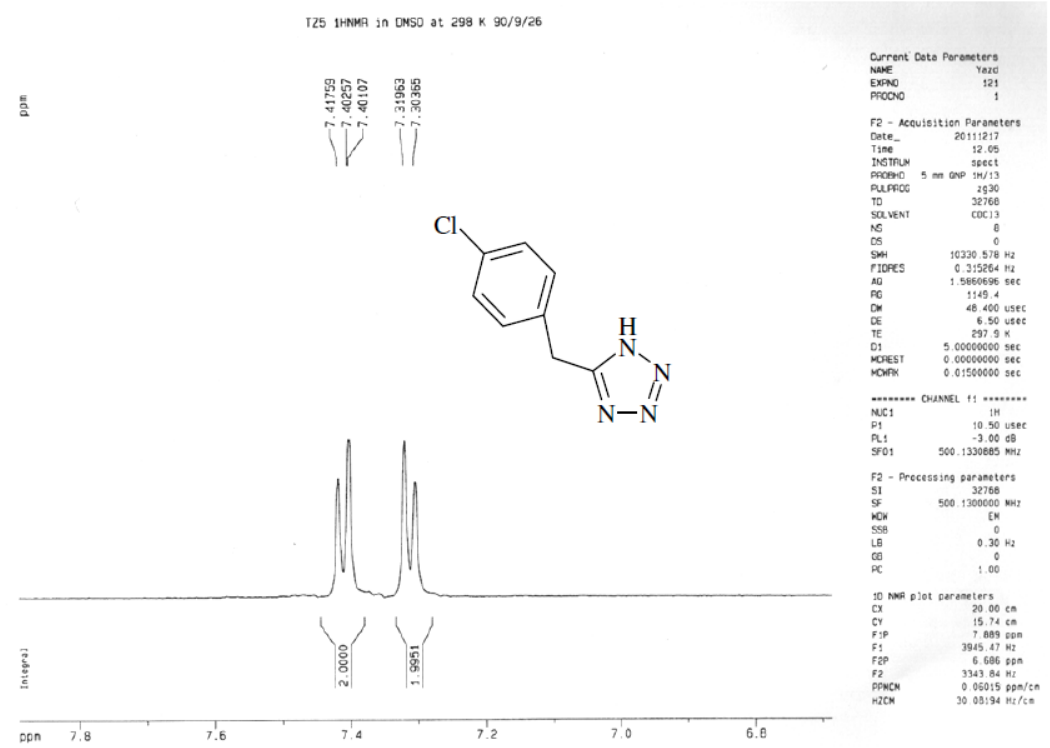

Figure S26. ^{1}H NMR (500 MHz, DMSO-d_6) 5-((4-Chlorophenyl)methyl)tetrazole (expand)

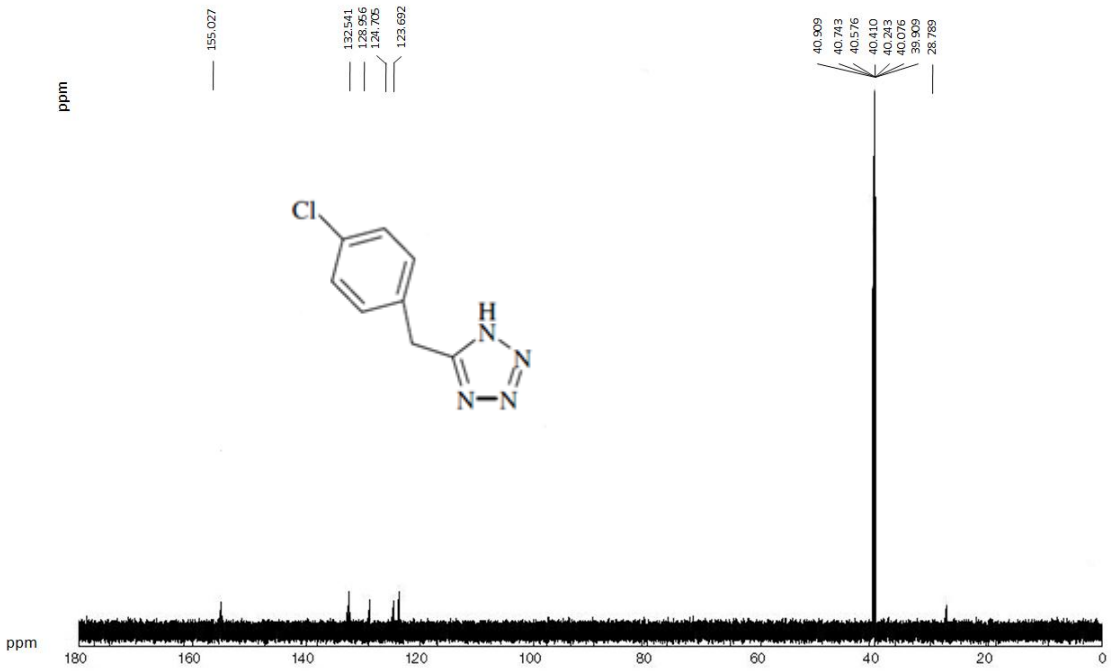

Figure S27. ^{13}C NMR (500 MHz, DMSO) of 5-((4-Chlorophenyl)methyl)tetrazole

5-Benzyhydryltetrazole

Yield: 88%, White crystal.

Figure S28. FT-IR: ῡ (KBr) = 2600-300, 1567, 1496, 1245, 745 cm^{-1}.

Figure S29, S30. ^{1}H NMR (500 MHz, $CDCl_3$): 5.82 (s, 1H), 8.128 (brs, 1H), 7.25-7.41 (m, 10H) ppm.

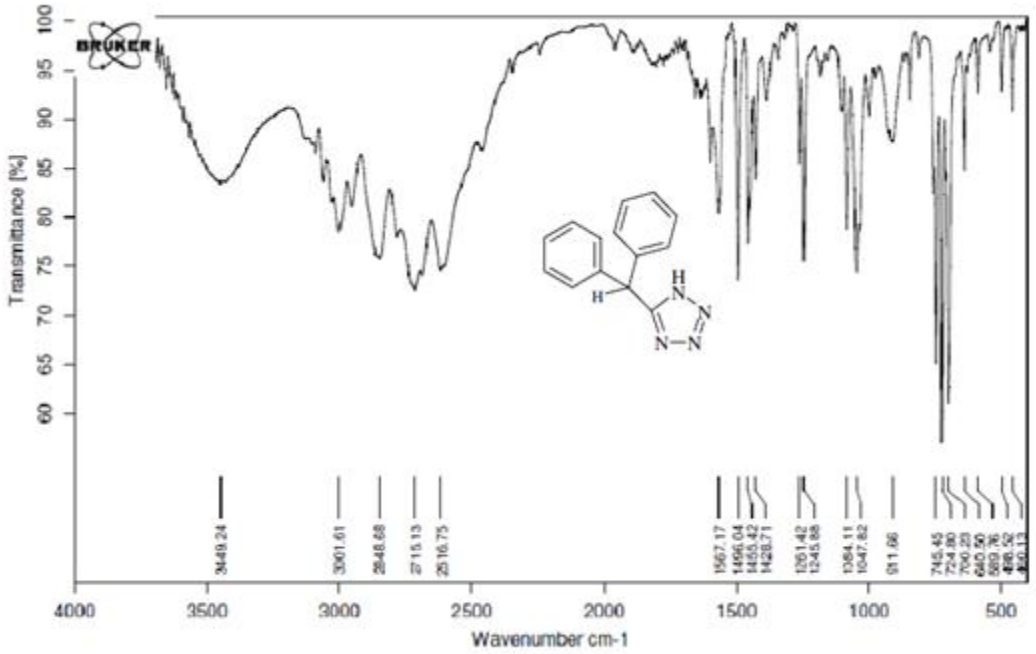

Figure S28. FT-IR (KBr) of 5-Benzyhydryltetrazole

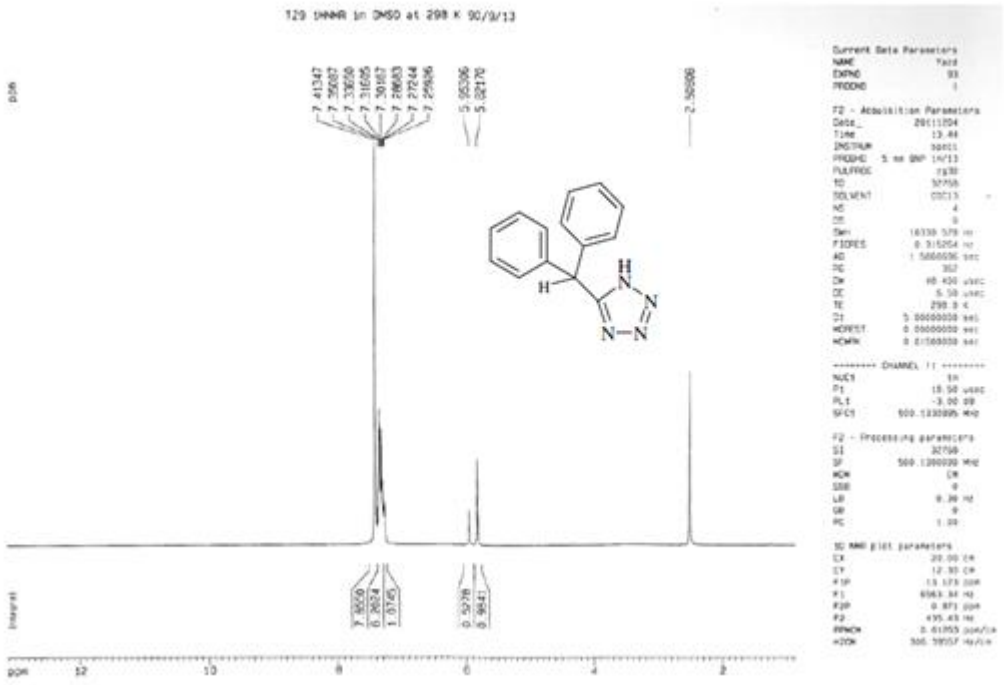

Figure S29. ^{1}H NMR (500 MHz, $CDCl_3$) of 5-Benzyhydryltetrazole

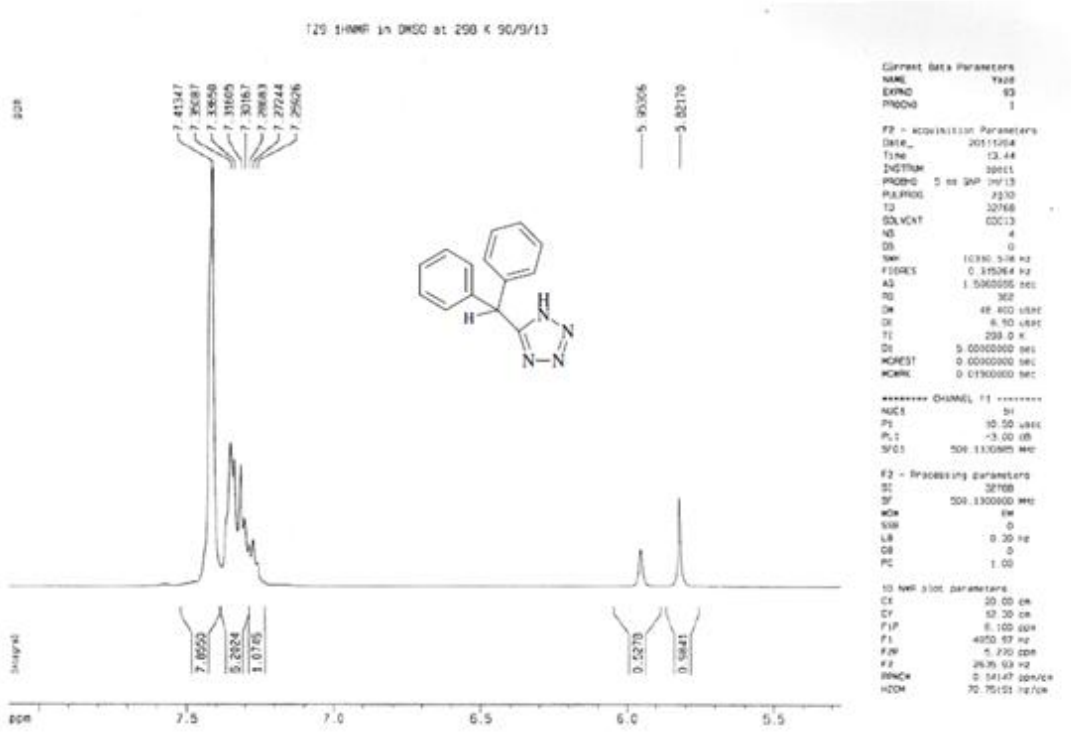

Figure S30. ^{1}H NMR (500 MHz, $CDCl_3$) of 5-Benzyhydryltetrazole (expand)

5-((3,4-dichlorophenyl)methyl)tetrazole

Yield: 84%, White crystal.

Figure S31. FT-IR: ῡ (KBr) = 2500-3300, 1560, 1472, 1440, 1260, 1210, 827, 766, 706, 674 cm^{-1}.

Figure S32, S33. ^{1}H NMR (500 MHz, $CDCl_3$): 7.61 (d, J= 8.45, 2H, 2H), 7.28 (d, J= 8.2, 1H), 4.32 (s, 2H) ppm.

Figure S34. ^{13}C-NMR (125 MHz, DMSO) δ = 28.8, 130.2, 131.6, 131.8, 174.8 ppm

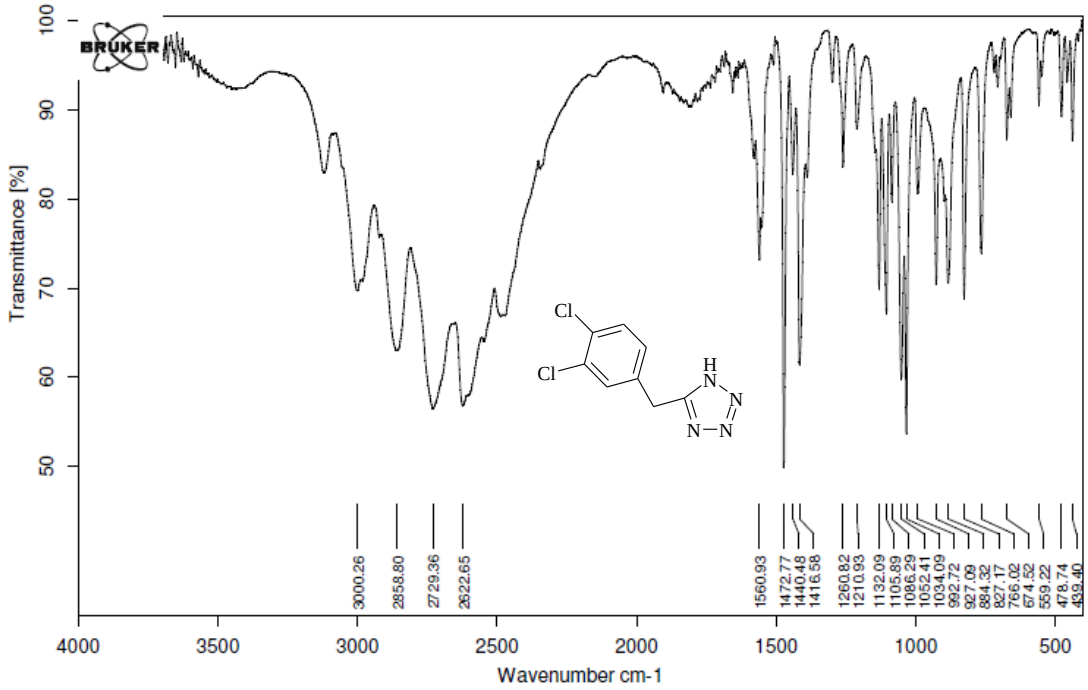

Figure S31: FT-IR (KBr) 5-((3,4-dichlorophenyl)methyl)tetrazole

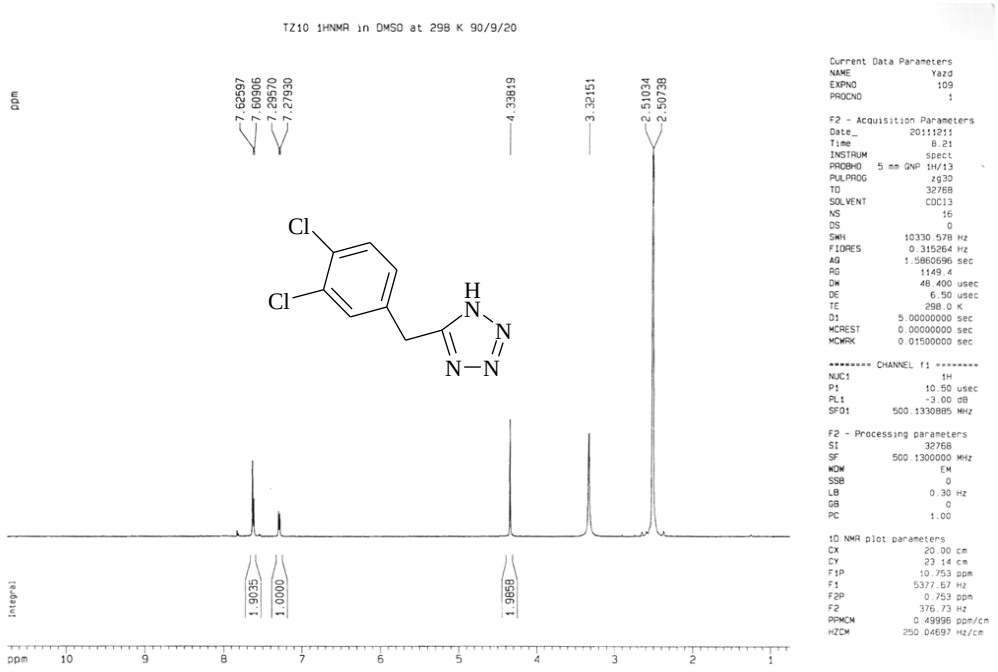

Figure S32. [1]H NMR (500 MHz, $CDCl_3$) 5-((3,4-dichlorophenyl)methyl)tetrazole

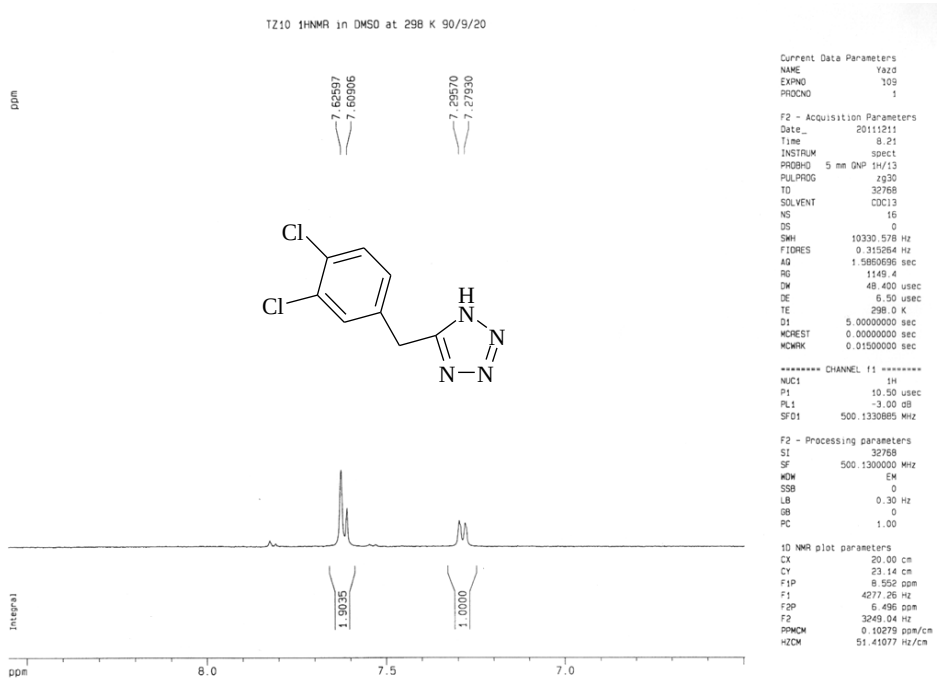

Figure S33. [1]H NMR (500 MHz, $CDCl_3$) 5-((3,4-dichlorophenyl)methyl)tetrazole (expand)

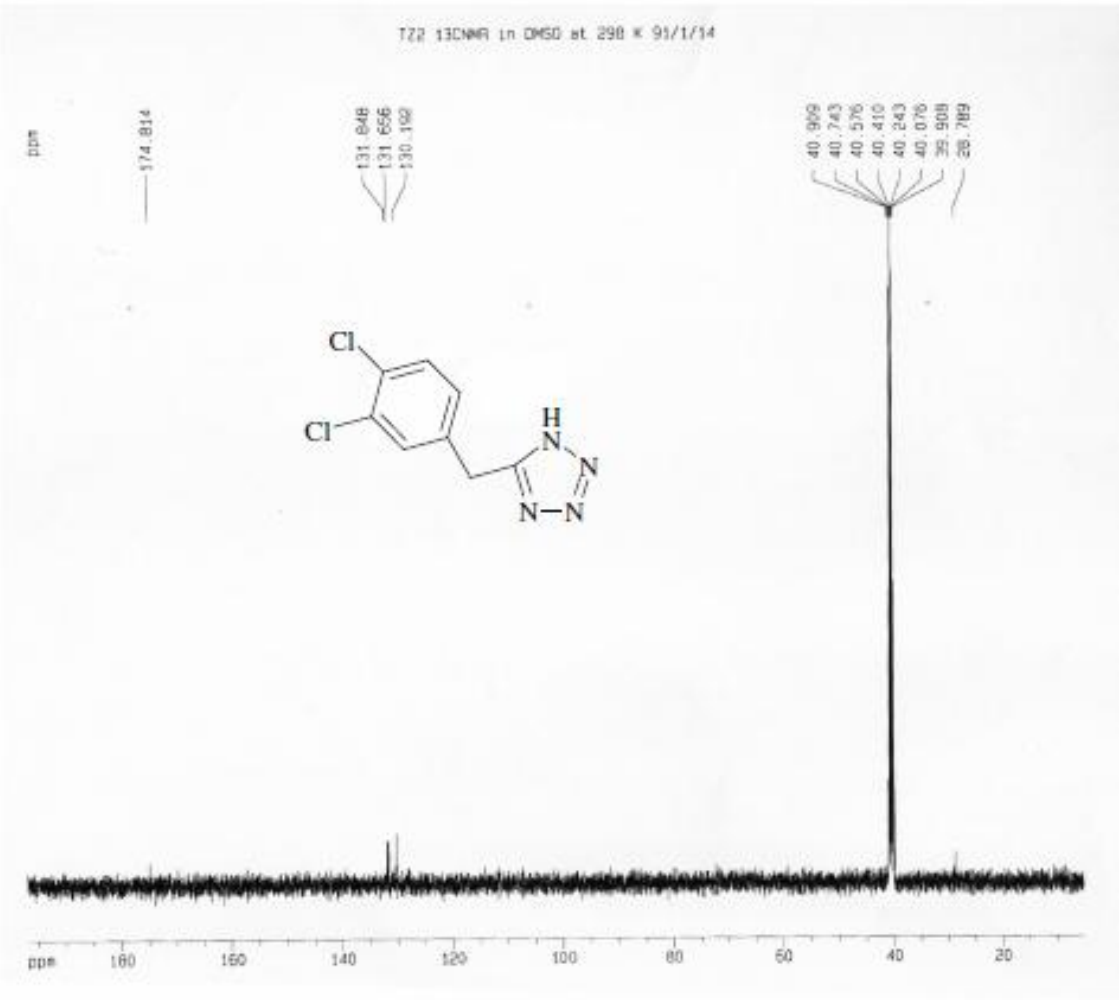

Figure S34. ^{13}C NMR (500 MHz, DMSO) 5-((3,4-dichlorophenyl)methyl)tetrazole

4-(1*H*-tetrazole-5-yl)pyridine (table 2, entry 12)

Yield: 92%, White crystal.

Figure S35. FT-IR:ῡ (KBr) =2500-3000, 1631, 1529, 1440, 1338, 1292, 1042, 990, 846, 751 cm^{-1}.

Figure S36, S37. ^{1}H NMR (500 MHz, $CDCl_3$): 8.00 (d, *J*= 7.89, 2H), 7.40 (d, *J*= 7.86, 2H) ppm.

Figure S38. ^{13}C-NMR (125 MHz, DMSO) δ = 127.9, 130.7 ppm.

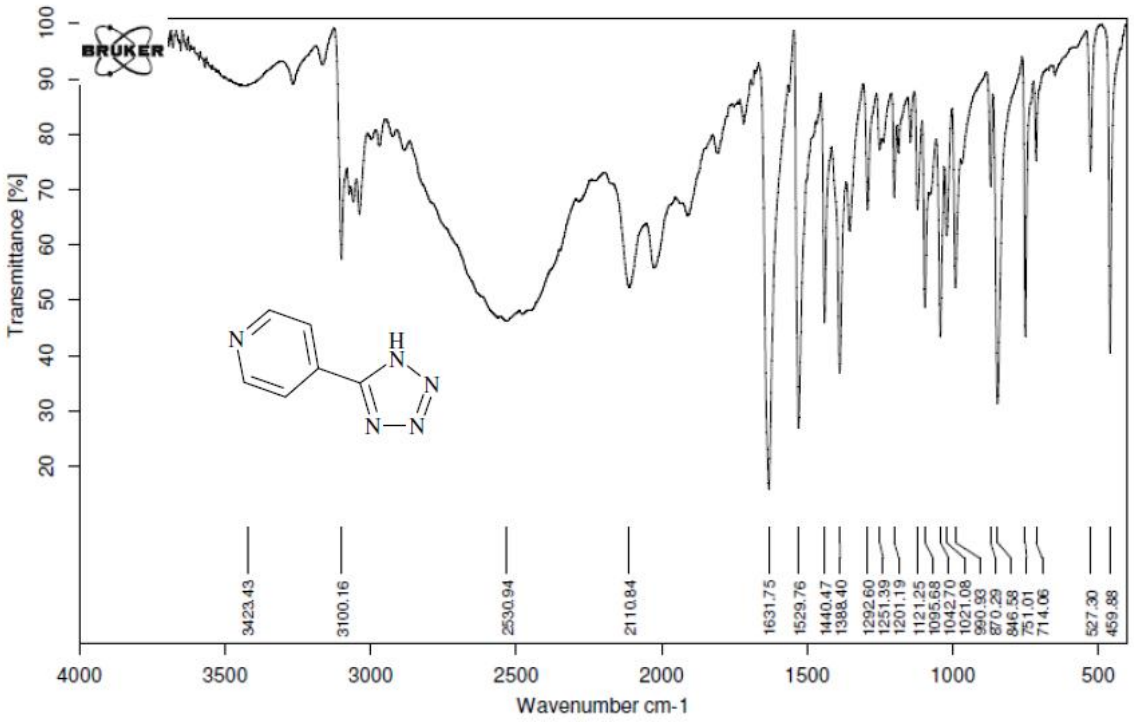

Figure S35. FT-IR (KBr) 4-(1*H*-tetrazole-5-yl)pyridine

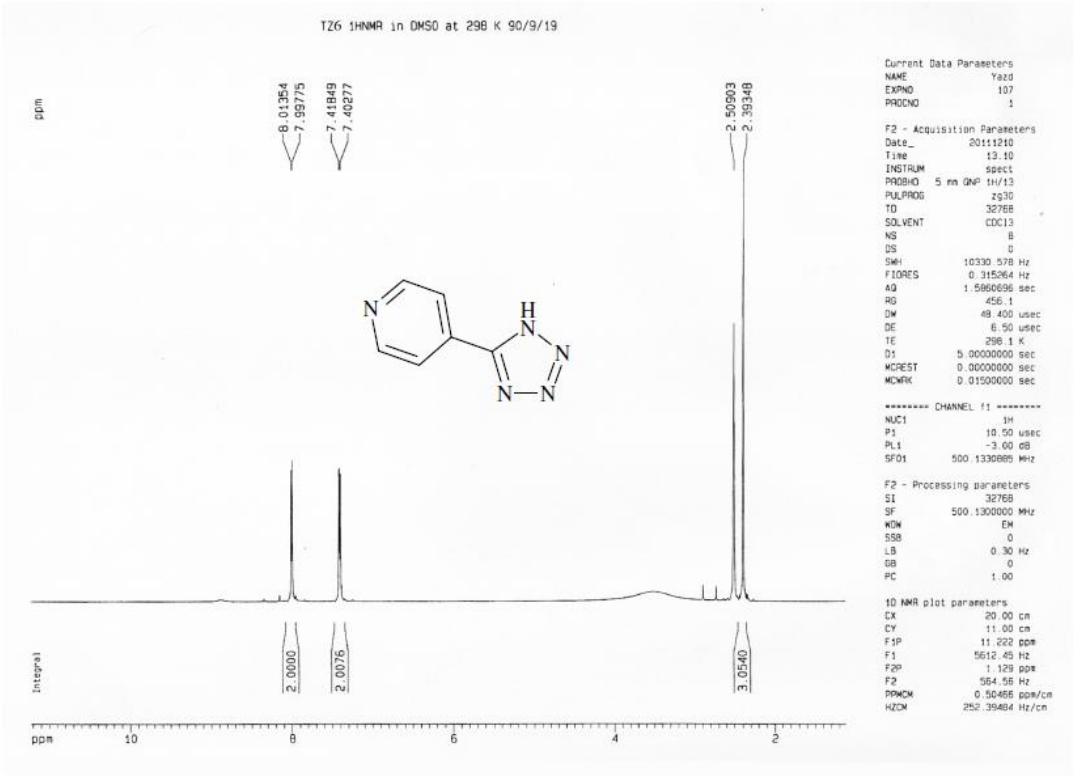

Figure S36. ^{1}H NMR (500 MHz, $CDCl_3$) 4-(1*H*-tetrazole-5-yl)pyridine

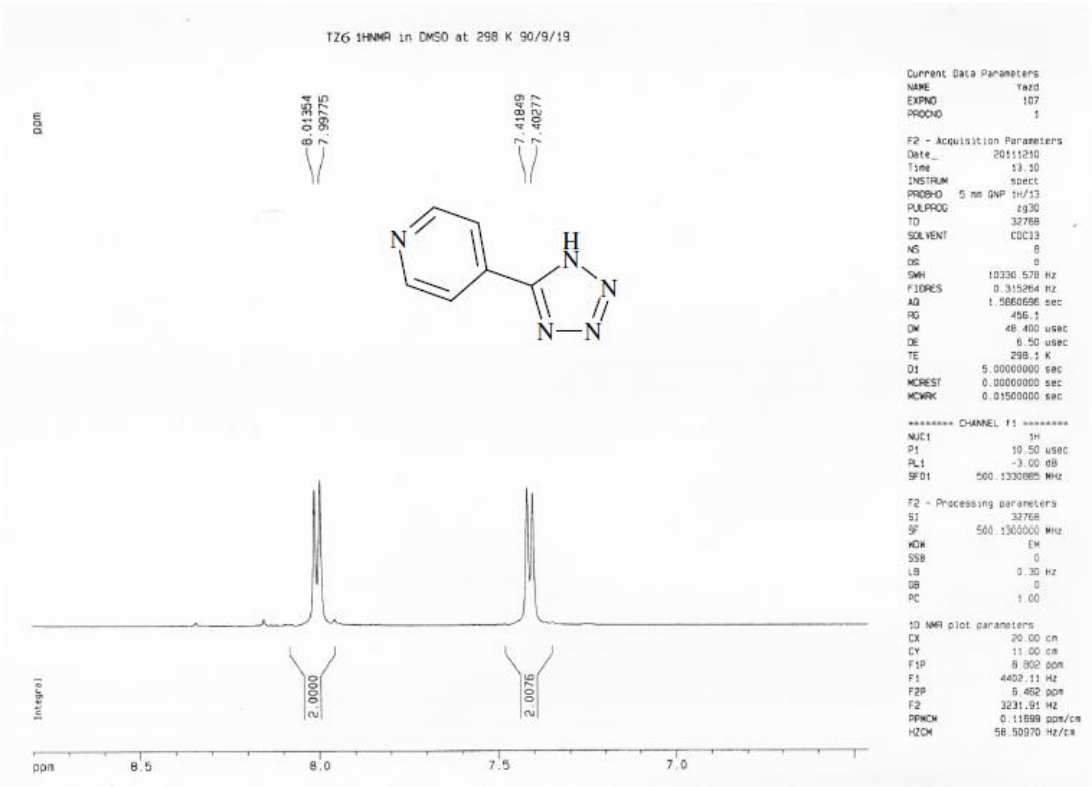

Figure S37. ^{1}H NMR (500 MHz, $CDCl_3$) 4-(1*H*-tetrazole-5-yl)pyridine (expand)

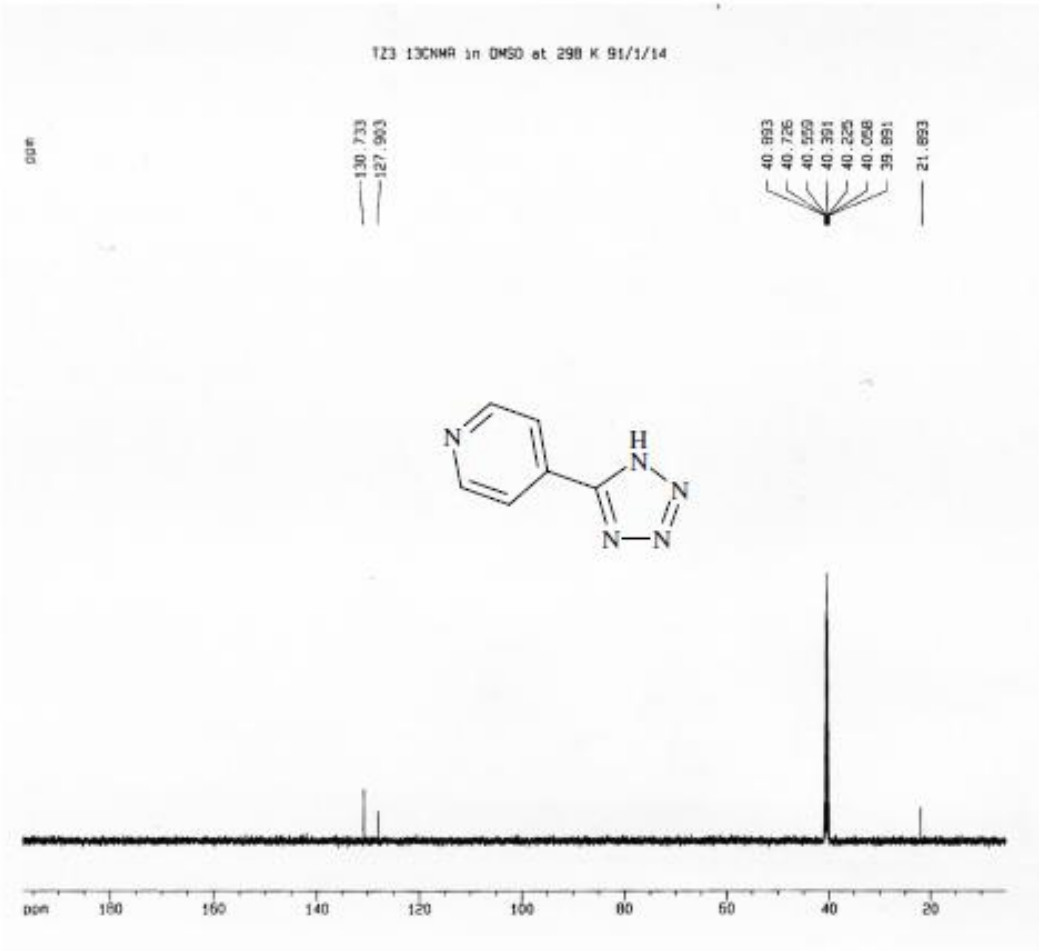

Figure S38. ^{13}C NMR (500 MHz, DMSO) 4-(1*H*-tetrazole-5-yl)pyridine

FT-IR, ^{1}H NMR and ^{13}C NMR Elucidation of 5-((3,4-dichlorophenyl)methyl)tetrazole

Characterization of 5-((3,4-dichlorophenyl)methyl)tetrazole was completed using FT-IR, ^{1}H NMR and ^{13}C NMR. The marked structure of 5-((3, 4-dichlorophenyl) methyl) tetrazole is showed in Figure 1.

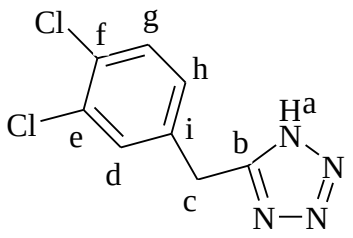

Figure 1

As can be seen in the IR spectrum, the stretching frequencies of C-H and N-H groups are indicated at 2500-3300 cm^{-1}, The stretching frequency of C=C group is demonstrated at 1560 cm^{-1}, The frequency absorption of tetrazole ring is specified at 1472 cm^{-1}, The stretching frequency of C-H benzyl group at 1260 cm^{-1} and the bending frequency of C-H phenyl ring is appeared at 827, 766, 706 cm^{-1} (Figure 1).

In the ^{1}H NMR spectrum, the appearance of the methylene protons (Hc, figure 1) as singlet at 4.32 ppm, integrating to two. The signal related to the one aromatic proton (Hh, figure 1) appear as a doublet at 7.28 ppm, integrating to one. In the appearance of a doublet at 7.61 ppm due to the two aromatic protons (Hg and Hd, figure 1), integrating to two, This signal appears at up filed due to the deshielding nature of the neighbouring chlorine atoms.

The ^{13}CNMR spectrum for 5-((3,4-dichlorophenyl)methyl) tetrazole displays signals characteristic various carbones (Cb, Cd, Cg, Ch, Cc, figure 1) at 28.8, 130.2, 131.6, 131.8, 174.8 ppm, respectively.

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