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Synthesis and Characterization of 5-Substituted 1H-tetrazoles in the Presence of Nano-TiCl₄·SiO₂

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Here, we describe the IR, ¹³C and ¹H NMR spectra of the novel 5-substituted 1H-tetrazole derivatives (Table 2, compounds 9, 11 and 12).

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Figure S38. $^{13}$C NMR of 4-(1H-tetrazole-5-yl)pyridine
5-Phenyl-1H-tetrazole

Yield: 95%, White crystal.

**Figure S1.** FT-IR: $\tilde{\nu}$ (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.
5-(4-Methylphenyl)-1H-tetrazole

Yield: 84%, White crystal.

Figure S3. FT-IR: $\tilde{\nu}$ (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)-1H-tetrazole](image-url)
**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)-1H-tetrazole

Yield: 82%, White crystal.

**Figure S6.** FT-IR: $\tilde{\nu}$ (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-Hydroxyphenyl)-1H-tetrazole](image-url)
Figure S7. $^1$H NMR (500 MHz, DMSO-d$_6$) 5-(4-Hydroxyphenyl)-1H-tetrazole

Figure S8. $^1$H NMR (500 MHz, DMSO-d$_6$) 5-(4-Hydroxyphenyl)-1H-tetrazole (expand)
5-(3-Nitropheryl)-1H-tetrazole
Yield: 92%, White crystal.

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

**Figure S10, S11.** \(^1\text{H} \) 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 S10. $^1$H NMR (500 MHz, DMSO-d$_6$) 5-(3-Nitrophenyl)-1H-tetrazole

Figure S11. $^1$H NMR (500 MHz, DMSO-d$_6$) 5-(3-Nitrophenyl)-1H-tetrazole (expand)
5-(4-Chlorophenyl)-1H-tetrazole

Yield: 91%, White crystal.

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

Figure S13, S14. $^1$H NMR (500 MHz, DMSO-d$_6$): 8.10 (d, $J = 10.53$, 2H), 7.69 (d, $J = 8.41$, 2H) ppm.

Figure S12: FT-IR (KBr) 5-(4-Chlorophenyl)-1H-tetrazole
Figure S13. $^1$H NMR (500 MHz, DMSO-d$_6$) 5-(4-Chlorophenyl)-1H-tetrazole

Figure S14. $^1$H NMR (500 MHz, DMSO-d$_6$) 5-(4-Chlorophenyl)-1H-tetrazole (expand)
5-(4-Bromophenyl)-1H-tetrazole
Yield: 90%, White crystal

**Figure S15.** FT-IR: $\tilde{\nu}$ (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)-1H-tetrazole
Figure S16. $^1$H NMR ((500 MHz, CDCl$_3$): 5-(4- Bromophenyl)-1H-tetrazole

Figure S17. $^1$H NMR ((500 MHz, CDCl$_3$): 5-(4- Bromophenyl)-1H-tetrazole (expand)
5-Benzyltetrazole

Yield: 78%, White crystal.

Figure S18. FT-IR: $\tilde{\nu}$ (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 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: $\tilde{\nu}$ (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 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: $\tilde{\nu}$ (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) $\delta$ = 155.0, 132.5, 128.9, 124.7, 123.6, 28.8 ppm.
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-Benzhydryltetrazole

Yield: 88%, White crystal.

**Figure S28.** FT-IR: $\tilde{\nu}$ (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-Benzhydryltetrazole](image)
Figure S29. $^1$H NMR (500 MHz, CDCl$_3$) of 5-Benzzyhydryltetrazole

Figure S30. $^1$H NMR (500 MHz, CDCl$_3$) of 5-Benzzyhydryltetrazole (expand)
5-((3,4-dichlorophenyl)methyl)tetrazole
Yield: 84%, White crystal.

**Figure S31.** FT-IR: $\tilde{\nu}$ (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) $\delta$ = 28.8, 130.2, 131.6, 131.8, 174.8 ppm

![Figure S31: FT-IR (KBr) 5-((3,4-dichlorophenyl)methyl)tetrazole](image-url)
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-(1H-tetrazole-5-yl)pyridine (table 2, entry 12)
Yield: 92%, White crystal.

Figure S35. FT-IR: $\tilde{\nu}$ (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) $\delta = 127.9$, 130.7 ppm.

Figure S35. FT-IR (KBr) 4-(1H-tetrazole-5-yl)pyridine
Figure S36. $^1$H NMR (500 MHz, CDCl$_3$) 4-(1H-tetrazole-5-yl)pyridine

Figure S37. $^1$H NMR (500 MHz, CDCl$_3$) 4-(1H-tetrazole-5-yl)pyridine (expand)
Figure S38. $^{13}$C NMR (500 MHz, DMSO) 4-(1H-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.

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.
References