

Supplementary Information

Effect of Sulfamic Acid on 1,3-Dipolar Cycloaddition Reaction: Mechanistic Studies and Synthesis of 4-Aryl-NH-1,2,3-triazoles from Nitroolefins

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Experimental characterization for compounds **2a-2t**

4-Phenyl-1H-1,2,3-triazole (**2a**)

White solid; yield 94%; mp 140-142 °C; ¹H NMR (300 MHz, DMSO-*d*₆ + CDCl₃) δ 7.27 (t, 1H, *J* 7.4, 12.1 Hz, Ph-H), 7.37 (t, 2H, *J* 7.2, 14.9 Hz, Ph-H), 7.74 (d, 2H, *J* 7.4 Hz, Ph-H), 7.87 (s, 1H, *Trz-H); ¹³C NMR (125 MHz, DMSO-*d*₆ + CDCl₃) δ 125.2, 127.5, 128.1, 129.7, 130.1, 146.2; HRMS (ESI) *m/z* calcd. for C₈H₇N₃ [M + H]⁺: 146.0713, found: 146.0721; *Trz-H: triazole hydrogen.

4-(*p*-Tolyl)-1H-1,2,3-triazole (**2b**)

White solid; yield 91%; mp 148-150 °C; ¹H NMR (500 MHz, DMSO-*d*₆ + CDCl₃) δ 2.37 (s, 3H, Ph-CH₃), 7.23 (d, 2H, *J* 7.9 Hz, Ph-H), 7.70 (d, 2H, *J* 7.8 Hz, Ph-H), 7.91 (s, 1H, Trz-H); ¹³C NMR (125 MHz, DMSO-*d*₆ + CDCl₃) δ 20.0, 124.5, 126.7, 128.2, 128.9, 136.5, 145.4; HRMS (ESI) *m/z* calcd. for C₉H₁₀N₃ [M + H]⁺: 160.0869, found: 160.0886.

4-(4-Methoxyphenyl)-1H-1,2,3-triazole (**2c**)

White solid; mp 160-163 °C; yield 87%; ¹H NMR (500 MHz, DMSO-*d*₆ + CDCl₃) δ 3.84 (s, 3H, Ph-OMe), 6.95 (s, 1H, Ph-H), 6.97 (s, 1H, Ph-H), 7.75 (s, 2H, Ph-H), 7.83 (s, 1H, Trz-H); ¹³C NMR (125 MHz, DMSO-*d*₆ + CDCl₃) δ 54.0, 113.0, 121.8, 125.9, 128.0, 158.3; HRMS (ESI) *m/z* calcd. for C₉H₉N₃O [M + H]⁺: 176.0818, found: 176.0809.

4-(3,4-Dimethoxyphenyl)-1*H*-1,2,3-triazole (**2d**)

White solid; yield 89%; mp 157-159 °C; ¹H NMR (300 MHz, DMSO-*d*₆ + CDCl₃) δ 3.91 (s, 3H, Ph-OMe), 3.95 (s, 3H, Ph-OMe), 6.95 (d, 1H, *J* 8.3 Hz, Ph-H), 7.41-7.33 (m, 2H, Ph-H), 7.88 (s, 1H, Trz-H); ¹³C NMR (125 MHz, DMSO-*d*₆ + CDCl₃) δ 55.2, 55.3, 108.6, 111.1, 117.9, 123.2, 129.4, 146.1, 147.7, 147.9; HRMS (ESI) *m/z* calcd. for C₁₀H₁₁N₃O₂ [M + H]⁺: 206.0924; found: 206.0914.

2-Methoxy-4-(1*H*-1,2,3-triazol-4-yl)phenol (**2e**)

White solid; yield 90%; mp 146-148 °C; ¹H NMR (300 MHz, DMSO-*d*₆ + CDCl₃) δ 3.94 (s, 3H, Ph-OMe), 6.93 (d, 1H, *J* 8.3 Hz, Ph-H), 7.23 (d, 1H, *J* 7.7 Hz, Ph-H), 7.38 (s, 1H, Ph-H), 7.82 (s, 1H, Trz-H), 8.14 (brs, 1H, Ph-OH); ¹³C NMR (125 MHz, DMSO-*d*₆ + CDCl₃) δ 55.4, 108.6, 114.8, 118.4, 128.9, 146.0, 147.0; HRMS (ESI) *m/z* calcd. for C₉H₉N₃O₂ [M + H]⁺: 192.0768, found: 192.0758.

4-(3,4,5-Trimethoxyphenyl)-1*H*-1,2,3-triazole (**2f**)

White solid; yield 88%; mp 163-165 °C; ¹H NMR (300 MHz, DMSO-*d*₆ + CDCl₃) δ 3.86 (s, 3H, Ph-OMe), 3.95 (s, 6H, 2Ph-OMe), 7.06 (s, 1H, Ph-H), 7.46 (s, 1H, Ph-H), 7.88 (s, 1H, Trz-H); ¹³C NMR (125 MHz, DMSO-*d*₆ + CDCl₃) δ 55.3, 59.8, 102.2, 125.6, 129.4, 137.0, 145.9, 152.6; HRMS (ESI) *m/z* calcd. for C₁₁H₁₃N₃O₃ [M + H]⁺: 236.1030, found: 236.1021.

4-(2,4,5-Trimethoxyphenyl)-1*H*-1,2,3-triazole (**2g**)

White solid; yield 91%; mp 167-169 °C; ¹H NMR (500 MHz, DMSO-*d*₆ + CDCl₃) δ 4.39-4.33 (m, 9H, 3Ph-OMe), 7.06 (s, 1H, Ph-H), 8.06 (s, 1H, Trz-H), 8.53 (s, 1H, Ph-H); ¹³C NMR (125 MHz, DMSO-*d*₆ + CDCl₃) δ 55.9, 56.1, 56.3, 97.3, 109.7, 110.5, 128.6, 142.6, 149.5, 150.7; HRMS (ESI) *m/z* calcd. for C₁₁H₁₃N₃O₃ [M + H]⁺: 236.1030, found: 236.1042.

4-(3-Bromophenyl)-1*H*-1,2,3-triazole (**2h**)

White solid; yield 60%; mp 125-127 °C; ¹H NMR (500 MHz, DMSO-*d*₆ + CDCl₃) δ 7.23-7.19 (m, 1H, Ph-H), 7.34 (d, 1H, *J* 7.6 Hz, Ph-H), 7.66 (d, 1H, *J* 7.5 Hz, Ph-H), 7.82 (s, 1H, Ph-H), 7.90 (s, 1H, Trz-H); ¹³C NMR (125 MHz, DMSO-*d*₆ + CDCl₃) δ 122.5, 124.2, 127.5, 128.5, 130.1, 130.7, 132.3, 144.5; HRMS (ESI) *m/z* calcd. for C₈H₆BrN₃ [M + H]⁺: 223.9818, found: 223.9870.

4-(4-Fluorophenyl)-1*H*-1,2,3-triazole (**2i**)

White solid; yield 81%; mp 162-164 °C; ¹H NMR (500 MHz, DMSO-*d*₆ + CDCl₃) δ 7.18-7.05 (m, 2H, Ph-H), 7.86-7.75 (m, 2H, Ph-H), 8.05 (s, 1H, Trz-H); ¹³C NMR (125 MHz, DMSO-*d*₆ + CDCl₃) δ 114.0 (d, *J*_{CF} 21.8 Hz), 125.3, 125.9 (q, *J*_{CF} 8.2 Hz), 144.3, 159.5, 161.5; HRMS (ESI) *m/z* calcd. for C₈H₆FN₃ [M + H]⁺: 164.0624, found: 164.0613.

4-(4-Chlorophenyl)-1*H*-1,2,3-triazole (**2j**)

White solid; yield 78%; mp 152-154 °C; ¹H NMR (500 MHz, DMSO-*d*₆ + CDCl₃) δ 7.43 (d, 2H, *J* 8.2 Hz, Ph-H), 7.76 (d, 2H, *J* 8.1 Hz, Ph-H), 7.95 (s, 1H, Trz-H); ¹³C NMR (125 MHz, DMSO-*d*₆ + CDCl₃) δ 125.9, 127.6, 127.9, 128.0, 132.1; HRMS (ESI) *m/z* calcd. for C₈H₆ClN₃ [M + H]⁺: 180.0323, found: 180.0368.

2-(1*H*-1,2,3-Triazol-4-yl)phenol (**2k**)

White solid; yield 85%; mp 150-152 °C; ¹H NMR (500 MHz, DMSO-*d*₆ + CDCl₃) δ 6.83 (t, 1H, *J* 7.3, 14.5 Hz, Ph-H), 6.97-6.91 (m, 1H, Ph-H), 7.13 (t, 1H, *J* 7.3, 14.3 Hz, Ph-H), 7.51 (d, 1H, *J* 6.7 Hz, Ph-H), 7.92 (s, 1H, Trz-H), 10.0 (brs, 1H, Ph-OH); ¹³C NMR (125 MHz, DMSO-*d*₆ + CDCl₃) δ 114.2, 116.9, 119.4, 126.1, 129.1, 129.4, 145.3, 155.0; HRMS (ESI) *m/z* calcd. for C₈H₇N₃O [M + H]⁺: 162.0662, found: 162.0655.

4-(Furan-2-yl)-1*H*-1,2,3-triazole (**2l**)

Brown solid; yield 80%; mp 80-82 °C; ¹H NMR (500 MHz, CDCl₃) δ 6.53-6.51 (m, 1H, *F-H), 6.85 (d, 1H, *J* 3.1 Hz, *F-H), 7.52 (d, 1H, *J* 1.1 Hz, *F-H), 7.97 (s, 1H, Trz-H); ¹³C NMR (125 MHz, CDCl₃) δ 107.8, 111.6, 128.6, 139.4, 142.8, 145.2; HRMS (ESI) *m/z* calcd. for C₆H₆N₃O [M + H]⁺: 136.0510, found: 136.0508; *F-H: furan hydrogens.

4-(Thiophen-2-yl)-1*H*-1,2,3-triazole (**2m**)

Brown solid; yield 85%; mp 88-90 °C; ¹H NMR (500 MHz, DMSO-*d*₆ + CDCl₃) δ 6.98 (s, 1H, *T-H), 7.21 (s, 1H, *T-H), 7.29 (s, 1H, Trz-H), 7.71 (s, 1H, *T-H); ¹³C NMR (125 MHz, DMSO-*d*₆ + CDCl₃) δ 124.0, 124.7, 126.7, 127.1, 132.3, 140.8; HRMS (ESI) *m/z* calcd. for C₆H₅N₃S [M + H]⁺: 152.0277, found: 152.0282; *T-H: thiophene hydrogens.

4-Phenyl-3,4-dihydrochromeno[3,4-*d*][1,2,3]triazole (**2n**)

White solid; yield 92%; mp 150-152 °C; ¹H NMR (500 MHz, DMSO-*d*₆ + CDCl₃) δ 6.54 (d, 1H, *J* 10.5 Hz, OCH), 6.97 (s, 2H, Ph-H), 7.18-7.14 (m, 1H, Ph-H), 7.30-7.23 (m, 3H, Ph-H), 7.37 (t, 2H, *J* 7.5, 16.9 Hz, Ph-H), 7.68 (t, 1H, *J* 7.8, 16.9 Hz, Ph-H); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 76.0, 115.7, 117.0, 119.6, 121.5, 122.5, 126.6, 128.0, 128.1, 129.4, 138.9, 152.9; HRMS (ESI) *m/z* calcd. for C₁₅H₁₁N₃O [M + H]⁺: 250.0975, found: 250.0976.

4-(*p*-Tolyl)-3,4-dihydrochromeno[3,4-*d*][1,2,3]triazole (**2o**)

White solid; yield 93%; mp 162-164 °C; ¹H NMR (500 MHz, DMSO-*d*₆ + CDCl₃) δ 2.77 (s, 3H, Ph-Me), 7.02 (s, 1H, OCH), 7.49 (t, 2H, *J* 8.4, 16.3 Hz, Ph-H), 7.61 (d, 2H, *J* 7.9 Hz, Ph-H), 7.69-7.66 (m, 1H, Ph-H), 7.77 (d, 2H, *J* 7.9 Hz, Ph-H), 8.21 (d, 1H, *J* 8.39 Hz, Ph-H); ¹³C NMR (125 MHz, DMSO-*d*₆ + CDCl₃) δ 21.0, 76.0, 116.2, 117.3, 122.0, 123.0, 127.2, 129.3, 129.9, 136.1, 137.3, 138.5, 141.2, 153.2; HRMS (ESI) *m/z* calcd. for C₁₆H₁₃N₃O [M + H]⁺: 264.1131, found: 264.1124.

5-Methyl-4-(3,4,5-trimethoxyphenyl)-1*H*-1,2,3-triazole (**2p**)

White solid; yield 85%; mp 120-122 °C; ¹H NMR (500 MHz, DMSO-*d*₆ + CDCl₃) δ 2.34 (s, 3H, Trz-Me), 3.85 (s, 3H, Ph-OMe), 3.88 (s, 6H, 2Ph-OMe), 6.91 (s, 2H, Ph-H), 7.27 (s, 1H, Ph-H); ¹³C NMR (125 MHz, DMSO-*d*₆ + CDCl₃) δ 29.5, 56.0, 60.8, 104.3, 126.7, 137.6, 142.4, 153.2; HRMS (ESI) *m/z* calcd. for C₁₂H₁₅N₃O₃ [M + H]⁺: 250.1186, found: 250.1170.

4-(3,6,7-Trimethoxyphenanthren-9-yl)-1*H*-1,2,3-triazole (**2q**)

Yellow solid; yield 83%; mp 211-213 °C; ¹H NMR (500 MHz, DMSO-*d*₆) δ 3.86 (s, 3H, Ph-OMe), 4.02 (s, 3H, Ph-OMe), 4.06 (s, 3H, Ph-OMe), 7.26 (d, 1H, *J* 7.6 Hz, Ph-H), 7.97-7.90 (m, 2H, Ph-H), 8.11 (d, 2H, *J* 12.4 Hz, Ph-H), 8.17 (s, 1H, Trz-H), 8.31-8.26 (m, 1H, Ph-H); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 55.1, 55.6, 55.8, 104.0, 104.6, 106.5,

116.3, 121.7, 123.1, 124.5, 125.0, 125.8, 130.3, 130.7, 133.3, 146.4, 148.9, 149.3, 158.5; HRMS (ESI) m/z calcd. for $C_{19}H_{17}N_3O_3$ $[M + H]^+$: 336.1343, found: 336.1342.

4-(2,3,6,7-Tetramethoxyphenanthren-9-yl)-1H-1,2,3-triazole (**2r**)

Brown solid; yield 85%; mp 268-270 °C; 1H NMR (500 MHz, $DMSO-d_6$) δ 3.86 (s, 3H, Ph-OMe), 3.91 (s, 3H, Ph-OMe), 4.06 (d, 6 H, J 2.7 Hz, 2Ph-OMe), 7.44 (s, 1H, Ph-H), 7.91 (s, 1H, Ph-H), 8.04-8.94 (m, 2H, Ph-H), 8.09 (s, 1H, Trz-H), 8.35 (s, 1H, Ph-H); ^{13}C NMR (125 MHz, $DMSO-d_6$) δ 55.1, 55.4, 55.8, 55.7, 103.5, 104.1, 106.4, 108.5, 123.4, 124.1, 124.7, 125.2, 125.3, 146.5, 148.7, 148.9, 149.6; HRMS (ESI) m/z calcd. for $C_{20}H_{19}N_3O_4$ $[M + H]^+$: 366.1454, found: 366.1514.

Ethyl 2-(2-methoxy-4-(1H-1,2,3-triazol-4-yl)phenoxy)acetate (**2s**)

Colorless liquid; yield 84%; 1H NMR (500 MHz, $CDCl_3$) δ 1.21 (t, 3H, J 7.0, 14.2 Hz, $-CH_3$), 3.85 (s, 3H, Ph-OMe), 4.22-4.17 (m, 2H, $-CH_2CH_3$), 4.66 (s, 2H, OCH_2CO), 6.80 (d, 1H, J 8.4 Hz, Ph-H), 7.20 (d, 1H, J 7.2 Hz, Ph-H), 7.34 (s, 1H, Ph-H), 7.83 (s, 1H, Trz-H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 13.1, 55.0, 60.5, 65.4, 108.8, 113.2, 117.6, 123.3, 127.5, 145.5, 146.5, 148.9, 168.0; HRMS (ESI) m/z calcd. for $C_{13}H_{15}N_3O_4$ $[M + H]^+$: 278.1135, found: 278.1135.

tert-Butyl 2-(2-methoxy-4-(1H-1,2,3-triazol-4-yl)phenoxy)acetate (**2t**)

Colorless liquid; yield 82%; 1H NMR (500 MHz, $CDCl_3$) δ 1.40 (s, 9H, *t*-butyl), 3.85 (s, 3H, Ph-OMe), 4.57 (s, 2H, OCH_2CO), 6.76 (d, 1H, J 8.2 Hz, Ph-H), 7.20 (d, 1H, J 7.8 Hz, Ph-H), 7.33 (s, 1H, Ph-H), 7.83 (s, 1H, Trz-H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 28.0, 56.0, 66.5, 82.5, 109.8, 113.7, 118.5, 124.0, 128.2, 146.3, 147.6, 149.7, 168.0; HRMS (ESI) m/z calcd. for $C_{15}H_{20}N_3O_4$ $[M + H]^+$: 306.1453, found: 306.1458.

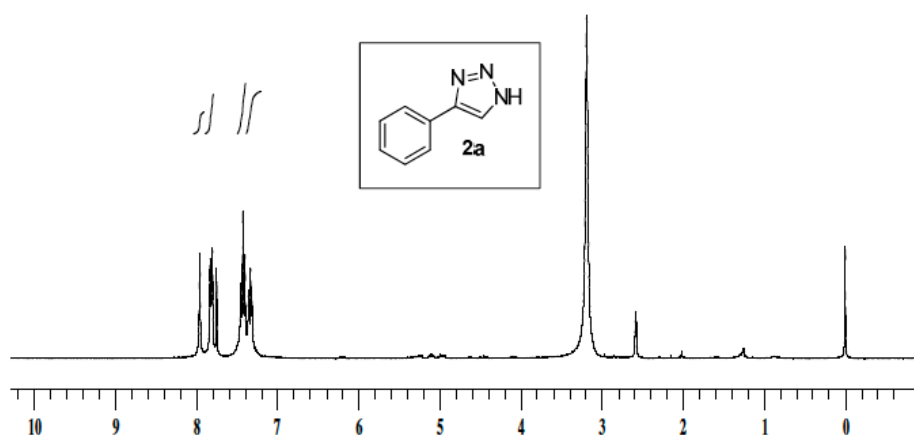


Figure S1. 1H NMR spectrum (300 MHz, $DMSO-d_6 + CDCl_3$) of compound **2a**.

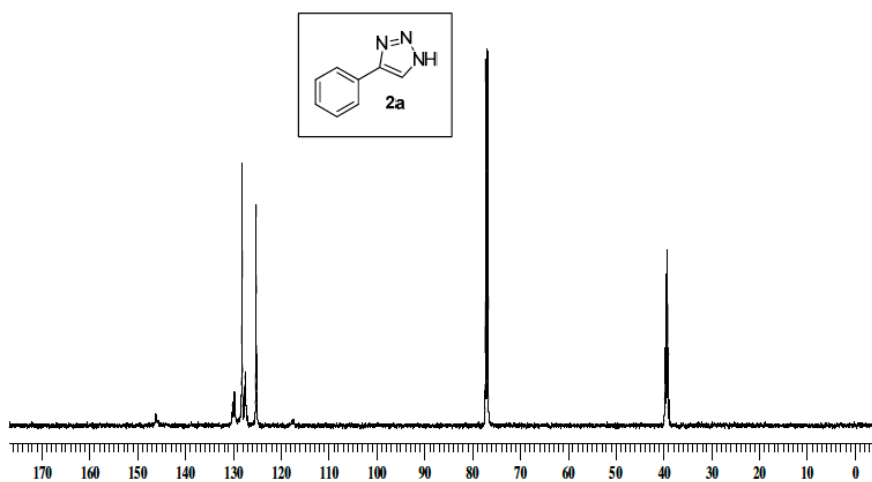


Figure S2. ¹³C NMR spectrum (125 MHz, DMSO-*d*₆ + CDCl₃) of compound **2a**.

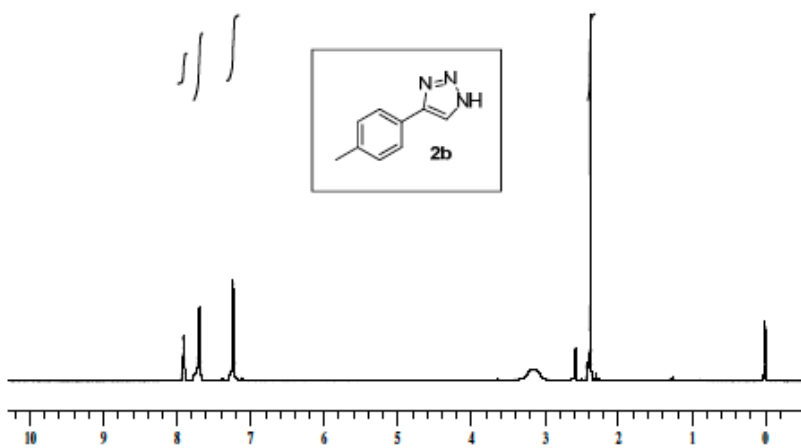


Figure S3. ¹H NMR spectrum (500 MHz, DMSO-*d*₆ + CDCl₃) of compound **2b**.

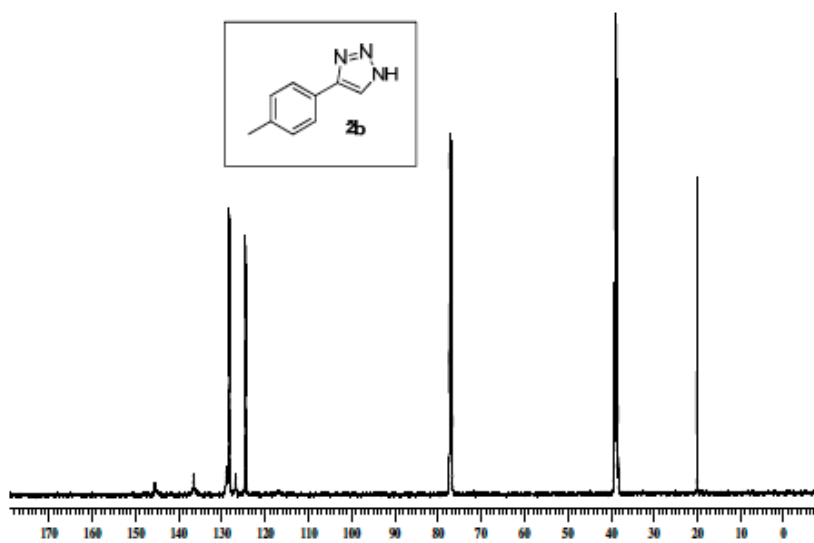


Figure S4. ¹³C NMR spectrum (125 MHz, DMSO-*d*₆ + CDCl₃) of compound **2b**.

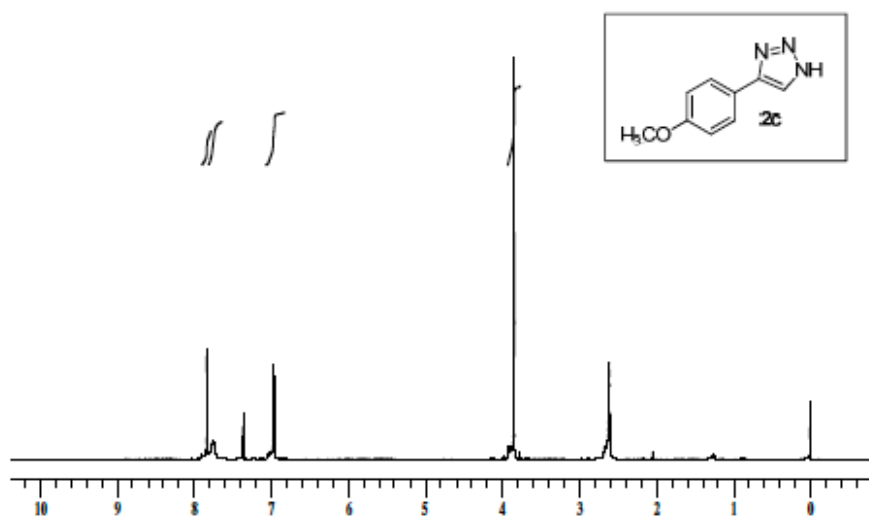


Figure S5. ^1H NMR spectrum (500 MHz, $\text{DMSO-}d_6 + \text{CDCl}_3$) of compound **2c**.

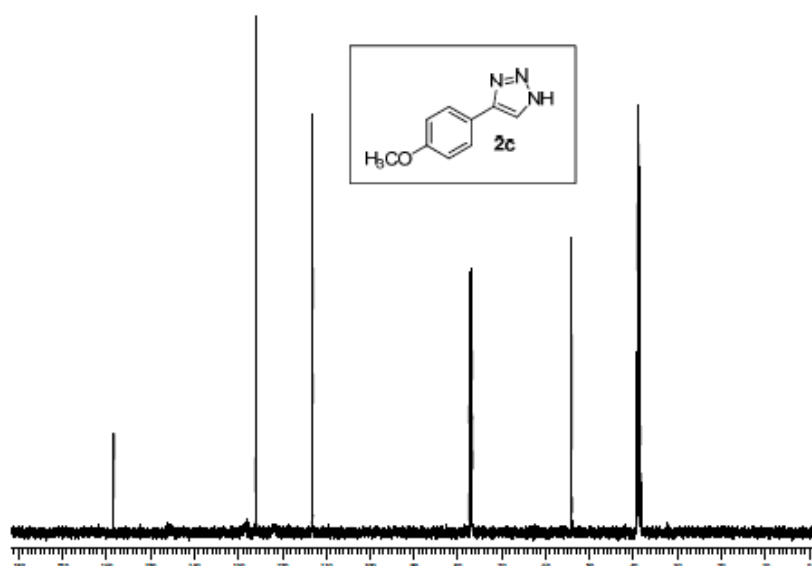


Figure S6. ^{13}C NMR spectrum (125 MHz, $\text{DMSO-}d_6 + \text{CDCl}_3$) of compound **2c**.

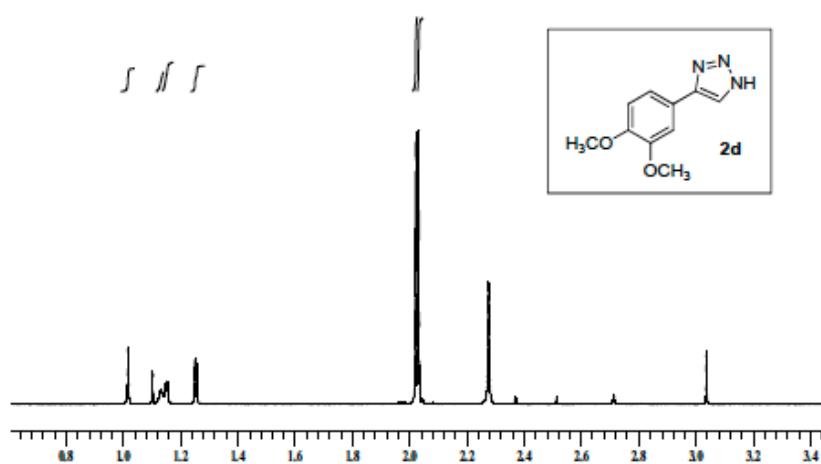


Figure S7. ^1H NMR spectrum (300 MHz, $\text{DMSO-}d_6 + \text{CDCl}_3$) of compound **2d**.

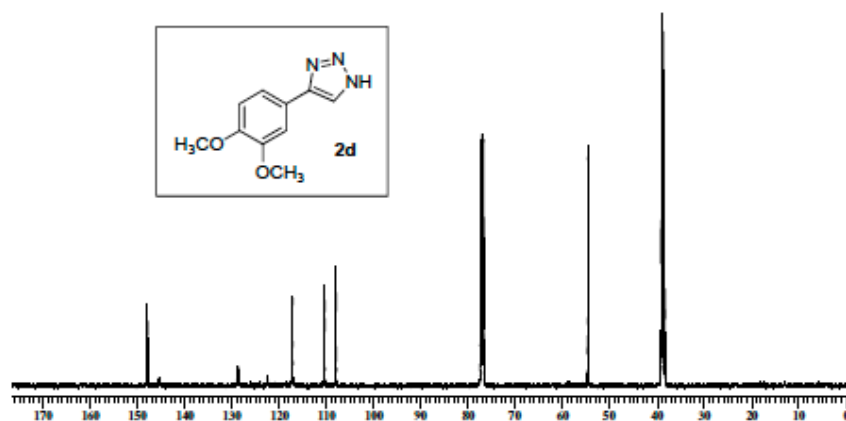


Figure S8. ¹³C NMR spectrum (125 MHz, DMSO-*d*₆ + CDCl₃) of compound 2d.

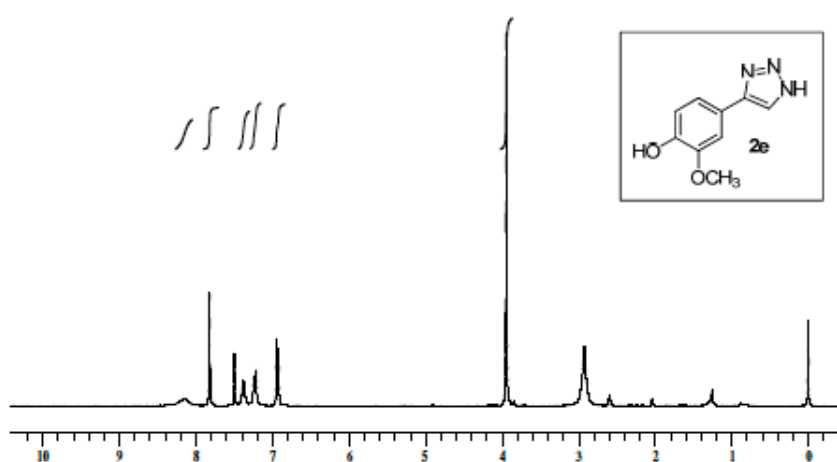


Figure S9. ¹H NMR spectrum (300 MHz, DMSO-*d*₆ + CDCl₃) of compound 2e.

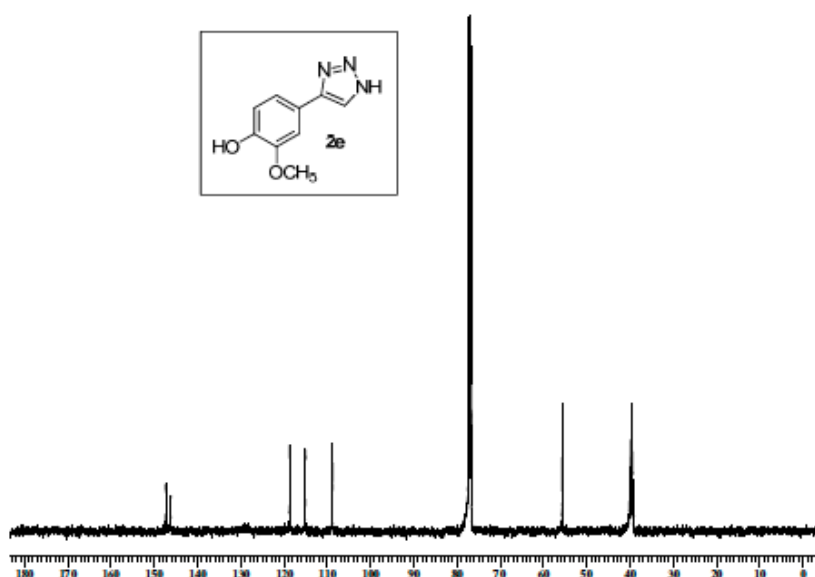


Figure S10. ¹³C NMR spectrum (125 MHz, DMSO-*d*₆ + CDCl₃) of compound 2e.

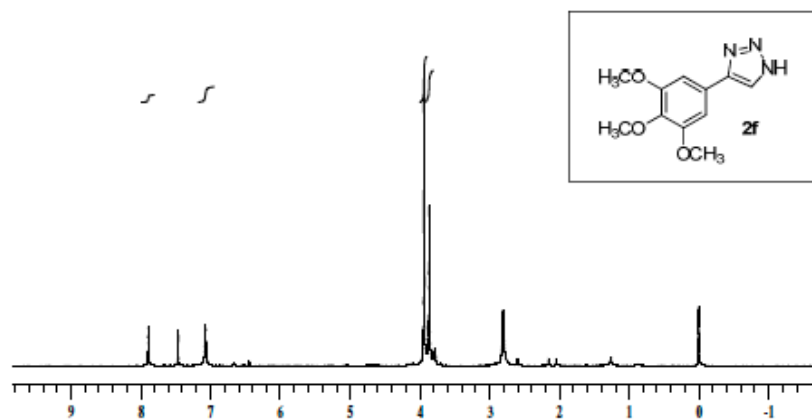


Figure S11. ^1H NMR spectrum (300 MHz, $\text{DMSO-}d_6 + \text{CDCl}_3$) of compound **2f**.

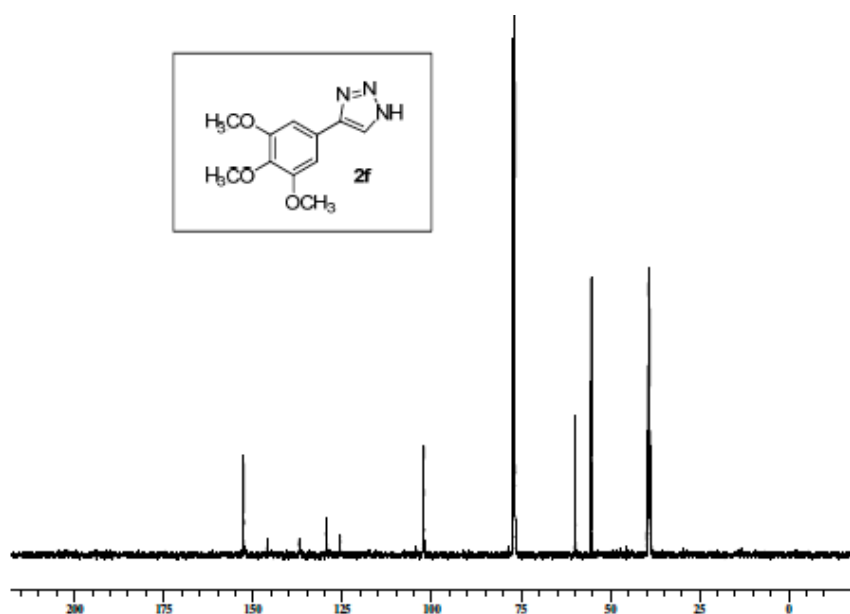


Figure S12. ^{13}C NMR spectrum (125 MHz, $\text{DMSO-}d_6 + \text{CDCl}_3$) of compound **2f**.

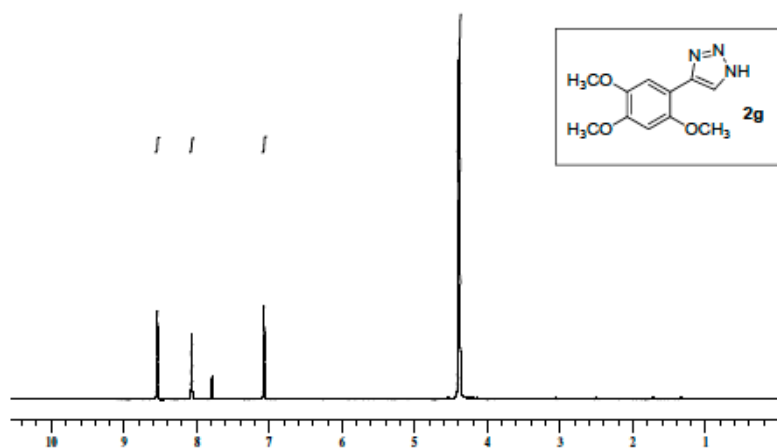


Figure S13. ^1H NMR spectrum (500 MHz, $\text{DMSO-}d_6 + \text{CDCl}_3$) of compound **2g**.

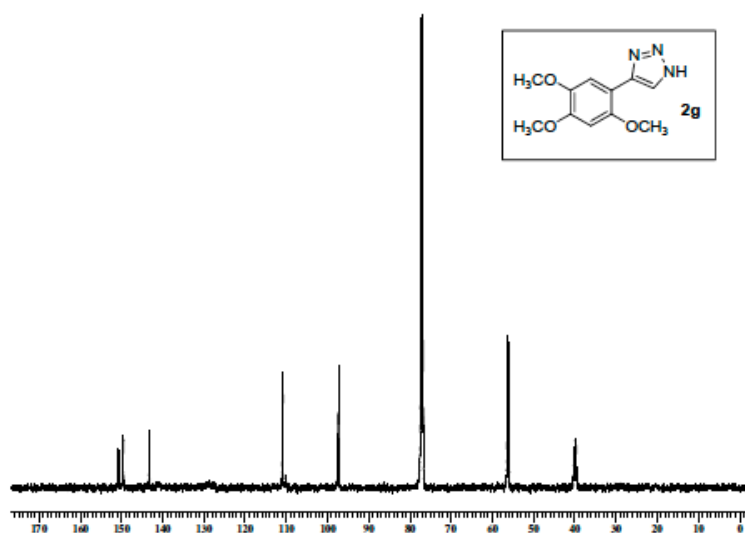


Figure S14. ^{13}C NMR spectrum (125 MHz, $\text{DMSO-}d_6 + \text{CDCl}_3$) of compound **2g**.

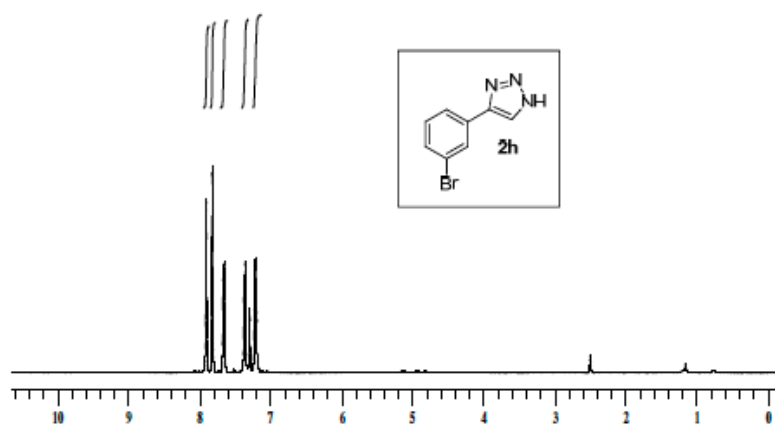


Figure S15. ^1H NMR spectrum (500 MHz, $\text{DMSO-}d_6 + \text{CDCl}_3$) of compound **2h**.

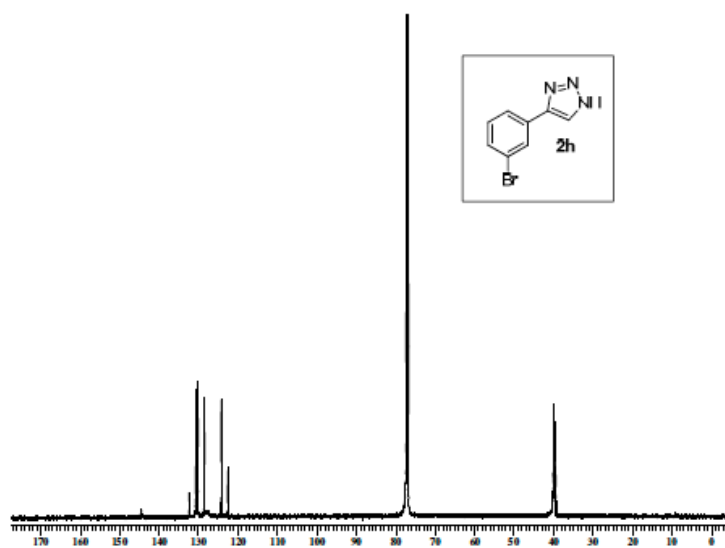


Figure S16. ^{13}C NMR spectrum (125 MHz, $\text{DMSO-}d_6 + \text{CDCl}_3$) of compound **2h**.

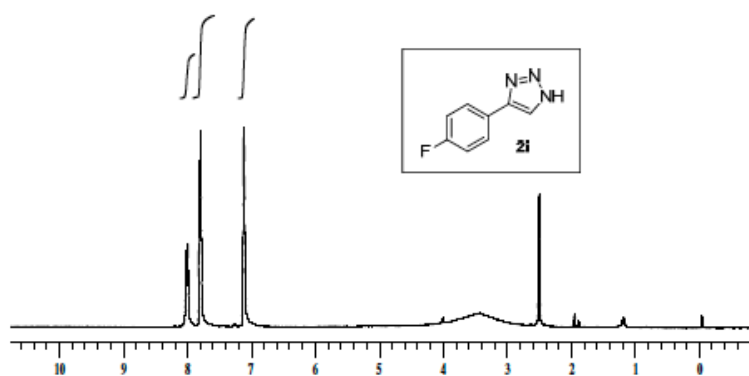


Figure S17. ^1H NMR spectrum (500 MHz, $\text{DMSO-}d_6 + \text{CDCl}_3$) of compound **2i**.

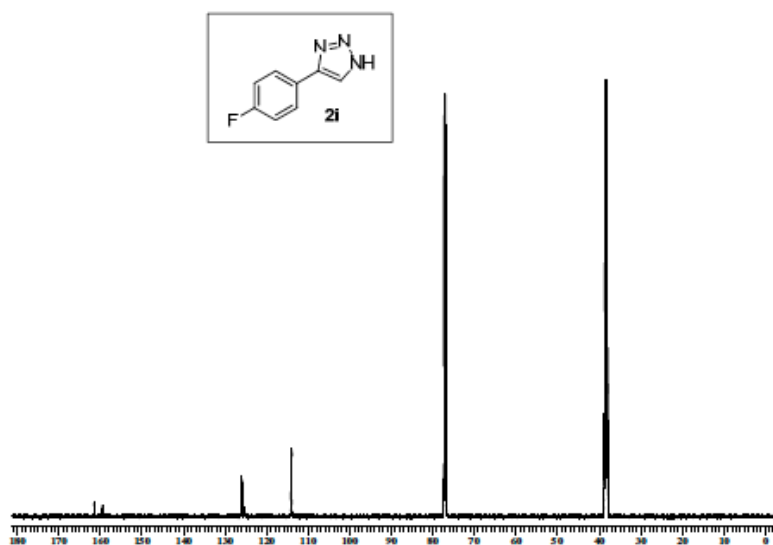


Figure S18. ^{13}C NMR spectrum (125 MHz, $\text{DMSO-}d_6 + \text{CDCl}_3$) of compound **2i**.

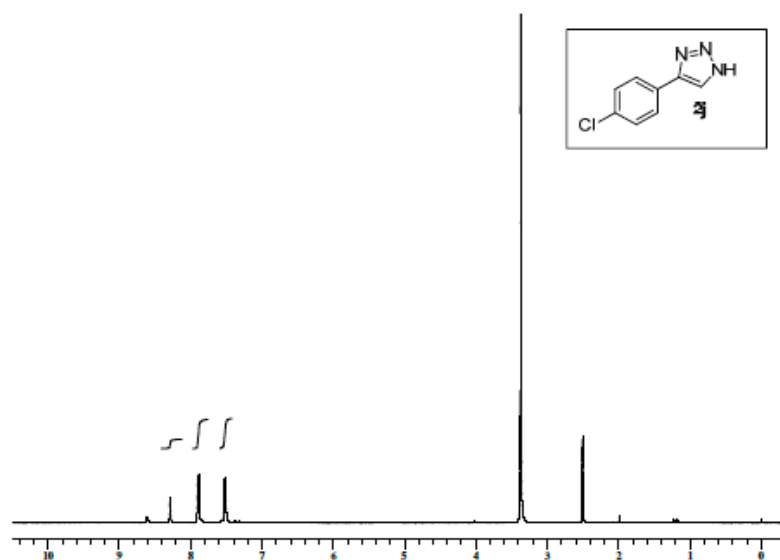


Figure S19. ^1H NMR spectrum (500 MHz, $\text{DMSO-}d_6 + \text{CDCl}_3$) of compound **2j**.

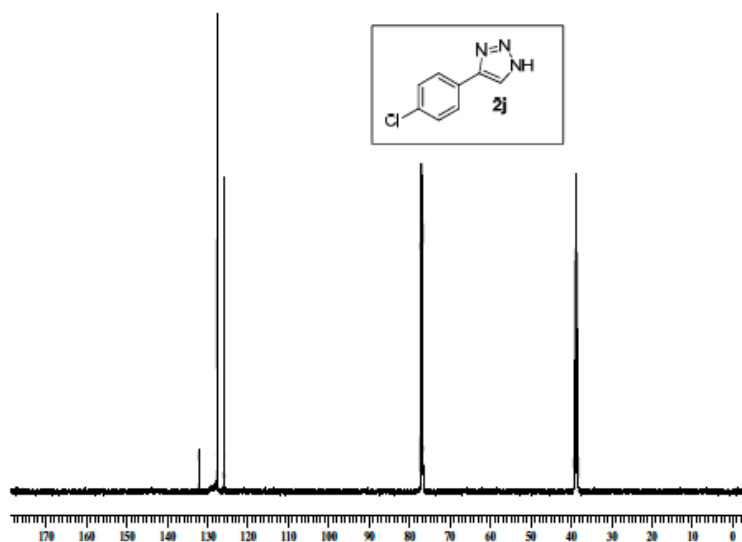


Figure S20. ¹³C NMR spectrum (125 MHz, DMSO-*d*₆ + CDCl₃) of compound **2j**.

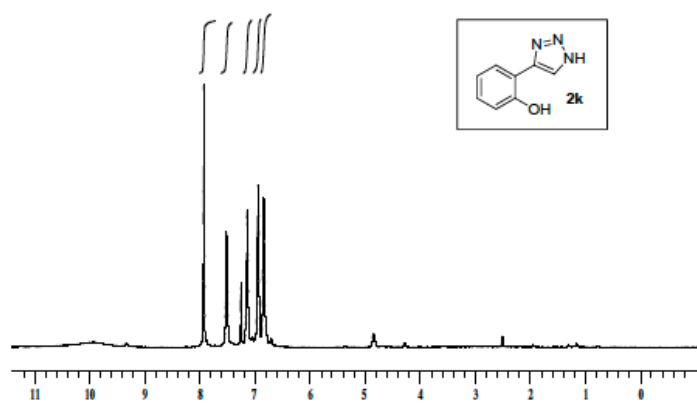


Figure S21. ¹H NMR spectrum (500 MHz, DMSO-*d*₆ + CDCl₃) of compound **2k**.

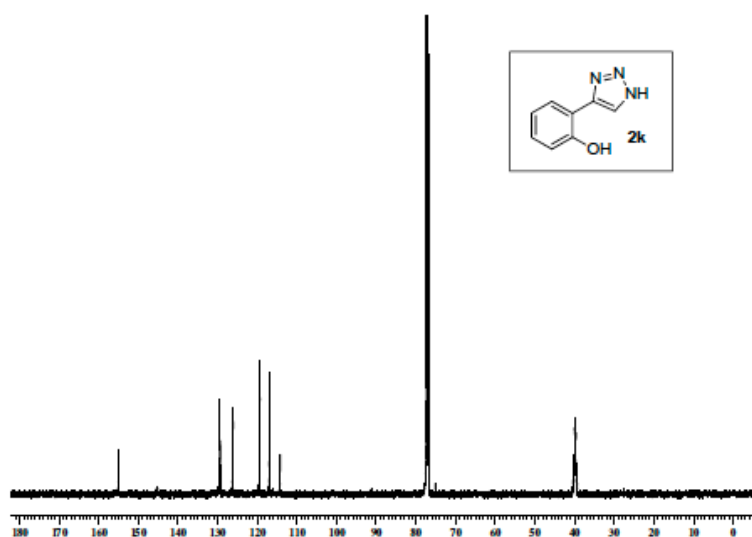


Figure S22. ¹³C NMR spectrum (125 MHz, DMSO-*d*₆ + CDCl₃) of compound **2k**.

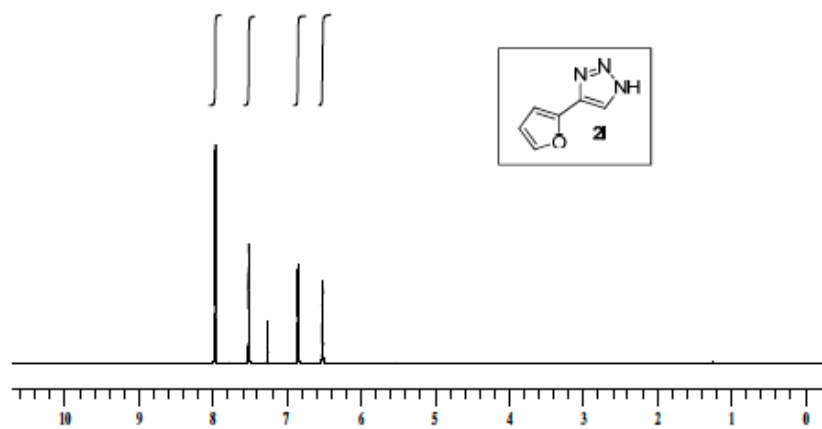


Figure S23. ¹H NMR spectrum (500 MHz, CDCl₃) of compound **2l**.

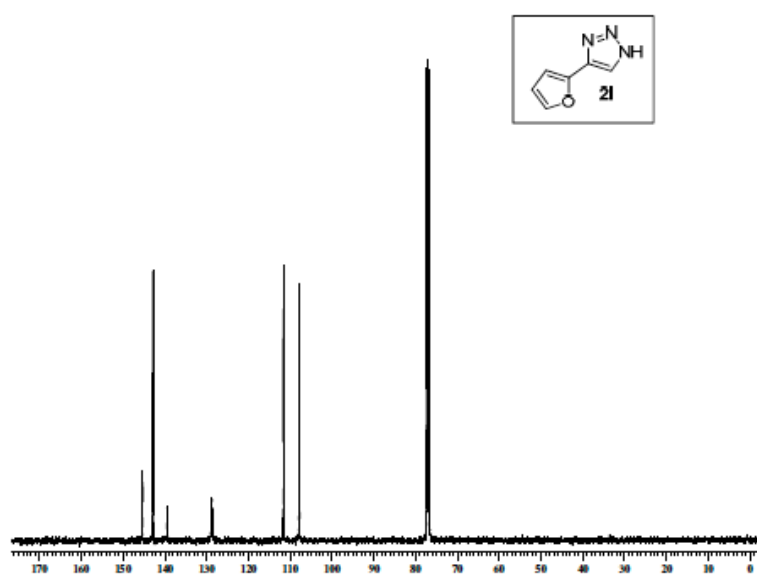


Figure S24. ¹³C NMR spectrum (125 MHz, CDCl₃) of compound **2l**.

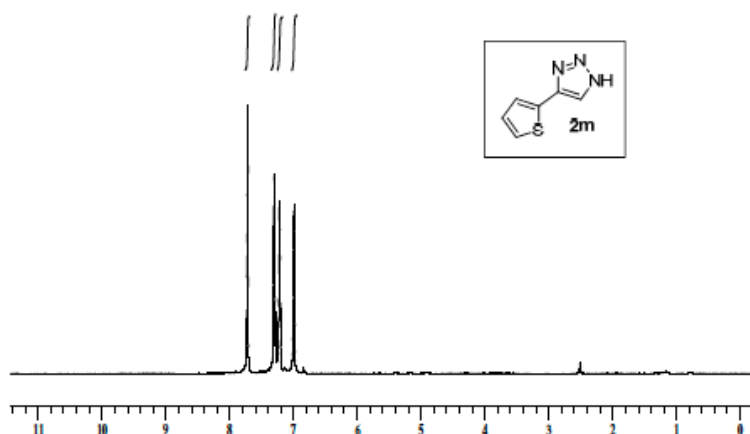


Figure S25. ¹H NMR spectrum (500 MHz, DMSO-*d*₆ + CDCl₃) of compound **2m**.

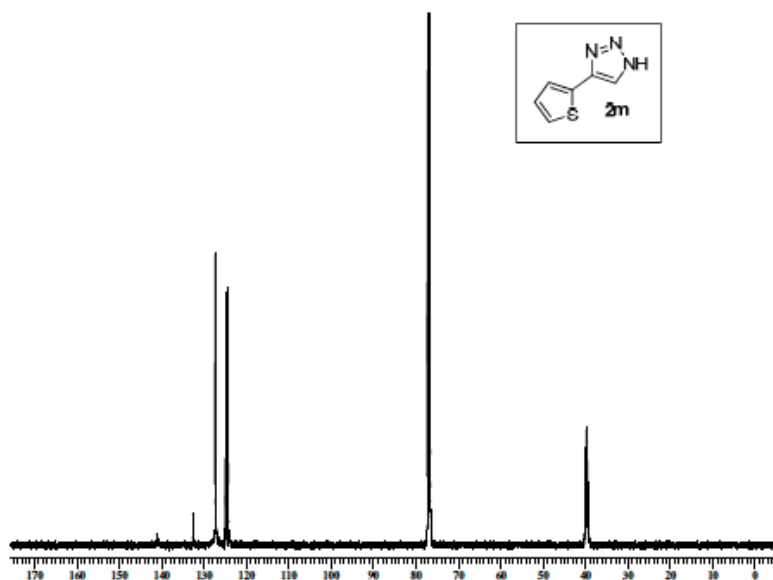


Figure S26. ^{13}C NMR spectrum (125 MHz, $\text{DMSO-}d_6 + \text{CDCl}_3$) of compound **2m**.

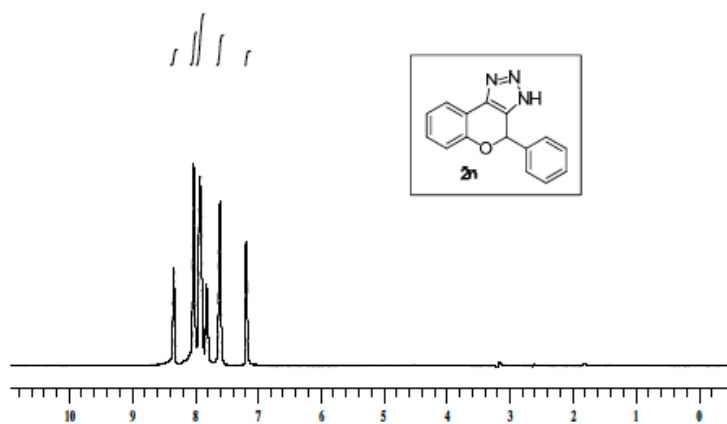


Figure S27. ^1H NMR spectrum (500 MHz, $\text{DMSO-}d_6 + \text{CDCl}_3$) of compound **2n**.

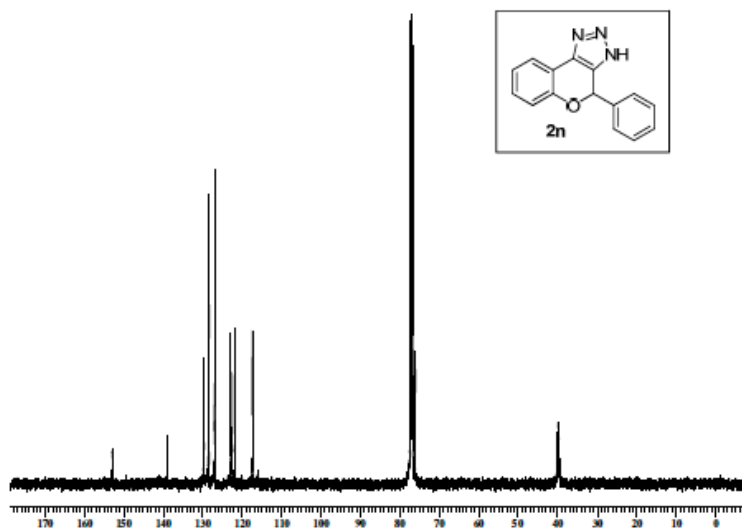


Figure S28. ^{13}C NMR spectrum (125 MHz, $\text{DMSO-}d_6 + \text{CDCl}_3$) of compound **2n**.

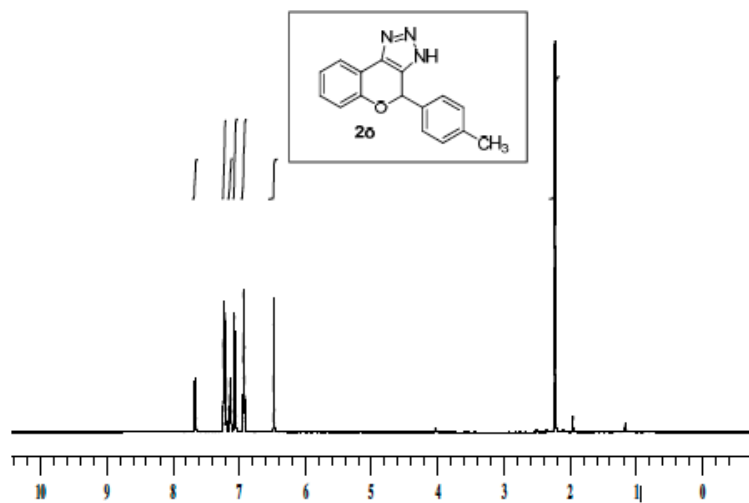


Figure S29. ^1H NMR spectrum (500 MHz, $\text{DMSO-}d_6 + \text{CDCl}_3$) of compound **2o**.

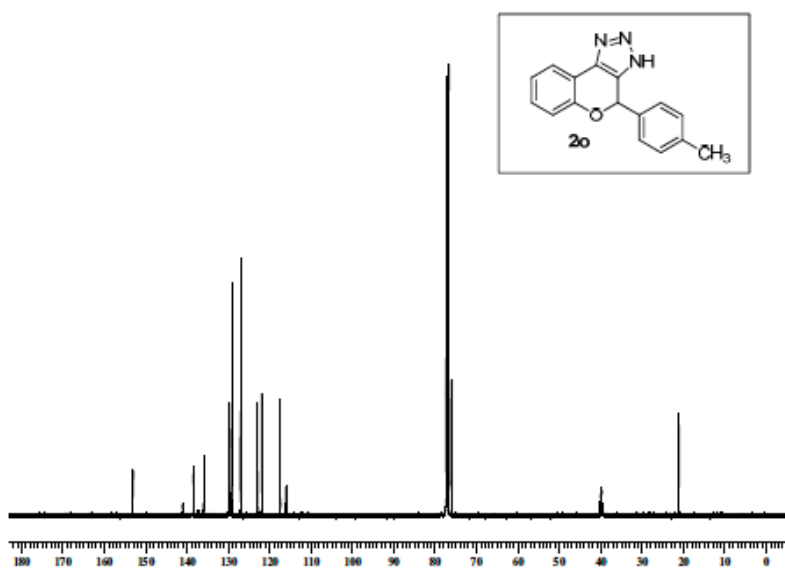


Figure S30. ^{13}C NMR spectrum (125 MHz, $\text{DMSO-}d_6 + \text{CDCl}_3$) of compound **2o**.

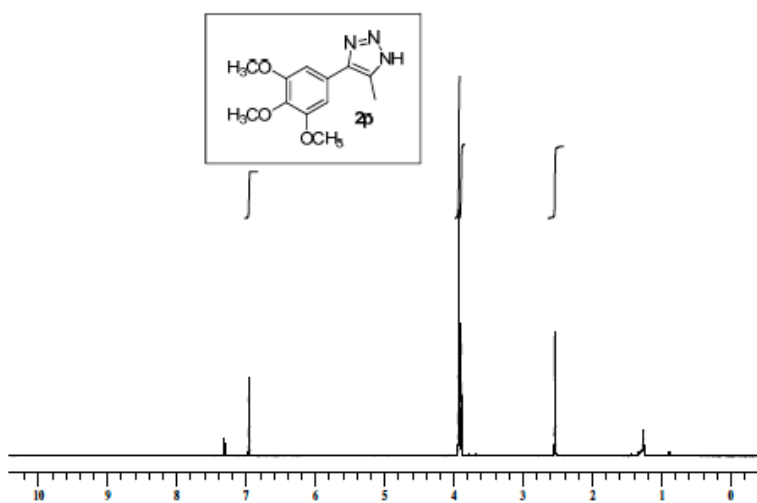


Figure S31. ^1H NMR spectrum (500 MHz, $\text{DMSO-}d_6 + \text{CDCl}_3$) of compound **2p**.

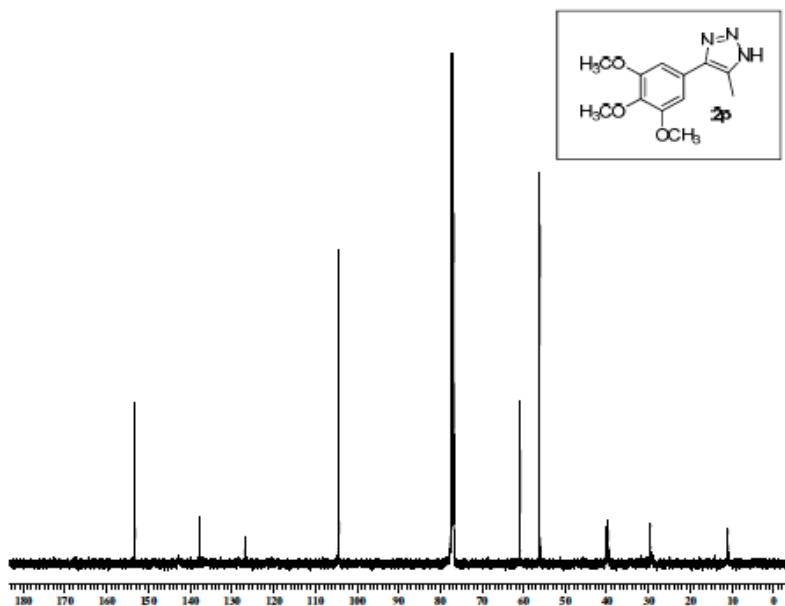


Figure S32. ^{13}C NMR spectrum (125 MHz, $\text{DMSO-}d_6 + \text{CDCl}_3$) of compound **2p**.

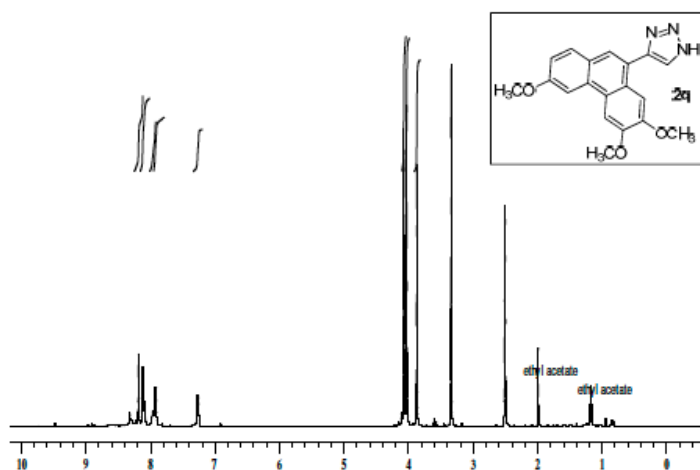


Figure S33. ^1H NMR spectrum (500 MHz, $\text{DMSO-}d_6$) of compound **2q**.

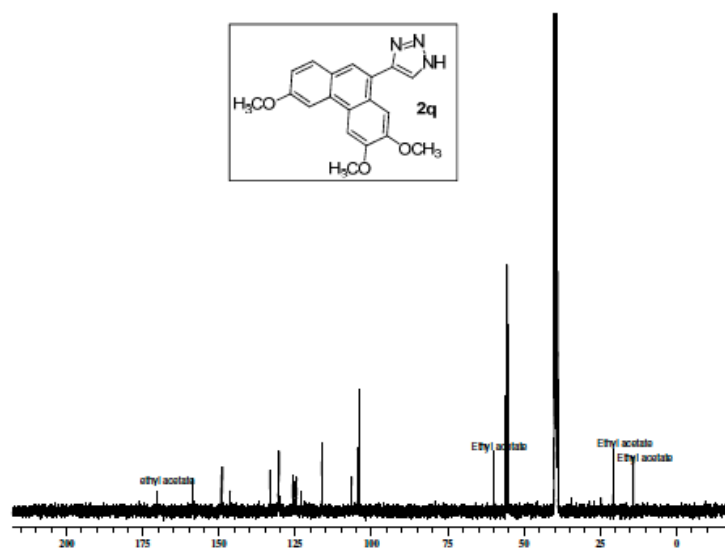


Figure S34. ^{13}C NMR spectrum (125 MHz, $\text{DMSO-}d_6$) of compound **2q**.

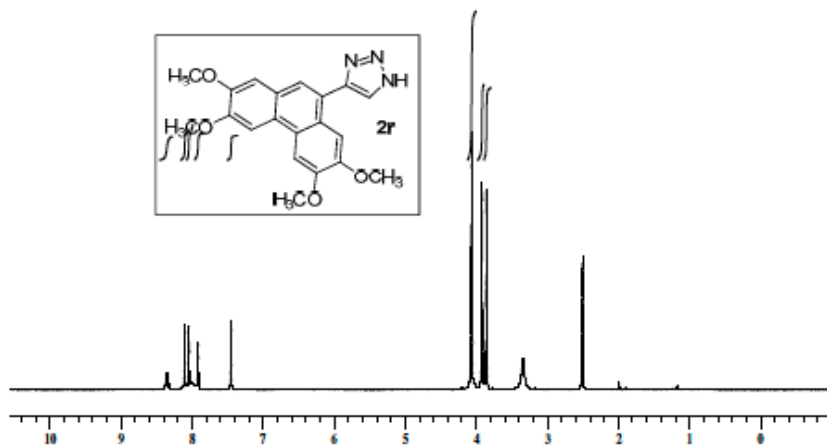


Figure S35. ^1H NMR spectrum (500 MHz, $\text{DMSO-}d_6$) of compound **2r**.

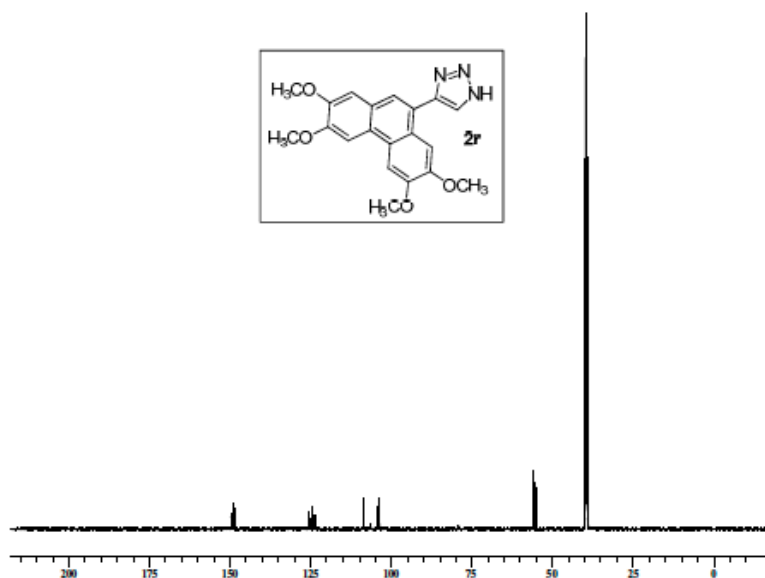


Figure S36. ^{13}C NMR spectrum (125 MHz, $\text{DMSO-}d_6$) of compound **2r**.

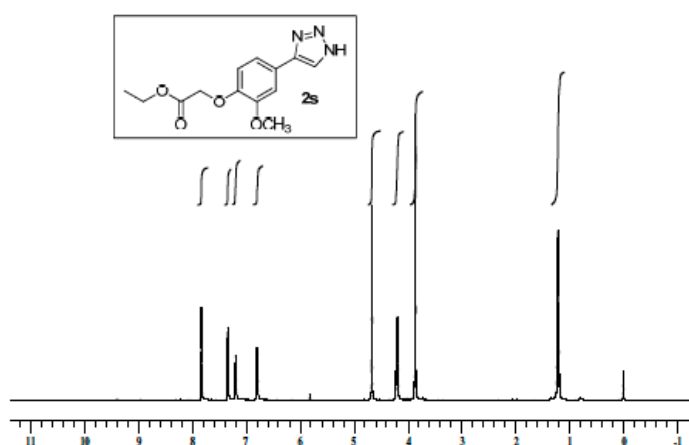


Figure S37. ^1H NMR spectrum (500 MHz, CDCl_3) of compound **2s**.

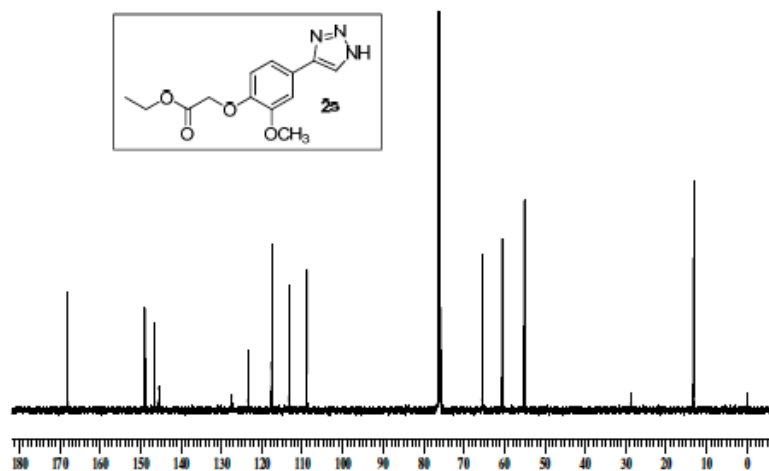


Figure S38. ^{13}C NMR spectrum (125 MHz, CDCl_3) of compound **2s**.

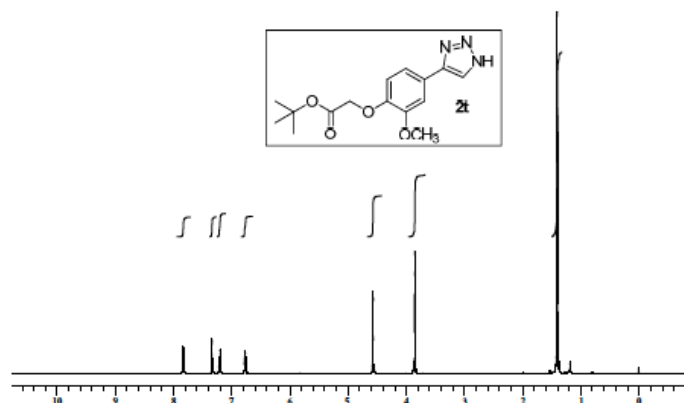


Figure S39. ^1H NMR spectrum (500 MHz, CDCl_3) of compound **2t**.

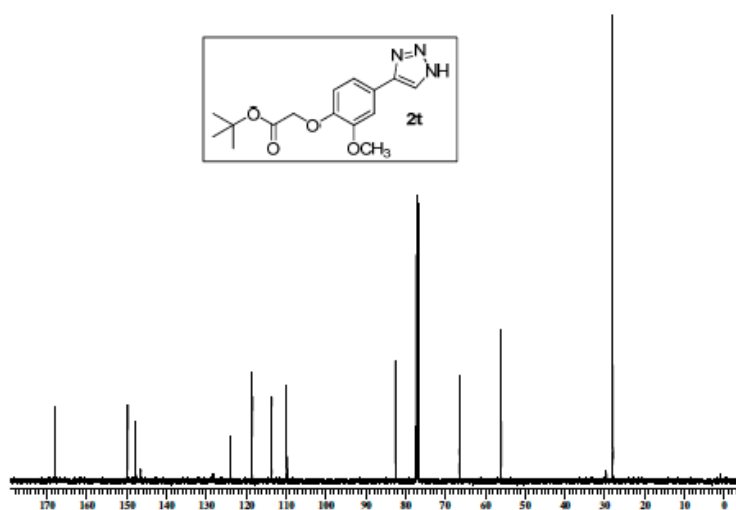


Figure S40. ^{13}C NMR spectrum (125 MHz, CDCl_3) of compound **2t**.