

Supporting Information for:

**Solvent- and catalyst-free, quantitative protection of
hydroxyl, thiol, carboxylic acid, amide and heterocyclic
amino functional groups**

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Tetrahydro-2H-pyran-2-yl benzoate. ^1H NMR (400 MHz, CDCl_3): δ 8.08 (dd, 2H, $^3J = 7.0$ Hz, $^3J = 1.4$ Hz, CH-phenyl), 7.56 (t, 1H, $^3J = 7.3$ Hz, CH-phenyl), 7.44 (t, 2H, $^3J = 8.0$ Hz, CH-phenyl), 6.25 (m, 1H, CH-THP), 3.96–4.02 (m, 1H, CH_2O -THP), 3.73–3.77 (m, 1H, CH_2O -THP), 1.53–1.92 (m, 6H, CH_2 -THP) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ 165.2, 133.2, 130.4, 129.8, 128.5, 93.2, 63.3, 29.4, 25.1, 18.7 ppm.

Tetrahydro-2H-pyran-2-yl stearate. ^1H NMR (400 MHz, CDCl_3): δ 5.95 (s, 1H, CH-THP), 3.85–3.91 (m, 1H, CH_2O -THP), 3.64–3.68 (m, 1H, CH_2O -THP), 2.33 (t, 2H, $^3J = 7.6$ Hz, $\text{C}(\text{O})\text{CH}_2$), 1.75–1.85 (m, 2H, $\text{C}(\text{O})\text{CH}_2\text{CH}_2$), 1.50–1.70 (m, 6H, CH_2 -THP), 1.21–1.28 (m, 28H, $\text{C}(\text{O})\text{CH}_2\text{CH}_2(\text{CH}_2)_{14}\text{CH}_3$), 0.86 (t, 3H, $^3J = 7.0$ Hz, $\text{C}(\text{O})\text{CH}_2\text{CH}_2(\text{CH}_2)_{14}\text{CH}_3$) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ 172.7, 92.5, 63.4, 34.6, 32.0, 29.7, 29.6, 29.5, 29.4, 29.35, 29.31, 29.21, 25.0, 24.9, 22.7, 18.8, 14.2 ppm. HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{23}\text{H}_{44}\text{NaO}_3$ 391.3182; found 391.3176.

2-(Phenylthio)tetrahydro-2H-pyran. ^1H NMR (400 MHz, CDCl_3): δ 7.48 (d, 2H, $^3J = 7.2$ Hz, CH-phenyl), 7.20–7.30 (m, 3H, CH-phenyl), 5.21 (t, 1H, $^3J = 5.2$ Hz, CH-THP), 4.16–4.20 (m, 1H, CH_2O -THP), 3.57–3.60 (m, 1H, CH_2O -THP), 2.00–2.05 (m, 1H, CH_2 -THP), 1.81–1.86 (m, 2H, CH_2 -THP), 1.62 (m, 3H, CH_2 -THP) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ 135.5, 130.8, 128.8, 126.7, 85.2, 64.5, 31.6, 25.6, 21.7 ppm.

1(2)-(Tetrahydro-2H-pyran-2-yl)-1(2)H-1,2,3-benzotriazole. ^1H NMR (400 MHz, CDCl_3): δ 8.05 (d, 1H, $^3J = 8.4$ Hz, CH-phenyl), 7.87–7.90 (m, 2H, CH-phenyl), 7.73 (d, 1H, $^3J = 8.8$ Hz, CH-phenyl), 7.47 (t, 1H, $^3J = 6.8$ Hz, CH-phenyl), 7.34–7.44 (m, 2H, CH-phenyl), 6.02 (dd, 1H, $^3J = 8.4$ Hz, $^3J = 3.2$ Hz, CH-THP), 3.90–3.95 (m, 1H, CH_2O -THP), 3.74–3.82 (m, 1H, CH_2O -THP), 2.56–2.65 (m, 1H, CH_2 -THP), 2.16–2.24 (m, 2H, CH_2 -THP), 1.71–1.88 (m, 3H, CH_2 -THP) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ 146.3, 144.2, 132.5, 127.6, 127.0, 124.4, 119.8, 118.6, 111.2, 90.8, 85.8, 67.6, 67.0, 30.0, 29.4, 24.9, 24.8, 21.7, 21.5 ppm.

1(2)-(Tetrahydro-2H-pyran-2-yl)-1(2)H-1,2,3-triazole. ^1H NMR (400 MHz, CDCl_3): δ 7.69 (s, 1H, 4-*H*-triazole), 7.62 (s, 1H, 5-*H*-triazole), 7.58 (s, 2H, 4-*H*-triazole and 5-*H*-triazole), 5.62–5.66 (m, 1H, CH-THP), 3.88–3.94 (m, 1H, CH_2O -THP), 3.61–3.67 (m, 1H, CH_2O -THP), 2.28–2.40 (m, 1H, CH_2 -THP), 1.92–2.10 (m, 2H, CH_2 -THP), 1.53–1.68 (m, 3H, CH_2 -THP) ppm.

4-Nitro-1-(tetrahydro-2H-pyran-2-yl)-1H-pyrazole. ^1H NMR (400 MHz, CDCl_3): δ 8.35 (s, 1H, 5-*H*-pz), 8.08 (s, 1H, 3-*H*-pz), 5.39 (dd, 1H, $^3J = 9.2$ Hz, $^3J = 2.6$ Hz, CH-THP), 4.05–4.09 (m, 1H, CH_2O -THP), 3.69–3.75 (m, 1H, CH_2O -THP), 2.11–2.17 (m, 1H, CH_2 -THP), 1.64–2.04 (m, 5H, CH_2 -THP) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ 135.6, 127.1, 88.5, 67.9, 30.7, 24.8, 21.8 ppm. HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_8\text{H}_{11}\text{N}_3\text{NaO}_3$ 220.0692; found 220.0711.

2-Phenoxytetrahydro-2H-pyran. ^1H NMR (400 MHz, CDCl_3): δ 7.27–7.31 (m, 2H, CH-phenyl), 7.06–7.08 (m, 2H, CH-phenyl), 5.44 (t, 1H, $^3J = 3.3$ Hz, CH-THP), 3.91–3.94 (m, 1H, CH_2O -THP), 3.60–3.63 (m, 1H, CH_2O -THP), 1.99–2.03 (m, 1H, CH_2 -THP), 1.80–1.90 (m, 2H, CH_2 -THP), 1.50–1.78 (m, 3H, CH_2 -THP) ppm. ^{13}C NMR (100 MHz, CDCl_3): 157.1, 129.2, 121.4, 116.4, 96.2, 61.8, 30.4, 25.3, 18.8 ppm.

1(2)-(Tetrahydro-2H-pyran-2-yl)-1(2)H-1,2,4-triazole. ^1H NMR (400 MHz, CDCl_3): 8.21 (s, 1H, 5-*H*-triazole), 8.08 (s, 2H, 3-*H*-triazole and 5-*H*-triazole), 7.87 (s, 1H, 3-*H*-triazole), 5.38

(dd, 1H, $^3J = 8.8$ Hz, $^3J = 4.4$ Hz, CH-THP), 3.95–3.99 (m, 1H, CH₂O-THP), 3.58–3.65 (m, 1H, CH₂O-THP), 1.88–2.05 (m, 3H, CH₂-THP), 1.52–1.64 (m, 3H, CH₂-THP) ppm.

2-(Decylthio)tetrahydro-2H-pyran. ¹H NMR (400 MHz, CDCl₃): δ 4.80–4.83 (m, 1H, CH-THP), 4.04–4.09 (m, 1H, CH₂O-THP), 3.45–3.51 (m, 1H, CH₂O-THP), 2.43–2.7 (m, 2H, SCH₂), 1.52–1.95 (m, 6H, CH₂-THP), 1.20–1.40 (m, 16H, CH₂(CH₂)₁₄CH₂), 0.85 (t, 3H, $^3J = 6.6$, CH₃) ppm. ¹³C NMR (100 MHz, CDCl₃): δ 94.7, 64.7, 31.9, 31.5, 30.4, 30.0, 29.64, 29.61, 29.40, 29.3, 29, 25.7, 22.7, 21.9, 14.2 ppm.

4-Chloro-1-(tetrahydro-2H-pyran-2-yl)-1H-pyrazole. ¹H NMR (400 MHz, CDCl₃): δ 7.59 (s, 1H, 5-H-pz), 7.46 (s, 1H, 3-H-pz), 5.29–5.33 (m, 1H, CH-THP), 4.01–4.04 (m, 1H, CH₂O-THP), 3.64–3.71 (m, 1H, CH₂O-THP), 2.00–2.09 (m, 3H, CH₂-THP), 1.58–1.69 (m, 3H, CH₂-THP) ppm. ¹³C NMR (100 MHz, CDCl₃): δ 137.9, 125.8, 110.7, 88.0, 67.7, 30.3, 24.7, 22.2 ppm. HRMS (ESI-TOF) *m/z*: [M + Na]⁺ calcd for C₈H₁₁ClN₂NaO 209.0452; found 209.0454.

4-Bromo-1-(tetrahydro-2H-pyran-2-yl)-1H-pyrazole. ¹H NMR (400 MHz, CDCl₃): δ 7.61 (s, 1H, 3-H-pz), 7.48 (s, 1H, 5-H-pz), 5.31–5.33 (m, 1H, CH-THP), 3.99–4.02 (m, 1H, CH₂O-THP), 3.64–3.69 (m, 1H, CH₂O-THP), 1.99–2.09 (m, 3H, CH₂-THP), 1.59–1.65 (m, 3H, CH₂-THP) ppm.

1-(Tetrahydro-2H-pyran-2-yl)-1H-benzimidazole. ¹H NMR (400 MHz, CDCl₃): δ 8.06 (s, 1H, CH-imidazole), 7.77–7.79 (m, 1H, CH-phenyl), 7.50–7.52 (m, 1H, CH-phenyl), 7.26–7.31 (m, 2H, CH-phenyl), 5.48 (dd, 1H, $^3J = 9.6$ Hz, $^3J = 2.0$ Hz, CH-THP), 4.10–4.12 (m, 1H, CH₂O-THP), 3.71–3.77 (m, 1H, CH₂O-THP), 2.08–2.21 (m, 3H, CH₂-THP), 1.68–1.81 (m, 3H, CH₂-THP) ppm.

3,5-Diphenyl-1-(tetrahydro-2H-pyran-2-yl)-1H-pyrazole. ¹H NMR (400 MHz, CDCl₃): δ 7.88 (d, 2H, $^3J = 8.4$ Hz, CH-phenyl), 7.58 (d, 2H, $^3J = 8.0$ Hz, CH-phenyl), 7.27–7.5 (m, 6H, CH-phenyl), 6.64 (s, 1H, 4-H-pz), 5.22 (dd, 1H, $^3J = 10.2$ Hz, $^3J = 2.2$ Hz, CH-THP), 4.16–4.18 (m, 1H, CH₂O-THP), 3.62 (t, 1H, $^3J = 11.7$ Hz, CH₂O-THP), 2.65–2.74 (m, 1H, CH₂-THP), 2.07–2.10 (m, 1H, CH₂-THP), 1.54–1.88 (m, 4H, CH₂-THP) ppm. ¹³C NMR (100 MHz, CDCl₃): δ 151.2, 145.6, 133.5, 130.6, 129.1, 128.8, 127.8, 126.0, 103.9, 84.5, 67.8, 29.8, 24.9, 23.1 ppm. HRMS (ESI-TOF) *m/z*: [M + Na]⁺ calcd for C₂₀H₂₀N₂NaO 327.1467; found 327.1478.

1(2)-(Tetrahydro-2H-pyran-2-yl)-1(2)H-indazole. ¹H NMR (400 MHz, CDCl₃): δ 8.15 (s, 1H, 5-H-pz), 8.03 (s, 1H, 3-H-pz), 7.71–7.73 (m, 1H, CH-phenyl), 7.58–7.6 (m, 1H, CH-phenyl), 7.37–7.41 (m, 1H, CH-phenyl), 5.72 (dd, 1H, $^3J = 9.6$ Hz, $^3J = 2.4$ Hz, CH-THP), 4.01–4.05 (m, 1H, CH₂O-THP), 3.72–3.78 (m, 1H, CH₂O-THP), 2.54–2.63 (m, 1H, CH₂-THP), 2.06–2.18 (m, 2H, CH₂-THP), 1.50–1.82 (m, 3H, CH₂-THP) ppm.

1-(Tetrahydro-2H-pyran-2-yl)-1H-pyrazole. ¹H NMR (400 MHz, CDCl₃): 7.57 (d, 1H, $^3J = 2.4$ Hz, 5-H-pz), 7.52 (d, 1H, $^3J = 1.6$ Hz, 3-H-pz), 6.26 (t, 1H, $^3J = 2$ Hz, 4-H-pz), 5.36 (dd, 1H, $^3J = 9.6$ Hz, $^3J = 2.4$ Hz, CH-THP), 4.00–4.04 (m, 1H, CH₂O-THP), 3.63–3.69 (m, 1H, CH₂O-THP), 1.97–2.13 (m, 3H, CH₂-THP), 1.48–1.69 (m, 3H, CH₂-THP) ppm.

1-(Tetrahydro-2H-pyran-2-yl)-1H-imidazole. ¹H NMR (400 MHz, CDCl₃): δ 7.58 (s, 1H, 2-H-imidazole), 6.96–7.00 (m, 2H, 4-H-imidazole, 5-H-imidazole), 5.12 (dd, 1H, $^3J = 2.4$ Hz, $^3J = 9.6$ Hz, CH-THP), 3.94–3.97 (m, 1H, CH₂O-THP), 3.54–3.60 (m, 1H, CH₂O-THP), 1.75–1.93

(m, 3H, CH₂-THP), 1.48–1.66 (m, 3H, CH₂-THP) ppm. ¹³C NMR (100 MHz, CDCl₃): δ 135.6, 129.1, 117.0, 100.8, 67.9, 31.5, 24.8, 22.5 ppm.

3(5)-Methyl-1-(tetrahydro-2H-pyran-2-yl)-1H-pyrazole. ¹H NMR (400 MHz, CDCl₃): δ 7.45 (d, 1H, ³J = 2.2 Hz, 5-*H*-pz), 7.42 (s, 1H, 3-*H*-pz), 6.05 (d, 1H, ³J = 2.2 Hz, 4-*H*-pz), 6.02 (d, 1H, ³J = 0.8 Hz, 4-*H*-pz), 5.25 (td, 1H, ³J = 7.7 Hz, ³J = 2.2 Hz, CH-THP) 4.00–4.07 (m, 1H, CH₂O-THP), 3.63–3.69 (m, 1H, CH₂O-THP), 2.41–2.50 (m, 1H, CH₂-THP), 2.32 (s, 3H, CH₃), 2.27 (s, 3H, CH₃), 2.05–2.12 (m, 1H, CH₂-THP), 1.92–2.03 (m, 2H, CH₂-THP), 1.51–1.71 (m, 3H, CH₂-THP) ppm. ¹³C NMR (100 MHz, CDCl₃): δ 149.2, 139.1, 139.0, 128.4, 106.2, 105.8, 87.5, 84.4, 68.1, 67.8, 30.5, 29.4, 25.1, 25.0, 22.88, 22.82, 13.7, 10.98 ppm.

4-Octyl-1-(tetrahydro-2H-pyran-2-yl)-1H-pyrazole. ¹H NMR (400 MHz, CDCl₃): δ 7.36 (s, 2H, 3-*H*-pz and 5-*H*-pz), 5.30 (dd, 1H, ³J = 10.0 Hz, ³J = 2.4 Hz, CH-THP), 4.02–4.06 (m, 1H, CH₂O-THP), 3.63–3.70 (m, 1H, CH₂O-THP), 2.42 (t, 2H, ³J = 11.6 Hz, CH₂(CH₂)₆CH₃), 2.00–2.12 (m, 3H, CH₂-THP), 1.49–1.72 (m, 5H, CH₂-THP, CH₂CH₂(CH₂)₅CH₃), 1.24–1.30 (m, 10H, CH₂CH₂(CH₂)₅CH₃), 0.86 (t, 3H, ³J = 6.8 Hz, (CH₂)₇CH₃) ppm. ¹³C NMR (100 MHz, CDCl₃): δ 139.5, 125.6, 122.6, 87.6, 67.9, 32.0, 30.9, 30.5, 29.4, 25.1, 24.3, 22.8, 14.2 ppm. HRMS (ESI-TOF) *m/z*: [M + Na]⁺ calcd for C₁₆H₂₈N₂NaO 287.2093; found 287.2083.

3,5-Dimethyl-1-(tetrahydro-2H-pyran-2-yl)-1H-pyrazole. ¹H NMR (400 MHz, CDCl₃): δ 5.79 (s, 1H, 4-*H*-pz), 5.11 (dd, 1H, ³J = 10.0 Hz, ³J = 2.0 Hz, CH₂-THP), 3.99–4.04 (m, 1H, CH₂O-THP), 3.56–3.62 (td, 1H, ³J = 11.6 Hz, ³J = 2.8 Hz, CH₂O-THP), 2.36–2.46 (m, 1H, CH₂-THP), 2.24 (s, 3H, CH₃), 2.18 (s, 3H, CH₃), 2.01–2.05 (m, 1H, CH₂-THP), 1.84–1.89 (m, 1H, CH₂-THP), 1.49–1.72 (m, 3H, CH₂-THP) ppm. ¹³C NMR (100 MHz, CDCl₃): δ 148.4, 139.9, 106.3, 84.4, 68.1, 29.7, 25.1, 23.2, 13.7, 10.9 ppm. HRMS (ESI-TOF) *m/z*: [M + Na]⁺ calcd for C₁₀H₁₆N₂NaO 203.1154; found 203.1178.

N-(Tetrahydro-2H-pyran-2-yl)benzamide. ¹H NMR (400 MHz, CDCl₃): δ 7.76–7.81 (m, 2H, CH-phenyl), 7.41–7.53 (m, 3H, CH-phenyl), 6.60 (br, 1H, ³J = 7.3 Hz, NH), 5.30 (t, 1H, ³J = 8.4 Hz, CH-THP), 4.01 (d, 1H, ³J = 11.0 Hz, CH₂O-THP), 3.63–3.69 (m, 1H, CH₂O-THP), 1.41–1.95 (m, 6H, CH₂-THP) ppm. ¹³C NMR (100 MHz, CDCl₃): δ 166.8, 131.8, 129.2, 127.2, 127, 78.4, 66.2, 29.9, 22.0 ppm.

2-Butoxytetrahydro-2H-pyran. ¹H NMR (400 MHz, CDCl₃): δ 4.52 (m, 1H, CH-THP), 3.80–3.83 (m, 1H, CH₂O-THP), 3.65–3.75 (m, 1H, OCH₂), 3.45–3.51 (m, 1H, CH₂O-THP), 3.33–3.40 (m, 1H, OCH₂), 1.48–1.90 (m, 8H, CH₂-THP, OCH₂CH₂), 1.30–1.40 (m, 2H, CH₂CH₂CH₃), 0.88 (t, 3H, ³J = 7.7 Hz, CH₃) ppm. ¹³C NMR (100 MHz, CDCl₃): δ 98.9, 67.4, 62.3, 31.9, 30.8, 25.6, 19.7, 19.5, 14.0 ppm.

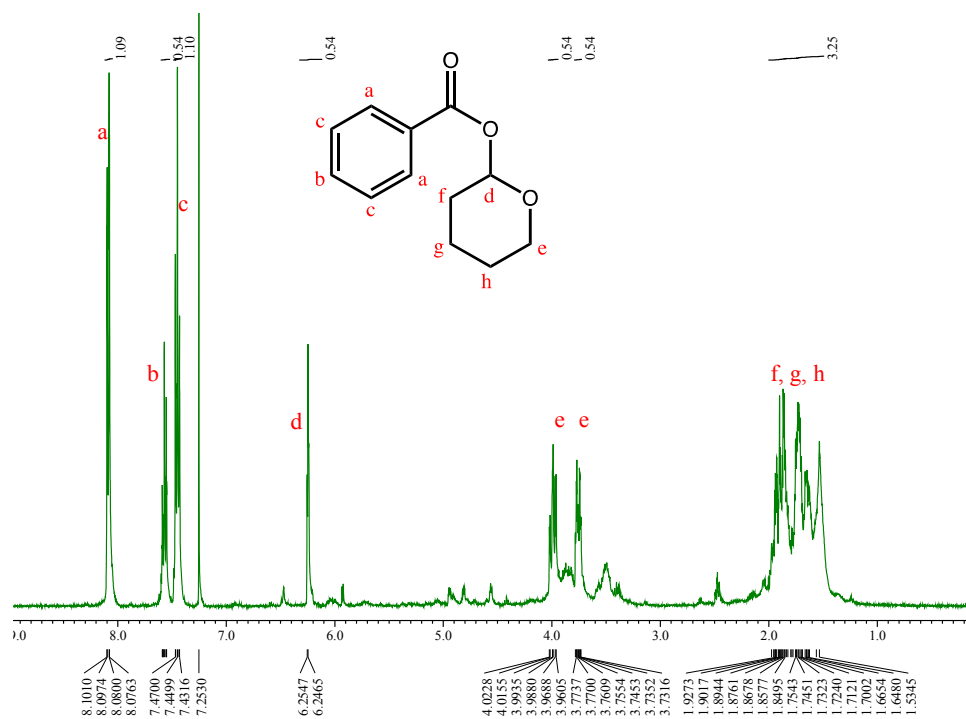


Figure S1. ¹H NMR spectrum of tetrahydro-2H-pyran-2-yl benzoate.

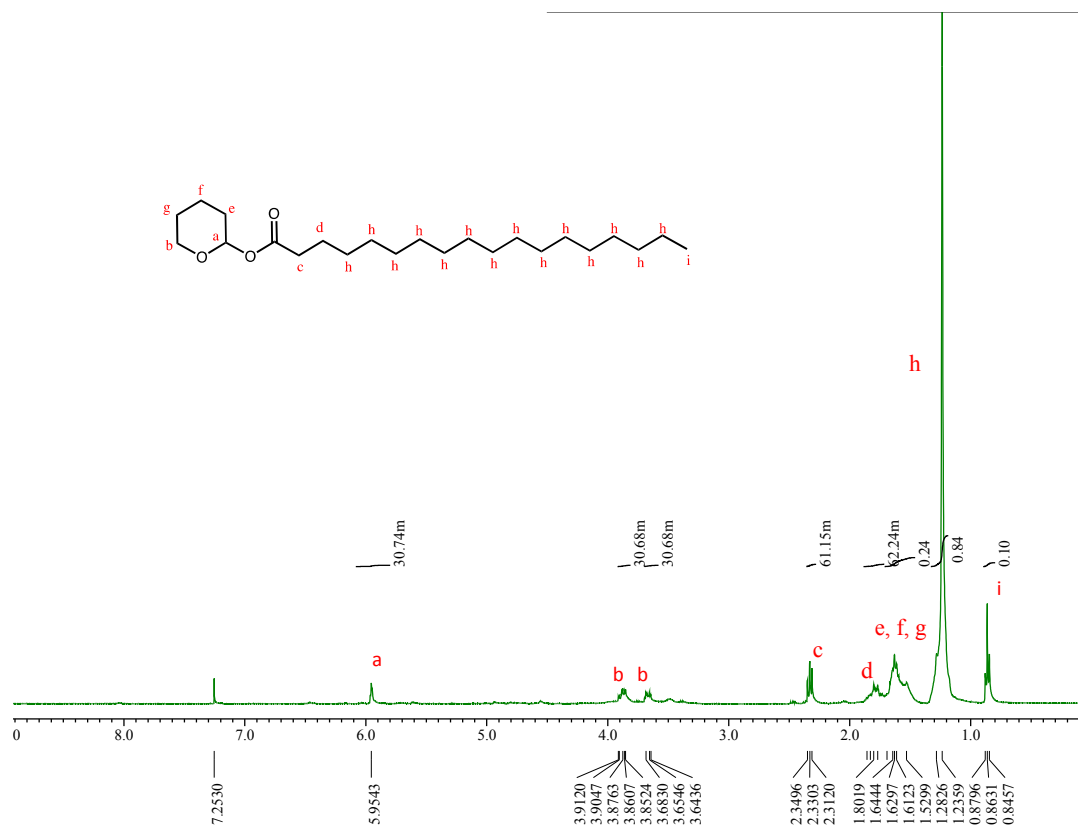


Figure S2. ¹H NMR spectrum of tetrahydro-2H-pyran-2-yl stearate.

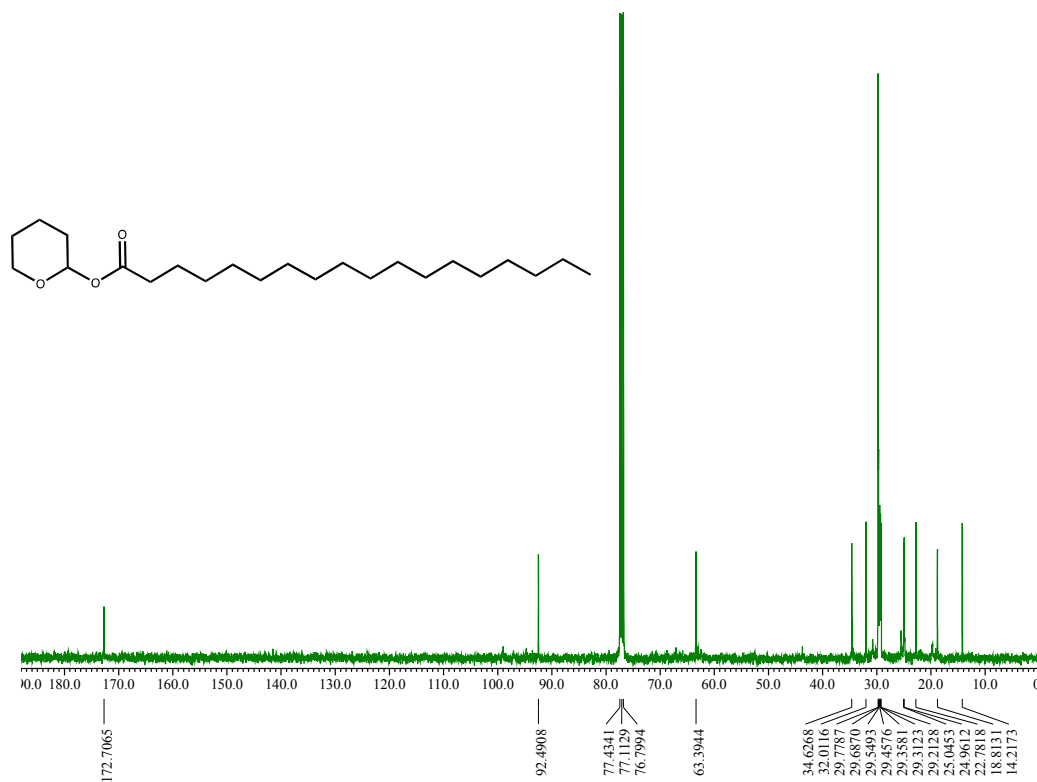


Figure S3. ¹³C NMR spectrum of tetrahydro-2*H*-pyran-2-yl stearate.

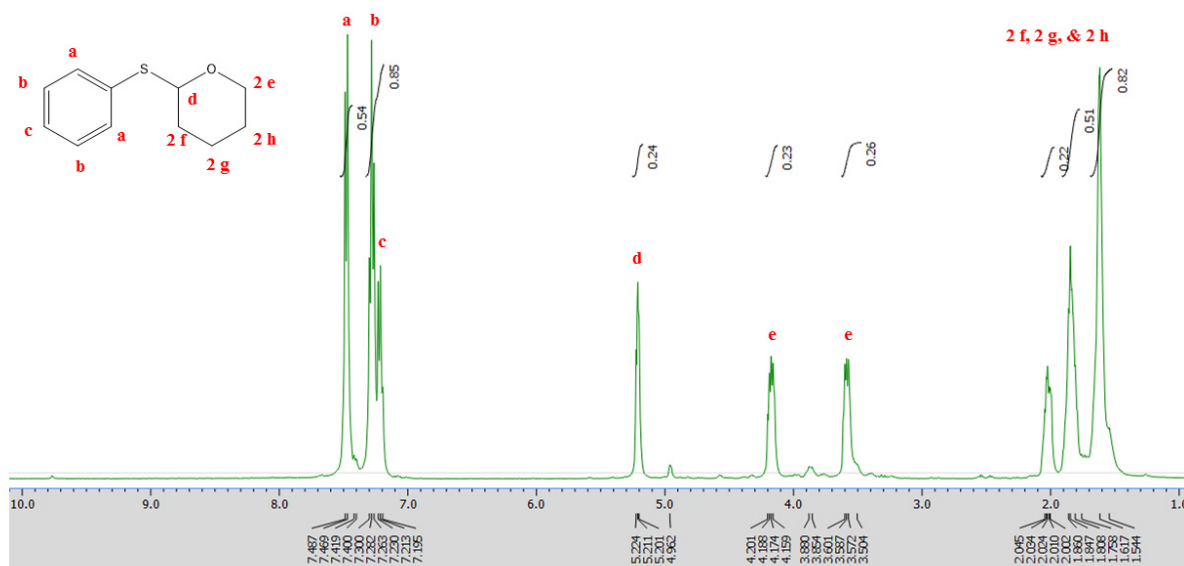


Figure S4. ¹H NMR spectrum of 2-(phenylthio)tetrahydro-2*H*-pyran.

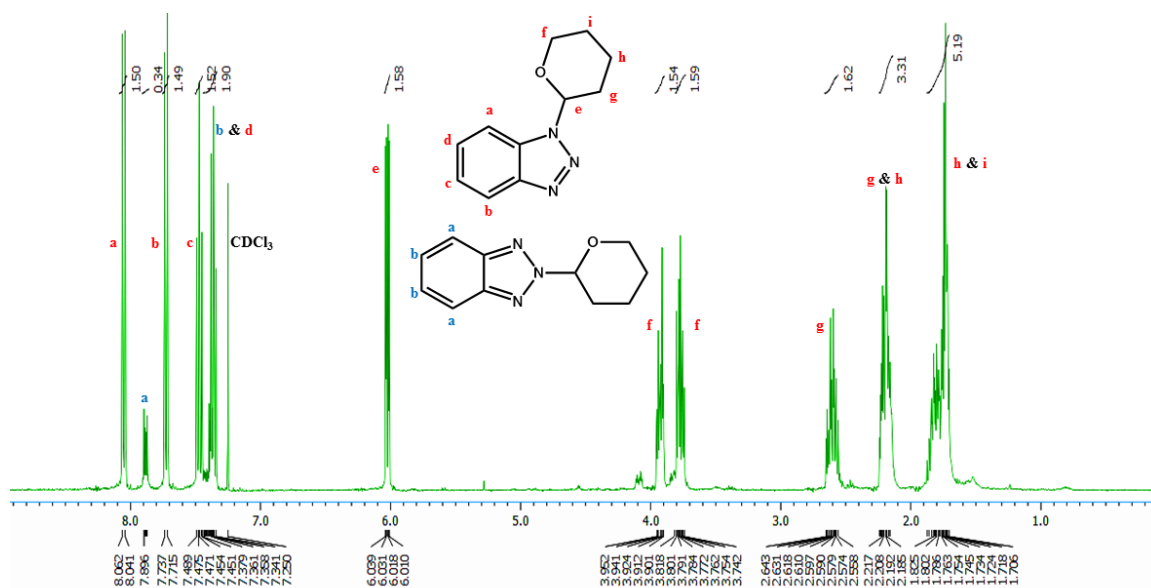


Figure S5. ¹H NMR spectrum of 1-(tetrahydro-2H-pyran-2-yl)-1H-1,2,3-benzotriazole (82%) and 2-(tetrahydro-2H-pyran-2-yl)-2H-1,2,3-benzotriazole (18%).

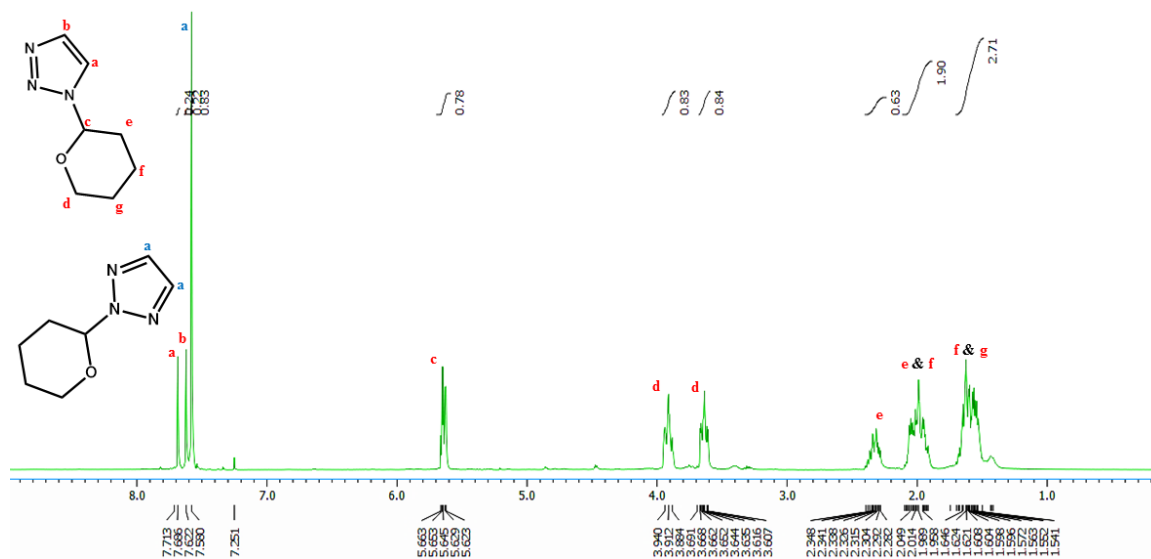


Figure S6. ¹H NMR spectrum of 1-(tetrahydro-2H-pyran-2-yl)-1H-1,2,3-triazole (22%) and 2-(tetrahydro-2H-pyran-2-yl)-2H-1,2,3-triazole (78%).

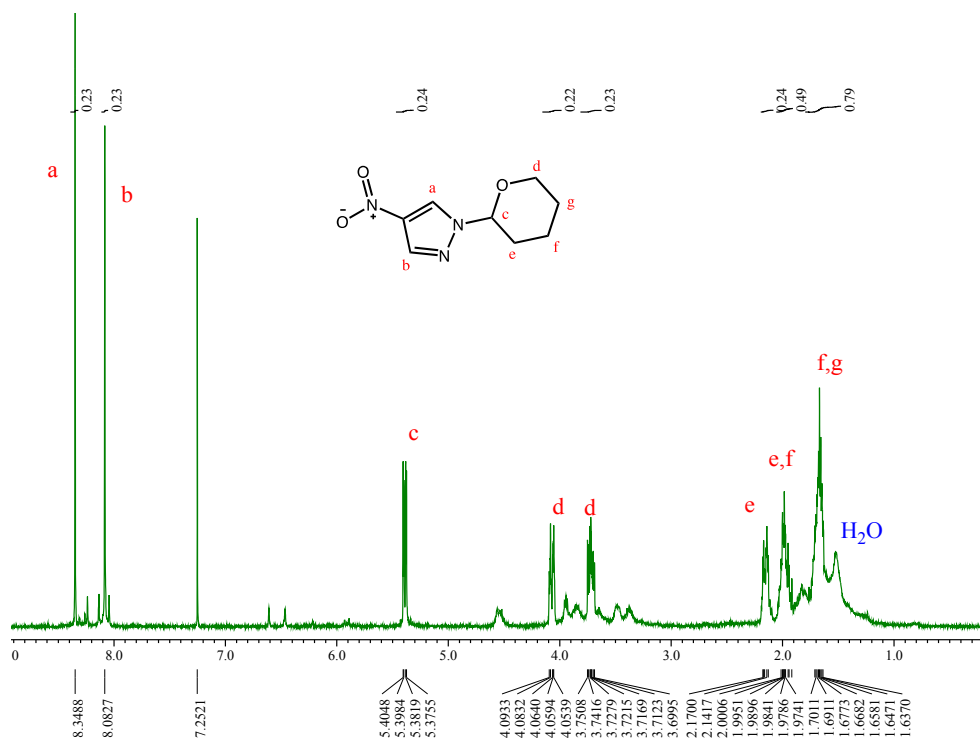


Figure S7. ¹H NMR spectrum of 4-nitro-1-(tetrahydro-2H-pyran-2-yl)-1H-pyrazole.

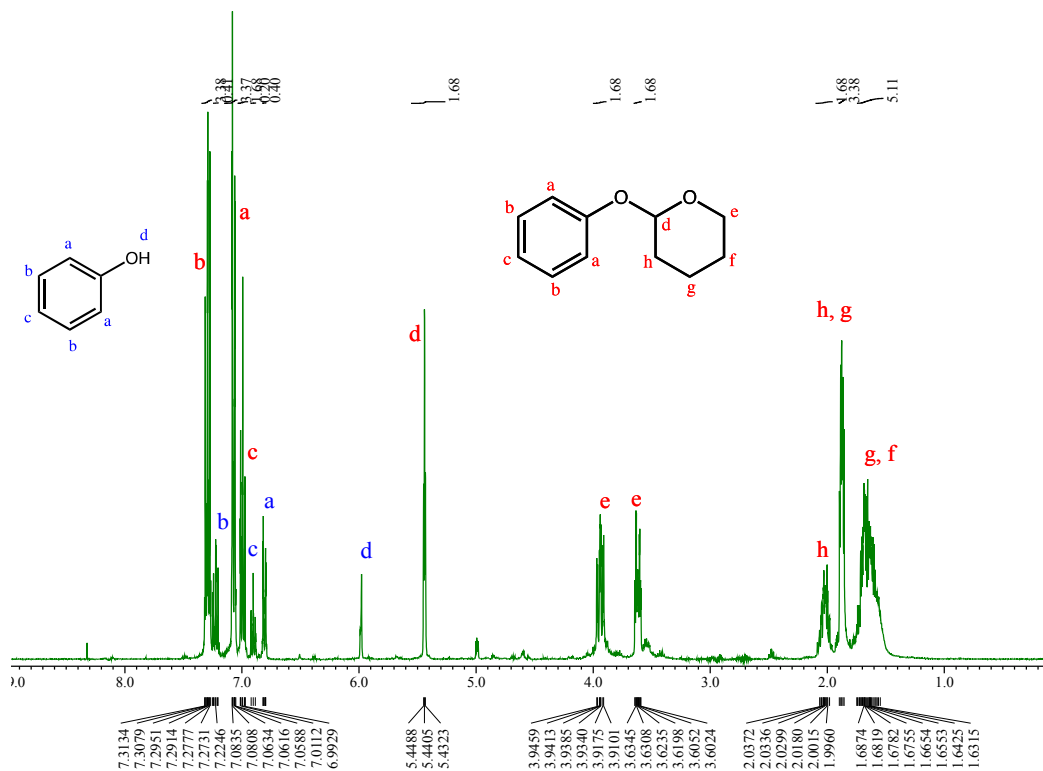


Figure S8. ¹H NMR spectrum of 2-phenoxytetrahydro-2H-pyran.

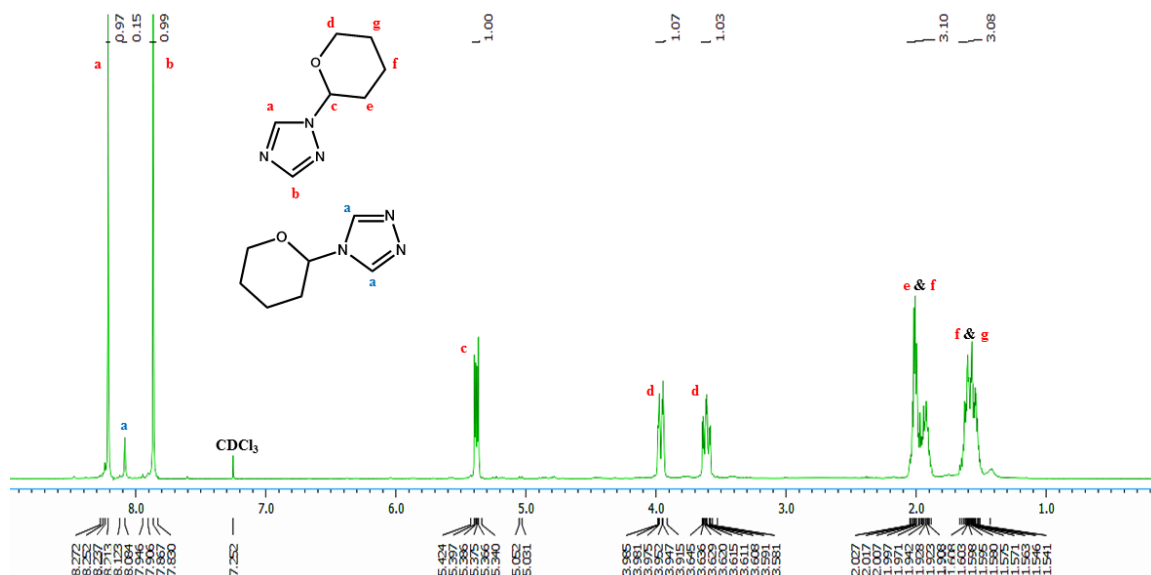


Figure S9. ^1H NMR spectrum of 1-(tetrahydro-2*H*-pyran-2-yl)-1*H*-1,2,4-triazole (87%) and 4-(tetrahydro-2*H*-pyran-2-yl)-4*H*-1,2,4-triazole (13%).

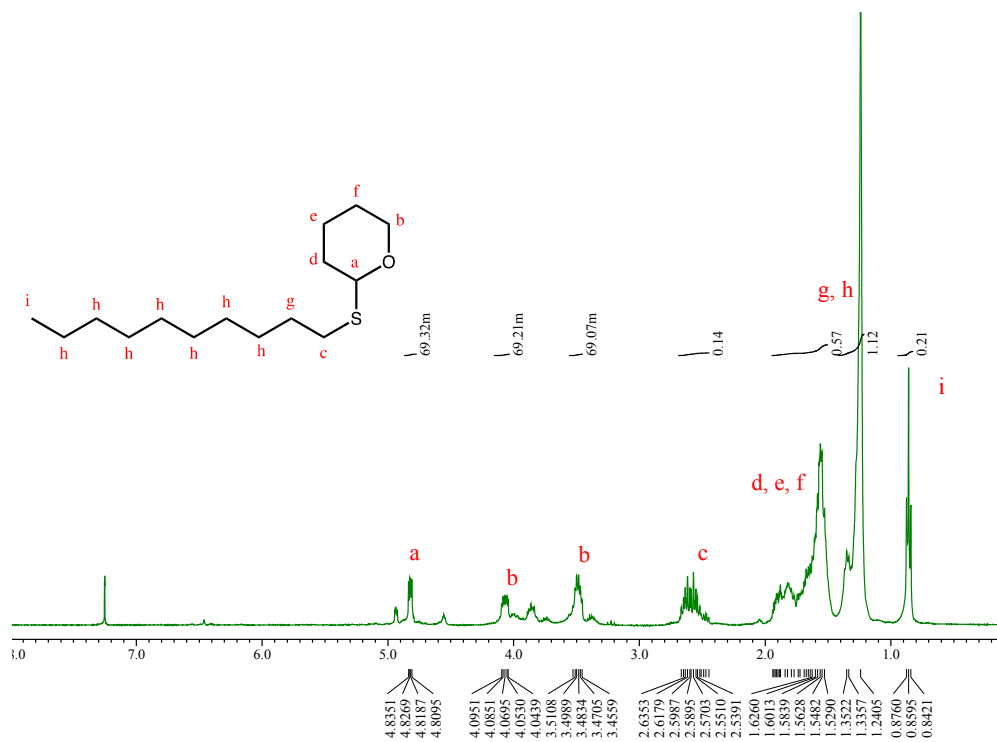


Figure S10. ^1H NMR spectrum of 2-(decylthio)tetrahydro-2*H*-pyran.

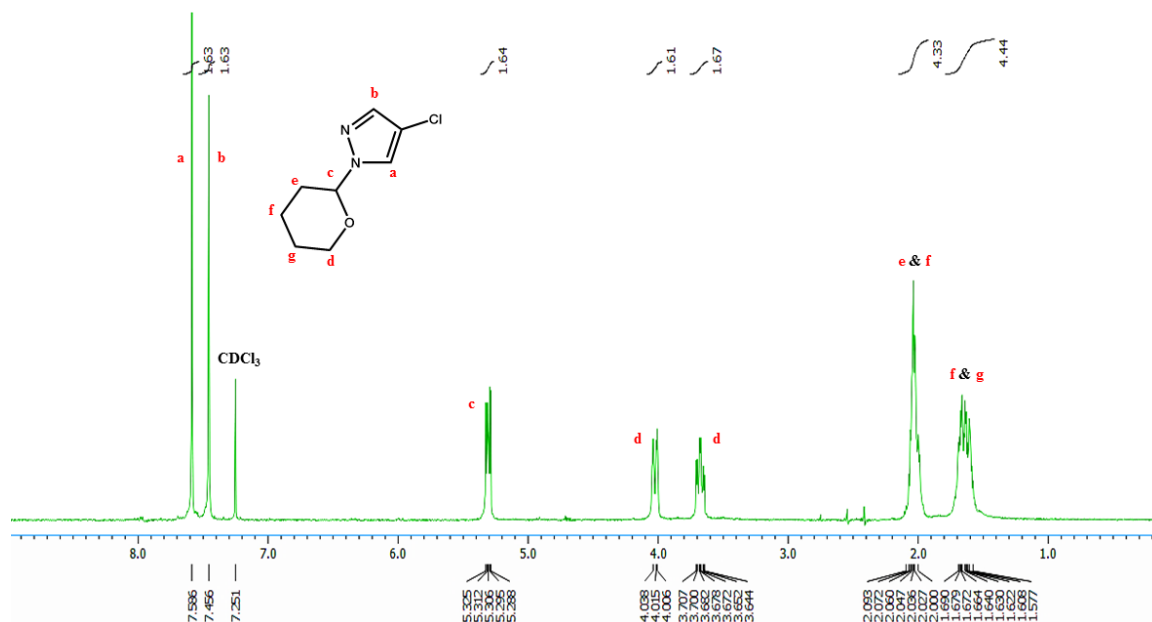


Figure S11. ^1H NMR spectrum of 4-chloro-1-(tetrahydro-2H-pyran-2-yl)-1H-pyrazole (100%).

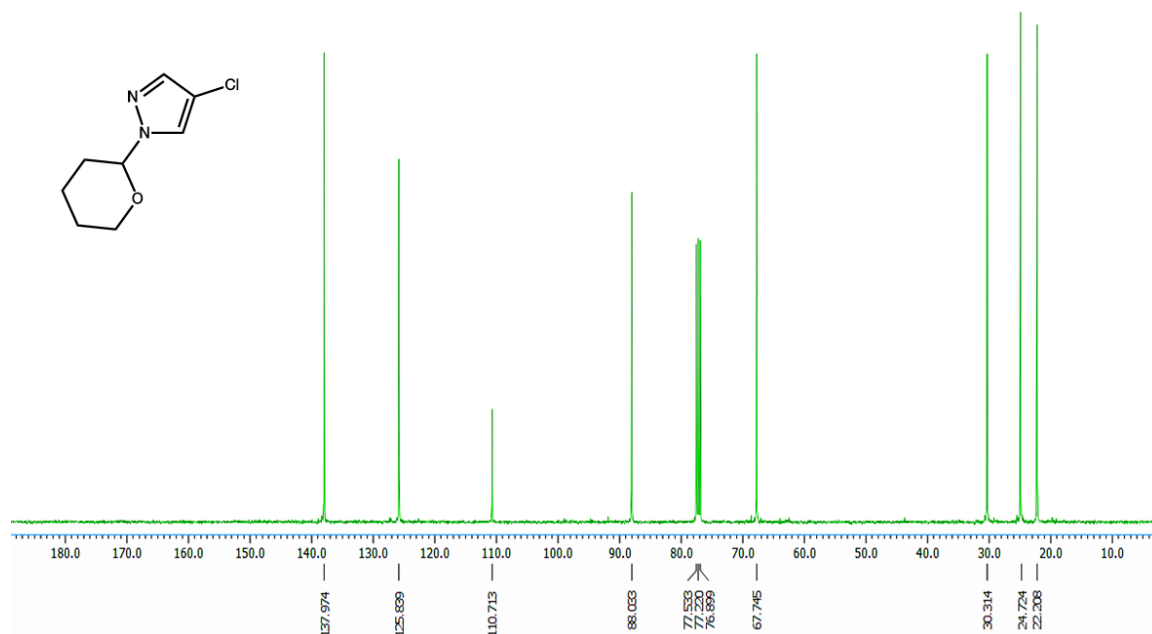


Figure S12. ^{13}C NMR spectrum of 4-chloro-1-(tetrahydro-2H-pyran-2-yl)-1H-pyrazole.

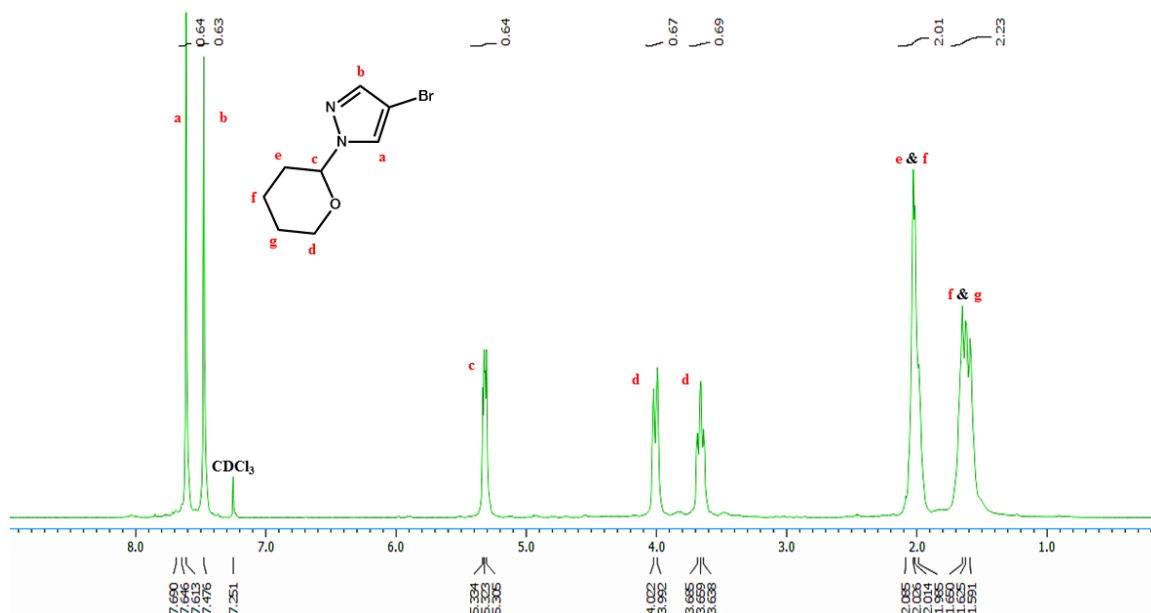


Figure S13. ¹H NMR spectrum of 4-bromo-1-(tetrahydro-2H-pyran-2-yl)-1H-pyrazole.

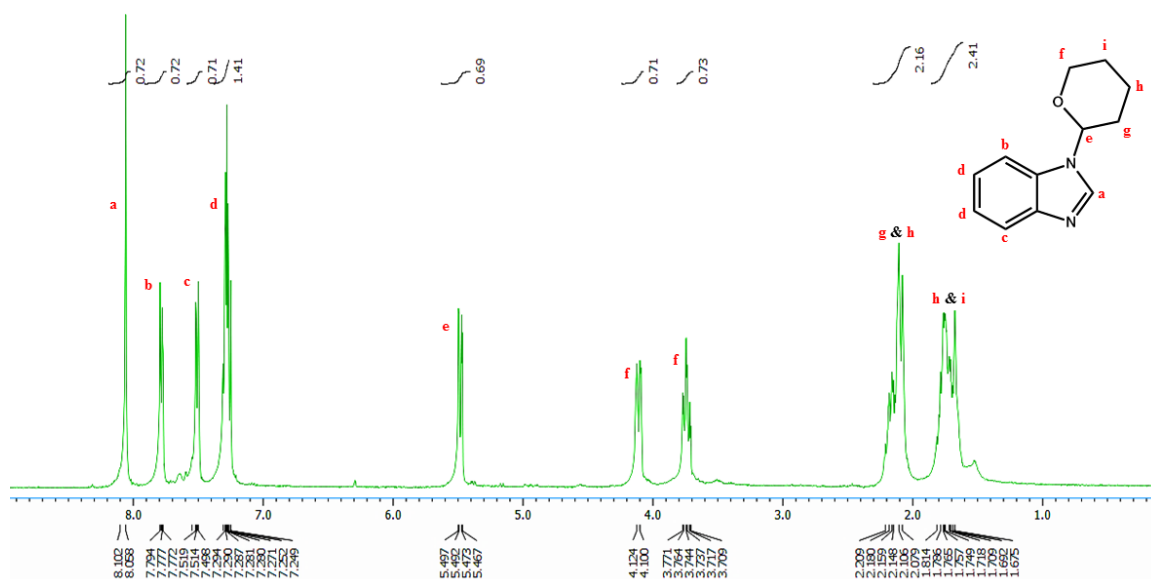


Figure S14. ¹H NMR spectrum of 1-(tetrahydro-2H-pyran-2-yl)-1H-benzimidazole.

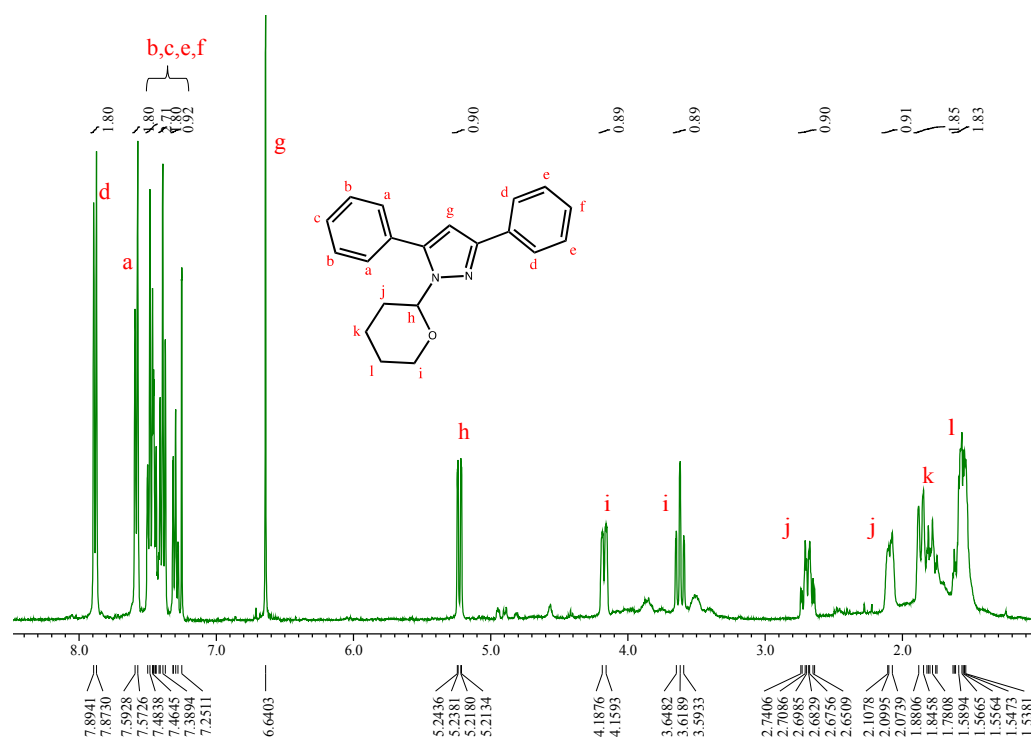


Figure S15. ¹H NMR spectrum of 3,5-diphenyl-1-(tetrahydro-2H-pyran-2-yl)-1H-pyrazole.

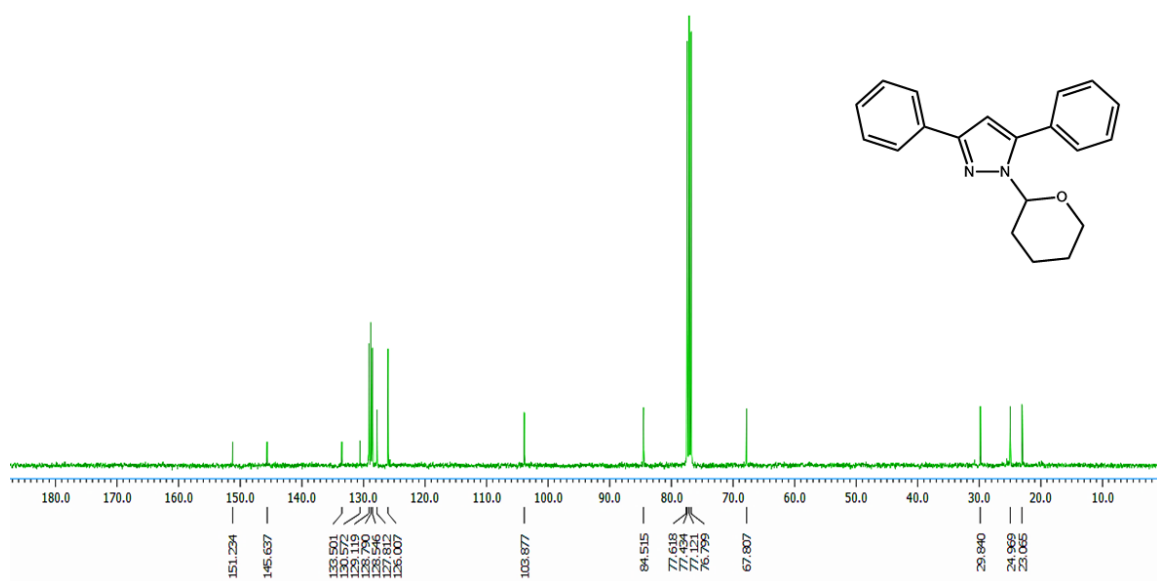


Figure S16. ¹³C NMR spectrum of 3,5-diphenyl-1-(tetrahydro-2H-pyran-2-yl)-1H-pyrazole.

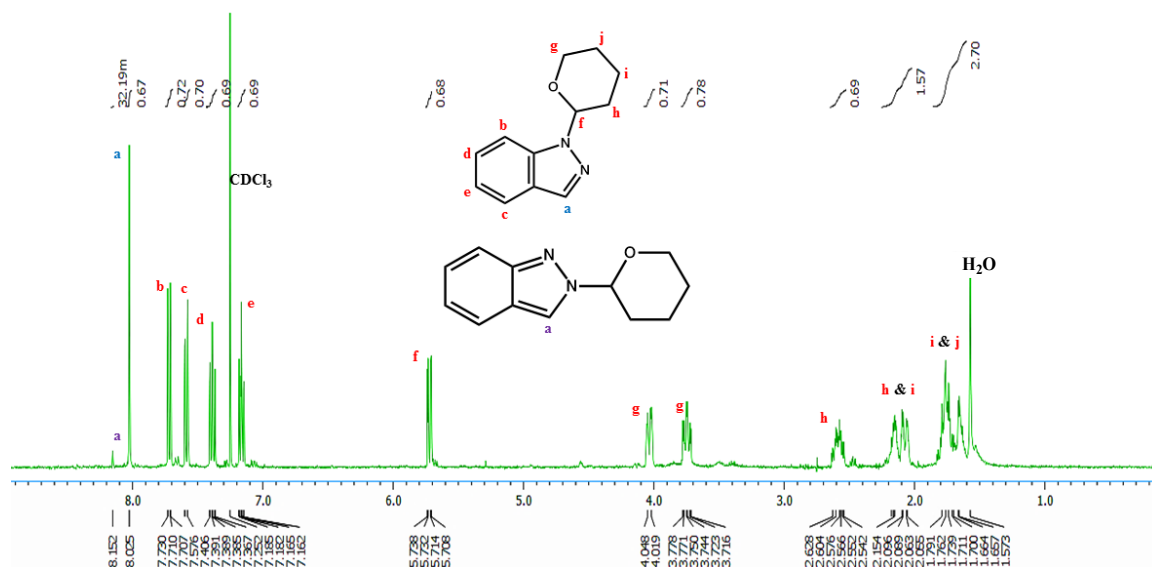


Figure S17. ^1H NMR spectrum of 1-(tetrahydro-2*H*-pyran-2-yl)-1*H*-indazole (5%) and 2-(tetrahydro-2*H*-pyran-2-yl)-2*H*-indazole (95%).

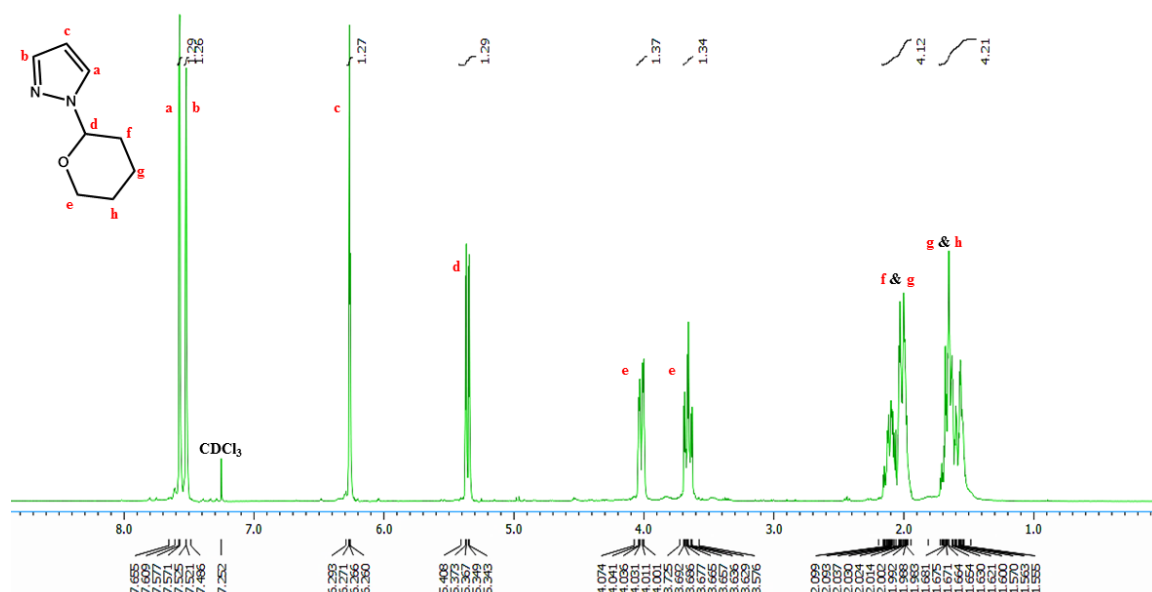


Figure S18. ^1H NMR spectrum of 1-(tetrahydro-2*H*-pyran-2-yl)-1*H*-pyrazole.

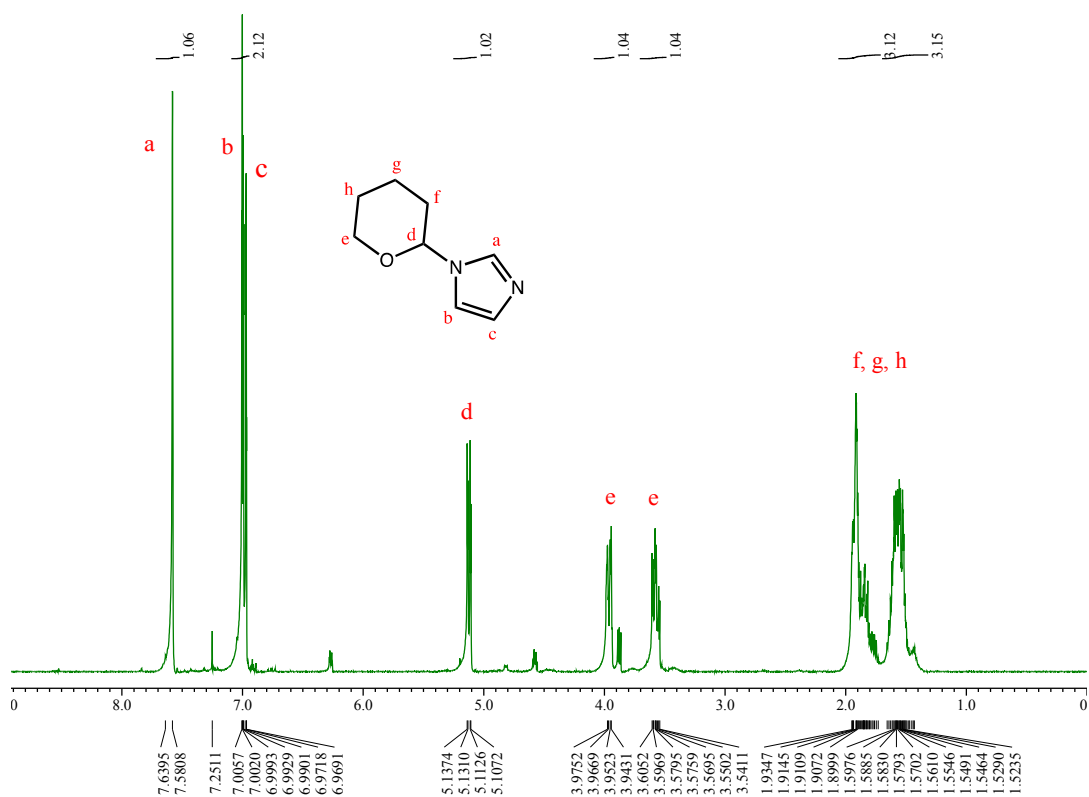


Figure S19. ^1H NMR spectrum of 1-(tetrahydro-2*H*-pyran-2-yl)-1*H*-imidazole.

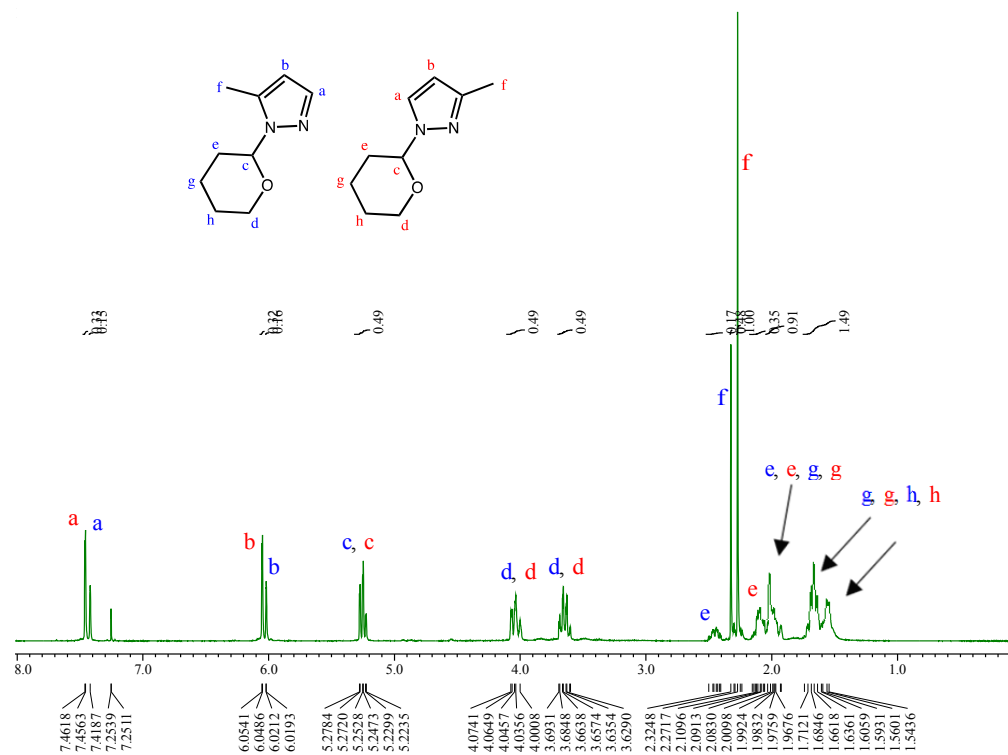


Figure S20. ^1H NMR spectrum of 3(5)-methyl-1-(tetrahydro-2*H*-pyran-2-yl)-1*H*-pyrazole.

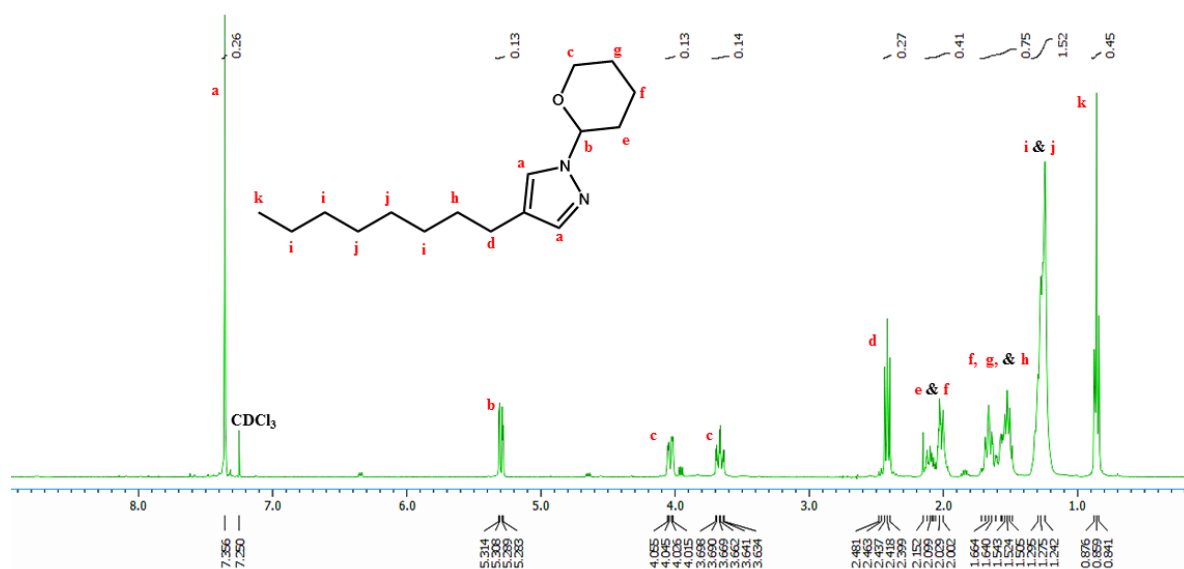


Figure S21. ¹H NMR spectrum of 4-octyl-1-(tetrahydro-2*H*-pyran-2-yl)-1*H*-pyrazole.

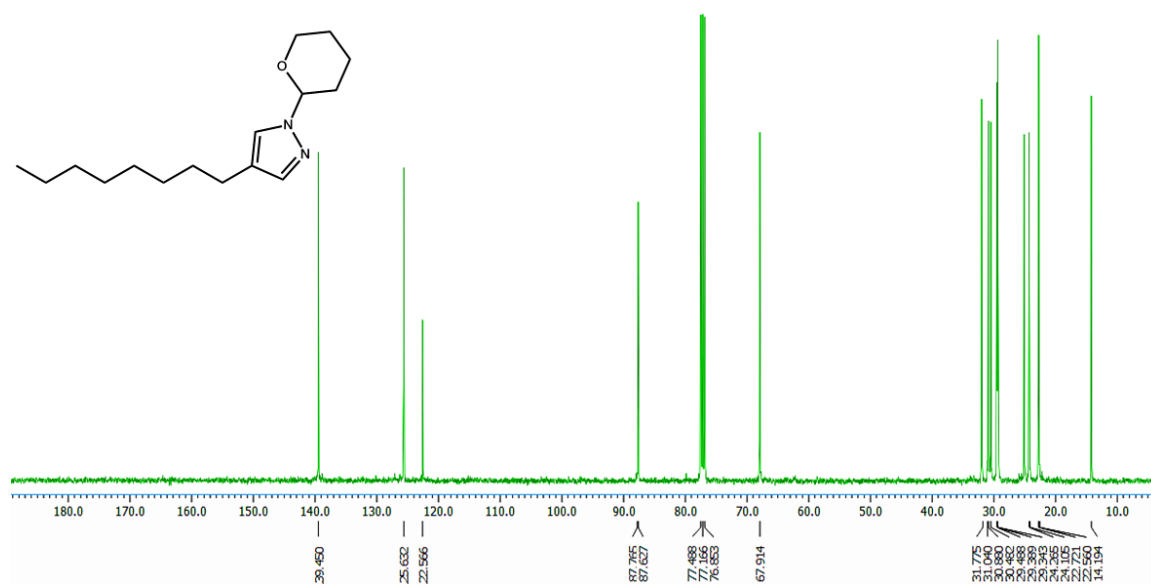


Figure S22. ¹³C NMR spectrum of 4-octyl-1-(tetrahydro-2*H*-pyran-2-yl)-1*H*-pyrazole.

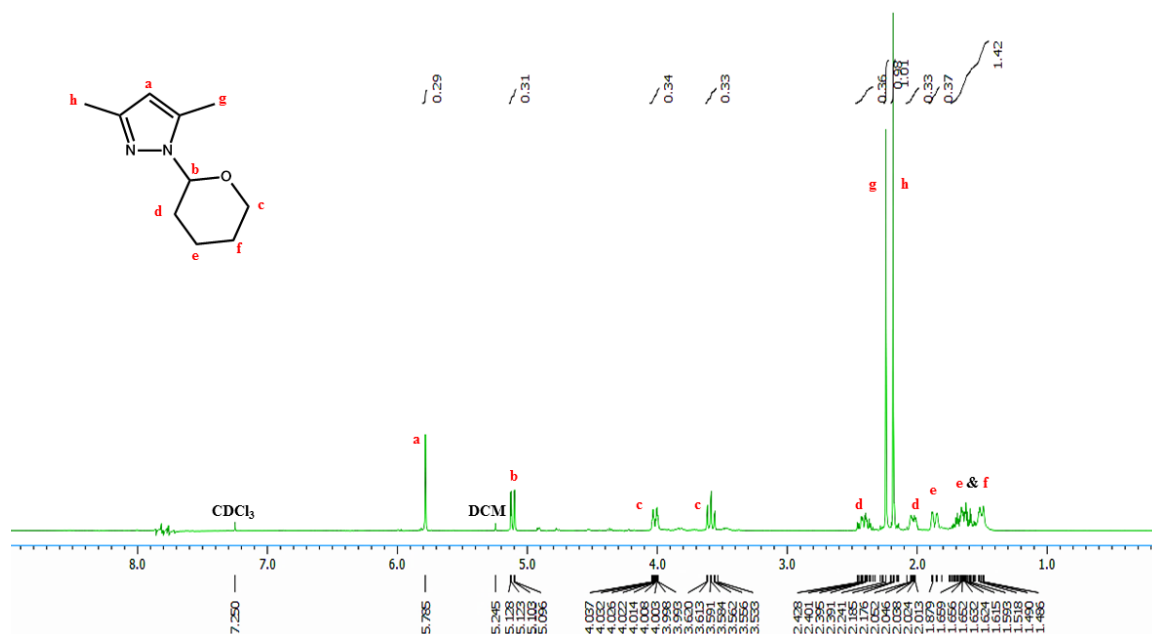


Figure S23. ¹H NMR spectrum of 3,5-dimethyl-1-(tetrahydro-2H-pyran-2-yl)-1H-pyrazole.

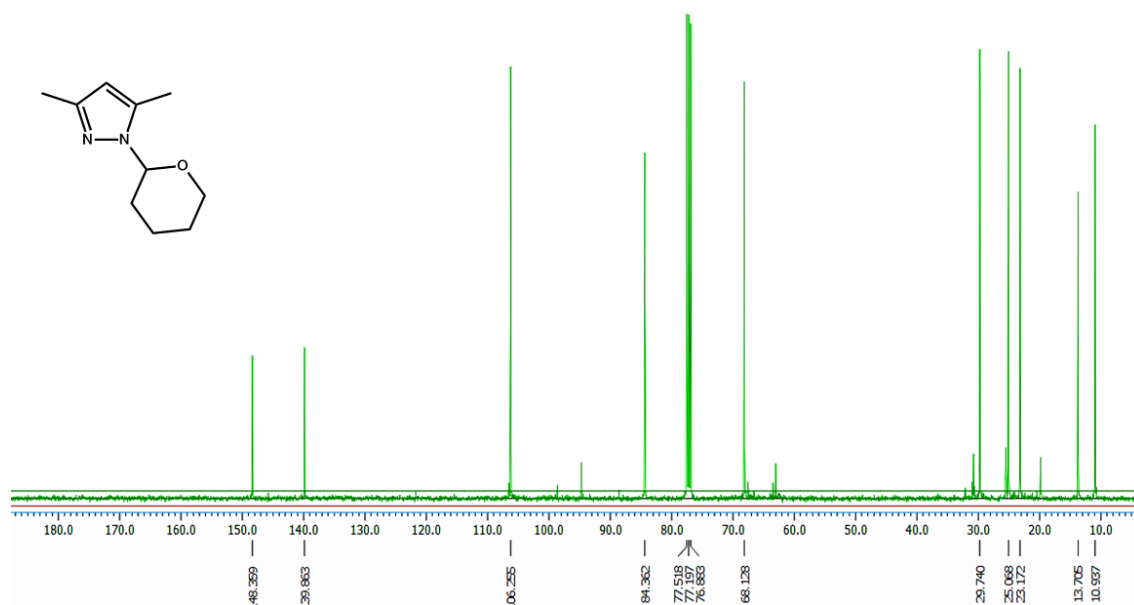


Figure S24. ¹³C NMR spectrum of 3,5-dimethyl-1-(tetrahydro-2H-pyran-2-yl)-1H-pyrazole.

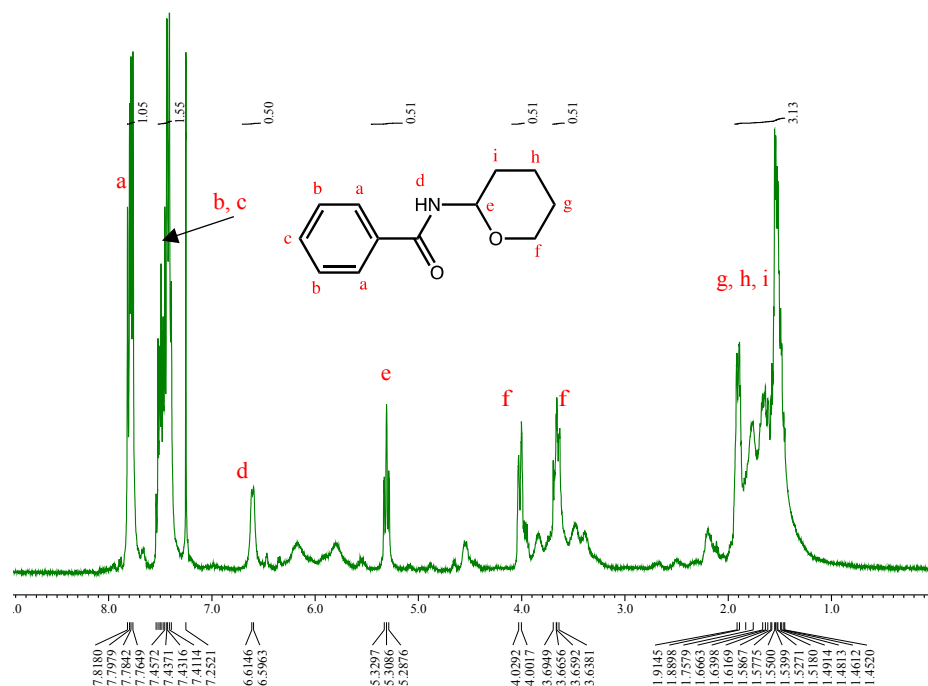


Figure S25. ¹H NMR spectrum of N-(tetrahydro-2H-pyran-2-yl)benzamide.