

An efficient synthesis of Nitrile, Tetrazole and Urea from carbonyl compounds

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Analytical Data

Benzonitrile (2a)¹: ¹H NMR (300 MHz, CDCl₃) δ 7.66 – 7.59 (m, 3H), 7.49 - 7.45 (m, 2H). ¹³C NMR (75 MHz, CDCl₃) δ 132.81, 132.10, 129.12, 118.84, 112.31. IR (neat, cm⁻¹); 2225.

4-methylbenzonitrile (2b)¹: ¹H NMR (300 MHz, CDCl₃) δ 7.55 (d, *J* = 8.2 Hz, 2H), 7.27 (d, *J* = 8.2 Hz, 2H), 2.43 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 143.76, 132.02, 129.86, 119.18, 109.23, 21.82. IR (neat, cm⁻¹); 2220.

4-chlorobenzonitrile (2c)¹: ¹H NMR (300 MHz, CDCl₃) δ 7.61 (d, *J* = 8.4 Hz, 2H), 7.48 (d, *J* = 8.4 Hz, 2H). ¹³C NMR (75 MHz, CDCl₃) δ 139.69, 133.51, 129.83, 118.10, 110.90. IR (neat, cm⁻¹); 2222.

4-fluorobenzonitrile (2d)¹: ¹H NMR (300 MHz, CDCl₃) δ 7.74 – 7.65 (m, 2H), 7.24 – 7.15 (m, 2H). ¹³C NMR (75 MHz, CDCl₃) δ 165.11 (*J* = 255.1 Hz), 134.77 (*J* = 9.3 Hz), 118.23, 116.94 (*J* = 22.5 Hz), 116.00, 108.64. IR (neat, cm⁻¹); 2229.

4-methoxybenzonitrile (2e)¹: ¹H NMR (300 MHz, CDCl₃) δ 7.60 (d, *J* = 8.8 Hz, 2H), 6.96 (d, *J* = 8.8 Hz, 2H), 3.87 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 162.88, 134.00, 119.29, 114.79, 103.89, 55.59. IR (neat, cm⁻¹); 2232,

1-naphthonitrile (2f)¹: ¹H NMR (300 MHz, CDCl₃) δ 8.25 (d, *J* = 8.3 Hz, 1H), 8.10 (d, *J* = 8.3 Hz, 1H), 7.97 – 7.89 (m, 2H), 7.76 – 7.67 (m, 1H), 7.67 – 7.60 (m, 1H), 7.57 – 7.50 (m, 1H). ¹³C NMR (75 MHz, CDCl₃) δ 133.42, 133.05, 132.78, 132.49, 128.79, 128.74, 127.69, 125.29, 125.06, 117.96, 110.31. IR (neat, cm⁻¹); 2220,

Thiophene-2-carbonitrile (2g)¹: ¹H NMR (300 MHz, CDCl₃) δ 7.65 (d, *J* = 3.7 Hz, 1H), 7.62 (d, *J* = 5.1 Hz, 1H), 7.16 – 7.12 (m, 1H). ¹³C NMR (75 MHz, CDCl₃) δ 137.51, 132.70, 127.73, 114.31, 109.90. IR (neat, cm⁻¹); 2229,

3,4,5-trimethoxybenzonitrile (2h)²: ¹H NMR (300 MHz, CDCl₃) δ 6.87 (s, 2H), 3.91 (s, 3H), 3.89 (s, 6H). ¹³C NMR (75 MHz, CDCl₃) δ 153.60, 142.34, 119.04, 109.47, 106.74, 61.10, 56.43. IR (neat, cm⁻¹); 2235.

3,4,-dimethoxybenzonitrile (2i)³: ¹H NMR (300 MHz, CDCl₃) δ 7.30 (d, *J* = 8.4 Hz, 1H), 7.09 (s, 1H), 6.91 (d, *J* = 8.4 Hz, 1H), 3.94 (s, 3H), 3.91 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 152.86, 149.16, 126.50, 119.27, 113.90, 111.25, 103.82, 56.15, 56.12. IR (neat, cm⁻¹); 2218,

Anthracene-9-carbonitrile (2j)⁴: ¹H NMR (300 MHz, CDCl₃) δ 8.69 (s, 1H), 8.43 (d, *J* = 8.7 Hz, 2H), 8.09 (d, *J* = 8.5 Hz, 2H), 7.78 – 7.67 (m, 2H), 7.64 – 7.55 (m, 2H). ¹³C NMR (75 MHz, CDCl₃) δ 133.41, 132.86, 130.72, 130.55, 130.01, 129.07, 126.47, 125.38, 117.40. IR (neat, cm⁻¹); 2225,

4-isopropylbenzonitrile (2k)⁵: ¹H NMR (300 MHz, CDCl₃) δ 7.58 (d, *J* = 8.3 Hz, 2H), 7.32 (d, *J* = 8.2 Hz, 2H), 3.03 -2.89 (m, 1H), 1.26 (d, *J* = 6.9 Hz, 6H). ¹³C NMR (75 MHz, CDCl₃) δ 154.19, 132.05, 127.12, 119.01, 109.39, 34.20, 23.35. IR (neat, cm⁻¹); 2229,

2-methoxybenzonitrile (2l)³: ¹H NMR (300 MHz, CDCl₃) δ 7.57-7.52 (m, 2H), 7.03-6.96 (m, 2H), 3.94 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 161.24, 134.50, 133.74, 120.80, 111.36, 56.04. IR (neat, cm⁻¹); 2235,

4-nitrobenzonitrile (2m)²: ¹H NMR (300 MHz, CDCl₃) δ 8.37 (d, *J* = 8.8 Hz, 1H), 7.90 (d, *J* = 8.8 Hz, 1H). ¹³C NMR (75 MHz, CDCl₃) δ 139.60, 133.46, 129.26, 117.93, 116.83, 111.00. IR (neat, cm⁻¹); 2210,

phenyl 2-(4-cyanophenoxy)acetate (2n): ¹H NMR (300MHz CDCl₃): δ 7.63 (d, *J* = 8.7Hz, 2H), 7.42 (t, *J*=7.8Hz, 2H), 7.28 (t, *J*= 7.8Hz, 1H), 7.12 (d, *J*=7.5Hz, 2H), 7.04 (d, *J*=8.7Hz, 2H), 4.93 (s, 2H). ¹³C NMR (75MHz CDCl₃): δ 166.52, 160.85, 149.92, 134.22, 129.70, 126.52, 121.17, 118.88, 115.49, 105.47, 65.19.

5-methyl-1-phenyl-1H-tetrazole (4a)⁶: ¹H NMR (300MHz CDCl₃): δ 7.63-7.59 (m, 3H), 7.49-7.46 (m, 2H), 2.63 (s, 3H). ¹³C NMR (75MHz CDCl₃): δ 151.64, 133.87, 130.41, 130.01, 124.60, 9.84. ESI-MS calculated.m/z 160.07 found 161.07 [M+1]⁺.

1-(4-methoxyphenyl)-5-methyl-1H-tetrazole (4b)⁶: ¹H NMR (300MHz CDCl₃): δ 7.38 (d, *J*= 8.7Hz, 2H), 7.08 (d, *J*=8.7Hz, 2H), 3.90 (s, 3H), 2.58 (s, 3H). ¹³C NMR (75MHz CDCl₃): δ 160.89, 151.78, 126.46, 126.11, 115.03, 55.75, 9.63. ESI-MS calculated.m/z 190.09 found 191.07 [M+1]⁺.

1-(4-chlorophenyl)-5-methyl-1H-tetrazole (4c)⁷: ¹H NMR (300MHz CDCl₃): δ 7.61-7.54 (m, 2H), 7.46-7.43 (m, 2H), 2.62 (s, 3H). ¹³C NMR (75MHz CDCl₃): δ 151.49, 136.13, 128.23, 127.45, 119.43, 9.55. ESI-MS calculated.m/z 194.04 found 195.04.

5-methyl-1-(*p*-tolyl)-1H-tetrazole (4d)⁶: ¹H NMR (300MHz CDCl₃): δ 7.40 (d, *J*=8.7Hz, 2H), 7.35 (d, *J*=8.7Hz, 2H), 2.60 (s, 3H), 2.47 (s, 3H). ¹³C NMR (75MHz CDCl₃): δ 151.60, 140.74, 131.27, 130.47, 124.39, 21.28, 9.75. ESI-MS calculated.m/z 174.09 found 175.08 [M+1]⁺.

5-methyl-1-(4-nitrophenyl)-1H-tetrazole (4e)⁷: ¹H NMR (300MHz CDCl₃): δ 8.43–8.38 (d, *J* = 7.9 Hz, 2H), 8.31(d, *J* = 7.9Hz, 2H), 2.70 (s, 3H). ¹³C NMR (75MHz CDCl₃): 151.04, 147.66, 133.69, 129.54, 123.75, 8.64. ESI-MS calculated.m/z 205.06 found 206.05 [M+1]⁺.

1-(2-methoxyphenyl)-5-methyl-1H-tetrazole (4f)⁶: ¹H NMR (300MHz CDCl₃): δ 7.56-7.53 (m, 1H), 7.34-7.37 (m, 1H), 7.16-7.11(m, 2H), 3.83 (s, 3H), 2.45 (s, 3H). ¹³C NMR (75MHz CDCl₃): δ 153.53, 153.30, 132.36, 127.85, 122.11, 121.06, 112.36, 55.86, 8.94. ESI-MS calculated.m/z 190.09 found 191.10 [M+1]⁺.

1-(3-methoxyphenyl)-5-methyl-1H-tetrazole (4g)⁶: ¹H NMR (300MHz CDCl₃): δ 7.52-7.46 (m, 1H), 7.12-7.08(m, 1H), 7.04-7.01(m, 2H), 3.88 (s, 3H), 2.63 (s, 3H). ¹³C NMR (75MHz CDCl₃): δ 160.70, 151.68, 134.88, 130.80, 116.52, 116.13, 110.61, 55.83, 9.96. ESI-MS calculated.m/z 190.09 found 191.10 [M+1]⁺.

1-([1,1'-biphenyl]-4-yl)-5-methyl-1H-tetrazole (4h)⁶: ¹H NMR (300MHz CDCl₃): δ 7.82- 7.78 (m, 2H), 7.65-7.62(m, 2H), 7.56-7.46 (m, 5H), 2.67 (s, 3H). ¹³C NMR (75MHz CDCl₃): δ 151.66, 143.47, 139.30, 132.86, 129.18, 128.60, 127.30, 124.91, 9.97. ESI-MS calculated.m/z 236.11 found 237.13. [M+1]⁺.

5-methyl-1-(naphthalen-2-yl)-1H-tetrazole (4i)⁶: ¹H NMR (300MHz CDCl₃): δ 8.13–8.11 (m, 1H), 8.02–8.01 (m, 1H), 7.66–7.61 (m, 2H), 7.58–7.55, (m, 1H), 7.51–7.49 (m, 1H), 7.17–7.15 (m, 1H), 2.44 (s, 3H). ¹³C NMR (75MHz CDCl₃): δ 153.4, 134.2, 131.6, 129.0, 128.5, 127.5, 125.0, 121.5, 9.1. ESI-MS calculated.m/z 210.09 found. 211.10 [M+1]⁺.

1-(5-bromothiophen-2-yl)-5-methyl-1H-tetrazole (4j): ¹H NMR (300MHz CDCl₃): δ 7.14 (d, 1H, *J*= 3.9Hz), 7.01 (d, 1H, *J*=3.9Hz), 2.64 (s, 3H). ¹³C NMR (75MHz CDCl₃): δ 152.42, 131.37, 129.36, 124.59, 113.86, 9.70. ESI-MS calculated.m/z 243.94 found 244.91[M+1]⁺.

1,3-diphenylurea (6a)⁸: ¹H NMR (300 MHz, DMSO) δ 8.25 (s, 2H), 7.46 (dd, J = 7.3, 6.3 Hz, 4H), 7.27 (t, J = 7.9 Hz, 4H), 6.98 (t, J = 7.4 Hz, 2H). ¹³C NMR (75 MHz, DMSO) δ 152.67, 139.03, 128.28, 121.67, 118.16. ESI-LC/MS calculated m/z 212.1, found 213.1. (M+1)⁺.

1-phenyl-3-(*p*-tolyl)urea (6b)⁹: ¹H NMR (300 MHz, CDCl₃+DMSO) δ 8.20 -8.03 (m, 2H), 7.45-7.33 (m, 4H), 7.25 – 7.05 (m, 4H), 7.07 (d, J = 7.8 Hz, 1H), 2.73 (s, 3H). ¹³C NMR (75 MHz, CDCl₃+DMSO) δ 153.24, 139.26, 136.46, 131.58, 129.09, 128.57, 122.00, 118.93, 118.62, 20.46. ESI-LC/MS calculated m/z 226.21, found 227.30. (M+1)⁺

1-(4-chlorophenyl)-3-phenylurea (6c)⁴⁰: ¹H NMR (300 MHz, CDCl₃+DMSO) δ 8.51 (s, 1H), 8.38 (s, 1H), 7.45 – 7.42 (m, 4H), 7.31 – 7.19 (m, 4H), 6.98 (t, J = 7.3 Hz, 1H). ¹³C NMR (75 MHz, CDCl₃+DMSO) δ 152.26, 138.66, 137.70, 128.07, 127.88, 125.72, 121.57, 119.08, 117.97. ESI-LC/MS calculated m/z 246.06, found 247.09 (M+1)⁺.

1,3-bis(4-chlorophenyl)urea (6d)⁸: ¹H NMR (300 MHz, CDCl₃+DMSO) δ 8.54 (s, 2H), 7.44 (d, J = 8.8 Hz, 4H), 7.22 (d, J = 8.8 Hz, 4H). ¹³C NMR (75 MHz, CDCl₃+DMSO) δ 151.85, 137.39, 127.69, 125.61, 118.94. ESI-LC/MS calculated m/z 280.02, found 279.11 (M-1)⁻.

1-(4-fluorophenyl)-3-phenylurea (6e)¹¹: ¹H NMR (300 MHz, CDCl₃+DMSO) δ 8.17 (d, J = 15.4 Hz, 3H), 7.46 – 7.37 (m, 6H), 7.30 – 7.24 (m, 3H), 7.01 – 6.93 (m, 4H). ¹³C NMR (75 MHz, CDCl₃+DMSO) δ 157.77 (J = 236.2 Hz), 153.67, 139.77, 135.85, 129.15, 122.68, 120.76 (J = 7.5 Hz), 119.50, 119.13, 115.63 (J = 23.4). ESI-LC/MS calculated m/z 230.09, found 229.09 (M-1)⁻.

1-(4-nitrophenyl)-3-phenylurea (6f)⁹: ¹H NMR (300 MHz, DMSO) δ 8.71 (s, 1H), 8.00 (d, J = 9.1 Hz, 1H), 7.50 (d, J = 8.4 Hz, 2H), 7.33 (t, J = 7.8 Hz, 2H), 7.02 (t, J = 7.3 Hz, 1H), 6.79 (s,

1H), 6.65 (d, $J = 9.1$ Hz, 1H). ^{13}C NMR (75 MHz, DMSO) δ 156.28, 153.16, 140.25, 129.39, 127.00, 122.46, 118.82, 118.74, 112.99. ESI-LC/MS calculated m/z 257.08, found 256.96 ($\text{M}-1$) $^-$.

1,3-bis(4-nitrophenyl)urea (6g)¹²: ^1H NMR (300 MHz, DMSO) δ 8.00 (d, $J = 9.0$ Hz, 2H), 6.79 (s, 2H), 6.65 (d, $J = 9.1$ Hz, 2H). ^{13}C NMR (75 MHz, DMSO) δ 156.10, 126.76, 126.23, 117.58, 112.89. ESI-LC/MS calculated m/z 302.07, found 301.08 ($\text{M}-1$) $^-$.

1-(3-methoxyphenyl)-3-phenylurea (6h)¹³: ^1H NMR (300 MHz, CDCl_3 +DMSO) δ 8.38 (br, 2H), 7.44 (d, $J = 7.9$ Hz, 2H), 7.27 (t, $J = 7.7$ Hz, 3H), 7.15 (t, $J = 8.1$ Hz, 1H), 6.98 (t, $J = 7.3$ Hz, 1H), 6.89 (d, $J = 8.1$ Hz, 1H), 6.53 (d, $J = 8.2$ Hz, 1H), 3.79 (s, 3H). ^{13}C NMR (75 MHz, CDCl_3 +DMSO) δ 159.34, 152.36, 140.19, 138.79, 128.74, 128.10, 121.50, 117.97, 110.12, 107.04, 103.59, 54.42. ESI-LC/MS calculated m/z 242.28, found 243.11 ($\text{M}+1$) $^+$.

1-(2-methoxyphenyl)-3-phenylurea (6i)¹⁰: ^1H NMR (300 MHz, CDCl_3 +DMSO) δ 8.72 (s, 1H), 8.23-8.20 (m, 1H), 8.00 (s, 1H), 7.46 (d, $J = 7.9$ Hz, 2H), 7.24 (t, $J = 7.8$ Hz, 2H), 7.03 – 6.80 (m, 4H), 3.82 (s, 3H). ^{13}C NMR (75 MHz, CDCl_3 +DMSO) δ 153.23, 147.93, 139.47, 128.73, 128.68, 122.15, 121.91, 120.94, 119.19, 118.83, 110.11, 55.59. ESI-LC/MS calculated m/z 242.11, found 243.13. ($\text{M}+1$) $^+$.

1-(4-phenoxyphenyl)-3-phenylurea (6j): ^1H NMR (300 MHz, CDCl_3) δ 7.34 – 7.20 (m, 12H), 7.09 – 7.04 (m, 2H), 6.94-6.89 (m, 4H). ^{13}C NMR (75 MHz, CDCl_3) δ 157.62, 154.67, 153.31, 138.26, 133.52, 129.84, 129.24, 123.81, 123.14, 122.88, 120.79, 119.73, 118.52. ESI-LC/MS calculated m/z 304.35, found 303.11 ($\text{M}+1$) $^+$.

1-(naphthalen-1-yl)-3-phenylurea (6k)¹⁰: ^1H NMR (300 MHz, CDCl_3 +DMSO) δ 8.71 (s, 1H), 8.52 (s, 1H), 8.11 (d, $J = 6.8$ Hz, 1H), 8.05 (d, $J = 7.6$ Hz, 1H), 7.87 – 7.83 (m, 1H), 7.58 (d, $J = 8.1$ Hz, 1H), 7.59 - 7.43 (m, 5H), 7.26 (d, $J = 7.5$ Hz, 2H), 6.98 (t, $J = 6.9$ Hz, 1H). ^{13}C NMR (75

MHz, CDCl₃+DMSO) δ 152.72, 139.08, 133.68, 133.59, 128.38, 128.33, 126.29, 125.98, 125.48, 125.07, 122.97, 121.70, 120.88, 120.77, 118.20. ESI-LC/MS calculated m/z 262.11, found 263.06 (M+1)⁺.

1-(tert-butyl)-3-phenylurea (6l)¹⁴:

¹H NMR (300 MHz, CDCl₃) δ 7.34 – 7.27 (m, 2H), 7.25 – 7.18 (m, 2H), 7.04 – 6.99 (m, 1H), 6.97 (bs, 1H), 5.18 (bs, 1H), 1.34 (s, 9H). ¹³C NMR (75 MHz, CDCl₃) δ 156.15, 139.67, 129.41, 123.24, 120.42, 50.88, 29.73. ESI-LC/MS calculated m/z 192.13, found 193.24 (M+1)⁺.

4-(1-chlorovinyl)phenyl methanesulfonate (Intermediate IV)

¹H NMR (300 MHz, CDCl₃) δ 7.69 (d, J = 9.0 Hz, 2H), 7.30 (d, J = 9.0 Hz, 2H), 5.78 (d, J = 2.0 Hz, 3H), 5.58 (d, J = 2.0 Hz, 3H), 3.17 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 149.57, 138.51, 136.29, 128.24, 121.99, 113.96, 37.64.

Diphenylmethanediimine (Intermediate XII)¹⁵

¹H NMR (300 MHz, CDCl₃) δ 7.37 – 7.27 (m, 4H), 7.26 - 7.20 (m, 6H). ¹³C NMR (75 MHz, CDCl₃) δ 138.66, 135.33, 129.65, 125.73, 124.32.

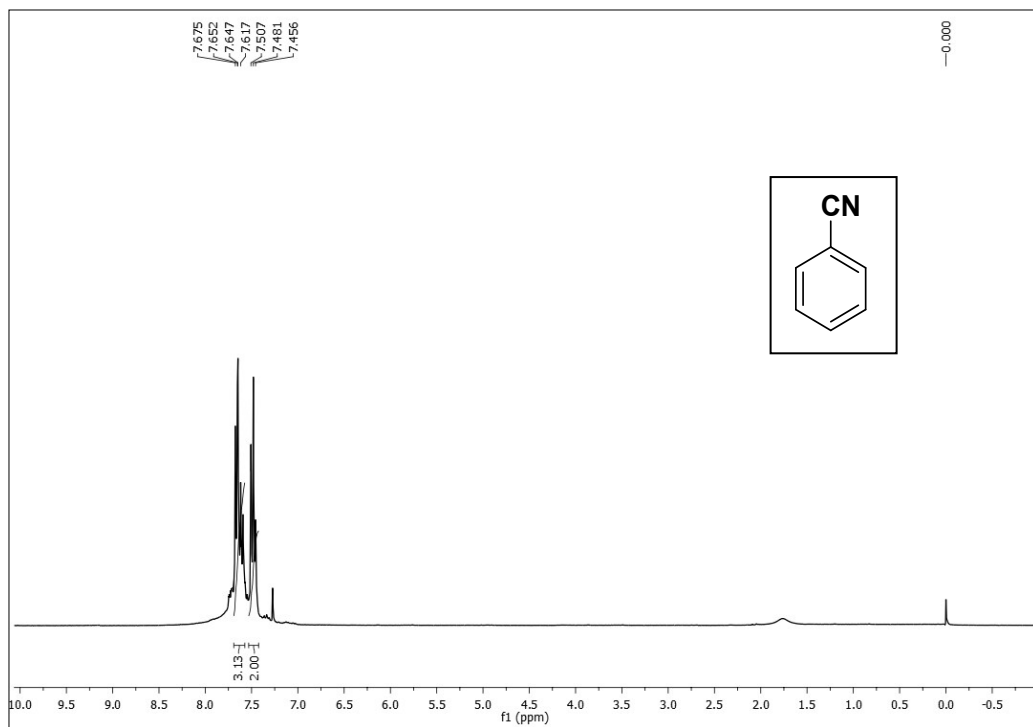
1,5-diphenyl-1H-tetrazole (minor product)

¹H NMR (300 MHz, CDCl₃) δ 7.57 – 7.47 (m, 6H), 7.42 - 7.29 (m, 4H). ¹³C NMR (75 MHz, CDCl₃) δ 153.71, 134.74, 131.34, 130.47, 129.96, 129.05, 129.00, 125.39, 123.79. ESI-LC/MS calculated m/z 223.0, found 224.0 (M+1)⁺. IR (KBr) $\nu_{\max}$; 1000, 1070, 1139, 1265, 1592, 3068.

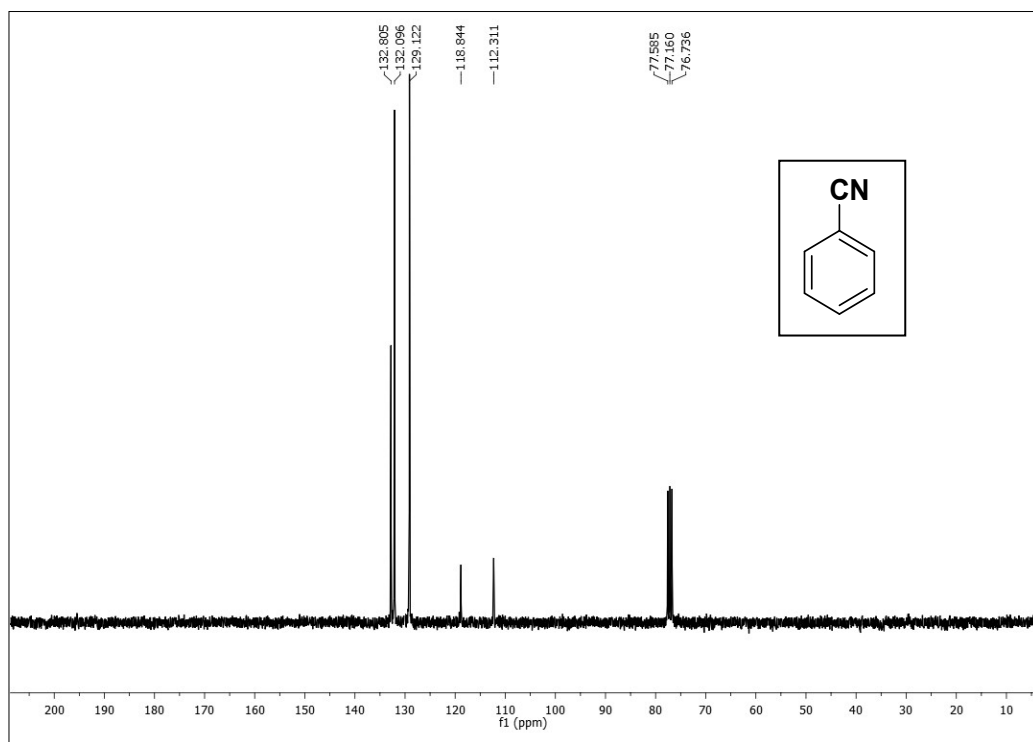
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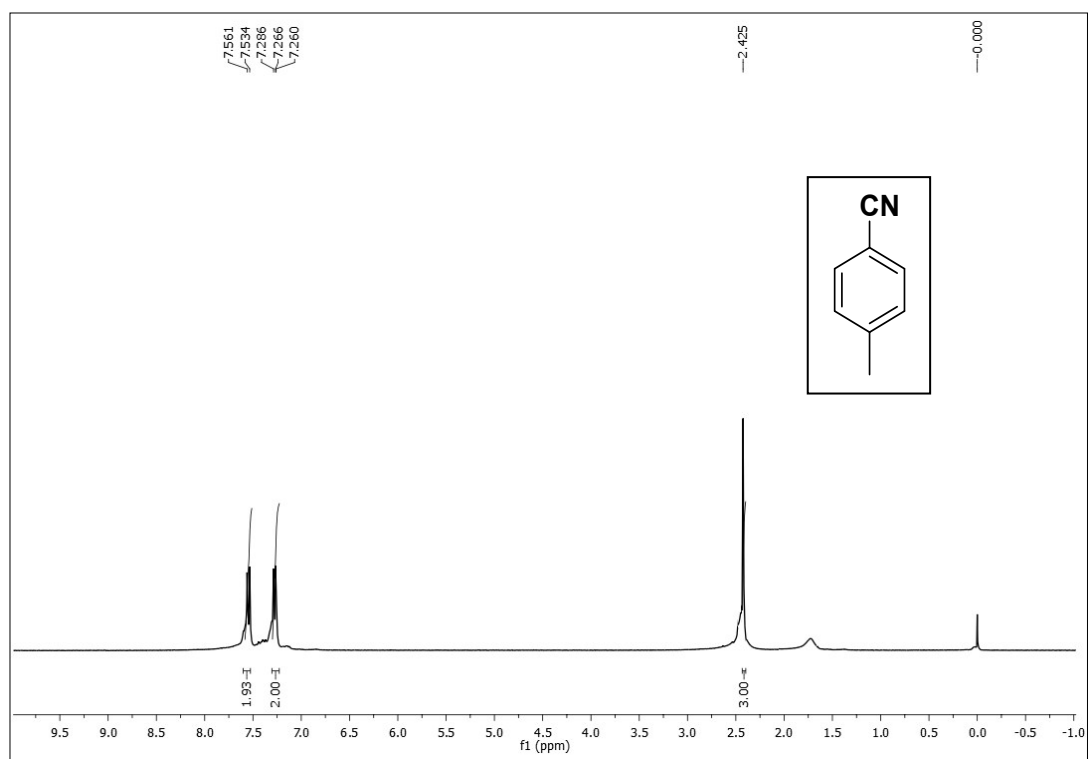
Model Spectrums



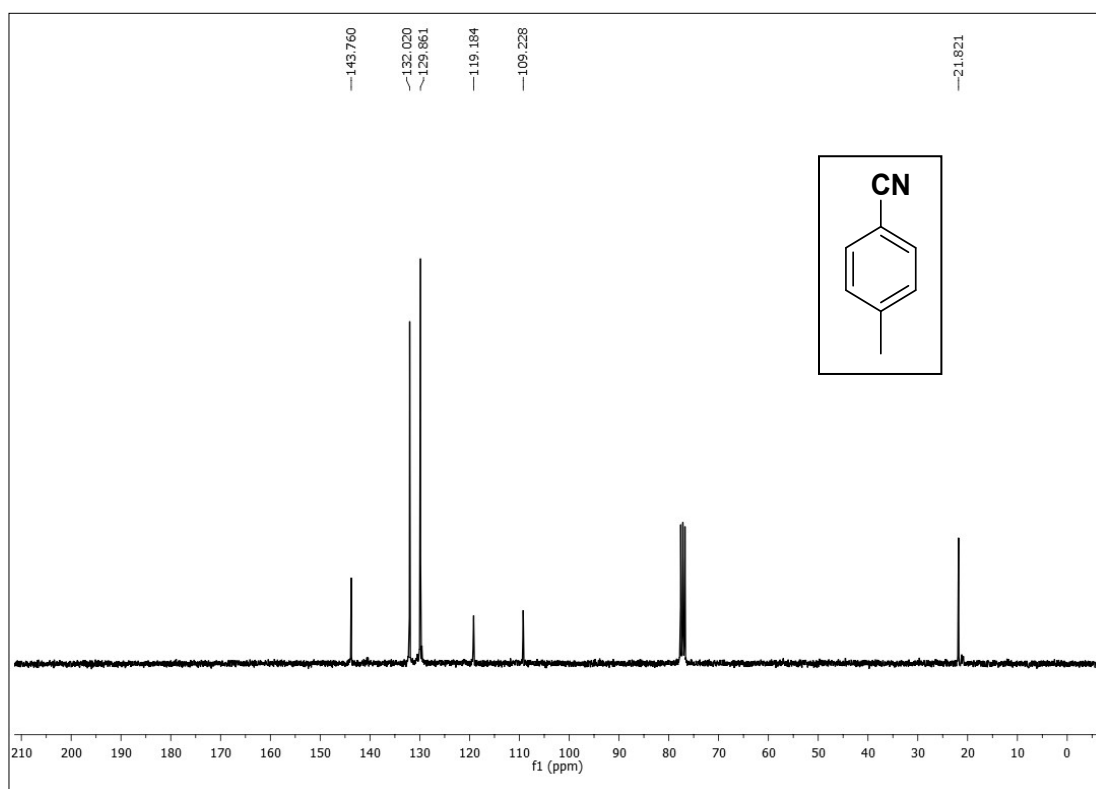
¹H NMR (300 MHz, CDCl₃) spectrum of compound **2a**



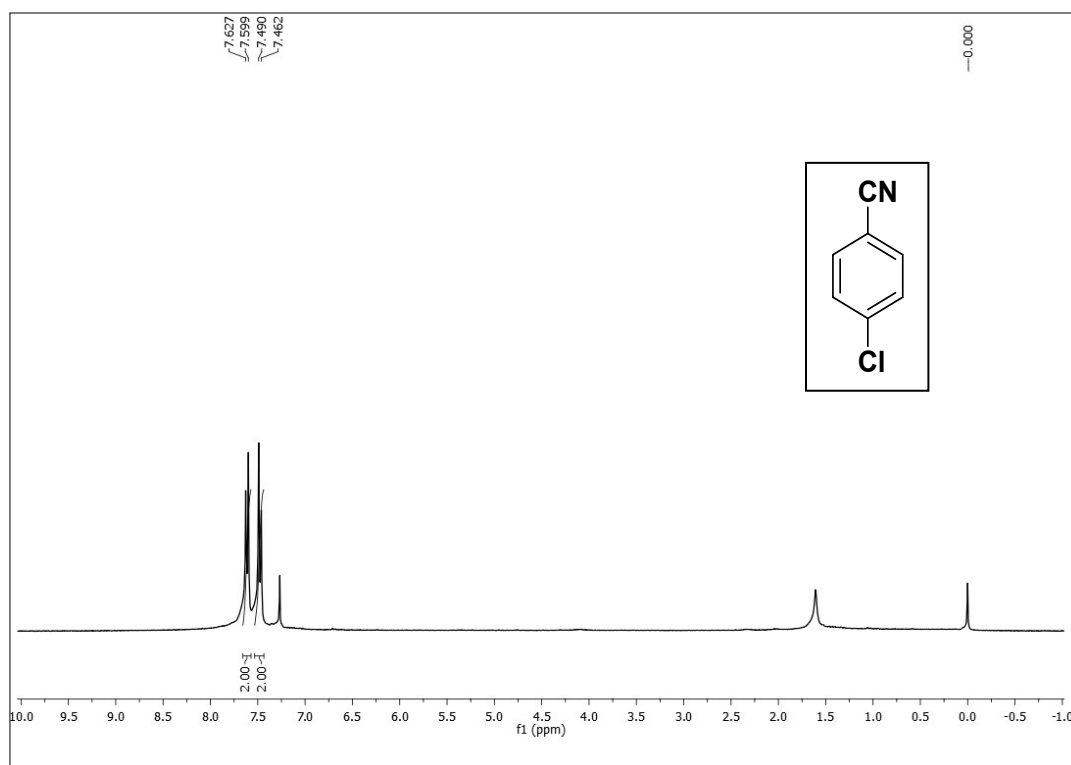
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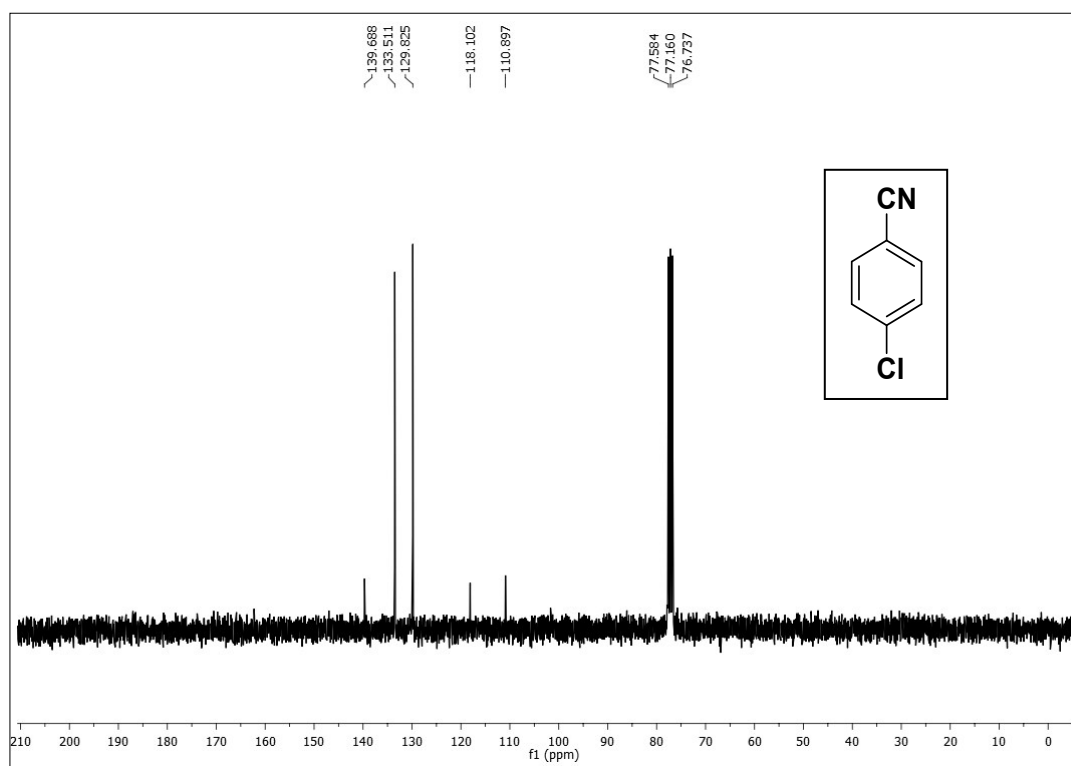
¹H NMR(300 MHz, CDCl₃) spectrum of compound **2b**



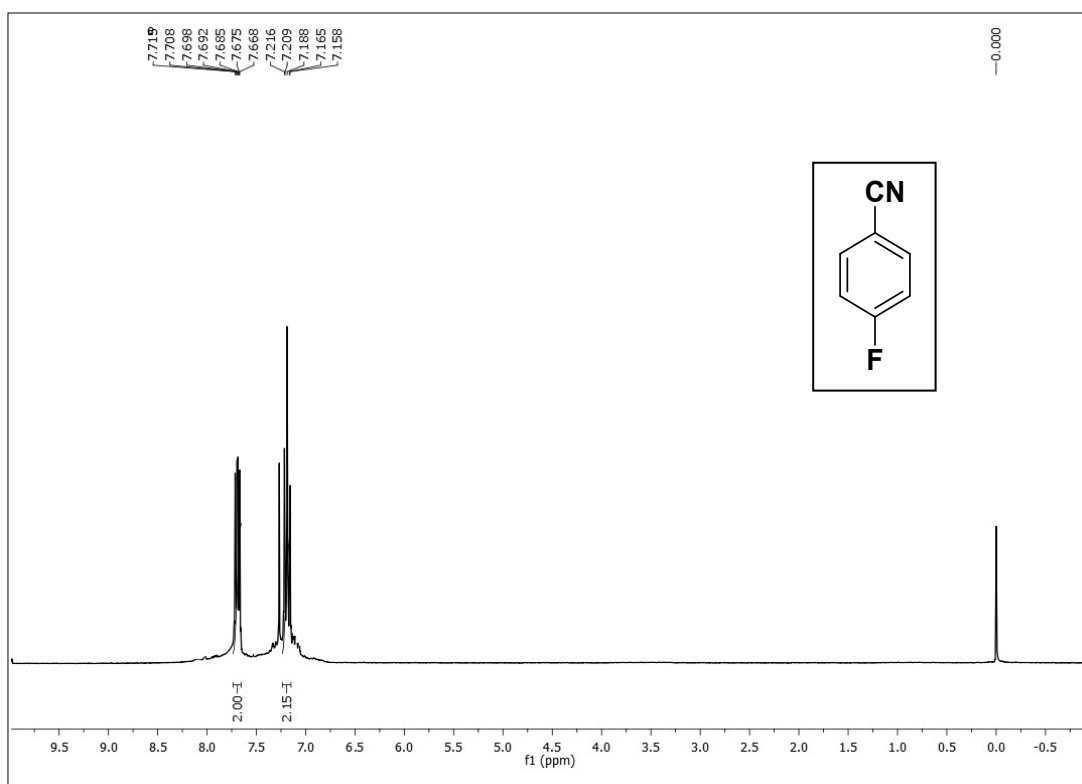
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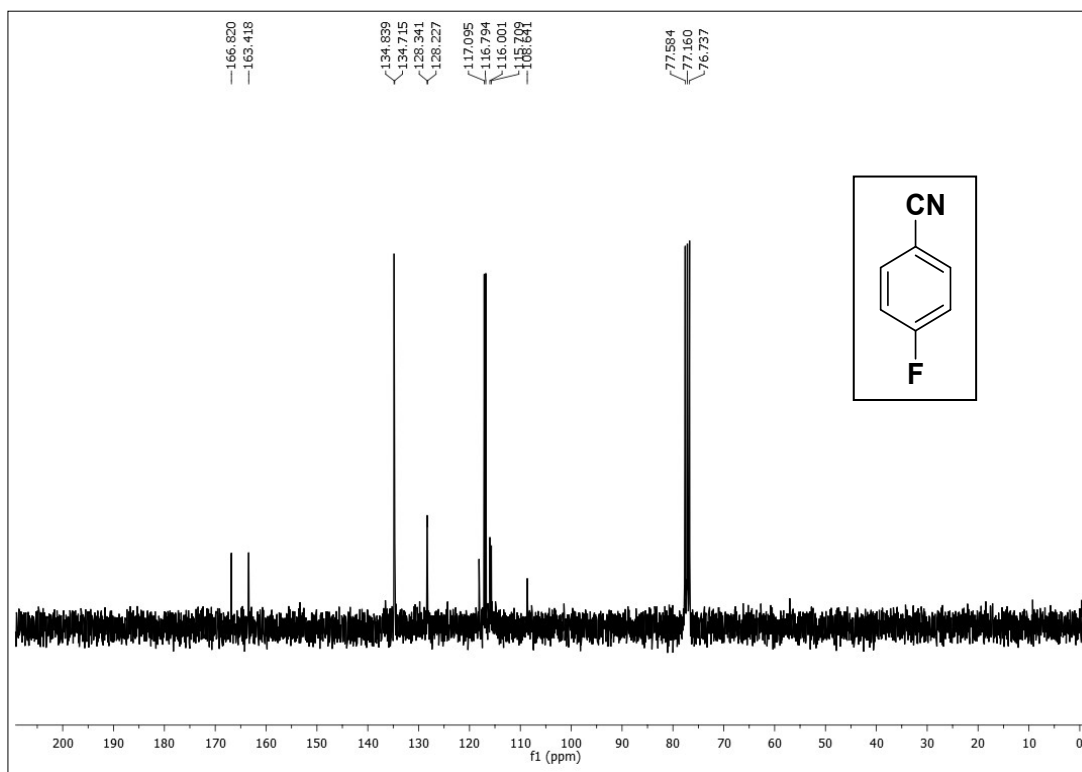
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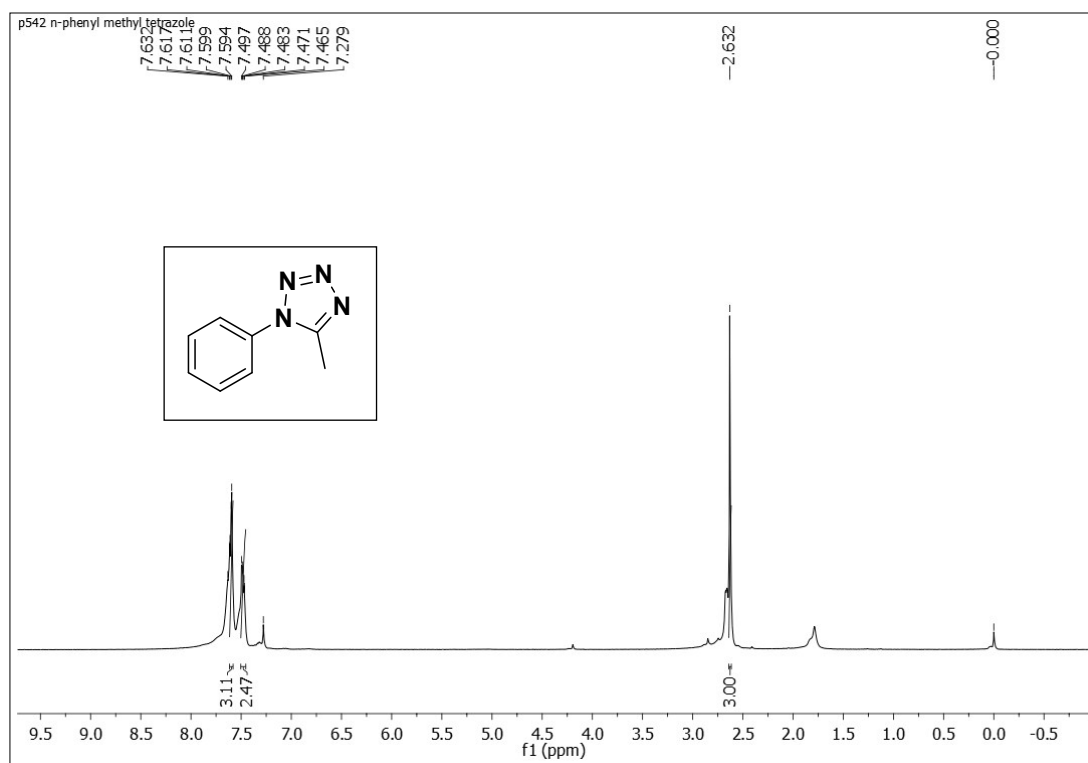
¹³C NMR(75 MHz, CDCl₃) spectrum of compound **2c**



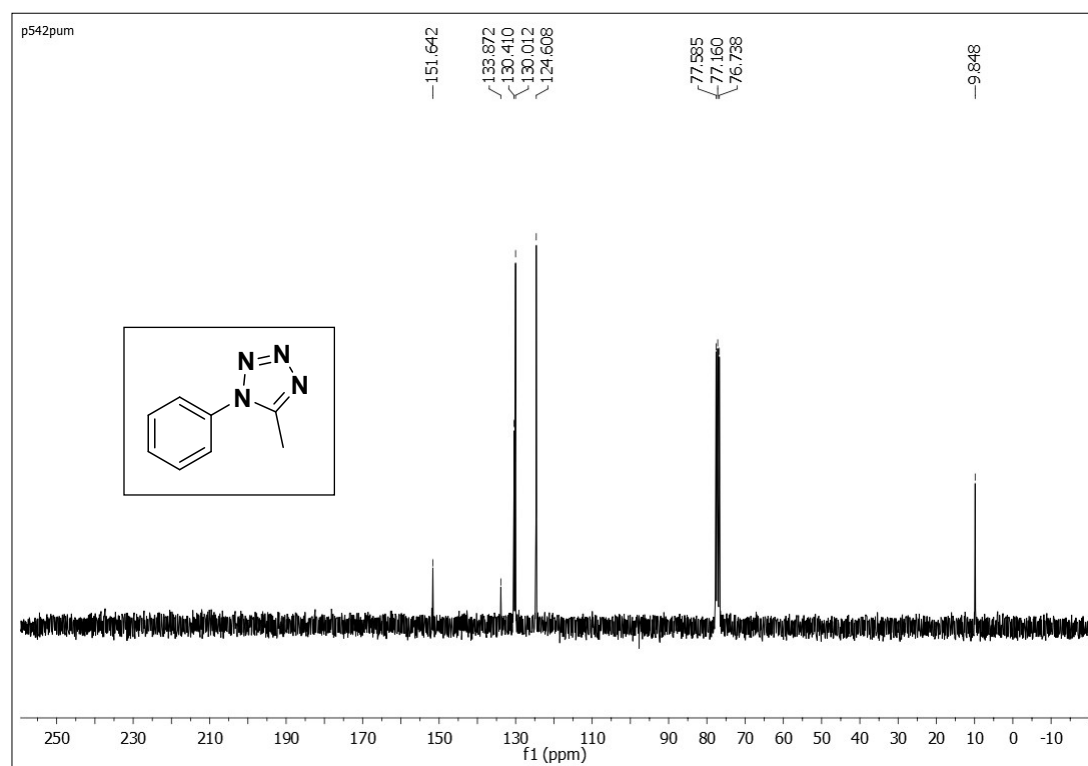
¹H NMR(300 MHz, CDCl₃) spectrum of compound **2d**



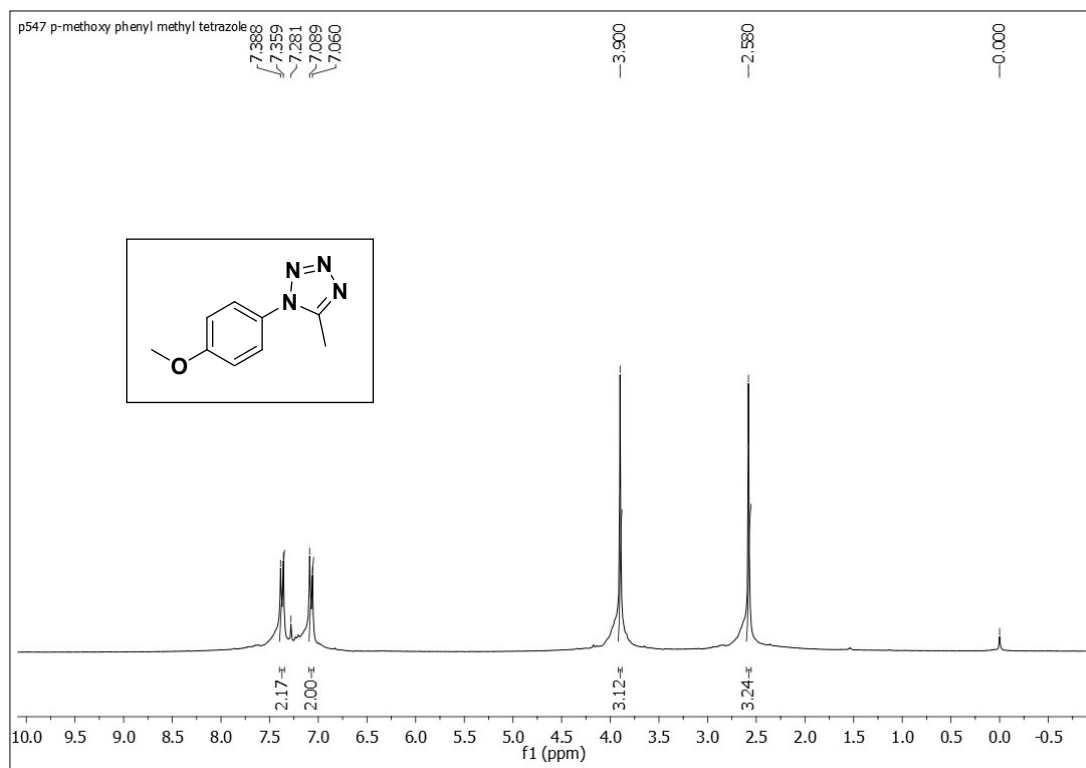
¹³C NMR(75 MHz, CDCl₃) spectrum of compound **2d**



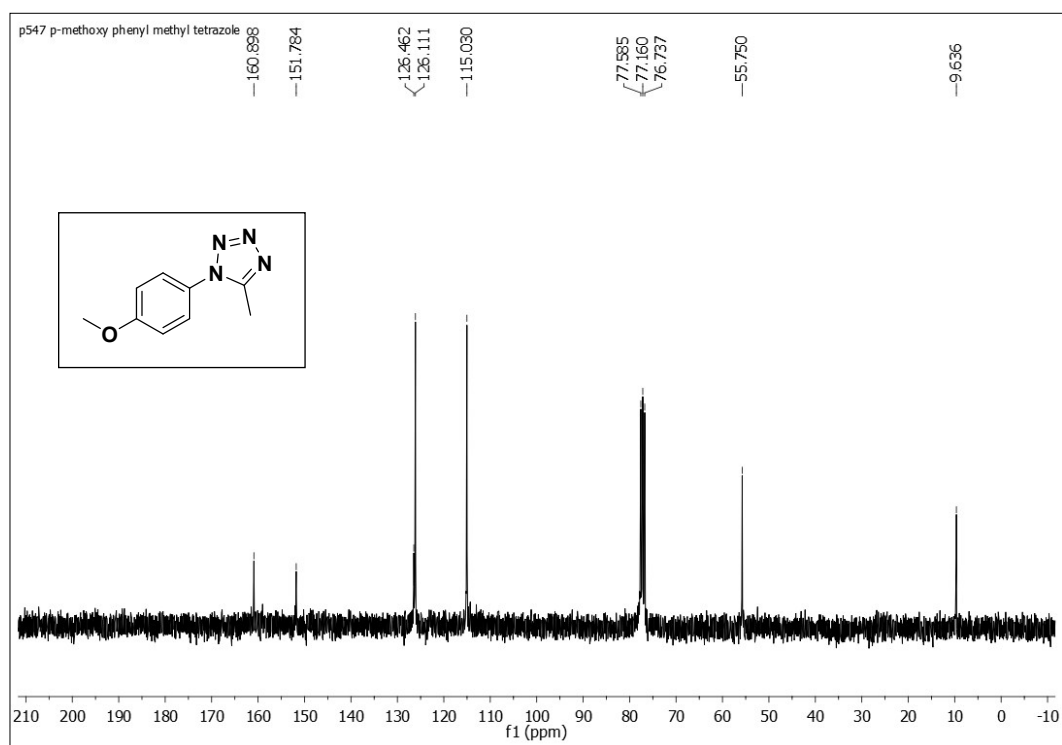
¹H NMR(300 MHz, CDCl₃) spectrum of compound **4a**



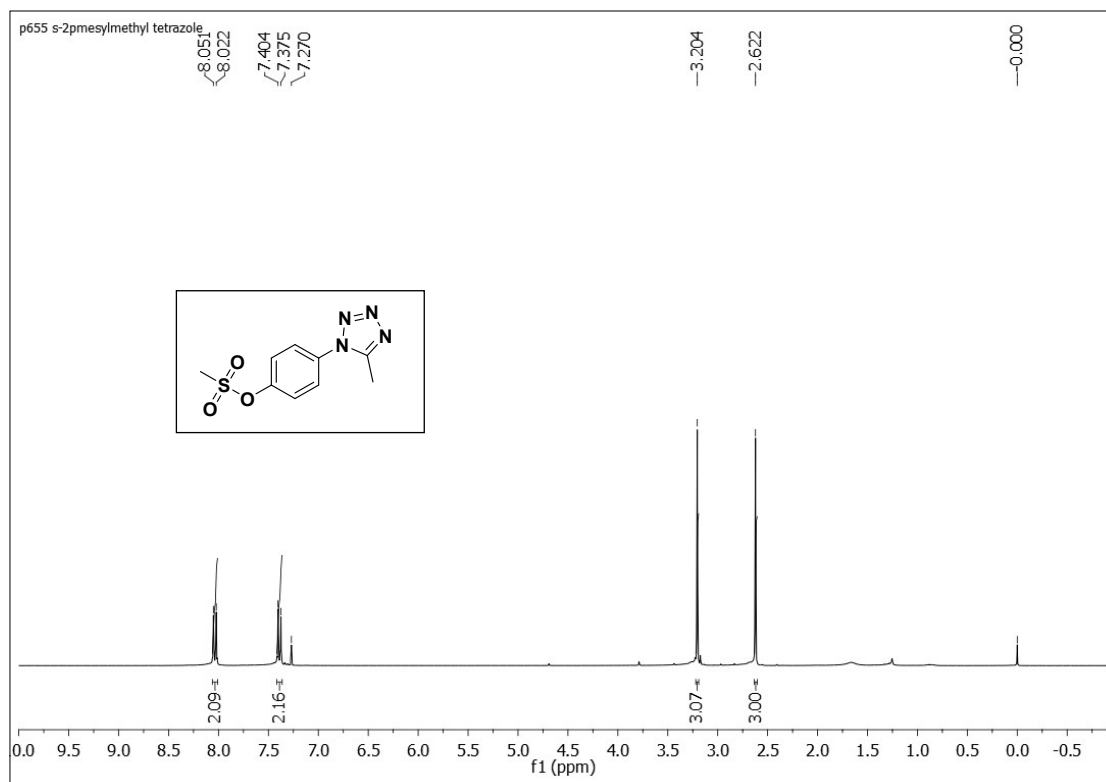
¹³C NMR(75 MHz, CDCl₃) spectrum of compound **4a**



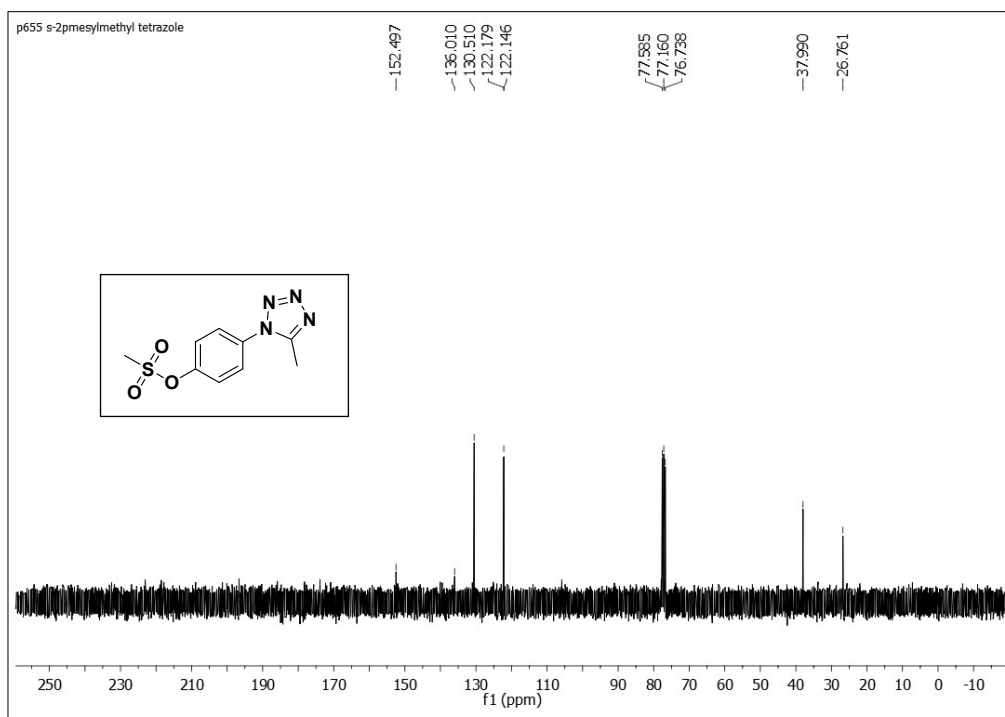
^1H NMR(300 MHz, CDCl_3) spectrum of compound **4b**



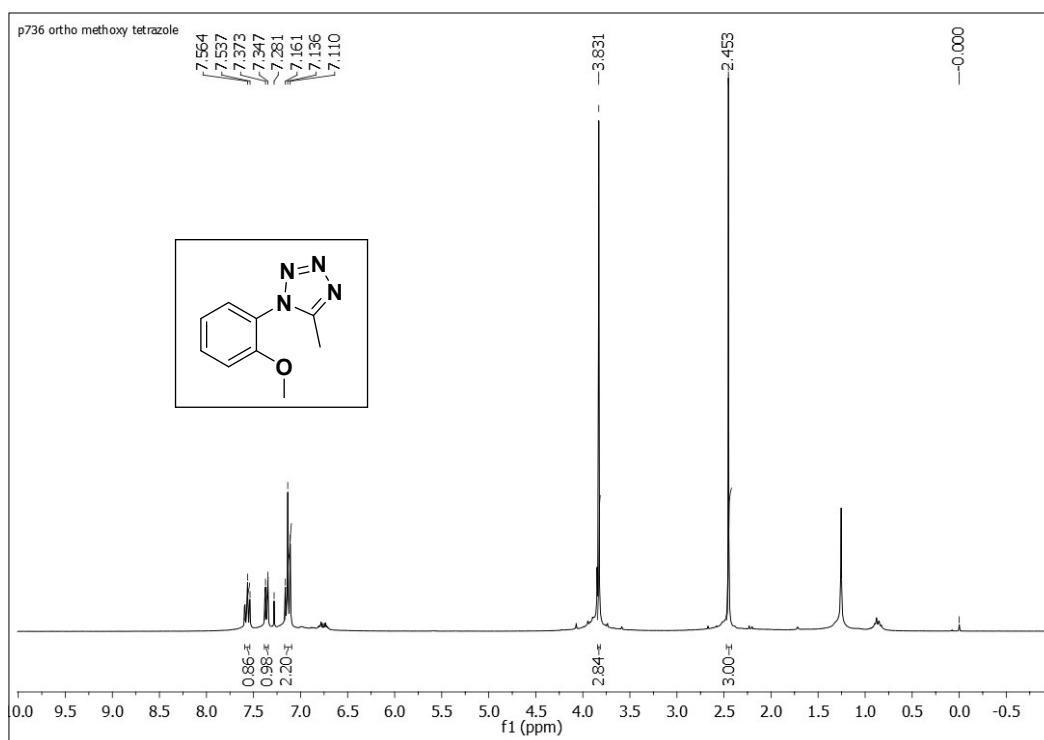
^{13}C NMR(75 MHz, CDCl_3) spectrum of compound **4b**



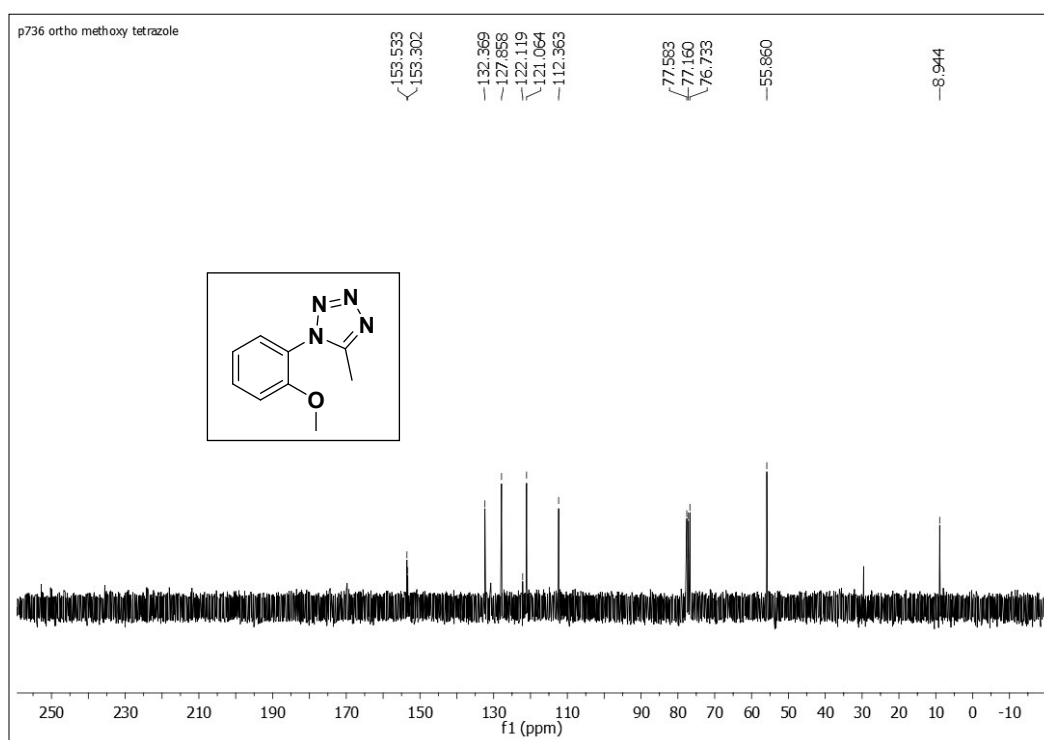
^1H NMR(300 MHz, CDCl_3) spectrum of compound **4e**



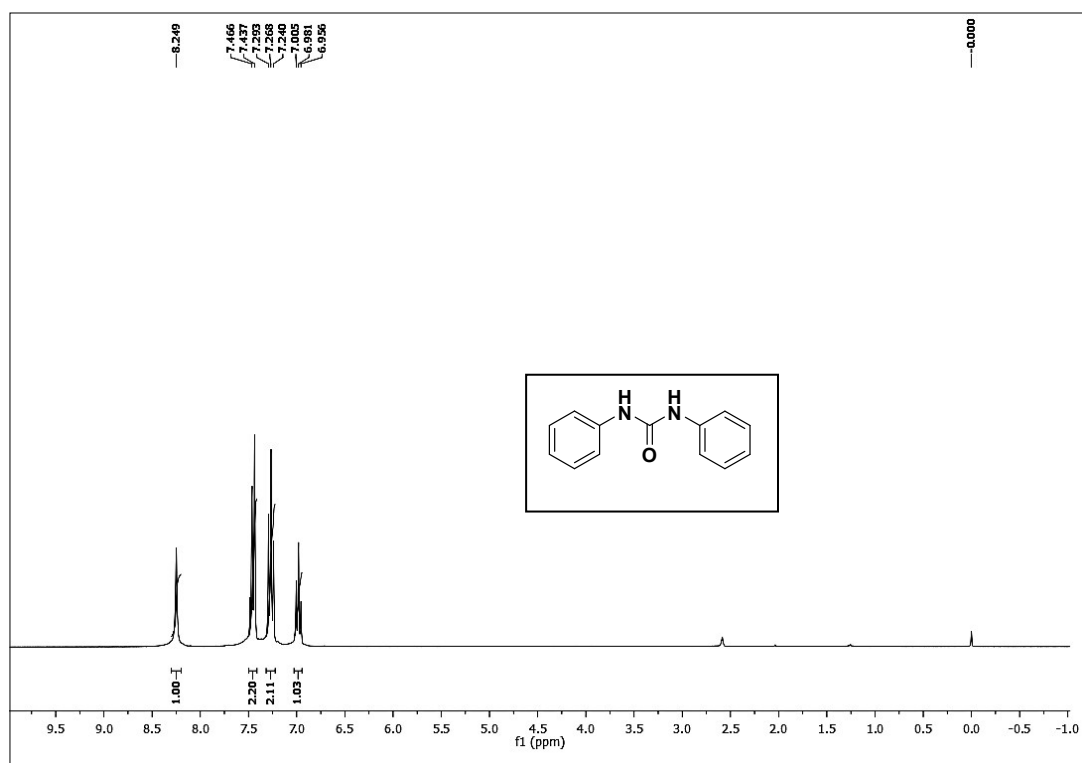
^{13}C NMR(75 MHz, CDCl_3) spectrum of compound **4e**



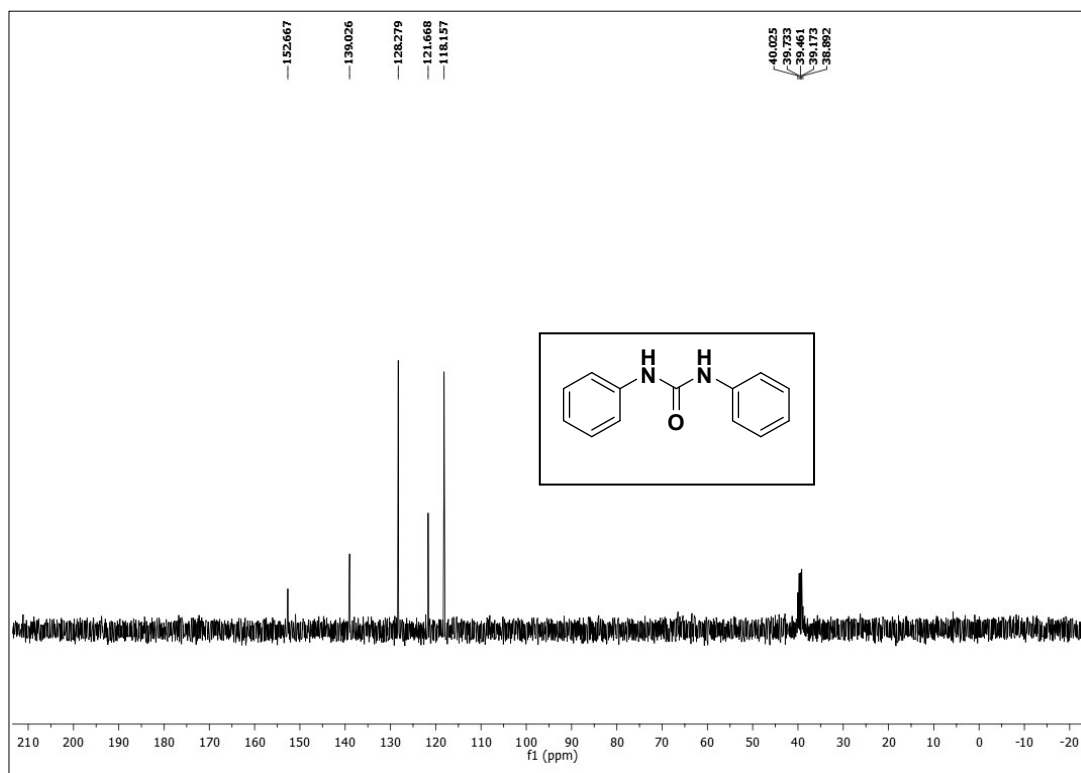
¹H NMR(300 MHz, CDCl₃) spectrum of compound **4g**



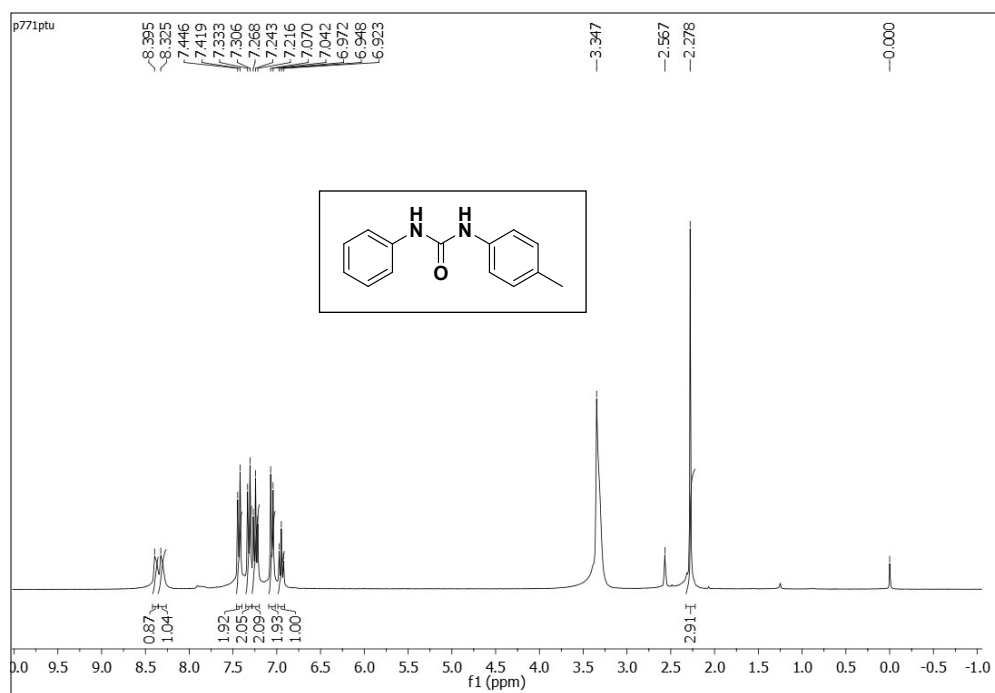
¹³C NMR(75 MHz, CDCl₃) spectrum of compound **4g**



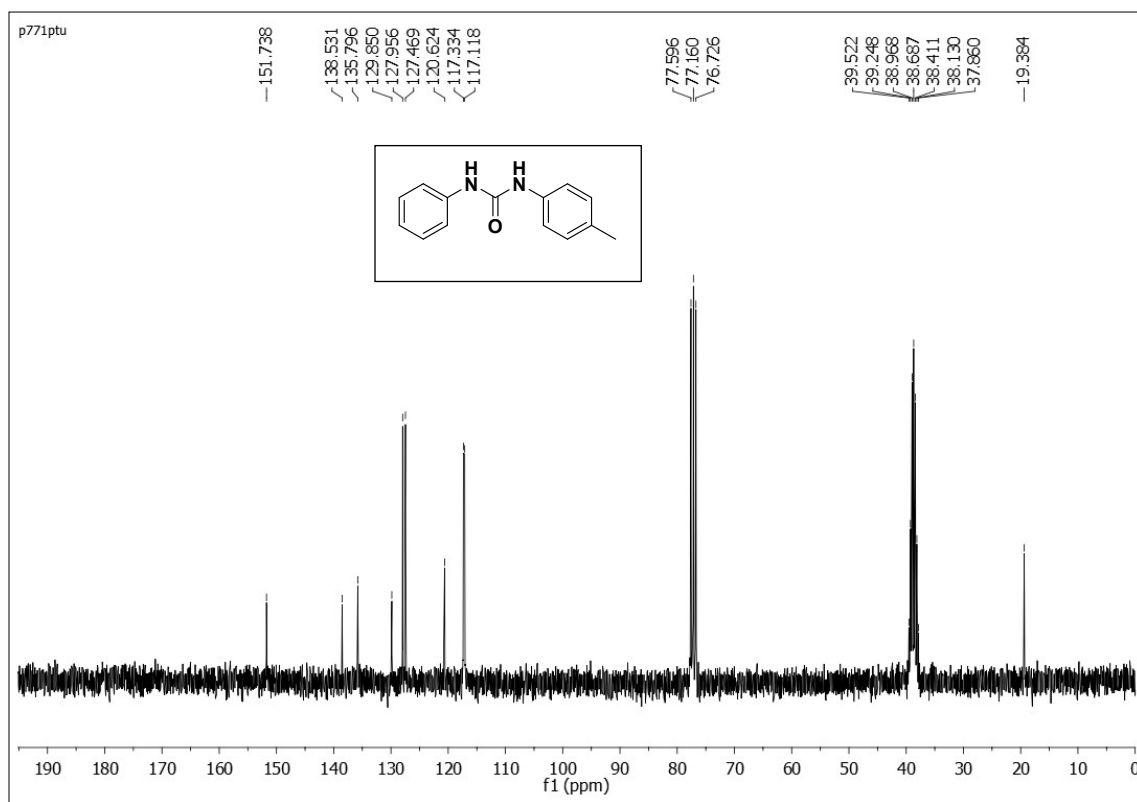
¹H NMR (300 MHz, DMSO-d₆) spectrum of compound **6a**



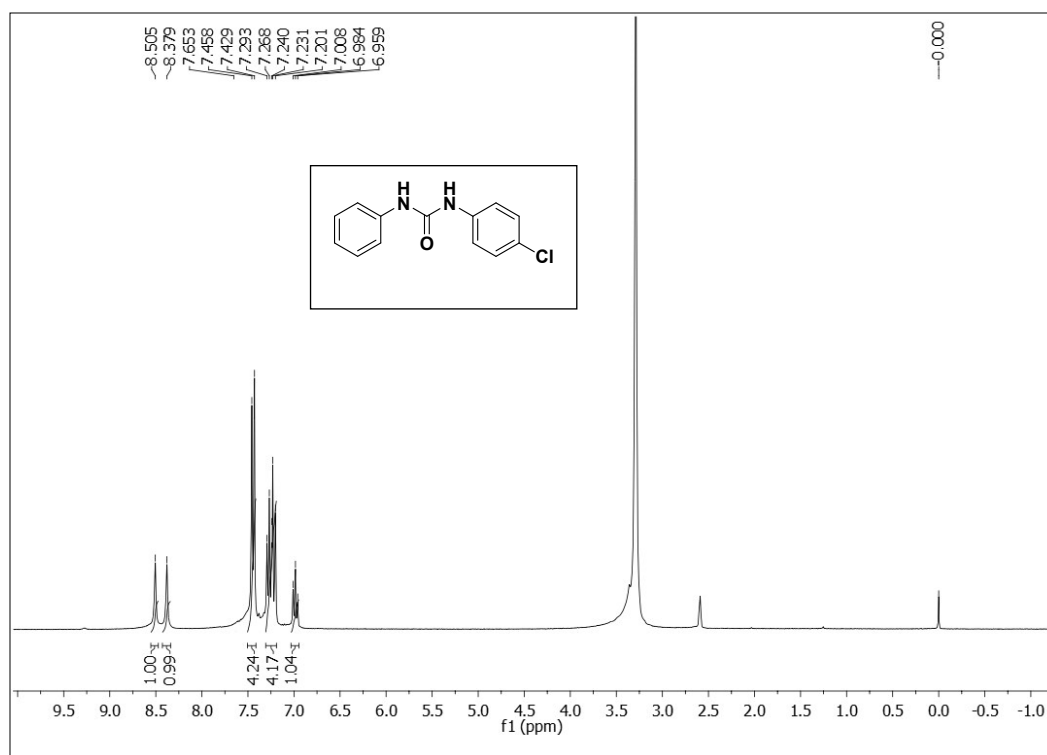
¹³C NMR (75 MHz, DMSO-d₆) spectrum of compound **6a**



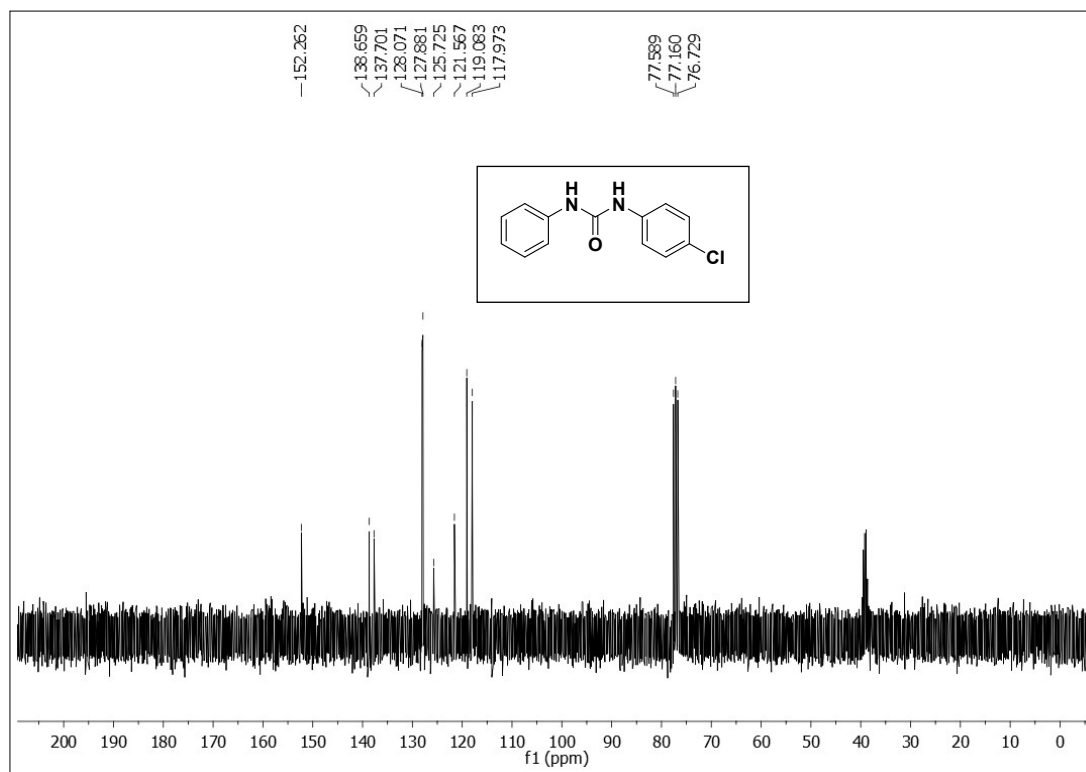
¹H NMR (300 MHz, CDCl₃) spectrum of compound **6b**



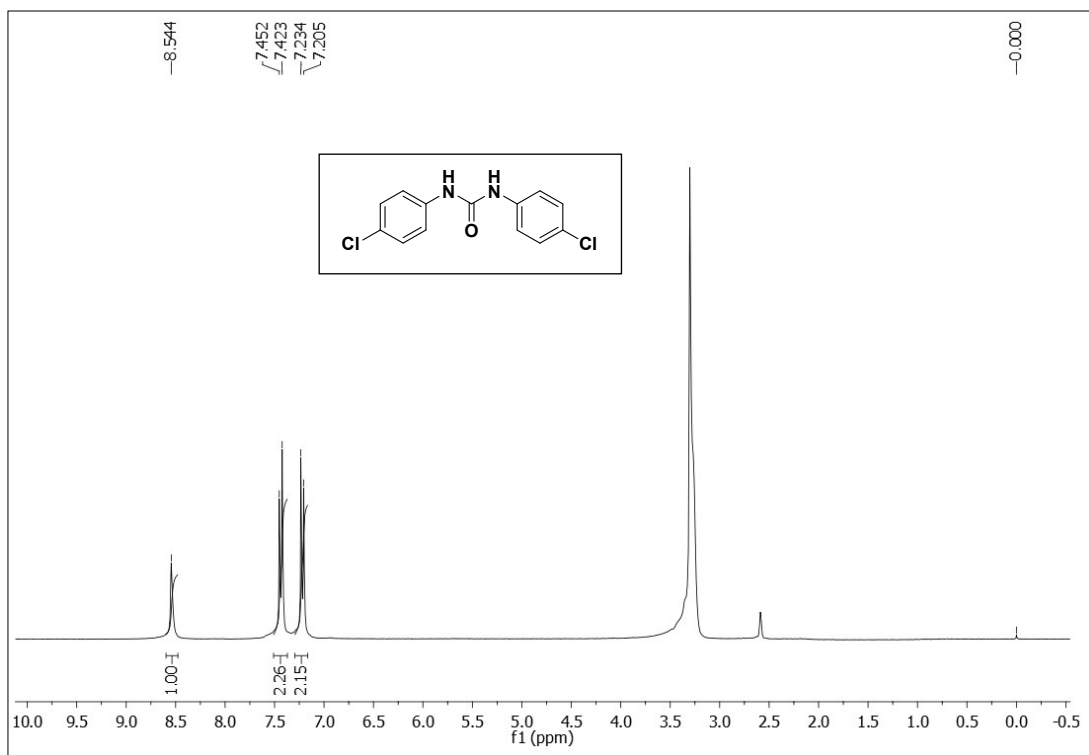
¹³C NMR (75 MHz, CDCl₃+DMSO-d₆) spectrum of compound **6b**



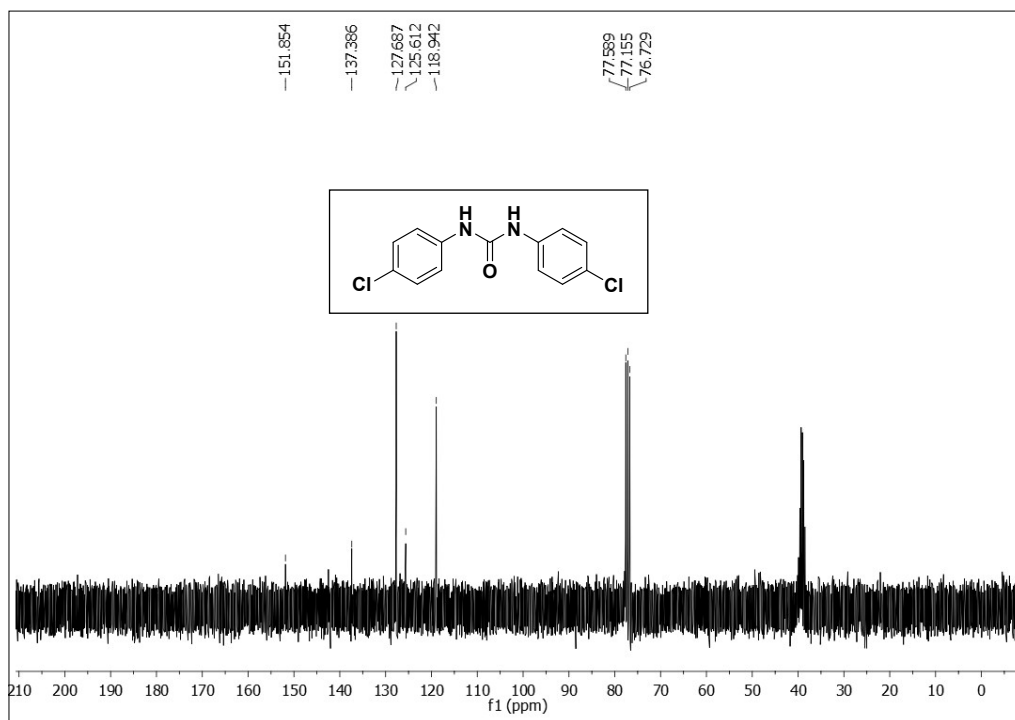
¹H NMR (300 MHz, CDCl₃+DMSO-d₆) spectrum of compound **6c**



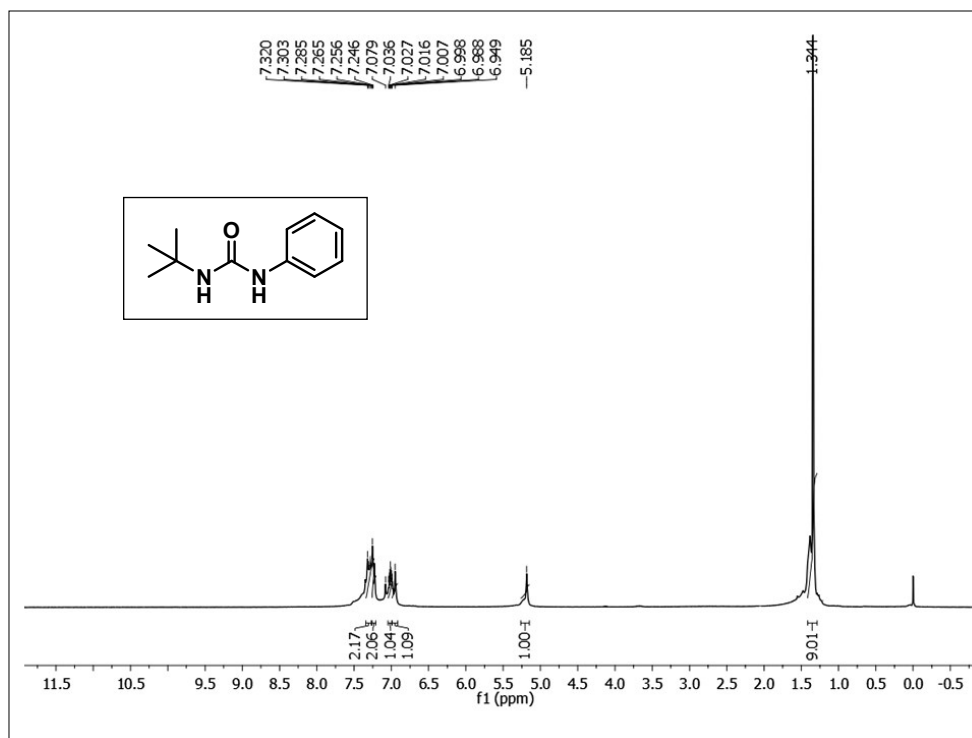
¹³C NMR (75 MHz, CDCl₃+DMSO-d₆) spectrum of compound **6c**



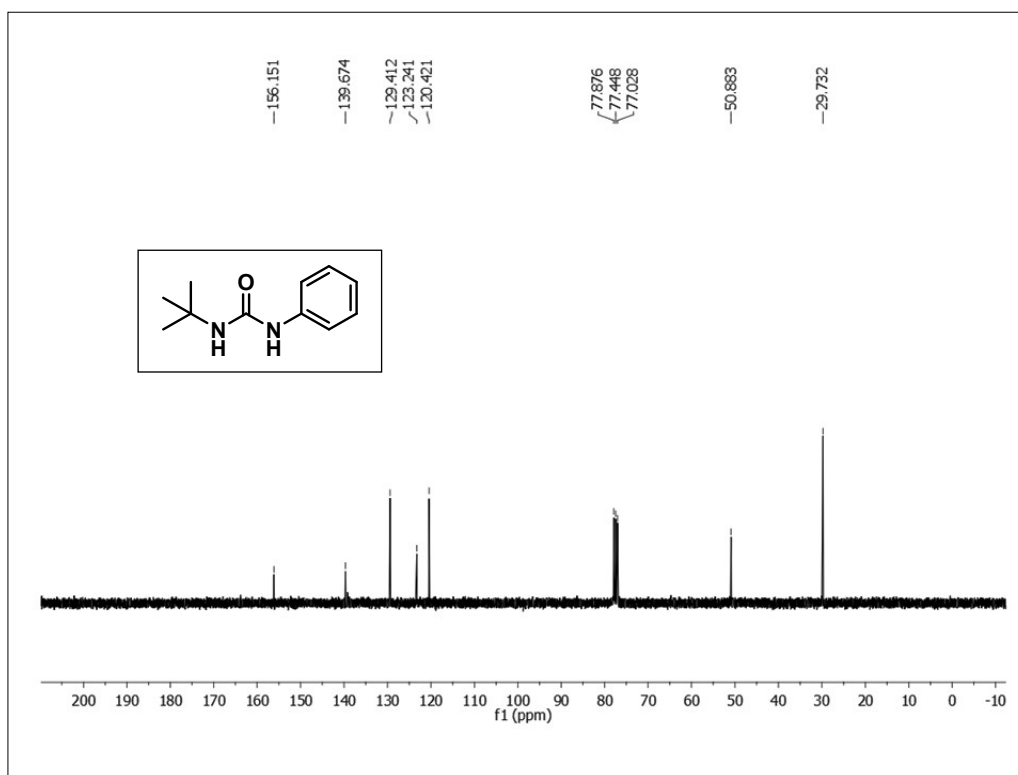
¹H NMR (300 MHz, CDCl₃+DMSO-d₆) spectrum of compound **6d**



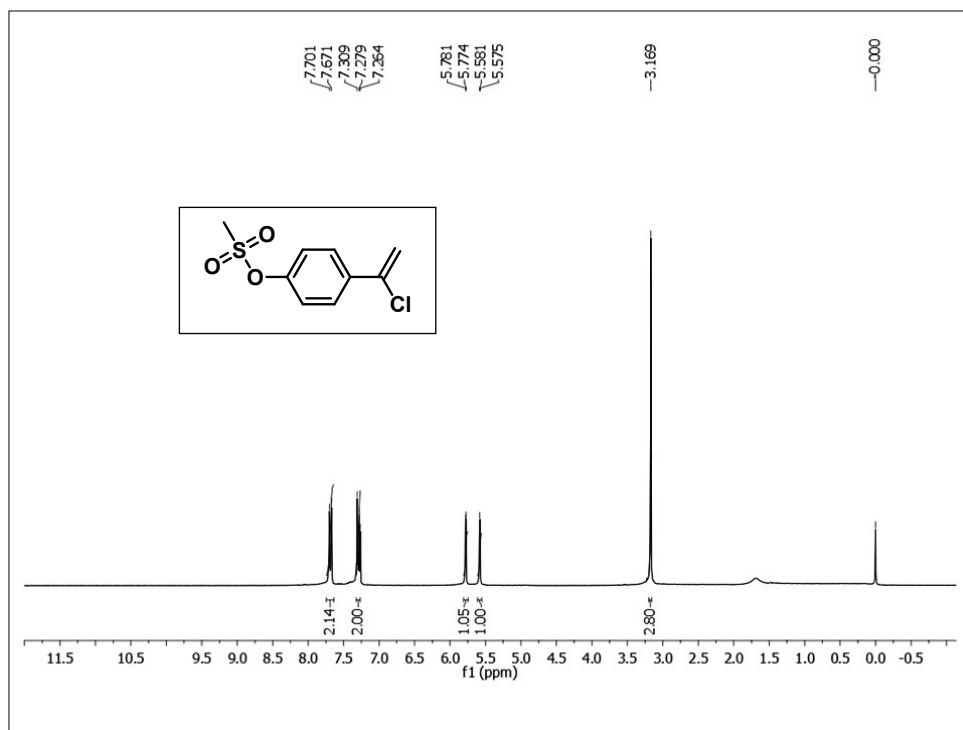
¹³C NMR (75 MHz, CDCl₃+DMSO-d₆) spectrum of compound **6d**



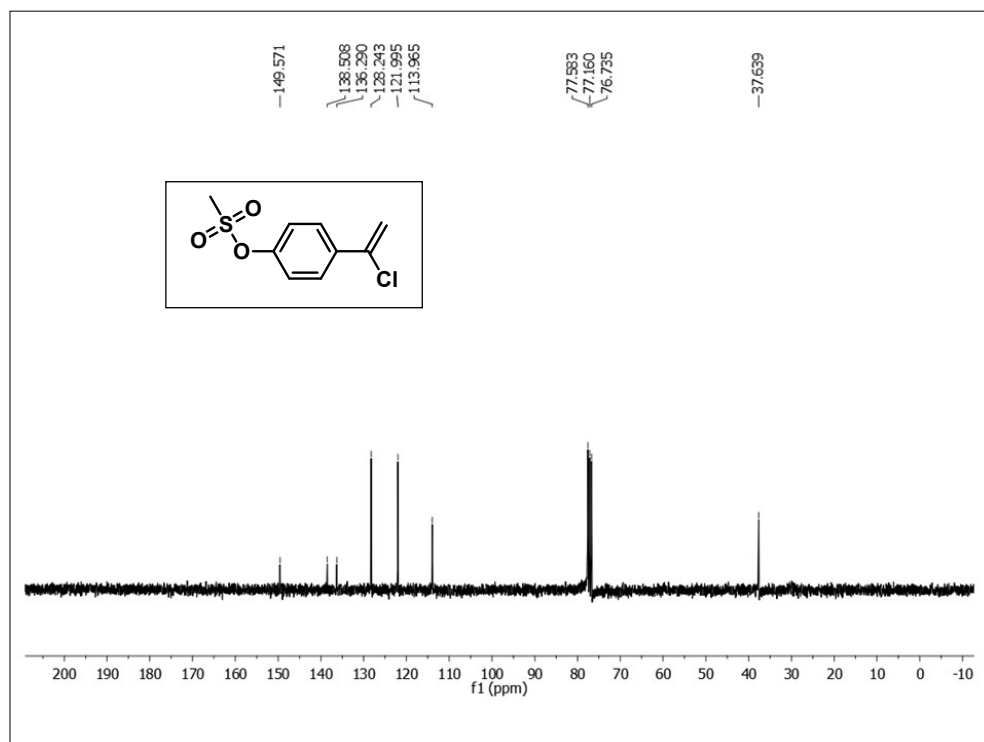
¹H NMR (300 MHz, CDCl₃) spectrum of compound 6I



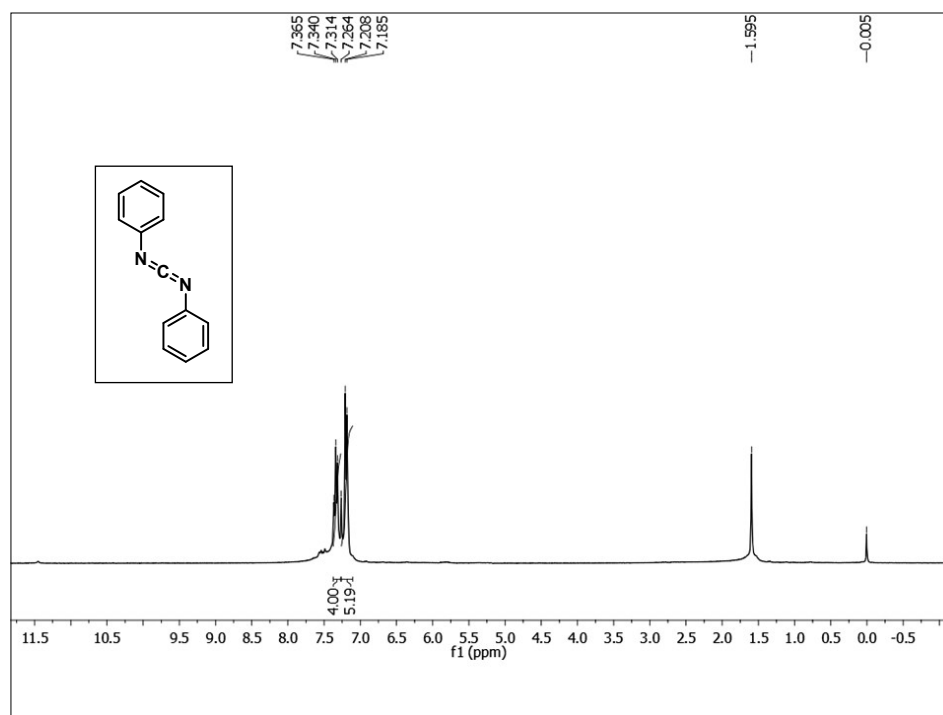
¹³C NMR (75 MHz, CDCl₃) spectrum of compound 6I



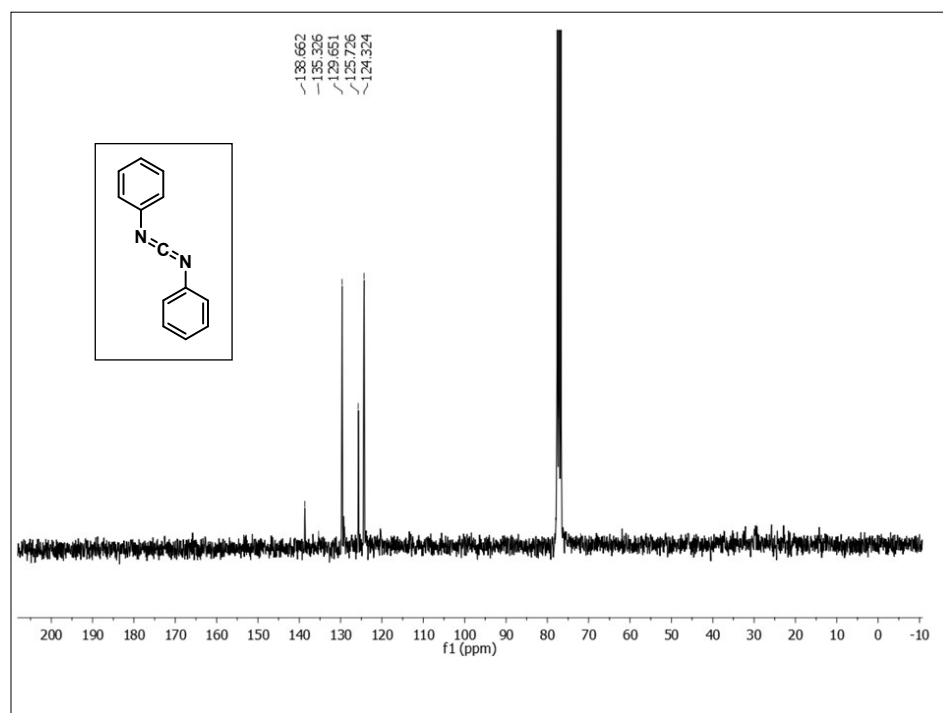
¹H NMR (300 MHz, CDCl₃) spectrum of Intermediate IV



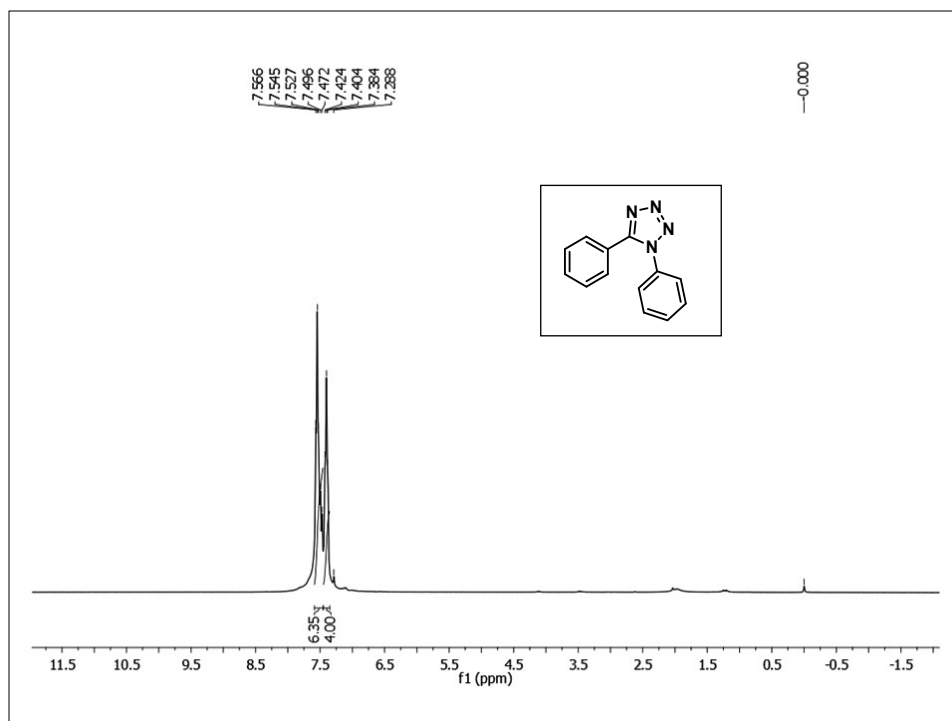
¹³C NMR (75 MHz, CDCl₃) spectrum of Intermediate IV



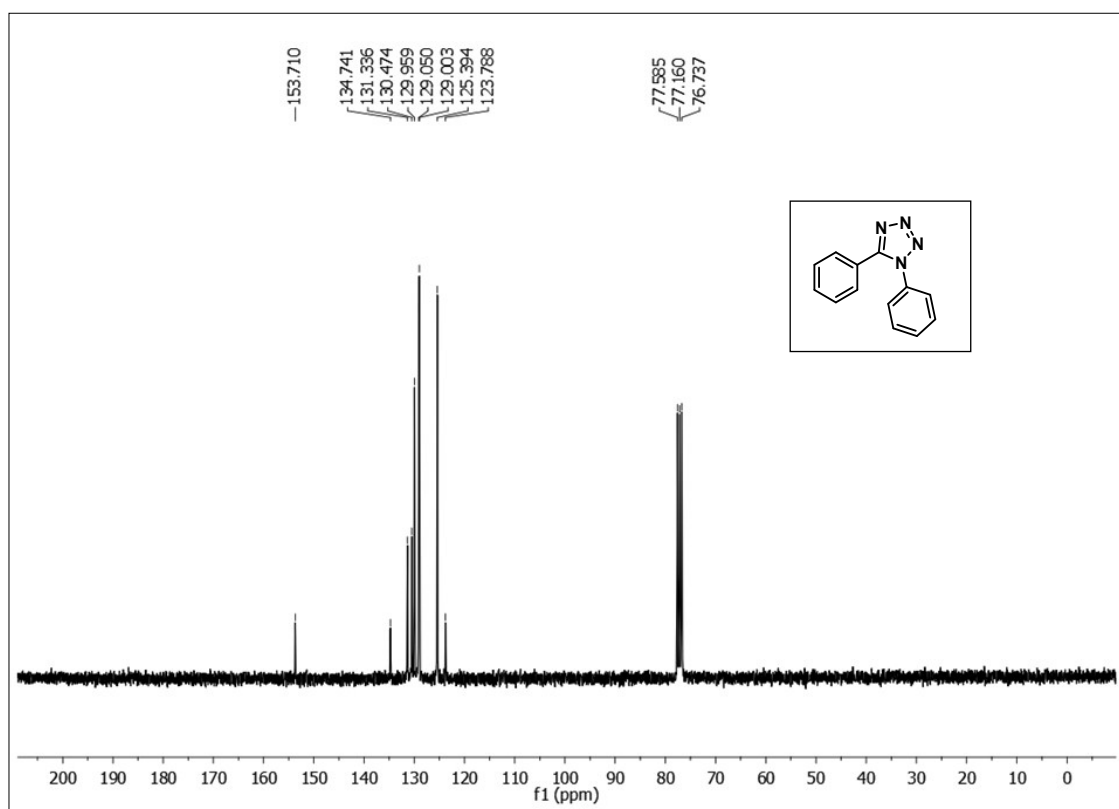
^1H NMR (300 MHz, CDCl_3) spectrum of Intermediate XII



^{13}C NMR (75 MHz, CDCl_3) spectrum of Intermediate XII



¹H NMR (300MHz, CDCl₃) Spectrum of diphenyltetrazole (minor product)



¹³C NMR (300MHz, CDCl₃) Spectrum of diphenyltetrazole (minor product)