Electronic Supplementary Material (ESI) for RSC Advances.

Supppрting Information

Highly Water-dispersible Magnetite Nanoparticles Supported-Palladium-β-Cyclodextrin as Efficient Catalyst for Suzuki-Miyaura and Sonogashira Coupling Reaction

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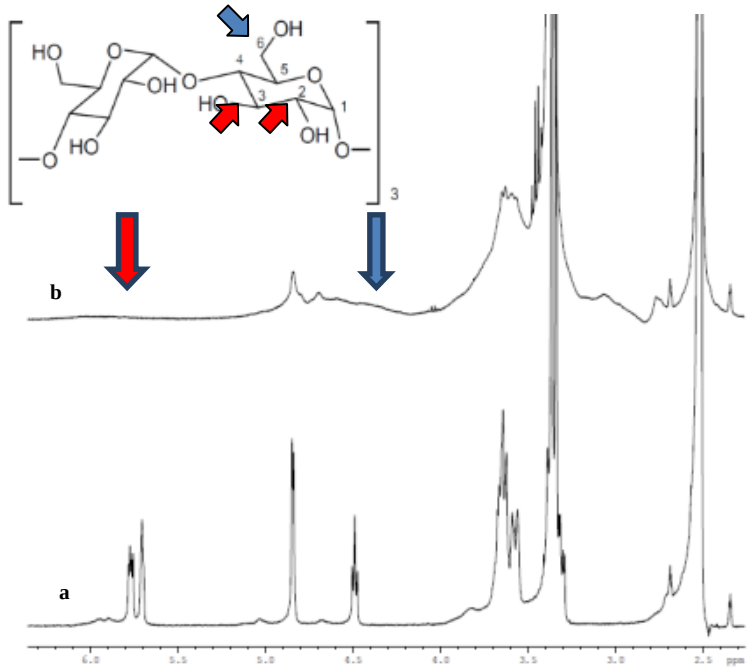


Figure s1. ^{1}H NMR spectrum (400 MHz) of (a) β-cyclodextrin and (b) Pd(II)-β-CD

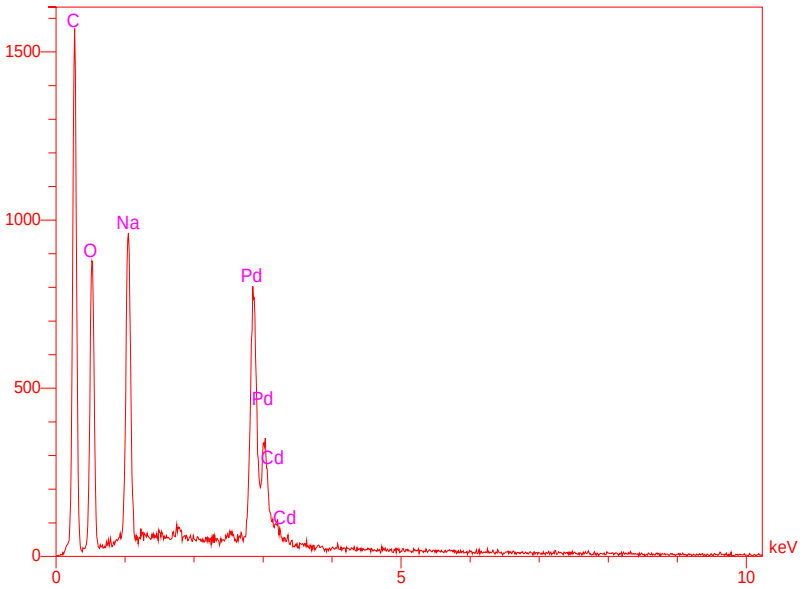


Figure s2. EDS spectrum of Pd(II)-β-CD complex

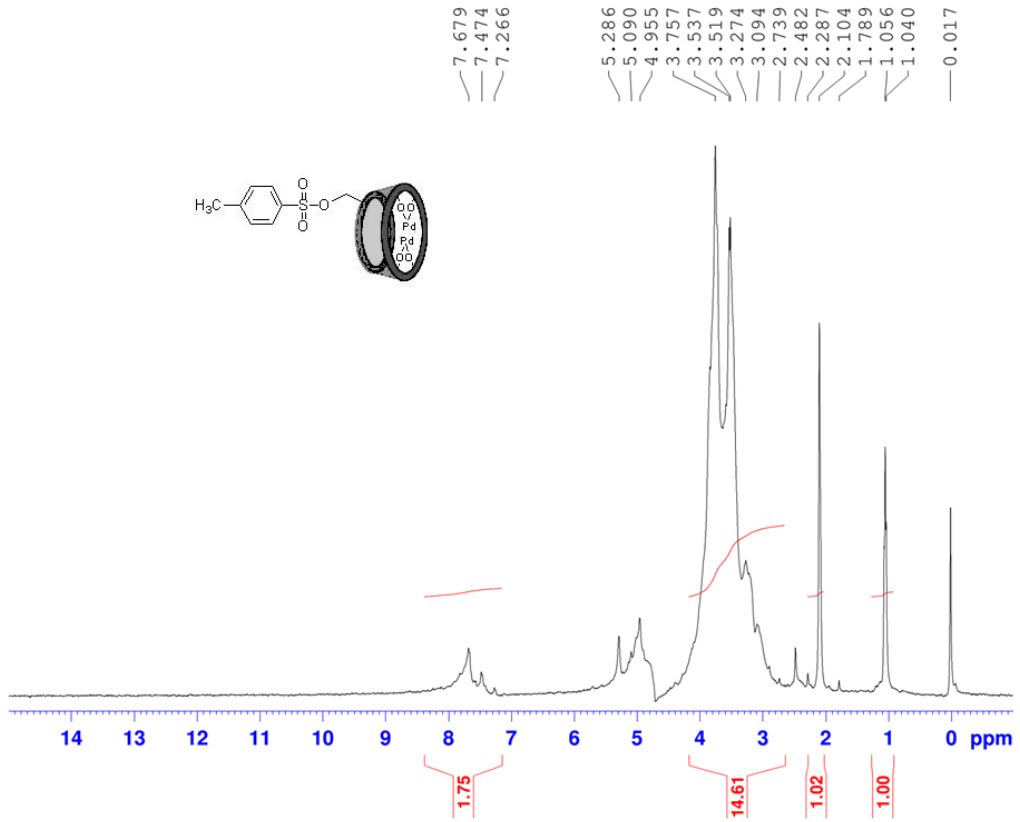


Figure s3. ^{1}H-NMR (400 MHz, D_2O): δ= 7.2-7.6 (aromatic hydrogens of p-toluene sulfunyl group)

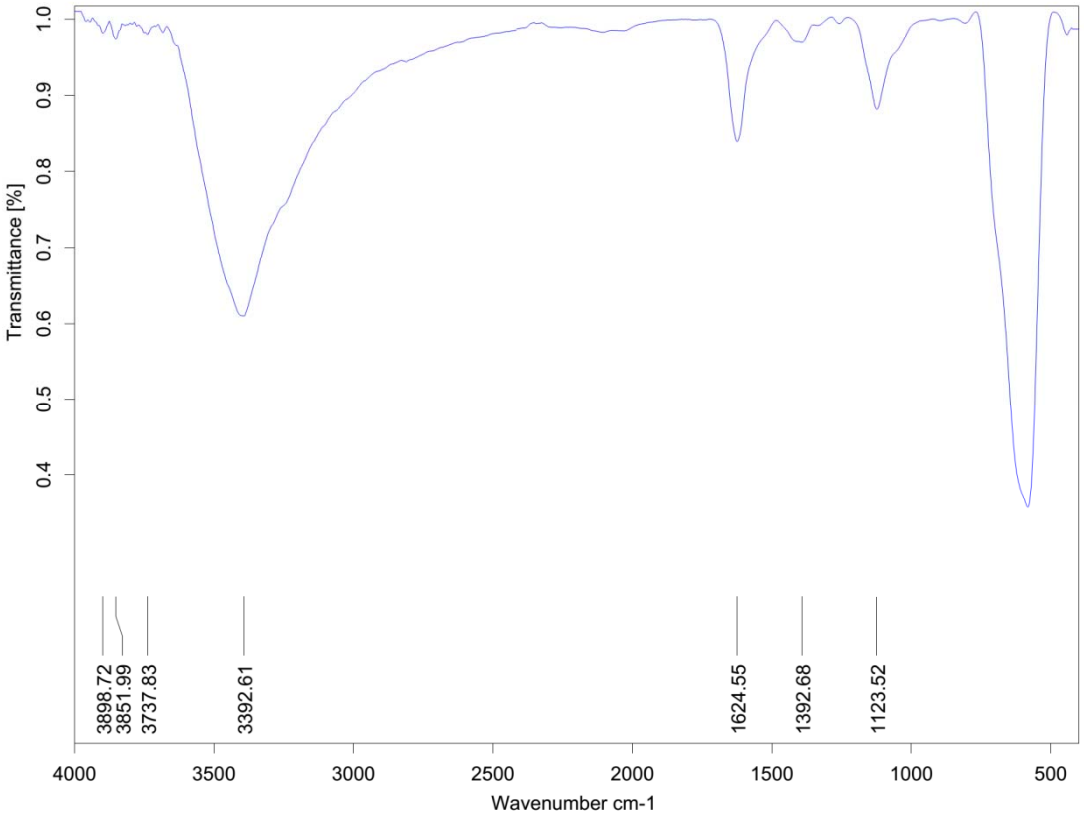


Figure s4. FT-IR: magnetite nanoparticles (MNP)

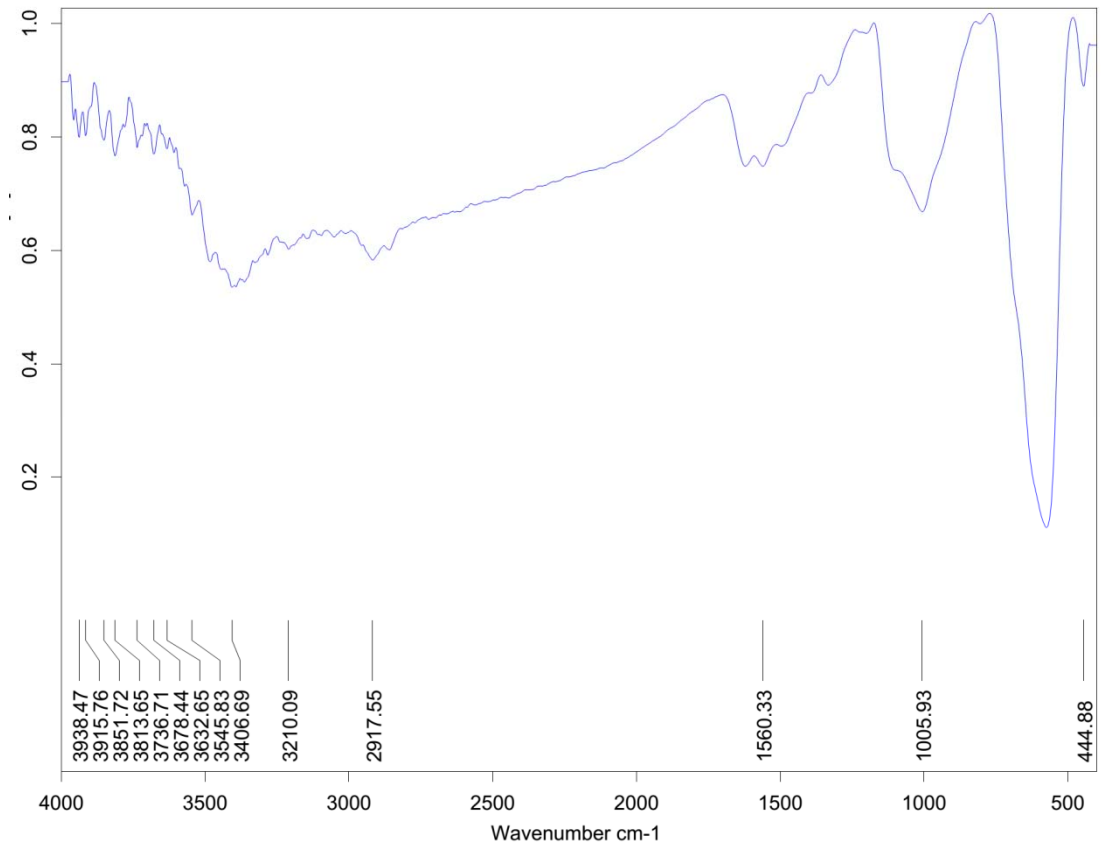


Figure s5. FT-IR: 3-aminopropyl triethoxy silane functionalized magnetite nanoparticles (MNP-AP)

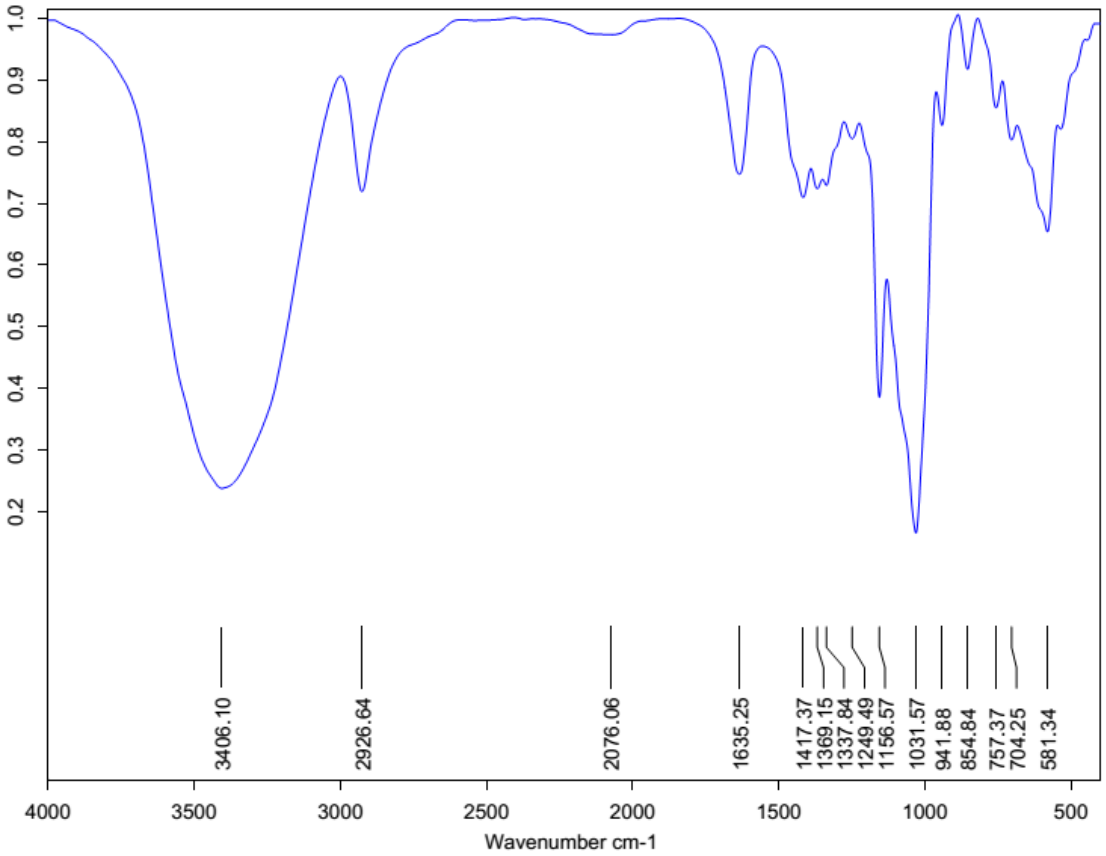


Figure s6. FT-IR: magnetite nanoparticle supported palladium-□-cyclodextrin (MNP-CD-Pd)

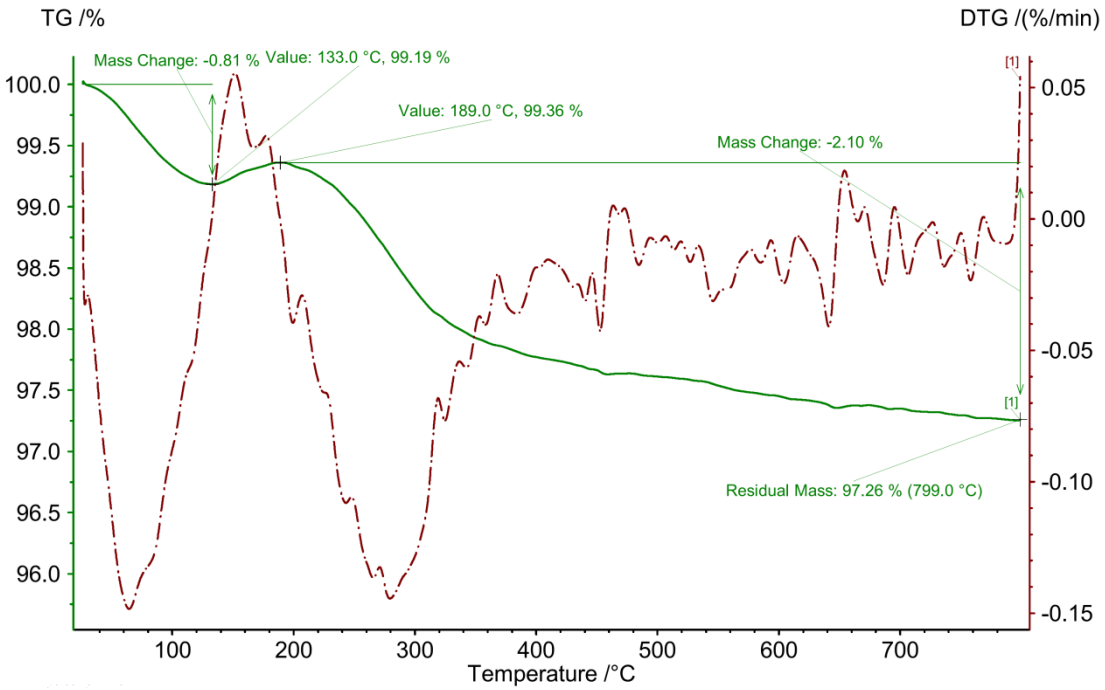


Figure s7. TG/DTG: MNP

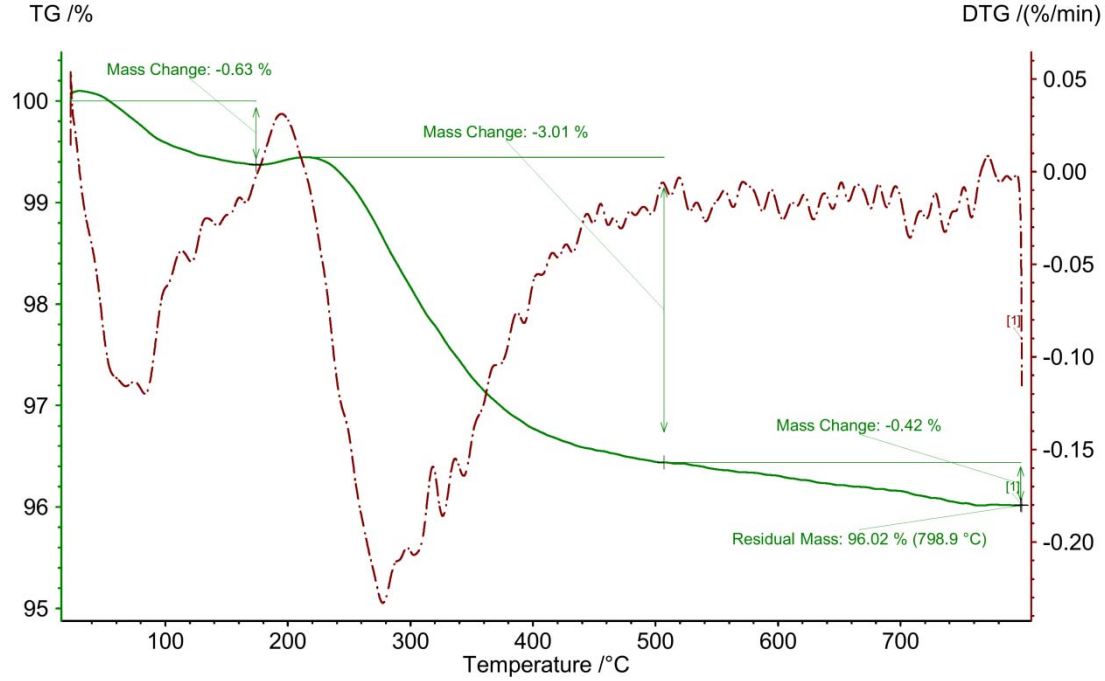


Figure s8. TG/DTG: MNP-AP

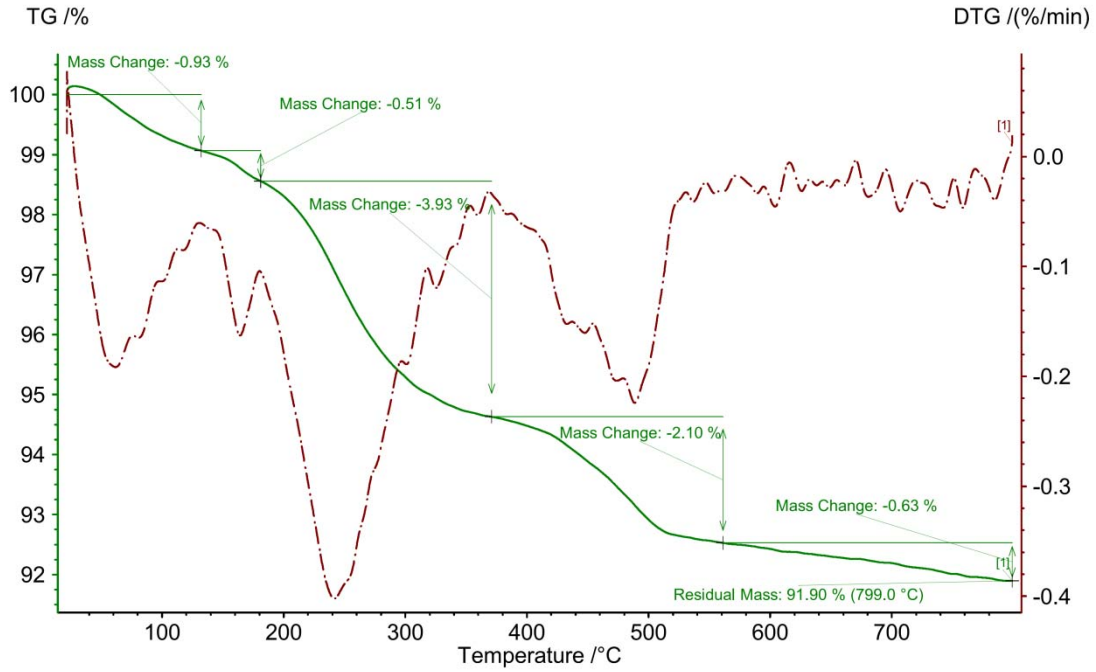


Figure s9. TG/DTG: MNP-CD-Pd

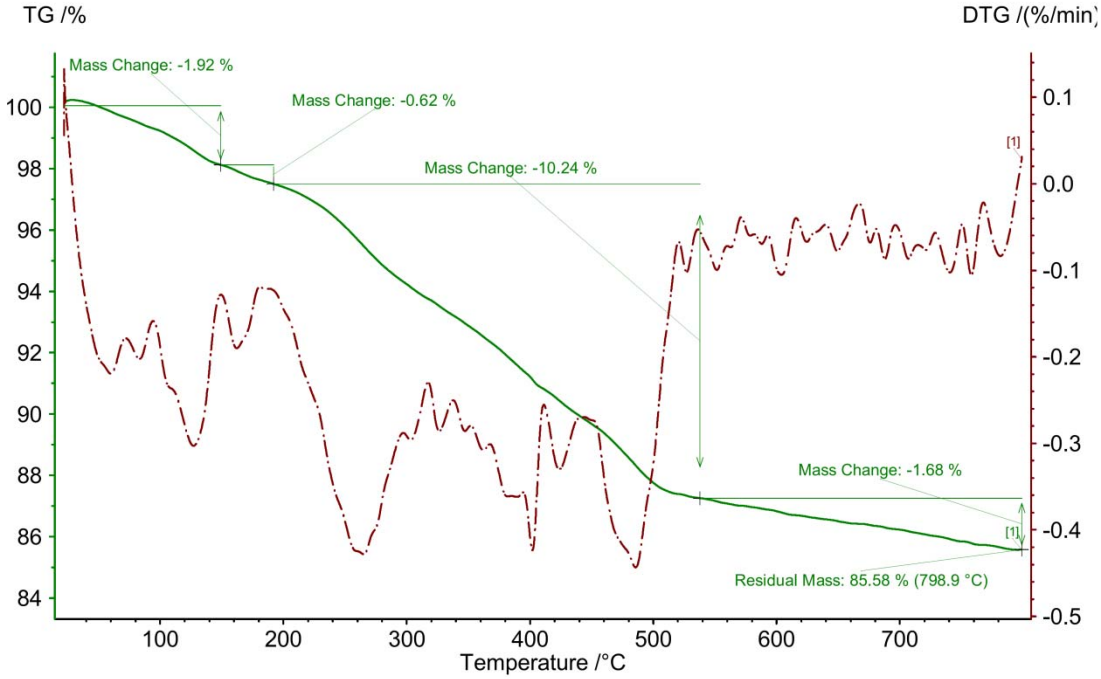


Figure s10. TG/DTG: Recycled MNP-CD-Pd

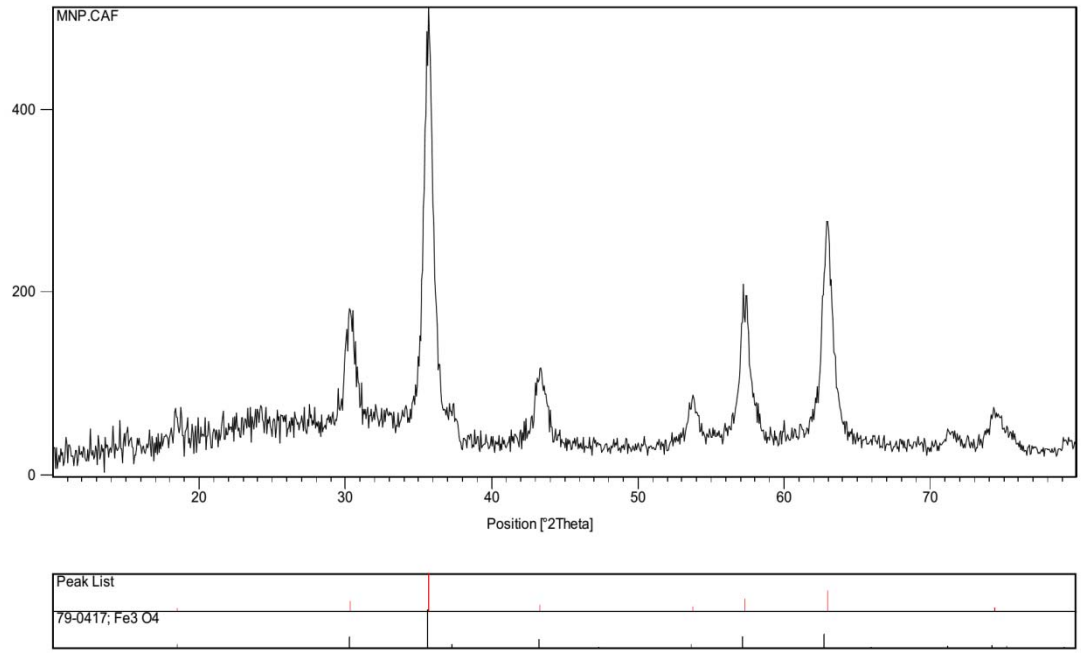


Figure s11. XRD: magnetite nanoparticles

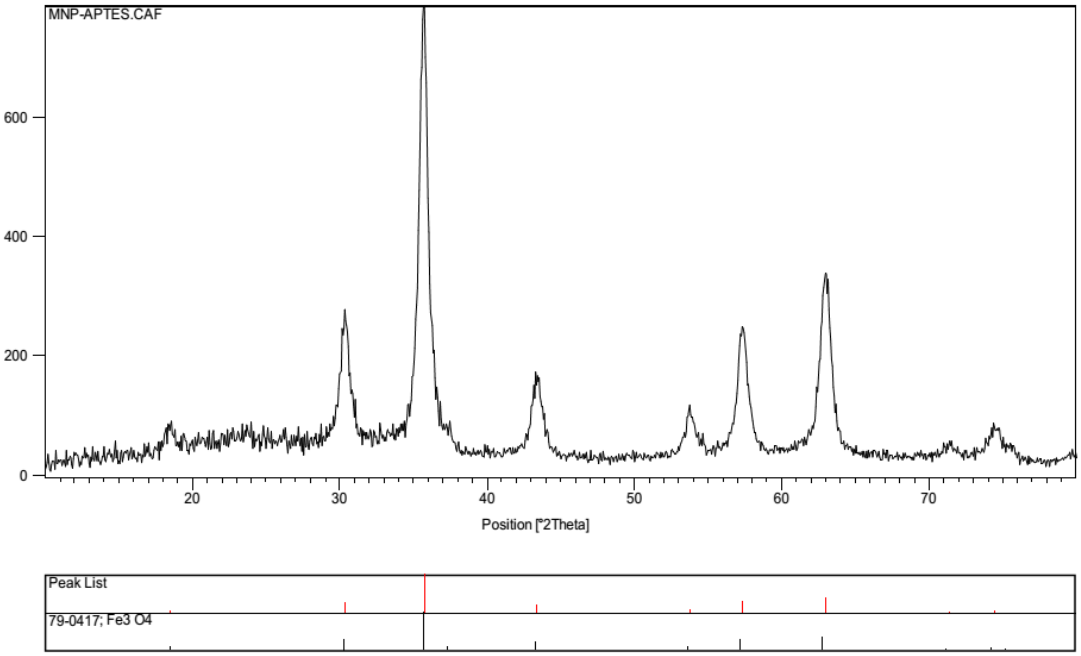


Figure s12. XRD: MNP-AP

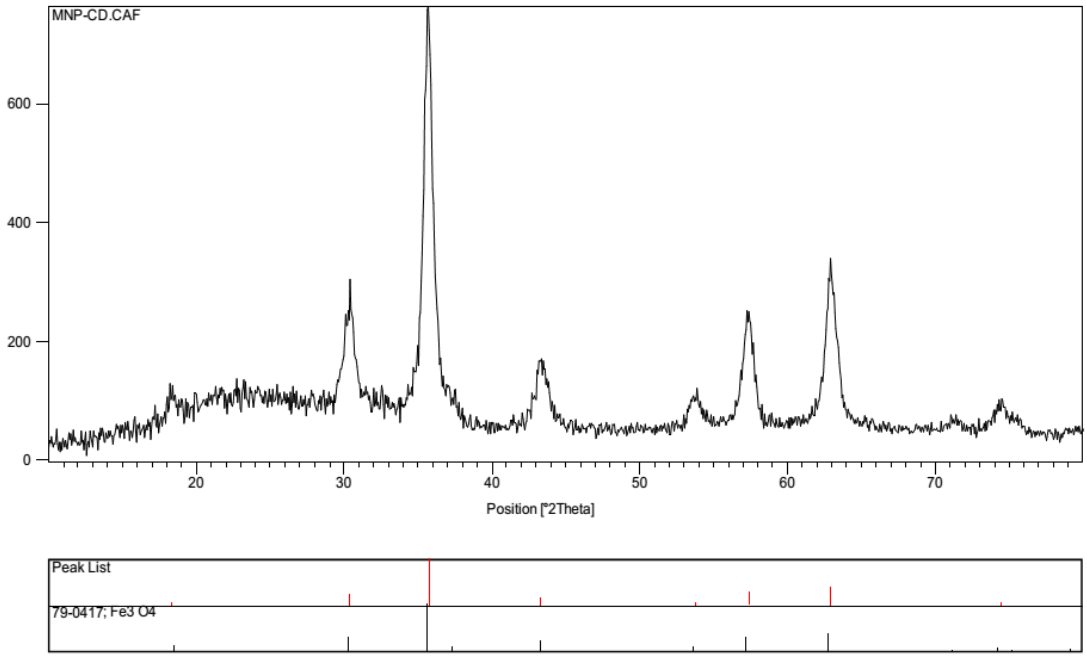


Figure s13. XRD: MNP-CD-Pd

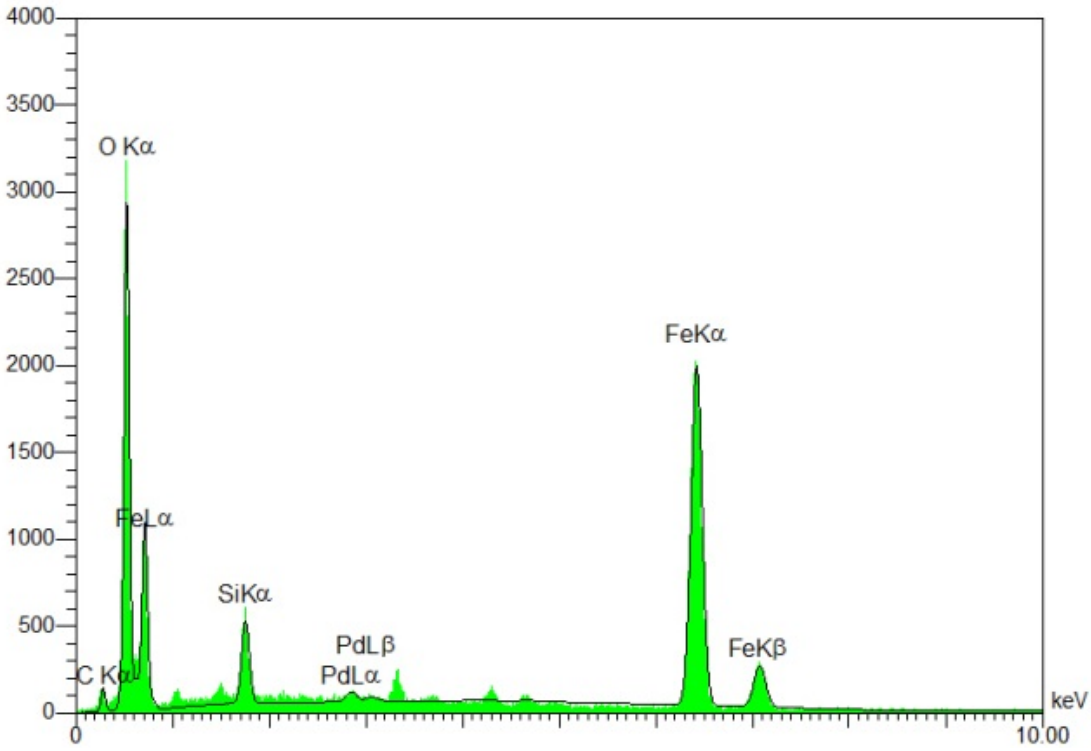
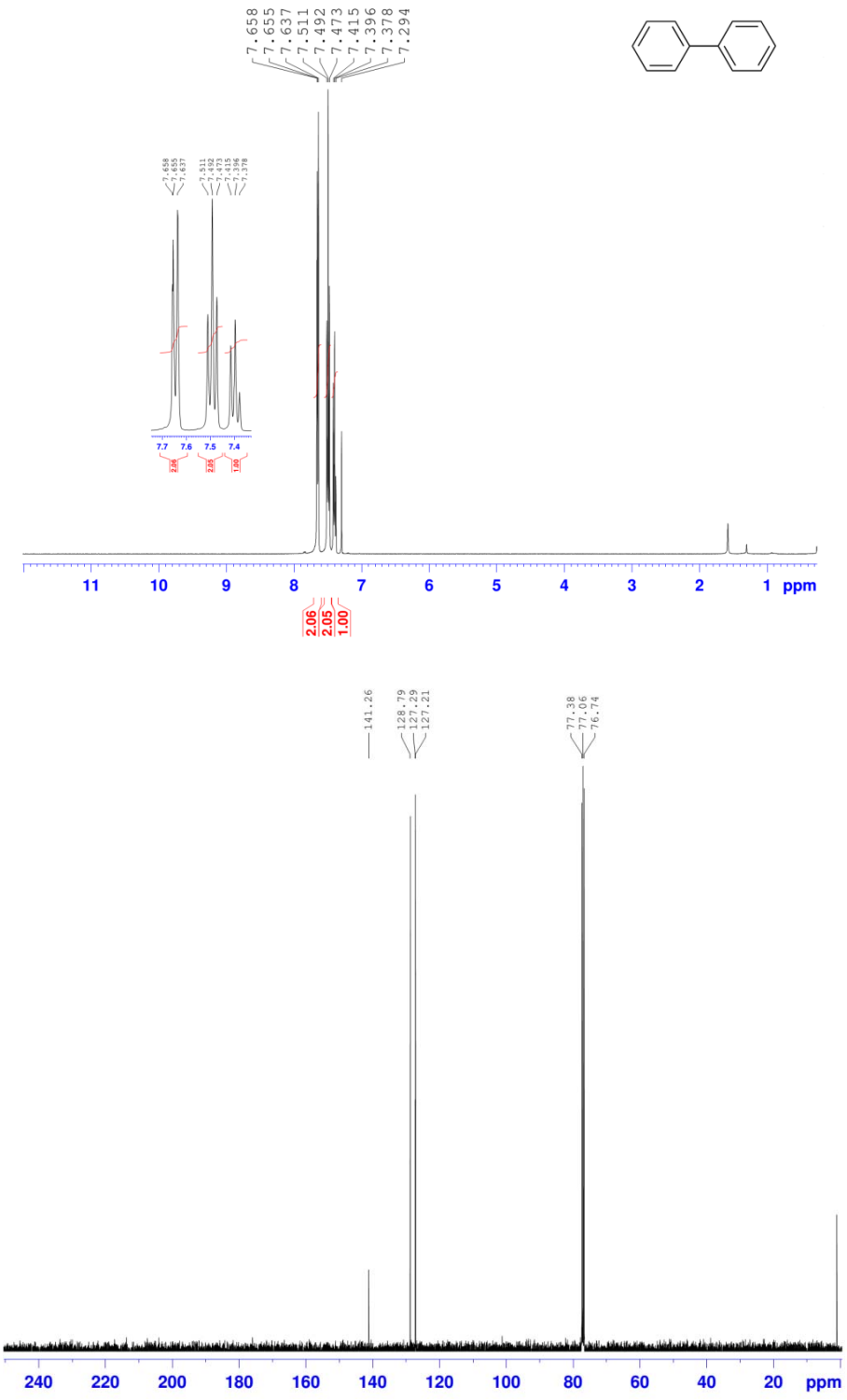
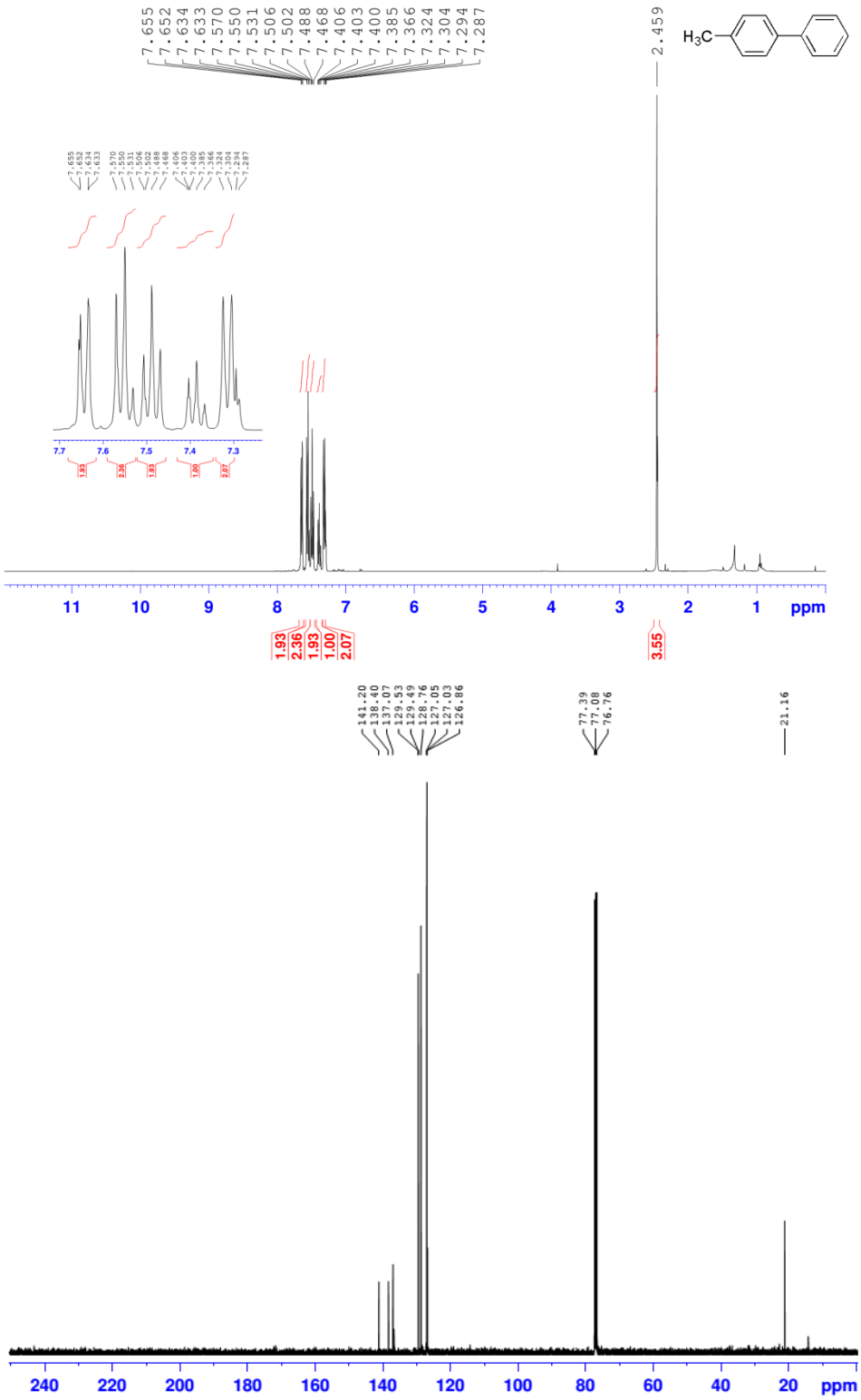


Figure s14. EDS spectrum of recycled catalyst after fifth run



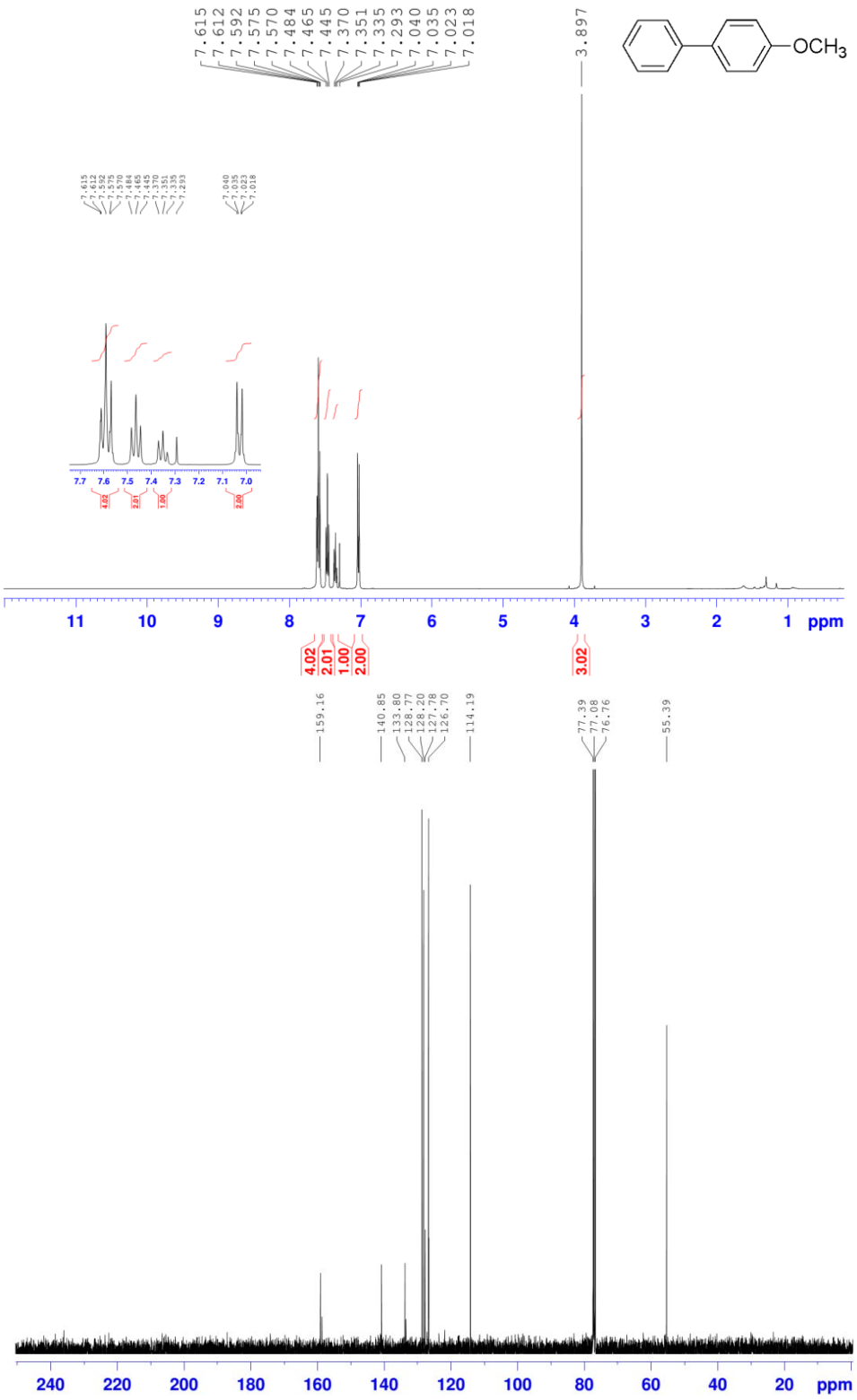
Biphenyl (3b):

^{1}H-NMR (400 MHz, $CDCl_3$): δ= 7.39 (2H, t), 7.49 (4H, t), 7.65 (4H, d).
^{13}C-NMR (100 MHz, $CDCl_3$): δ= 127.2, 127.3, 128.8, 141.2.



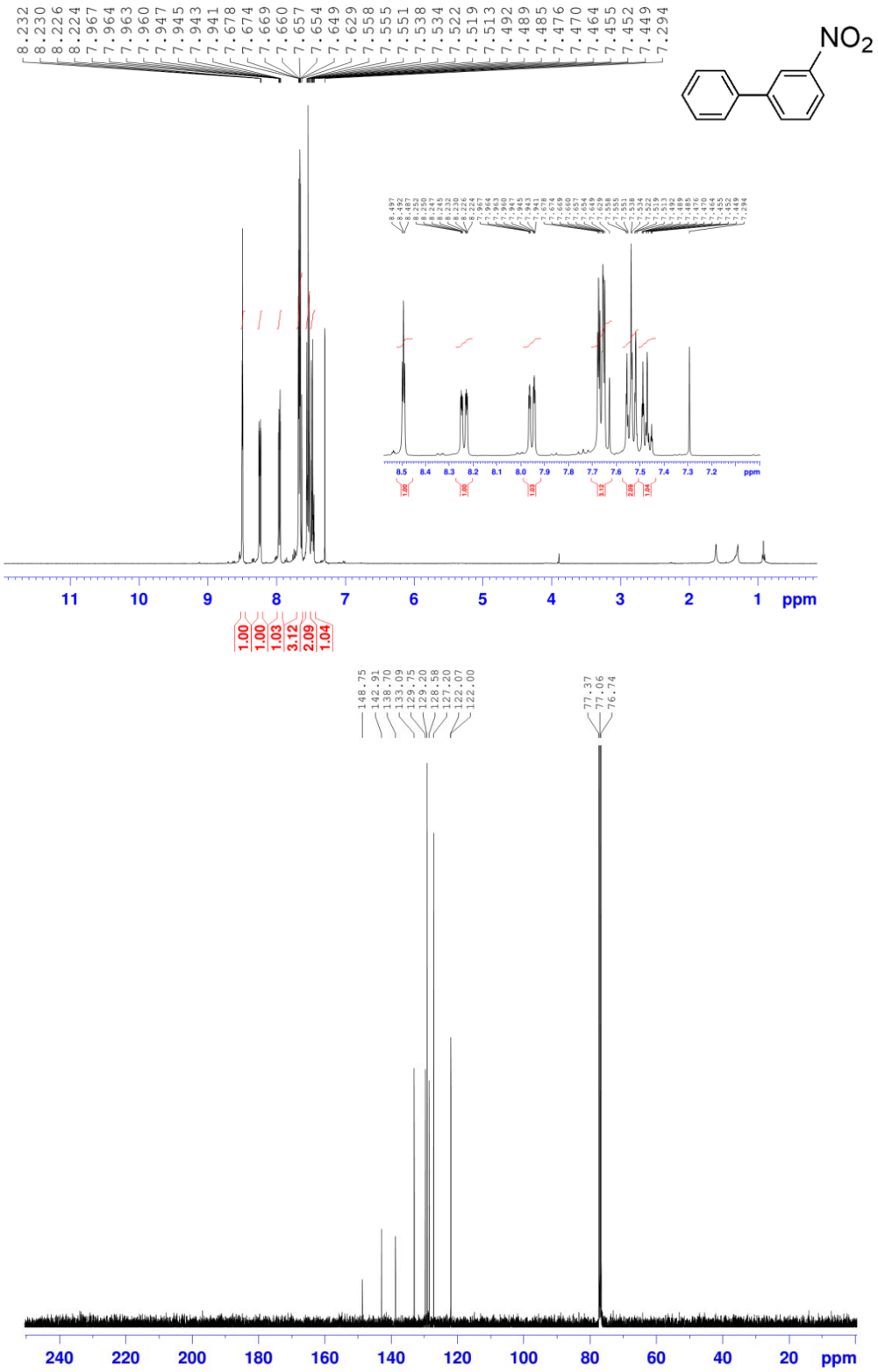
4-Methylbiphenyl (3c):

^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.45 (3H, s), 7.30 (2H, m), 7.38 (1H, m), 7.48 (2H, m), 7.55 (2H, m), 7.64 (2H, d).
^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.1, 126.8, 127, 127, 128.7, 129.5, 137, 138.4, 141.2.



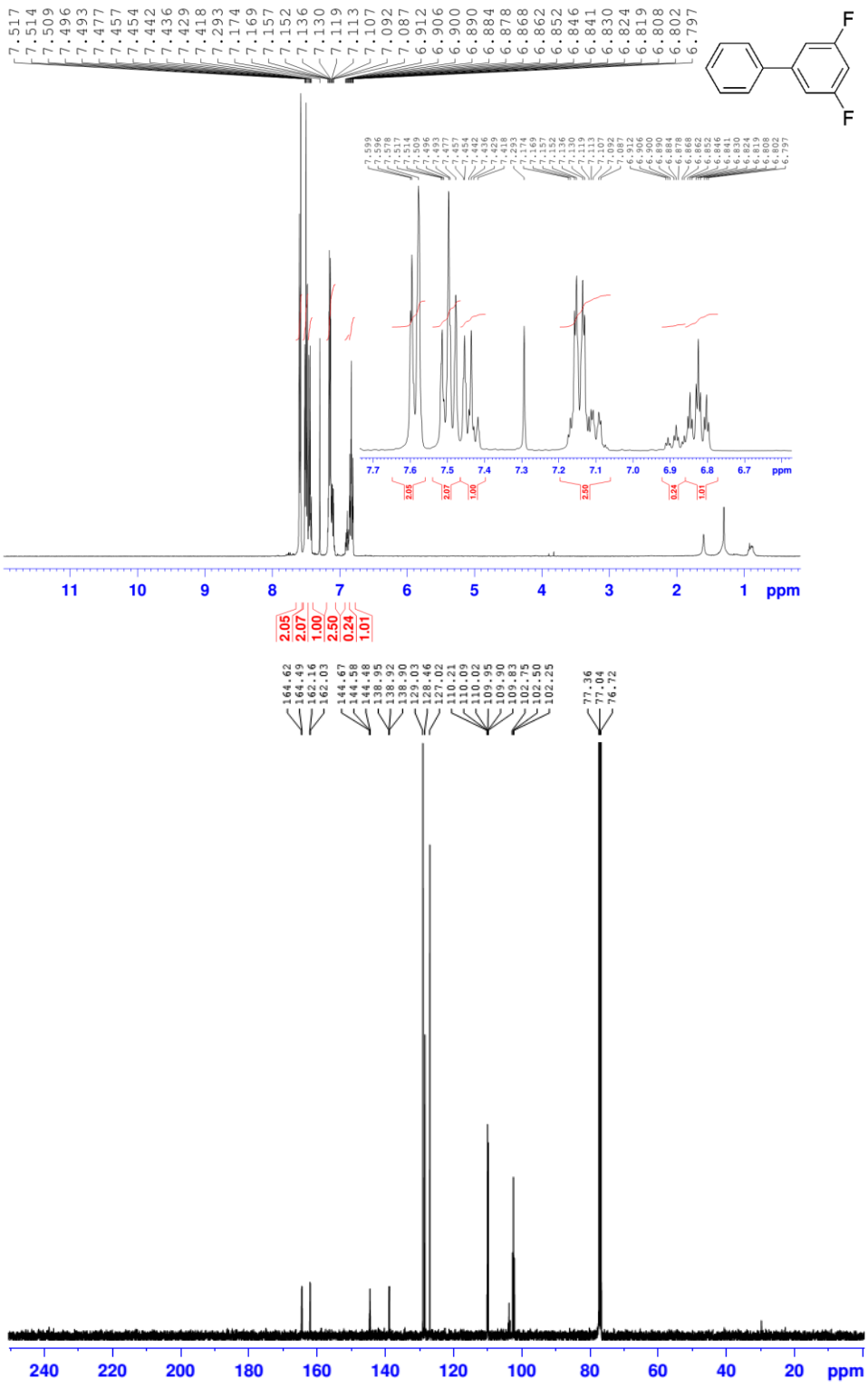
4-Phenylanisole (3a):

^{1}H-NMR (400 MHz, $CDCl_3$): δ= 3.89 (3H, s), 7.01 (2H, d), 7.33 (1H, t), 7.45 (2H, t), 7.55 (4H, m).
^{13}C-NMR (100 MHz, CDCl3): δ= 55.4, 114.2, 126.7, 126.7, 128.2, 128.7, 133.8, 140.8, 159.1.



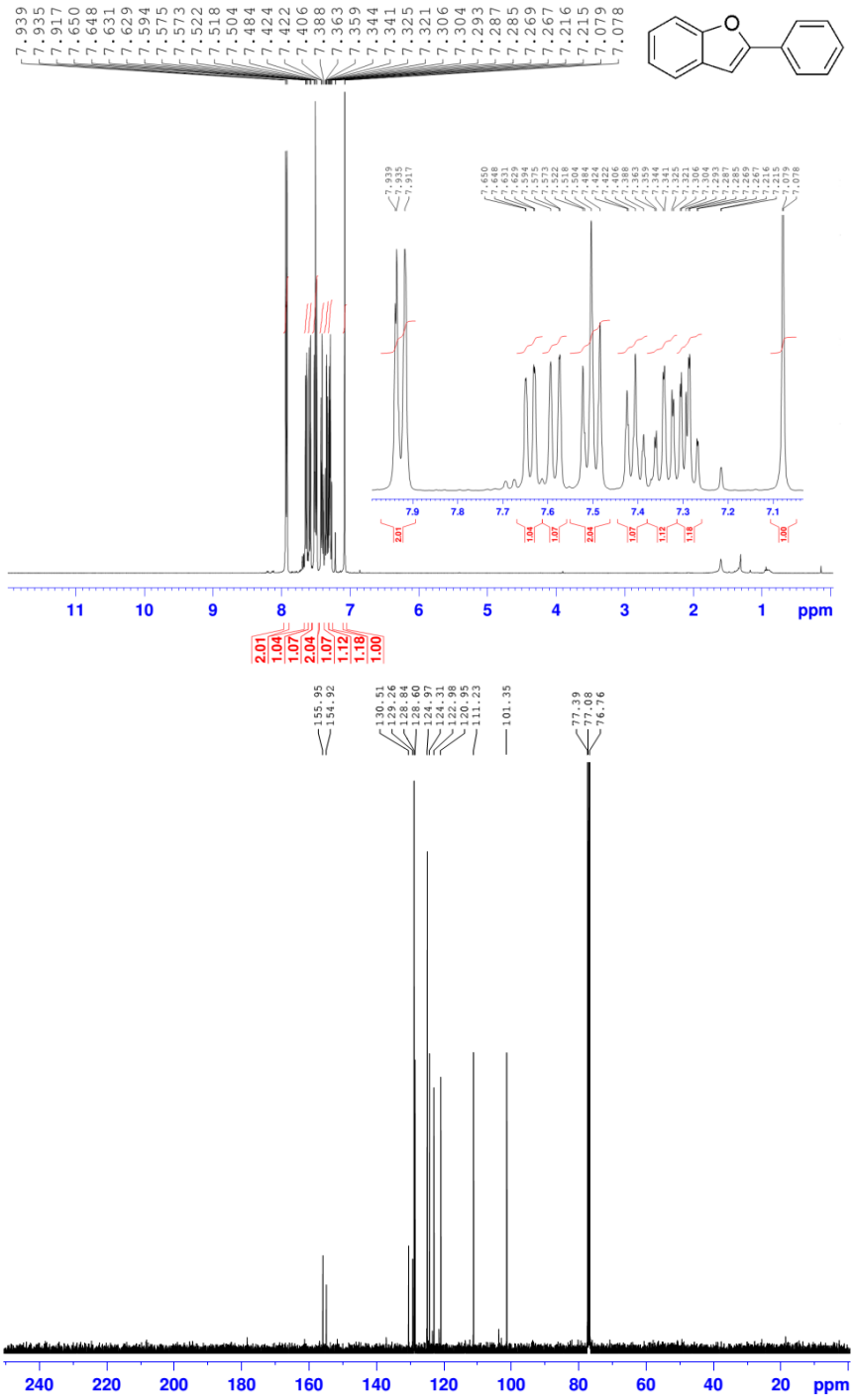
3-Nitrobiphenyl (3d):

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.47 (1H, m), 7.53 (2H, m), 7.65 (3H, m), 7.95 (2H, m), 8.25 (1H, m), 8.49 (1H, m).
^{13}C NMR (100 MHz, $CDCl_3$): δ = 122.0, 122.0, 127.2, 128.6, 129.2, 129.7, 133.0, 138.7, 142.9, 148.7.



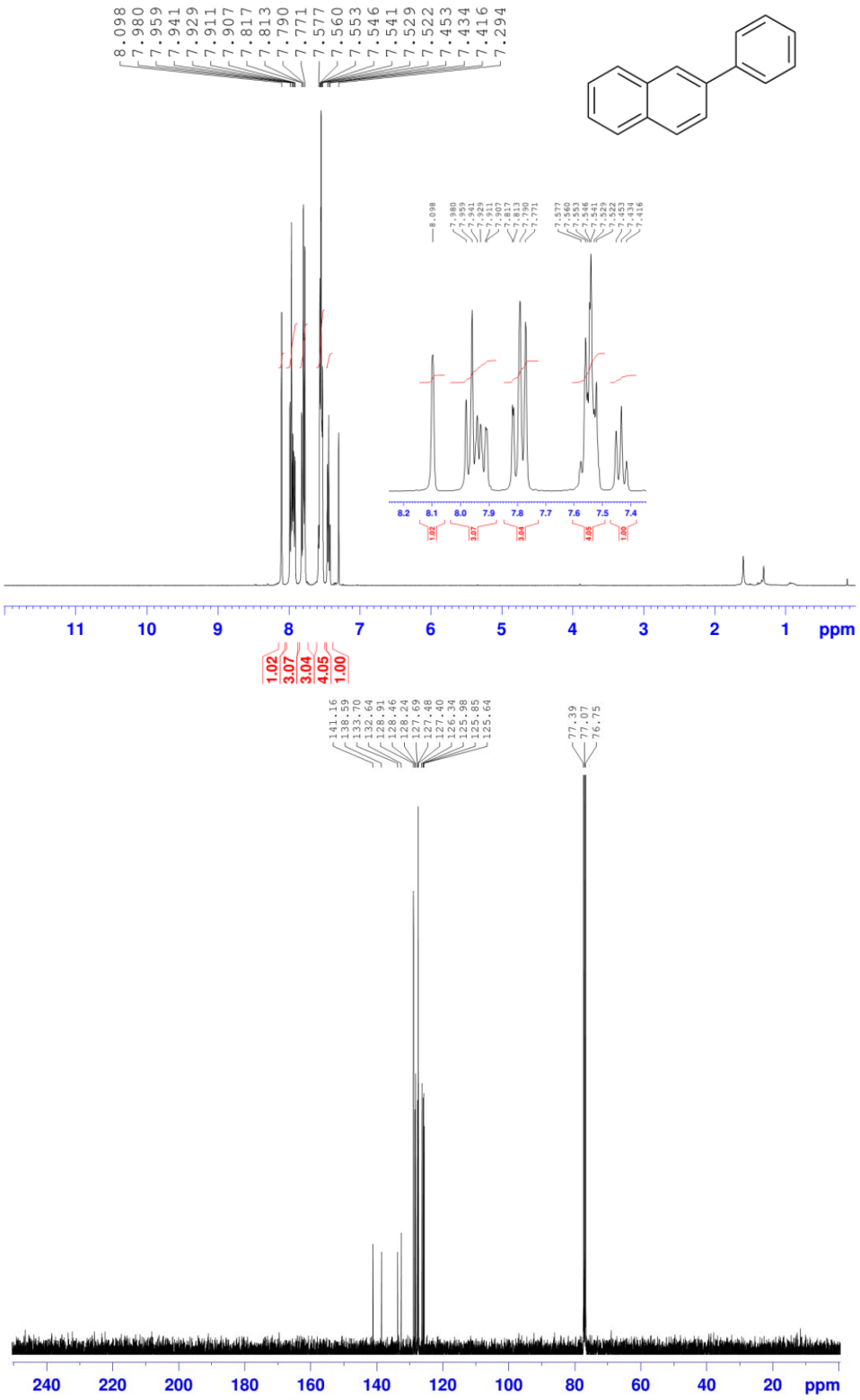
3,5-Difluorobiphenyl (3e):

^{1}H NMR (400 MHz, $CDCl_3$): δ = 6.82 (1H, tt), 7.10 (2H, dd), 7.43 (1H, t), 7.50 (2H, t), 7.60 (2H, d).
^{13}C NMR (100 MHz, $CDCl_3$): δ = 102.5 (t), 109.9 (dd), 127.0, 128.4, 129.0, 138.9, 144.5 (t), 163.3 (dd).



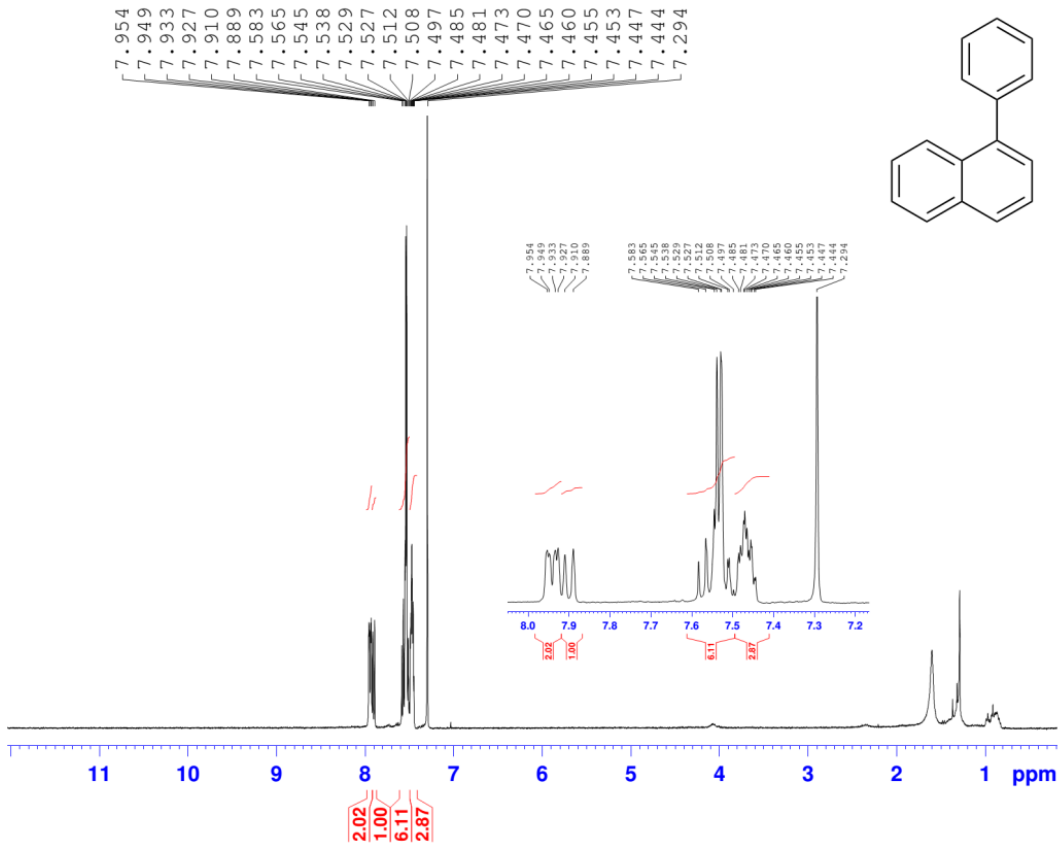
2-Phenylbenzo[b]furan (3f):

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.07 (1H, d), 7.21-7.29 (1H, m), 7.30-7.34 (1H, m), 7.35-7.42 (1H, m), 7.48-7.52 (2H, m), 7.57-7.59 (1H, m), 7.62-7.65 (1H, m), 7.91-7.93 (2H, m).
^{13}C NMR (100 MHz, $CDCl_3$): δ = 101.3, 111.2, 120.9, 122.9, 124.3, 124.9, 128.6, 128.8, 129.2, 130.5, 154.9, 155.9.



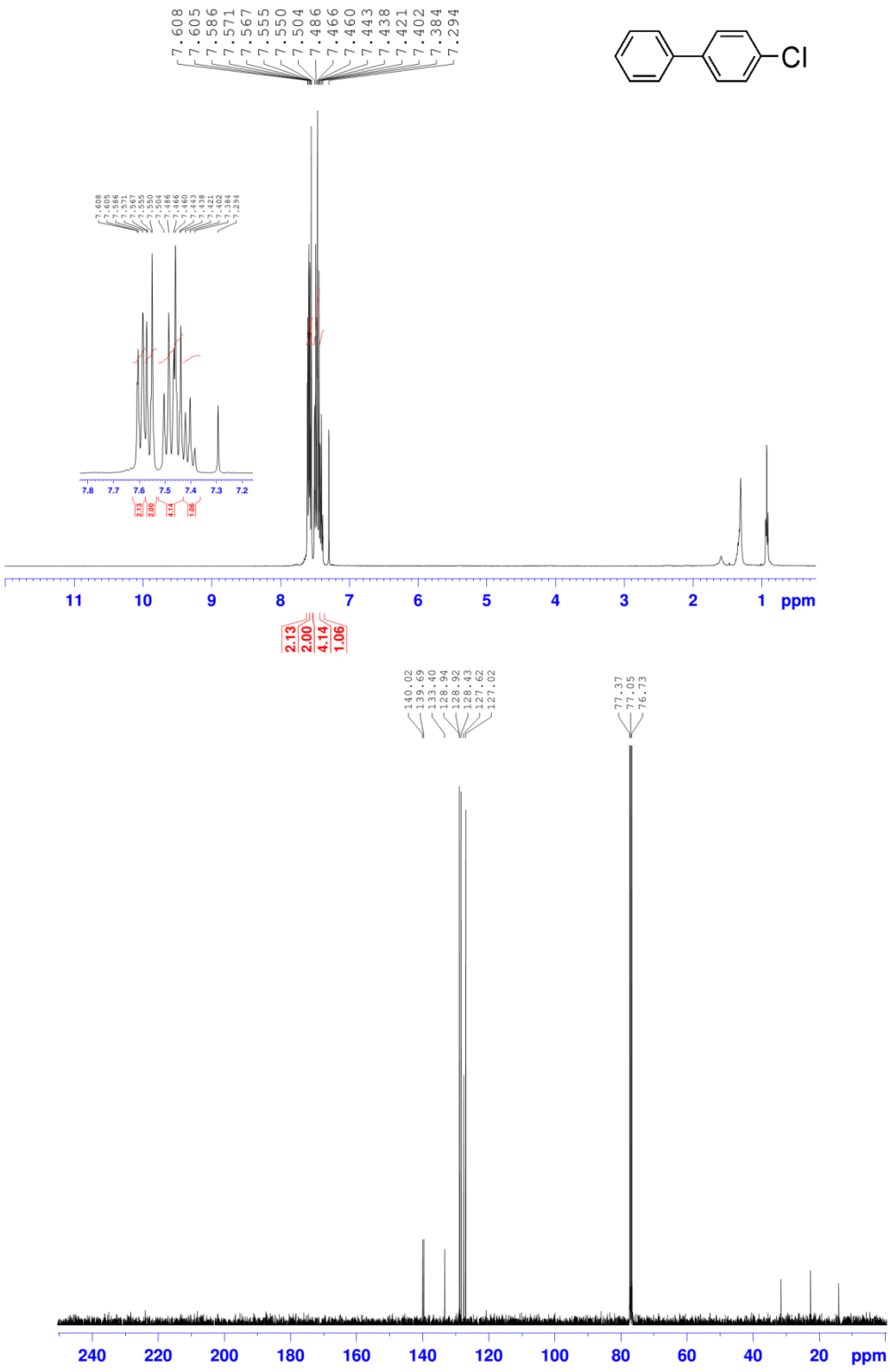
2-Phenylnaphthalene (3g):

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.41 (1H, t), 7.52-7.57 (4H, m), 7.77-7.90 (3H, m), 7.91-7.98 (3H, m), 8.09 (1H, s).
^{13}C NMR (100 MHz, $CDCl_3$): δ = 125.6, 125.8, 125.9, 126.3, 127.4, 127.7, 128.2, 128.4, 128.9, 132.6, 133.7, 138.6, 141.1.



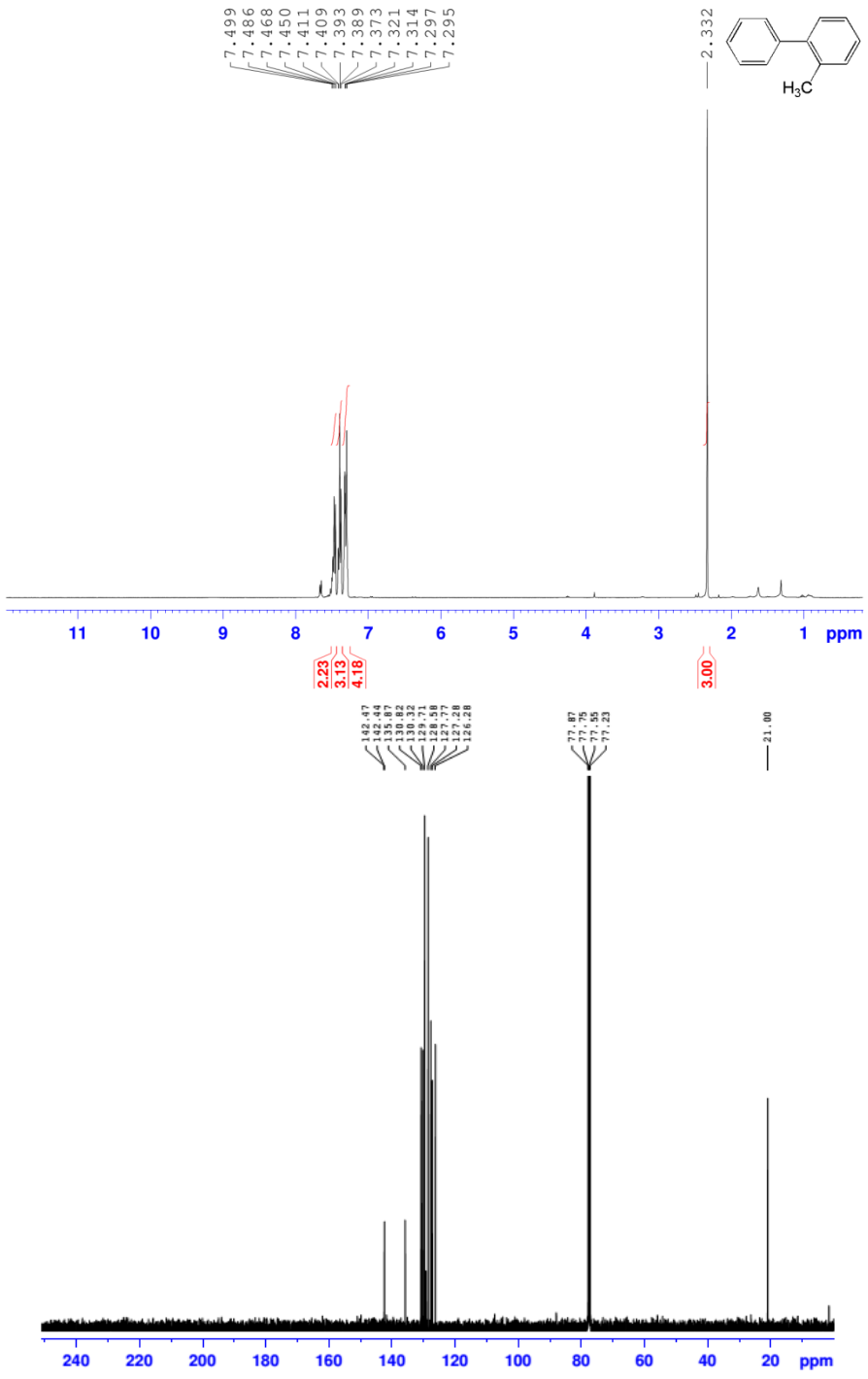
1-Phenylnaphthalene (3i):

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.44-7.49 (3H, m), 7.50-7.58 (6H, m), 7.88-7.95 (3H, m).



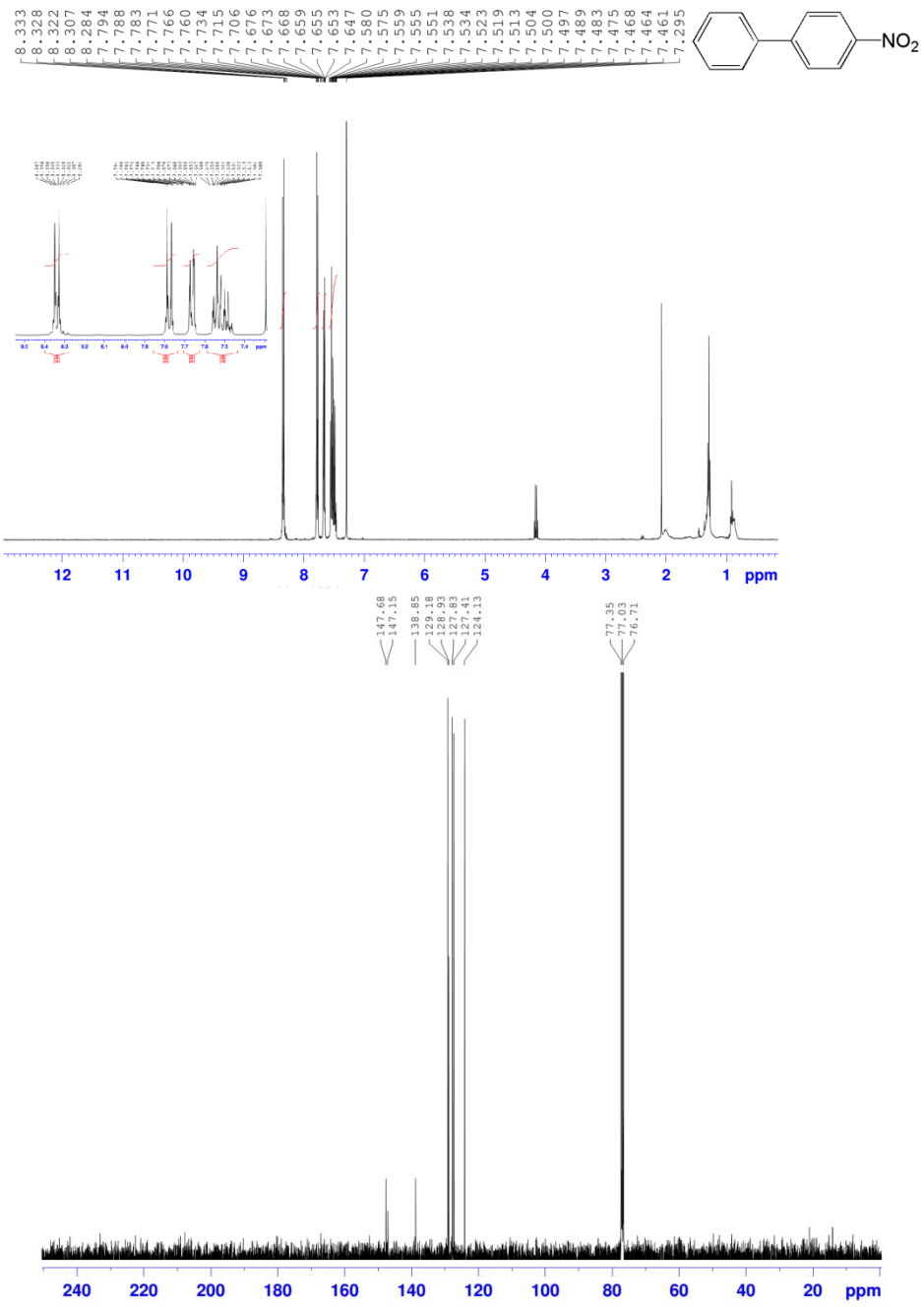
4-Chlorobiphenyl (3j):

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.35-7.46 (5H, m), 7.49-7.56 (4H, m).
^{13}C NMR (100 MHz, CDCl3): δ = 127, 127.6, 128.4, 128.9, 128.9, 133.4, 139.7, 140.



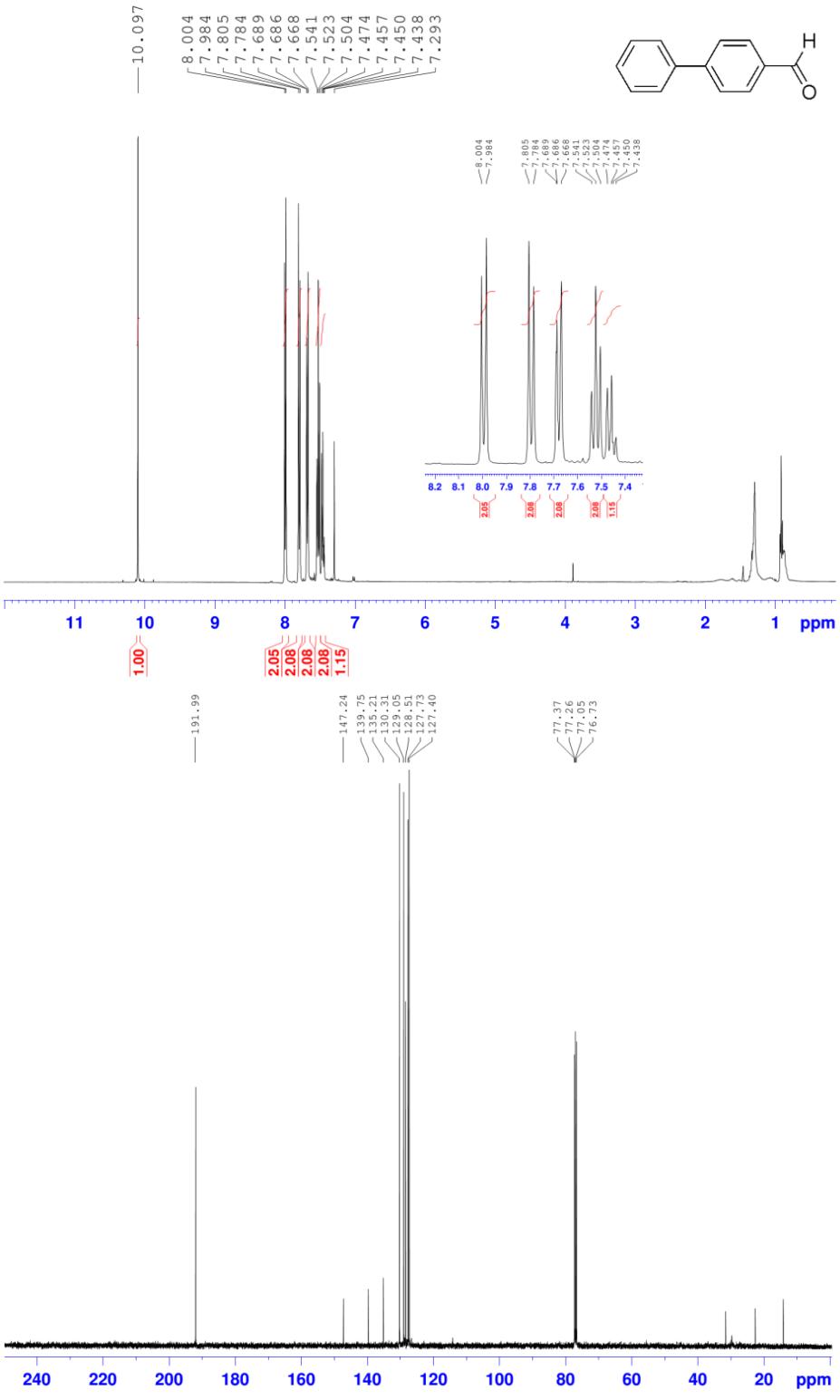
2-Methylbiphenyl (3k):

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.29-7.32 (4H, m), 7.37-7.41 (3H, m), 7.45-7.50 (2H, m).
^{13}C NMR (100 MHz, CDCl3): δ = 21.0, 126.2, 127.2, 127.7, 128.5, 129.7, 130.3, 130.8, 135.8, 142.4, 142.5.



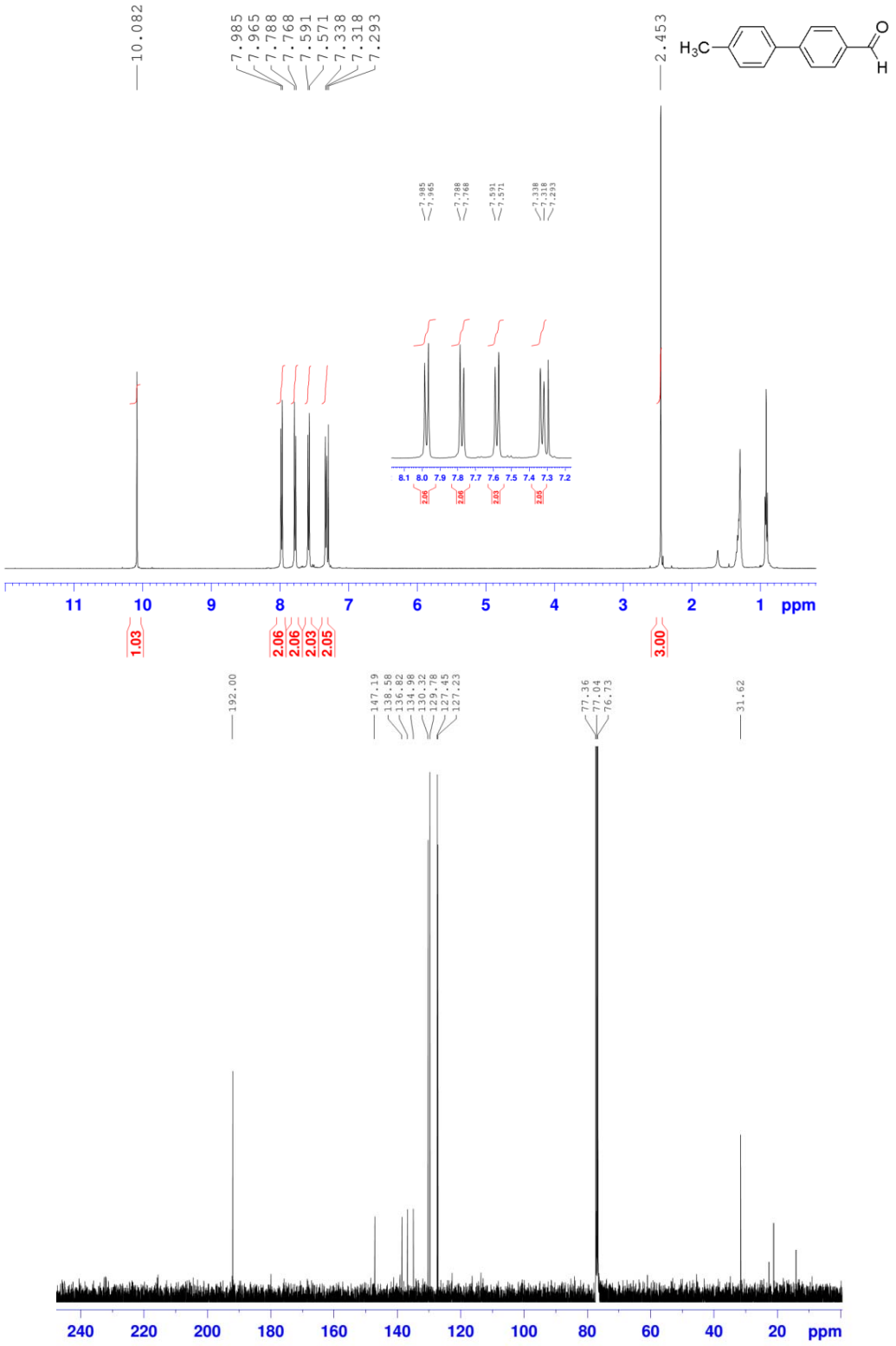
4-Nitrobiphenyl (3l):

^{1}H-NMR (400 MHz, $CDCl_3$): δ= 7.50-7.58 (3H, m), 7.64-7.67 (2H, m), 7.70-7.79 (2H, m), 8.28-8.36 (2H, m).
^{13}C-NMR (100.6 MHz, CDCl3): δ= 124.1, 127.4, 127.8, 128.9, 129.1, 138.8, 147.1, 147.6.



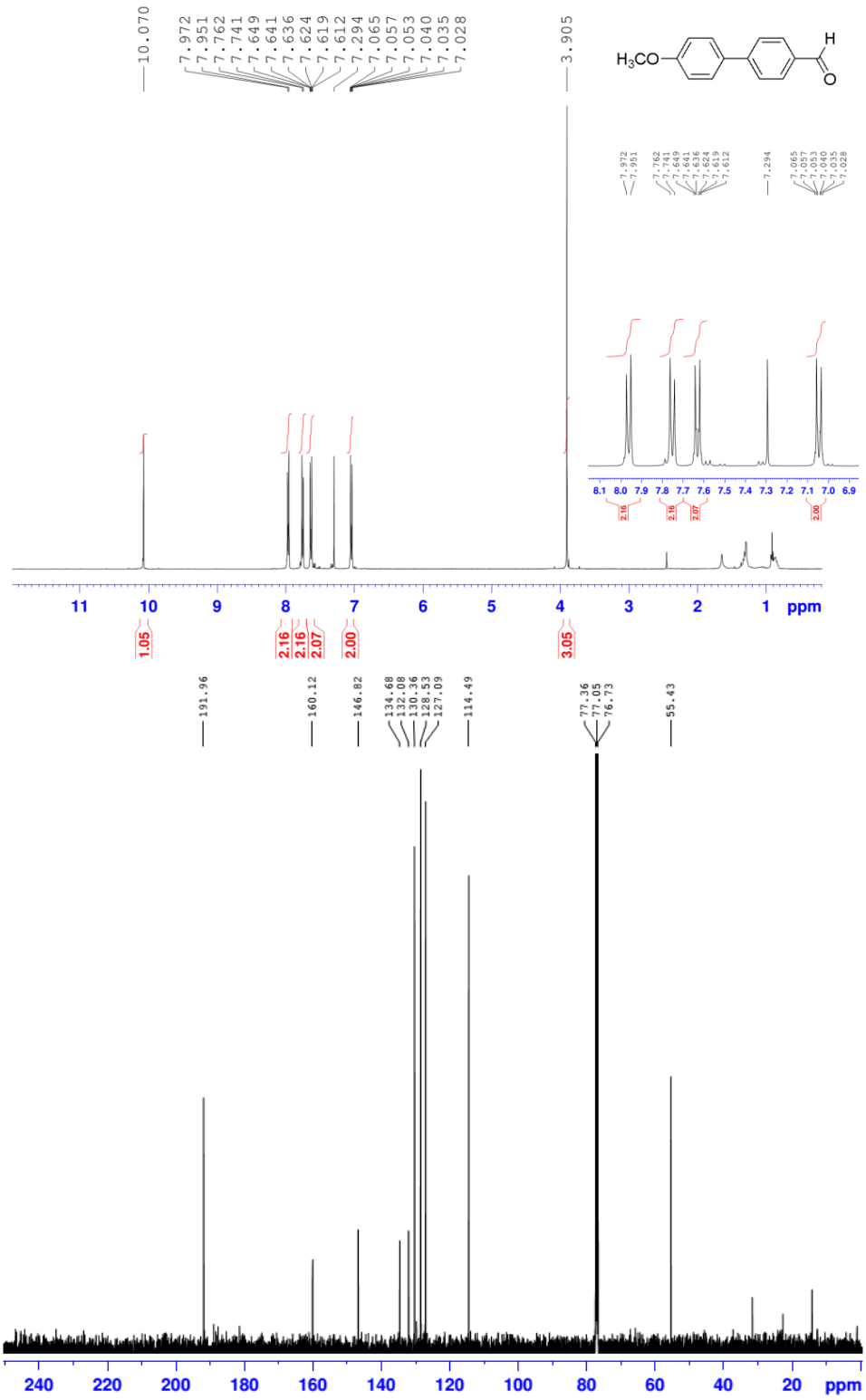
4-Phenylbenzaldehyde (3m):

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.45 (1H, t), 7.52 (2H, t), 7.67 (2H, d), 7.79 (2H, d), 7.99 (2H, d), 10.09 (1H, s)
^{13}C NMR (100 MHz, $CDCl_3$): δ = 127.4, 127.7, 128.5, 129.0, 130.3, 135.2, 139.7, 147.2, 192.0.



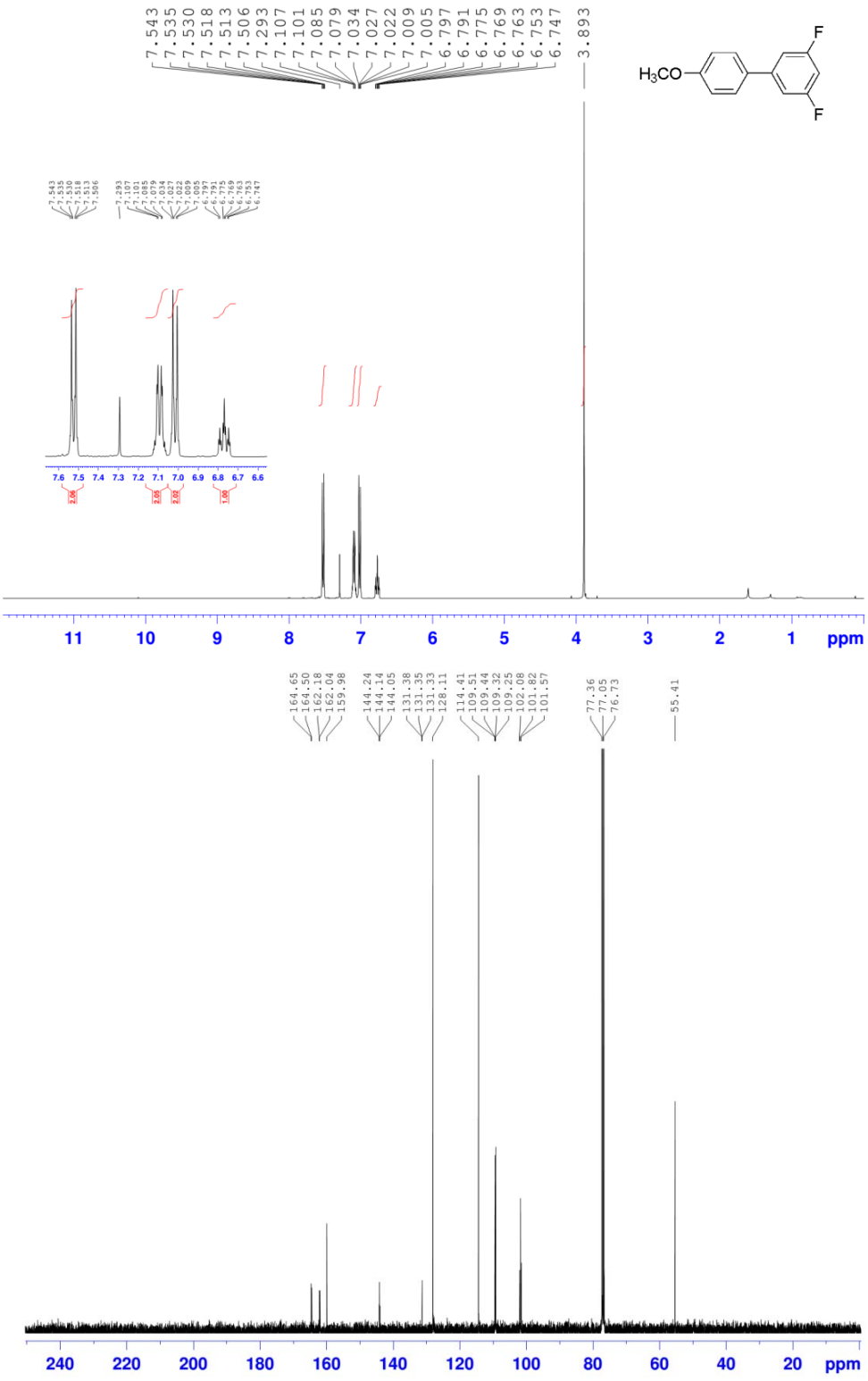
4’-methyl-biphenyl-4-carbaldehyde (3n):

^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.45 (3H, s), 7.31 (2H, t), 7.58 (2H, d), 7.77 (2H, d), 7.97 (2H, d), 10.08 (1H, s)
^{13}C NMR (100 MHz, $CDCl_3$): δ = 31.6, 127.2, 127.4, 129.7, 130.3, 134.9, 136.8, 138.5, 147.1, 192.0.



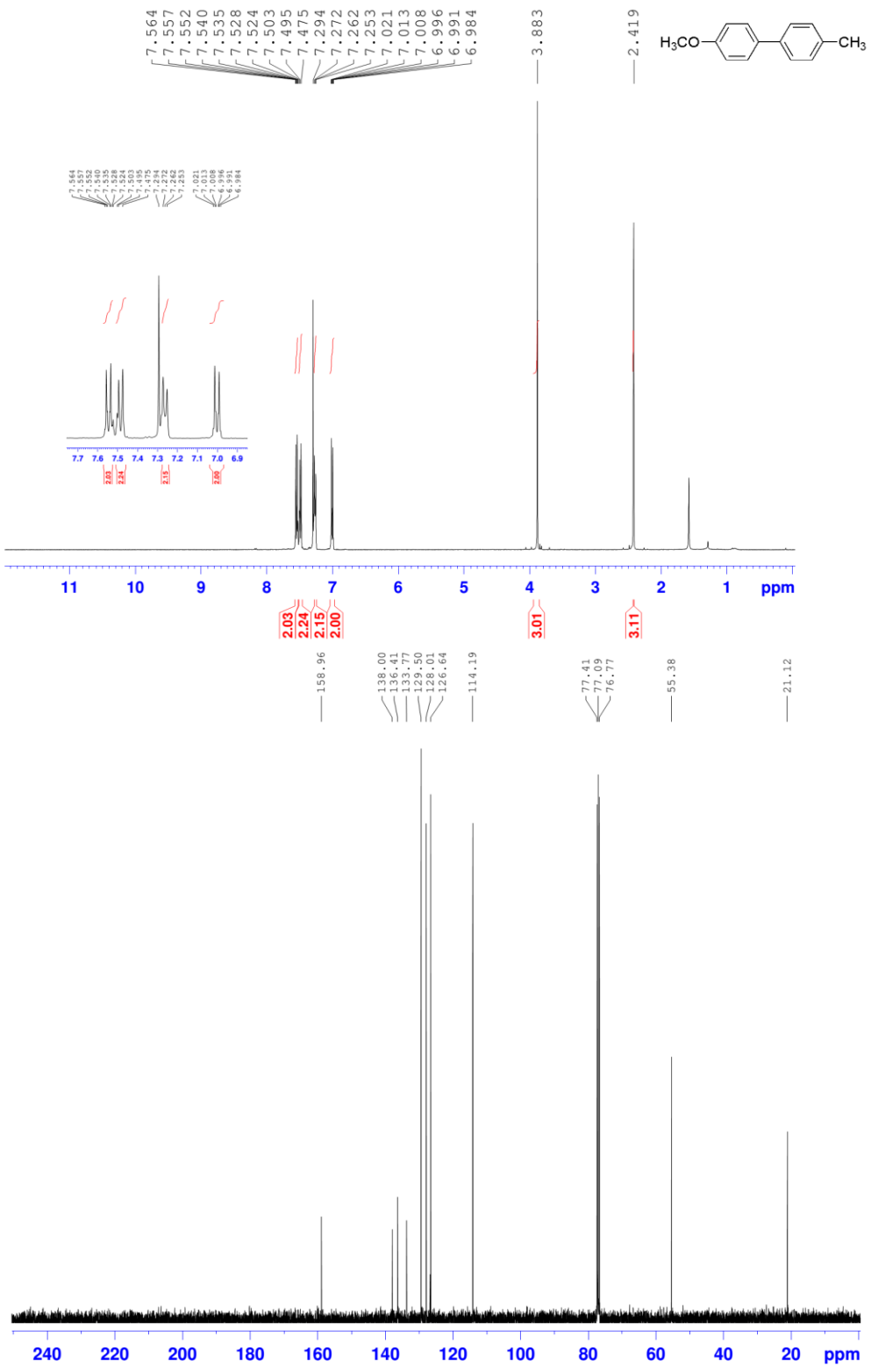
4’-Methoxy-biphenyl-4-carboxaldehyde (3o):

^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.90 (3H, s), 7.05 (2H, d), 7.63 (2H, d), 7.75 (2H, d), 7.96 (2H, d), 10.07 (1H, s).
^{13}C NMR (100 MHz, $CDCl_3$): δ = 55.43, 114.5, 127.1, 128.5, 130.6, 132.1, 134.7, 146.8, 160.1, 191.9.



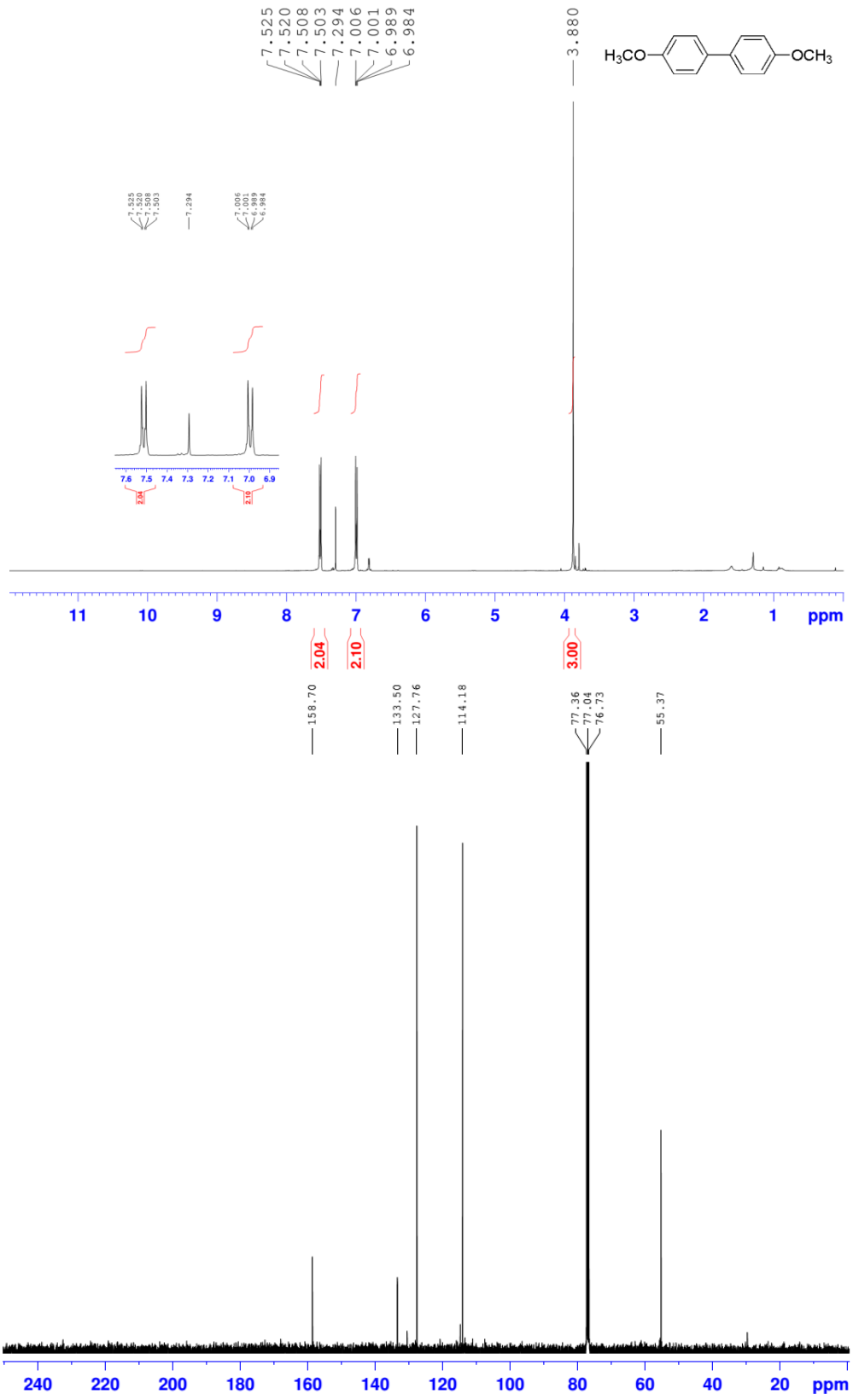
4-(3,5-difluorophenyl)-anisole (3p):

^{1}H NMR (400 MHz, $CDCl_3$): 3.89 (3H, s), 6.77 (1H, tt), 7.02 (2H, d), 7.10 (2H, m), 7.52 (2H, d).
^{13}C NMR (100 MHz, $CDCl_3$): 55.4, 101.8 (t), 109.3 (dd), 141.4, 128.1, 131.3 (t), 144.1 (t), 159.9, 163.3 (dd).



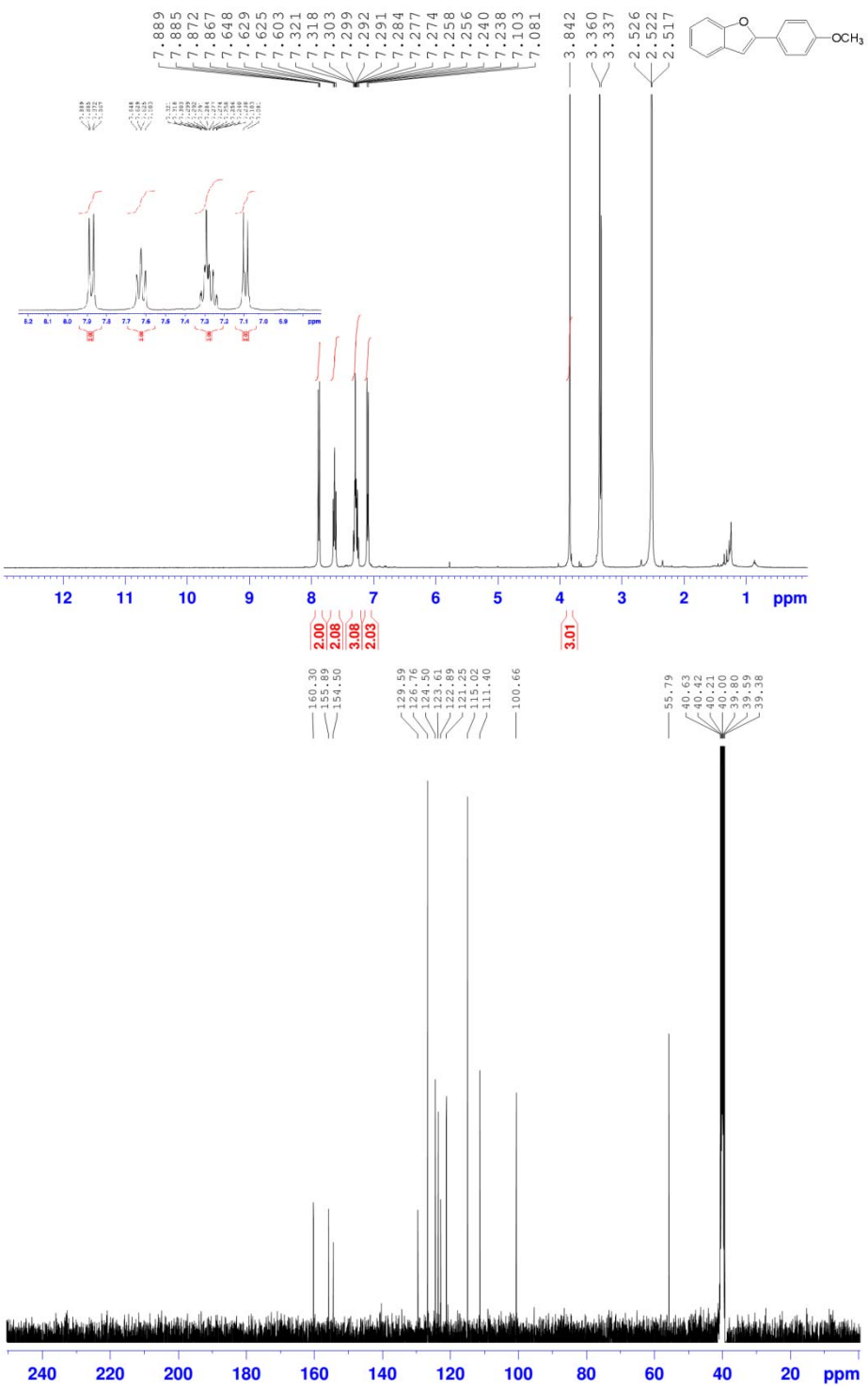
4-Methoxy-4’-methyl-biphenyl (3q):

^{1}H NMR (400 MHz, $CDCl_3$): 2.41 (3H, s), 3.88 (3H, s), 7.00 (2H, d), 7.26 (2H, m), 7.50 (2H, m), 7.55 (2H, m).
^{13}C NMR (100 MHz, $CDCl_3$): 21.1, 55.3, 114.1, 126.6, 128.0, 129.5, 133.7, 136.4, 138.0, 158.9.



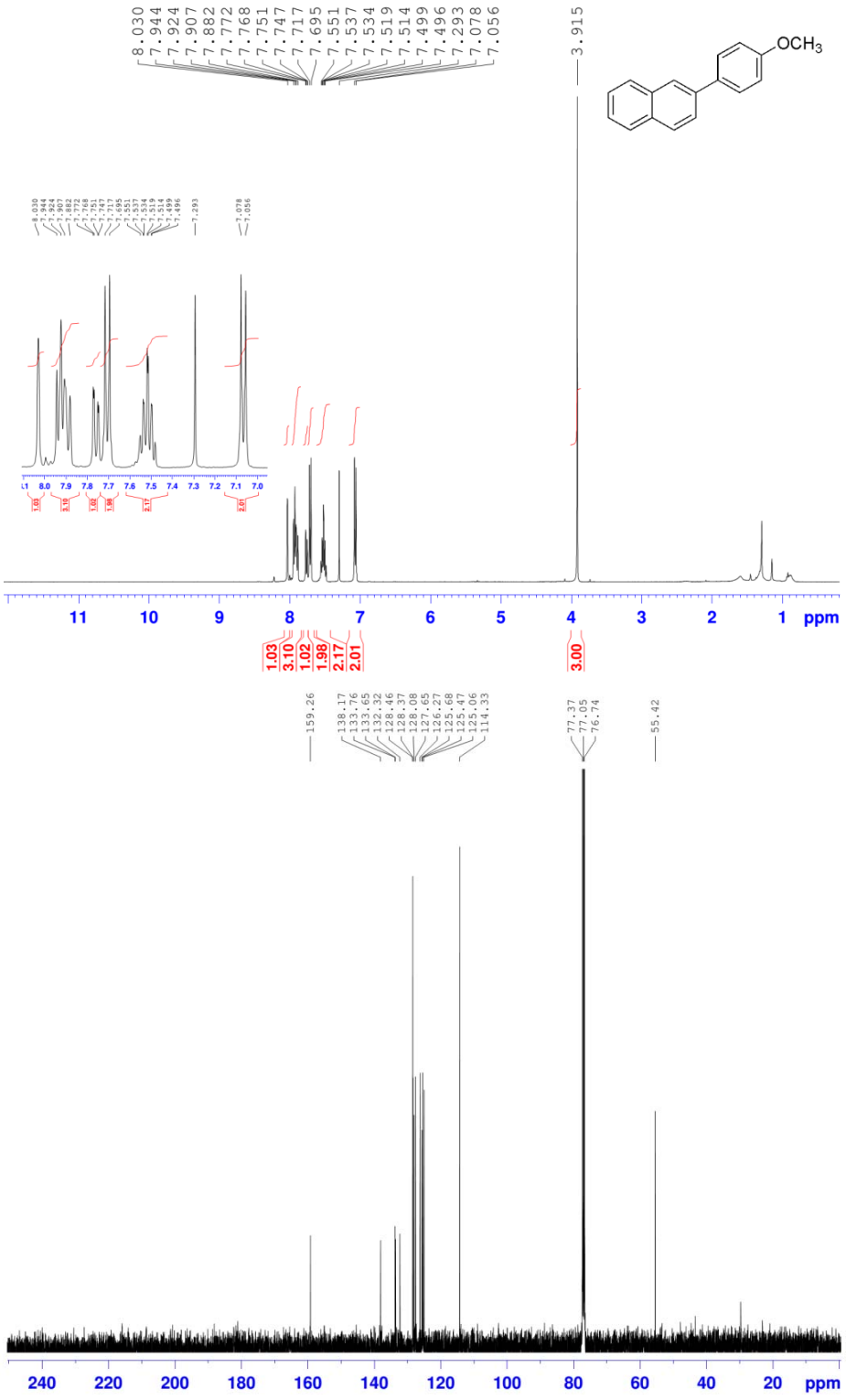
4, 4’-Dimethoxy-biphenyl (3r):

^{1}H NMR (400 MHz, $CDCl_3$): 3.88 (6H, s), 7.00 (4H, d), 7.51 (4H, d).
^{13}C NMR (100 MHz, $CDCl_3$): 55.3, 114.1, 127.7, 133.5, 158.7.



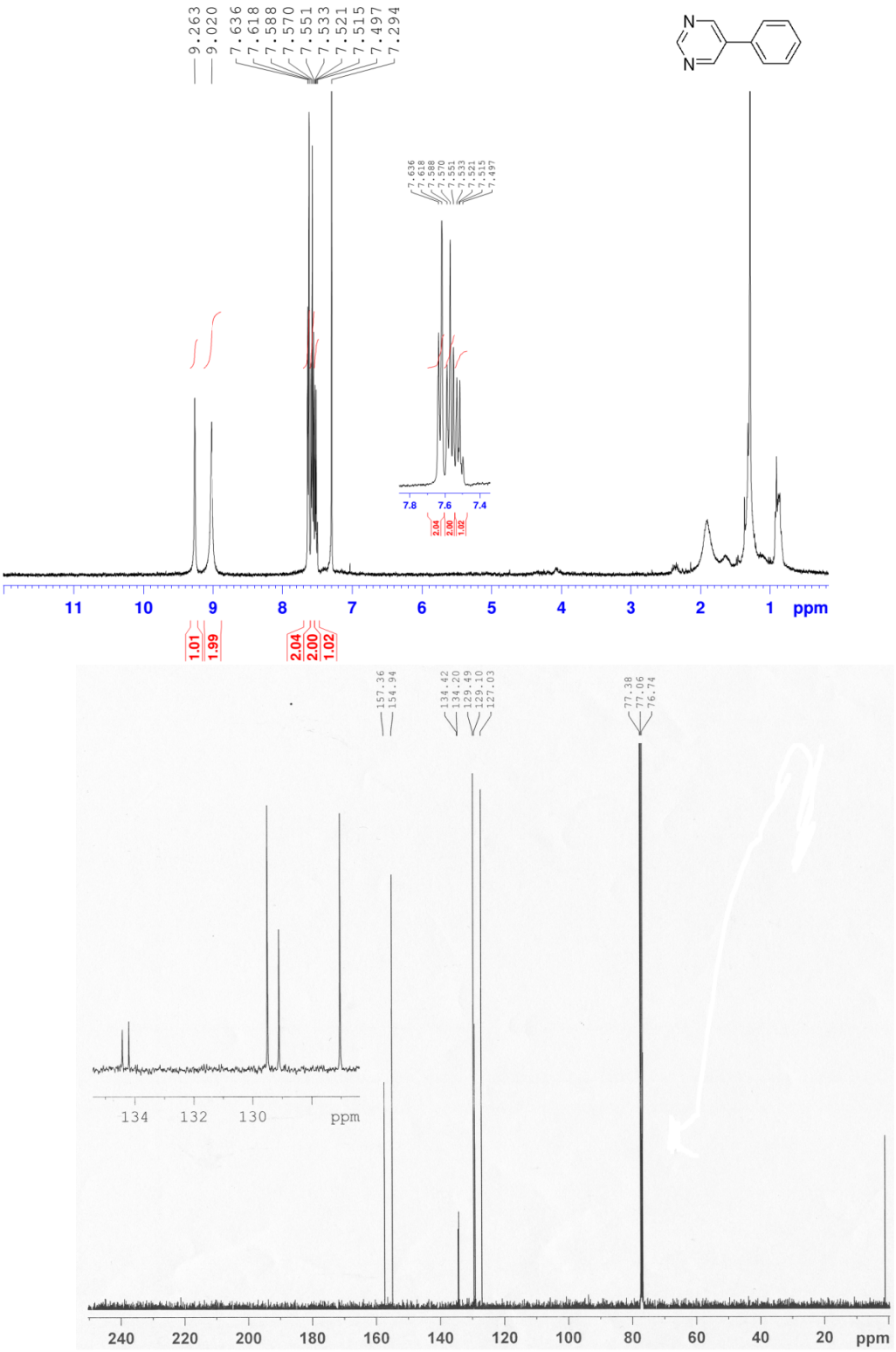
2-(4-methoxyphenyl)benzo[b]furan (3s):

^{1}H NMR (400MHz, DMSO): 3.84(3H, s), 7.09(2H, d), 7.23-7.32(3H, m), 7.60-7.87(2H, m), 7.88(2H, d)
^{13}C NMR (100MHz, DMSO): 55.8, 100.6, 111.4, 115.0, 121.2, 122.9, 123.6, 124.5, 126.7, 129.6, 154.5, 155.9, 160.3



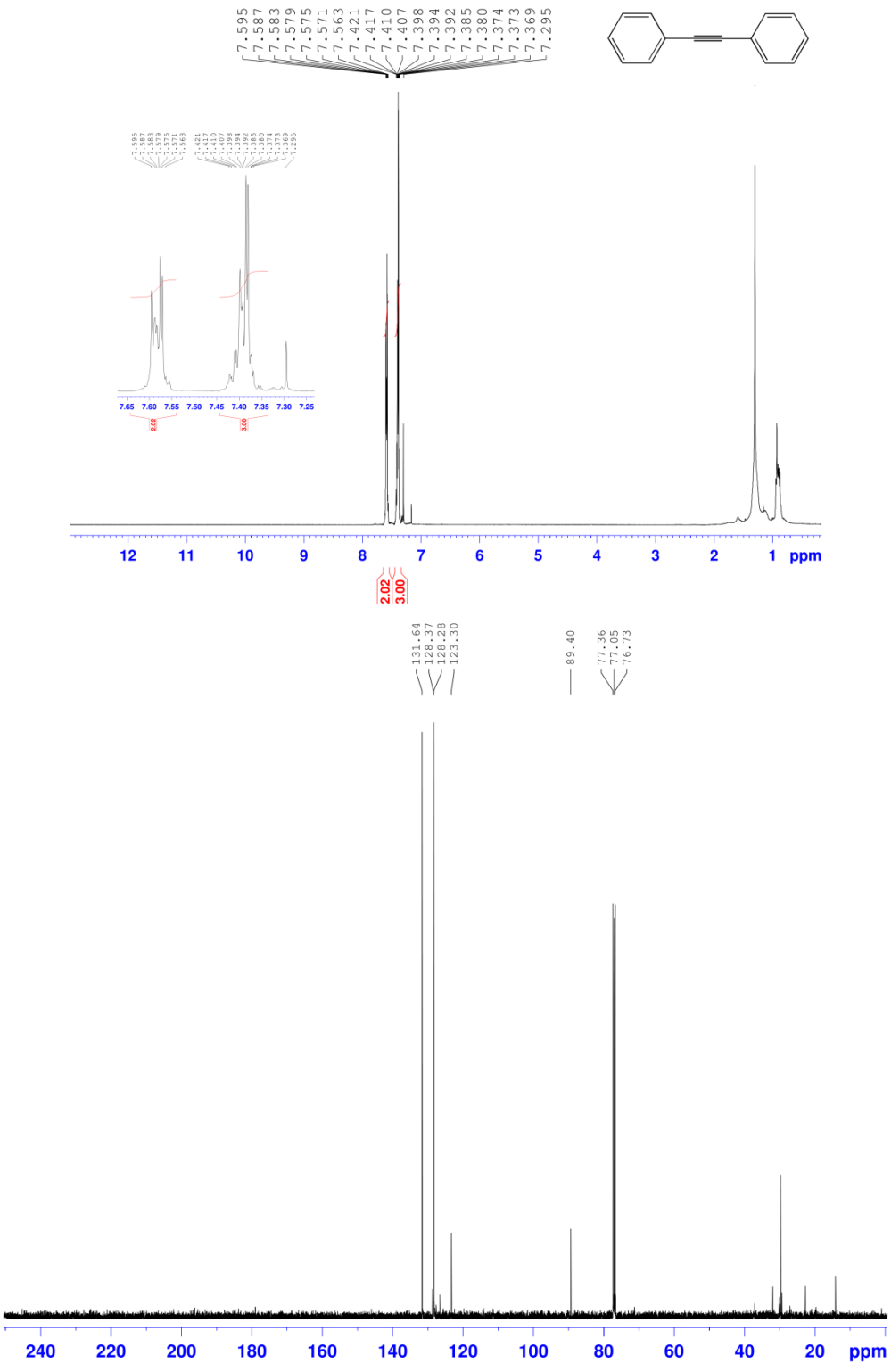
2-(4-methoxyphenyl)naphthalene (3t):

^{1}H NMR (400 MHz, $CDCl_3$): δ= 3.91 (3H, s), 7.06 (2H, d), 7.53 (2H, m), 7.70 (2H, d), 7.76 (1H, dd), 7.90 (2H, m), 8.03 (1H, s).
^{13}C NMR (100 MHz, $CDCl_3$): δ= 55.4, 114.3, 125.0, 125.4, 125.6, 126.2, 127.6, 128.0, 128.3, 128.4, 132.2, 133.6, 133.8, 138.1, 159.3.



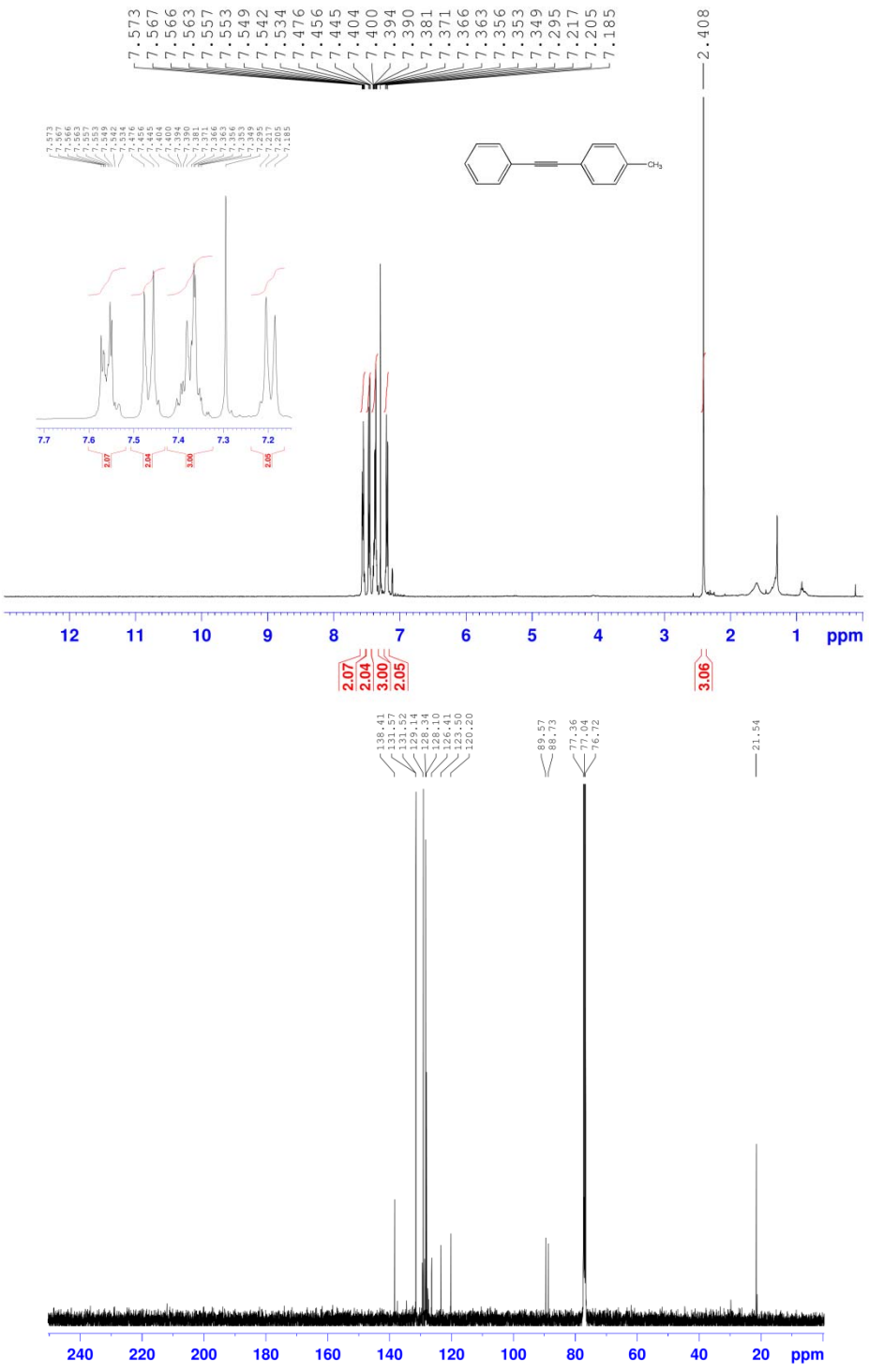
5-Phenylpyrimidine (3u):

^{1}H NMR (400 MHz, $CDCl_3$): δ= 7.49-7.63 (5H, m), 9.02 (2H, s), 9.26 (1H, s).
^{13}C NMR (100MHz, CDCl3): δ= 127.0, 129.1, 129.5, 134.2, 134.4, 154.9, 157.3.



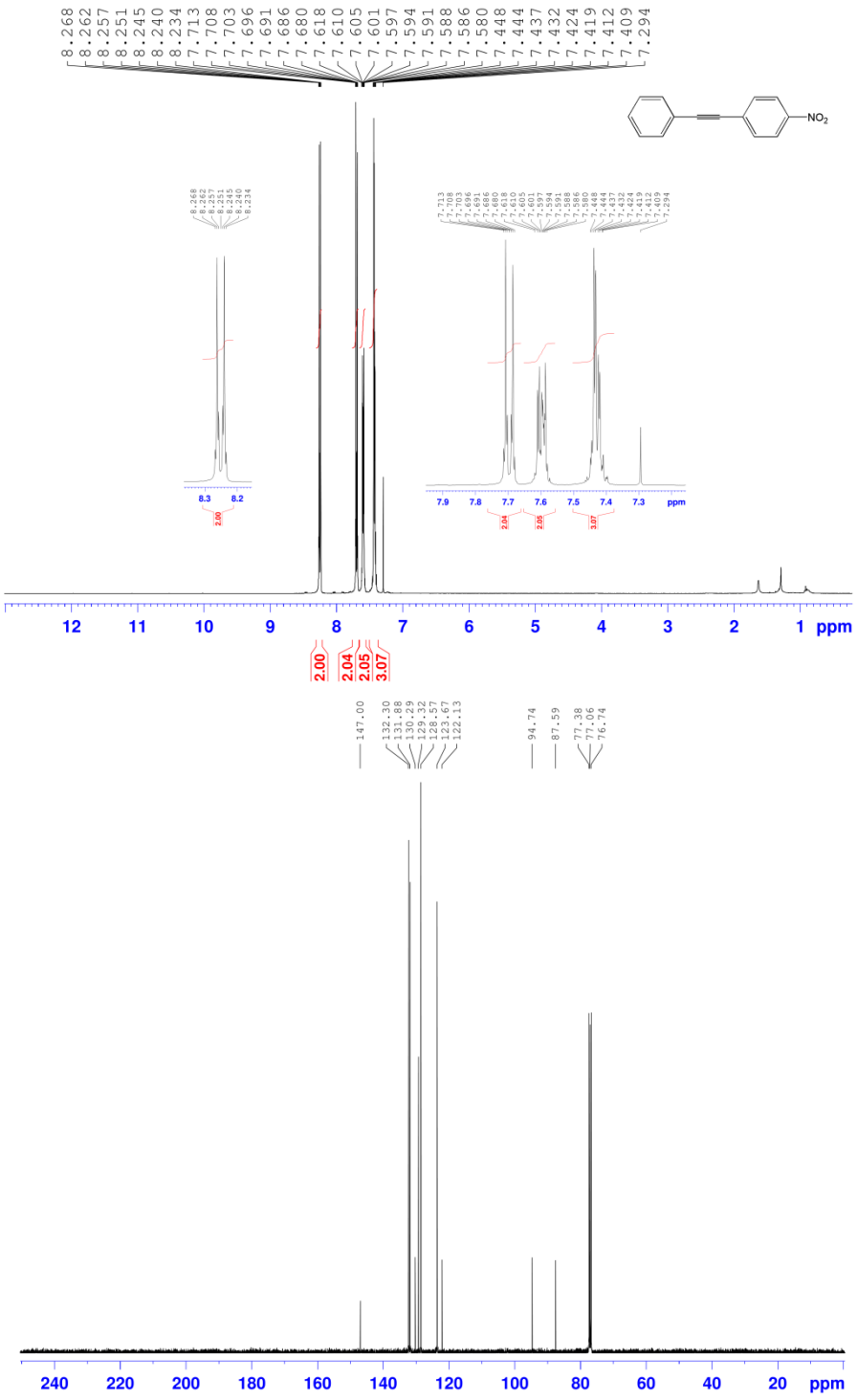
1,2-diphenylethyne (6a):

^{1}H NMR (400 MHz, $CDCl_3$): δ= 7.29-7.42 (6H, m), 7.56-7.59 (4H, m)
^{13}C NMR (100MHz, $CDCl_3$): δ= 89.40, 123.30, 128.28, 128.37, 131.64.



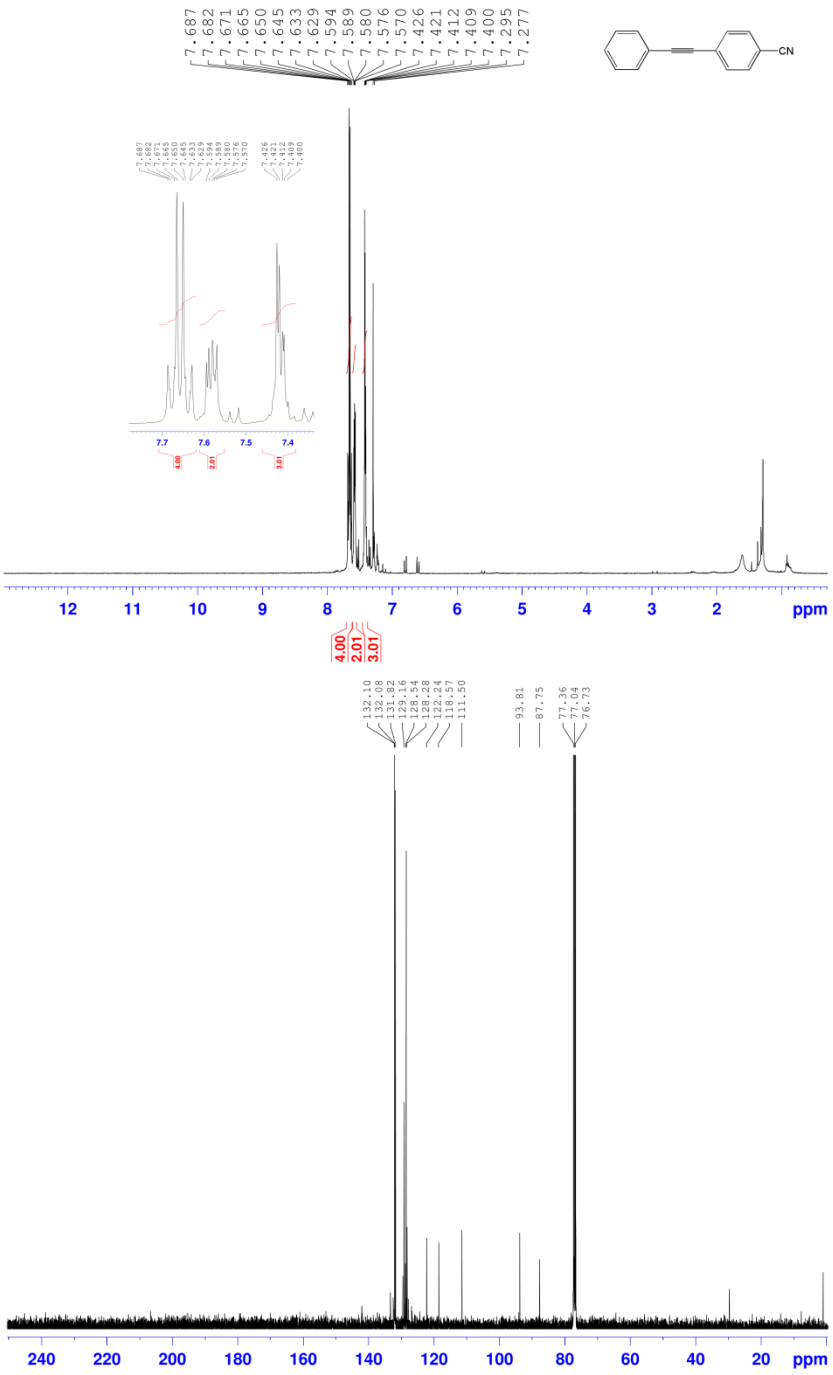
1-methyl-4-(phenylethynyl)benzene (6b):

^{1}H NMR (400 MHz, $CDCl_3$): δ= 2.40 (3H, s), 7.19 (2H, d), 7.35-7.37 (3H, 3), 7.46 (2H, d), 7.53-7.55 (2H m)
^{13}C NMR (100MHz, $CDCl_3$): δ= 21.54, 88.73, 89.57, 120.20, 123.50, 128.10, 128.34, 129.19, 131.52, 131.57, 138.41.



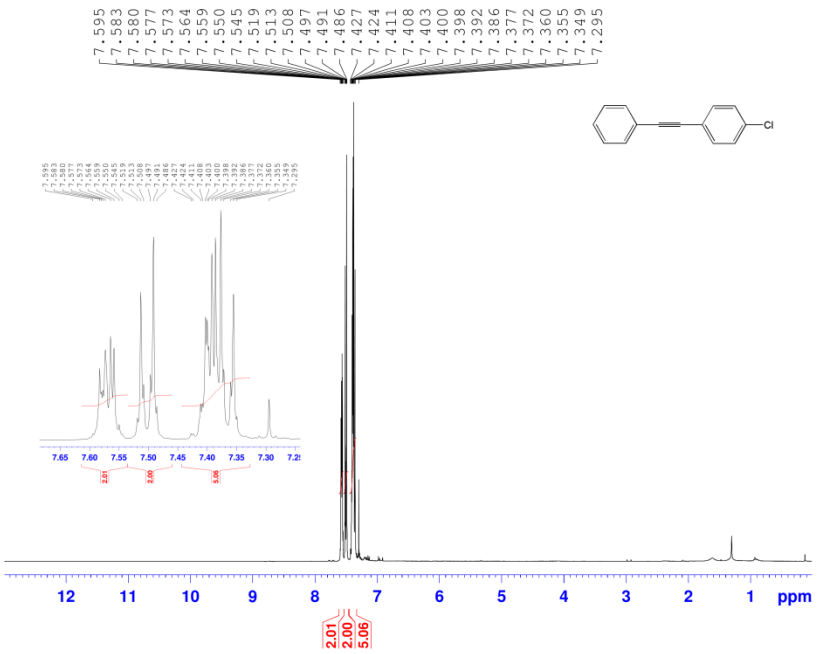
1-nitro-4-(phenylethynyl)benzene (6c):

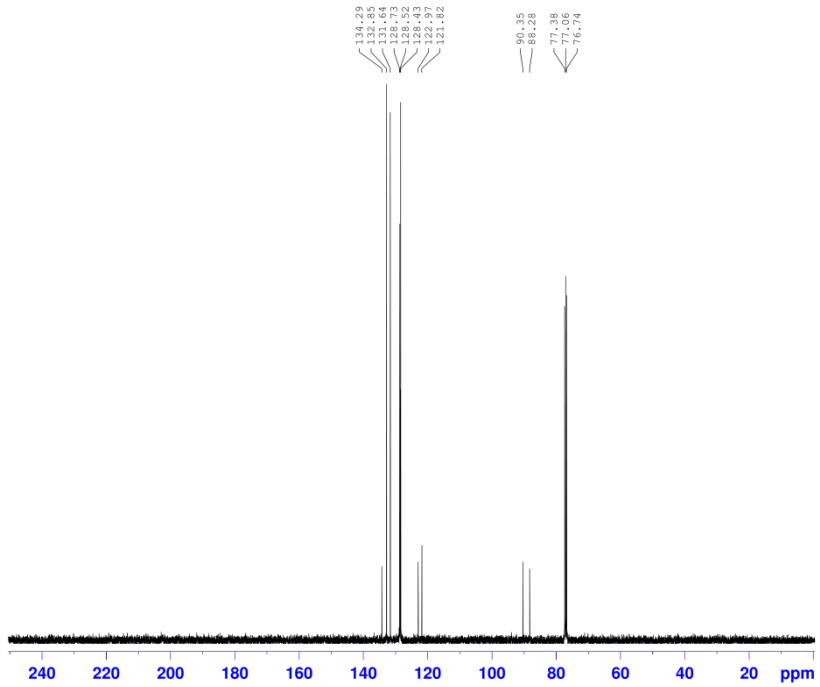
^{1}H NMR (400 MHz, $CDCl_3$): δ= 7.40-7.44 (3H, m), 7.58-7.61 (3H, m), 7.68-7.71 (2H, m), 8.23-8.26 (2H, m).
^{13}C NMR (100MHz, $CDCl_3$): δ= 87.59, 94.74, 122.13, 123.67, 128.57, 129.32, 130.29, 131.88, 132.30, 147.00.



4-(phenylethynyl)benzonitrile (6d):

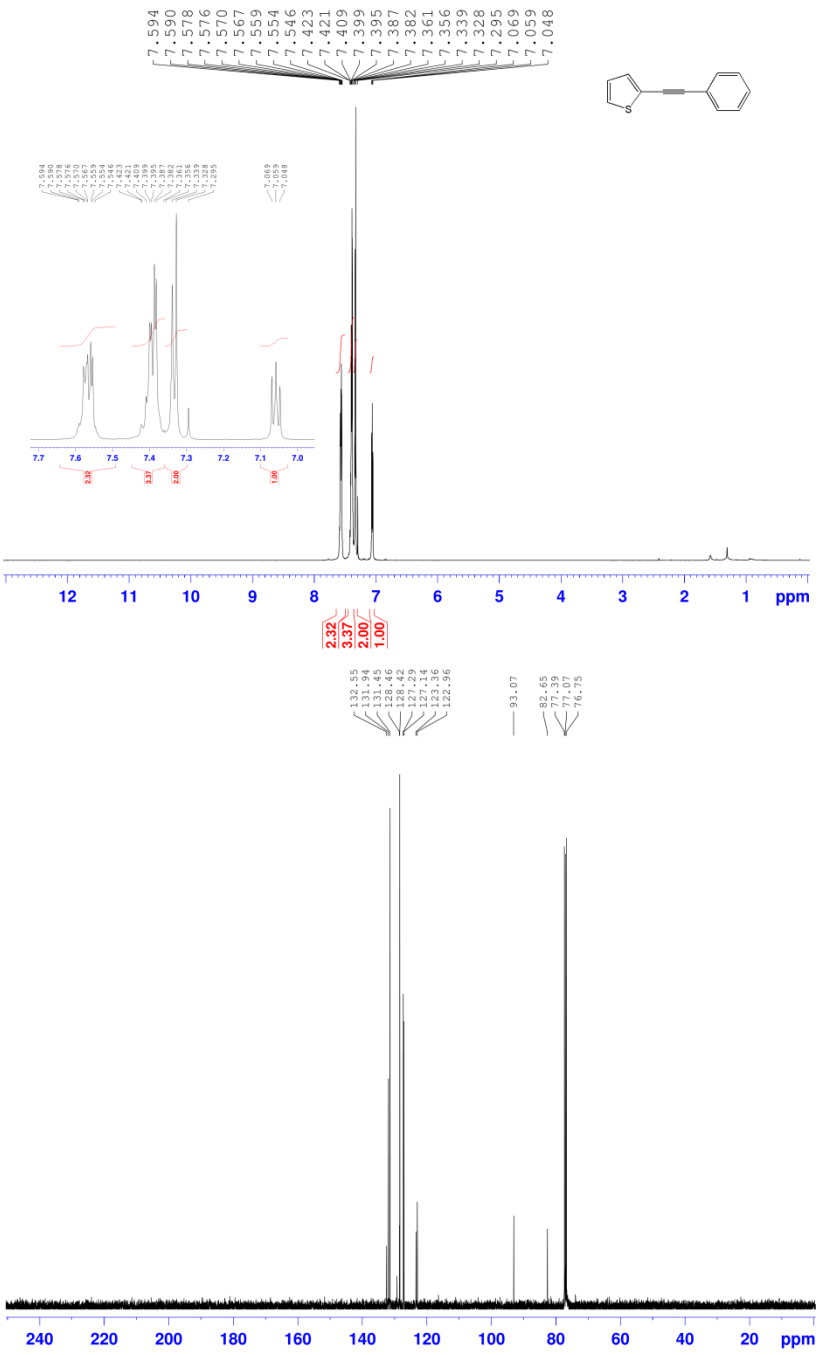
^{1}H NMR (400 MHz, $CDCl_3$): δ= 7.40-7.42 (3H, m), 7.57-7.59 (2H, m), 7.62-7.68 (4H, m)
^{13}C NMR (100MHz, $CDCl_3$): δ= 87.75, 93.81, 111.50, 118.57, 122.24, 128.28, 128.54, 129.16, 131.82, 132.08, 132.10.





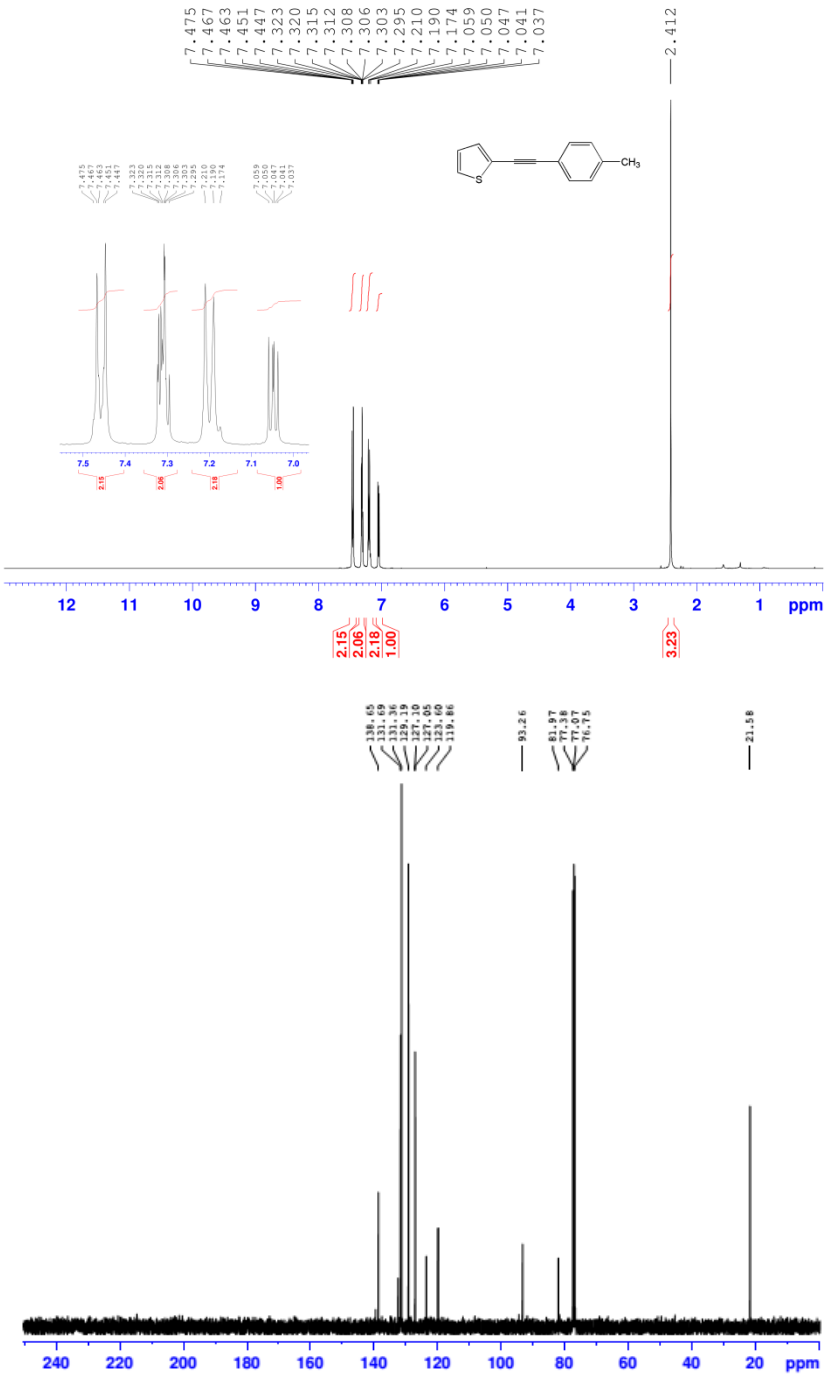
1-chloro-4-(phenylethynyl)benzene (6e):

^{1}H NMR (400 MHz, $CDCl_3$): δ= 7.34-7.42 (5H, m), 7.49 (2H, d), 7.54-7.59 (2H, m).
^{13}C NMR (100MHz, $CDCl_3$): δ= 88.28, 90.35, 121.82, 122.97, 128.43, 128.52, 128.73, 131.64, 132.85, 134.29.



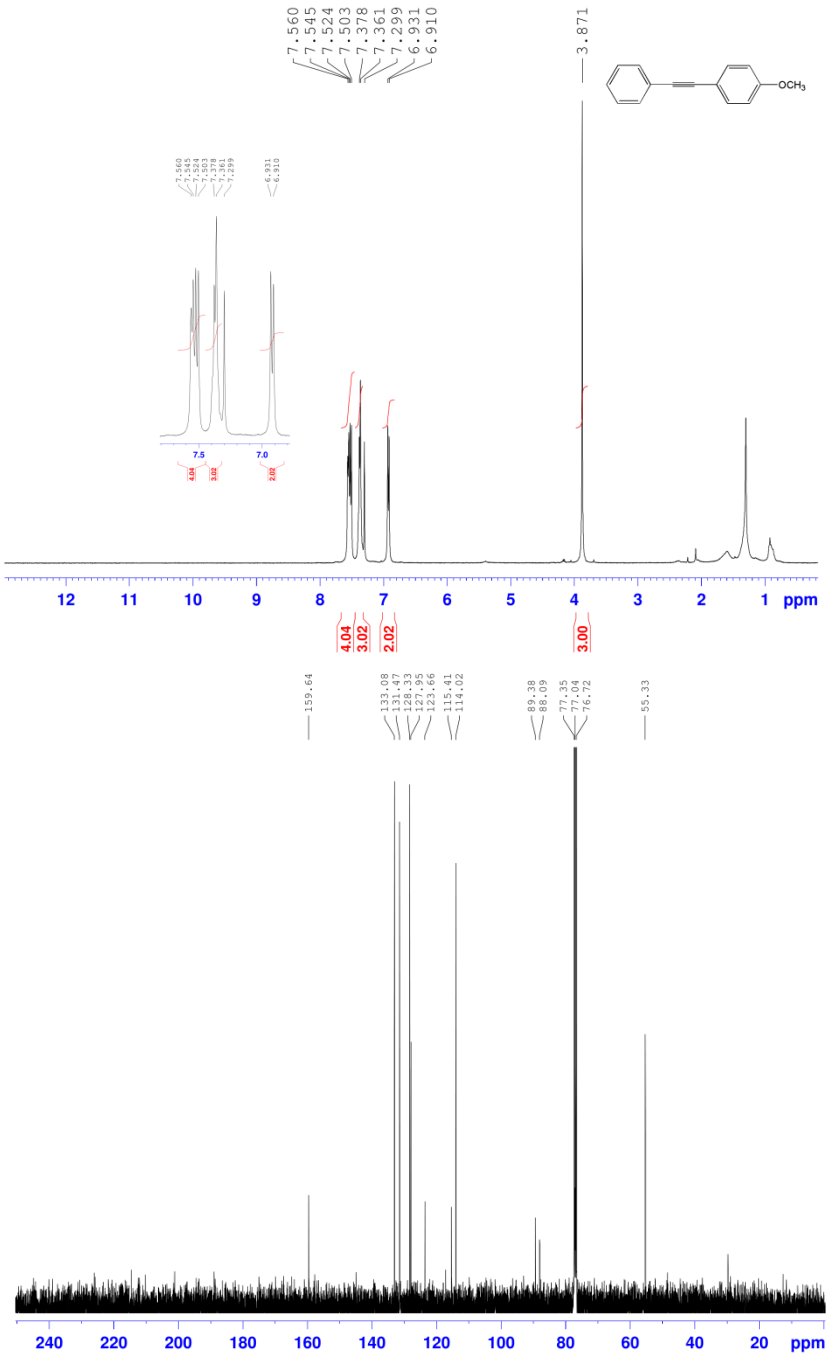
2-(phenylethynyl)thiophene (6f):

^{1}H NMR (400 MHz, $CDCl_3$): δ= 7.05 (1H, t), 7.32 (2H, d), 7.35-7.42 (3H, m), 7.54-7.59 (2H, m).
^{13}C NMR (100MHz, $CDCl_3$): δ= 82.65, 93.07, 122.96, 123.36, 127.29, 128.42, 128.46, 131.45, 132.55.



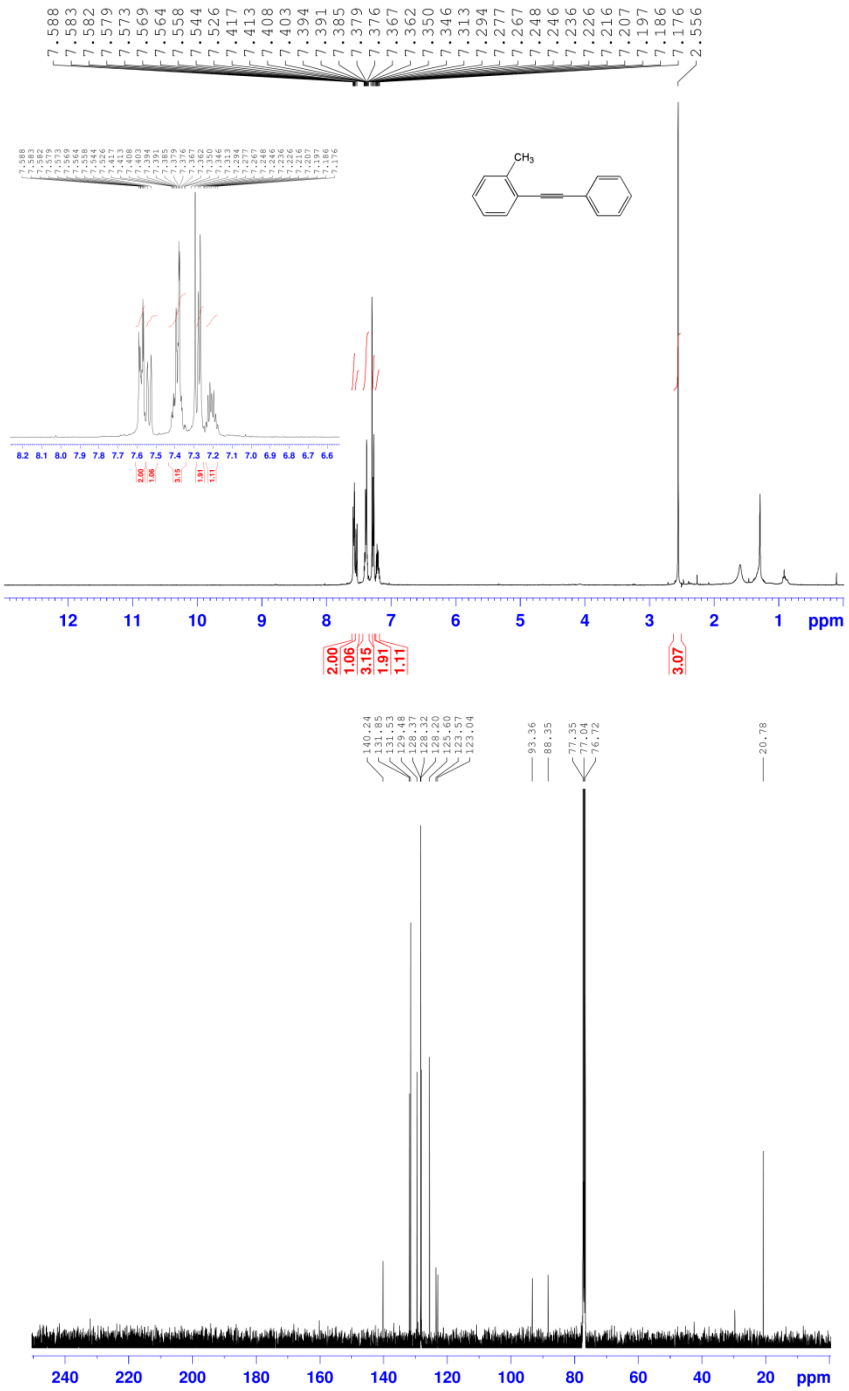
2-(p-tolylethynyl)thiophene (6g):

^{1}H NMR (400 MHz, $CDCl_3$): δ= 3.23 (3H, s), 7.04 (1H, dd), 7.19 (2H, d), 7.29-7.32 (2H, m), 7.46 (2H, d).
^{13}C NMR (100MHz, $CDCl_3$): δ= 21.58, 81.97, 93.26, 119.86, 123.60, 127.05, 127.10, 129.19, 131.36, 131.69, 138.65.



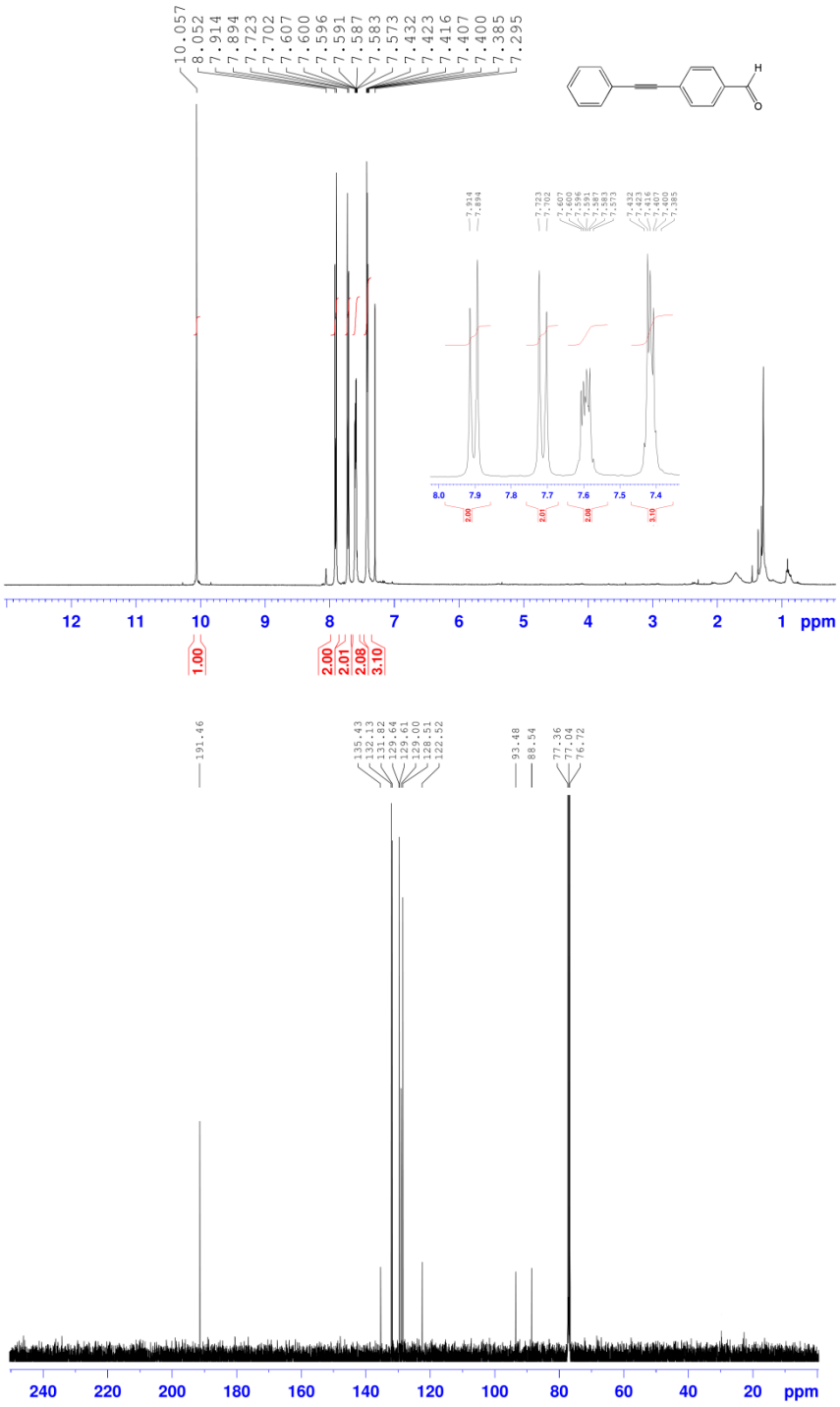
1-methoxy-4-(phenylethynyl)benzene (6h):

^{1}H NMR (400 MHz, $CDCl_3$): δ= 3.87 (3H, s), 6.92 (2H, d), 7.36-7.37 (3H, m), 7.50-7.56 (4H, m).
^{13}C NMR (100MHz, $CDCl_3$): δ= 55.33, 88.09, 89.38, 114.02, 115.41, 123.66, 127.95, 128.33, 131.47, 133.08, 159.64.



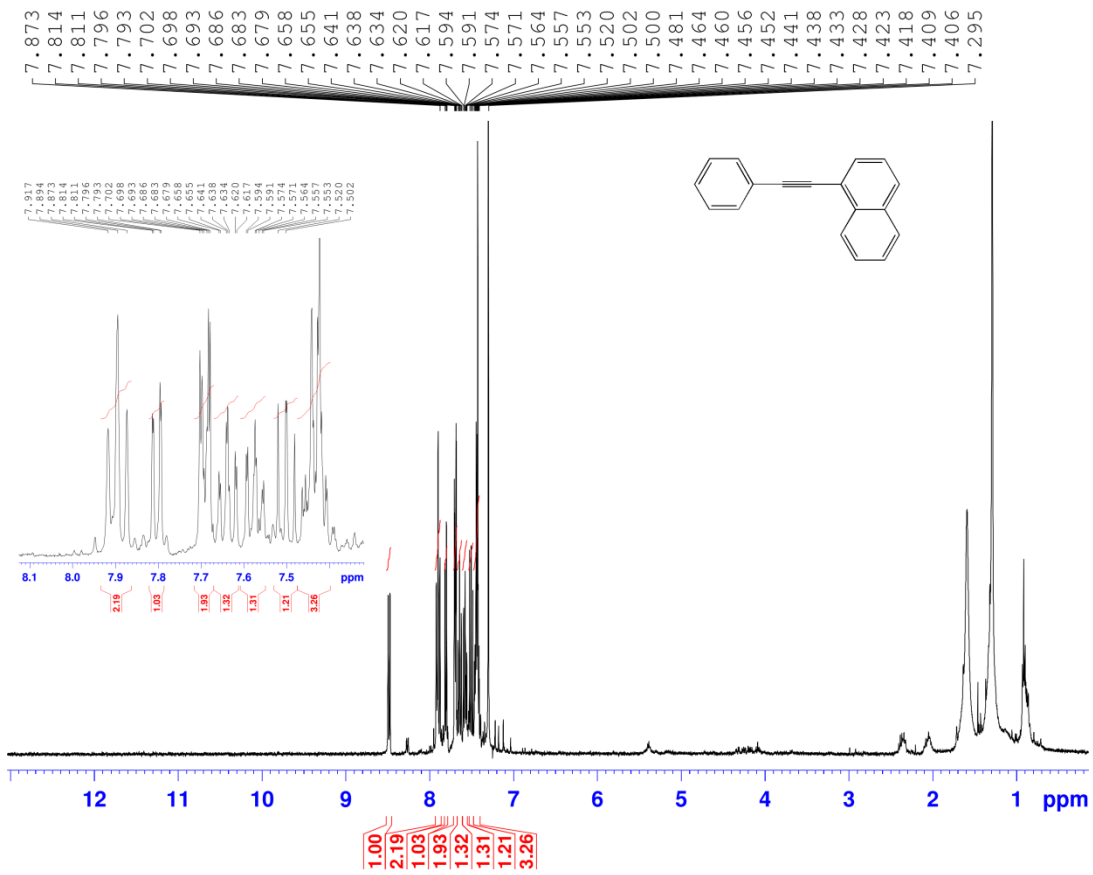
2-methyl-1-(phenylethynyl)benzene (6i):

^{1}H NMR (400 MHz, $CDCl_3$): 2.55 (3H, s): δ= 7.17-7.26 (1H, m), 7.28 (2H, d), 7.35-7.14 (3H, m), 7.53 (1H, d), 7.55-7.58 (2H, m).
^{13}C NMR (100MHz, $CDCl_3$): δ= 20.78, 88.35, 93.36, 123.04, 123.57, 125.60, 128.20, 128.32, 128.37, 129.48, 131.53, 131.85, 140.24.



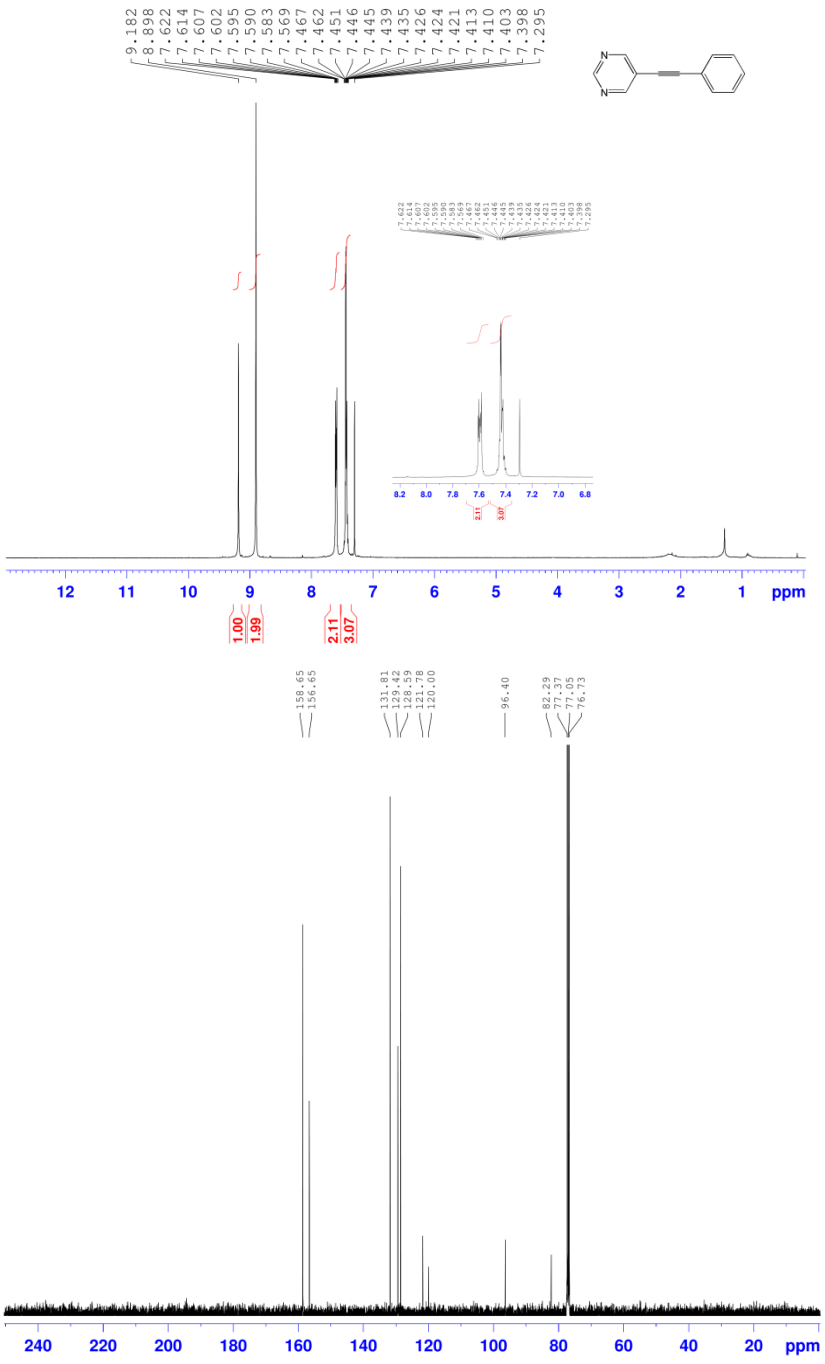
4-(phenylethynyl)benzaldehyde (6j):

^{1}H NMR (400 MHz, $CDCl_3$): δ= 7.38-7.43 (3H, m), 7.57-7.60 (2H, m), 7.71 (2H, d), 7.90 (2H, d), 10.05 (1H, s)
^{13}C NMR (100MHz, $CDCl_3$): δ= 88.54, 93.48, 122.52, 128.51, 129.00, 129.61, 129.64, 131.82, 132.13, 135.43, 191.46.



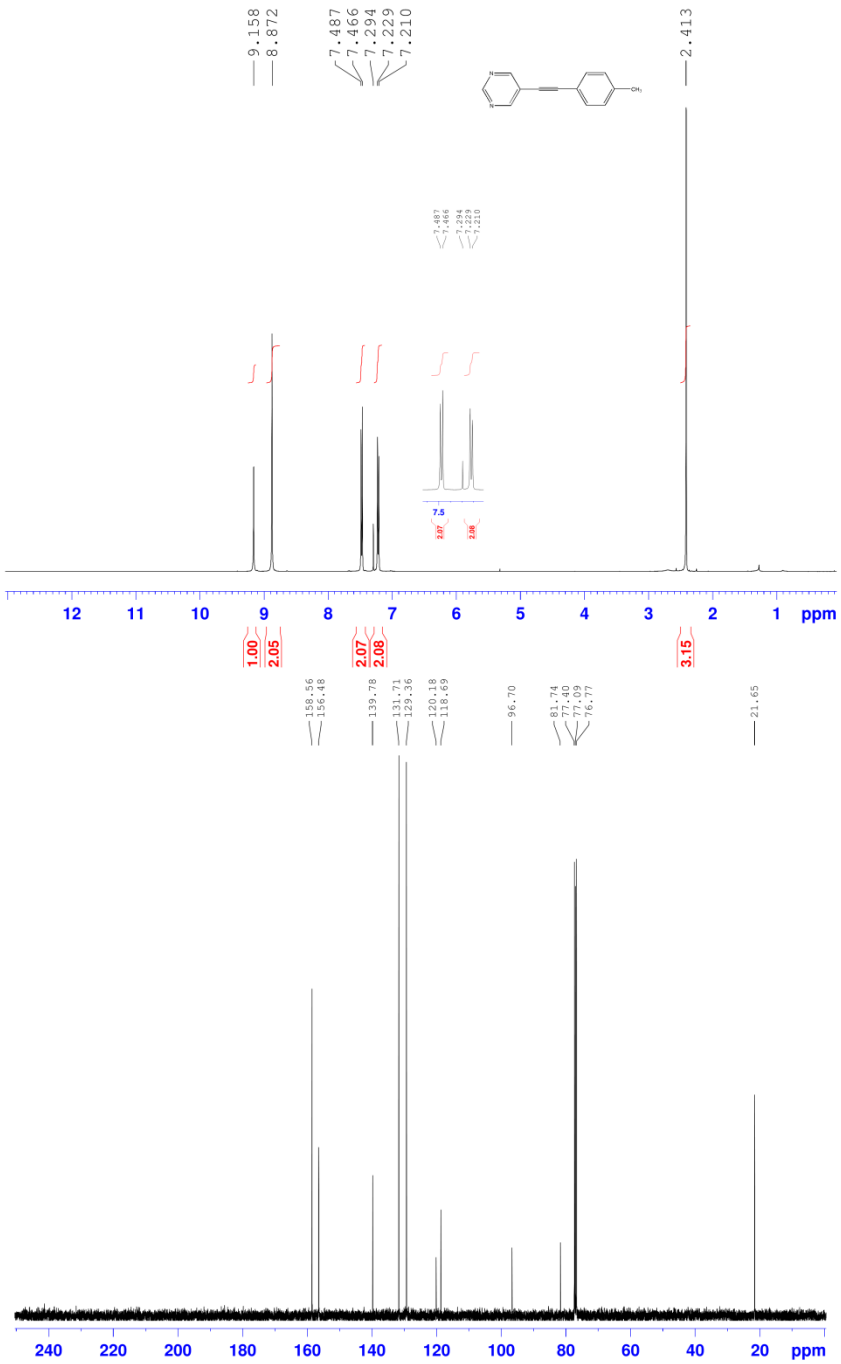
1-(phenylethynyl)naphthalene (6k):

^{1}H NMR (400 MHz, $CDCl_3$): δ= 7.45-7.46 (3H, m), 7.48-7.52 (1H, m), 7.55-7.77 (1H, m), 7.59-7.65 (1H, m), 7.67-7.70 (2H, m), 7.80 (1H, dd), 7.89 (2H, t), 8.48 (1H, d).



5-(phenylethynyl)pyrimidine (6l):

^{1}H NMR (400 MHz, $CDCl_3$): δ= 7.40-7.46 (3H, m), 7.56-7.62 (2H, m), 8.89 (2H, s), 9.18 (1H, s).
^{13}C NMR (100MHz, $CDCl_3$): δ= 82.29, 96.40, 120.00, 121.78, 128.59, 129.42, 131.81, 156.65, 158.65.



5-(p-tolylethynyl)pyrimidine (6m):

^{1}H NMR (400 MHz, $CDCl_3$): δ= 2.41 (3H, s), 7.22 (2H, d), 7.47 (2H, d), 8.87 (2H, s), 9.15 (1H, s).
^{13}C NMR (100MHz, $CDCl_3$): δ= 21.65, 81.74, 96.70, 118.69, 120.18, 129.36, 131.71, 139.78, 156.48, 158.46.