

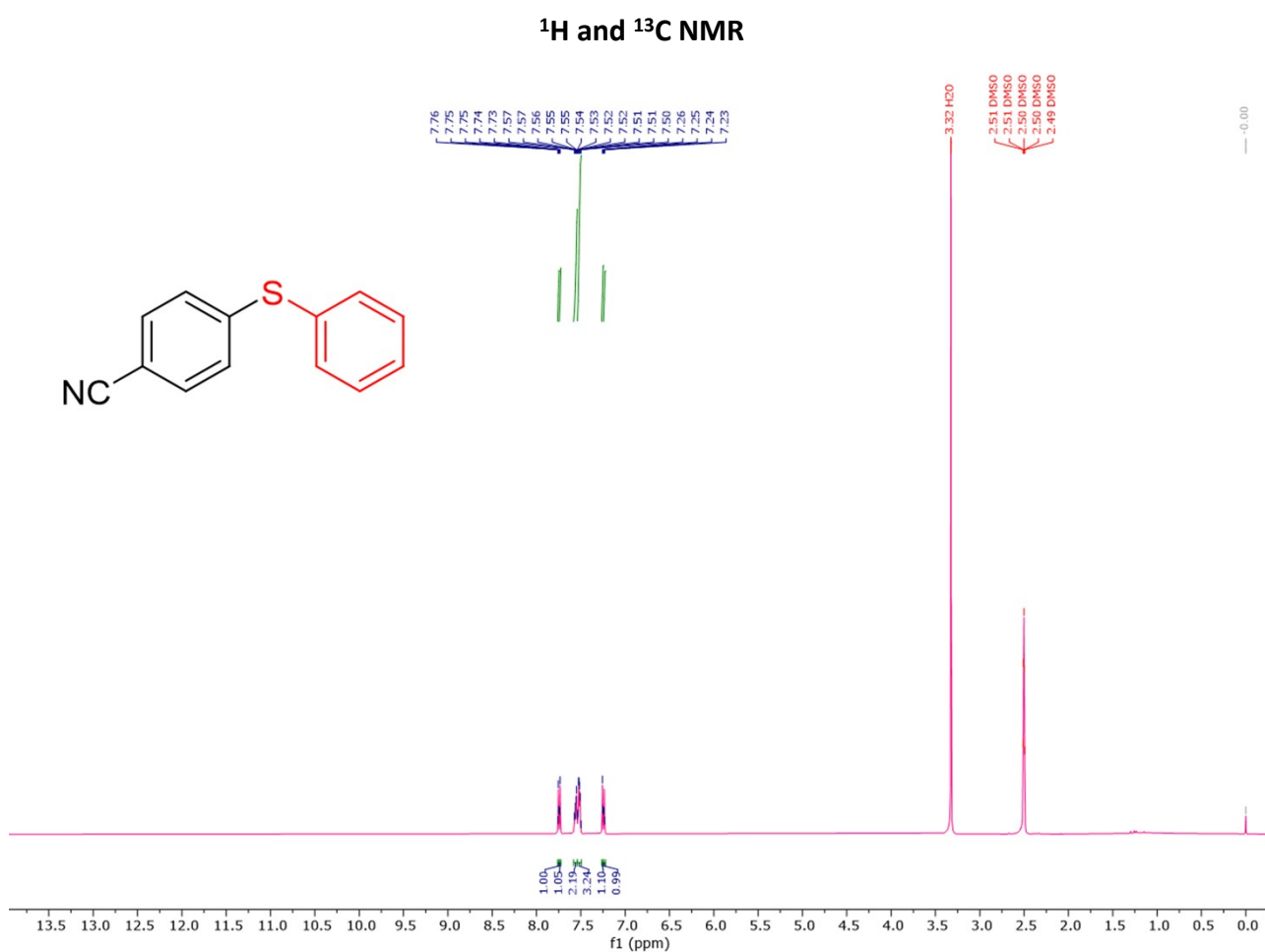
Copper Immobilized on Layered Magnetite-based Nanocatalyst for Sustainable Ullmann Cross-coupling Reaction

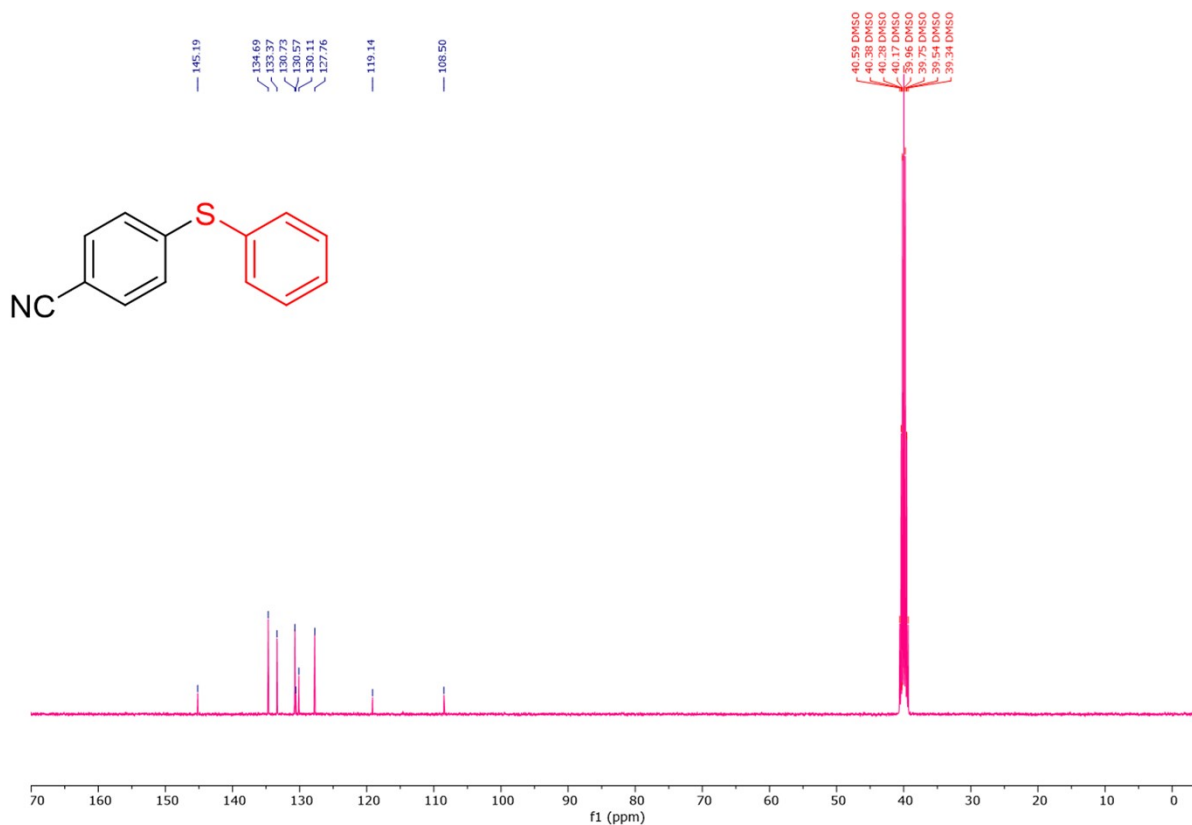
Eden Sinchana D Souza,^a Aatika Nizam^{a*}

^aDepartment of Chemistry, Christ University, Bangalore, India- 560029.

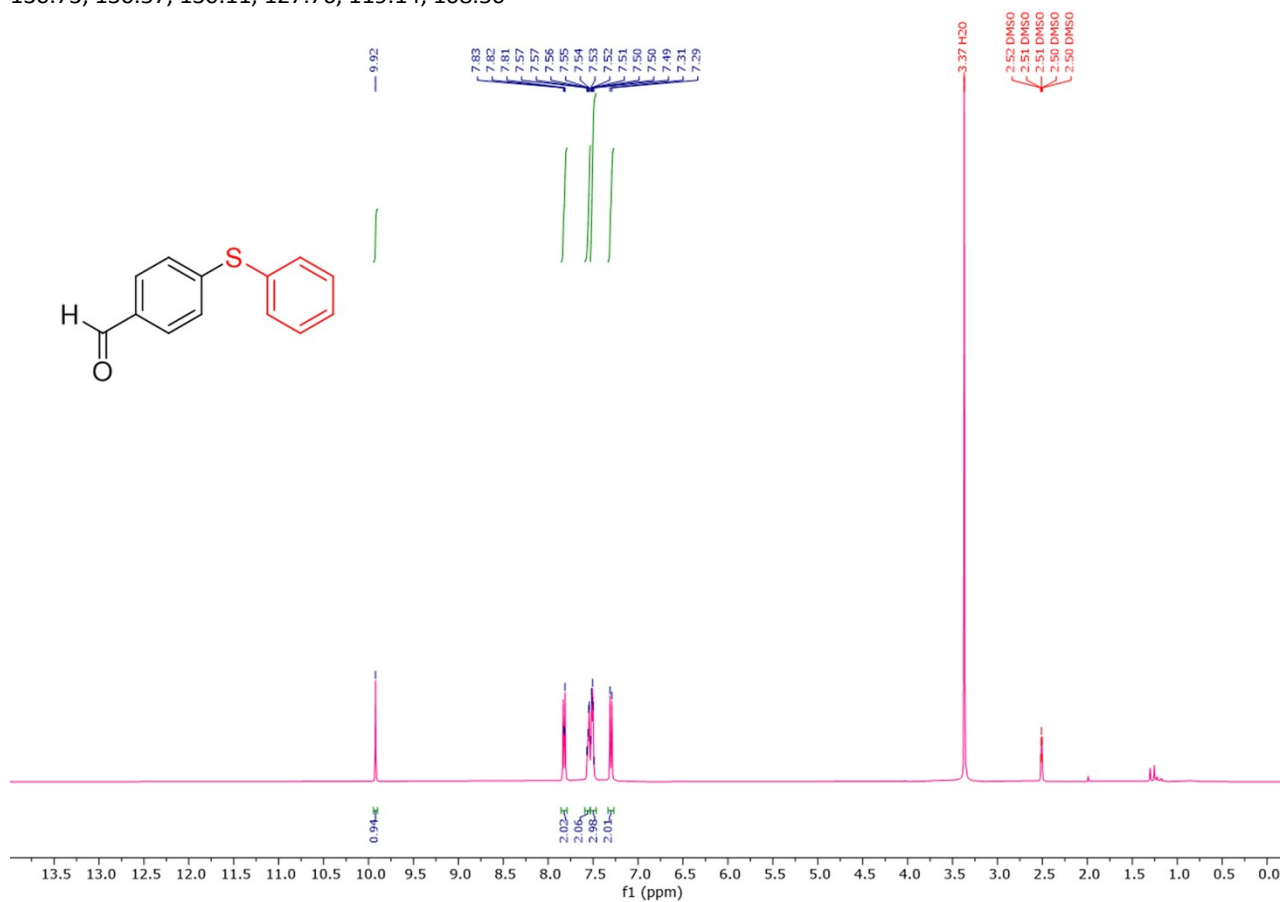
*E-mail: aatika.nizam@christuniversity.in

3a, 3h, 3j, 3k, 3m were characterized by ¹H NMR spectral analysis. The remaining compounds were identified by comparison of their TLC profiles with samples prepared following our previously reported method.¹ The corresponding NMR spectra for these compounds, already published in our earlier work, are provided in the supplementary information.

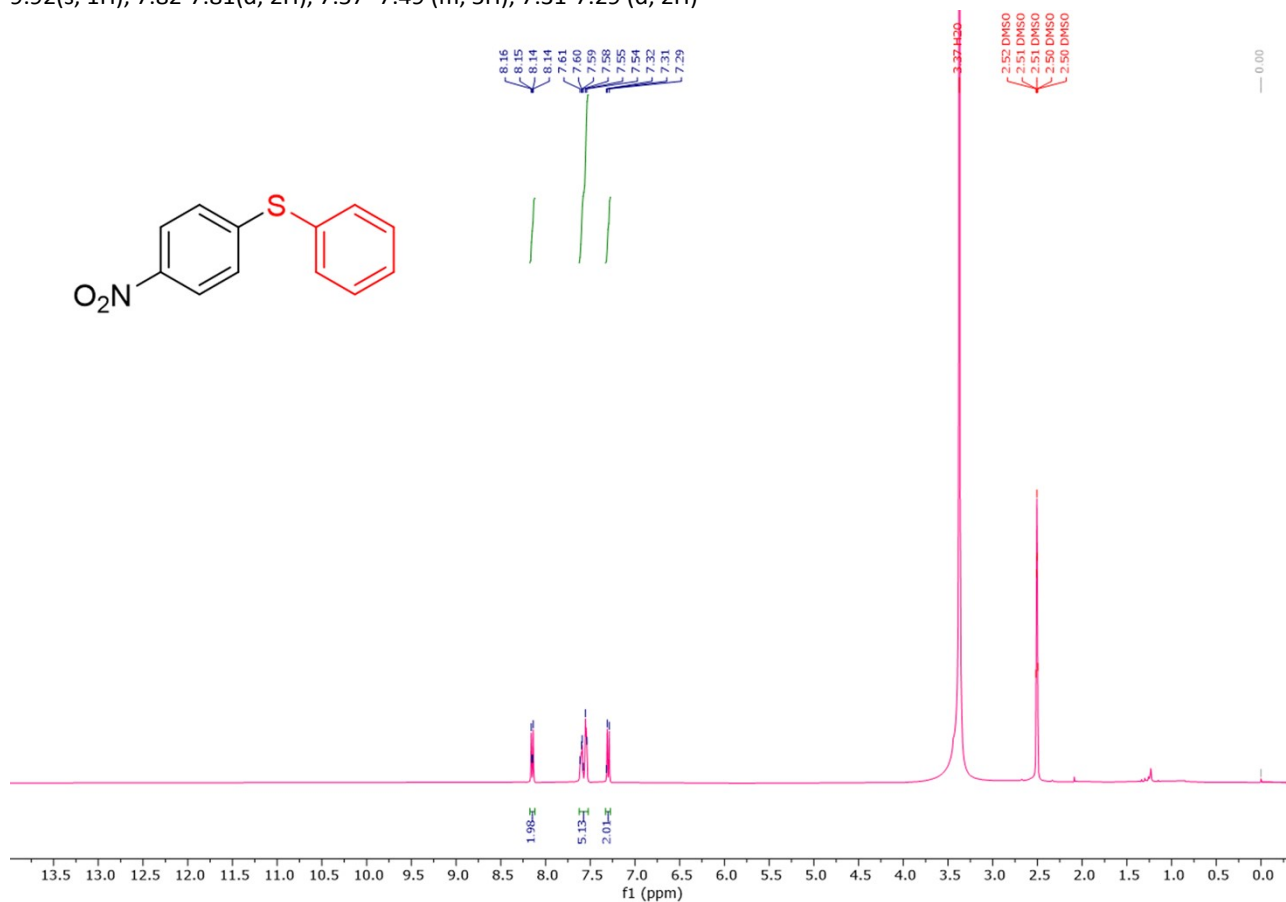




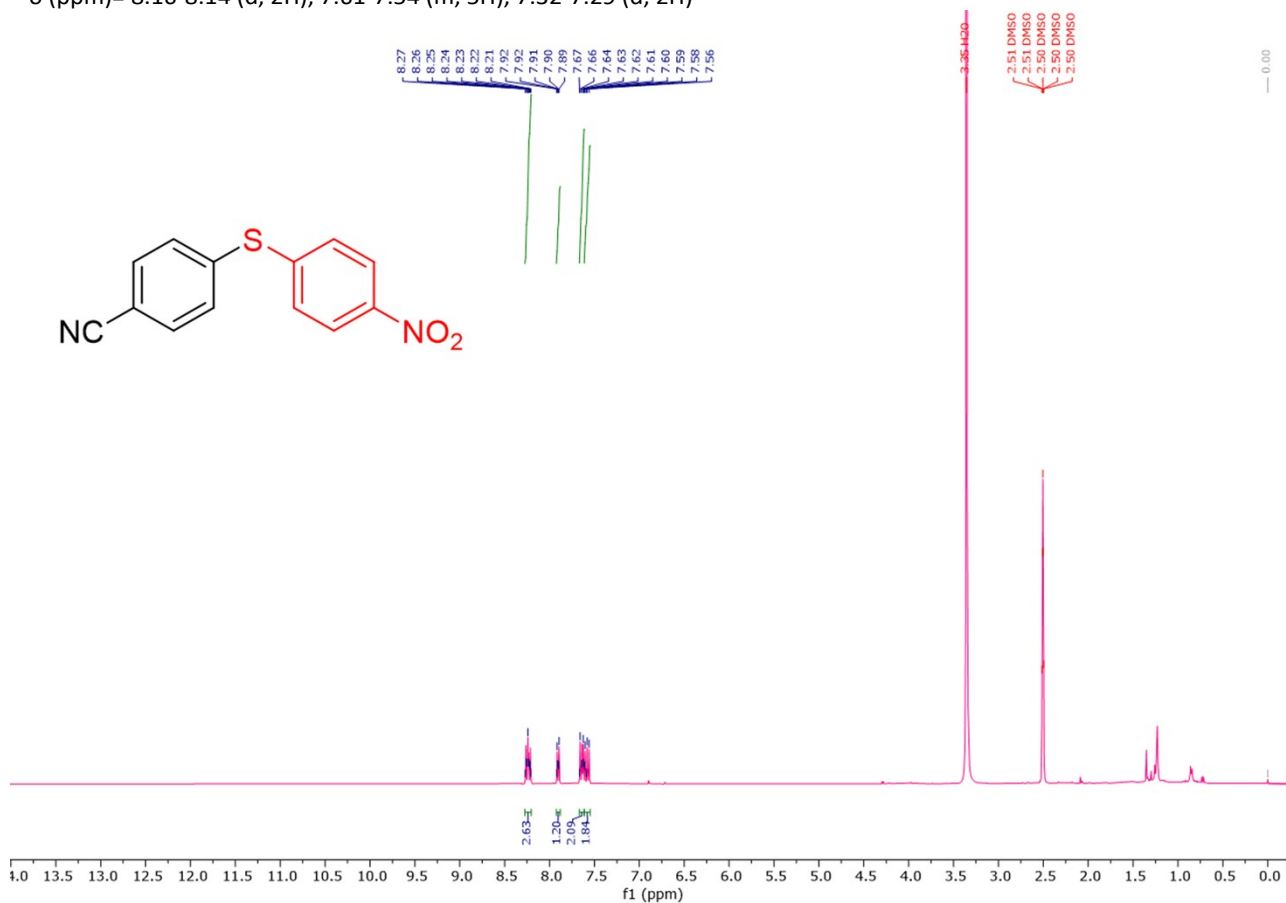
4-(phenylthio)benzonitrile (**Scheme 3, 3a and 3i**). Colorless oil (90% and 75%). ^1H NMR (400MHz, DMSO- d_6): δ (ppm) = 7.75-7.73 (d, 2H), 7.58-7.50 (m, 5H), 7.25-7.23 (d, 2H). ^{13}C NMR (400MHz, DMSO- d_6): δ (ppm) = 145.19, 134.69, 133.37, 130.73, 130.57, 130.11, 127.76, 119.14, 108.50



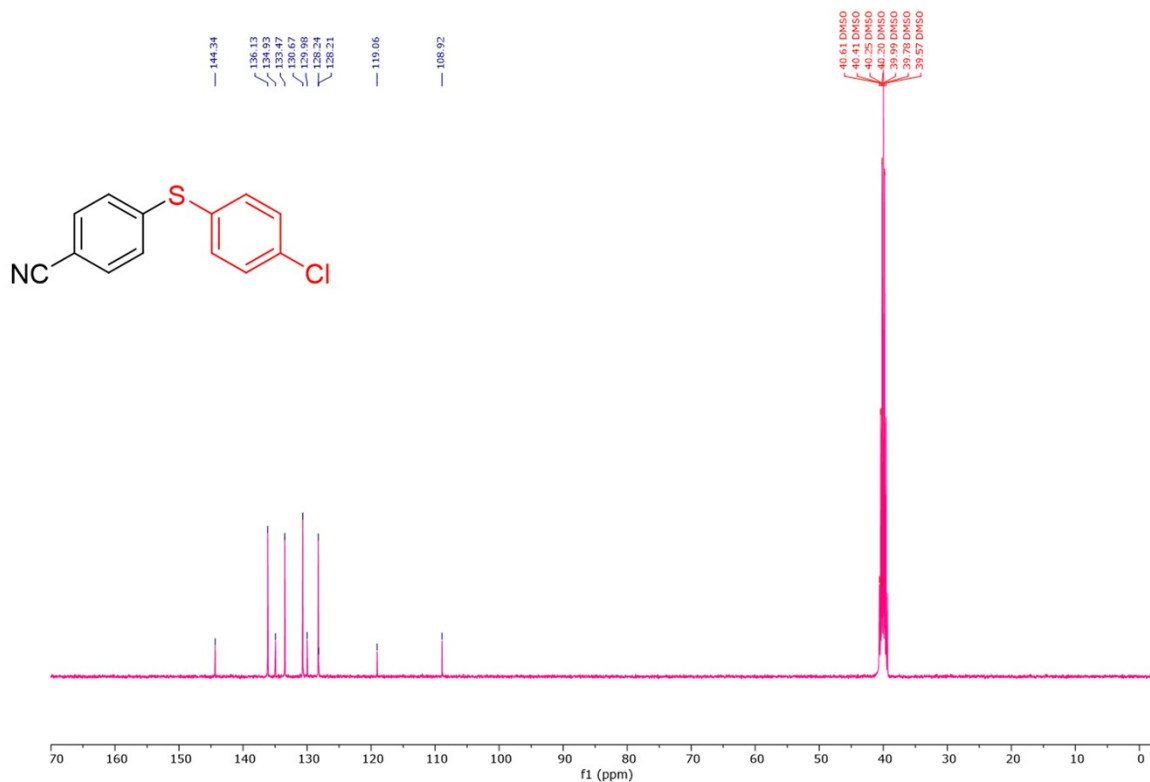
4-(phenylthio)benzaldehyde (**Scheme 3, 3c and 3g**). Colourless oil (53% and 87%). $^1\text{H NMR}$ (400MHz, $\text{DMSO-}d_6$): δ (ppm)= 9.92(s, 1H), 7.82-7.81(d, 2H), 7.57- 7.49 (m, 5H), 7.31-7.29 (d, 2H)



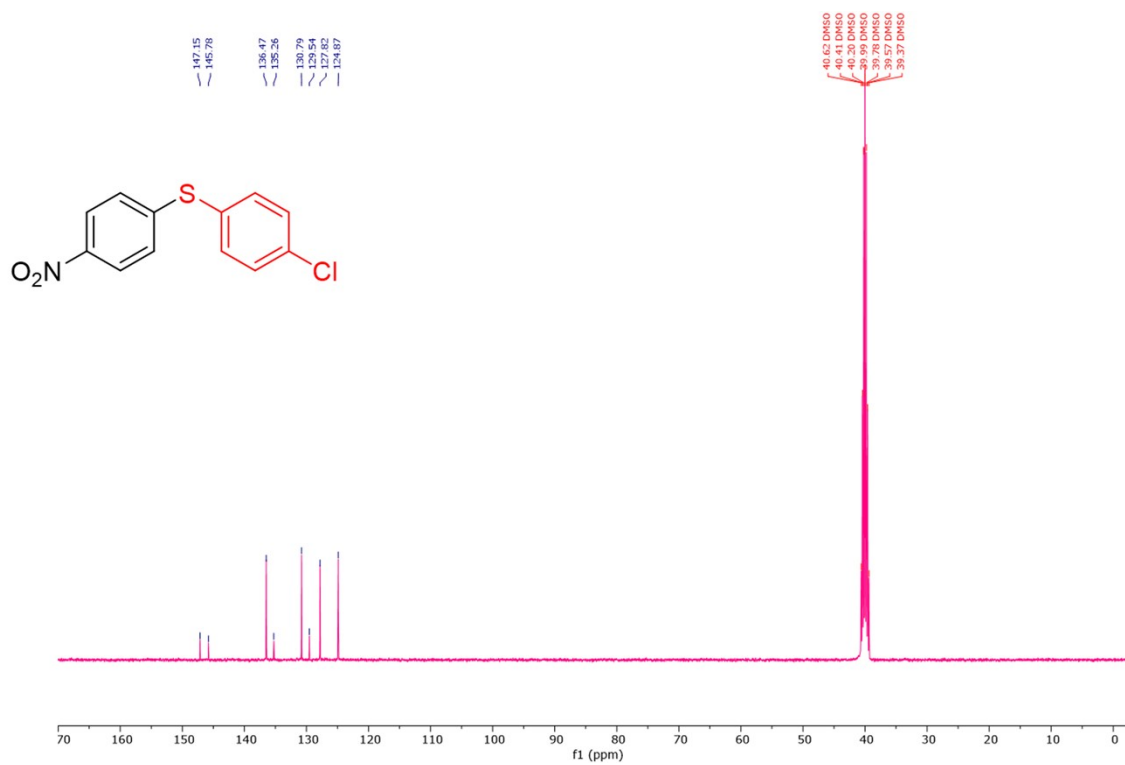
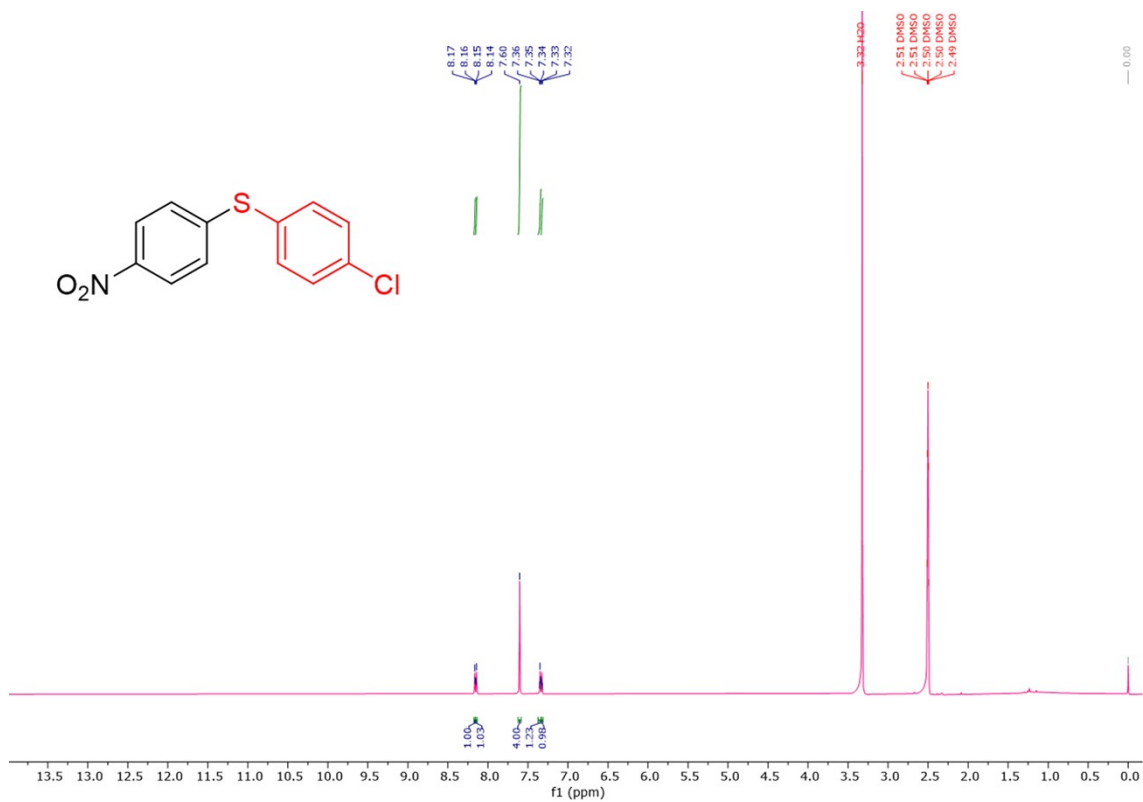
(4-nitrophenyl)(phenyl)sulfane (**Scheme 3, Entry 3d and 3o**). Yellow solid (92% and 54%). $^1\text{H NMR}$ (400MHz, $\text{DMSO-}d_6$): δ (ppm)= 8.16-8.14 (d, 2H), 7.61-7.54 (m, 5H), 7.32-7.29 (d, 2H)



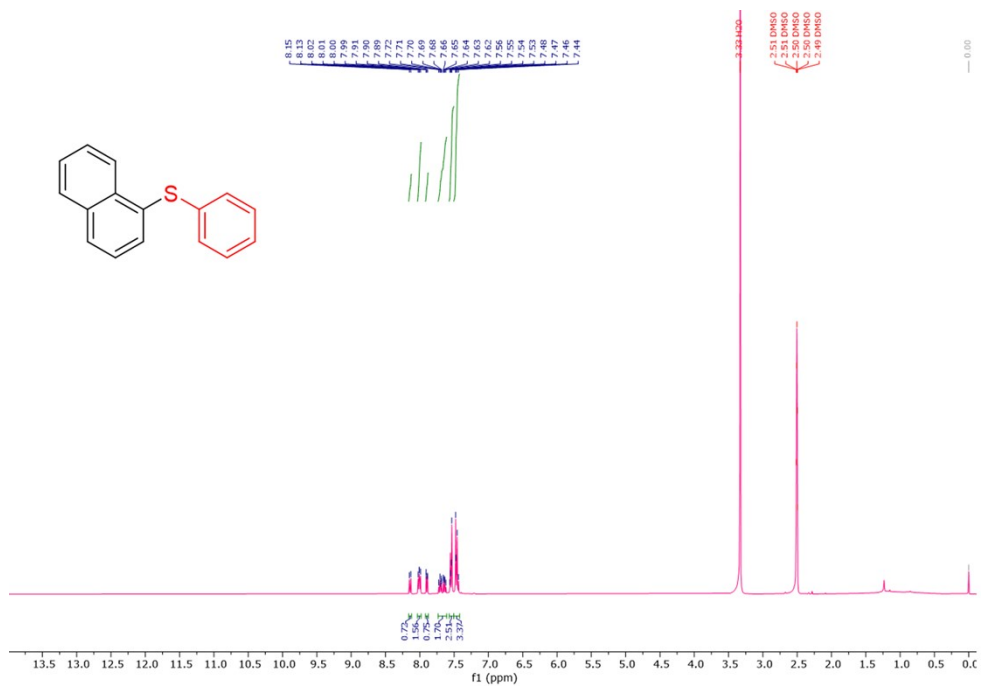
4-((4-nitrophenyl)thio)benzonitrile (**Scheme 3, Entry 3e**). Slightly yellow solid (74%). $^1\text{H NMR}$ (400MHz, $\text{DMSO-}d_6$): δ (ppm)= 8.27- 8.21 (d, 2H), 7.92-7.89 (m, 5H), 7.67- 6.56 (d, 2H)



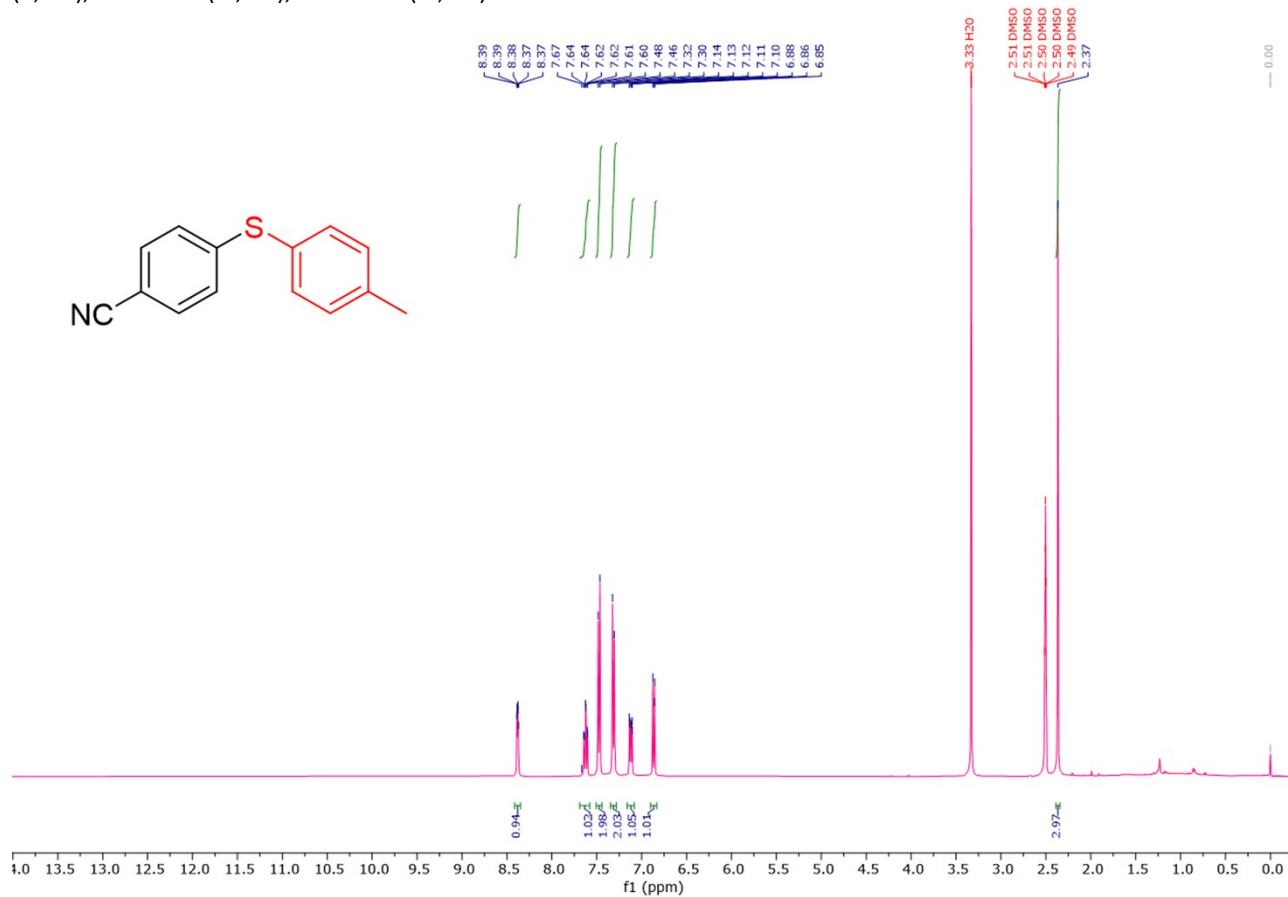
4-((4-chlorophenyl)thio)benzonitrile (**Scheme 3, Entry 3f**). White solid (91%). $^1\text{H NMR}$ (400 MHz, DMSO-d_6): δ (ppm) = 7.78-7.75 (d, 2H), 7.57-7.56 (m, 4H), 7.31-7.28 (d, 2H). $^{13}\text{C NMR}$ (400 MHz, DMSO-d_6): δ (ppm) = 144.34, 136.13, 134.93, 133.47, 130.67, 129.98, 128.24, 128.21, 119.06, 108.92



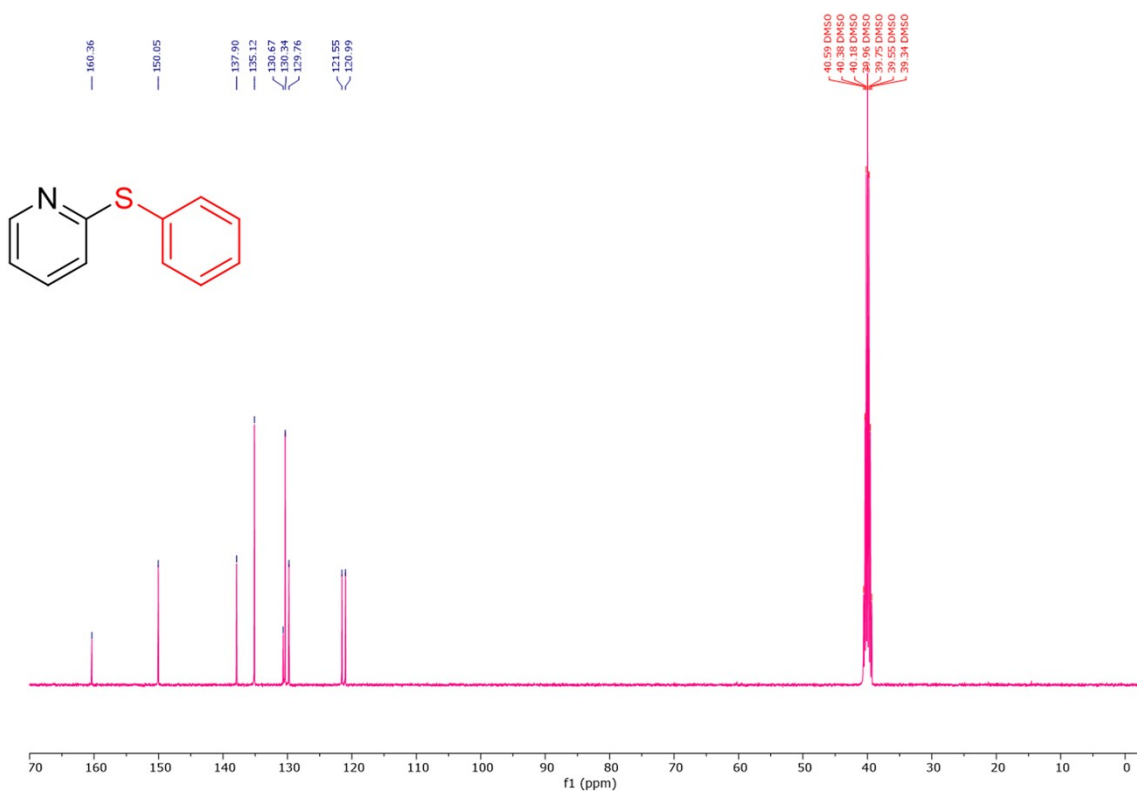
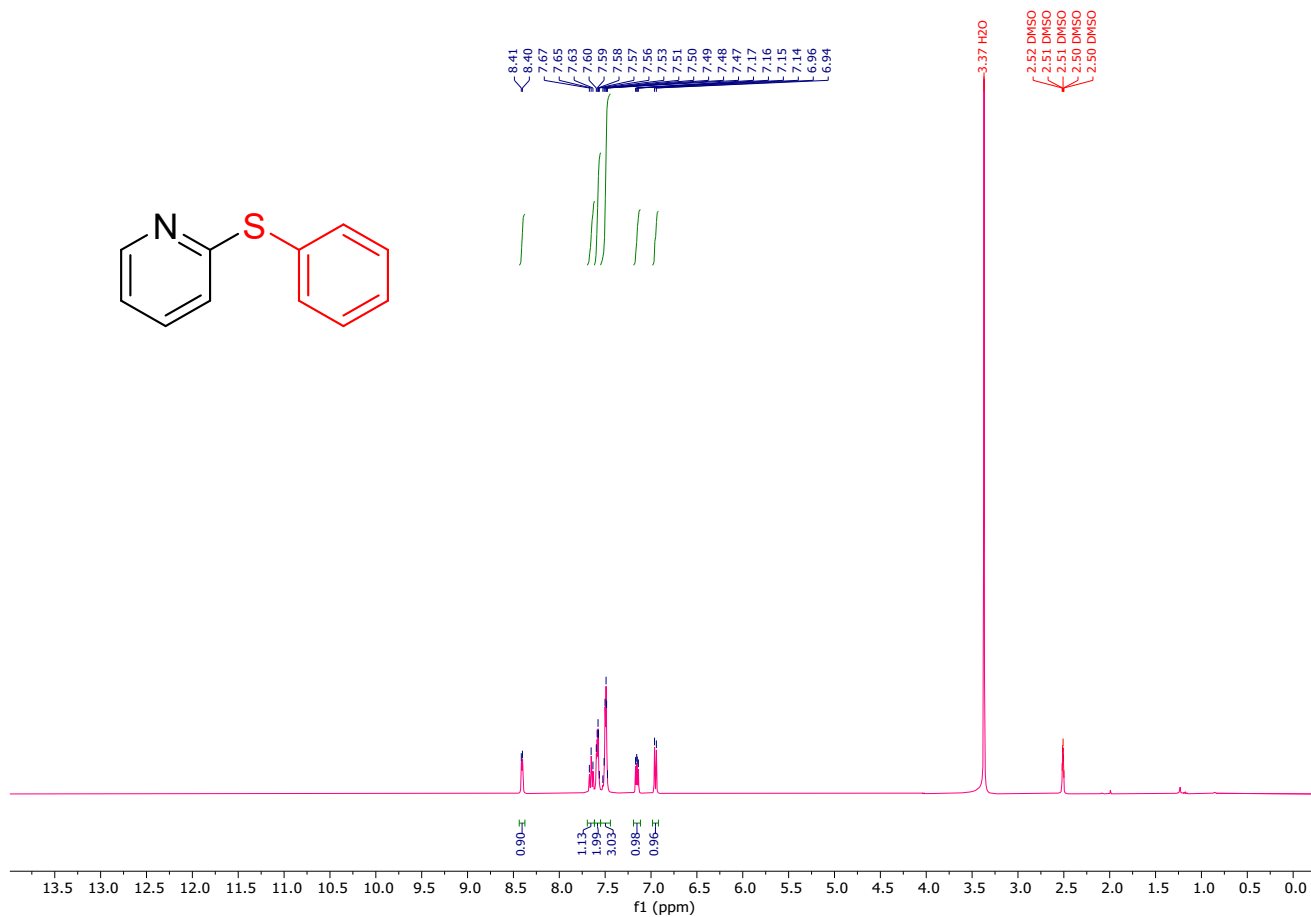
(4-chlorophenyl)(4-nitrophenyl)sulfane (**Scheme 3, Entry 3h**). Yellow solid (87%). ¹H NMR (400MHz, DMSO-*d*₆): δ (ppm)= 8.17-8.14 (d, 2H), 7.58-7.50 (m, 4H), 7.25-7.23 (d, 2H). ¹³C NMR (400MHz, DMSO-*d*₆): δ (ppm)= 147.15, 145.78, 136.47, 135.26, 130.79, 129.54, 127.82, 124.87



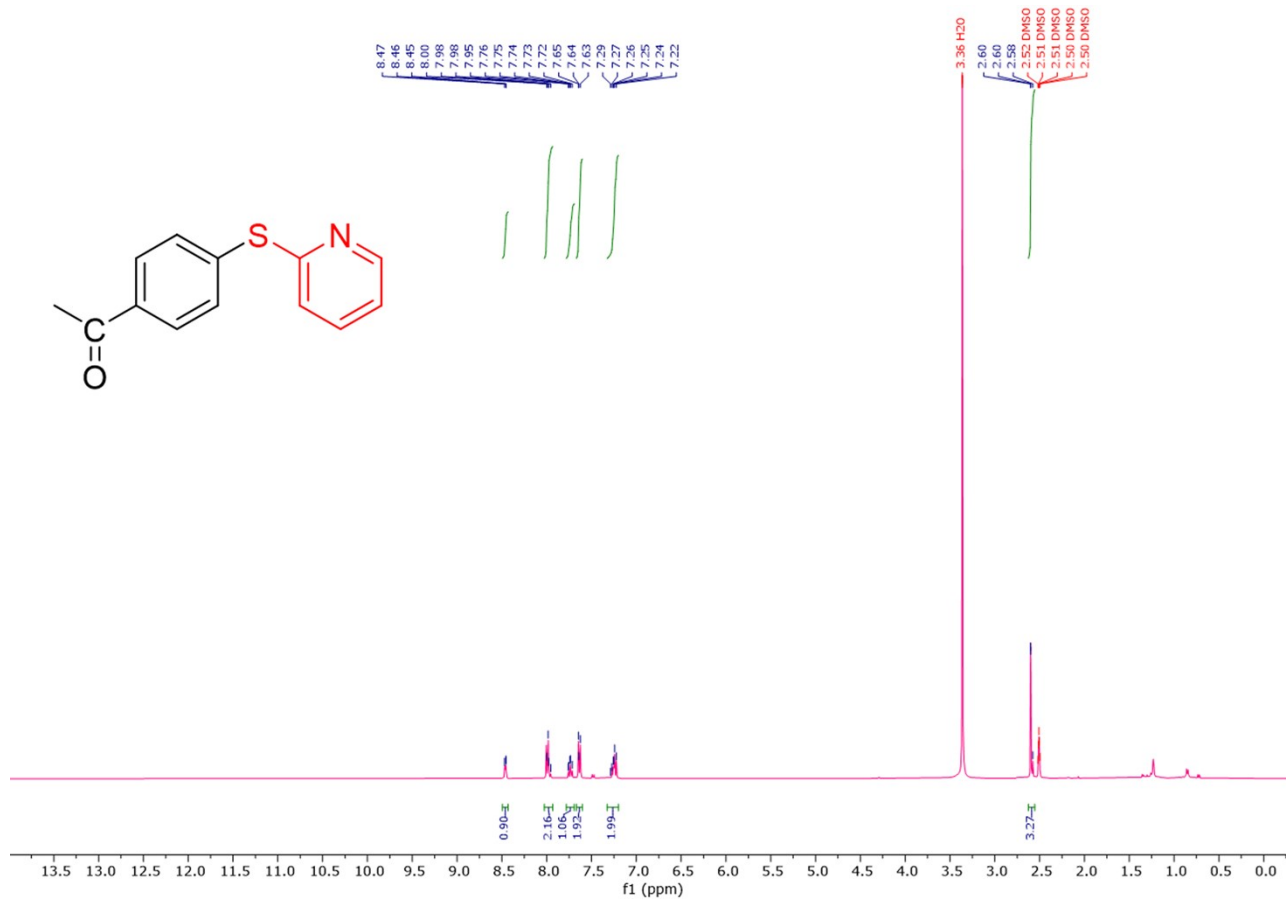
Naphthalen-1-yl(phenyl)sulfane (**Scheme 3, Entry 3j**). White solid (77%). $^1\text{H NMR}$ (400MHz, DMSO-d_6): δ (ppm)= 8.15- 8.13 (d, 1H), 8.02- 7.89 (m, 3H), 7.73- 7.44 (m, 8H)



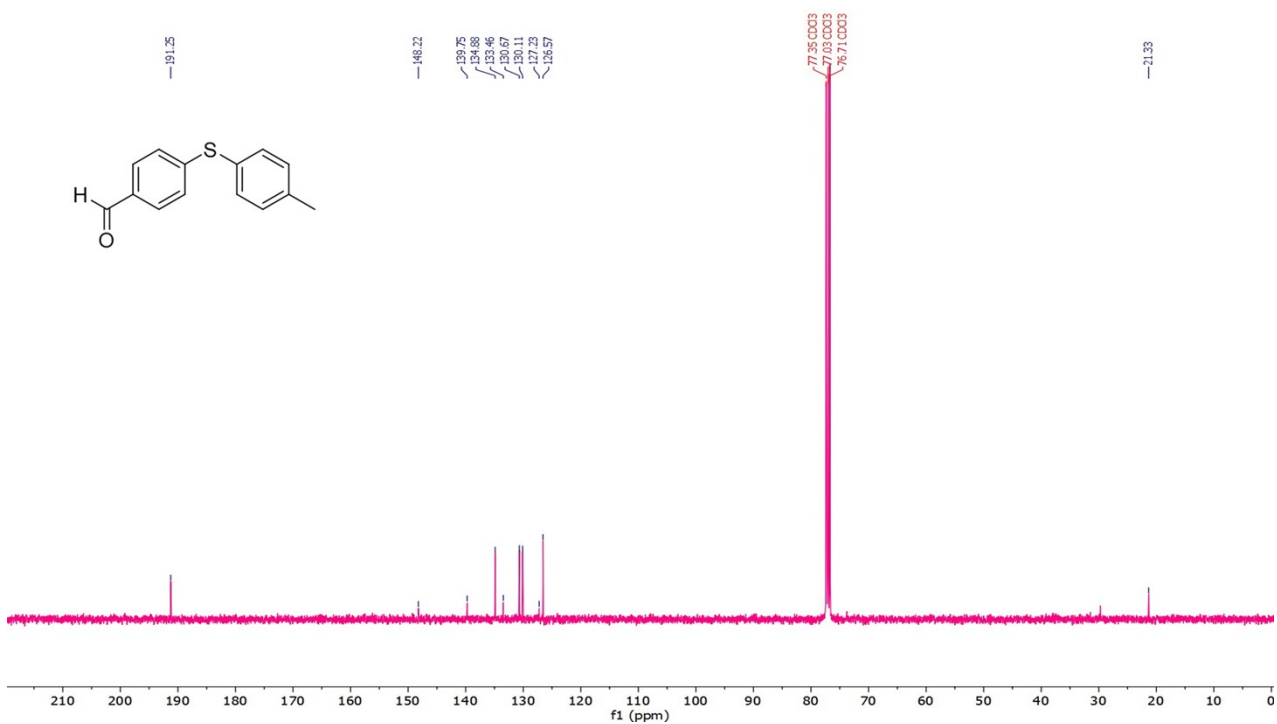
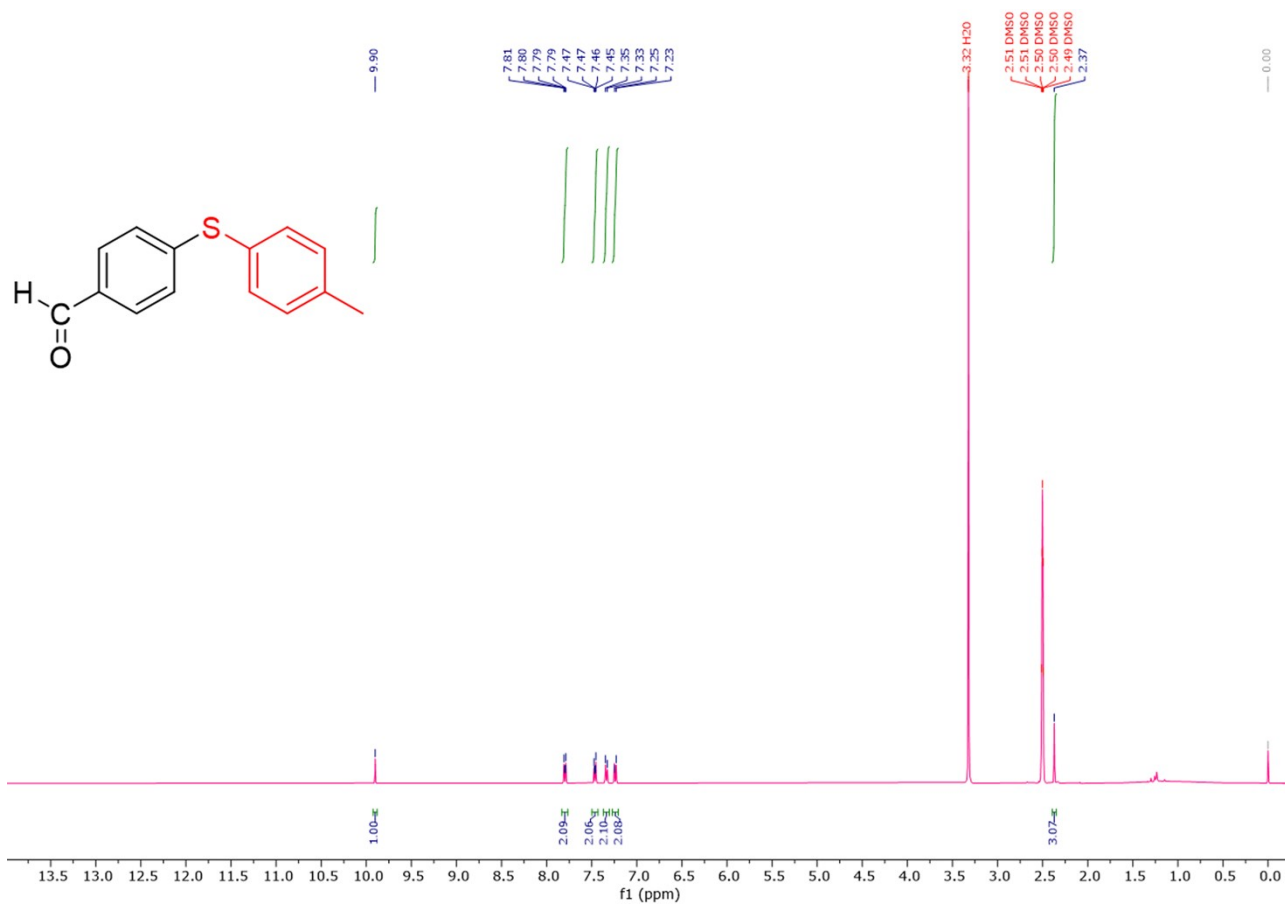
(4-tolylthio)benzotrile (**Scheme 3, Entry 3k**). Colorless liquid (94%). $^1\text{H NMR}$ (400MHz, DMSO-d_6): δ (ppm)= 8.39- 8.17 (d, 2H), 7.64-7.30 (m, 5H), 7.14- 6.86 (d, 2H), 2.37 (s, 3H)



2-(phenylthio)pyridine (**Scheme 3, Entry 3I**). White solid (81%). ¹H NMR (400MHz, DMSO- *d*₆): 8.41-8.40 (d, 1H), 7.67-7.47 (m, 6H), 7.17- 6.94 (d, 2H). ¹³C NMR (400MHz, DMSO- *d*₆): δ (ppm)= 160.36, 150.05, 137.90, 135.12, 130.67, 130.34, 129.76, 121.55, 120.99



1-(4-(pyridine-2-ylthio)phenyl)ethan-1-one (**Scheme 3, Entry 3p**). Colourless liquid (50%). ¹H NMR (400MHz, DMSO- *d*₆): δ (ppm)= 8.47- 8.45 (d, 1H), 8.00- 7.47 (m, 5H), 7.29- 7.22 (d, 2H), 2.6- 2.58 (s, 3H)



4-(*p*-tolylthio)benzaldehyde (**Scheme 3, Entry 3q**). white solid (92%). ¹H NMR (400MHz, DMSO-*d*₆): δ (ppm)= 9.90 (s, 1H), 7.81-7.79 (d, 2H), 7.47- 7.33 (m, 4H), 7.25- 7.23 (d, 2H), 2.37 (s, 3H). ¹³C NMR (400MHz, CDCl₃): δ (ppm)= 191.25, 148.22, 139.75, 134.88, 133.46, 130.67, 130.11, 127.23, 126.57, 21.33

References

- 1 E. S. D Souza and A. Nizam, *Catal Today*, 2025, 460, 115486.

