

Electronic Supplementary Information

for the manuscript entitled

Carbon-sulphur cross coupling reactions catalyzed by nickel-based coordination polymers based on metalloligands

Gulshan Kumar, Firasat Hussain and Rajeev Gupta*

Department of Chemistry, University of Delhi, Delhi – 110 007 (India)

Fax: +91-11-2766 6605; E-mail: rgupta@chemistry.du.ac.in

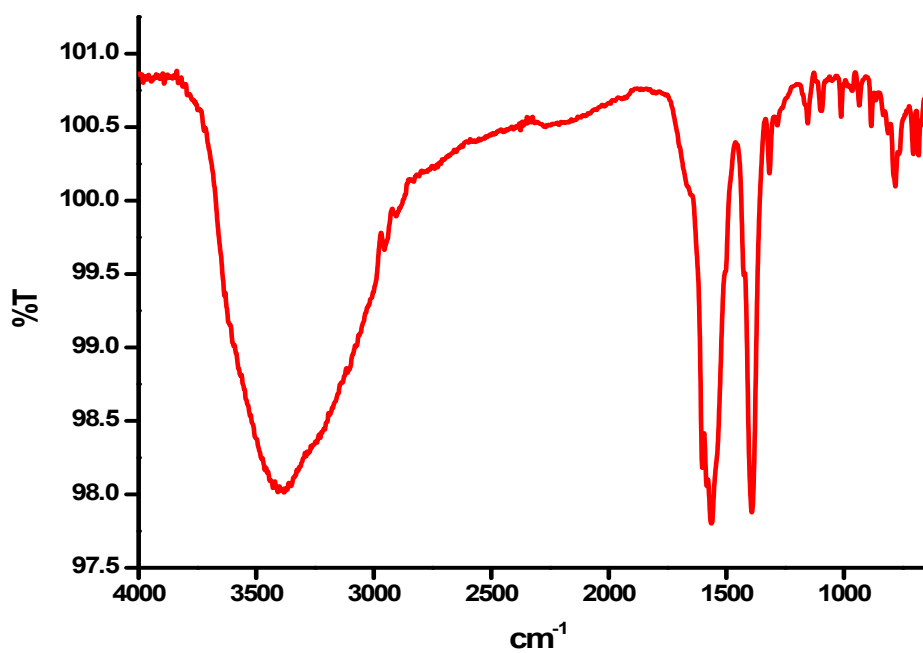


Figure S1. FTIR spectrum of **1-Ni**.

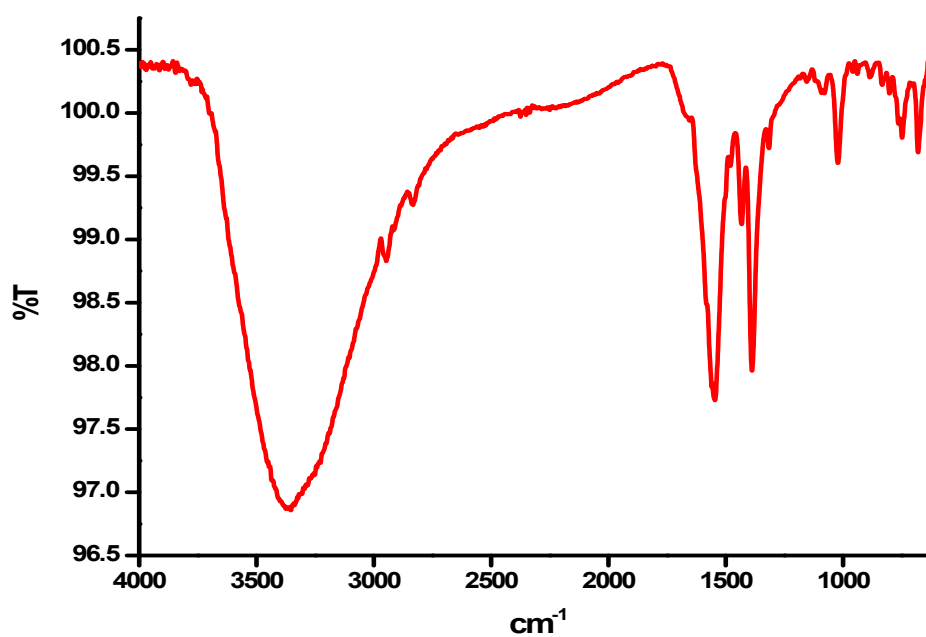


Figure S2. FTIR spectrum of **2-Ni**.

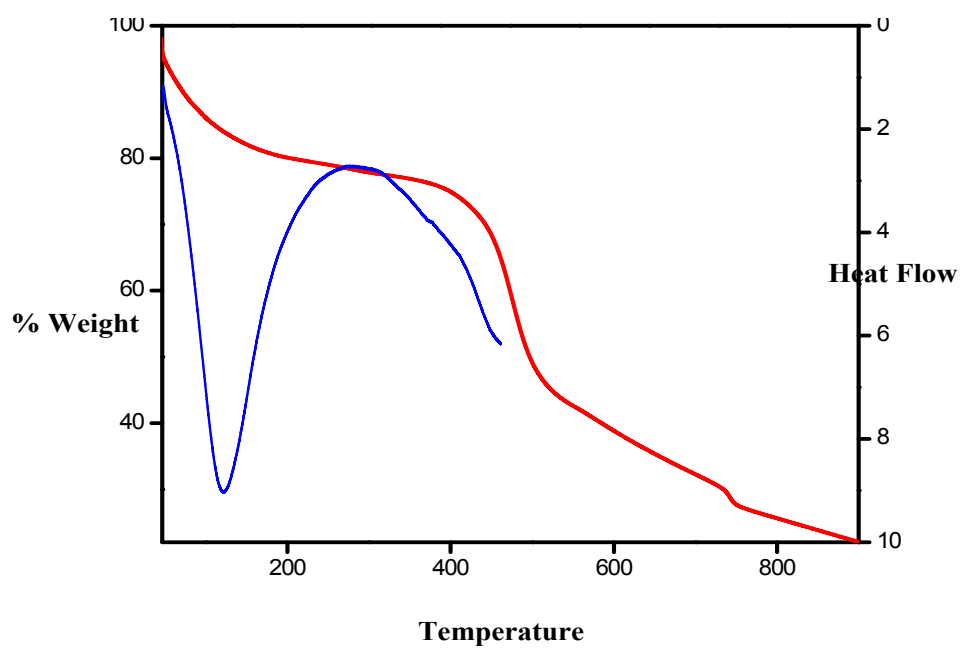


Figure S3. Thermal Gravimetric Analysis (TGA, red trace) and Differential Scanning Calorimetric (DSC, blue trace) plots for **1-Ni**.

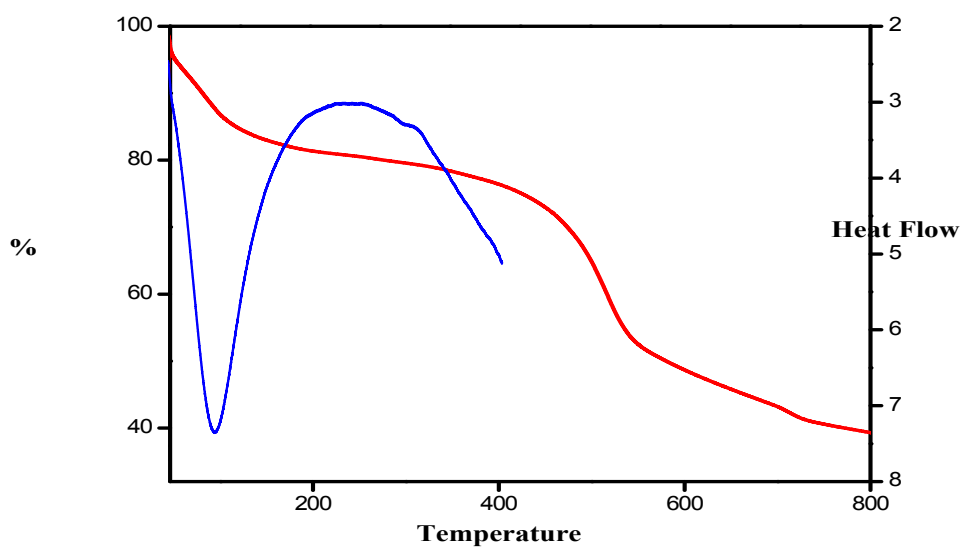


Figure S4. Thermal Gravimetric Analysis (TGA, red trace) and Differential Scanning Calorimetric (DSC, blue trace) plots for **2-Ni**.

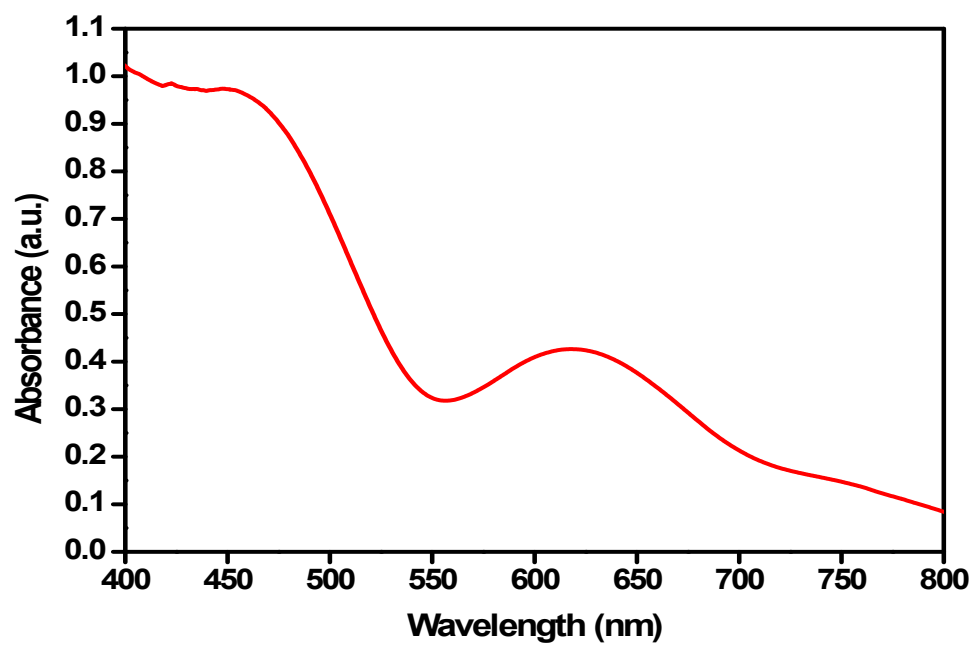


Figure S5. Diffuse reflectance UV-Vis spectrum of **1-Ni**.

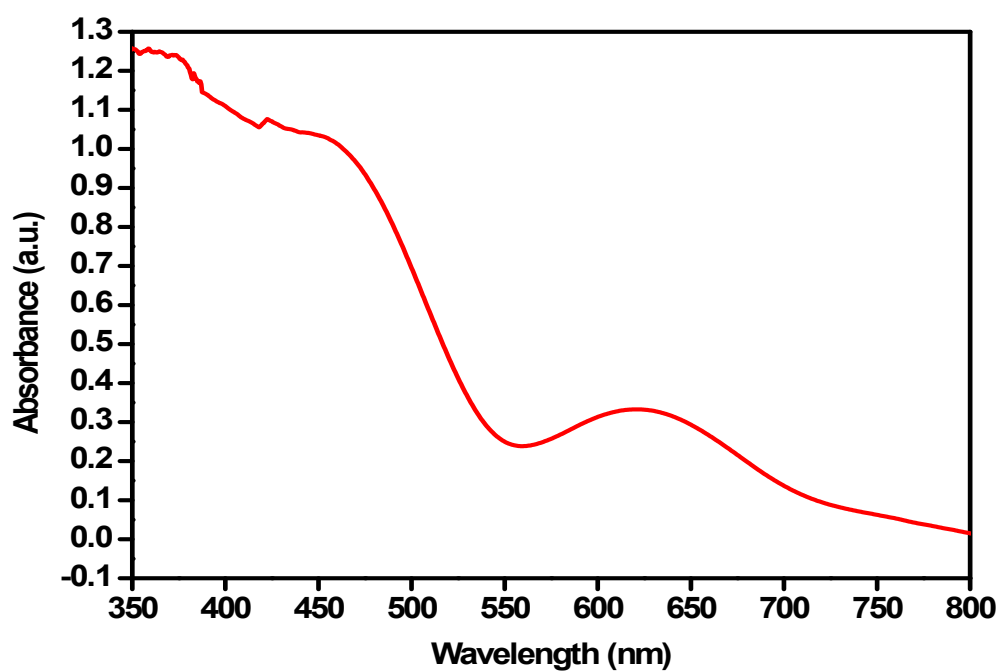


Figure S6. Diffuse reflectance UV-Vis spectrum of **2-Ni**.

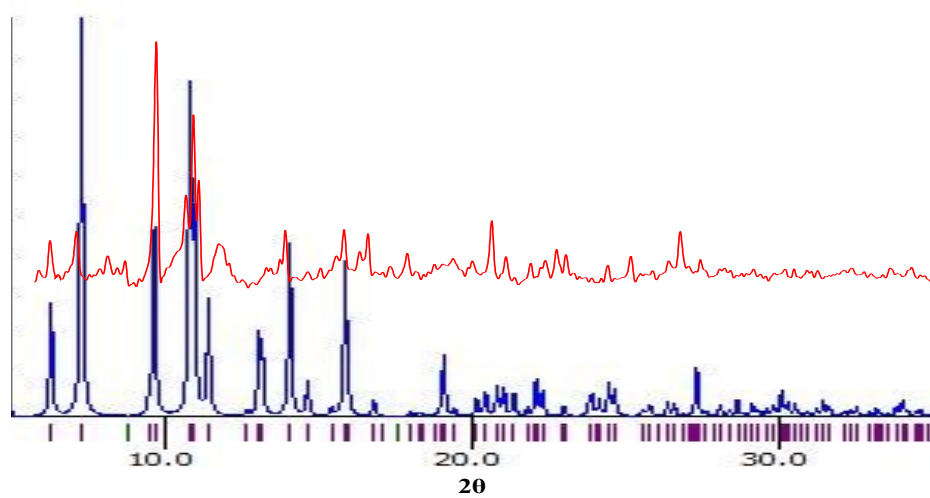


Figure S7. X-ray Powder Diffraction (XRPD) pattern for as-synthesized **1-Ni** (red trace) and the one simulated from the Mercury 3.8 using the single crystal data (blue trace).

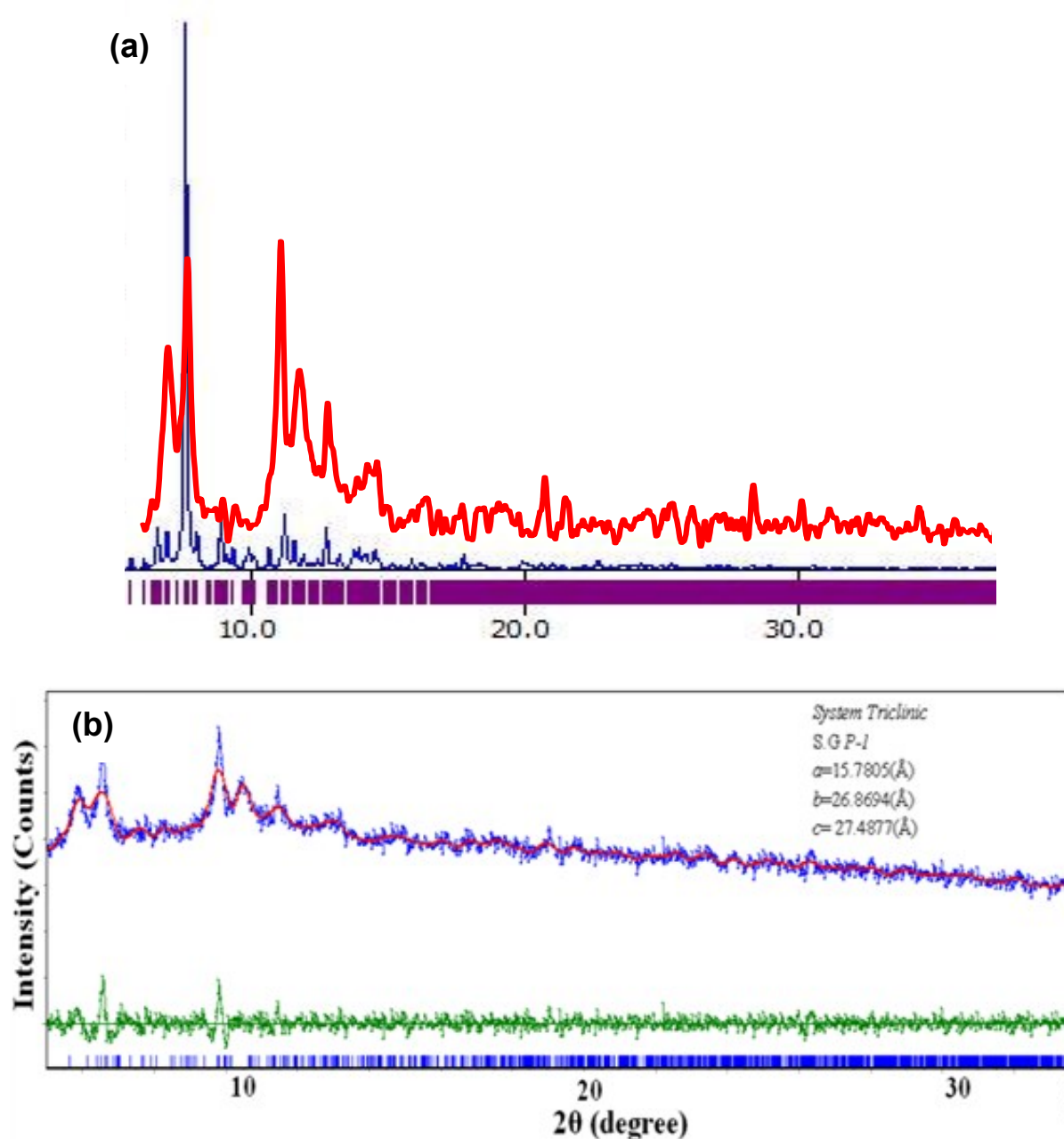


Figure S8. (a) X-ray Powder Diffraction (XRPD) pattern for as-synthesized **2-Ni** (red trace) and the one simulated from the Mercury 3.8 using the single crystal data (blue trace). (b) Le Bail refinement of XRPD pattern of **2-Ni**. The observed, calculated (profile matching), and difference profiles are given in blue, red, and olive lines, respectively. Generated Bragg positions are provided as blue vertical lines.

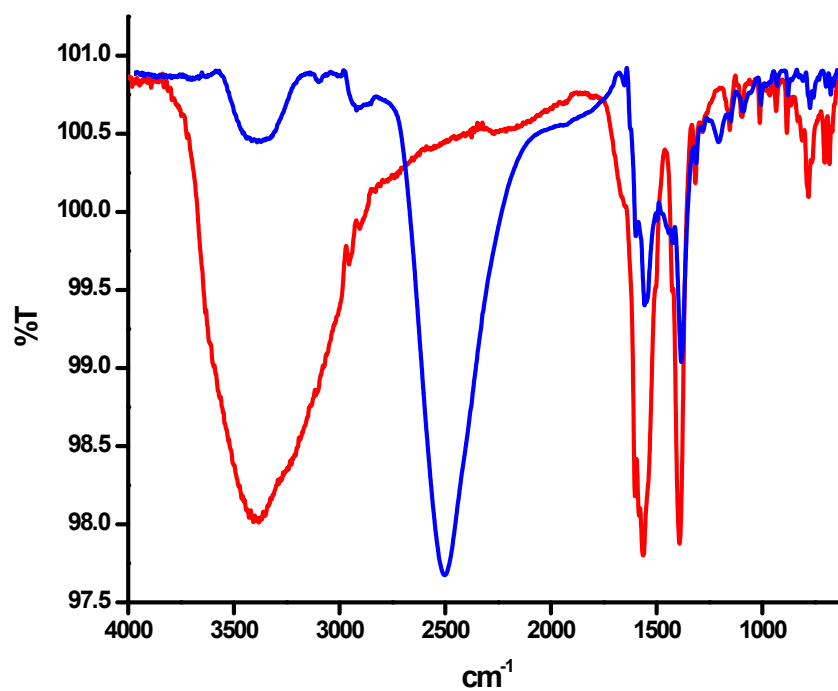


Figure S9. FTIR spectra of as synthesized **1-Ni** (red trace) and after D₂O exchange experiment (blue trace).

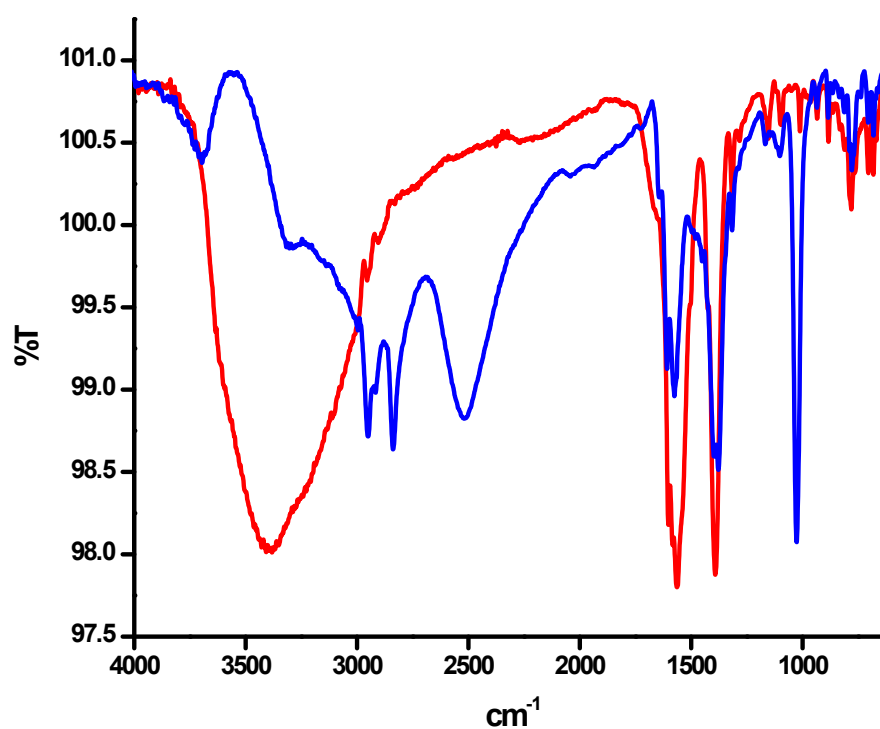


Figure S10. FTIR spectra of as synthesized **1-Ni** (red trace) and after CH₃OH exchange experiment (blue trace).

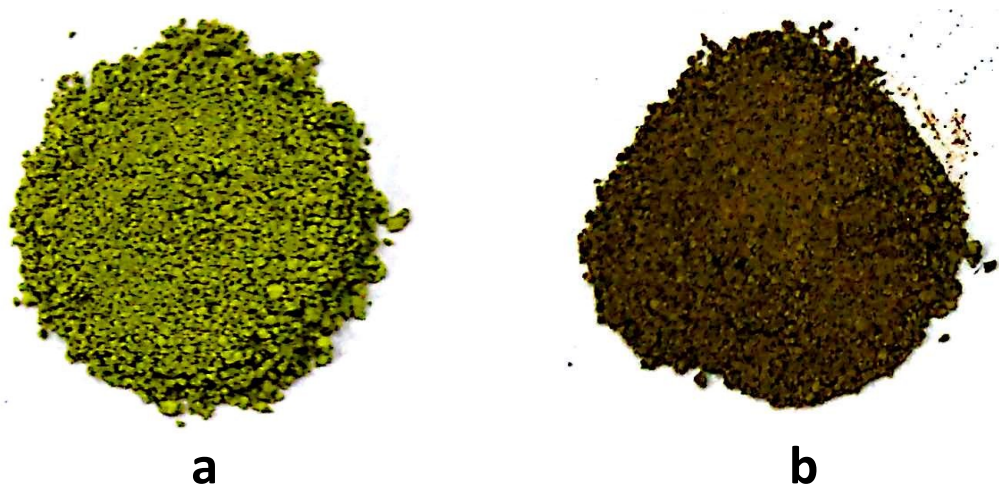


Figure S11. Optical images of a solid sample of (a) as synthesized **1-Ni** and after (b) iodine adsorption.

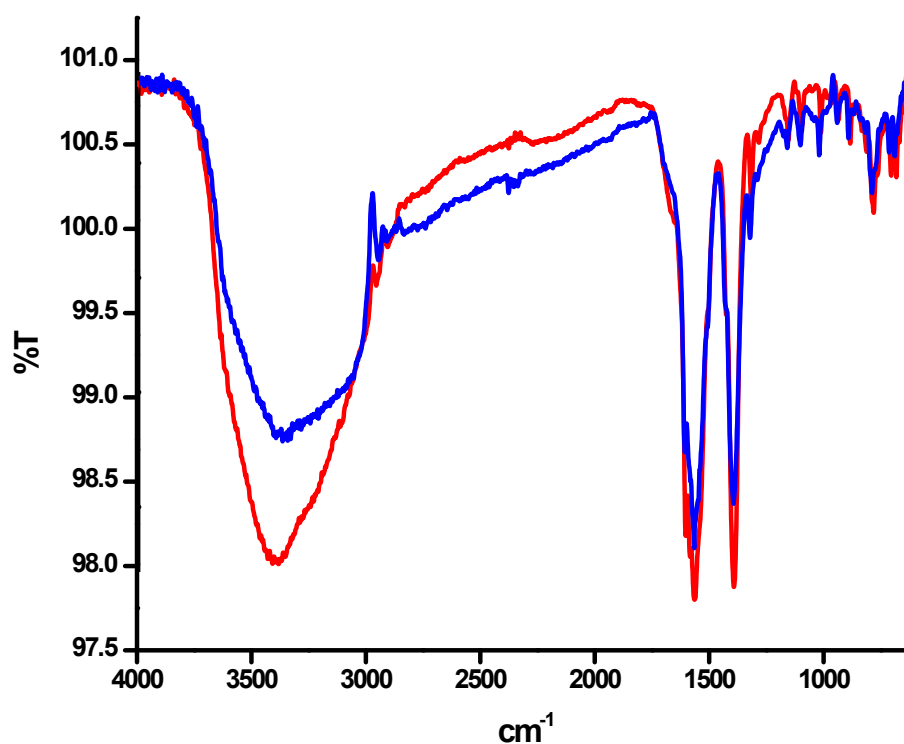


Figure S12. FTIR spectra of as synthesized **1-Ni** (red trace) and after C-S cross coupling reaction between 4-iodotoluene and thiophenol (blue trace).

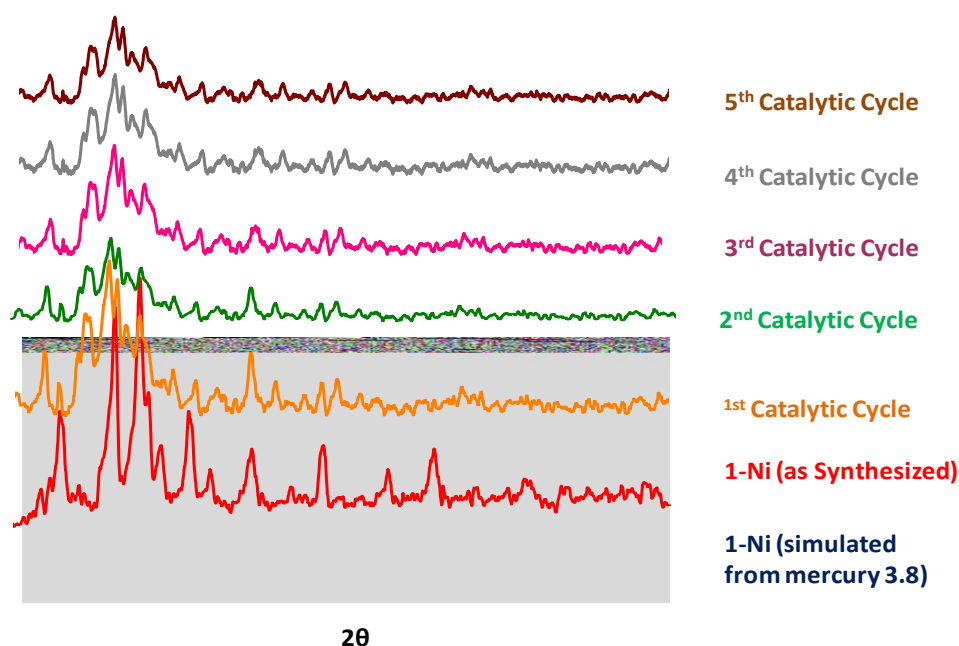


Figure S13. X-ray Powder Diffraction (XRPD) pattern for 1-Ni simulated from mercury 3.8 (blue trace), as synthesized **1-Ni** (red Trace) and after 5 cycles of C-S cross coupling reaction between 4-iodotoluene and thiophenol.

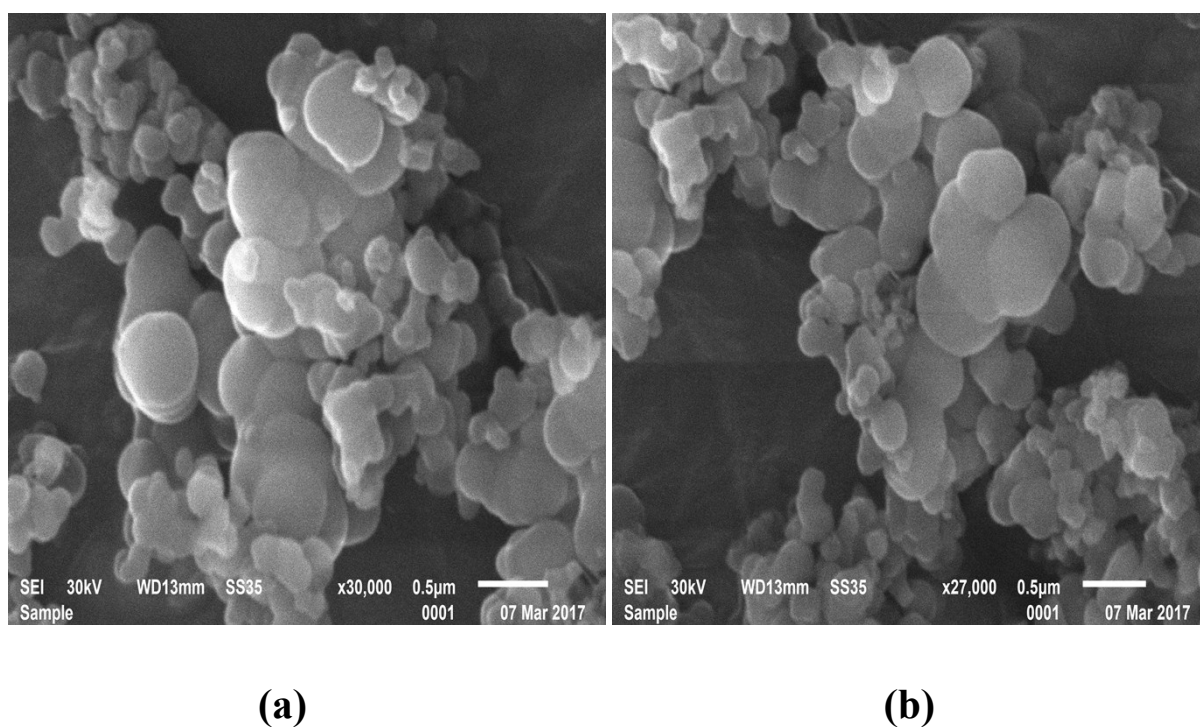


Figure S14. SEM images of as synthesized **1-Ni** (a) before and (b) after C-S cross coupling reaction between 4-iodotoluene and thiophenol.

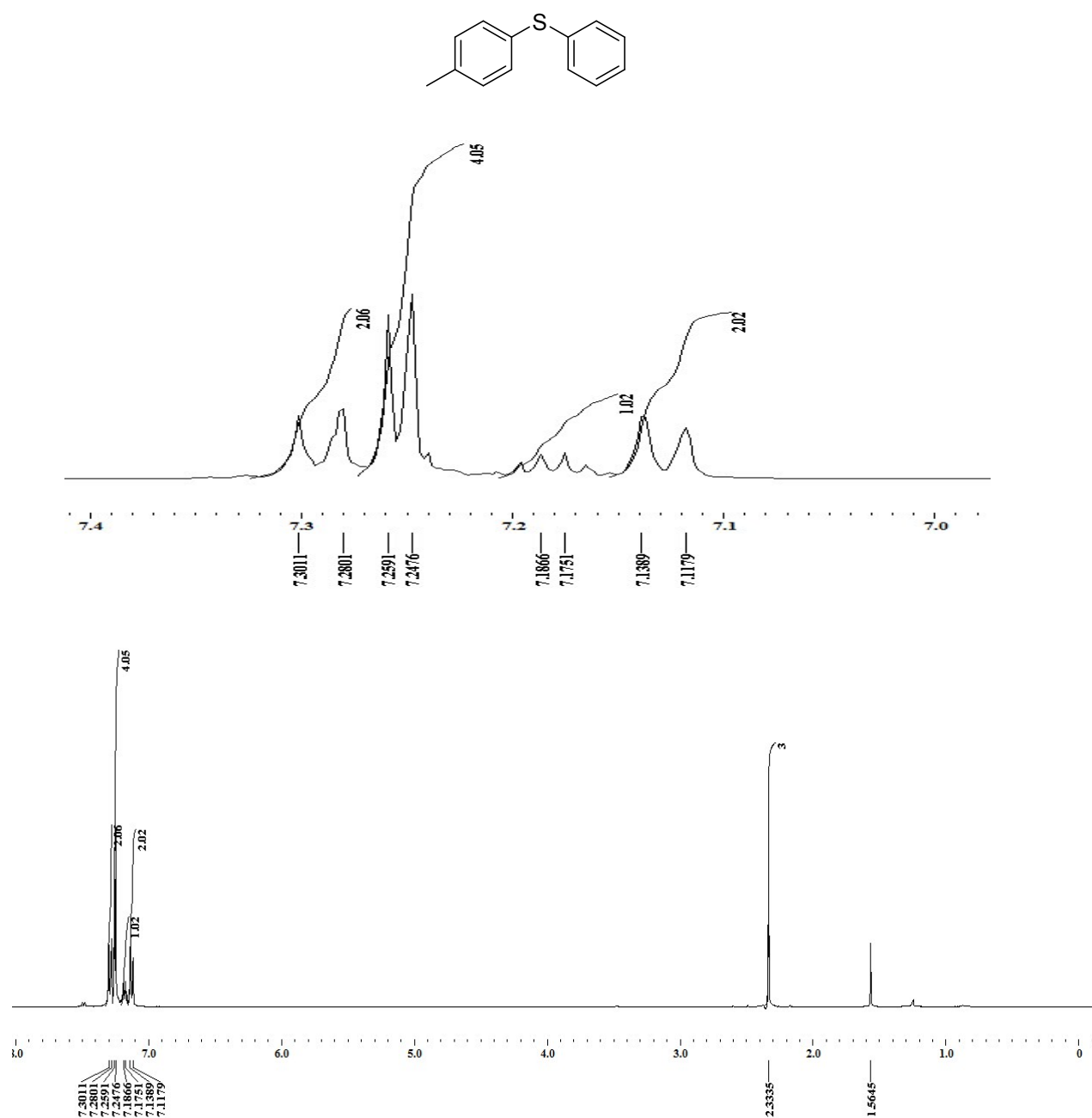


Figure S15. ^1H NMR spectrum of phenyl(4-tolyl)sulfane in CDCl_3 .

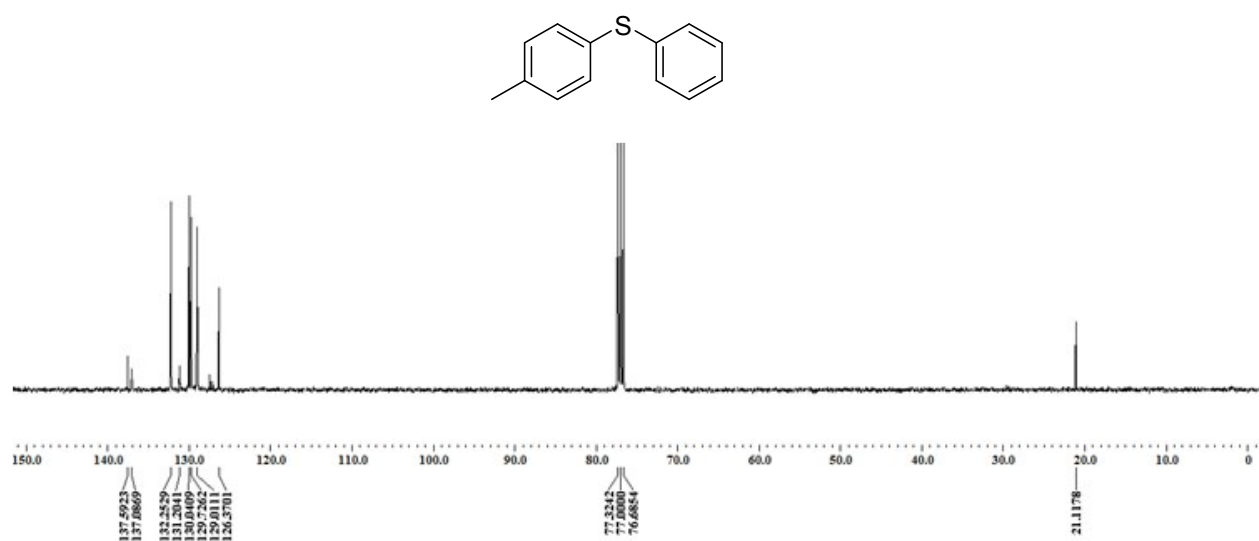


Figure S16. ^{13}C NMR spectrum of phenyl(4-tolyl)sulfane in CDCl_3 .

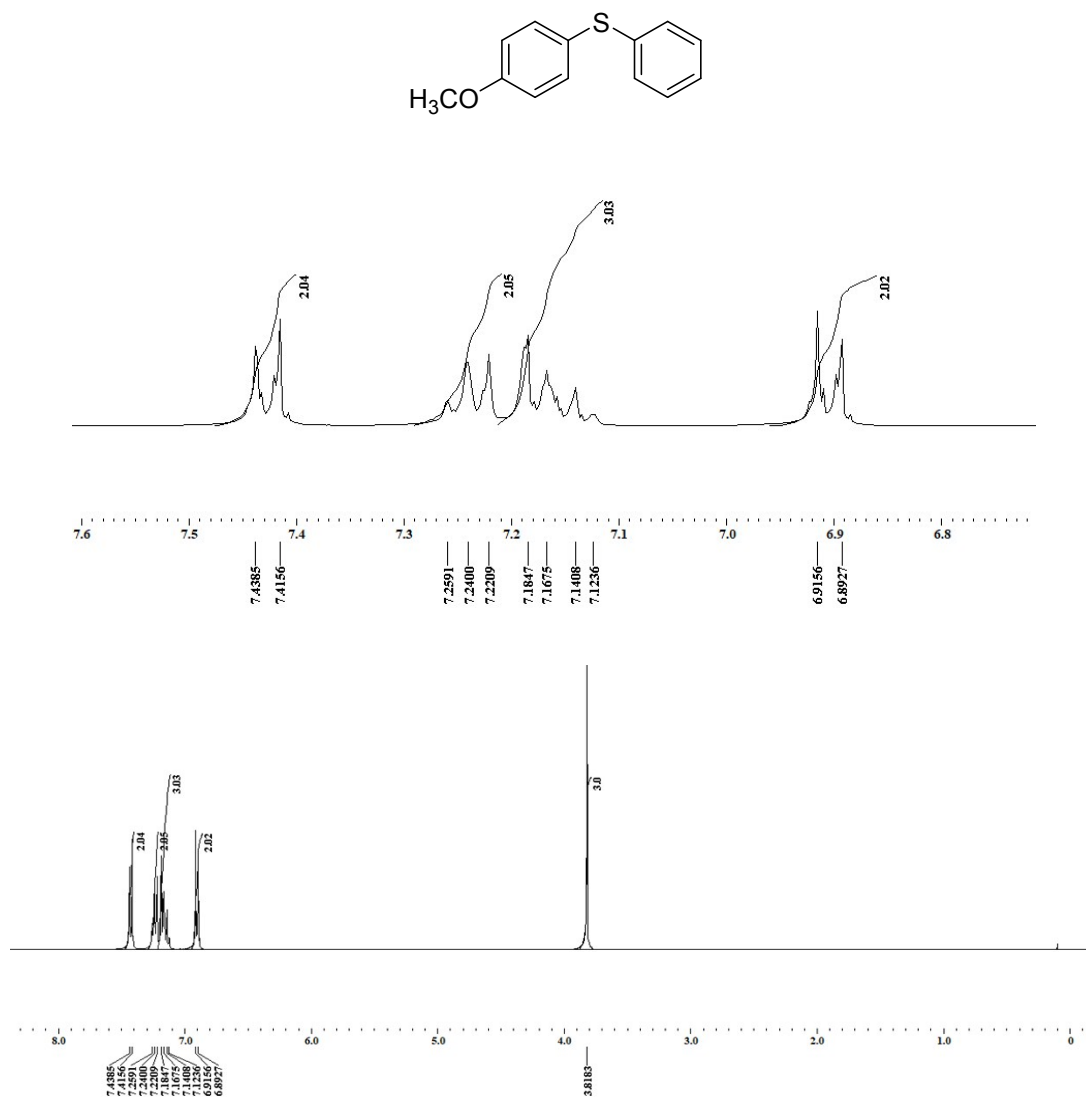


Figure S17. ¹H NMR spectrum of (4-methoxyphenyl)(phenyl)sulfane in CDCl₃.

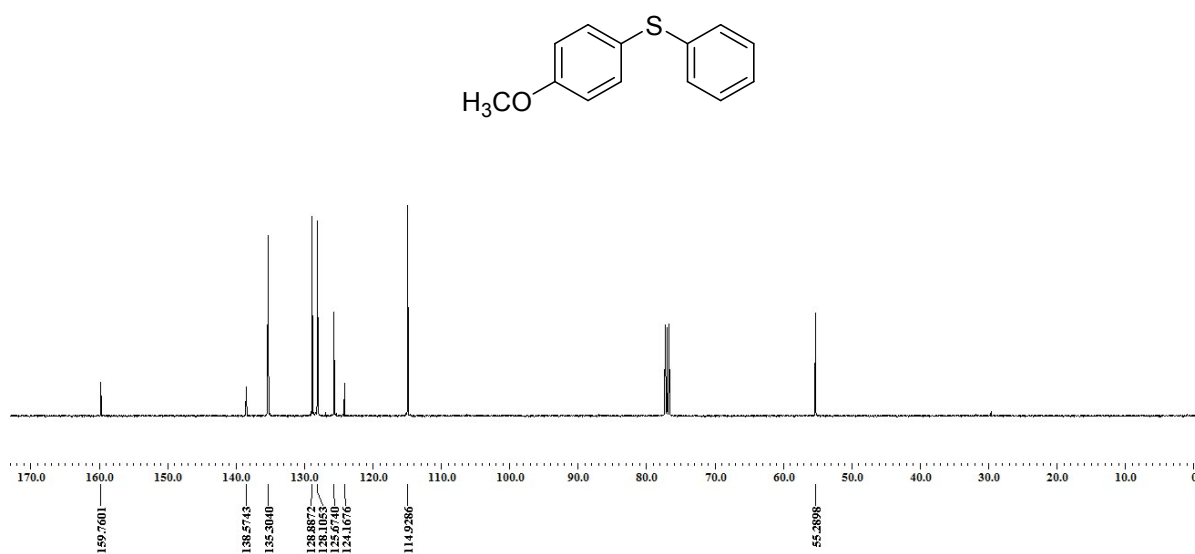


Figure S18. ¹³C NMR spectrum of (4-methoxyphenyl)(phenyl)sulfane in CDCl₃.

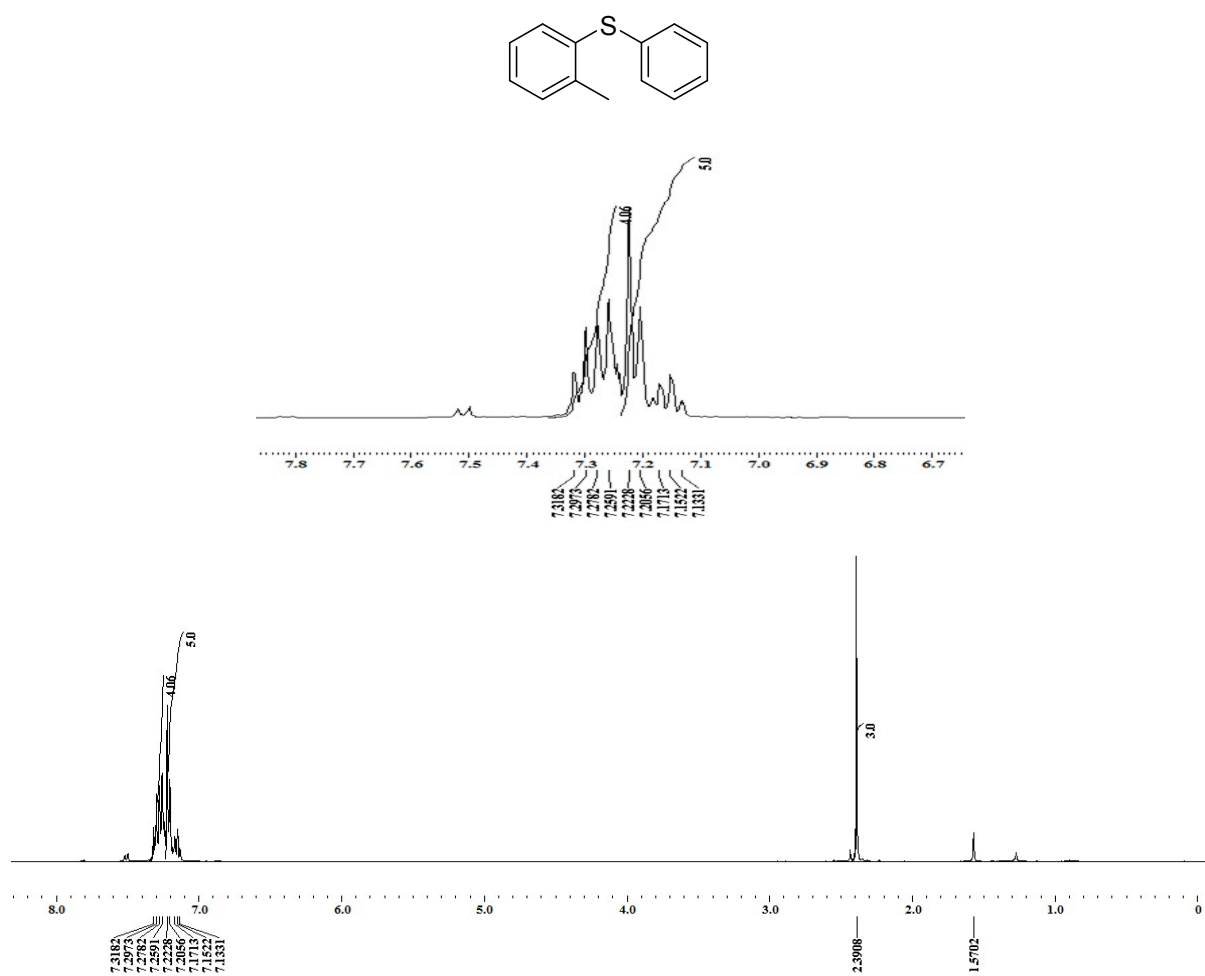


Figure S19. ¹H NMR spectrum of phenyl(2-tolyl)sulfane in CDCl₃.

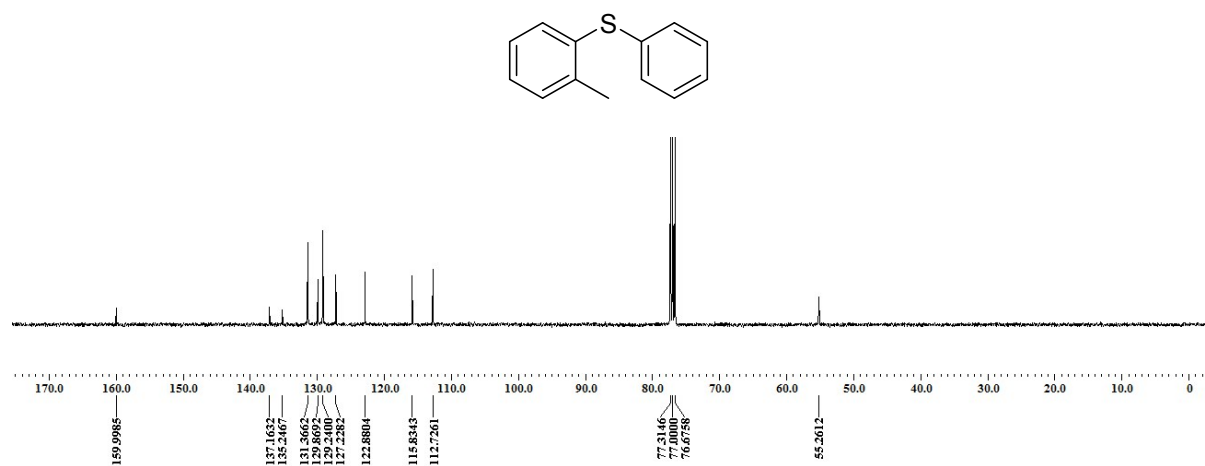


Figure S20. ¹³C NMR spectrum of phenyl(2-tolyl)sulfane in CDCl₃.

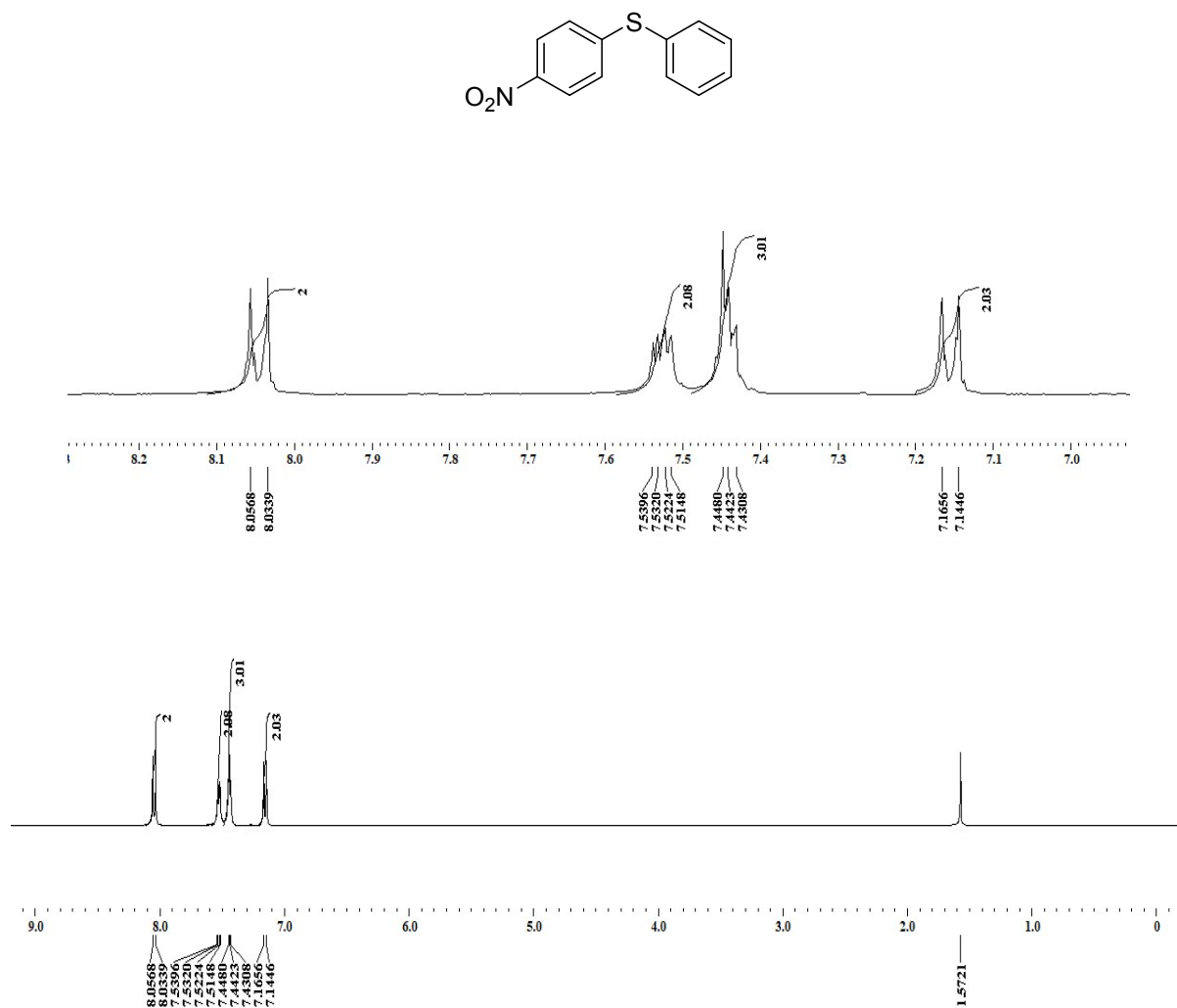


Figure S21. ¹H NMR spectrum of (4-nitrophenyl)(phenyl)sulfane in CDCl₃.

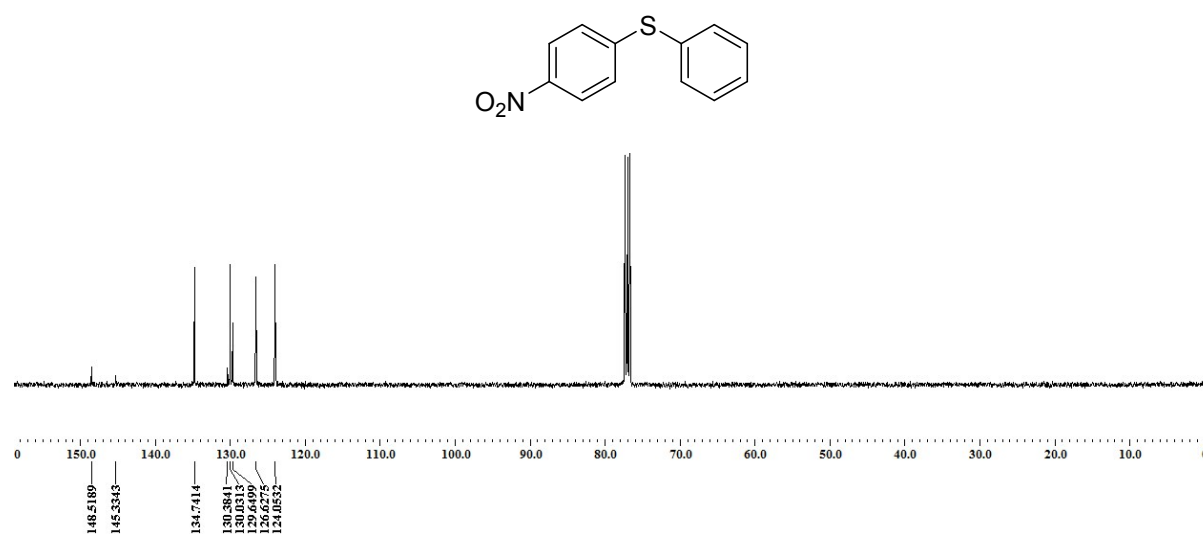


Figure S22. ¹³C NMR spectrum of (4-nitrophenyl)(phenyl)sulfane in CDCl₃.

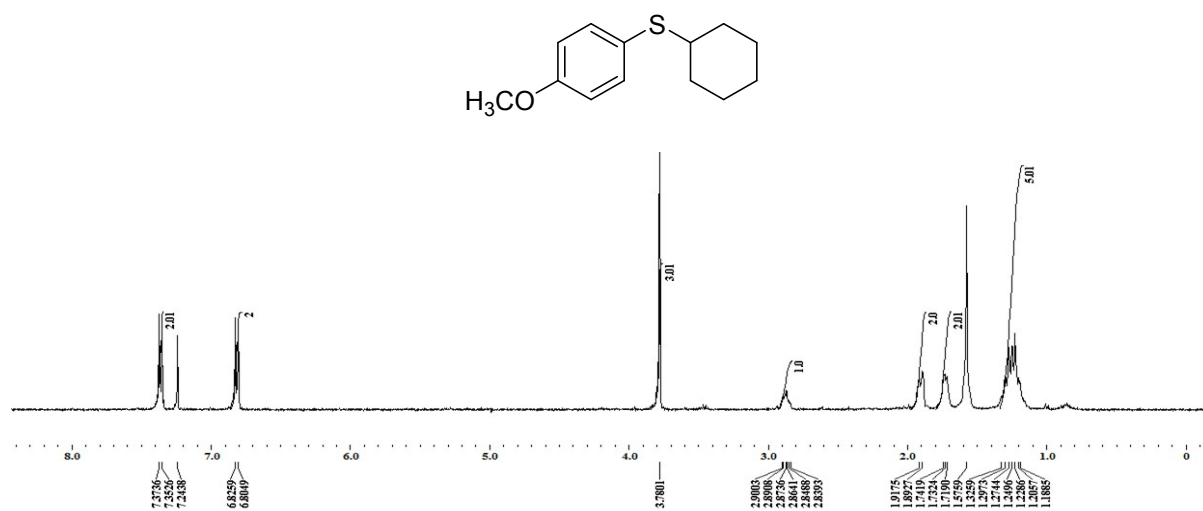


Figure S23. ¹H NMR spectrum of cyclohexyl(4-methoxyphenyl)sulfane in CDCl₃.

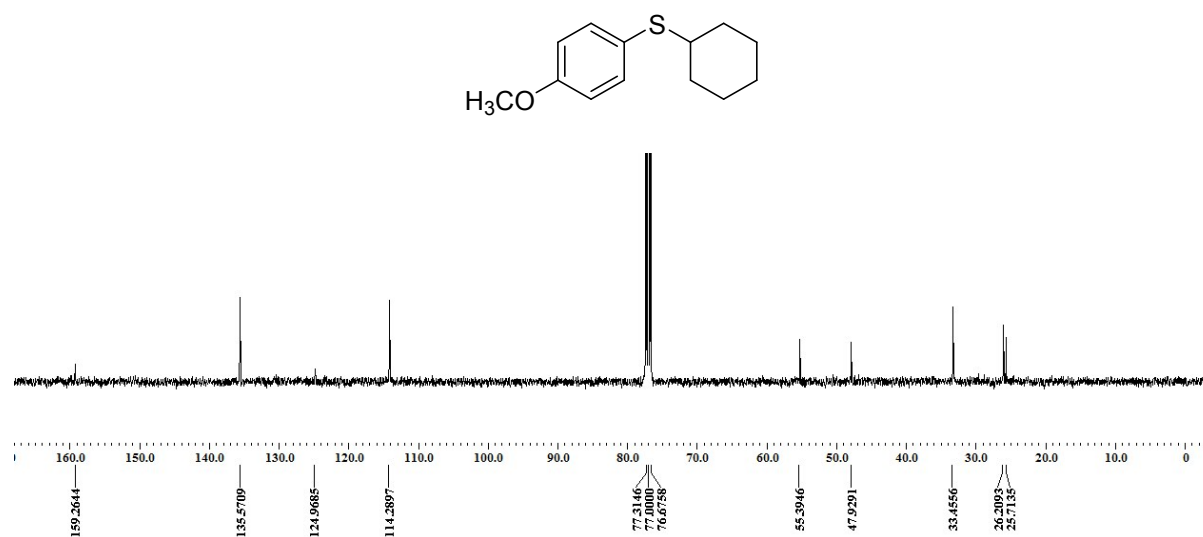


Figure S24. ¹³C NMR spectrum of cyclohexyl(4-methoxyphenyl)sulfane in CDCl₃.

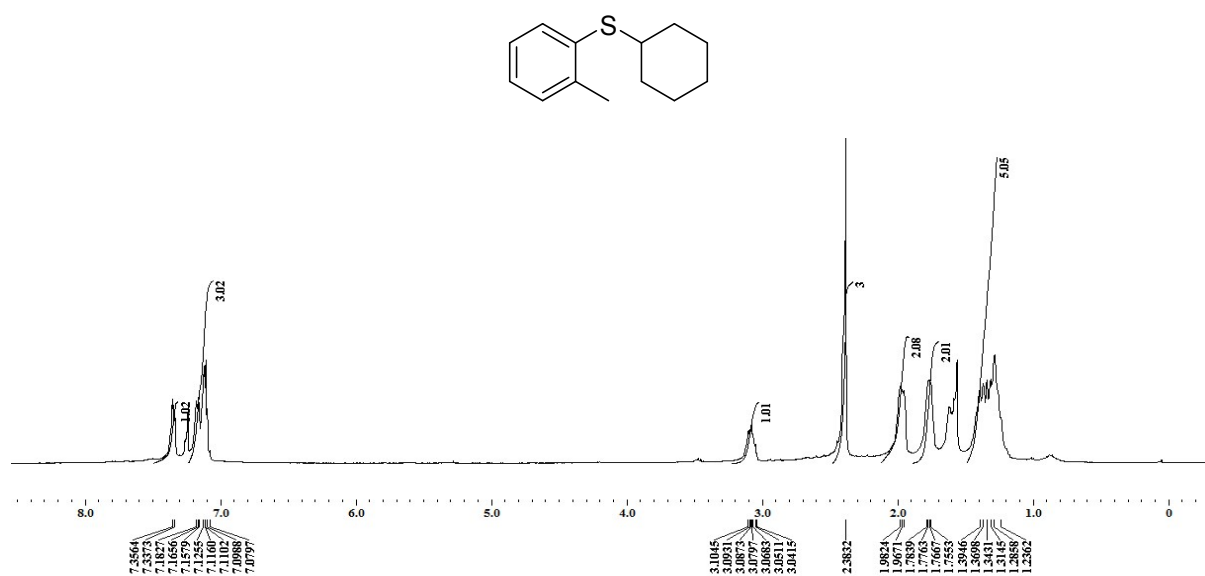


Figure S25. ¹H NMR spectrum of cyclohexyl(2-tolyl)sulfane in CDCl₃.

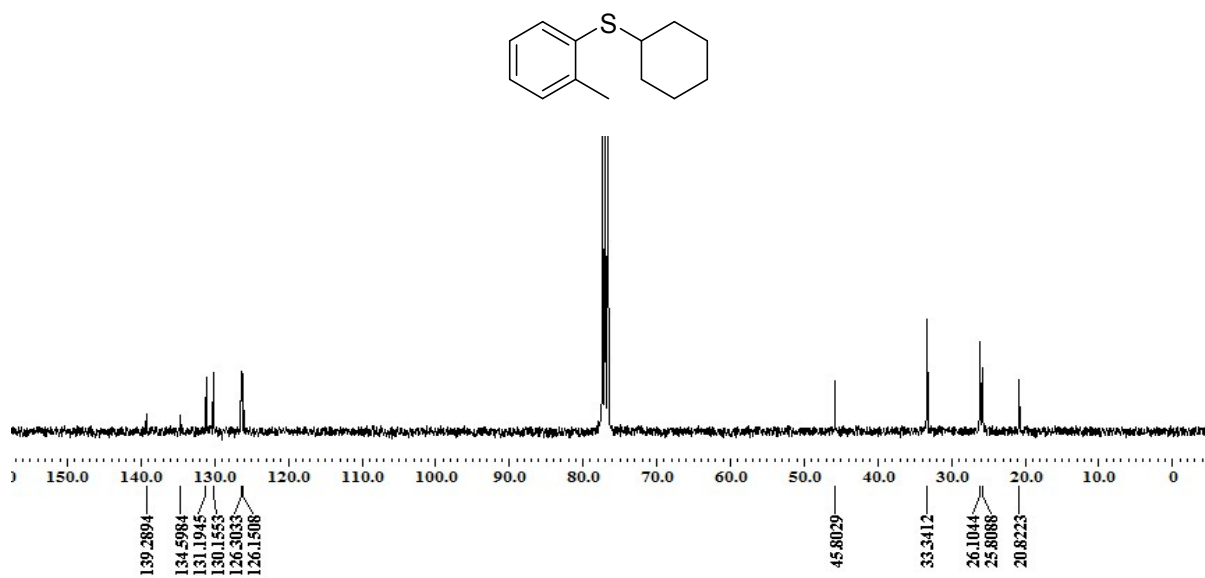


Figure S26. ¹³C NMR spectrum of cyclohexyl(2-tolyl)sulfane in CDCl₃.

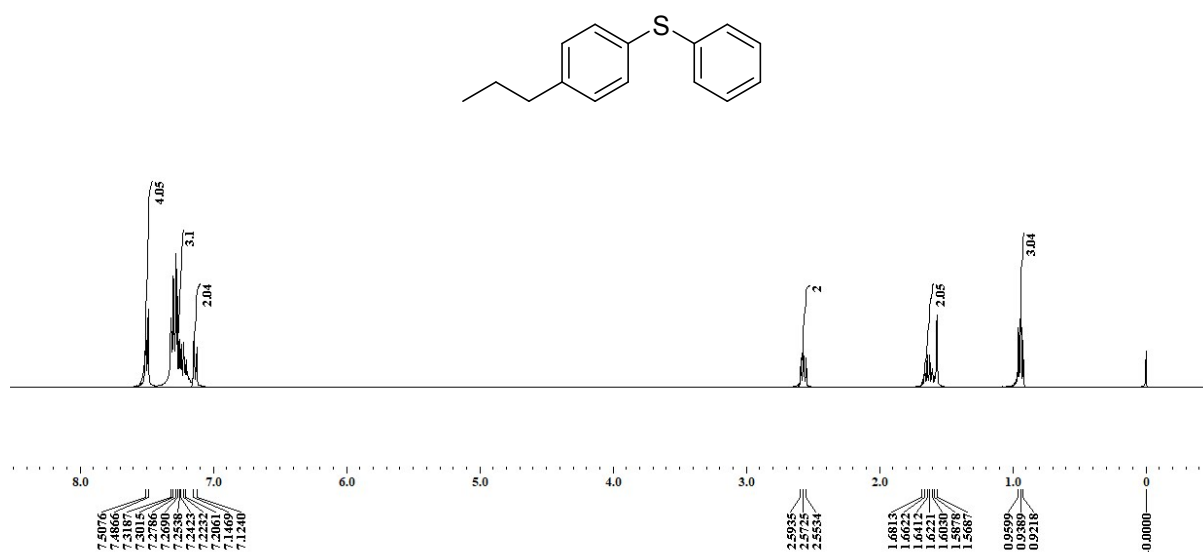


Figure S27. ¹H NMR spectrum of phenyl(4-propylphenyl)sulfane in CDCl₃.

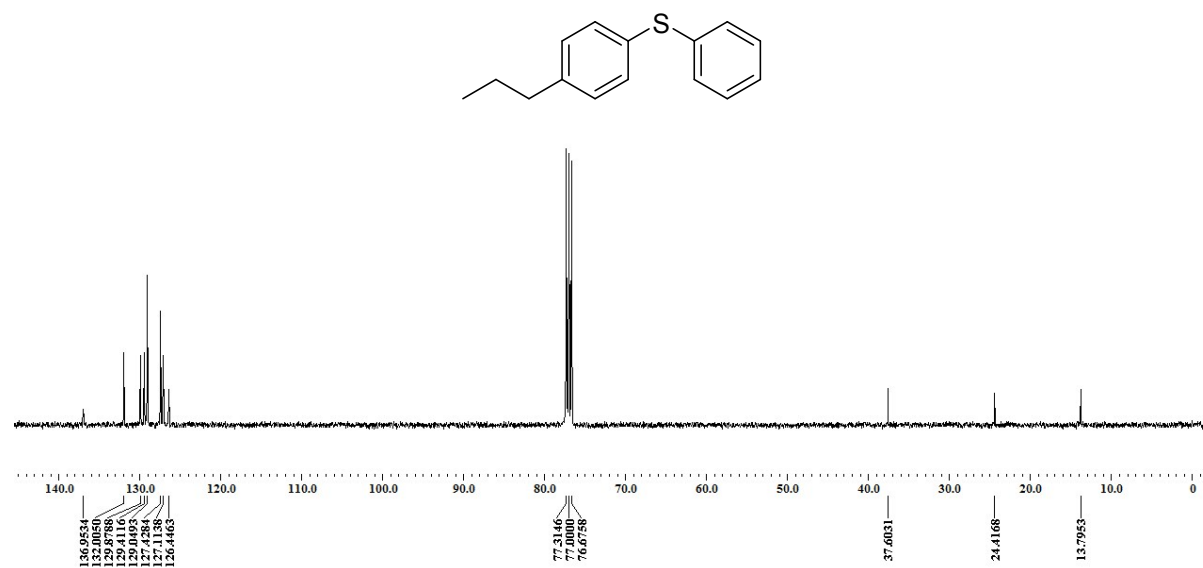


Figure S28. ¹³C NMR spectrum of phenyl(4-propylphenyl)sulfane in CDCl₃.

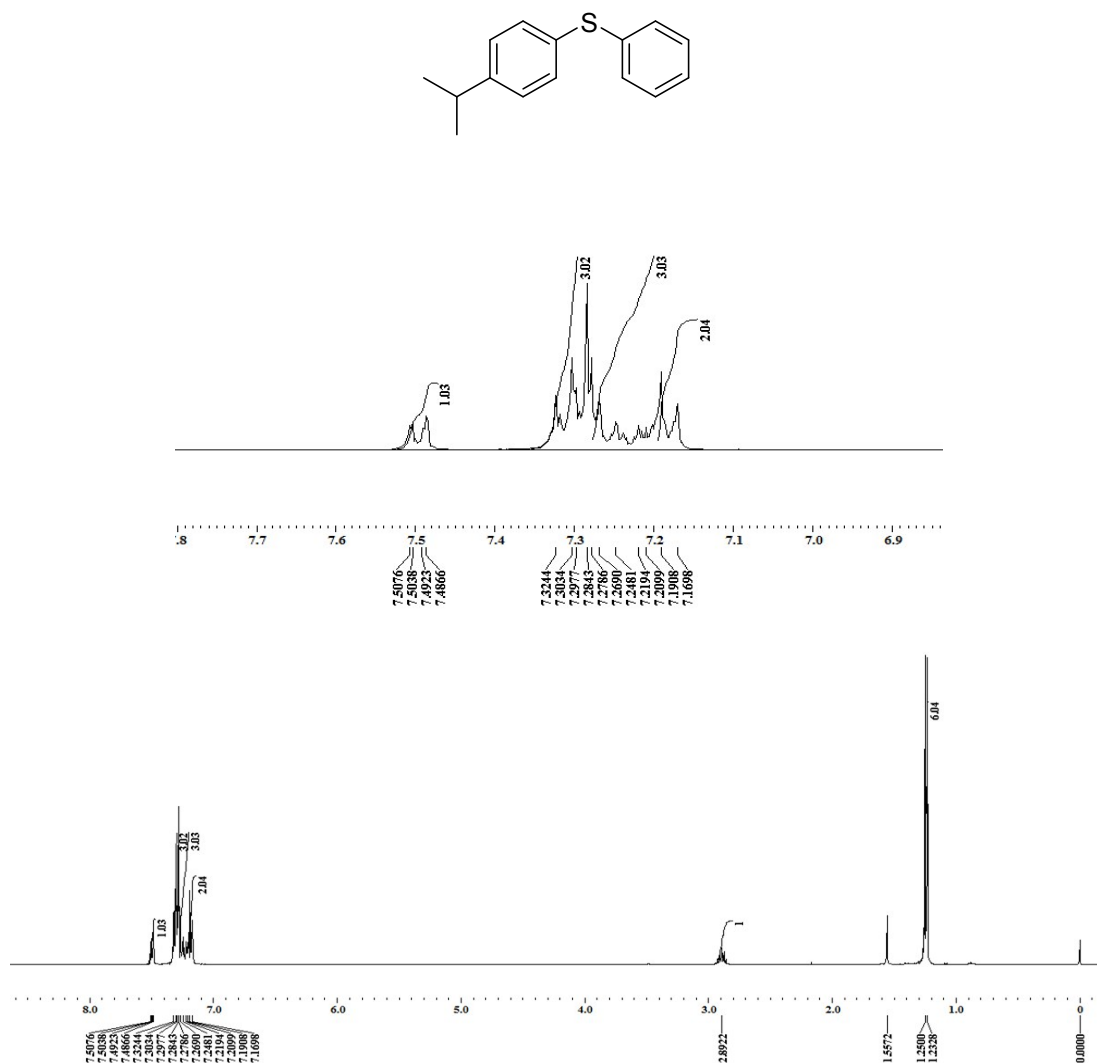


Figure S29. ¹H NMR spectrum of (4-isopropylphenyl)(phenyl)sulfane in CDCl₃.

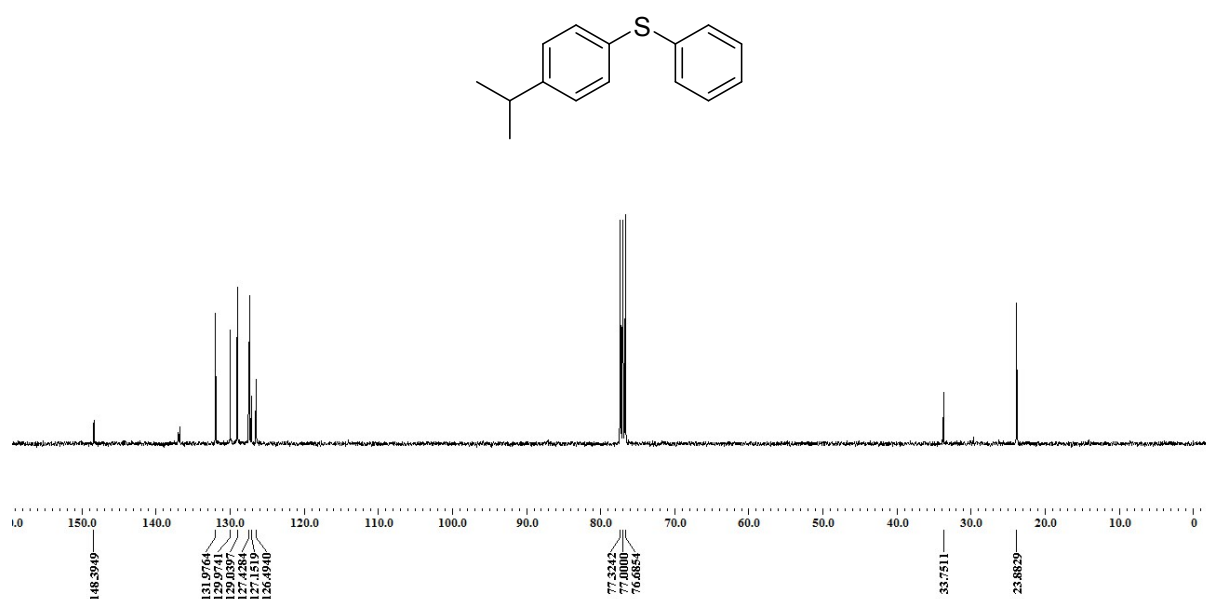


Figure S30. ¹³C NMR spectrum of (4-isopropylphenyl)(phenyl)sulfane in CDCl₃.

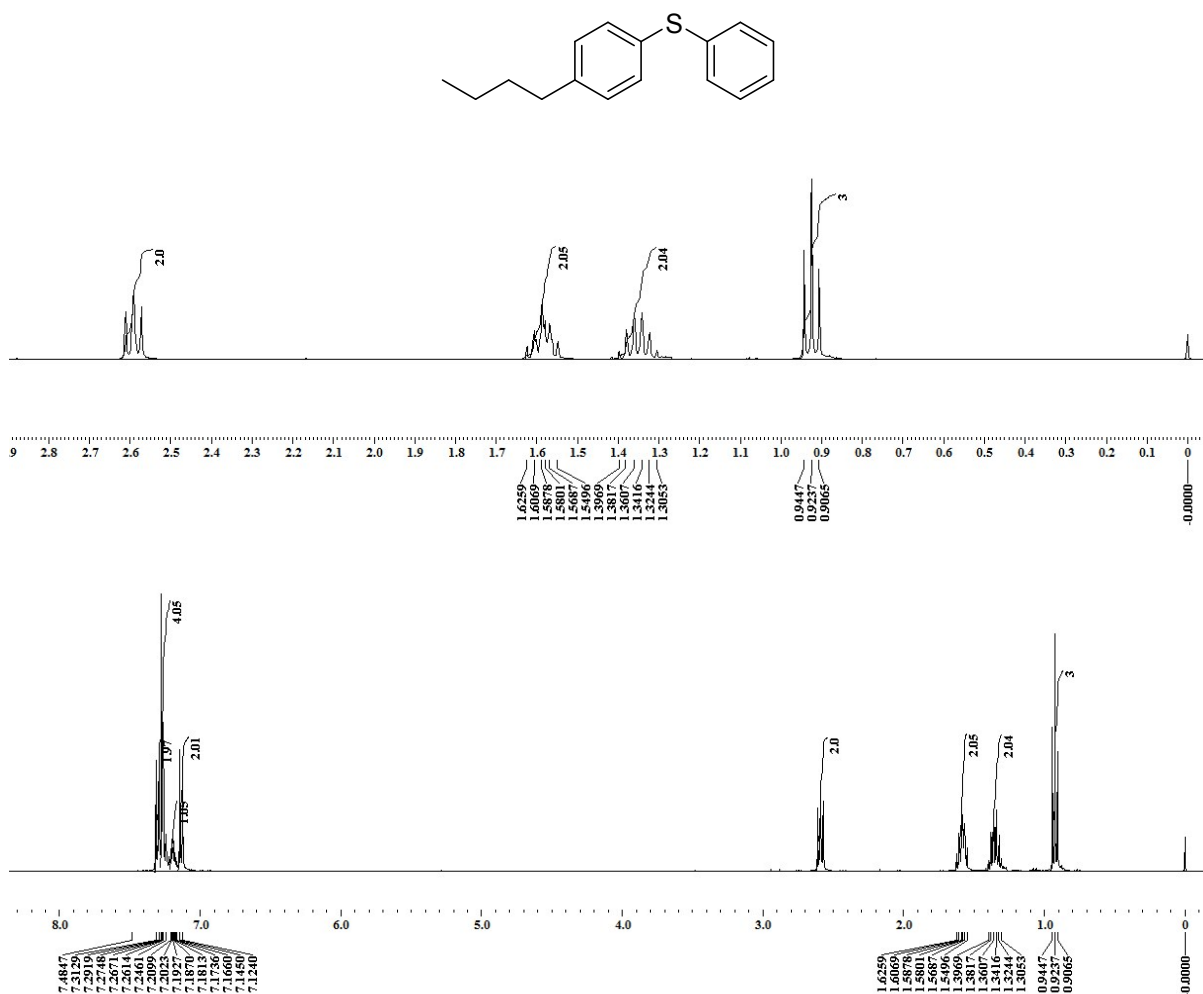


Figure S31. ¹H NMR spectrum of (4-butylphenyl)(phenyl)sulfane in CDCl₃.

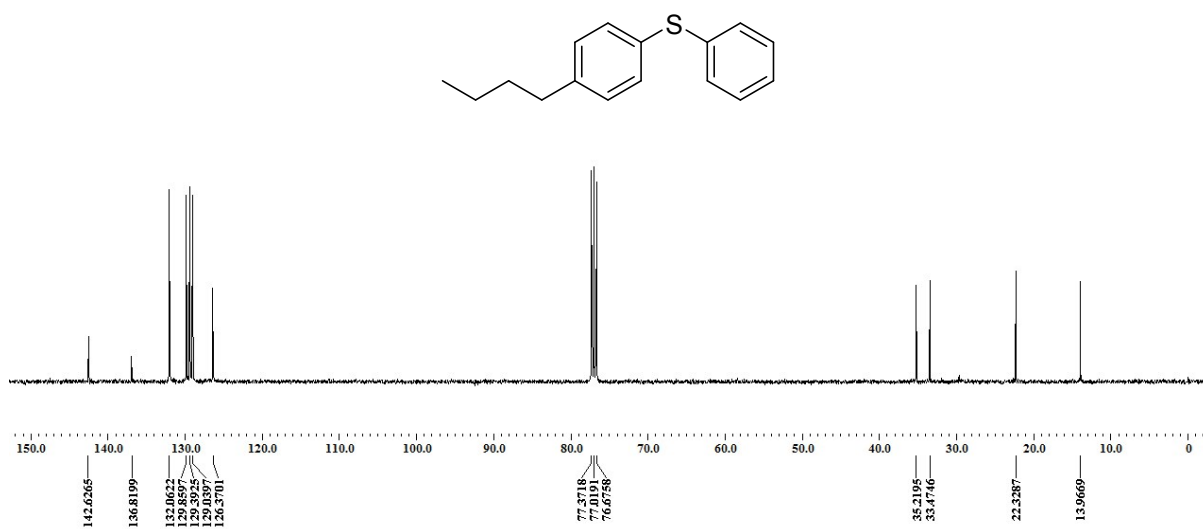


Figure S32. ¹³C NMR spectrum of (4-butylphenyl)(phenyl)sulfane in CDCl₃.

Table S1. Crystallographic data collection and structural refinement parameters for **1-Ni**.

1	1-Ni
Empirical formula	C ₄₂ H ₄₆ CoN ₆ Ni ₂ O ₂₄
Formula weight	1195.20
T(K)	173(2)
Crystal system	Hexagonal
Space group	P 6 ₂ 2 2
<i>a</i> (Å)	27.857(2)
<i>b</i> (Å)	27.857(2)
<i>c</i> (Å)	10.0365(7)
$\alpha(^{\circ})$	90
$\beta(^{\circ})$	90
$\gamma(^{\circ})$	120
<i>V</i> (Å) ³	6745.2(11)
<i>Z</i>	3
Crystal Size (mm ³)	0.2 x 0.15 x 0.12
<i>d</i> [g cm ⁻³]	0.883
μ [mm ⁻¹]	0.648
<i>F</i> (000)	1845.0
<i>R</i> (int.)	0.1768
Final <i>R</i> indices ^a	R1 = 0.0543
[<i>I</i> > 2 σ (<i>I</i>)]	wR2 = 0.0995
<i>R</i> indices	R1 = 0.0712
All data	wR2 = 0.1054
GOF on <i>F</i> ²	0.944
CCDC No.	

$$R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|. \text{ } ^b\text{wR}_2 = \{\Sigma[w(|F_o|^2 - |F_c|^2)^2]/\Sigma[wF_o^4]\}^{1/2}$$