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Supporting Information

The recyclable heterogeneous nanocatalyst of copper-grafted natural asphalt sulfonate (NAS@Cu): Characterization, synthesis and application in the Suzuki-Miyaura coupling reaction

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Supporting information includes Elemental mapping for NAS@Cu, SEM images of NA, Na-NAS, NAS@Cu and NAS@Cu after recovery, FT-IR spectra of natural asphalt and natural asphalt sulfonate, TEM image of NAS@Cu , selected original spectrums and Selected spectral data of the synthesized biphenyl derivatives.

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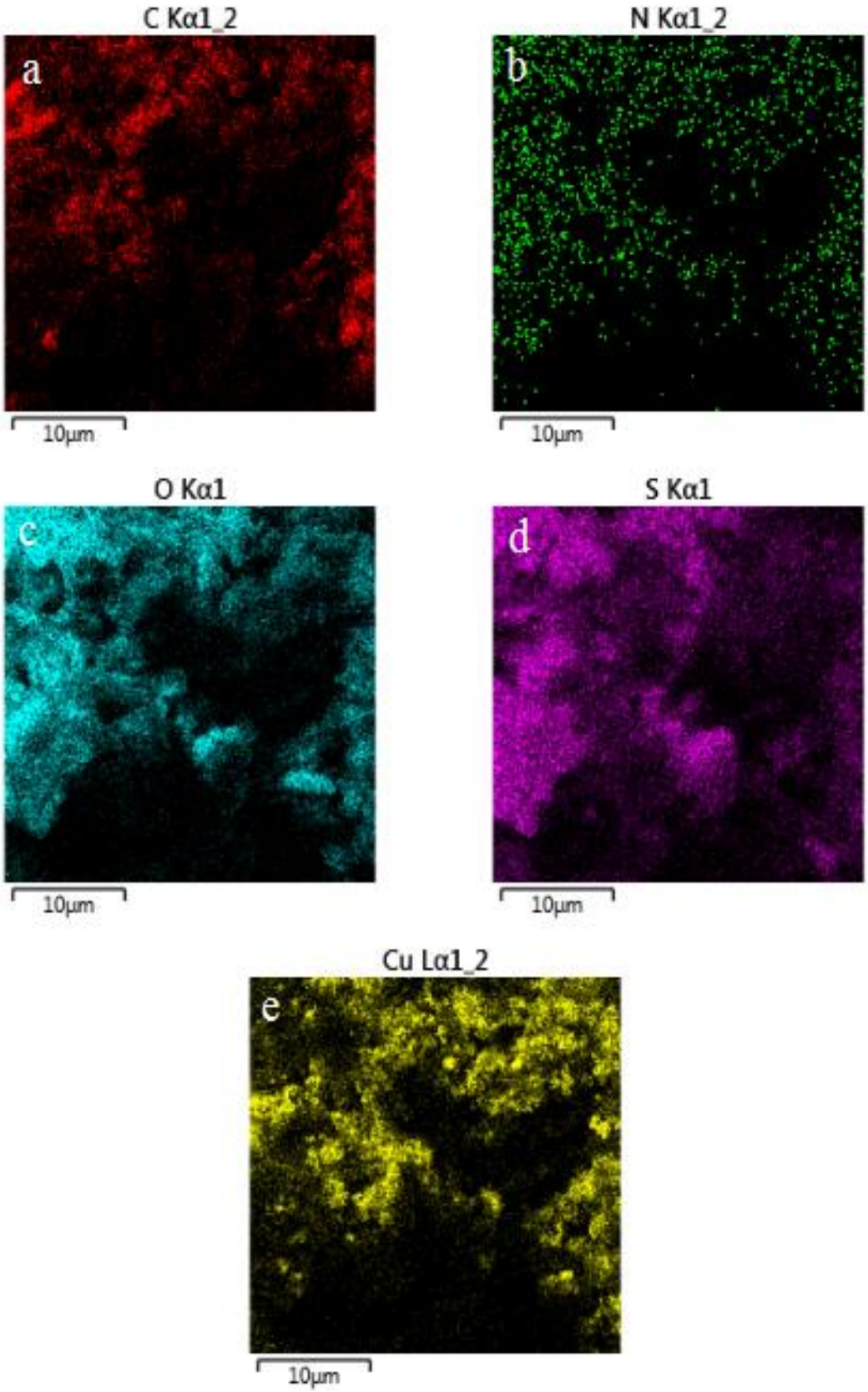
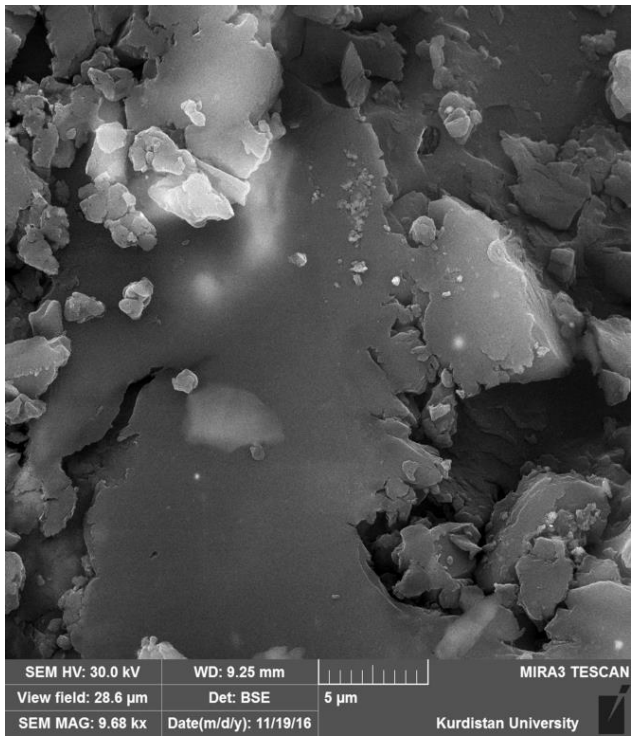


Figure S1. Elemental mapping of C (a), N (b), O (c), S (d) and Cu (e) for NAS@Cu



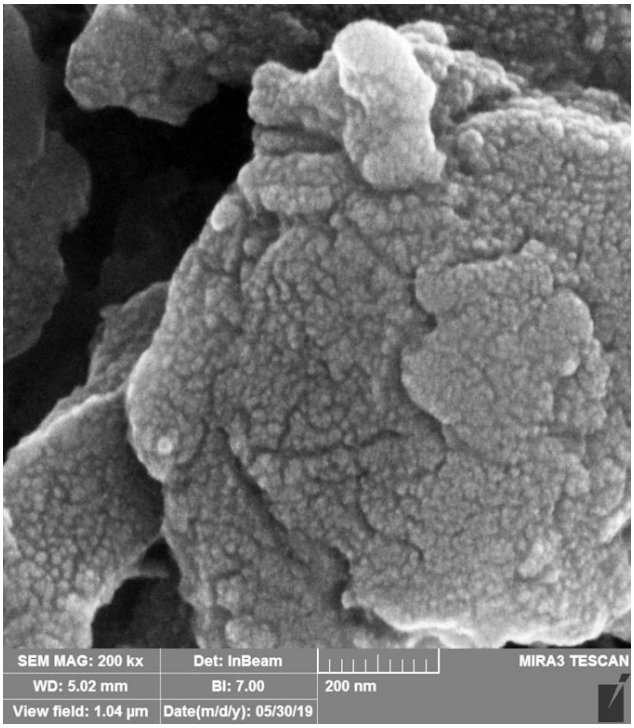


Figure S2. SEM images of NA and Na- NAS

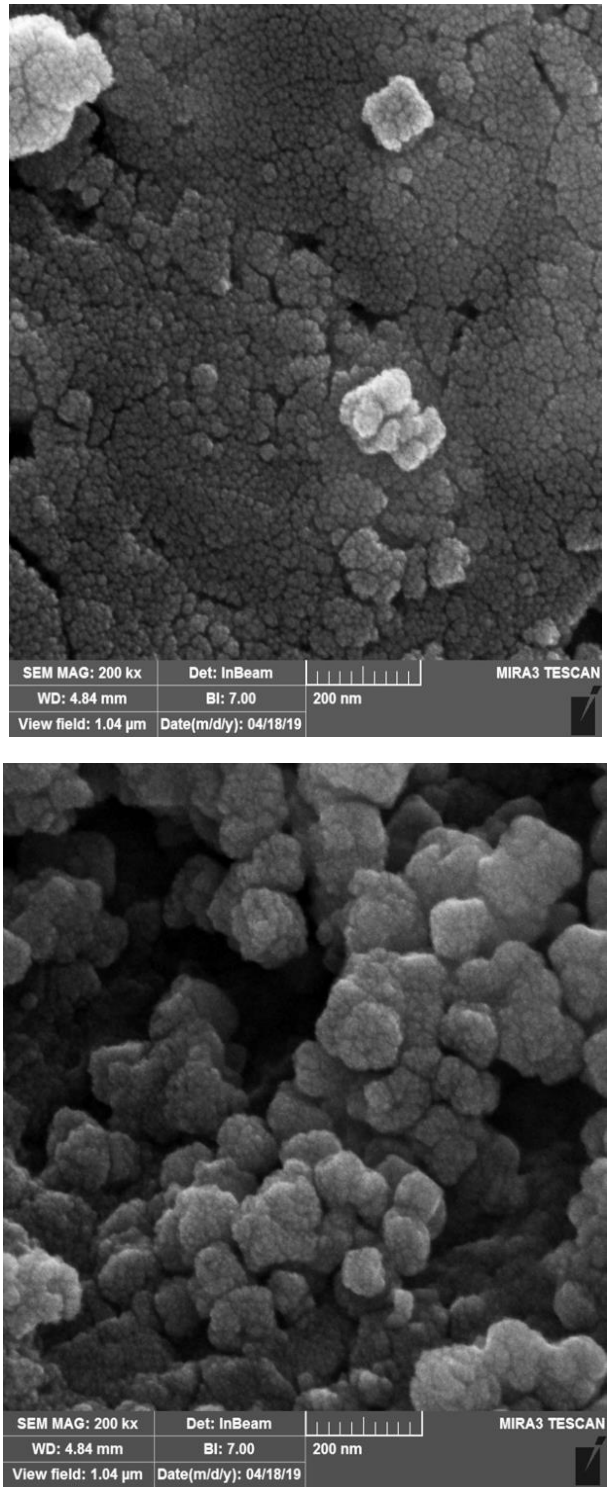


Figure S3. SEM images of NAS@Cu and NAS@Cu after the recovery

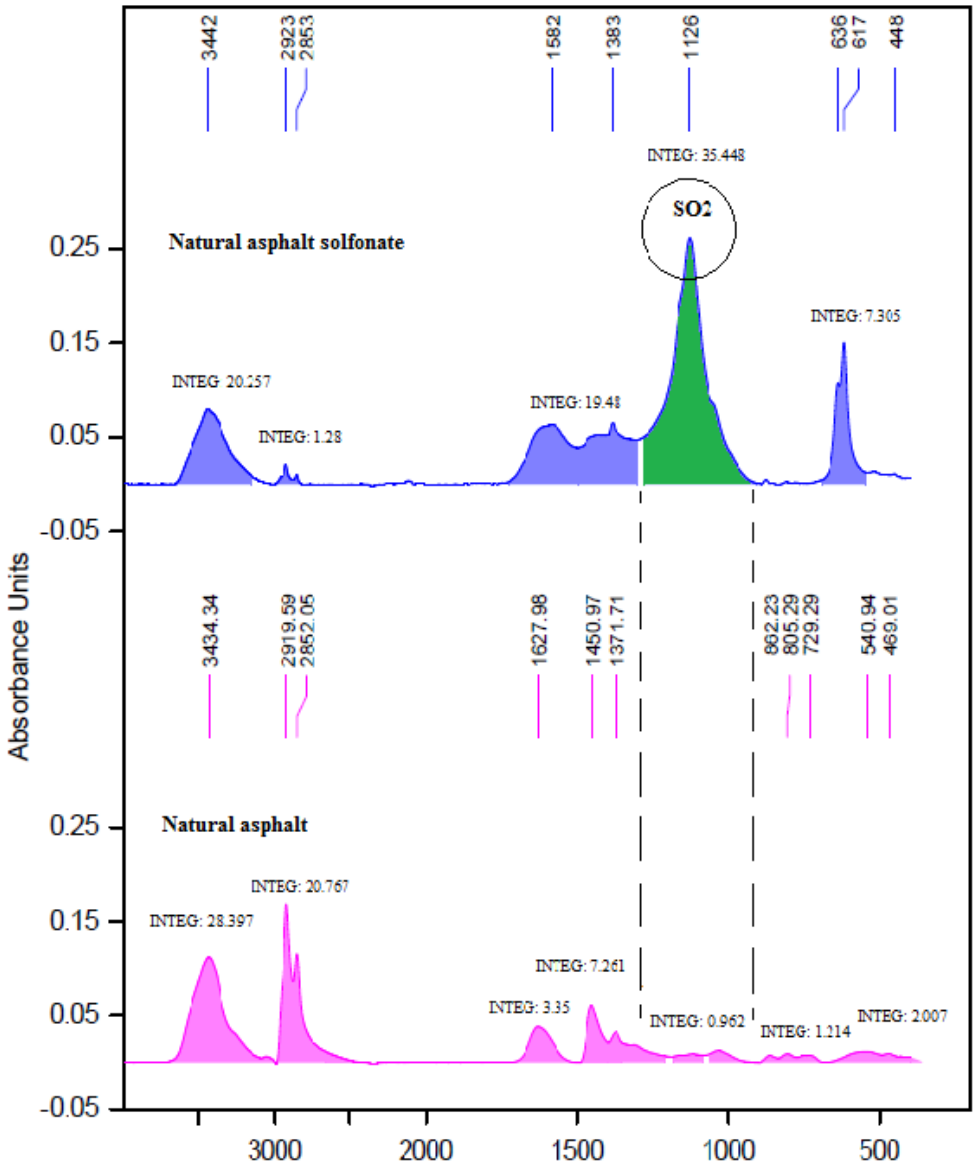


Figure S4. FT-IR spectra of natural asphalt and natural asphalt sulfonate

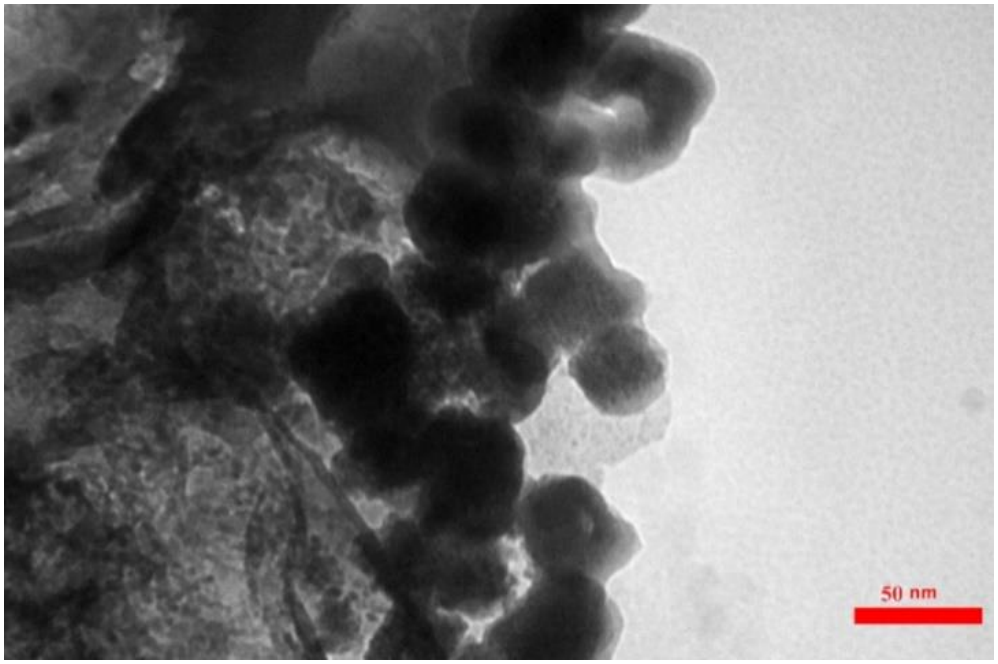


Figure S5.TEM image of NAS@Cu

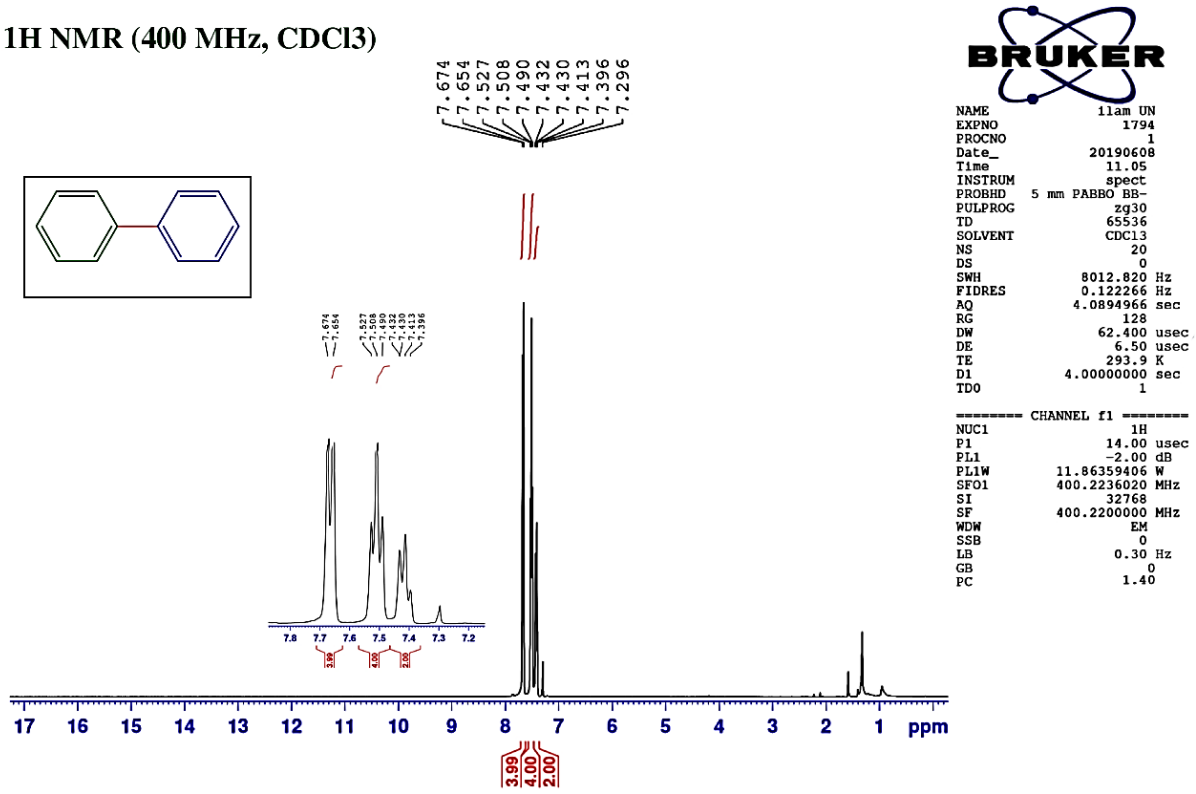


Fig S6. ^{1}H NMR spectrum of 1,1'-Biphenyl

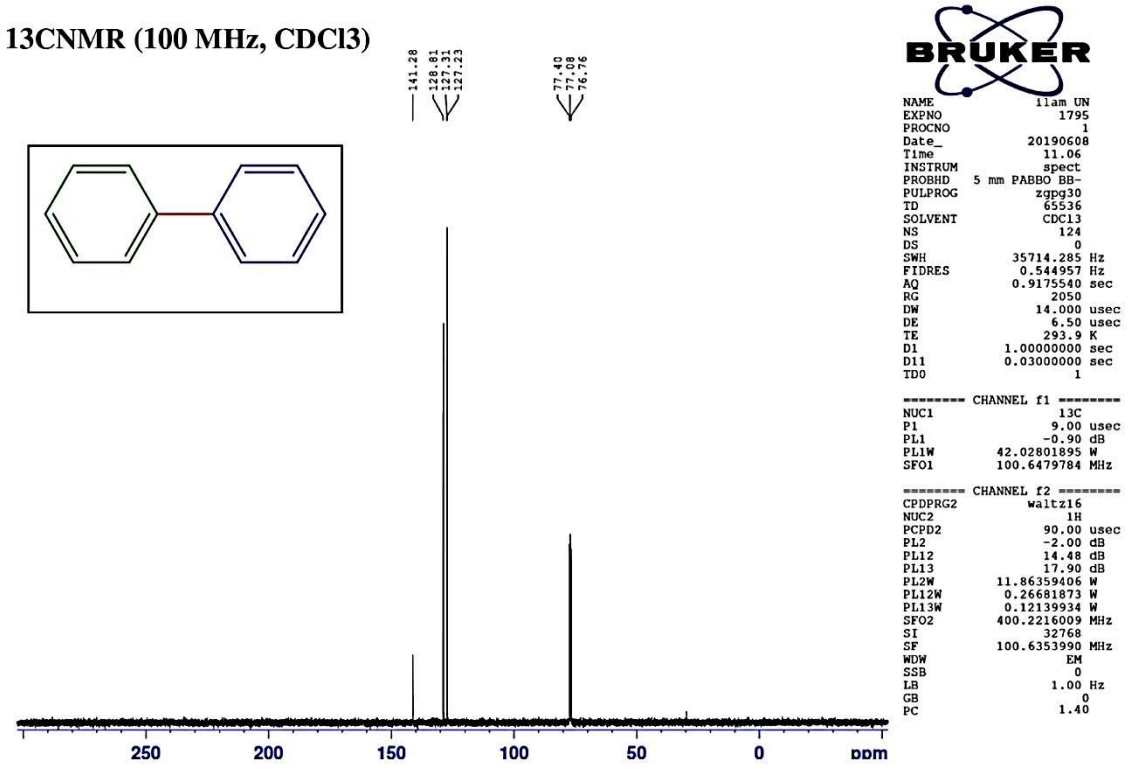


Fig S7. ^{13}C NMR spectrum of 1,1'-Biphenyl

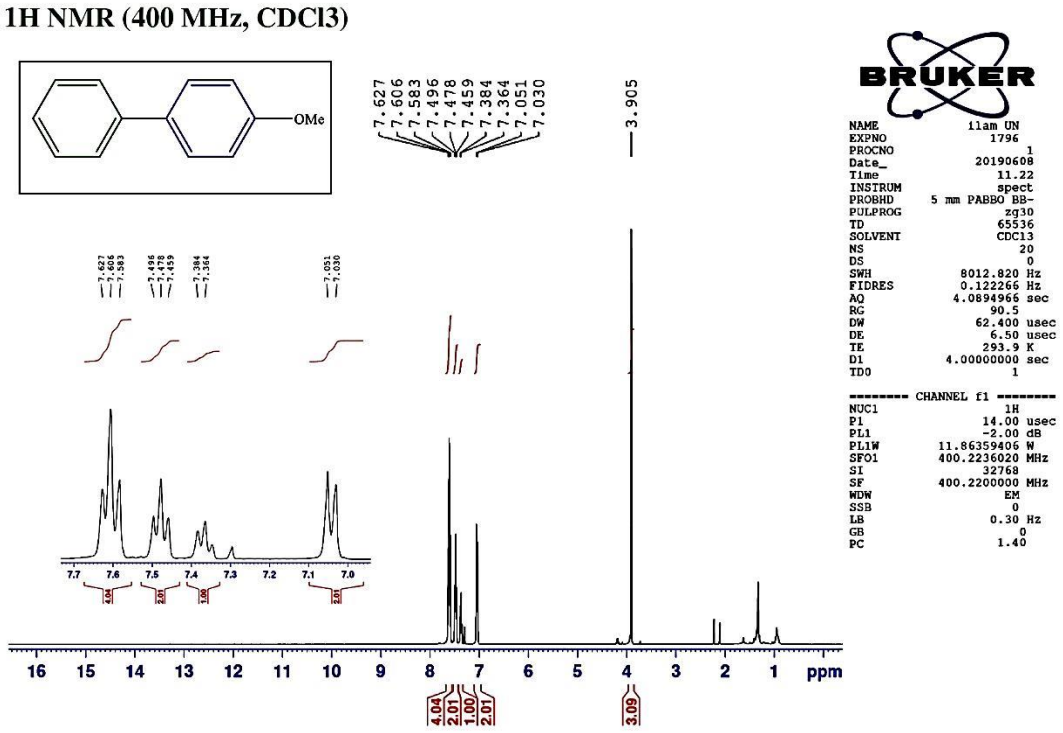


Fig S8. ^{1}H NMR spectrum 4-Methoxy-1,1'-biphenyl

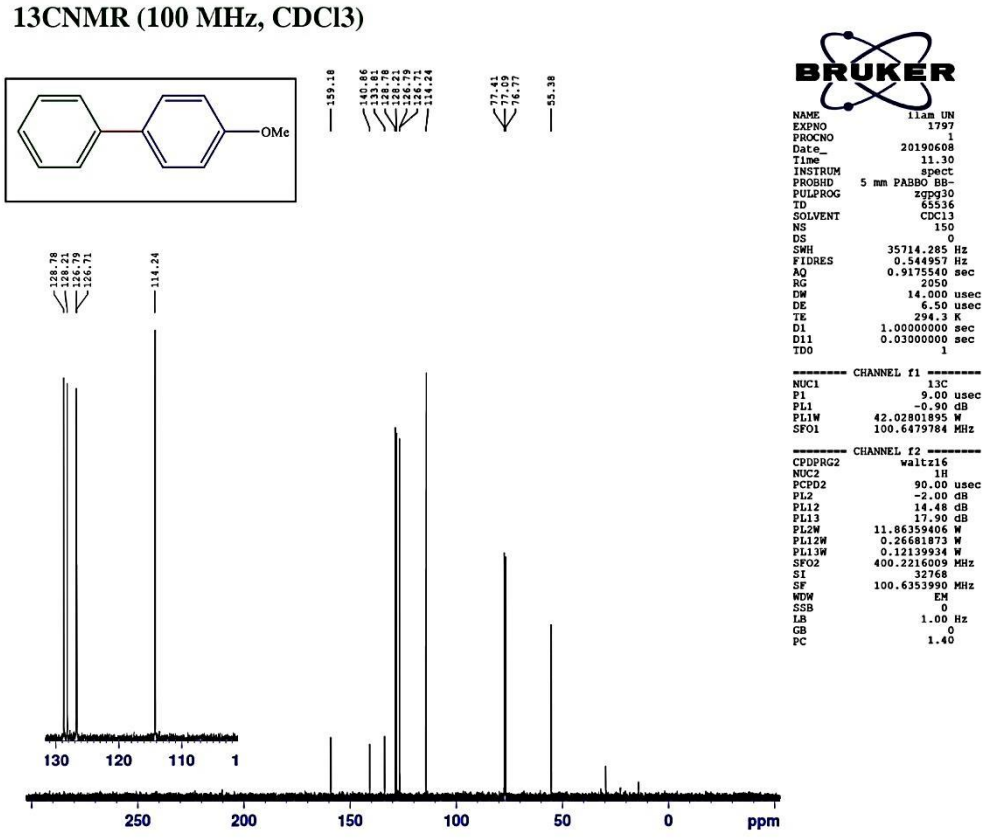


Fig S9. ^{13}C NMR spectrum of 4-Methoxy-1,1'-biphenyl

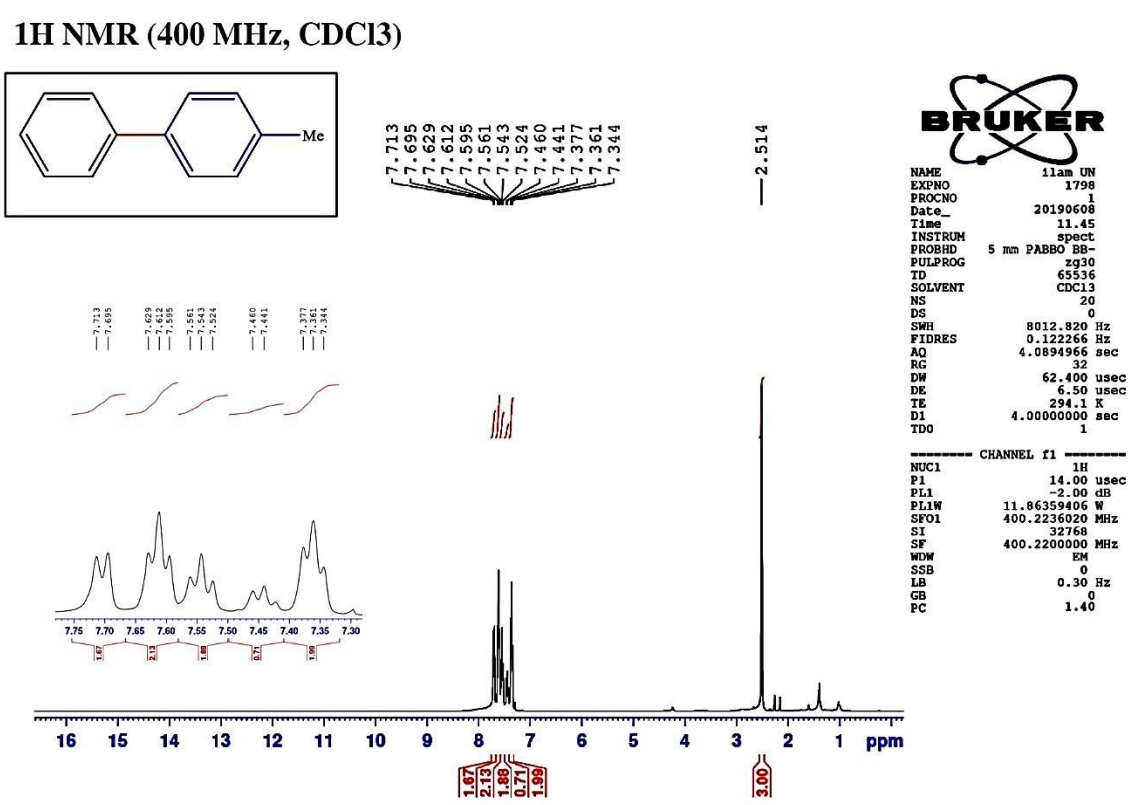


Fig S10. ^{1}H NMR spectrum of 4-Methyl-1,1'-bipheny

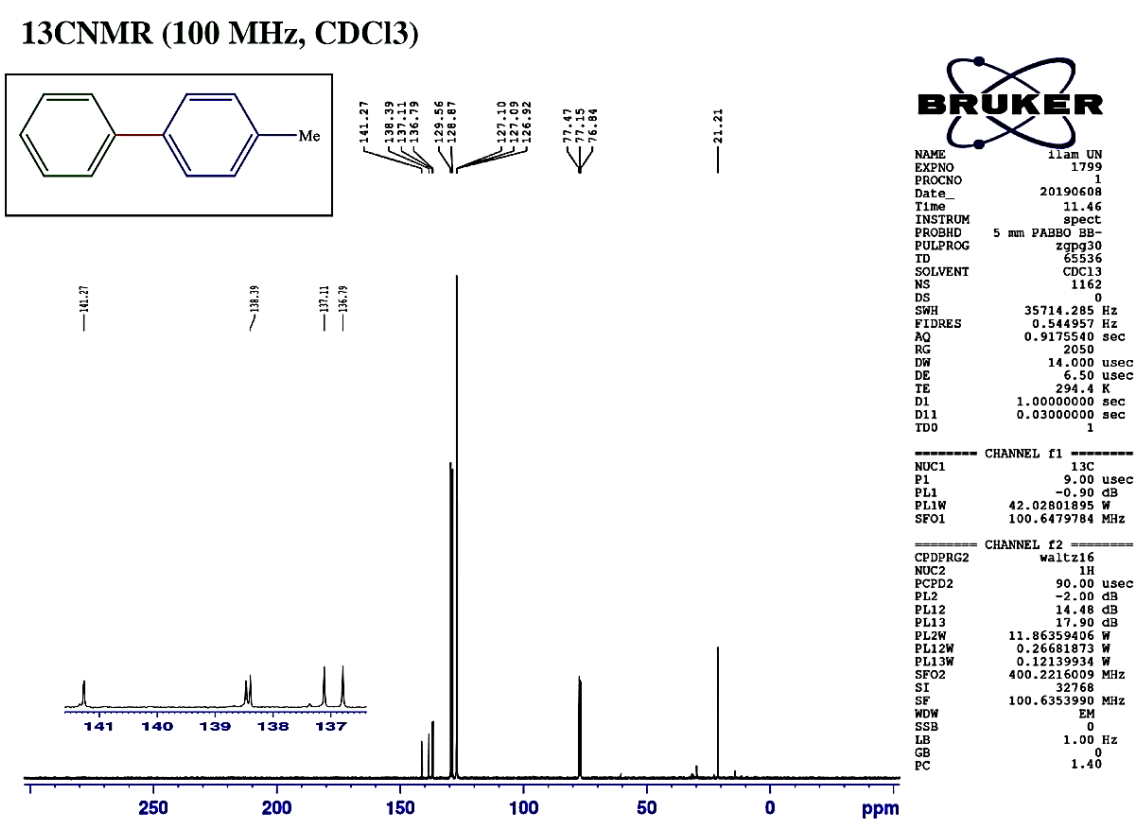


Fig S11. ^{13}C NMR spectrum of 4-Methyl-1,1'-bipheny

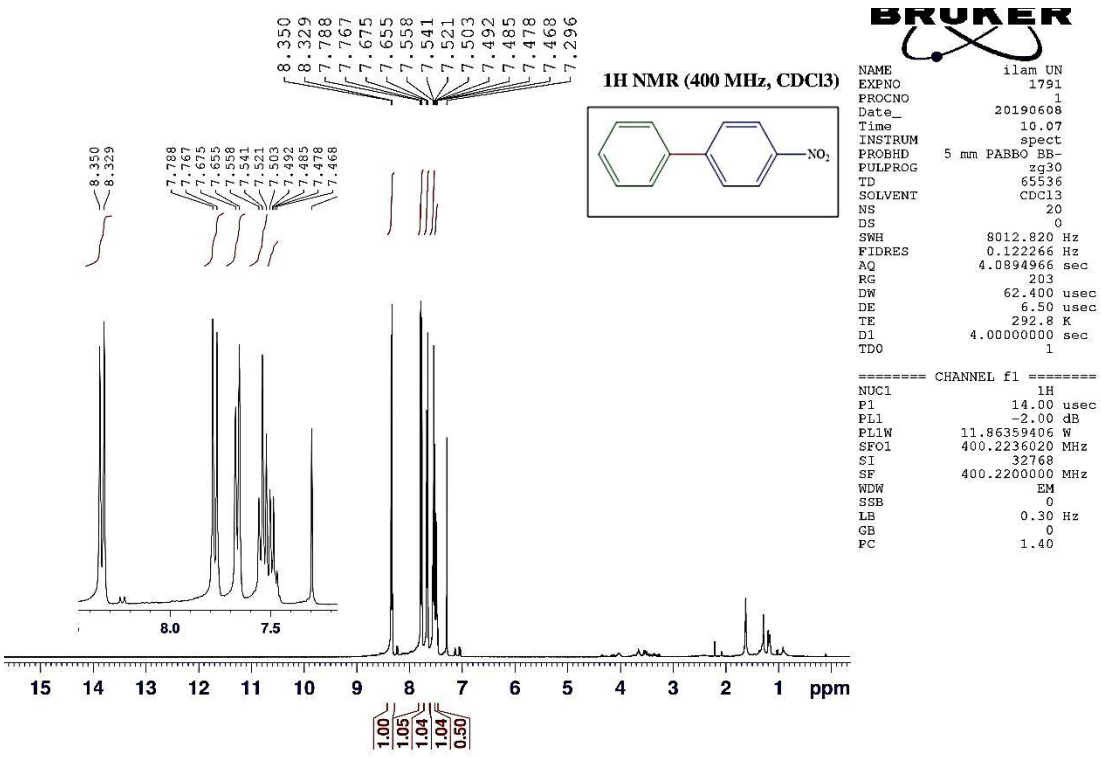


Fig S12. ^{1}H NMR spectrum of 4-Nitro-1,1'-biphenyl

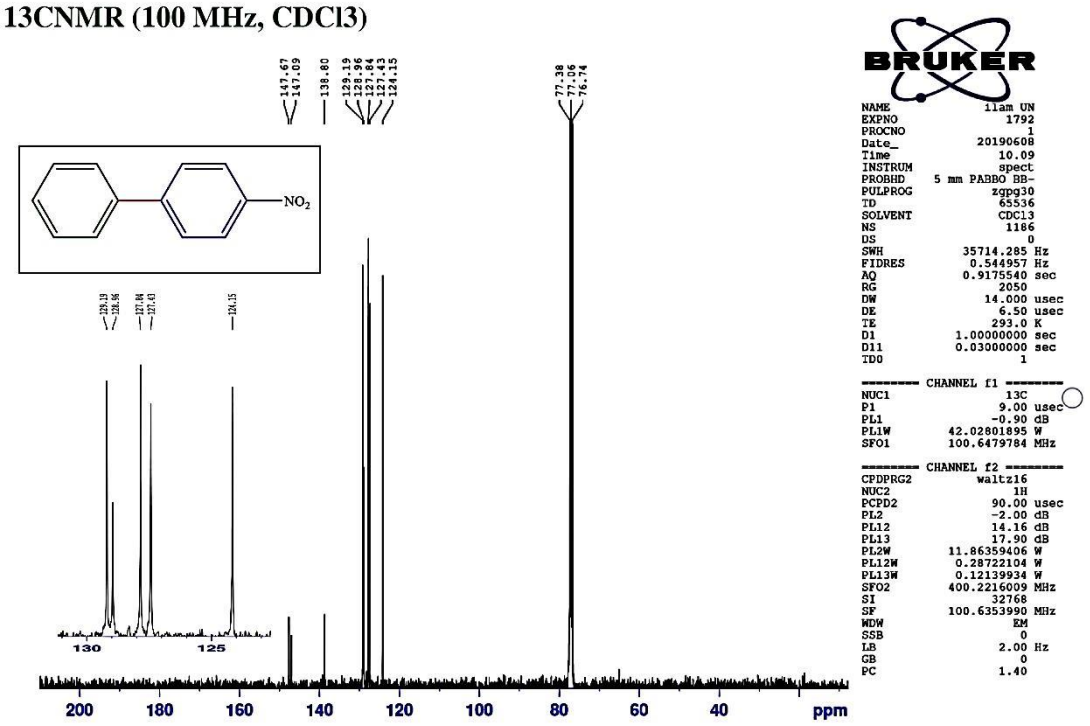


Fig S13. ^{13}C NMR spectrum of 4-Nitro-1,1'-biphenyl

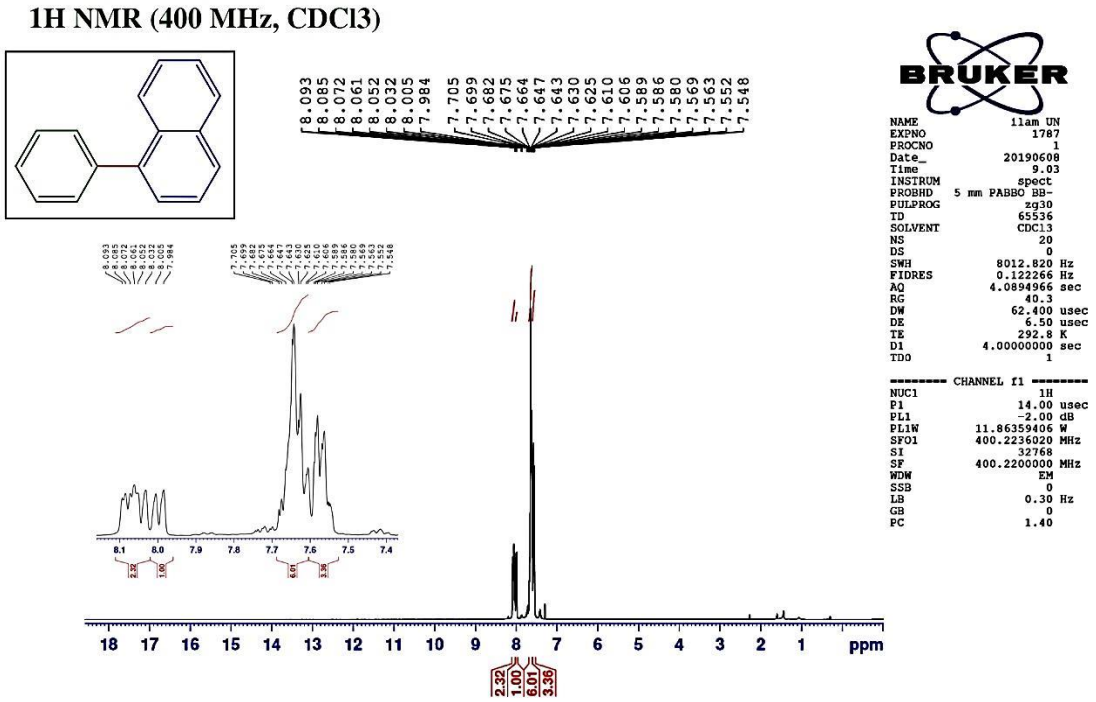


Fig S14. ^{1}H NMR spectrum of 2-Phenylnaphthalene

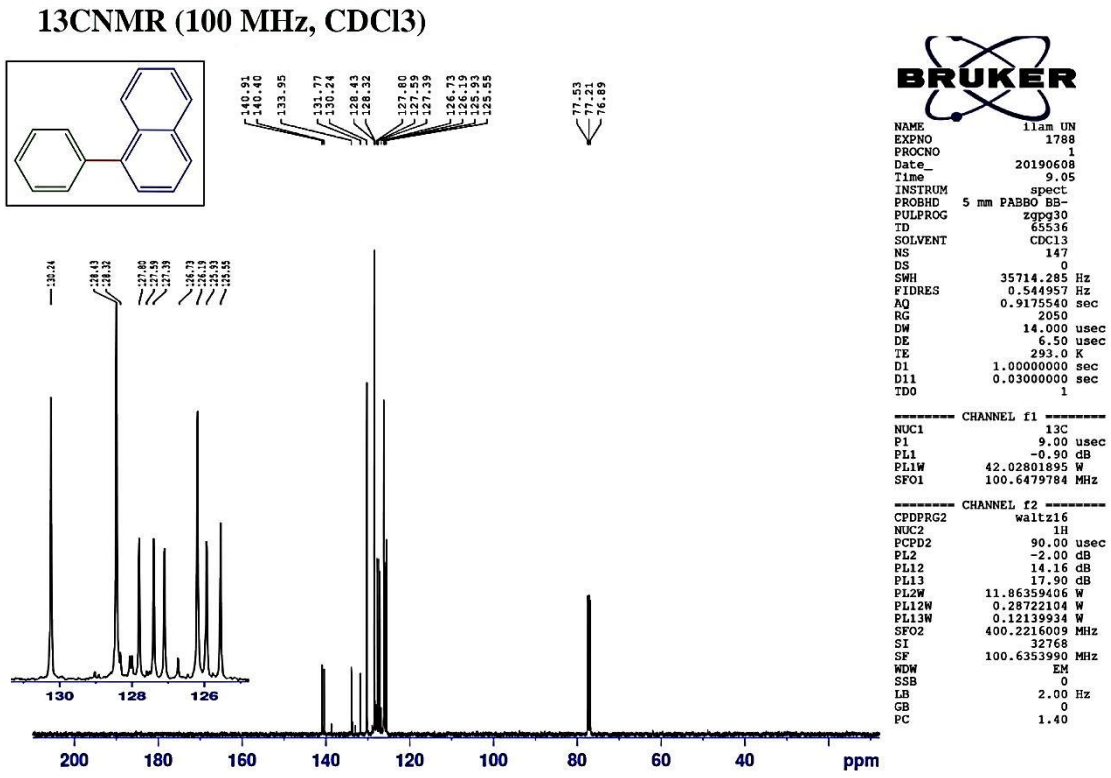


Fig S15. ^{13}C NMR spectrum of 2-Phenylnaphthalene

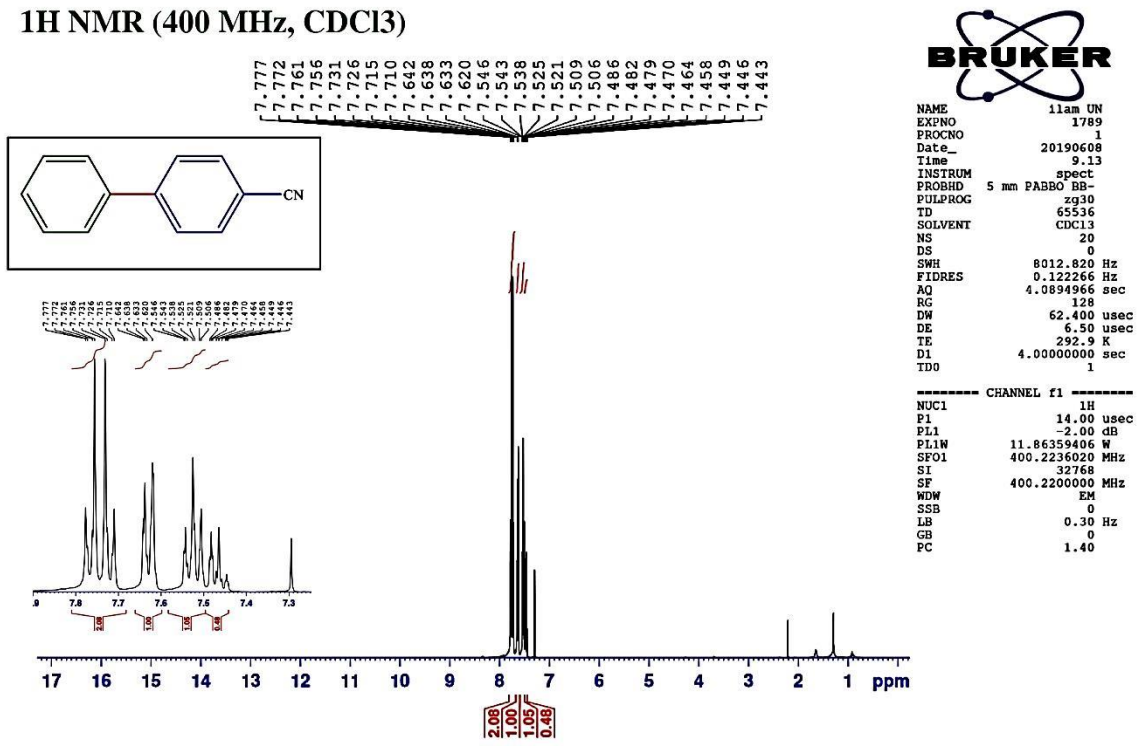


Fig S16. ^{1}H NMR spectrum of [1,1'- Biphenyl]-4-carbonitrile

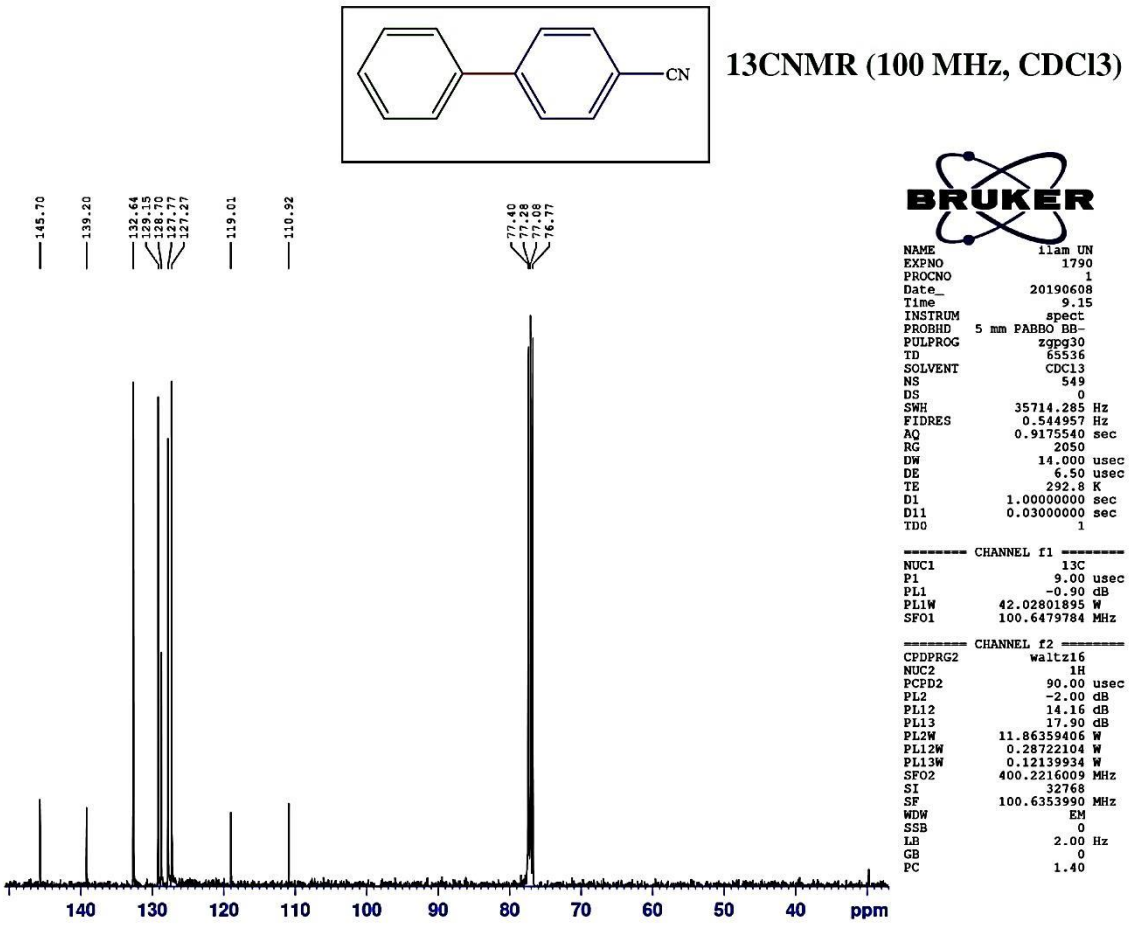


Fig S17. ^{13}C NMR spectrum of [1,1'- Biphenyl]-4-carbonitrile

Selected spectral data

1,1'-Biphenyl: White solid, Mp (°C): 63-65, [1] ^{1}H NMR (400 MHz, $CDCl_3$): δH= 7.41 (t, J= 8Hz, 2H), ^{13}CNMR (100 MHz, CDCl3): δ = 127.2, 127.3, 129.8, 141.3.

4-Methoxy-1,1'-biphenyl: White solid, Mp (°C):83-85, [2] ^{1}H NMR (400 MHz, $CDCl_3$): δH= 3.91(s, 3H), 7.03-7.05 (d, J= 8Hz, 2H), 7.47 (t, J= 8Hz, 2H), 7.60 (t, J= 8Hz, 2H) ppm. 13CNMR (100 MHz, $CDCl_3$): δ = 55.4, 114.2, 126.7, 126.8, 128.2, 128.8, 133.8, 140.9, 159.1.

4-Nitro-1,1'-biphenyl: Pale yellow solid, Mp (°C): 110-114, [1] ^{1}H NMR (400 MHz, $CDCl_3$): δH= 7.29-7.78 (m, 5H), 7.76-7.78 (d, J= 8 Hz, 2H), 8.29-8.50 (d, J= 8Hz, 2H) ppm. ^{13}CNMR (100 MHz, $CDCl_3$): δ = 124.15, 127.43, 127.84, 128.96, 129.19, 138.80, 147.09, 147.67.

2-Phenylnaphthalene: oil, [3] ^{1}H NMR (400 MHz, $CDCl_3$): δH= 7.54-7.58 (m,3H), 7.60-7.70 (m, 6H), 7.98-8.01 (m, 1H) ppm. ^{13}CNMR (100 MHz, $CDCl_3$): δ = 125.55, 125.93, 126.19, 126.73, 127.39, 127.59, 127.80, 128.32, 128.43, 130.24, 131.77, 133.95, 140.40, 140.91.

[1,1'-Biphenyl]-4-carbonitrile: White solid, Mp (°C) 80-82, [1] 1H NMR (400 MHz, $CDCl_3$): δH= 7.46 (t, J= 8Hz, 1H), 7.52 (t, J= 8Hz, 2H), 7.62-7.64 (d, J= 8 Hz, 2H),7.71-7.77 (q, J= 8Hz, 4H) ppm. ^{13}CNMR (100 MHz, $CDCl_3$): δ = 110.92, 119.01, 127.27, 127.77, 128.70, 129.15, 132.64, 139.20, 145.70.

4-Methyl-1,1'-bipheny: White solid, Mp (°C): 44-46, [1] ^{1}H NMR (400 MHz, $CDCl_3$): δH= 2.51(s, 3H), 7.36 (t, J= 8Hz, 2H), 7.54 (t, J= 8Hz, 2H), 7.61 (t, J= 8Hz, 2H), 7.69-7.71 (d, J= 8Hz, 2H) ppm.^{13}CNMR (100 MHz, $CDCl_3$): δ =21.1, 126.9, 127.1, 128.9, 129.5, 136.8, 137.1, 138.4, 141.2.

References

1. Q. Zhang, H. Su, J. Luo and Y. Wei, Tetrahedron. 14 (2013) 447-54.
2. J. F. Wei, J. Jiao, J. J, Feng, J. Lv, X. R. Zhang, X. Y. Shi and Z. G. Chen, J. Org. Chem. 74 (2009) 6283-6.
3. L. Bai and J.X. Wang, Adv. Synth. Catal. 25 (2008) 315-20.