Electronic Supplementary Material (ESI)

for

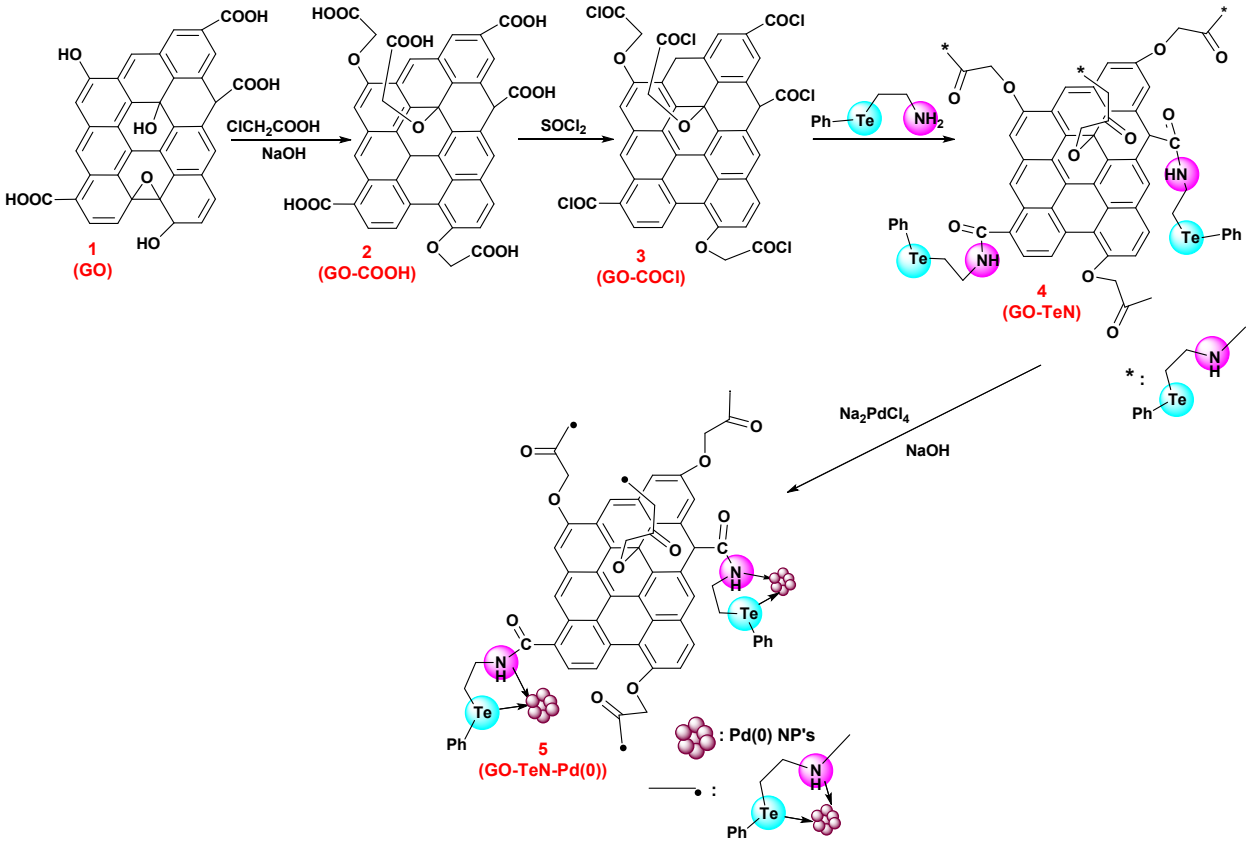
Catalytic system having organotellurium ligand on graphene oxide: immobilization of Pd(0) nanoparticles and application in heterogeneous catalysis of cross-coupling reactions

Suraj Purohit,[a] Preeti Oswal,[a] Anurag Bahuguna,[a] AnupmaTyagi,[a] NeerajBhatt,[a] and Arun Kumar[a]*

[a]*Department of Chemistry, School of Physical Sciences, Doon University, Dehradun-248001, India*

Corresponding author: Arun Kumar, E-mail:arunkaushik@gmail.com, akumar.ch@doonuniversity.ac.in

Table of Contents



Scheme S1. Methodology for the synthesis of GO, GO−COOH, GO−COCl, GO−TeN and GO−TeN−Pd(0)

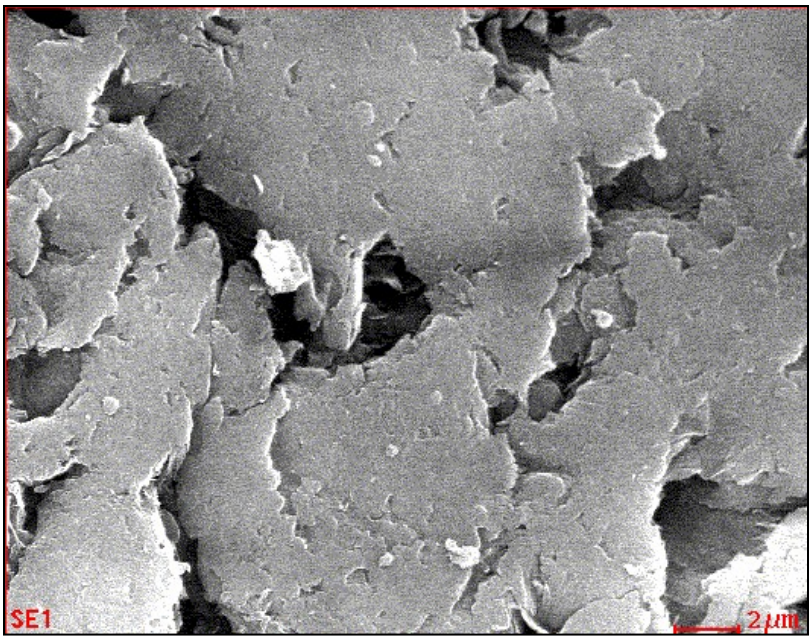


Fig. S1. Area under SEM-EDS analysis for graphene oxide (GO)

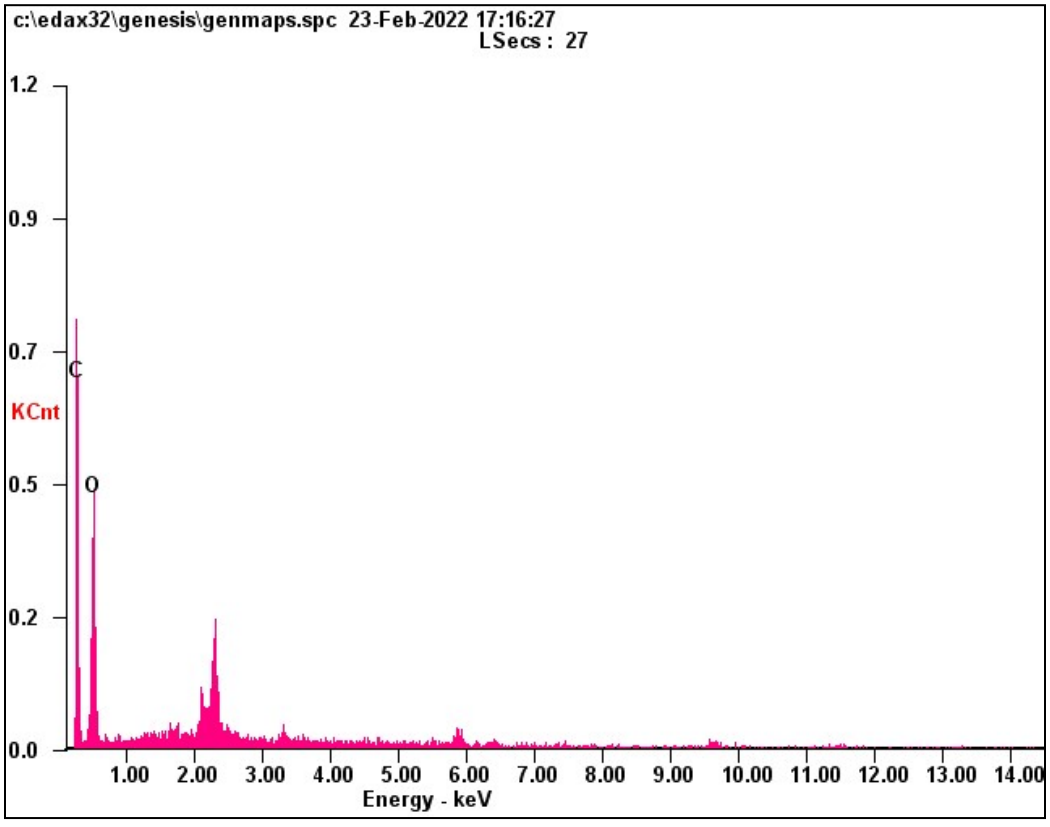


Fig. S2. EDS spectrum (composition plot) of graphene oxide (GO)

Table S1. EDS analysis of graphene oxide (GO)

Element	*Wt%*	*At%*
CK	56.04	62.93
OK	43.96	37.07
Matrix	Correction	ZAF

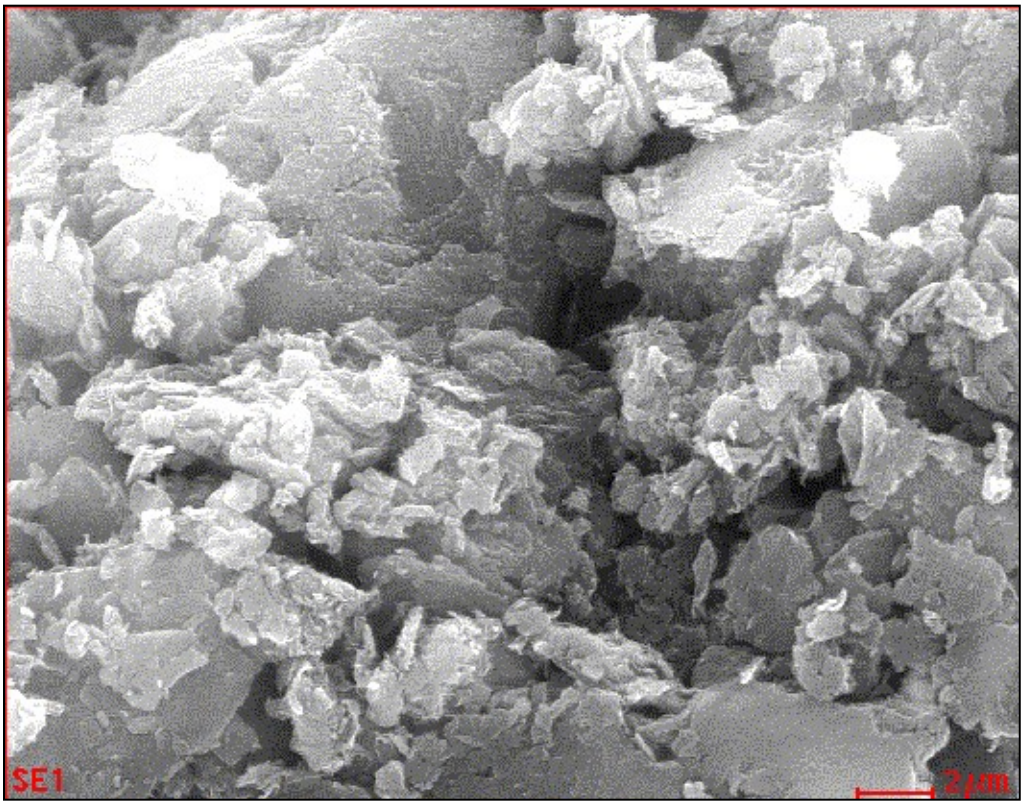


Fig S3. Area under SEM-EDS analysis for GO-TeN-Pd(0)

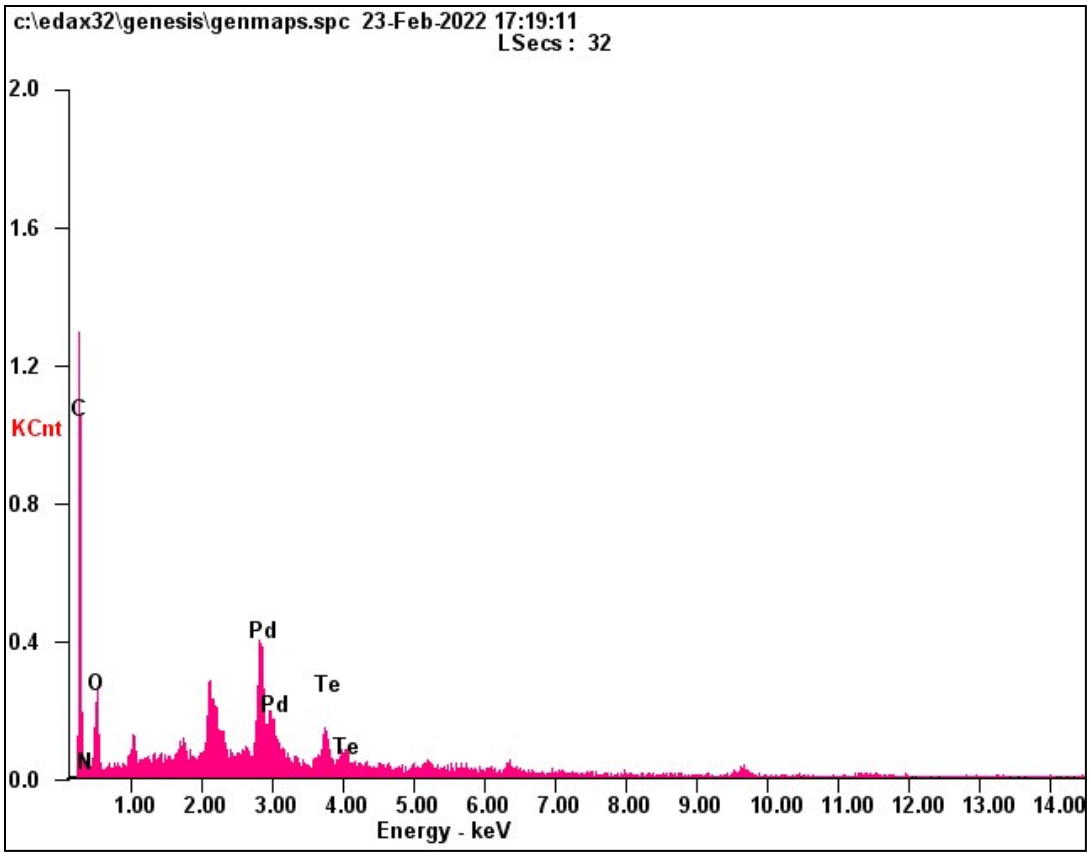


Fig S4. EDS spectrum (composition plot) of GO-TeN-Pd(0)

Table S2. EDS analysis of GO-TeN-Pd(0)

Element	***Wt%***	***At%***
CK	57.84	71.64
NK	05.23	05.55
OK	22.47	20.90
PdL	09.86	01.38
TeL	04.61	00.54
Matrix	Correction	ZAF

.

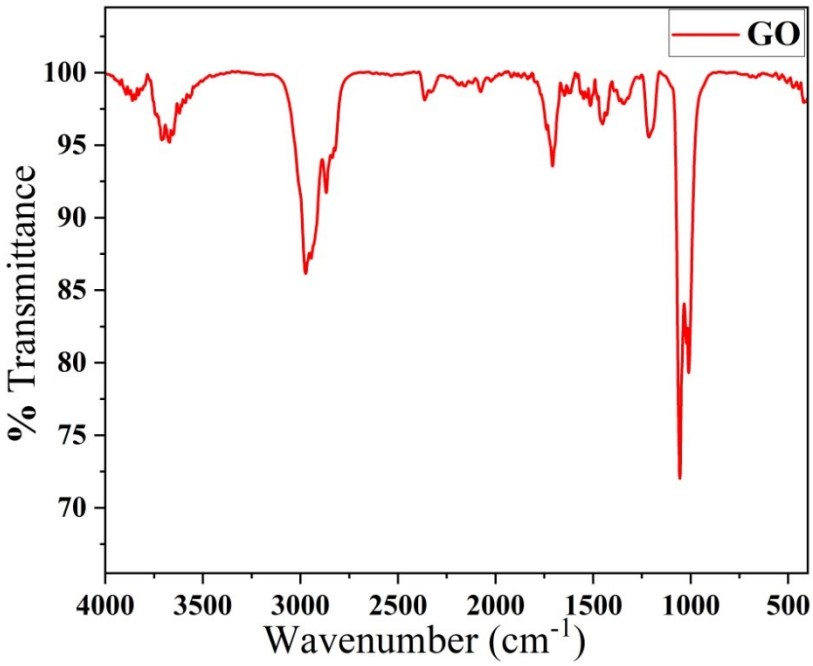


Fig S5. FT-IR spectrum of graphene oxide (GO)

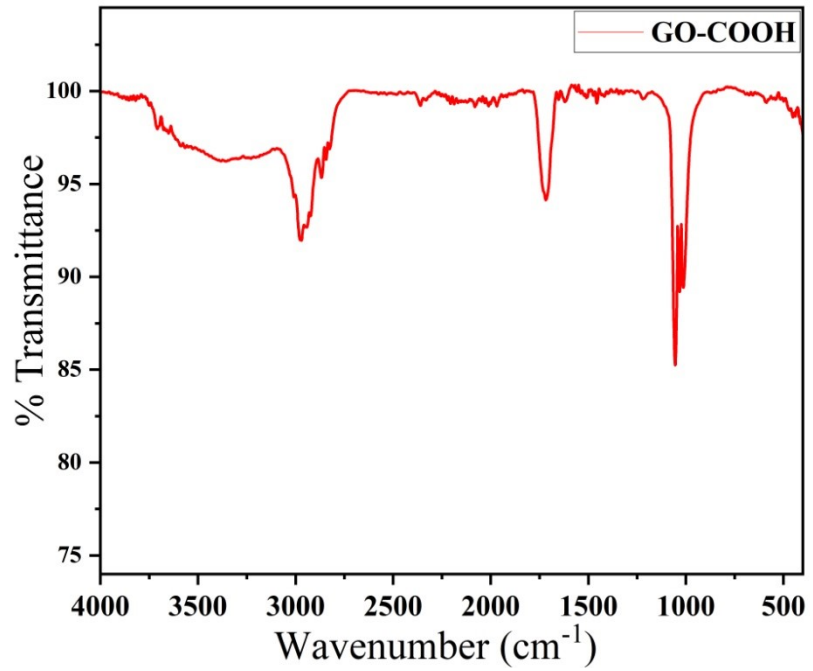


Fig S6. FT-IR spectrum of GO-COOH

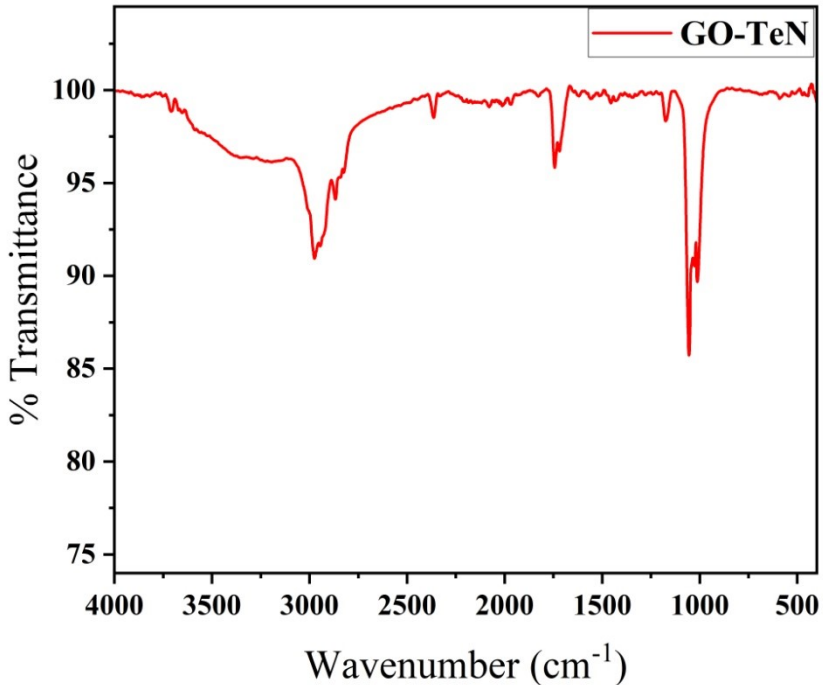


Fig S7. FT-IR spectrum of GO−TeN

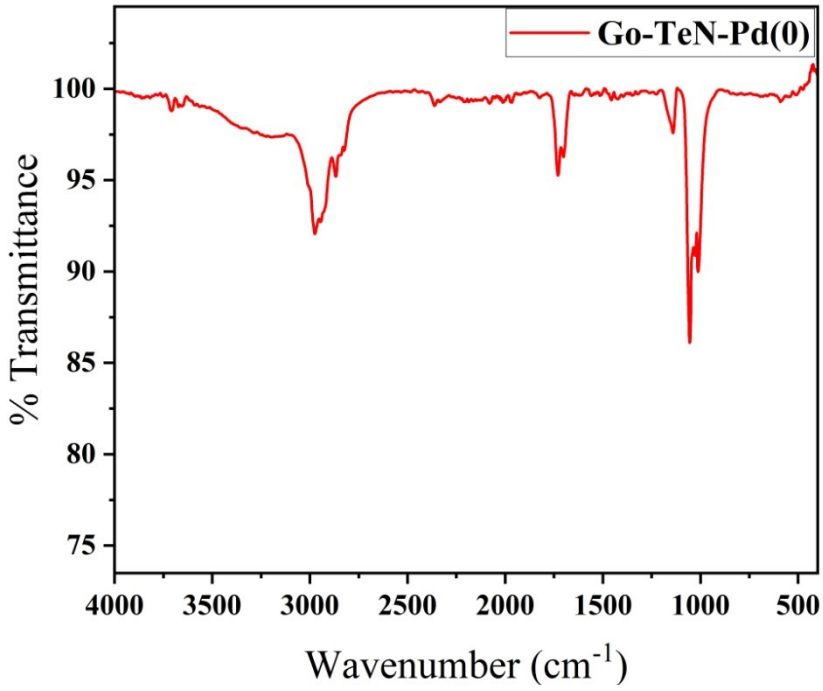


Fig S8. FT-IR spectrum of GO−TeN−Pd(0)

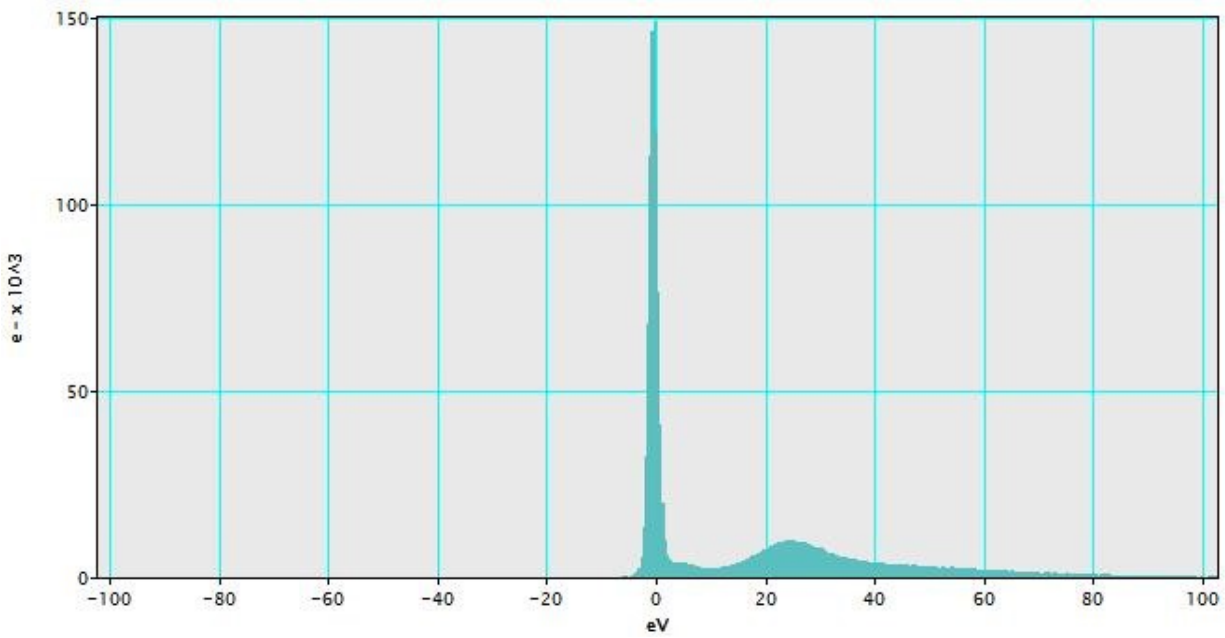


Fig S9. Low-loss EELS spectrum of graphene oxide (GO)

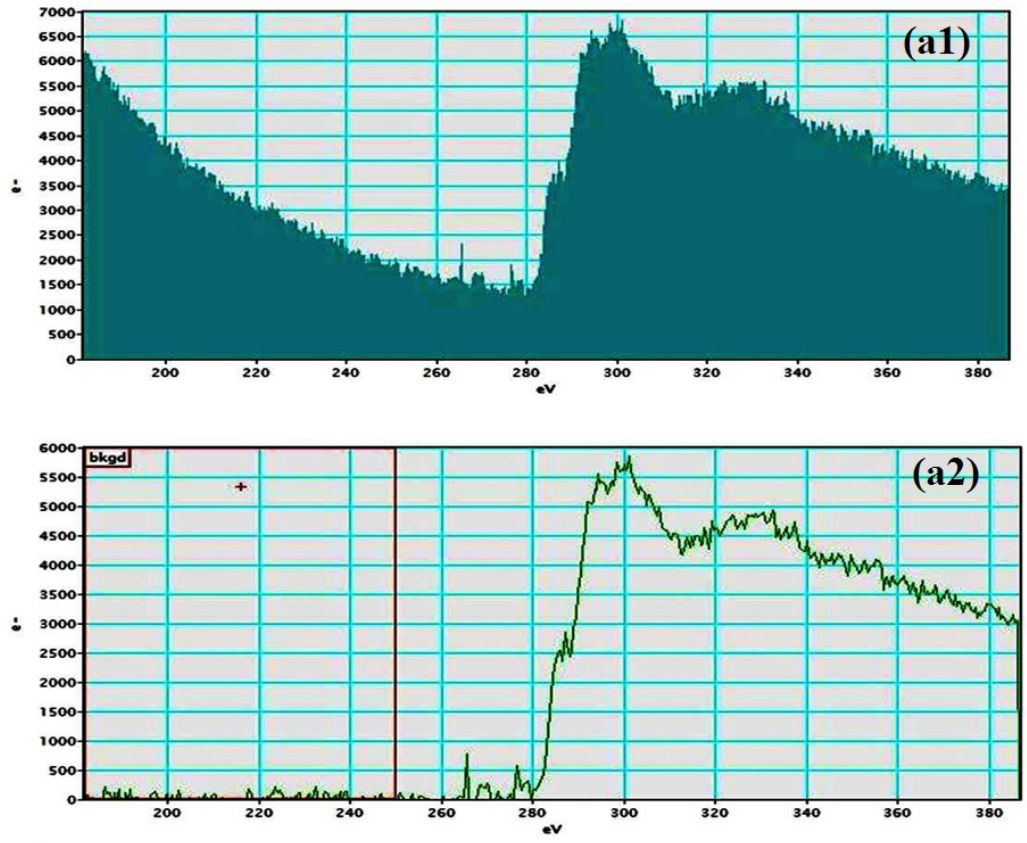


Fig S10. Carbon K-edge (a1, a2) EELS spectrum of graphene oxide (GO)

Fig S11. Oxygen K-edge (b1, b2) EELS spectrum of graphene oxide (GO)

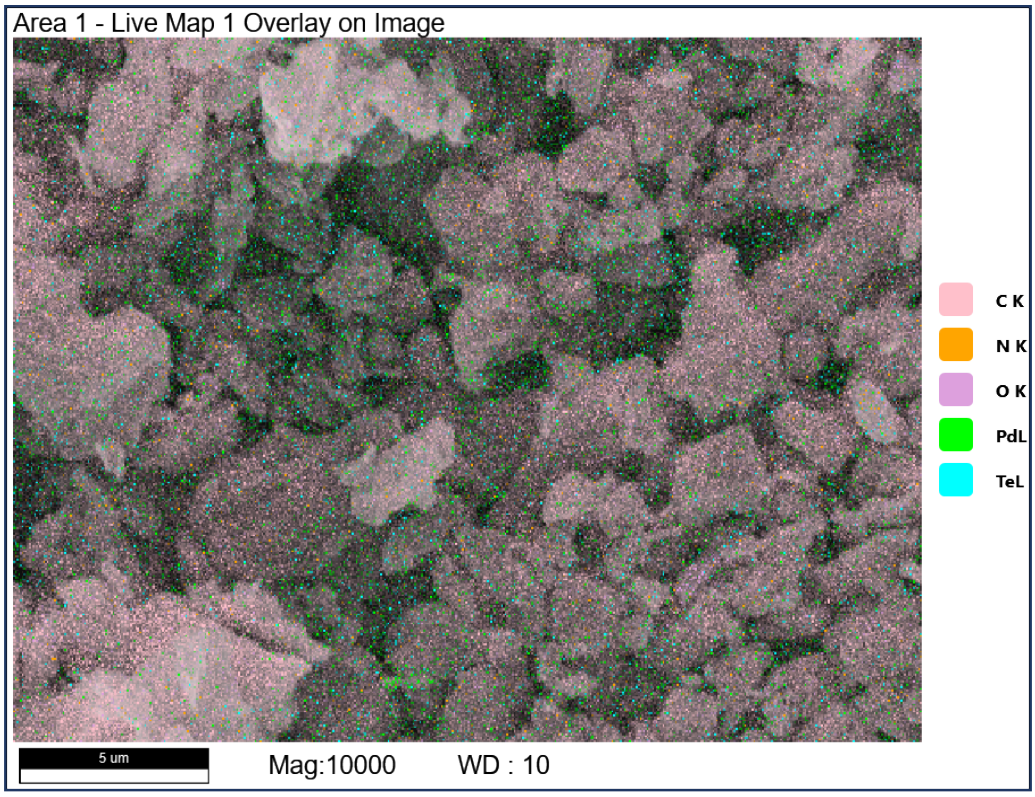


Fig S12: SEM-EDS (elemental mapping) layered view of recycled GO-TeN-Pd(0) nanocatalyst

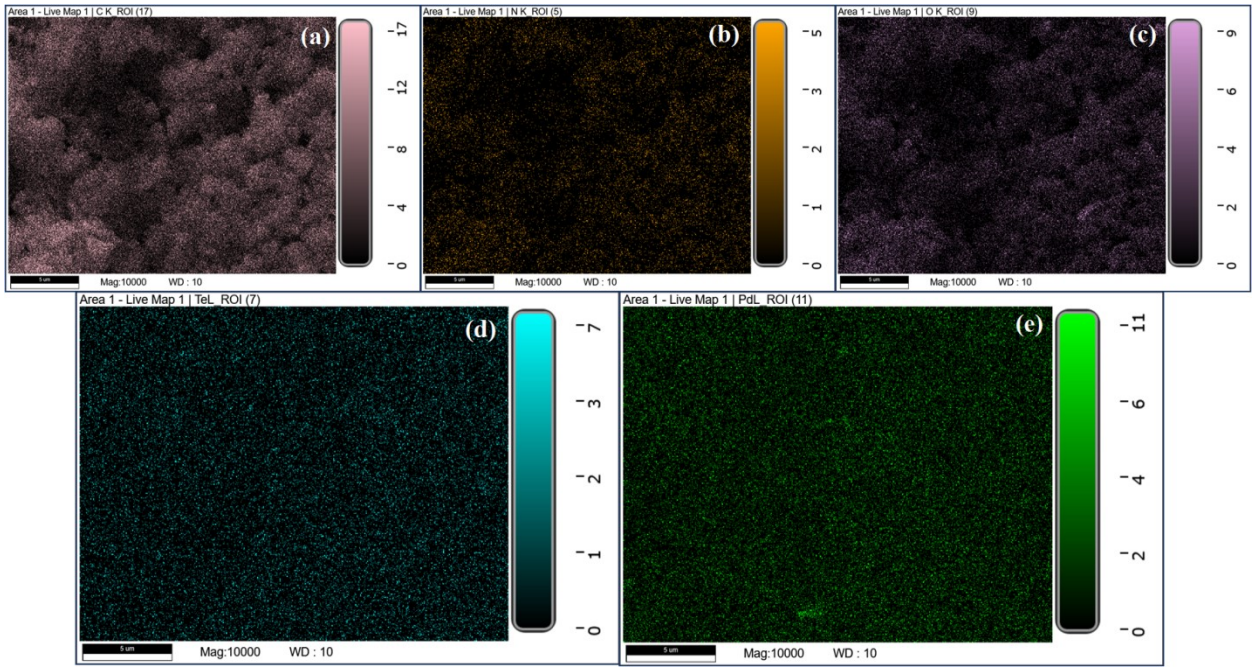


Fig S13: SEM-EDS (elemental mapping) of (a) carbon (b) nitrogen (c) oxygen (d) tellurium and (e) palladium of recycled GO-TeN-Pd(0) nanocatalyst

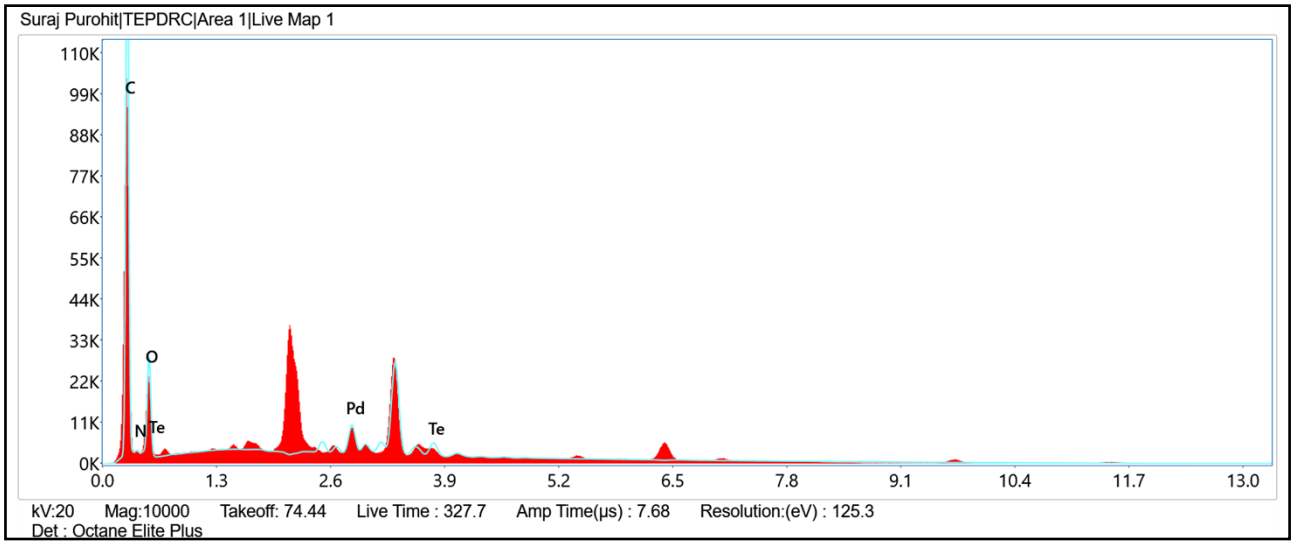


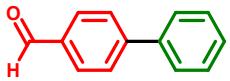
Fig. S14: SEM-EDS data (composition plot) of recycled GO-TeN-Pd(0) nanocatalyst

Table S3. EDS analysis of recycled GO-TeN-Pd(0)nanocatalyst

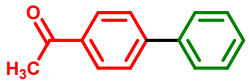
eZAF Quant Result - Analysis Uncertainty: 14.78 %		
Element	***Weight %***	***Atomic %***
C K	*70.27*	*77.71*
N K	*3.22*	*3.05*
O K	*22.64*	*18.79*
Pd L	*2.24*	*0.28*
Te L	*1.64*	*0.17*

[1]H-NMR data of C-C cross-coupled products (6-15) of Suzuki-Miyaura coupling

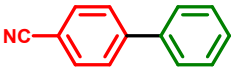
---

Compound 6: [1,1'-Biphenyl]-4-carbaldehyde :[1] 

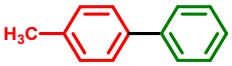
[1]H-NMR (500 MHz, CDCl3): δ (ppm) 10.08(s, 1H), 7.97(dt, J = 8.0, 1.5 Hz, 2H), 7.77(dd, J = 7.0, 1.5 Hz, 2H), 7.65(dt, J = 8.5, 1.5 Hz, 2H), 7.52-7.48(m, 2H), 7.44(tt, J = 7.0, 1.0 Hz, 1H).[13]C-NMR (125 MHz, CDCl3): δ (ppm) 192.0, 147.1, 141.1, 139.6, 135, 130.2, 128.9, 127.6. 127.3

Compound 7: 1-([1,1'-Biphenyl]-4-yl)ethanone :[1] 

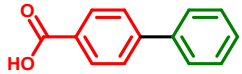
[1]H-NMR (500 MHz, CDCl3): δ (ppm) 8.05(dt, J = 10.0, 2.0 Hz, 2H), 7.70(dt, J = 9.0, 1.8 Hz, 2H), 7.64(dd, J = 9.0, 2.0 Hz, 2H), 7.49(t, J = 7.2 Hz, 2H), 7.42(tt, J = 7.5, 1.5 Hz, 1H), 2.66(s, 3H). [13]C-NMR (125 MHz, CDCl3): δ (ppm) 173.6, 131.9, 127.2, 123.9, 118.53, 118.50, 117.95, 117.17, 117.13, 36.71

Compound 8: [1,1'-Biphenyl]-4-carbonitrile :[1] 

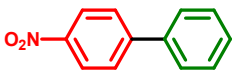
[1]H-NMR (500 MHz, CDCl3): δ (ppm) 7.72 (d, 2H, J = 8.0 Hz), 7.68 (d, 2H, J = 8.0 Hz), 7.59 (d, 2H, J = 6.8 Hz), 7.51-7.41 (m, 3H). [13]C-NMR (125 MHz, CDCl3): δ (ppm) 131.85, 126.64, 121.41, 118.63, 118.27, 117.51, 117.12, 110.52, 104.02

Compound 9: 4-Methyl-1,1'-biphenyl :[1] 

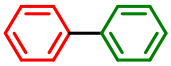
[1]H-NMR (500 MHz, CDCl3): δ (ppm) 7.62(t, J = 4.2 Hz, 2H), 7.53(d, J = 8.0 Hz, 2H), 7.46(t, J = 7.5 Hz, 2H), 7.36(t, J = 11.5 Hz, 1H), 7.28(t, J = 5.8 Hz, 2H), 2.43(s, 3H). [13]C-NMR (125 MHz, CDCl3): δ (ppm) 128.36, 125.99, 124.74, 118.93, 118.38, 117.11, 116.82, 107.59, 32.25

Compound 10: [1,1'-Biphenyl]-4-carboxylic acid :[2] 

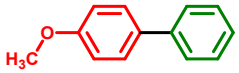
[1]H NMR (500 MHz, DMSO-d6): δ (ppm) 13.17(s, 1H), 8.03(d, J = 8.5 Hz, 2H), 7.79(d, J = 8.0 Hz, 2H), 7.74(t, J = 4.2 Hz, 2H), 7.51(t, J = 7.5 Hz, 2H), 7.42(t, J = 7.2 Hz, 1H).[13]C-NMR (125 MHz, CDCl3): δ (ppm) 172.03, 146.50, 141.36, 133.75, 130.17, 129.55, 129.32, 128.46, 124.91

Compound 11: 4-Nitro-1,1'-biphenyl :[1] 

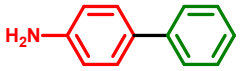
^{1}H-NMR (500 MHz, CDCl3): δ (ppm)8.30 (d, J = 9.0 Hz, 2H),7.74 (d, J = 9.0 Hz, 2H),7.63 (d, J = 9.0 Hz, 2H), 7.45-7.53 (m, 3H). ^{13}C-NMR (125 MHz, CDCl3): δ (ppm) 147.56, 146.99, 138.68, 129.11, 128.88, 127.74, 127.33, 124.05

Compound 12: Biphenyl :[1] 

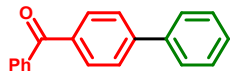
^{1}H-NMR (500 MHz, CDCl3): δ (ppm) 7.59(dd, J = 8.5, 2.0 Hz, 4H), 7.44(t, J = 8.0 Hz, 4H), 7.34(t, J = 7.5 Hz, 2H). ^{13}C-NMR (125 MHz, CDCl3): δ (ppm) 141.21, 128.74, 127.15, 115.24

Compound 13: 4-Methoxy-1,1'-biphenyl :[1] 

^{1}H-NMR (500 MHz, CDCl3): δ (ppm) 7.59-7.53(m, 4H), 7.43(t, J = 7.8 Hz, 2H), 7.32(tt, J = 7.5, 3.0 Hz, 1H), 7.00(dt, J = 9.5, 3.0 Hz, 2H), 3.87(s, 3H). ^{13}C-NMR (125 MHz, CDCl3): δ (ppm) 137.42, 135.45, 132.28, 130.92, 129.80, 129.47, 127.63, 21.99

Compound 14: [1,1'-Biphenyl]-4-amine :[2] 

^{1}H-NMR (500 MHz, CDCl3): δ (ppm) 7.52−7.56 (m, 2H), 7.37−7.43(m, 4H), 7.21−7.25 (m, 1H), 6.75 (d, *J* = 8.4 Hz, 2H), 3.71 (s, 2H). ^{13}C-NMR (125 MHz, CDCl3): δ (ppm) 145.33, 141.17, 131.97, 129.62, 128.72, 127.13, 116.69, 110.19

Compound 15: [1,1'-Biphenyl]-4-yl(phenyl)methanone : 

^{1}H-NMR (500 MHz, CDCl3): δ (ppm)7.42-7.49 (m, 6H), 7.33 (d, *J*=7.2Hz, 2H), 2.28 (s, 6H). ^{13}C-NMR (125 MHz, CDCl3): δ (ppm) 172.46, 131.53, 127.31, 125.53, 124.31, 121.27, 119.95, 119.36, 118.54, 118.01, 117.19, 116.93

NMR Spectra of Suzuki-Miyaura cross coupled products.

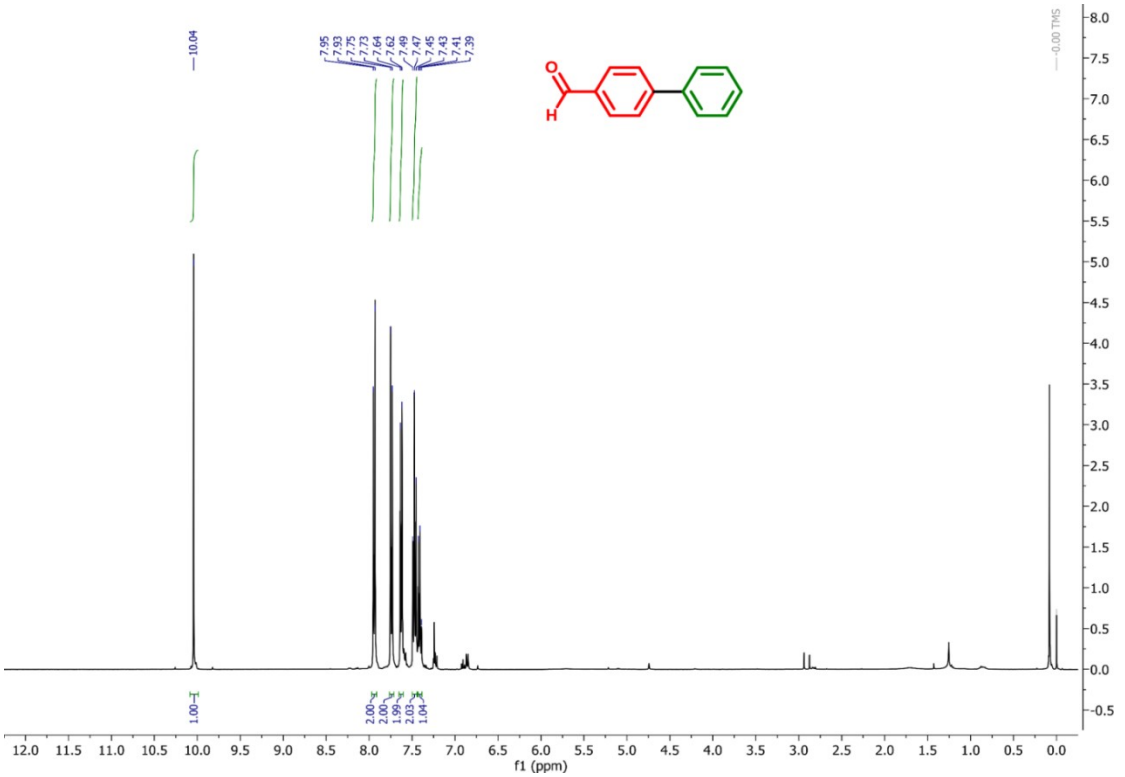


Fig S15. Full view of ^{1}H NMR spectrum of [1,1'-biphenyl]-4-carbaldehyde

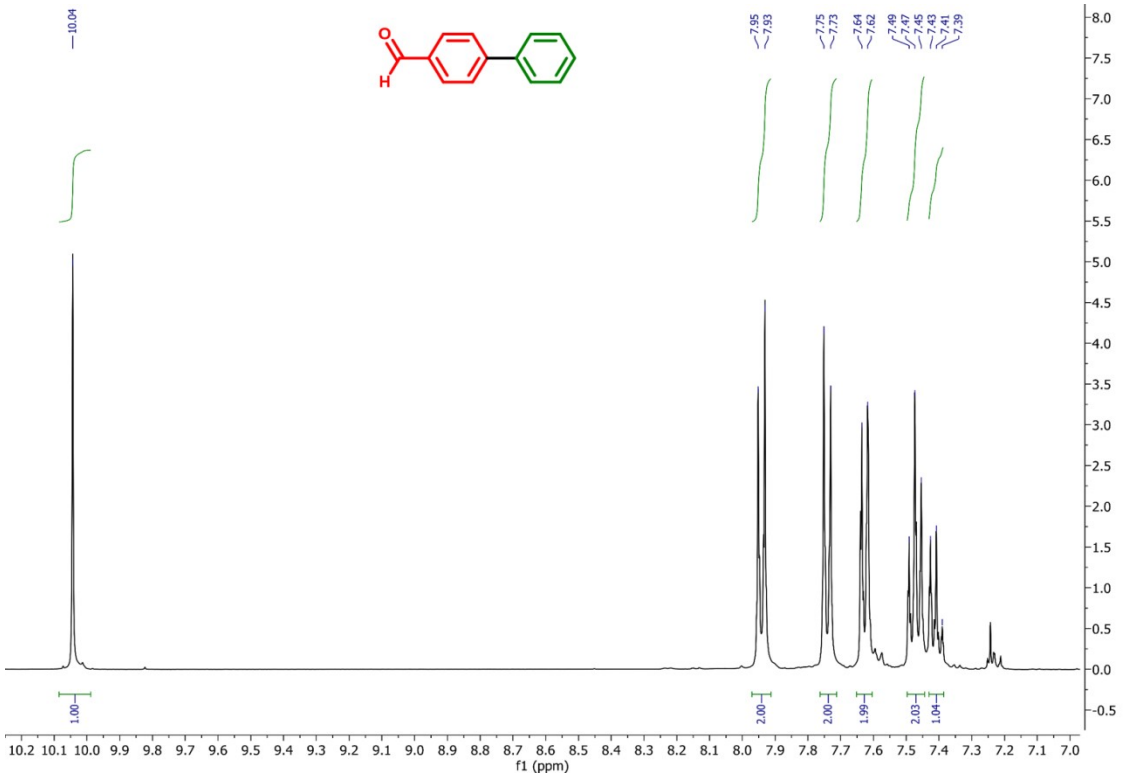


Fig S16. Zoomed view of ^{1}H NMR spectrum of [1,1'-biphenyl]-4-carbaldehyde

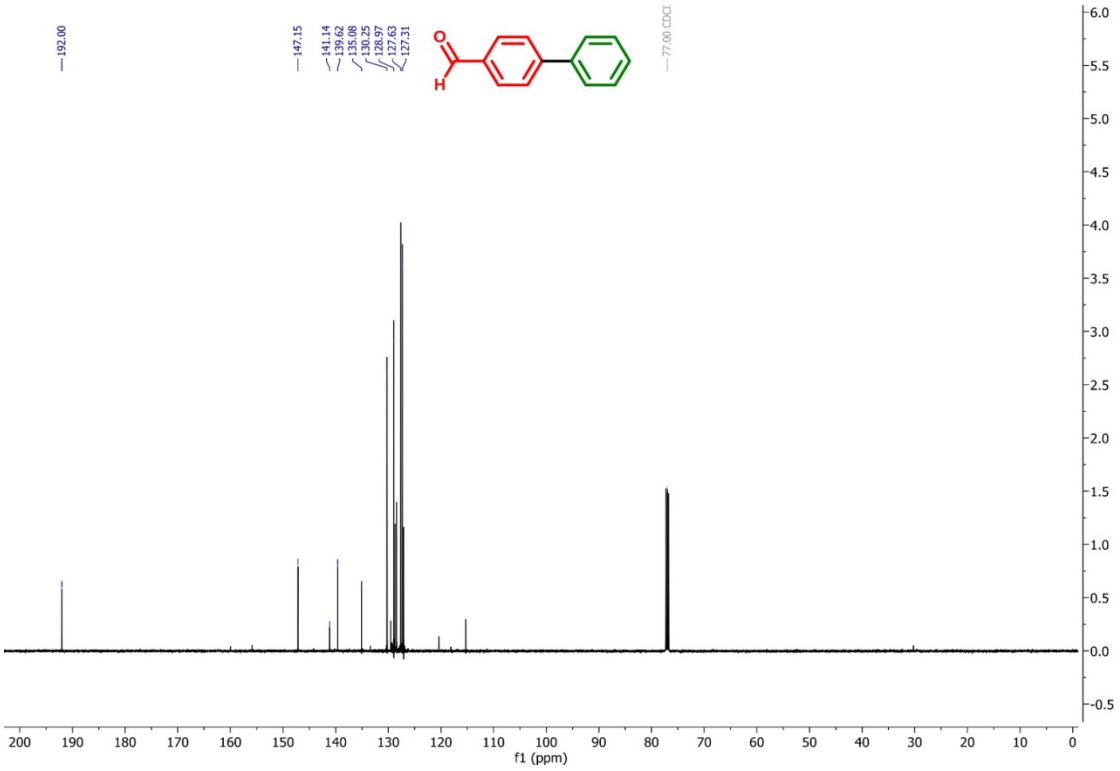


Fig S17. ^{13}C NMR spectrum of [1,1'-biphenyl]-4-carbaldehyde

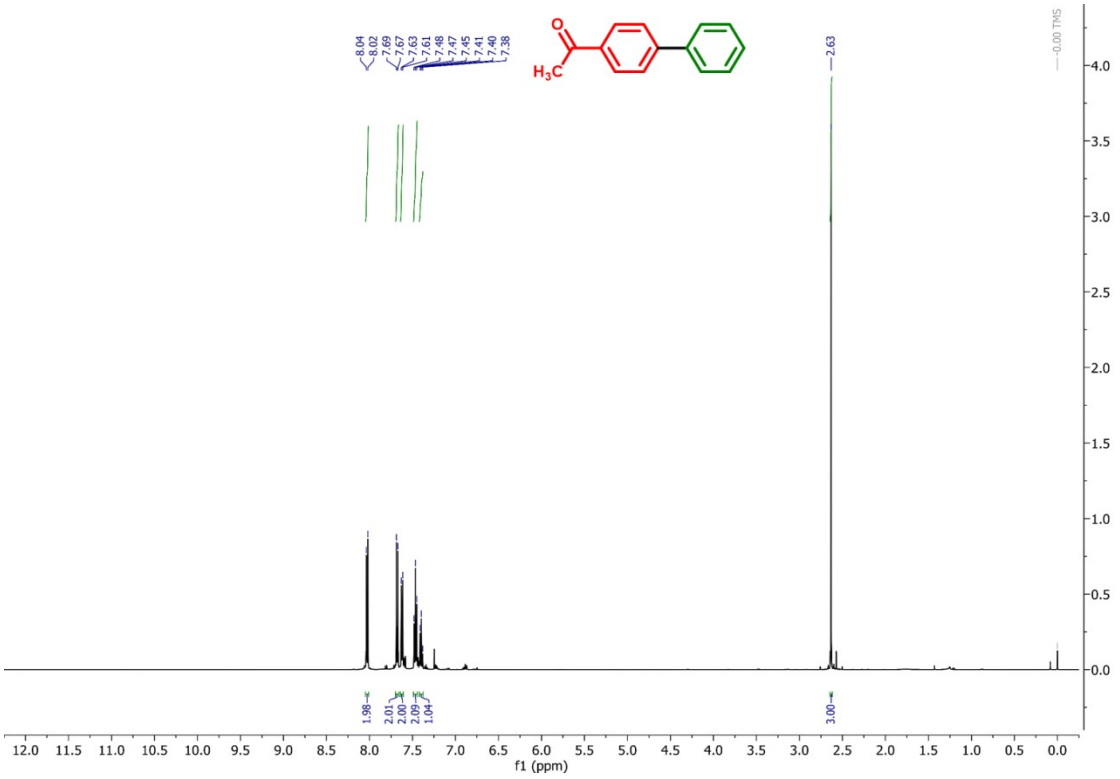


Fig S18. Full view of ^{1}H NMR spectrum of 1-([1,1'-biphenyl]-4-yl)ethanone

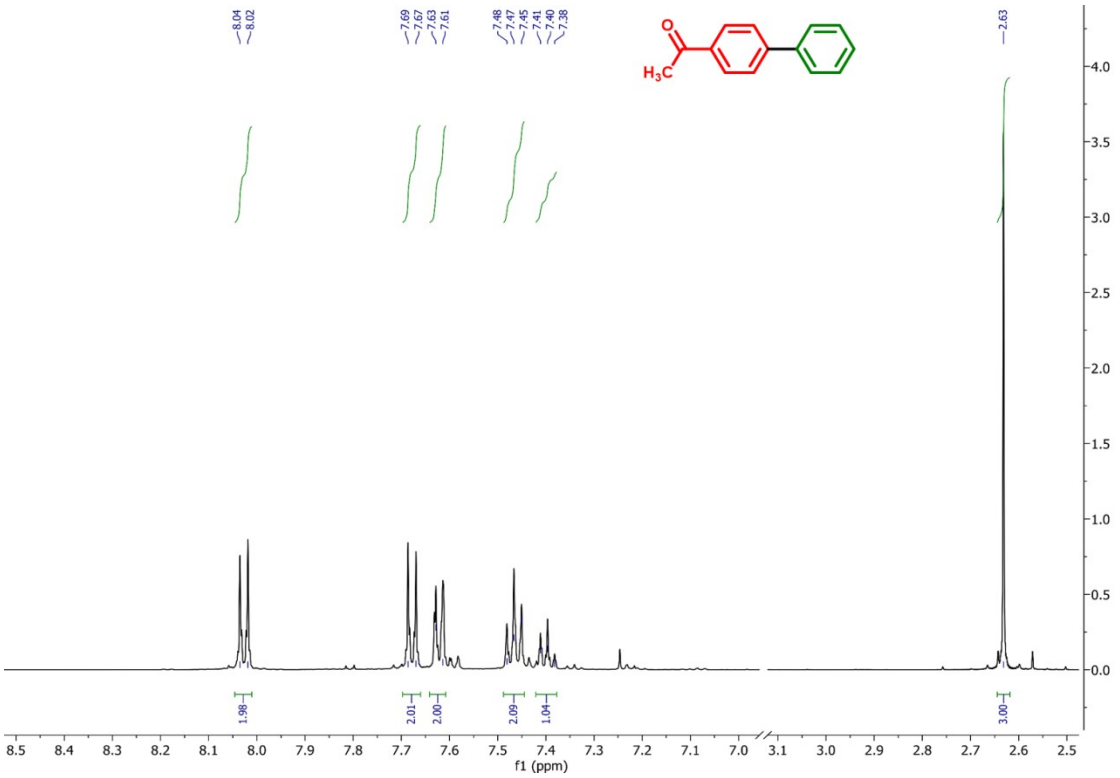


Fig S19. Zoomed view of ^{1}H NMR spectrum of 1-([1,1'-biphenyl]-4-yl)ethanone

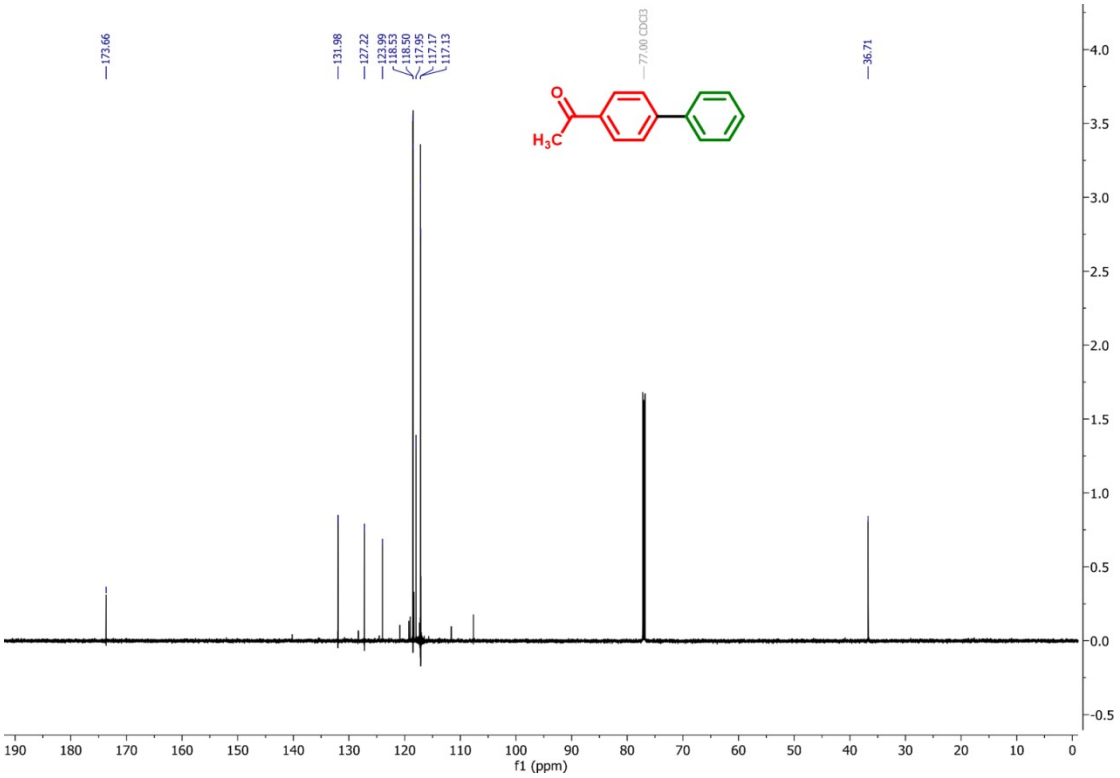


Fig S20. ^{13}C NMR spectrum of 1-([1,1'-biphenyl]-4-yl)ethanone

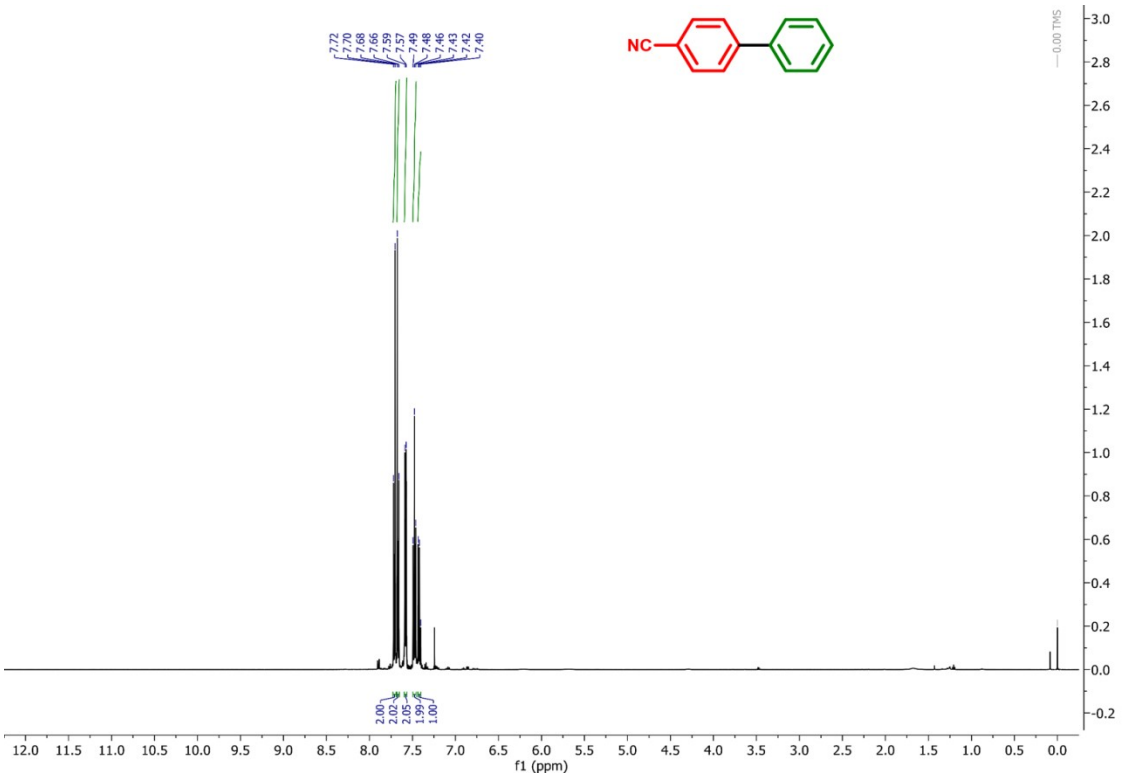


Fig S21. Full view of ^{1}H NMR spectrum of [1,1'-biphenyl]-4-carbonitrile

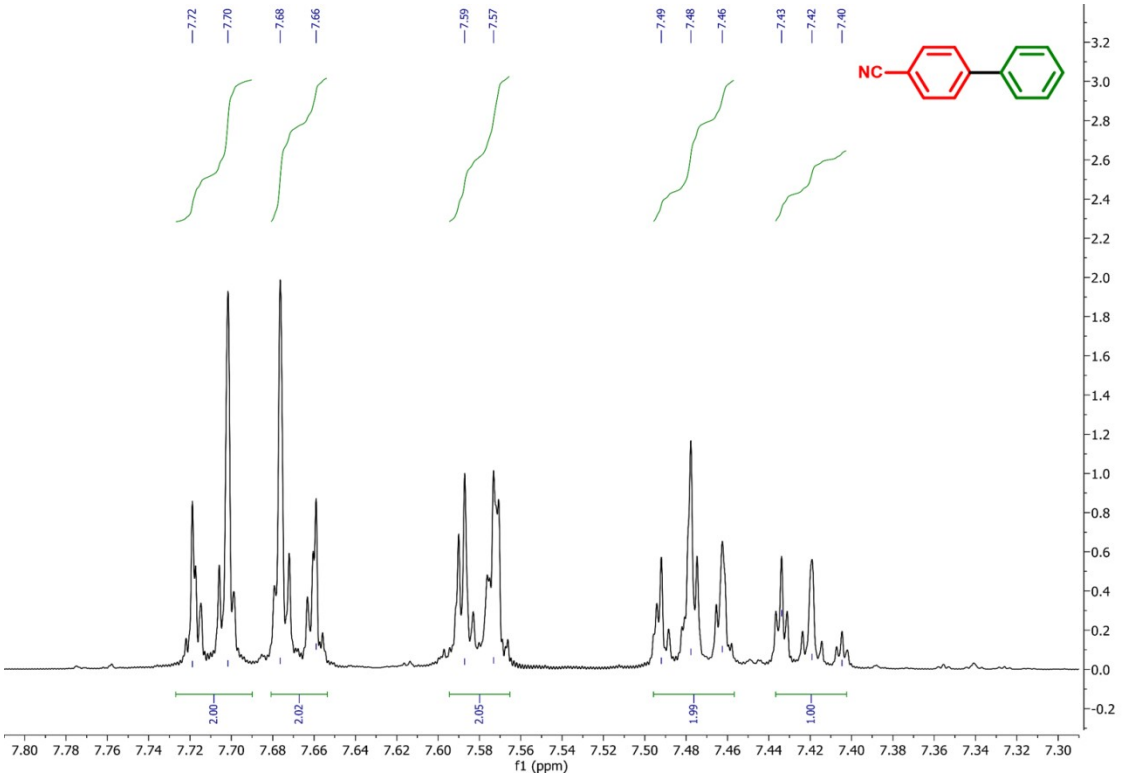


Fig S22. Zoomed view of ^{1}H NMR spectrum of [1,1'-biphenyl]-4-carbonitrile

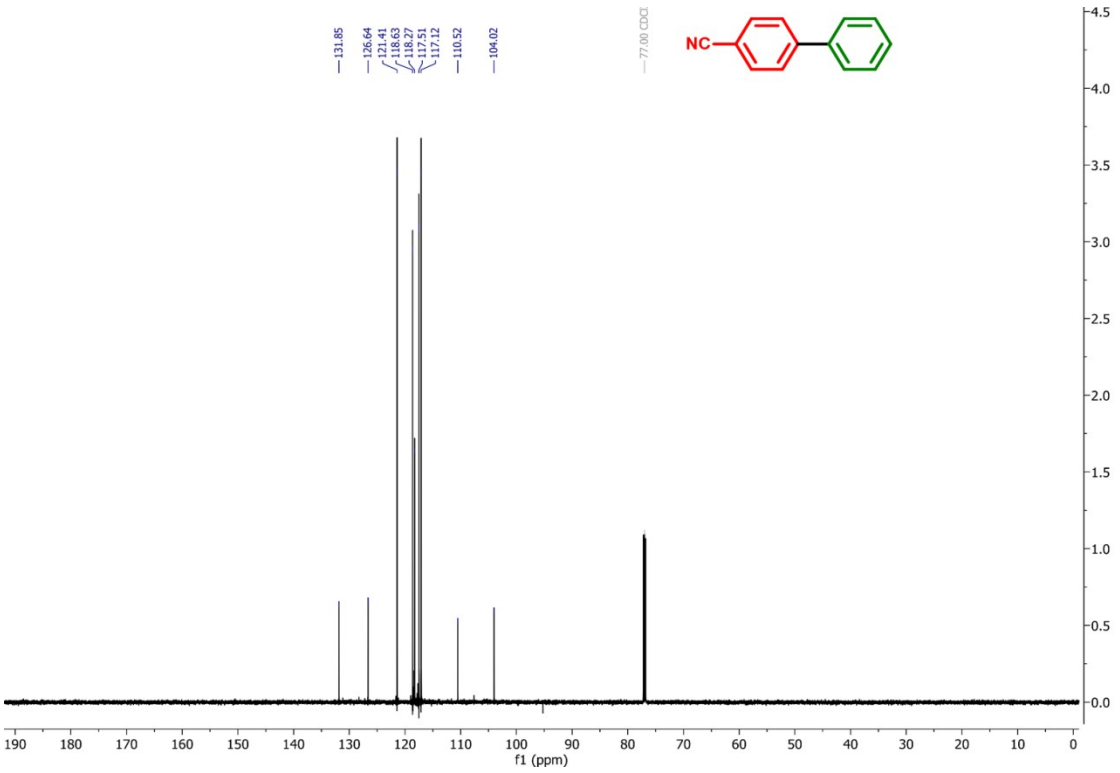


Fig S23. ^{13}C NMR spectrum of [1,1'-biphenyl]-4-carbonitrile

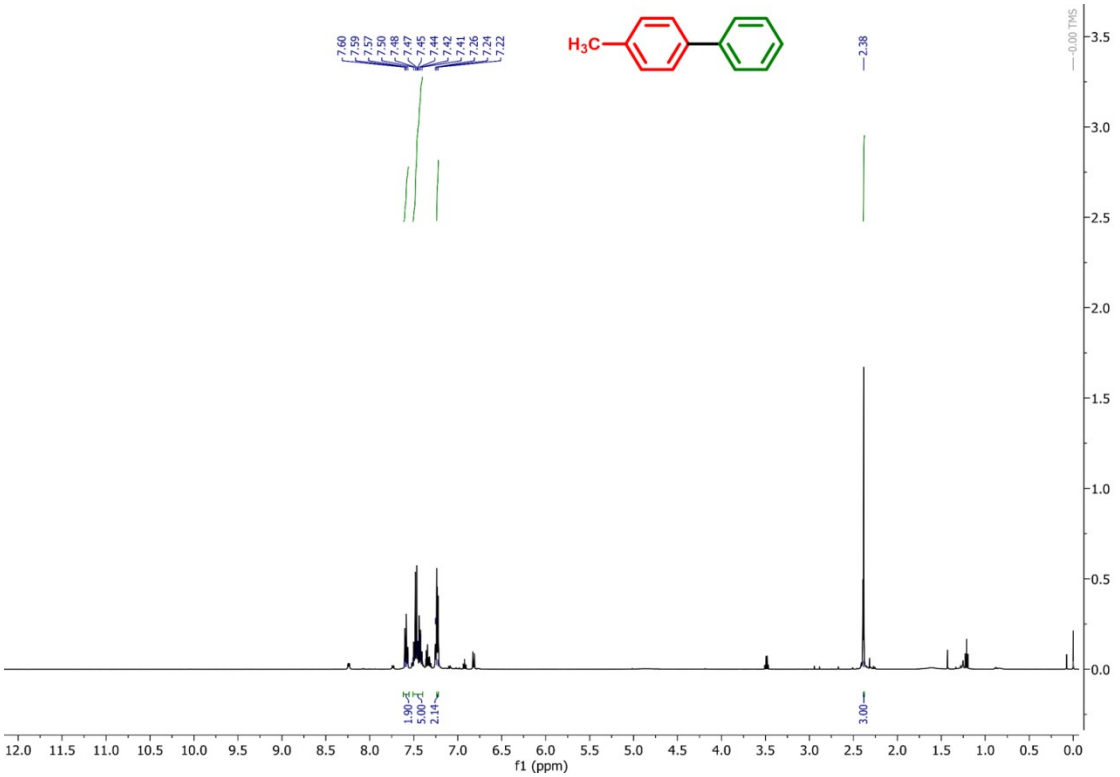


Fig S24. Full view of ^{1}H NMR spectrum of 4-methyl-1,1'-biphenyl

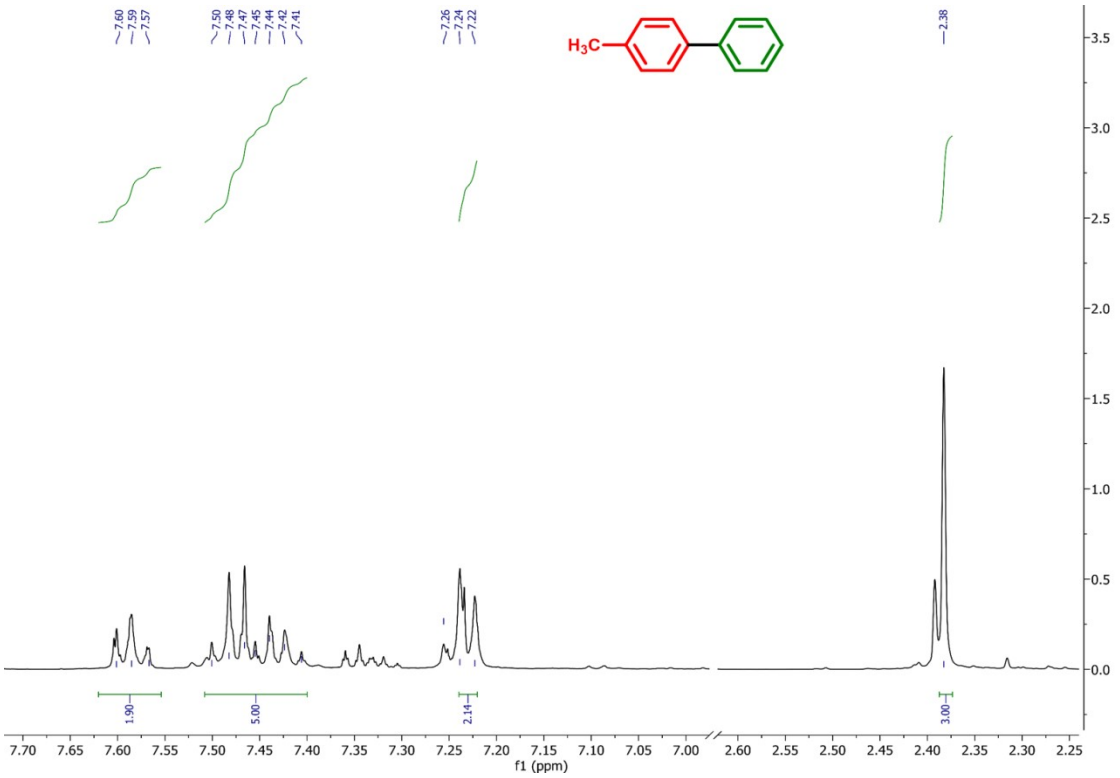


Fig S25. Zoomed view of ^{1}H NMR spectrum of 4-methyl-1,1'-biphenyl

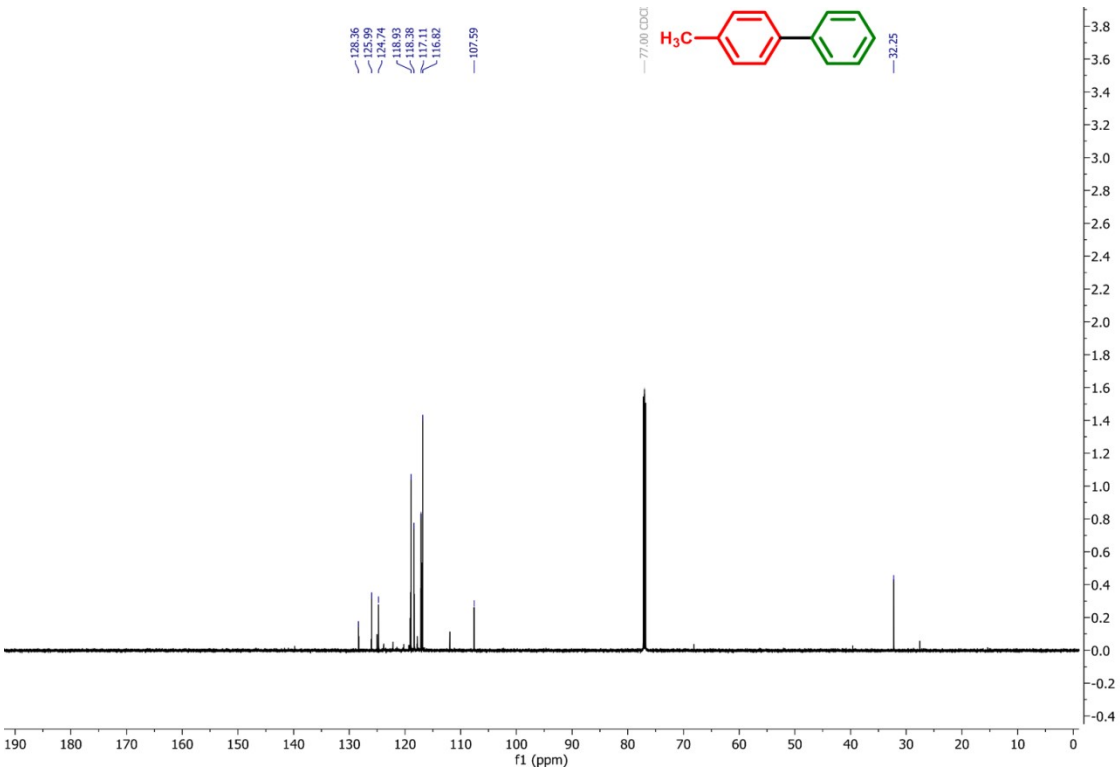


Fig S26. ^{13}C NMR spectrum of 4-methyl-1,1'-biphenyl

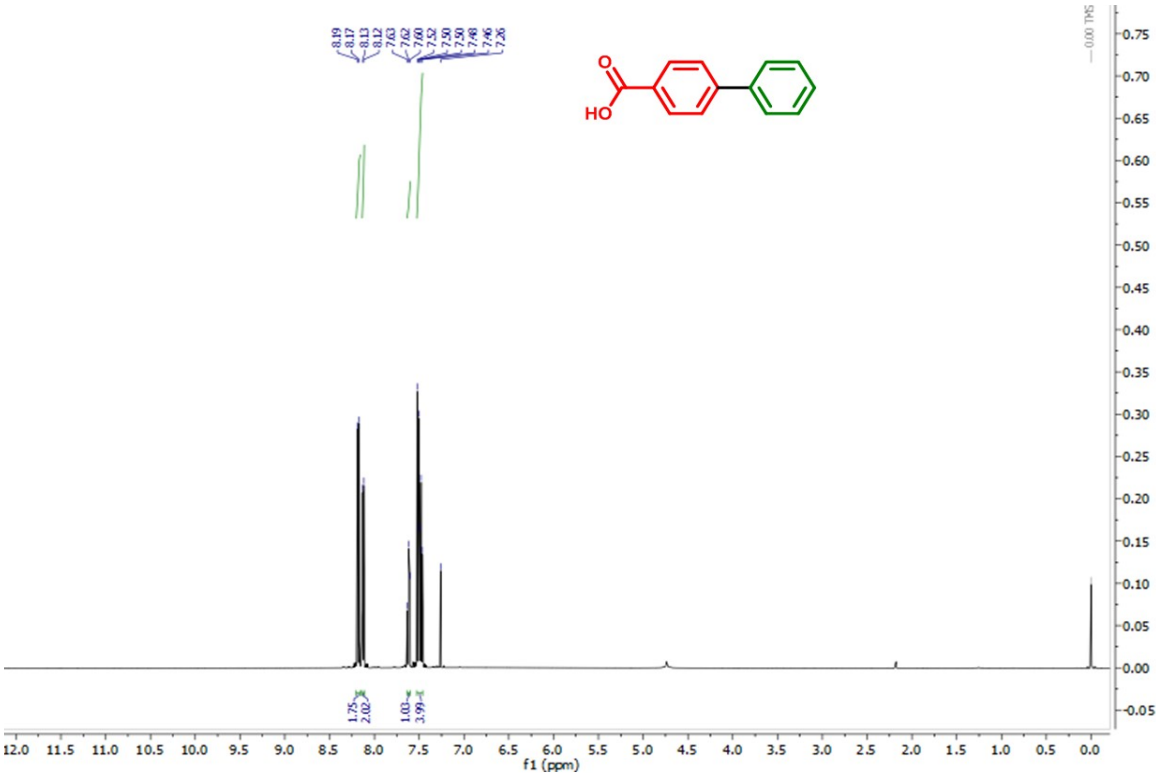


Fig S27.Full view of ^{1}H NMR spectrum of [1,1'-biphenyl]-4-carboxylic acid

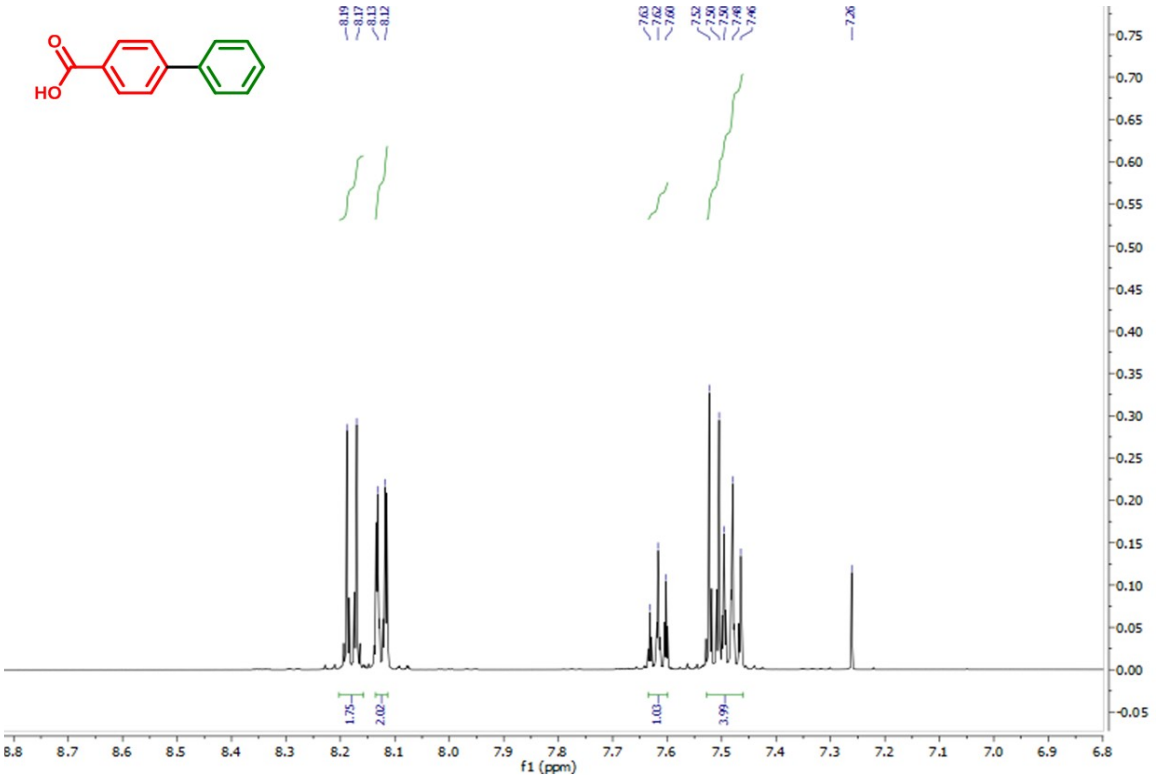


Fig S28. Zoomed view of ^{1}H NMR spectrum of [1,1'-biphenyl]-4-carboxylic acid

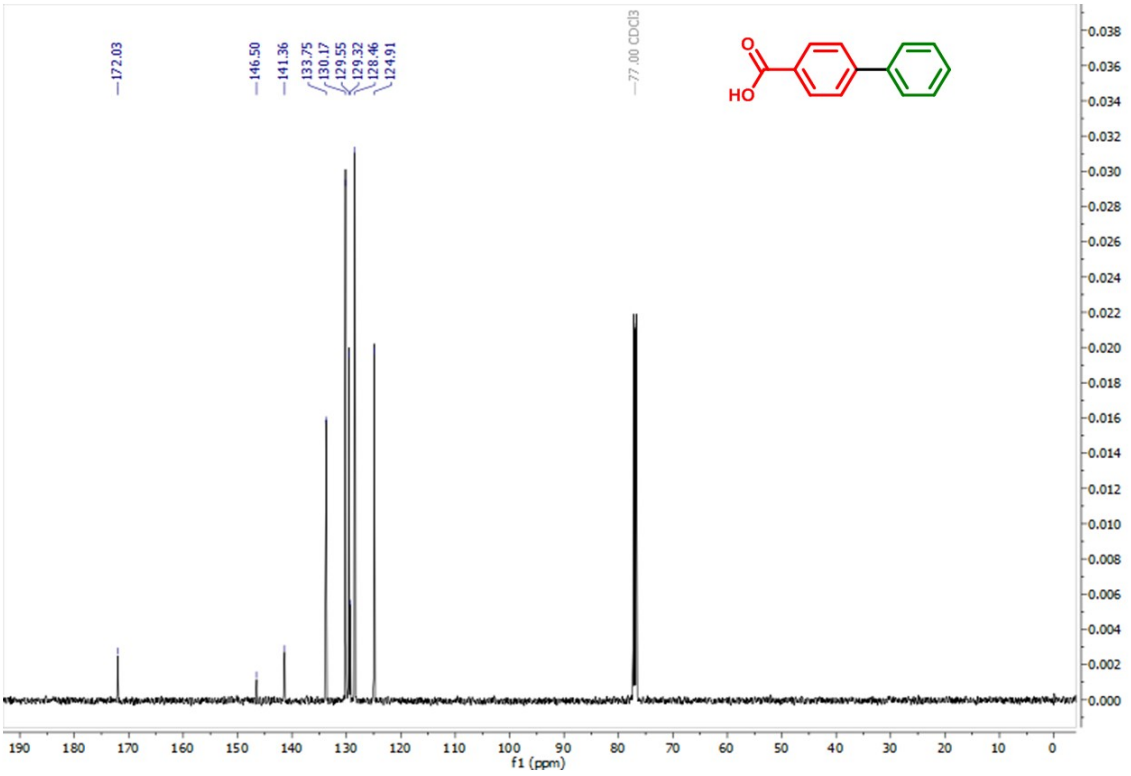


Fig S29. ^{13}C NMR spectrum of [1,1'-biphenyl]-4-carboxylic acid

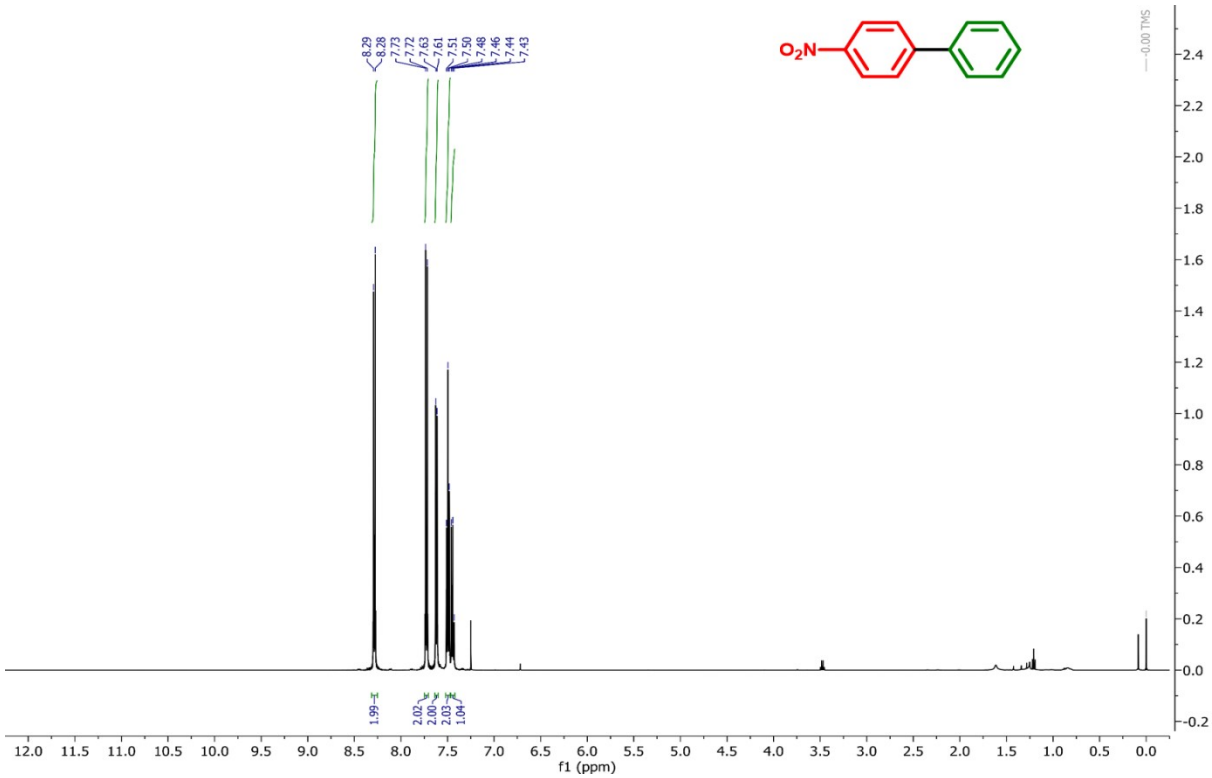


Fig S30. Full view of ^{1}H NMR spectrum of 4-nitro-1,1'-biphenyl

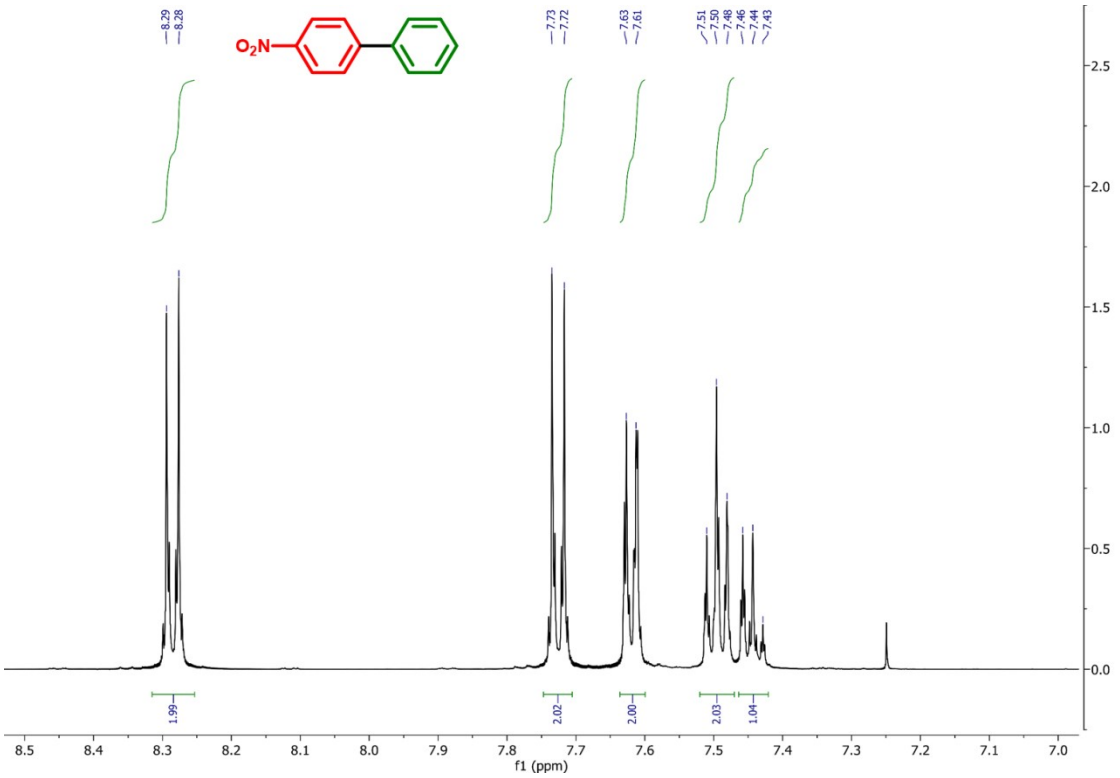


Fig S31. Zoomed view of ^{1}H NMR spectrum of 4-nitro-1,1'-biphenyl

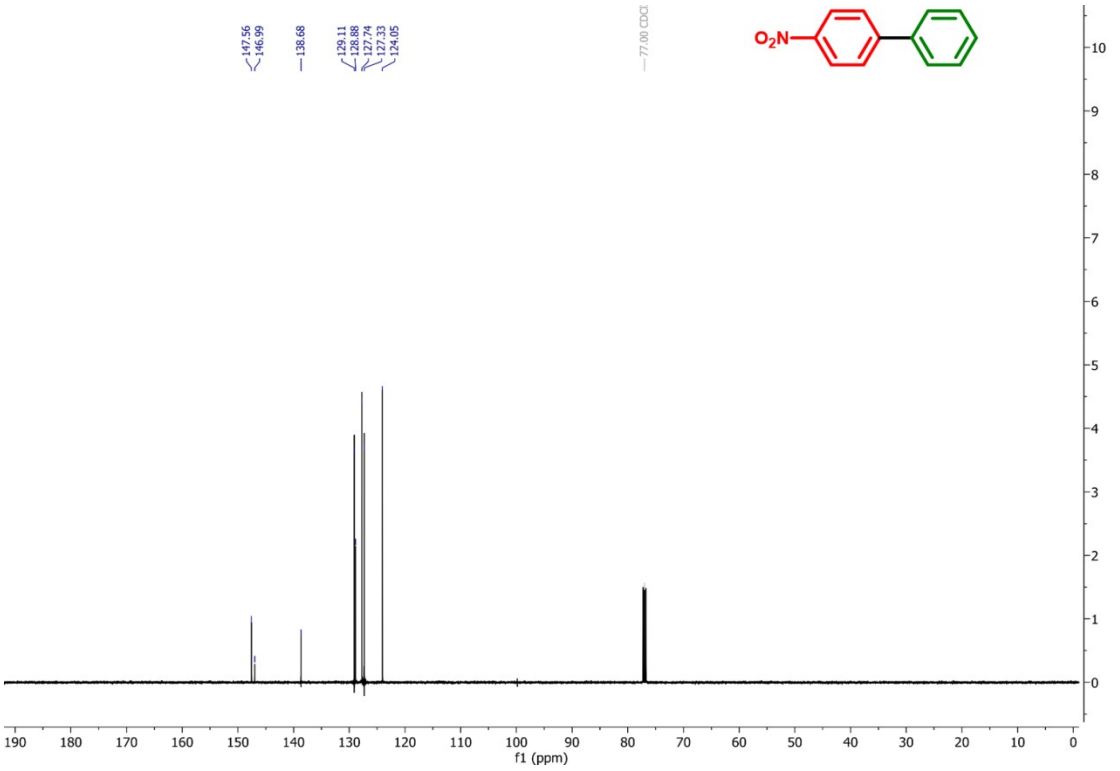


Fig S32. ^{13}C NMR spectrum of 4-nitro-1,1'-biphenyl

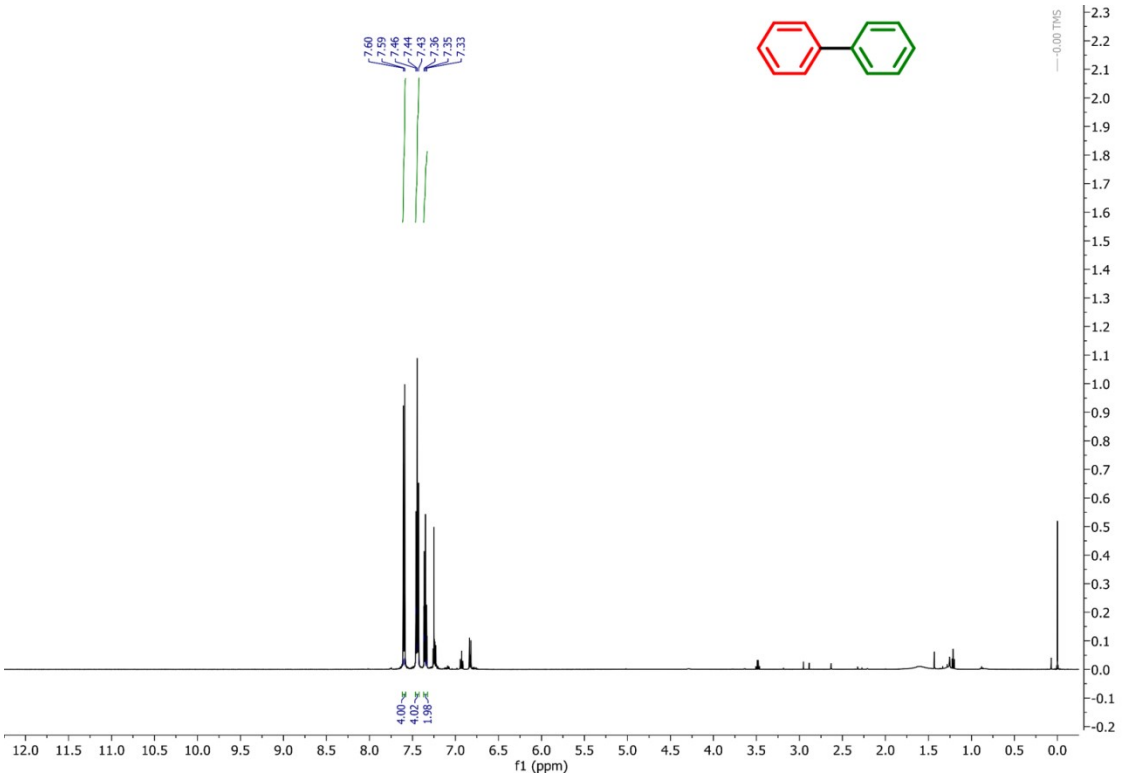


Fig S33. Full view of ^{1}H NMR spectrum of biphenyl

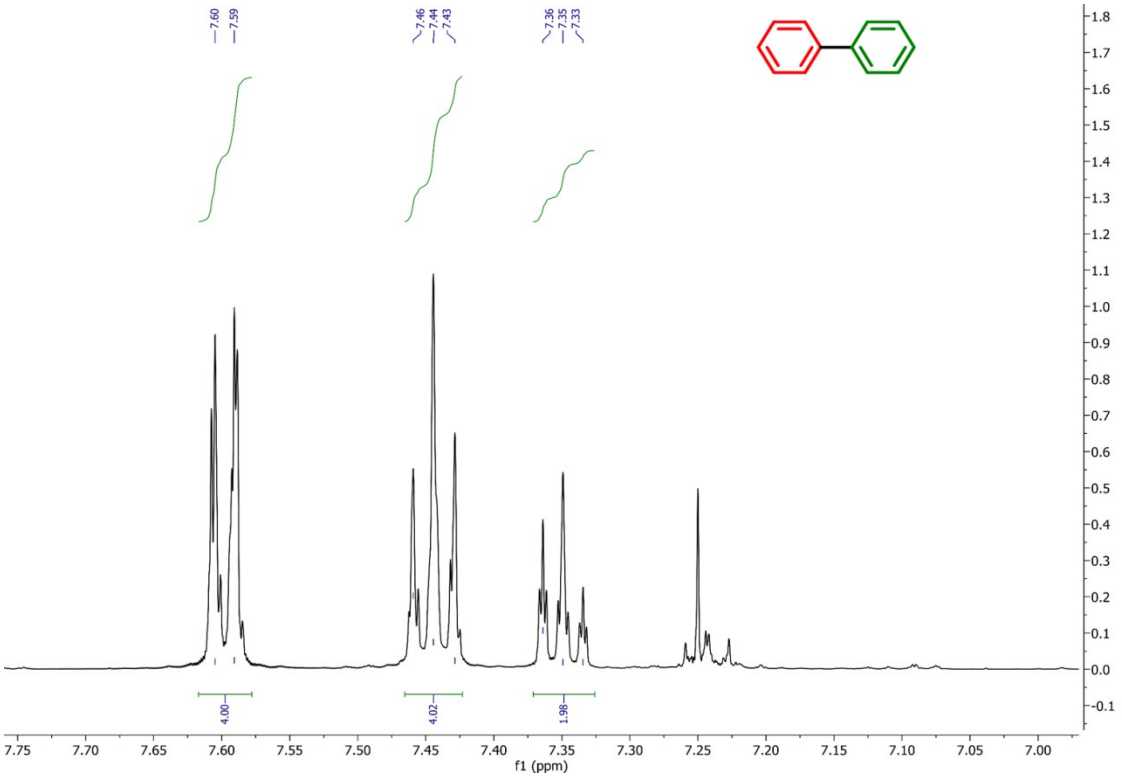


Fig S34. Zoomed view of ^{1}H NMR spectrum of Biphenyl

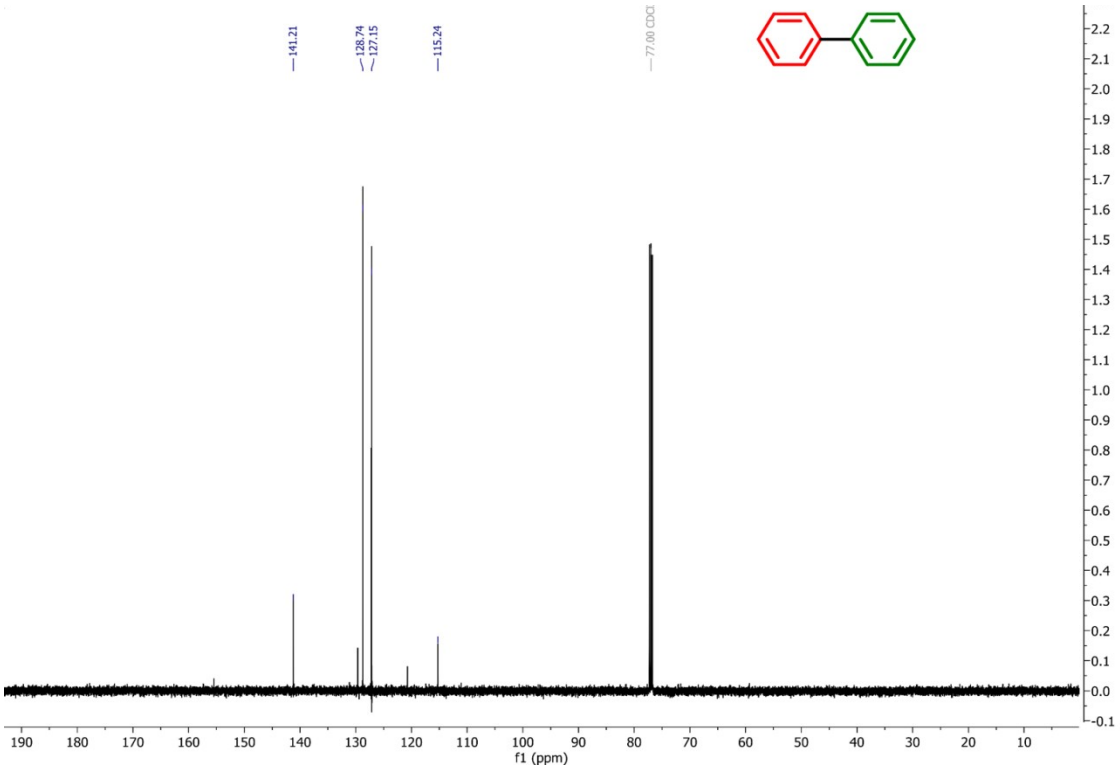


Fig S35. ^{13}C NMR spectrum of biphenyl

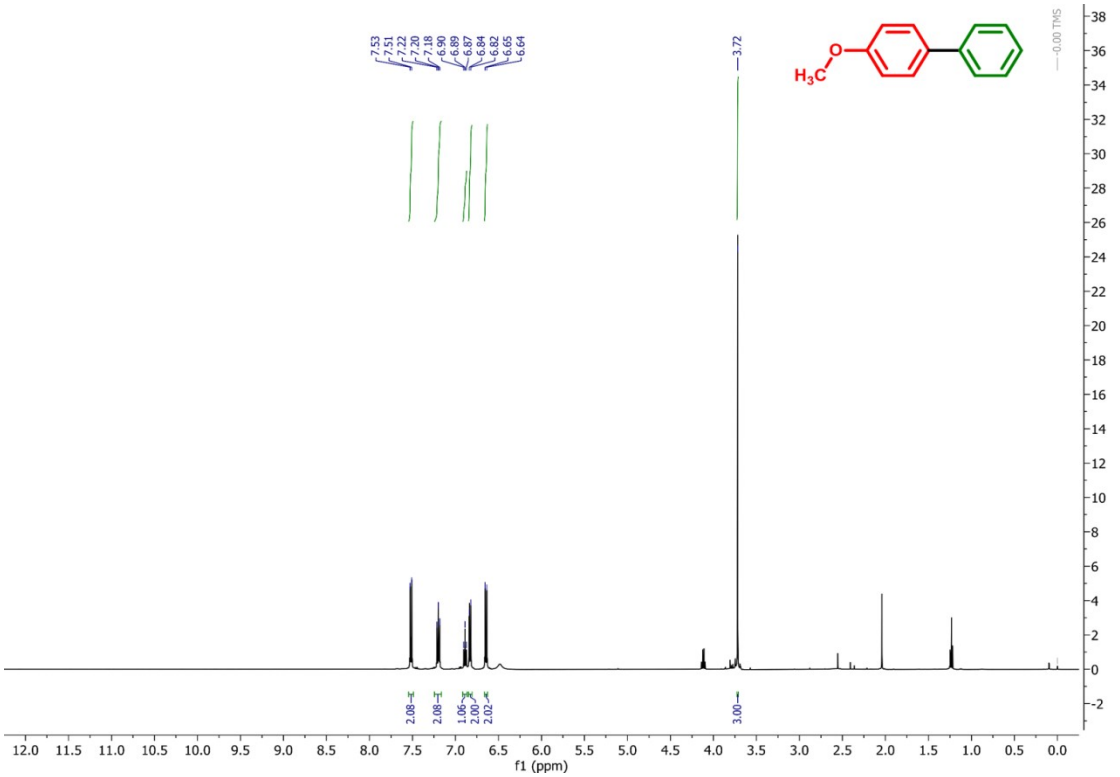


Fig S36. Full view of ^{1}H NMR spectrum of 4-methoxy-1,1'-biphenyl

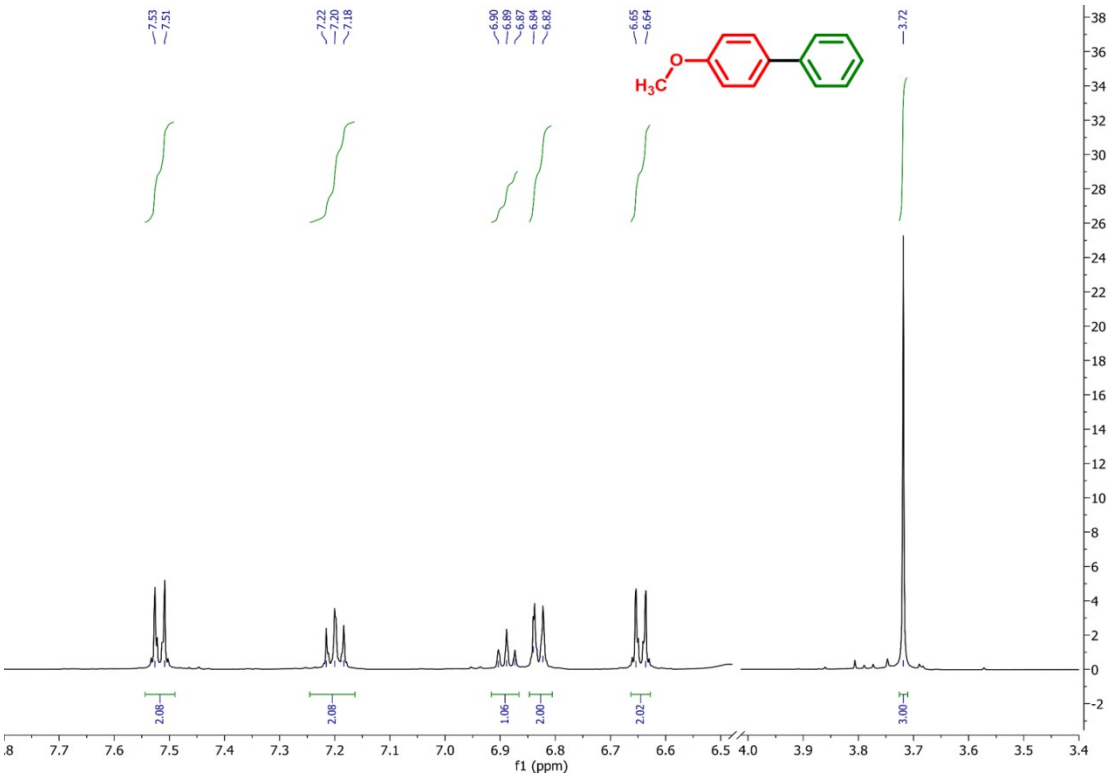


Fig S37.Zoomed view of ^{1}H NMR spectrum of 4-methoxy-1,1'-biphenyl

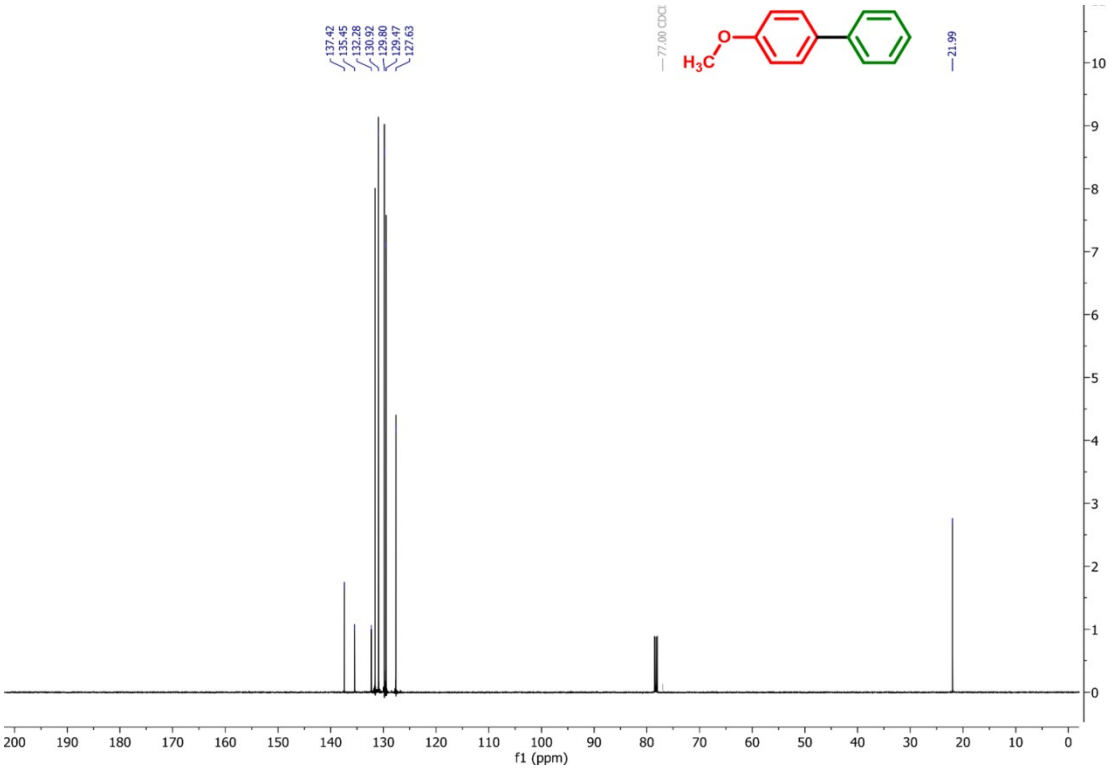


Fig S38. ^{13}C NMR spectrum of 4-methoxy-1,1'-biphenyl

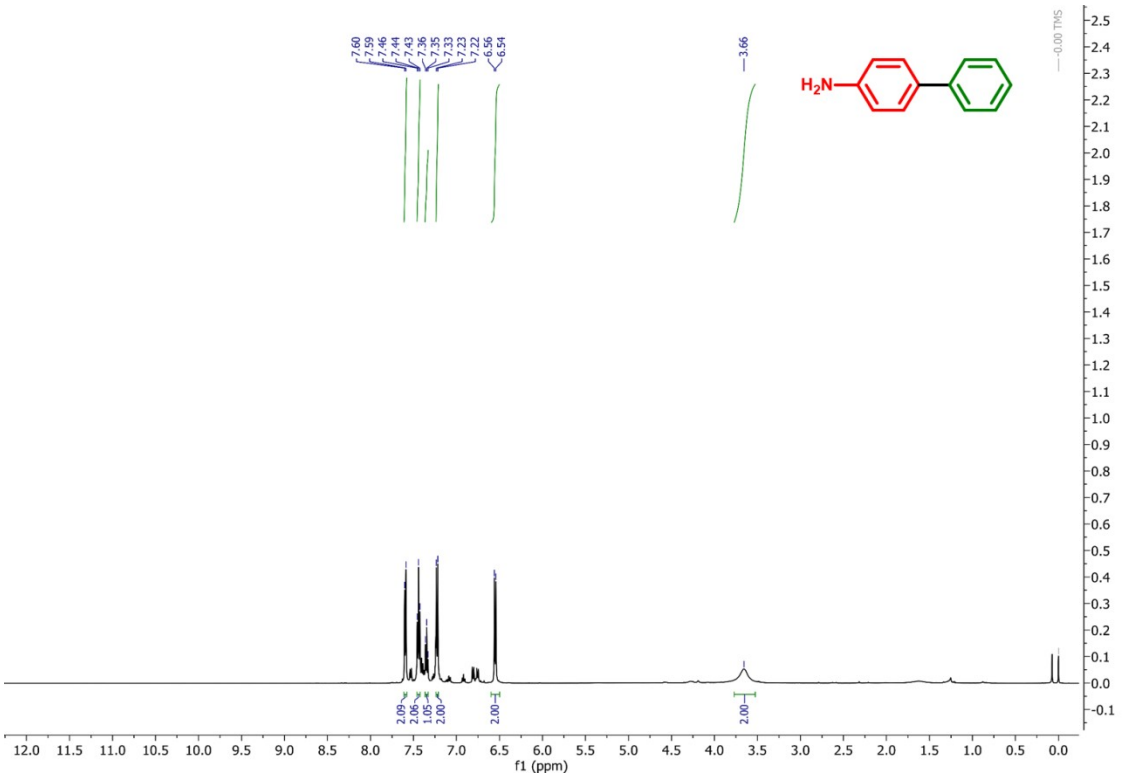


Fig S39. Full view of ^{1}H NMR spectrum of [1,1'-biphenyl]-4-amine

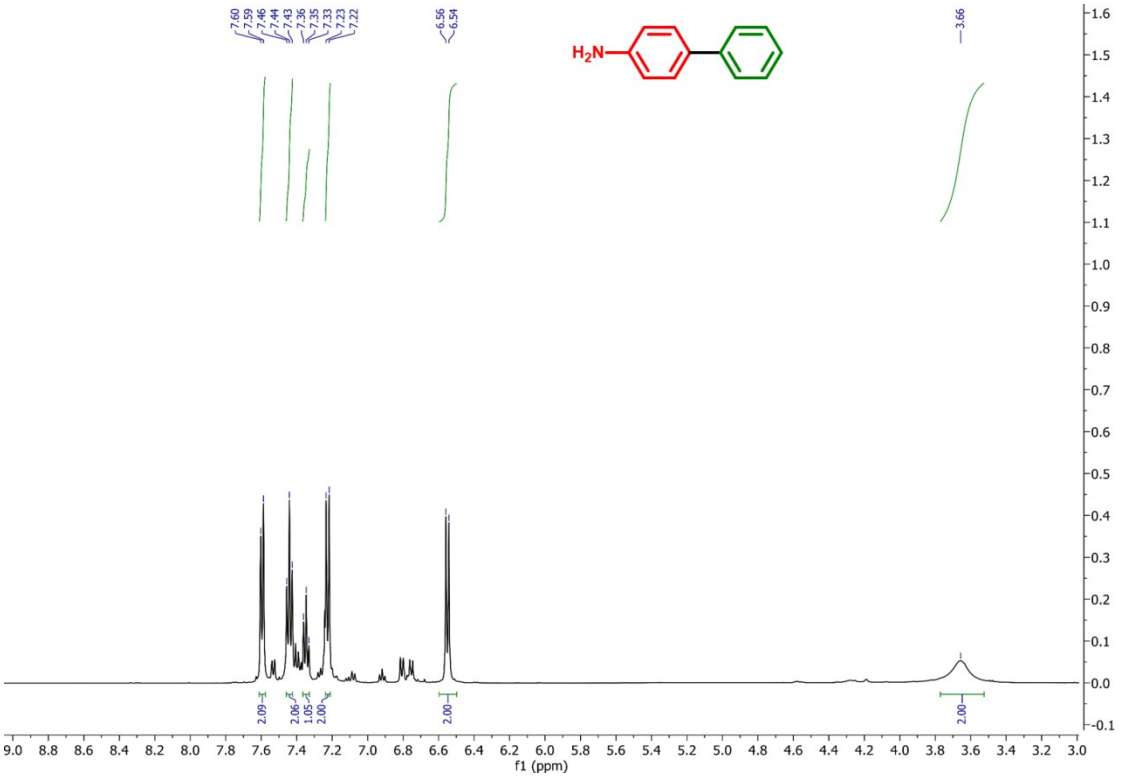


Fig S40. Zoomed view of ^{1}H NMR spectrum of [1,1'-biphenyl]-4-amine

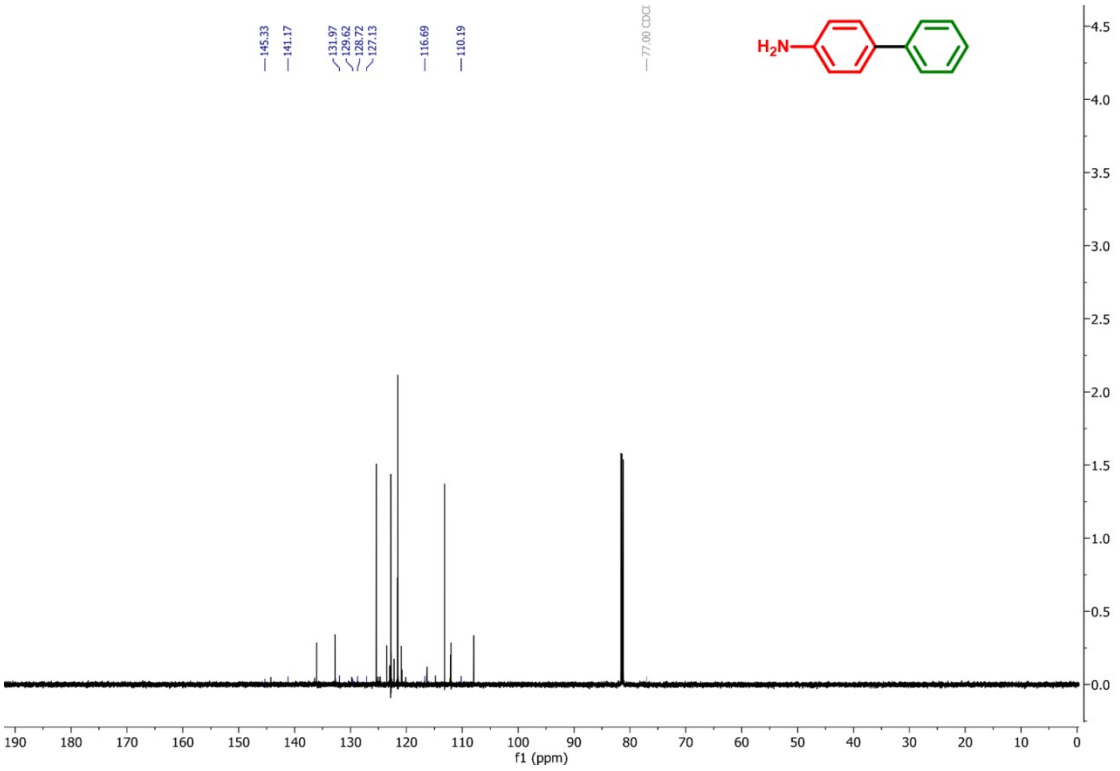


Fig S41. ^{13}C NMR spectrum of [1,1'-biphenyl]-4-amine

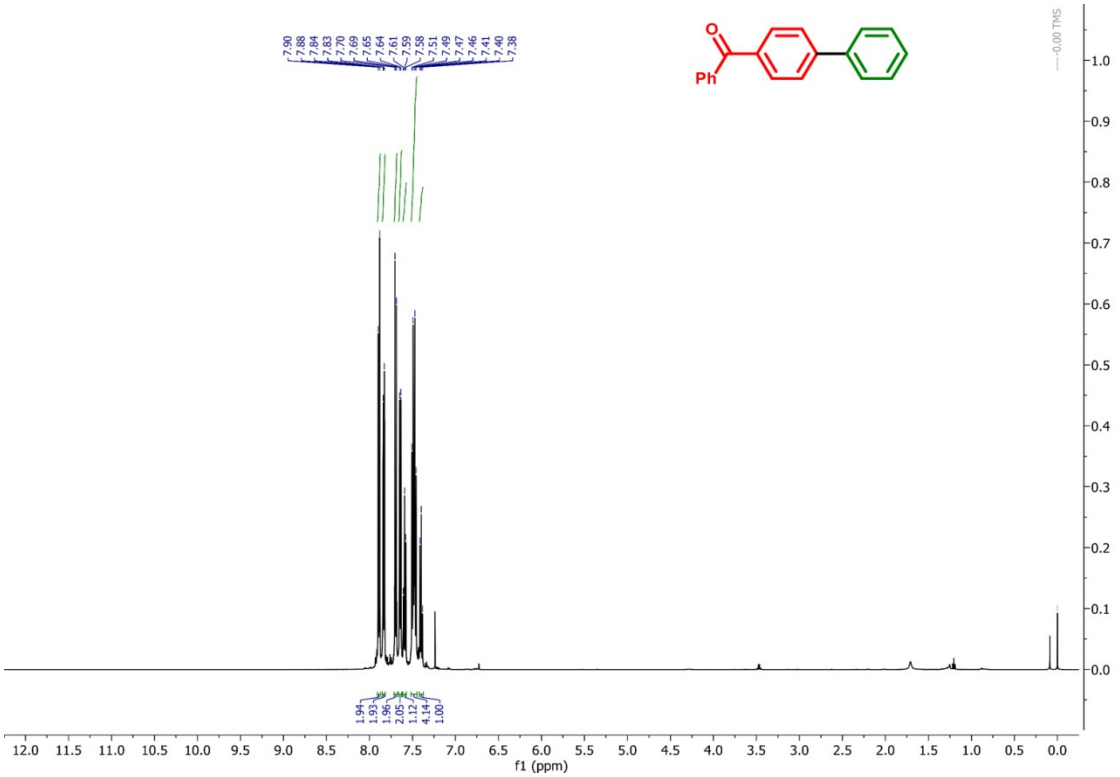


Fig S42. Full view of ^{1}H NMR spectrum of [1,1'-biphenyl]-4-yl(phenyl)methanone

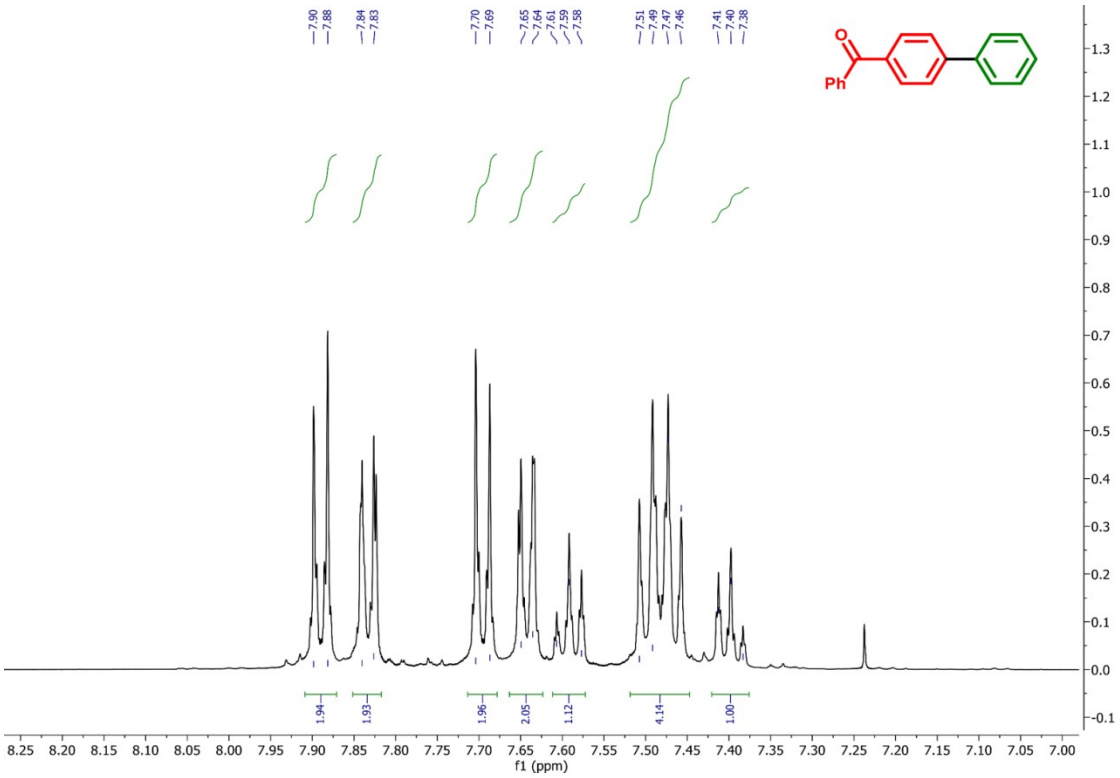


Fig S43. Zoomed view of ^{1}H NMR spectrum of [1,1'-biphenyl]-4-yl(phenyl)methanone

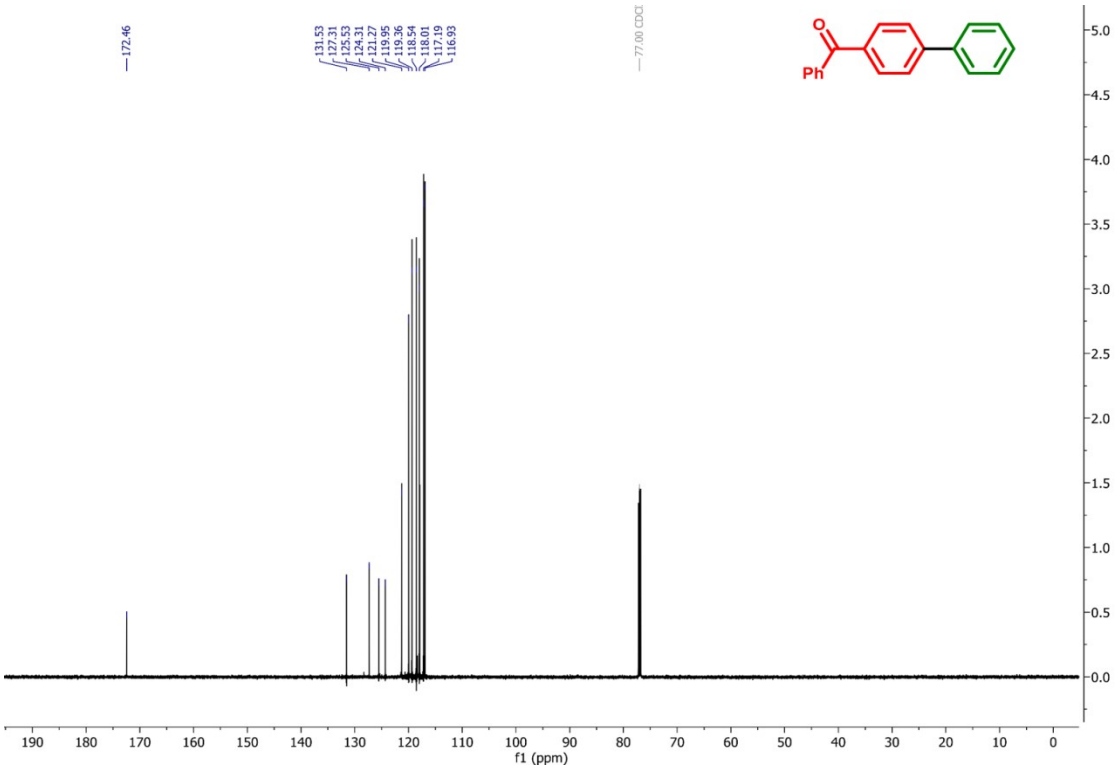
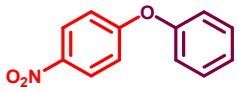


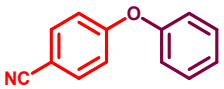
Fig S44. ^{13}C NMR spectrum of [1,1'-biphenyl]-4-yl(phenyl)methanone

[1]H-NMR data of *O*-arylated cross-coupled products (16-19) of C−O coupling reactions

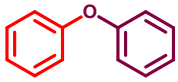
---

Compound 16: 1-Nitro-4-phenoxy benzene :[1] 

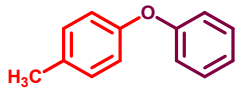
[1]H NMR (500 MHz, $CDCl_3$, 25°C vs TMS), δ(ppm): 8.11-8.14 (d, 2H), 7.34-7.40 (t, 2H), 7.16-7.21 (t, 1H), 7.01-7.03 (d, 2H), 6.92-6.95 (d, 2H). [13]C-NMR (125 MHz, CDCl3): δ (ppm) 146.07, 139.09, 129.42, 119.62, 116.12, 115.71, 111.81, 109.01

Compound 17: 4-Phenoxybenzonitrile :[1] 

[1]H NMR (500 MHz, $CDCl_3$), δ(ppm): 7.55 (d, 2H), 7.43 (t, 2H), 7.20 (t, 1H), 7.00 (d, 2H), 6.95 (d, 2H).[13]C-NMR (125 MHz, CDCl3): δ (ppm) 168.32, 154.74, 134.12, 131.88, 130.22, 128.95, 125.14, 120.41, 117.86

Compound 18: Diphenyl ether :[1] 

[1]H NMR (500 MHz, $CDCl_3$), δ(ppm): 7.37 (d, 4H), 7.25 (t, 4H), 6.93 (t, 2H).[13]C-NMR (125 MHz, CDCl3): δ (ppm) 134.20, 129.68, 128.57, 126.39

Compound 19: 1-Methyl-4-phenoxybenzene :[1] 

[1]H NMR (500 MHz, $CDCl_3$, 25°C vs TMS), δ(ppm): 7.46-7.48 (m, 2H), 7.23-7.26 (t, 2H), 7.06-7.07 (m, 1H), 6.98-7.04 (m, 2H), 6.89-6.91 (m, 2H), 2.26 (s, 3H).[13]C-NMR (125 MHz, CDCl3): δ (ppm) 134.14, 134.18, 131.01, 130.31, 129.64, 128.53, 128.20, 126.36, 20.71

NMR Spectra of C-O cross coupling products.

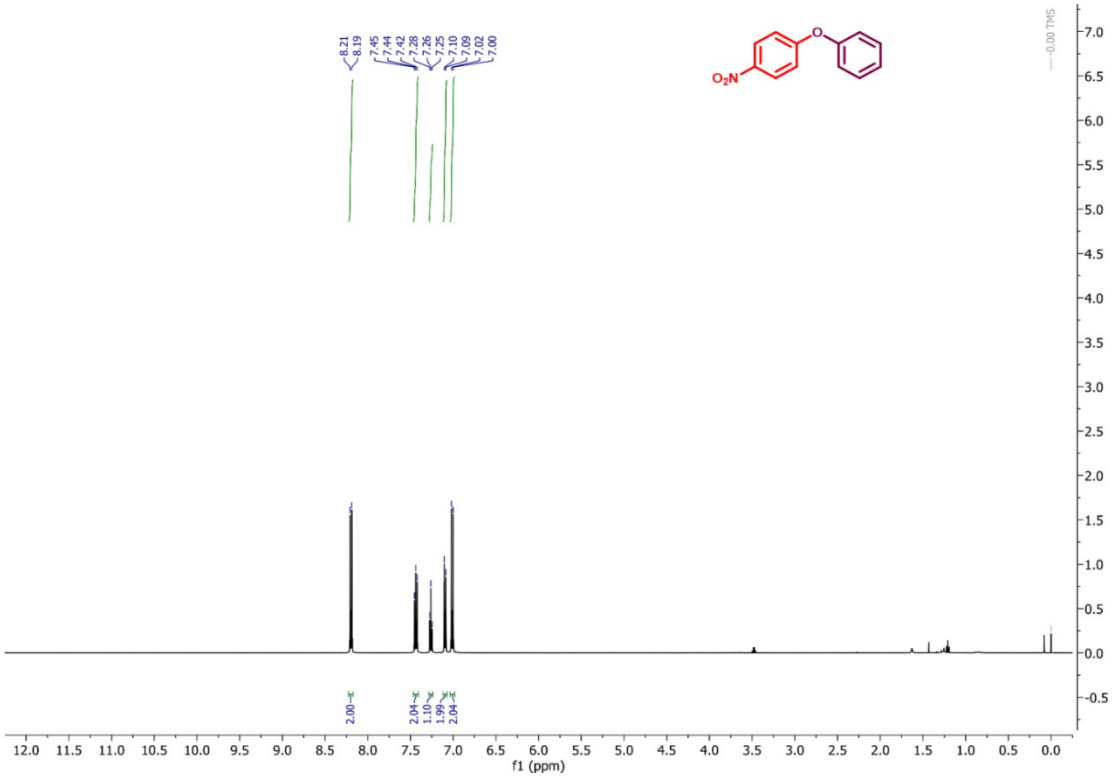


Fig. S45. Full view of ^{1}H NMR spectrum of 1-nitro-4-phenoxy benzene

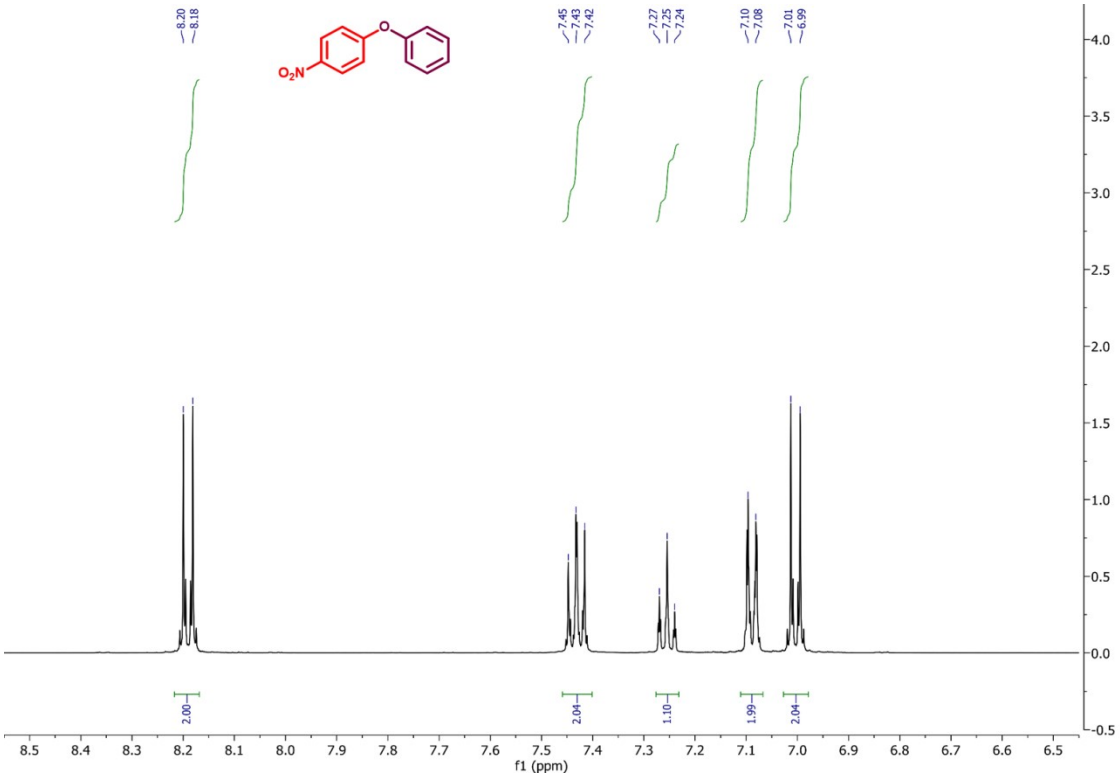


Fig. S46. Zoomed view of ^{1}H NMR spectrum of 1-nitro-4-phenoxy benzene

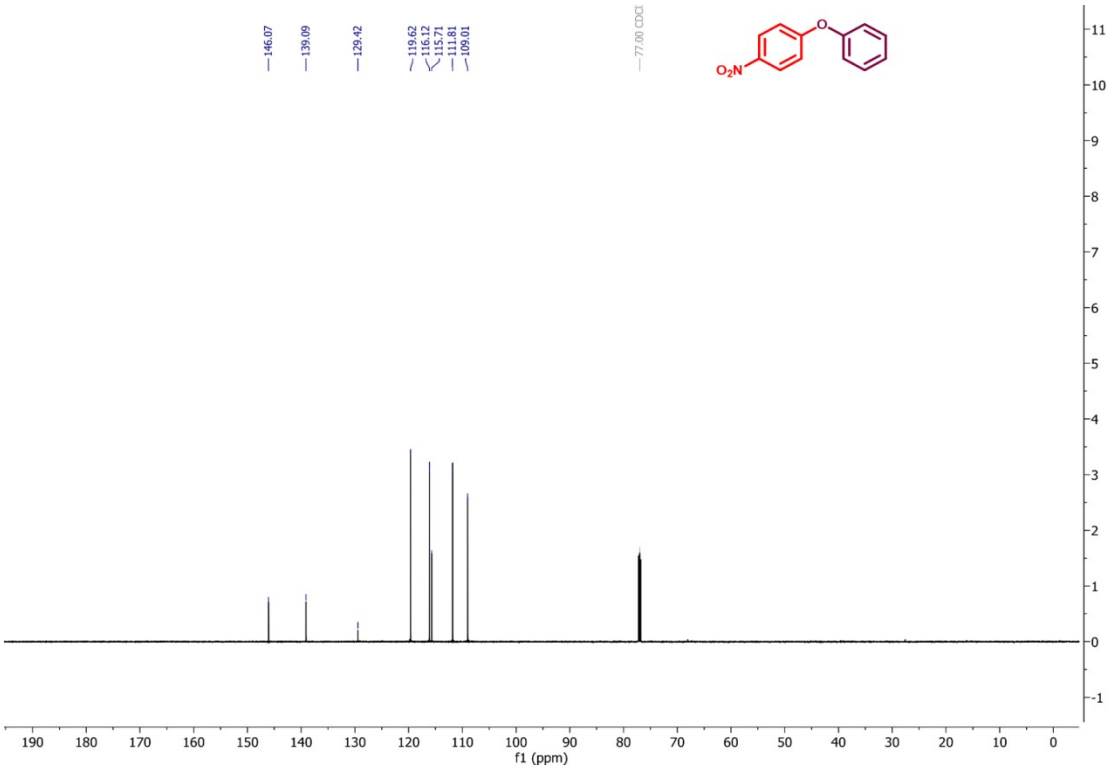


Fig. S47.[13]C NMR spectrum of 1-nitro-4-phenoxy benzene

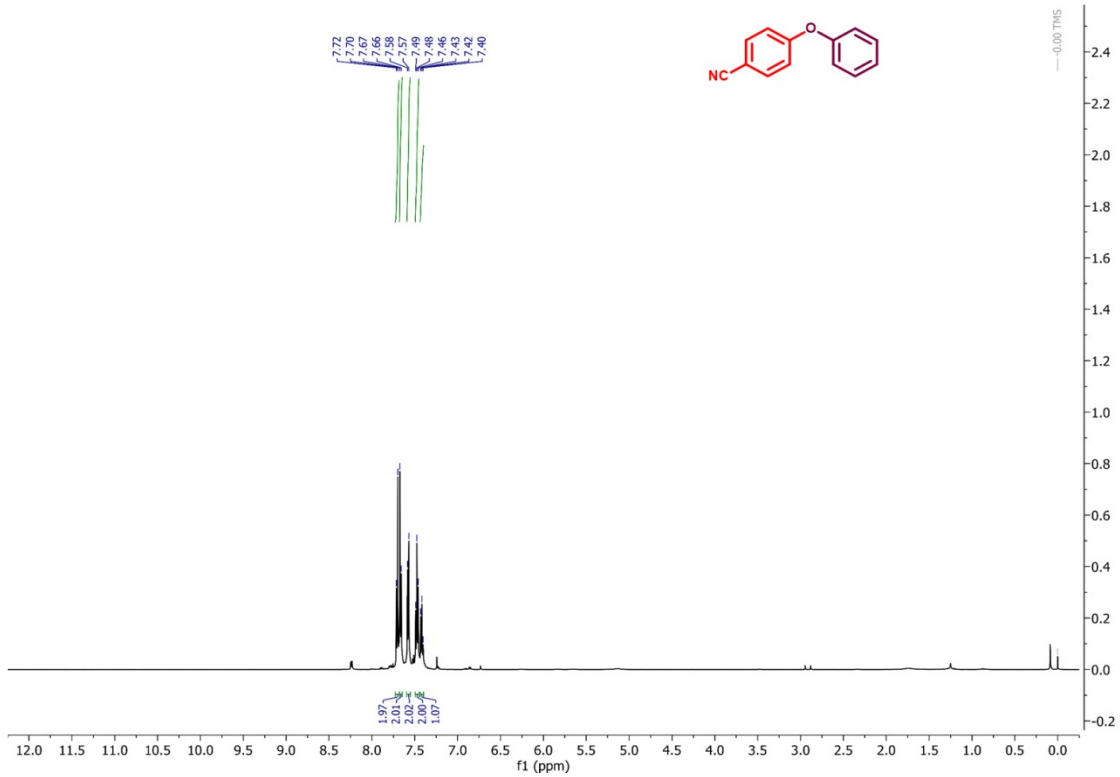


Fig. S48. Full view of [1]H NMR spectrum of 4-phenoxybenzonitrile

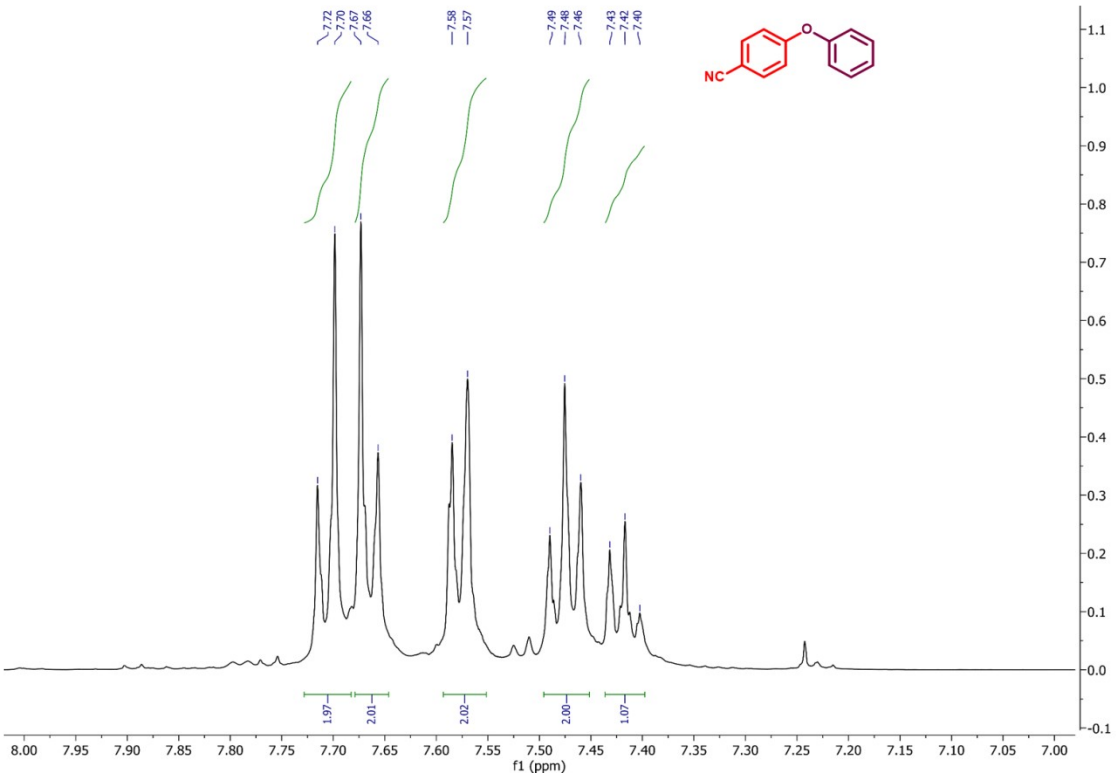


Fig. S49. Zoomed view of ^{1}H NMR spectrum of 4-phenoxybenzonitrile

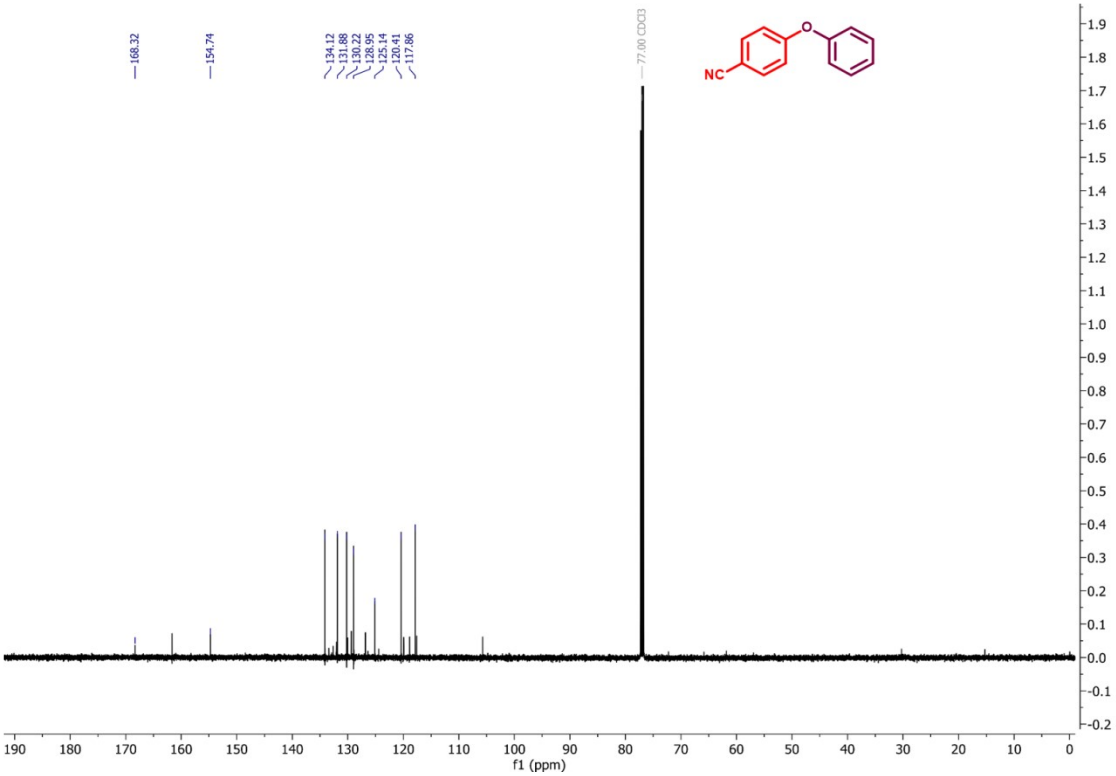


Fig. S50. ^{13}C NMR spectrum of4-phenoxybenzonitrile

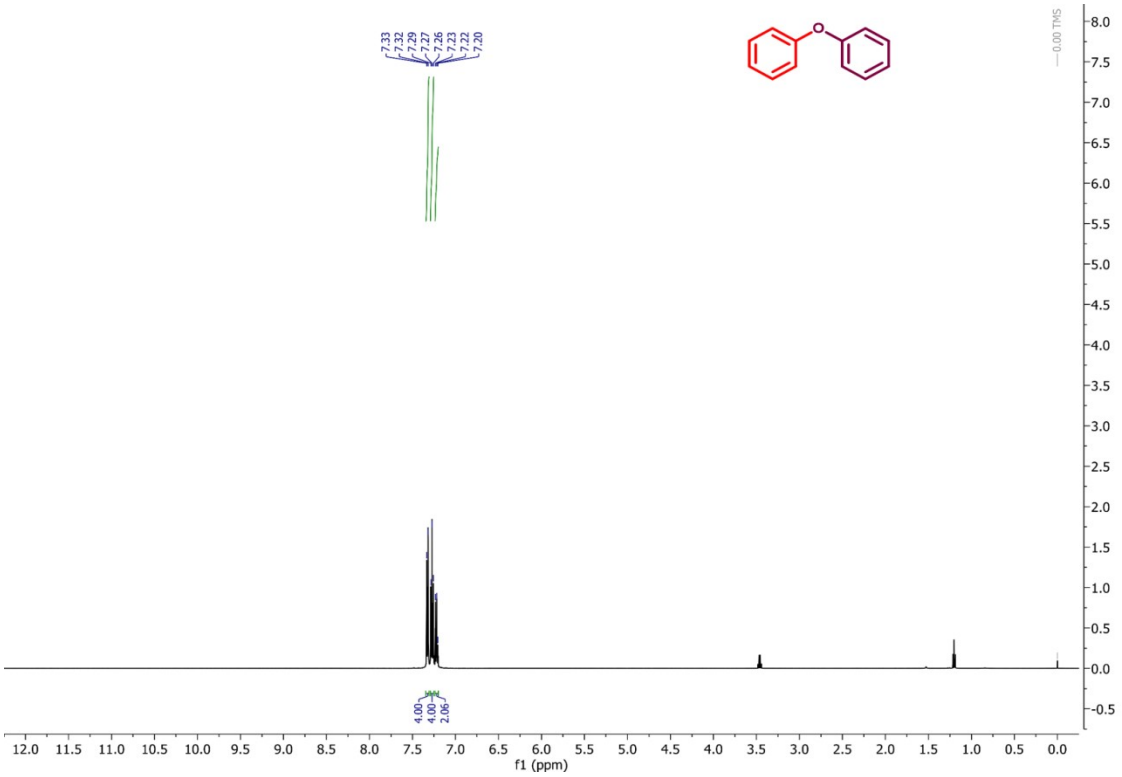


Fig. S51. Full view of ^{1}H NMR spectrum of diphenyl ether

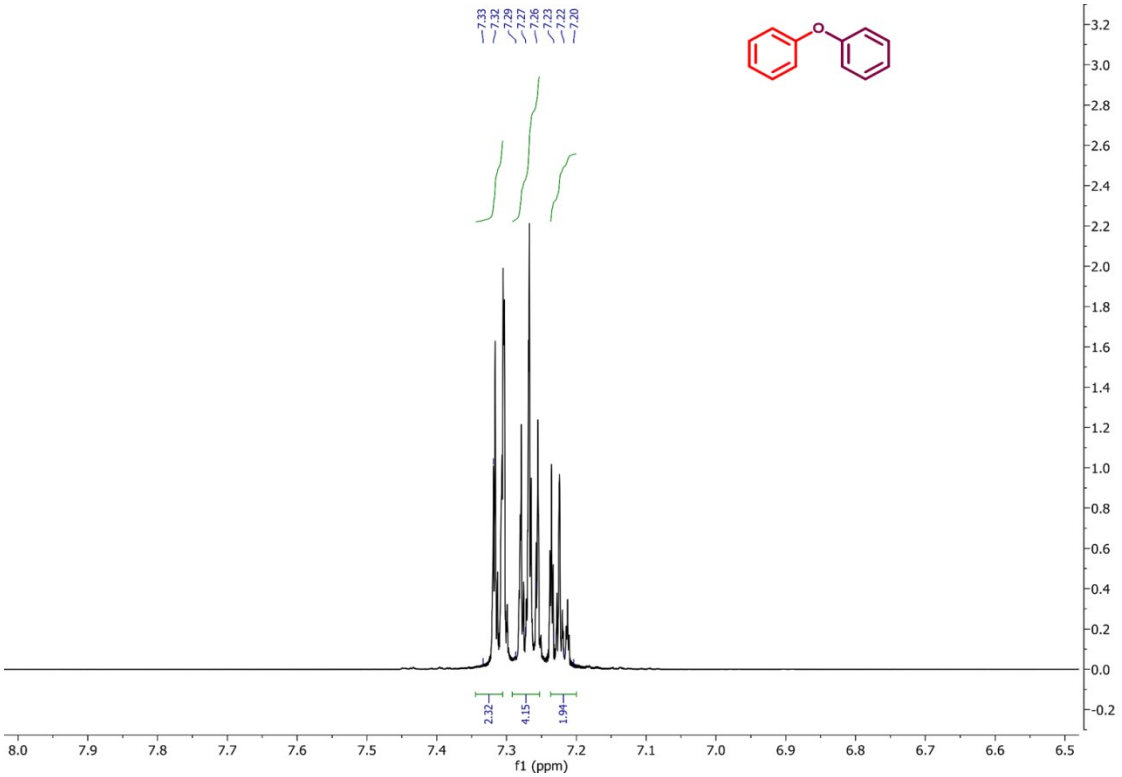


Fig. S52. Zoomed view of ^{1}H NMR spectrum of diphenyl ether

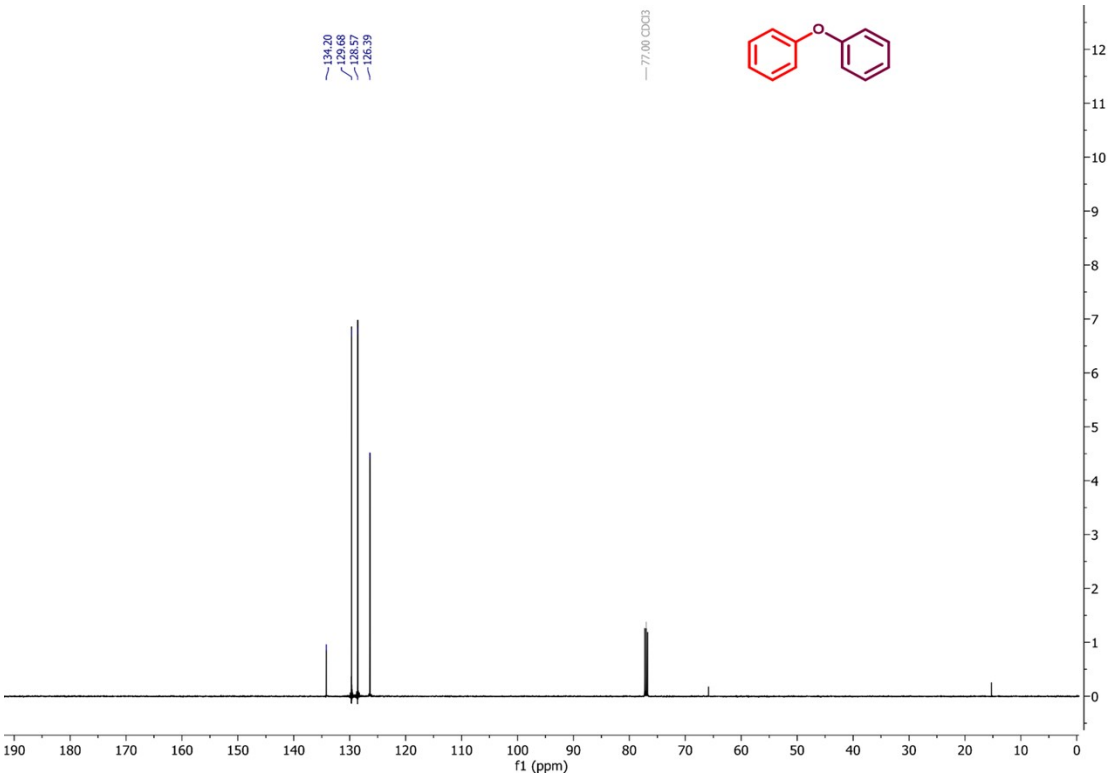


Fig. S53. ^{13}C NMR spectrum of diphenyl ether

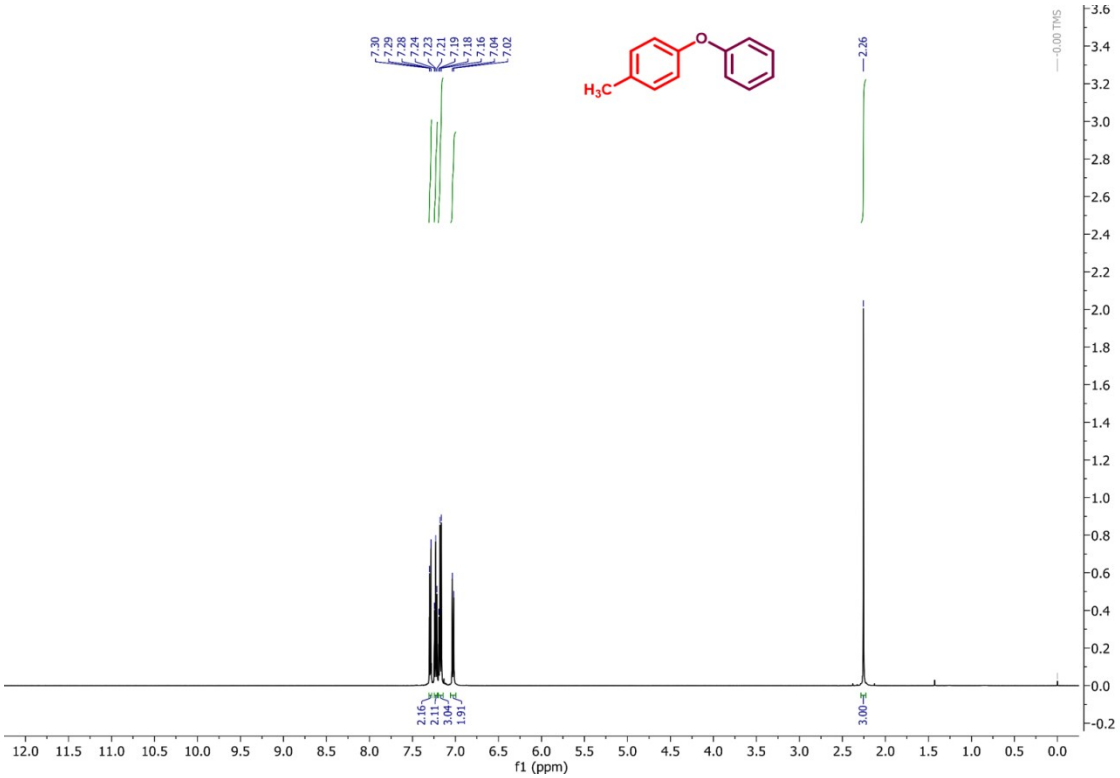


Fig. S54. Full view of ^{1}H NMR spectrum of 1-methyl-4-phenoxybenzene

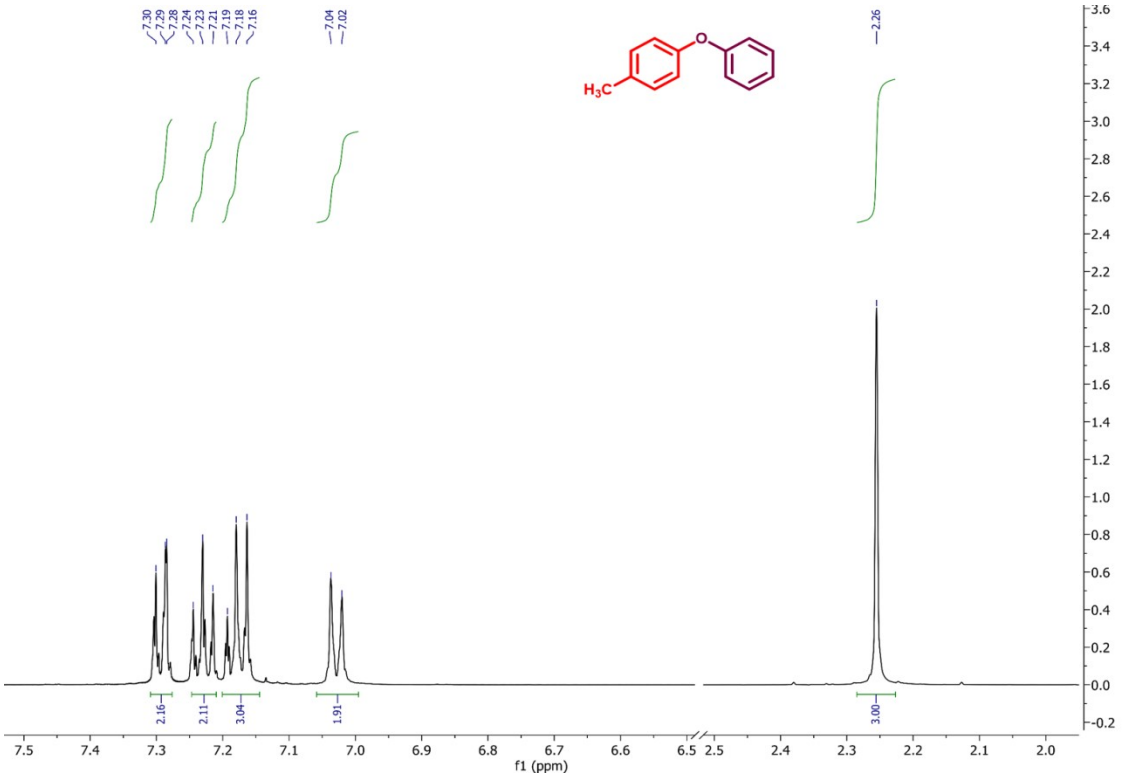


Fig. S55. Zoomed view of ^{1}H NMR spectrum of 1-methyl-4-phenoxybenzene

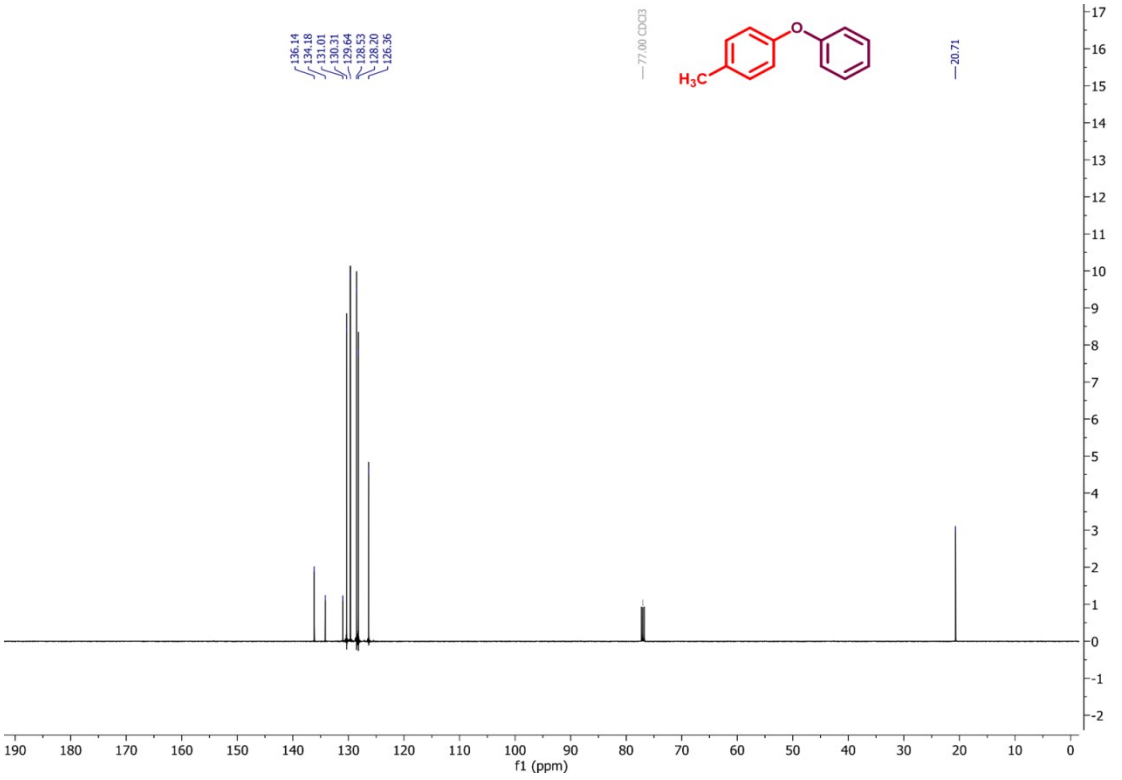


Fig. S56. ^{13}C NMR spectrum of 1-methyl-4-phenoxybenzene

References:

1. A. K. Sharma, H. Joshi, and A. K. Singh, *RSC Adv.*, 2020, **10**, 6452-6459.

2. P. Oswal, A. Arora, J. Kaushal, G. K. Rao, S. Kumar, A. K. Singh, and A. Kumar, *RSC Adv.*, 2019, **9**, 22313-22319.