

Supplementary Information

Efficient preparation of hybrid biofuels from biomass-derived 5-(acetoxymethyl)furfural and petroleum-derived aromatic hydrocarbons

Abhishek Kumar Yadav^a, Navya Subray Bhat^a, Sonal Kaushik^a, Asiful H. Seikh^b, and Saikat Dutta^{a}*

^aDepartment of Chemistry, National Institute of Technology Karnataka (NITK), Surathkal, Mangalore-575025, Karnataka, India. E-mail: sdutta@nitk.edu.in

^bMechanical Engineering Department, College of Engineering, King Saud University, Riyadh-11421, Saudi Arabia.

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Characterization of 5-(arylmethyl)furfurals (AMFFs)

5-(Benzenylmethyl)furfural (**1a**): light-yellow liquid (0.328 g, 59%), ¹H-NMR (CDCl₃, 400 MHz) δ (ppm): 4.06 (s, 2H), 6.18 (d, 1H, *J* = 3.2 Hz), 7.15 (d, 1H, *J* = 3.6 Hz), 7.24-7.33 (m, 5H), 9.54 (s, 1H). ¹³C-NMR (CDCl₃, 100 MHz) δ (ppm): 35.1, 110.0, 123.4, 127.3, 129.0, 129.1, 136.4, 152.5, 162.3, 177.5. FTIR (cm⁻¹): 3123, 3031, 2925, 2853, 1678, 1582, 1516, 1025.

5-(Toluenylmethyl)furfural (**1b**): light-yellow liquid (0.454 g, 76%), ¹H-NMR (CDCl₃, 400 MHz) δ (ppm): 2.29 (s, 3H), 2.33 (s, 3H), 4.02 (s, 2H), 4.05 (s, 2H), 6.05 (d, 1H, *J* = 3.6 Hz), 6.16 (d, 1H, *J* = 3.6 Hz), 7.14-7.19 (m, 10H), 9.53 (s, 2H). ¹³C-NMR (CDCl₃, 100 MHz) δ (ppm): 19.7, 21.3, 32.9, 34.7, 109.8, 109.9, 110.0, 123.5, 126.6, 127.7, 129.0, 129.7, 130.0, 130.8, 133.3, 134.6, 136.7, 136.9, 152.4, 162.1, 162.7, 177.4. FTIR (cm⁻¹): 3122, 3047, 2921, 2820, 1677, 1581, 1514, 1022.

5-(*p*-Xylenylmethyl)furfural (**1c**): light-yellow liquid (0.522 g, 82%), ¹H-NMR (CDCl₃, 400 MHz) δ (ppm): 2.24 (s, 3H), 2.30 (s, 3H), 4.01 (s, 2H), 6.05 (d, 1H, *J* = 3.6 Hz), 6.98-7.08 (m, 3H), 7.14 (d, 1H, *J* = 3.6 Hz), 9.54 (s, 1H). ¹³C-NMR (CDCl₃, 100 MHz) δ (ppm): 19.2, 21.1, 32.9, 109.9, 123.4, 128.3, 130.7, 130.8, 133.5, 134.4, 136.1, 152.3, 162.3, 177.4. FTIR (cm⁻¹): 3116, 3042, 2819, 1677, 1580, 1512, 1022.

5-(*m*-xylenylmethyl)furfural (**1d**): light-yellow liquid (0.492 g, 77%), ¹H-NMR (CDCl₃, 400 MHz) δ (ppm): 2.25 (s, 9H), 2.31 (s, 15H), 4.00 (s, 6H), 4.07 (s, 2H), 5.91 (d, 1H, *J* = 3.6 Hz), 6.05 (d, 3H, *J* = 3.6 Hz), 6.97-7.14 (m, 18H), 9.51 (s, 4H). ¹³C-NMR (CDCl₃, 100 MHz) δ (ppm): 19.3, 20.0, 20.9, 28.9, 32.2, 109.2, 109.6, 123.2, 127.0, 127.1, 128.4, 129.8, 131.3, 131.3, 132.7, 136.2, 136.9, 152.1, 152.1, 161.5, 162.1, 177.0, 177.1. FTIR (cm⁻¹): 3004, 2920, 2821, 1674, 1580, 1279, 1195, 1020.

5-(*o*-Xylenylmethyl)furfural (**1e**): light-yellow liquid (0.497 g, 78%), ¹H-NMR (CDCl₃, 400 MHz) δ (ppm): 2.18 (s, 3H), 2.24 (s, 12H), 2.30 (s, 3H), 3.99 (s, 4H), 4.07 (s, 2H), 6.03 (d, 1H, *J* = 3.6 Hz), 6.17 (d, 2H, *J* = 3.2 Hz), 7.00-7.16 (m, 12H), 9.53 (s, 3H). ¹³C-NMR (CDCl₃, 100 MHz) δ (ppm): 19.6, 20.0, 20.9, 33.6, 34.7, 109.8, 109.9, 123.4, 126.0, 126.5, 128.1, 129.4, 130.2, 130.4, 133.7, 134.5, 135.3, 135.6, 137.2, 137.6, 152.3, 152.3, 162.5, 162.8, 177.5. FTIR (cm⁻¹): 3116, 3003, 2947, 2819, 1677, 1581, 1511, 1196, 1023.

5-(Mesitylmethyl)furfural (**1f**): light-yellow liquid (0.551 g, 81%), $^1\text{H-NMR}$ (CDCl_3 , 300 MHz) δ (ppm): 2.27 (s, 9H), 4.04 (s, 2H), 5.92 (s, 1H), 6.89 (s, 2H), 7.11 (s, 1H), 9.51 (s, 1H). $^{13}\text{C-NMR}$ (CDCl_3 , 75 MHz) δ (ppm): 20.1, 21.0, 28.8, 109.3, 123.5, 129.3, 129.8, 136.8, 136.9, 152.3, 162.1, 177.2. FTIR (cm^{-1}): 3117, 3013, 2818, 1680, 1581, 1513, 1197, 1021.

5-(Cumenylmethyl)furfural (**1g**): light-yellow liquid (0.538 g, 79%), $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ (ppm): 1.18 (d, 6H, $J = 6.8$ Hz), 1.223 (d, 6H, $J = 7.2$ Hz), 2.85-2.92 (m, 1H), 3.06-3.13 (m, 1H), 4.01 (s, 2H), 4.10 (s, 2H), 6.02 (d, 1H, $J = 3.6$ Hz), 6.18 (d, 1H, $J = 3.6$ Hz), 7.12-7.17 (m, 8H), 7.24-7.33 (m, 2H), 9.51 (s, 1H), 9.52 (s, 1H). $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ (ppm): 23.8, 24.0, 29.2, 32.2, 33.8, 34.5, 109.7, 109.8, 123.2, 125.1, 125.8, 126.1, 126.3, 126.8, 127.2, 127.9, 128.8, 128.8, 130.3, 132.8, 133.5, 147.1, 147.7, 152.1, 152.2, 162.4, 162.6, 177.2. FTIR (cm^{-1}): 3116, 3020, 2830, 1679, 1514, 1195, 1022. HRMS (ESI) calculated for $\text{C}_{15}\text{H}_{16}\text{O}_2\text{Na}$ $[\text{M}+\text{Na}]^+$ 251.104, found 251.094.

5-(Ethylbezenylmethyl)furfural (**1h**): light-yellow liquid (0.479 g, 75%), $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ (ppm): 1.21 (t, 3H, $J = 7.6$ Hz), 1.26 (t, 5H, $J = 7.6$ Hz), 2.63-2.69 (m, 5H), 4.05 (s, 2H), 4.11 (s, 2H), 6.07 (d, 1H, $J = 3.6$ Hz), 6.20 (d, 1H, $J = 3.6$ Hz), 7.19-7.20 (m, 10H), 7.25-7.28 (m, 2H), 9.56 (s, 2H). $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ (ppm): 15.2, 15.8, 25.9, 28.7, 32.2, 34.7, 109.9, 110.0, 123.3, 126.5, 127.8, 128.5, 129.0, 130.3, 133.5, 133.8, 142.6, 143.3, 152.3, 152.4, 162.5, 162.6, 177.4. FTIR (cm^{-1}): 3120, 3033, 2821, 1677, 1512, 1196, 1023. FTIR (cm^{-1}): 3119, 3033, 2925, 1676, 1583, 1023. HRMS (ESI) calculated for $\text{C}_{14}\text{H}_{14}\text{O}_2\text{Na}^+$ $[\text{M}+\text{Na}]$ 237.0886, found 237.0867.

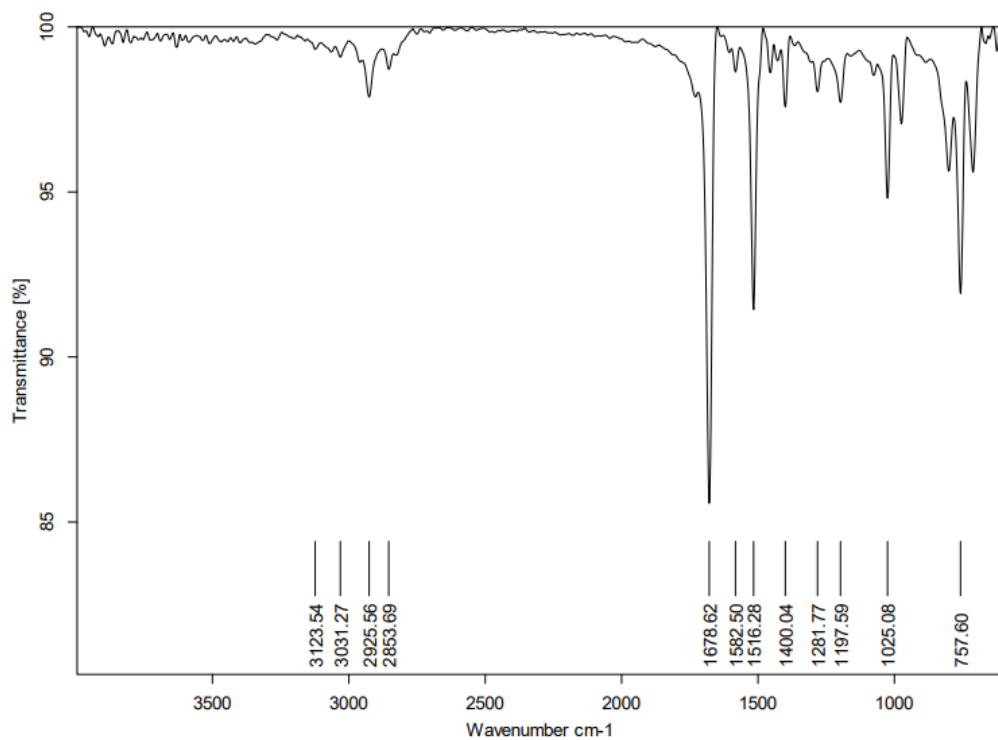


Figure S1. The FTIR spectrum of **1a**.

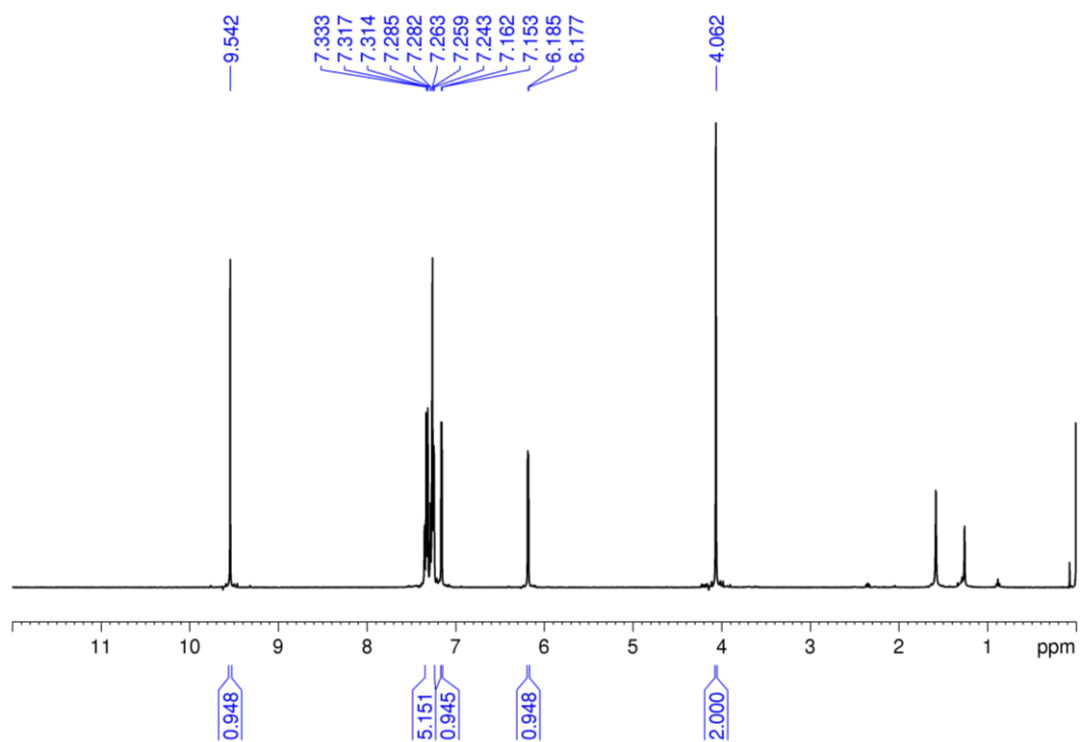


Figure S2. The $^1\text{H-NMR}$ spectrum of **1a**.

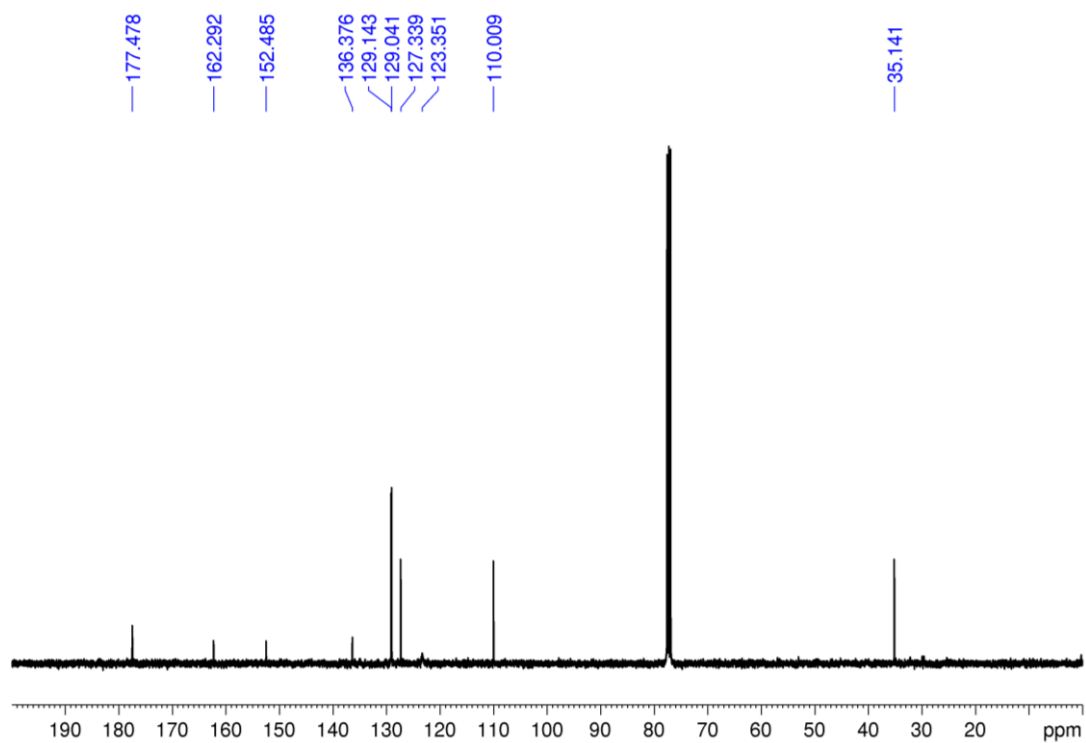


Figure S3. The ^{13}C -NMR spectrum of **1a**.

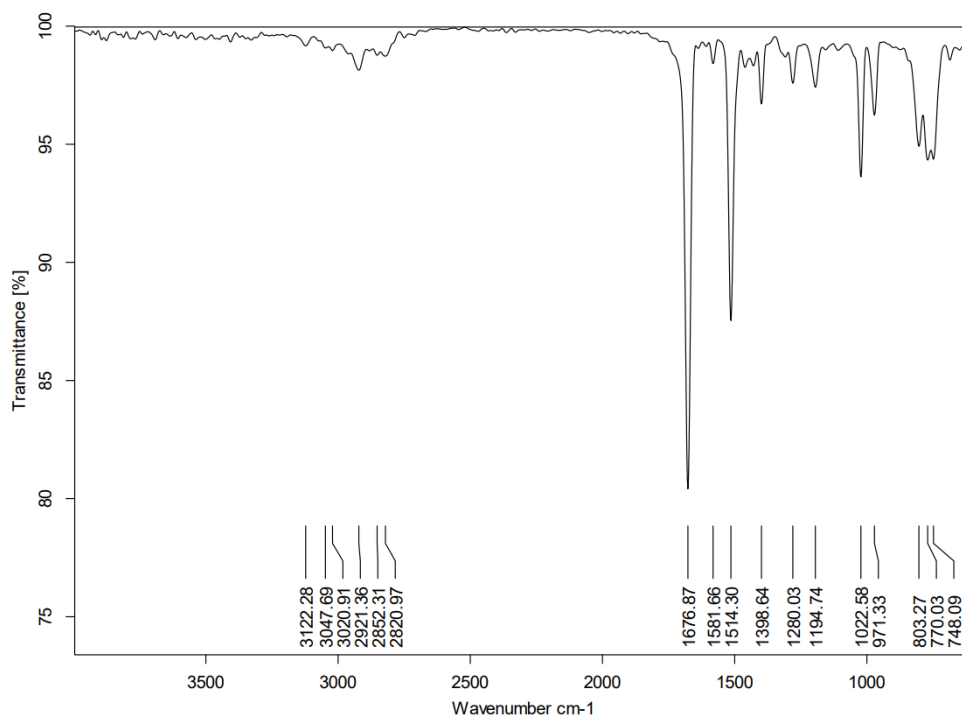


Figure S4. The FTIR spectra of **1b**.

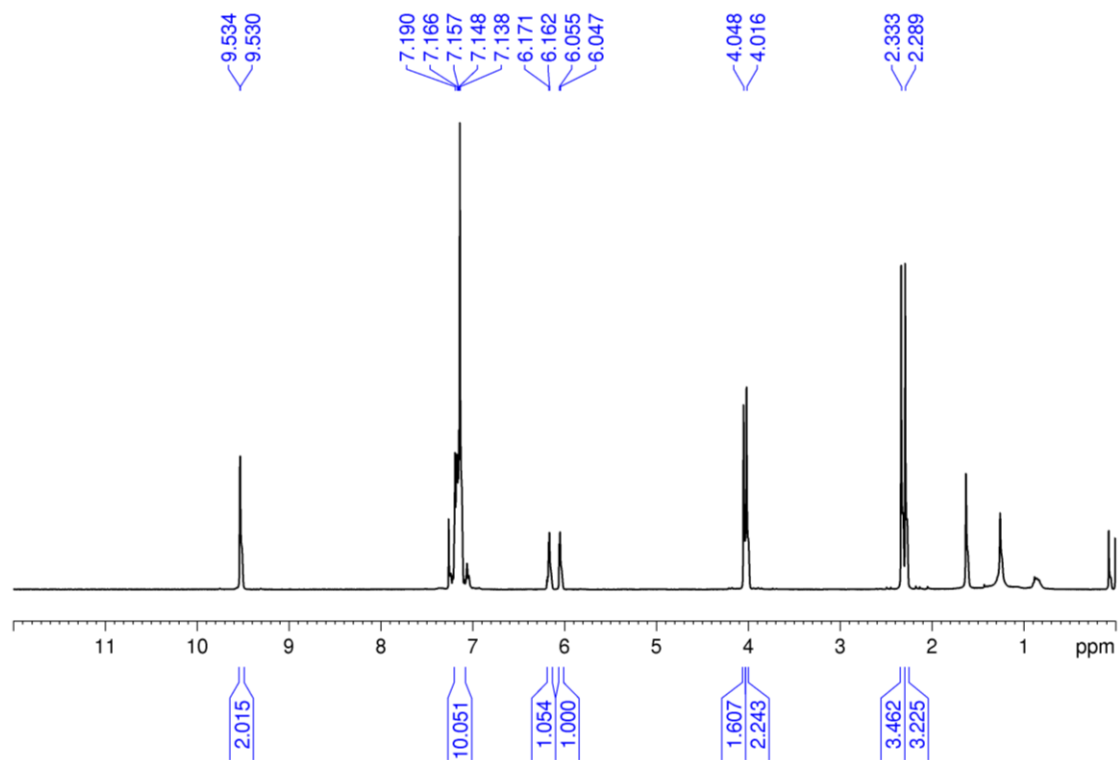


Figure S5. The ^1H -NMR spectrum of **1b**.

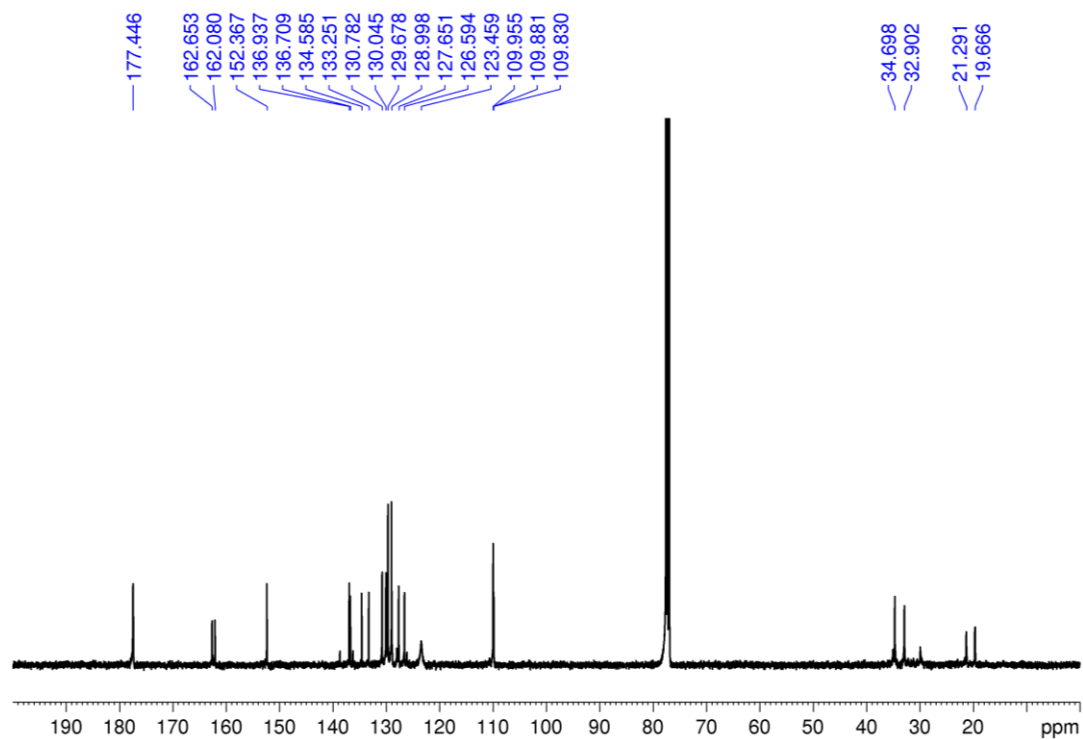


Figure S6. The ^{13}C -NMR spectrum of **1b**.

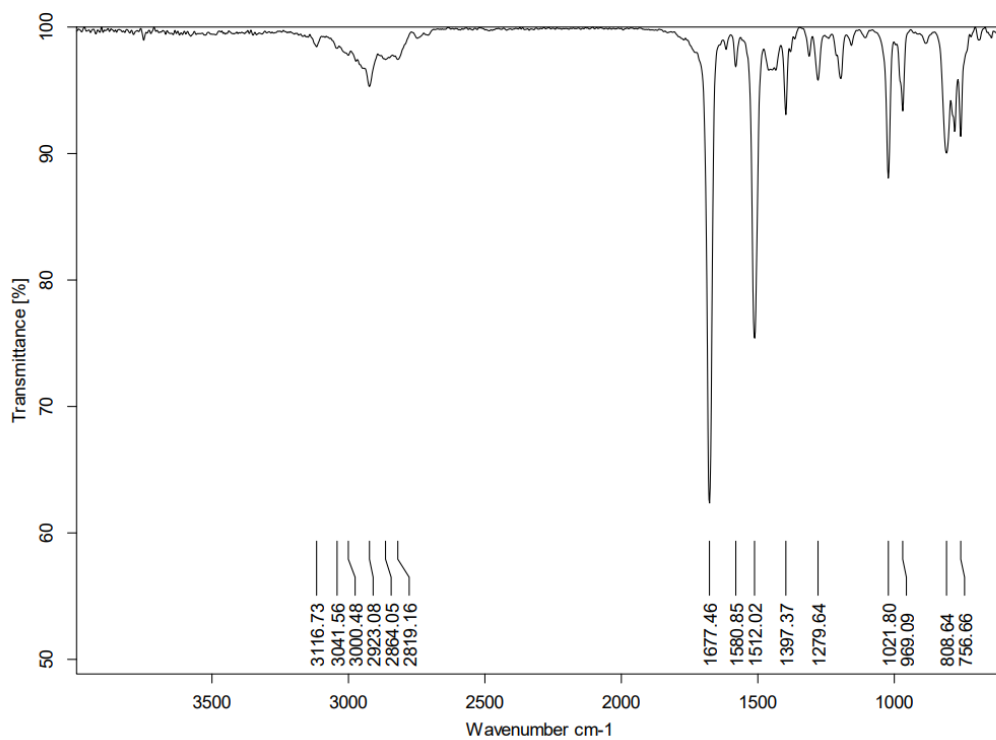


Figure S7. The FTIR spectra of **1c**.

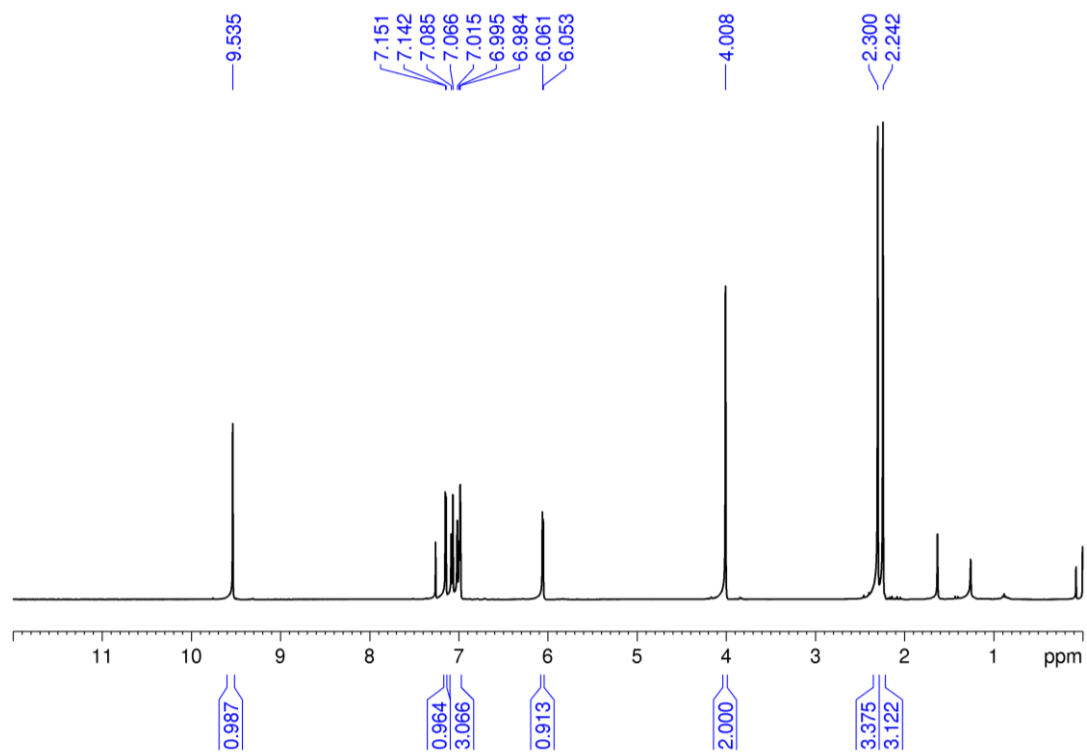


Figure S8. The ¹H-NMR spectrum of **1c**.

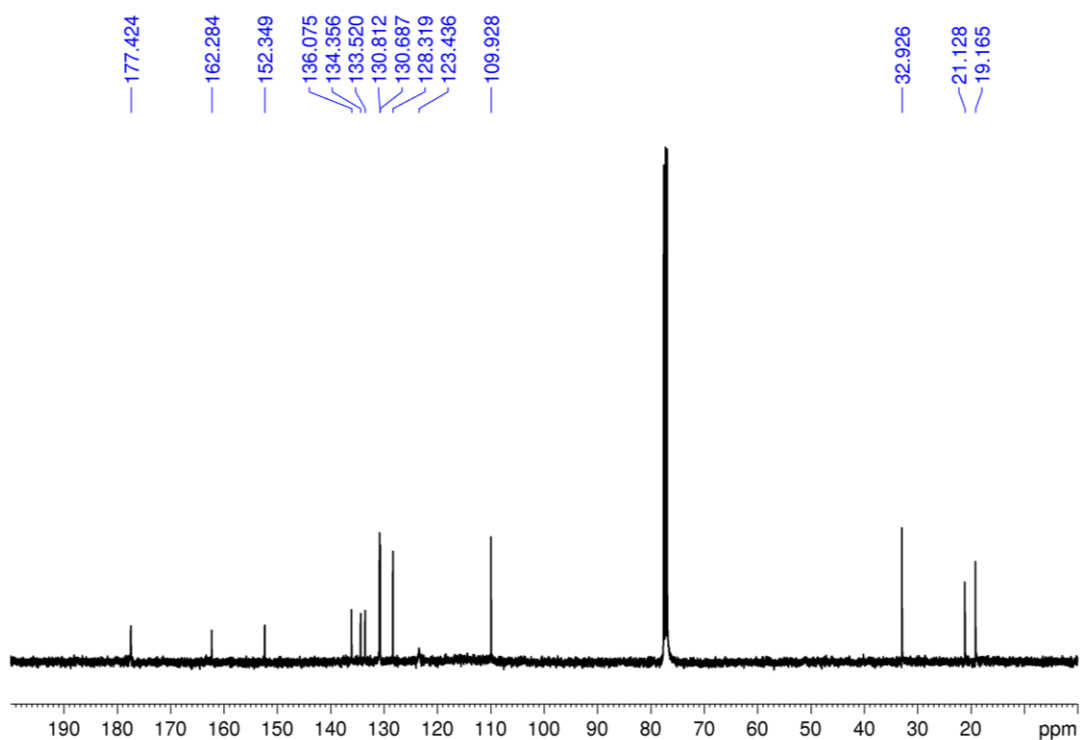


Figure S9. The ^{13}C -NMR spectrum of **1c**.

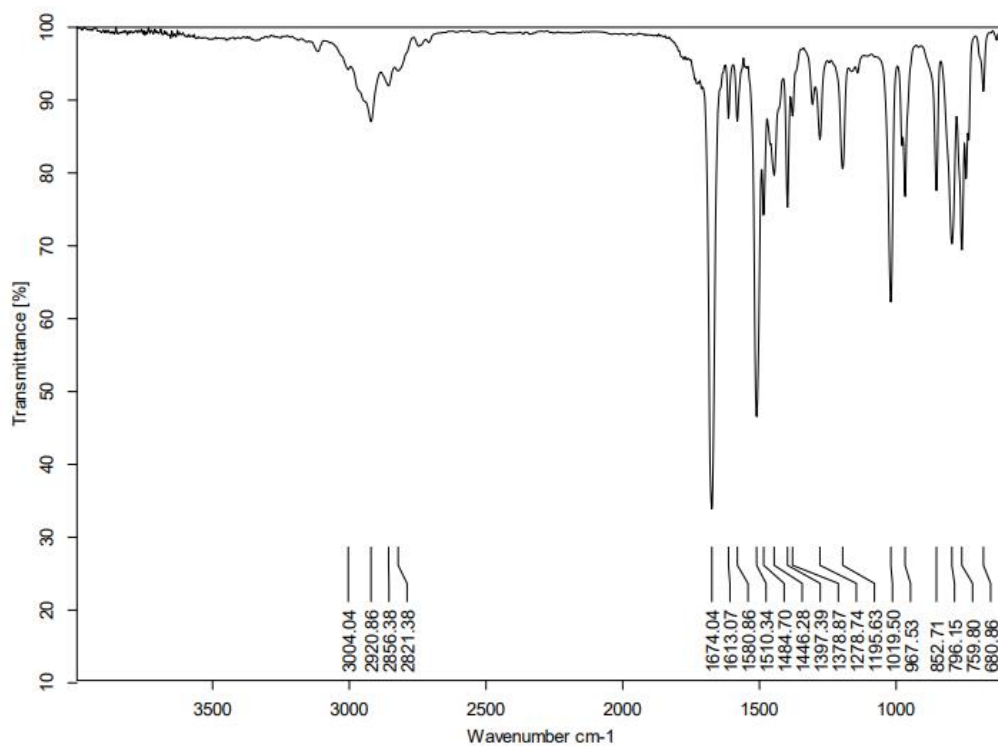


Figure S10. The FTIR spectrum of **1d**.

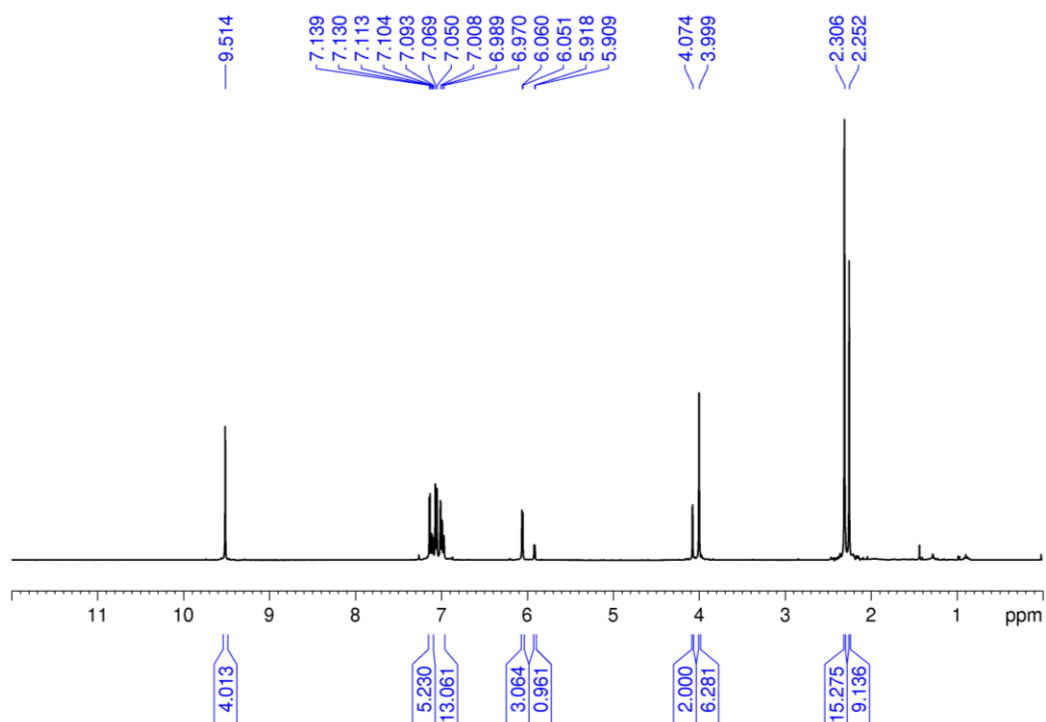


Figure S11. The ^1H -NMR spectrum of **1d**.

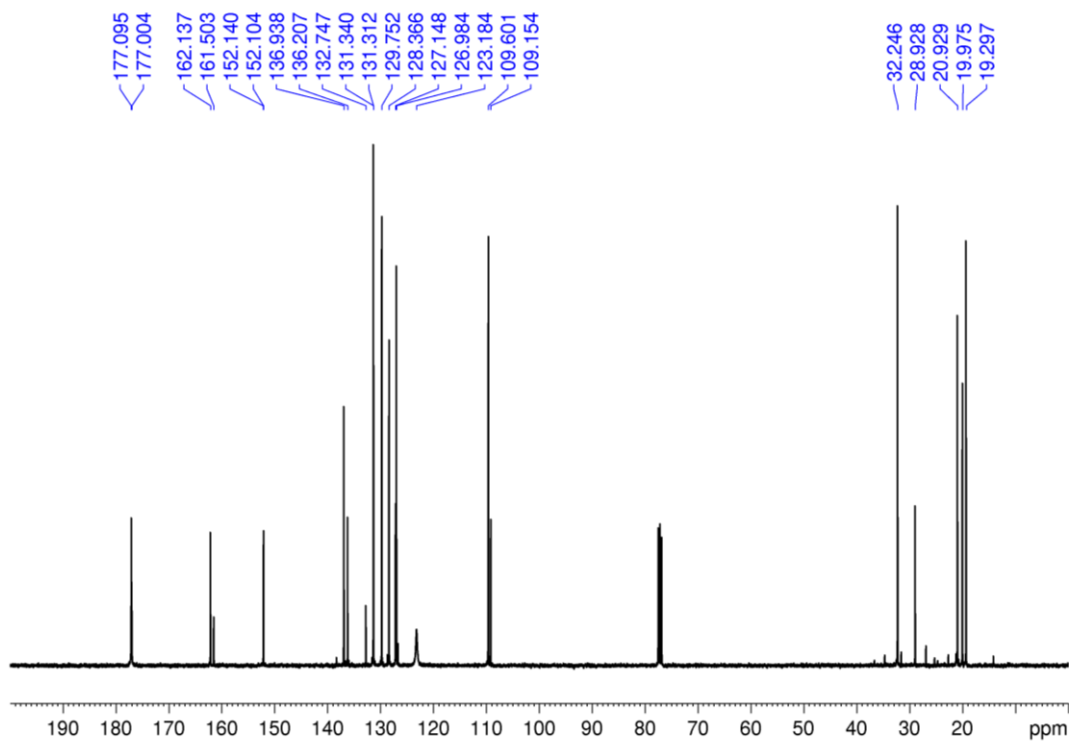


Figure S12. The ^{13}C -NMR spectrum of **1d**.

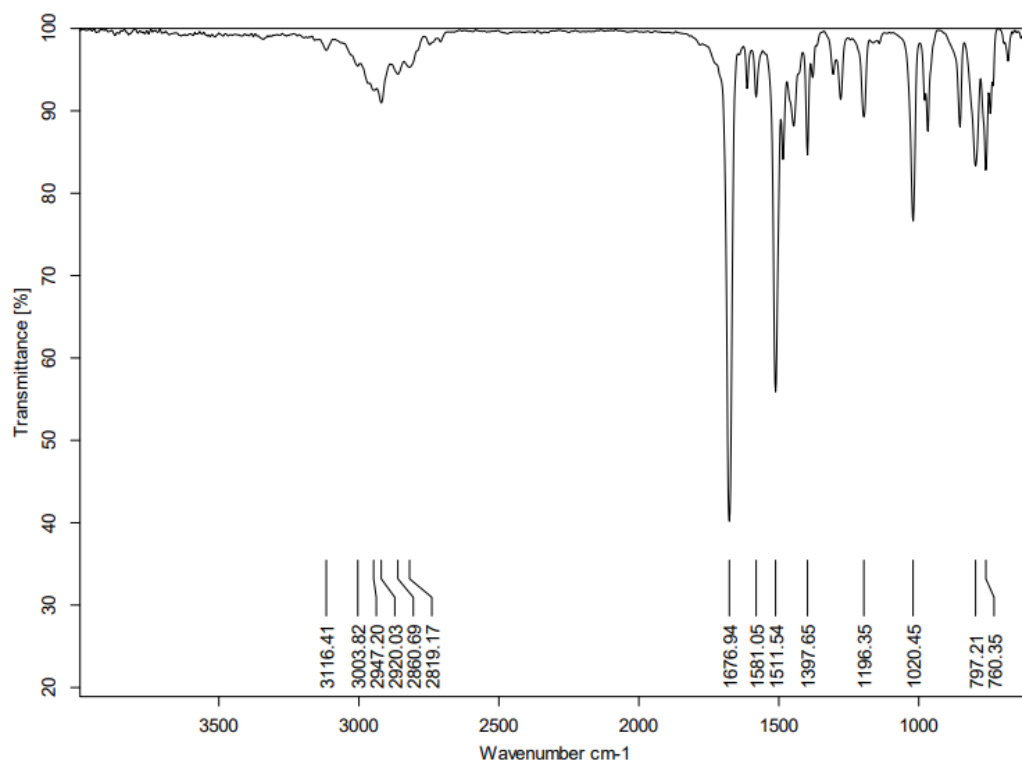


Figure S13. The FTIR spectrum of **1e**.

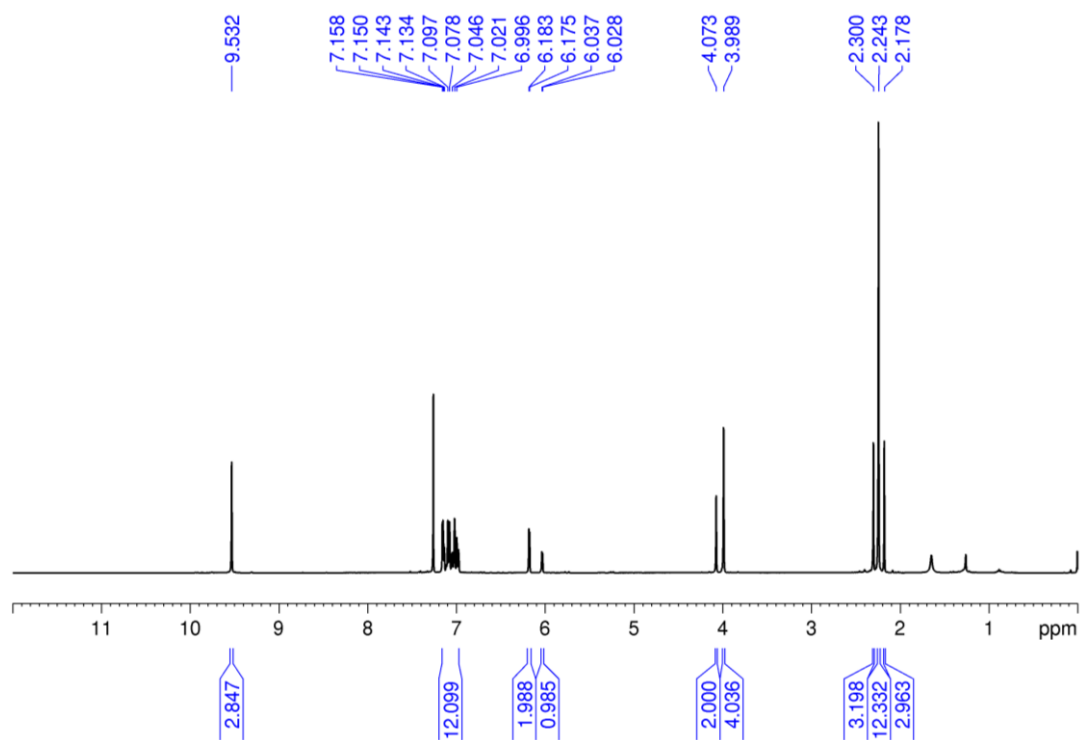


Figure S14. The ¹H-NMR spectrum of **1e**.

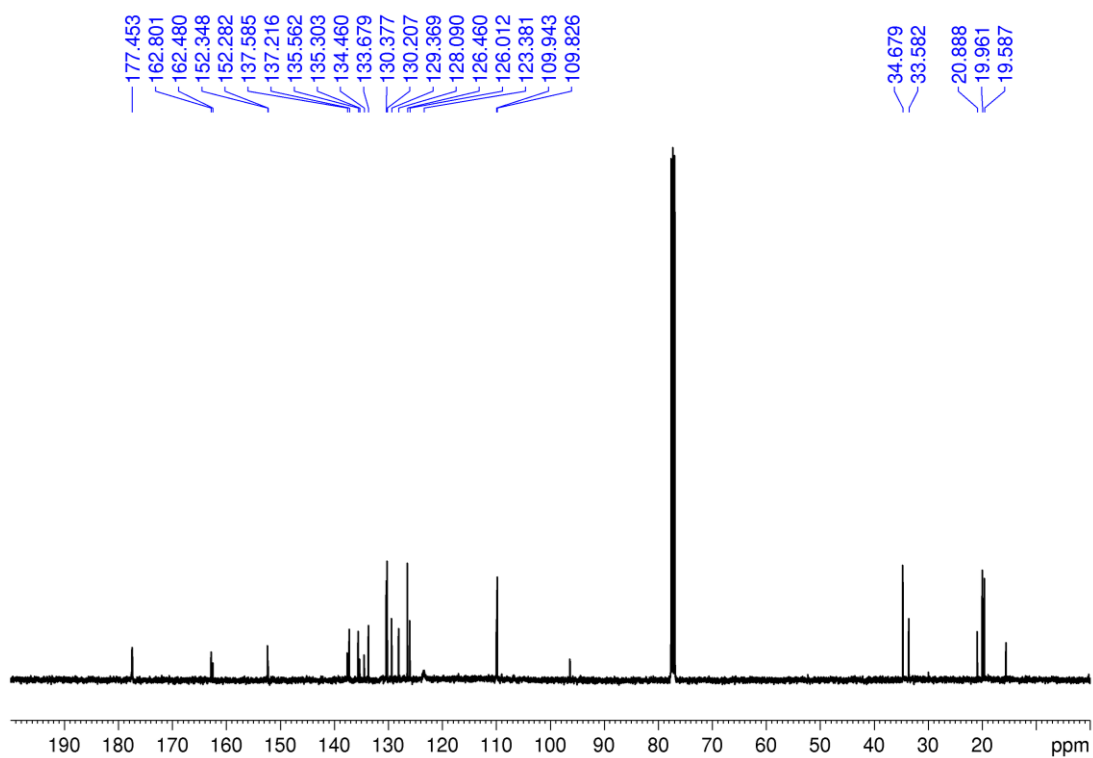


Figure S15. The ^{13}C -NMR spectrum of **1e**.

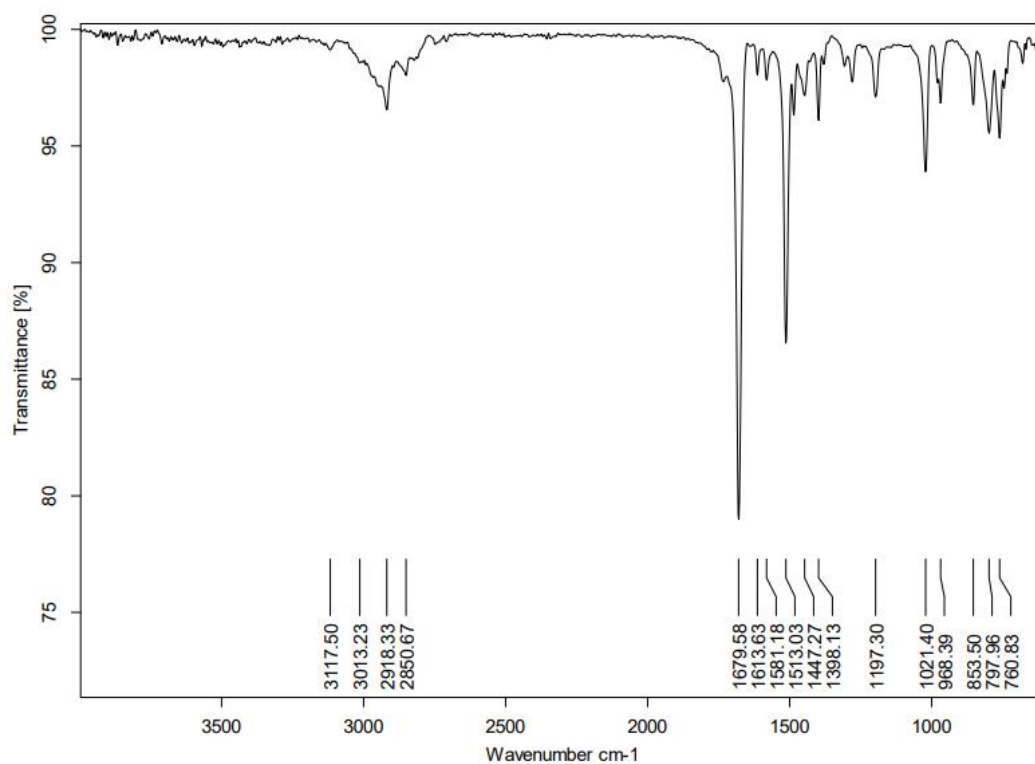


Figure S16. The FTIR spectrum of **1f**.

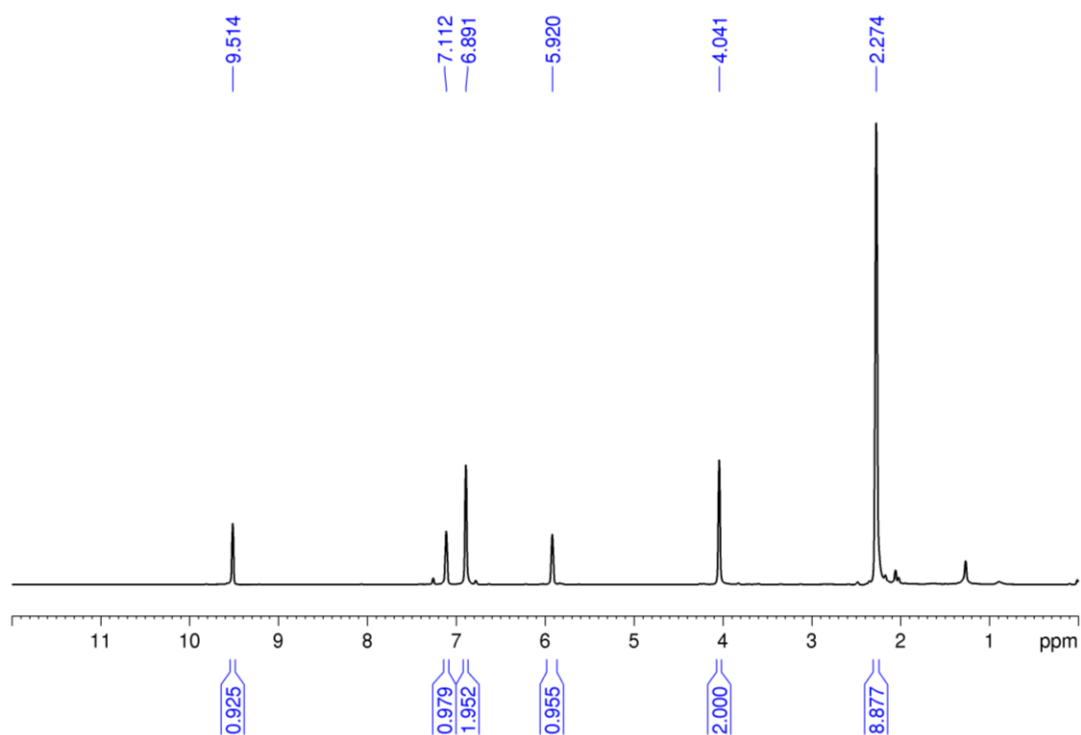


Figure S17. The ^1H -NMR spectrum of **1f**.

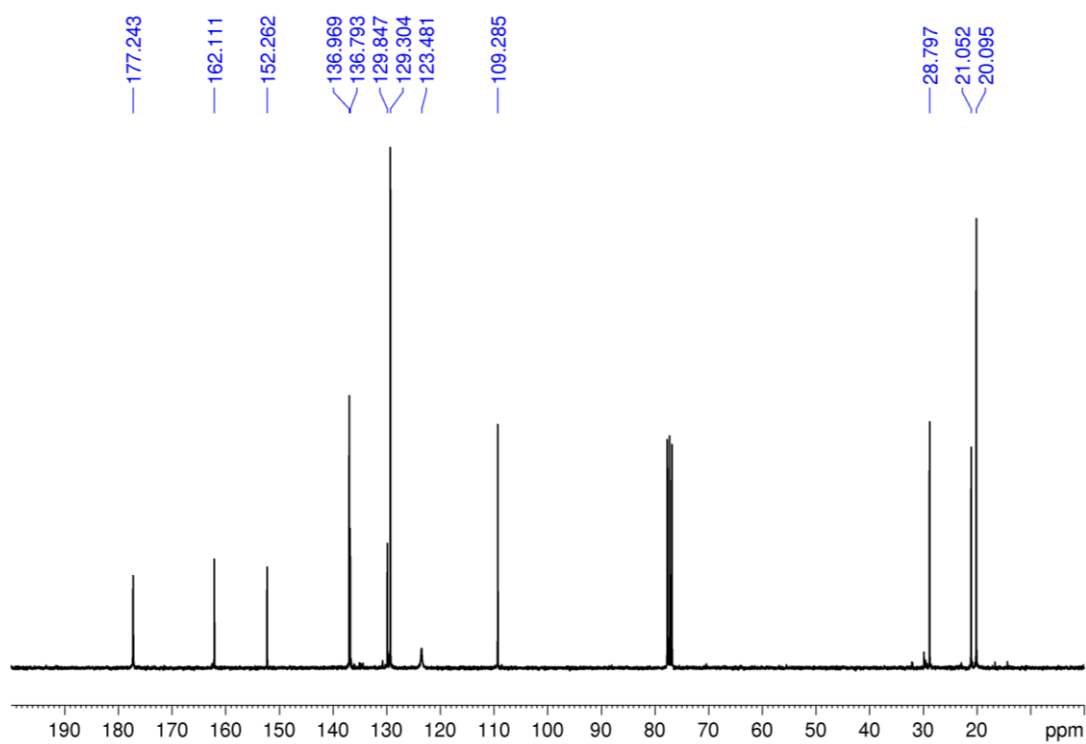


Figure S18. The ^{13}C -NMR spectrum of **1f**.

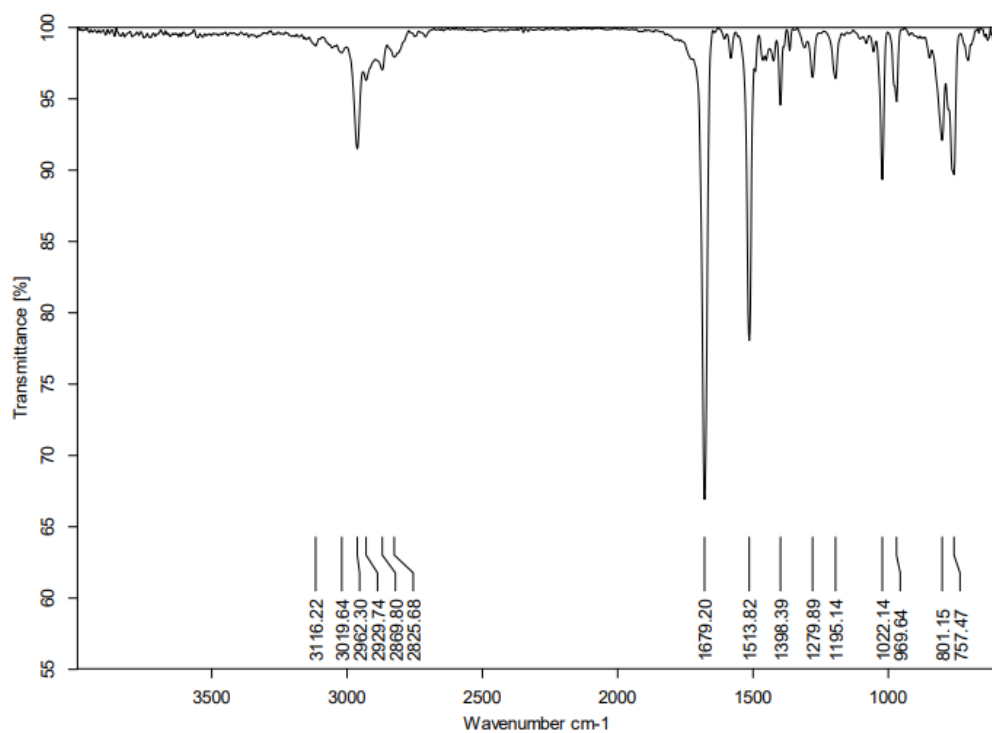


Figure S19. The FTIR Spectrum of **1g**.

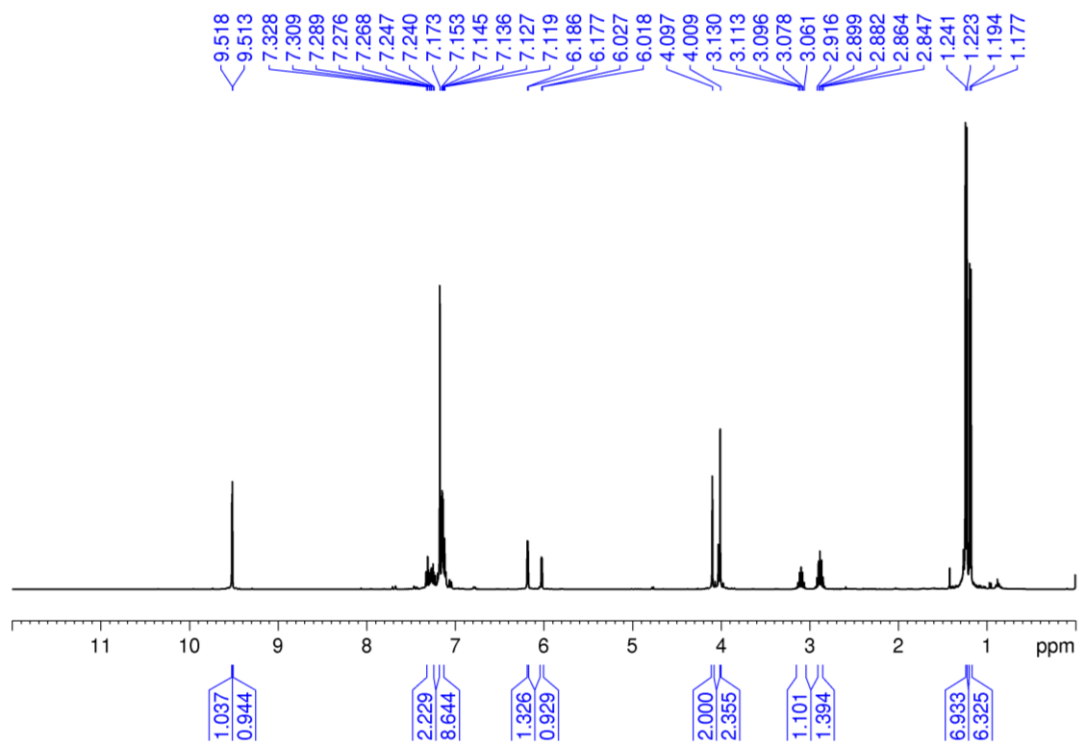


Figure S20. The ¹H-NMR spectrum of **1g**.

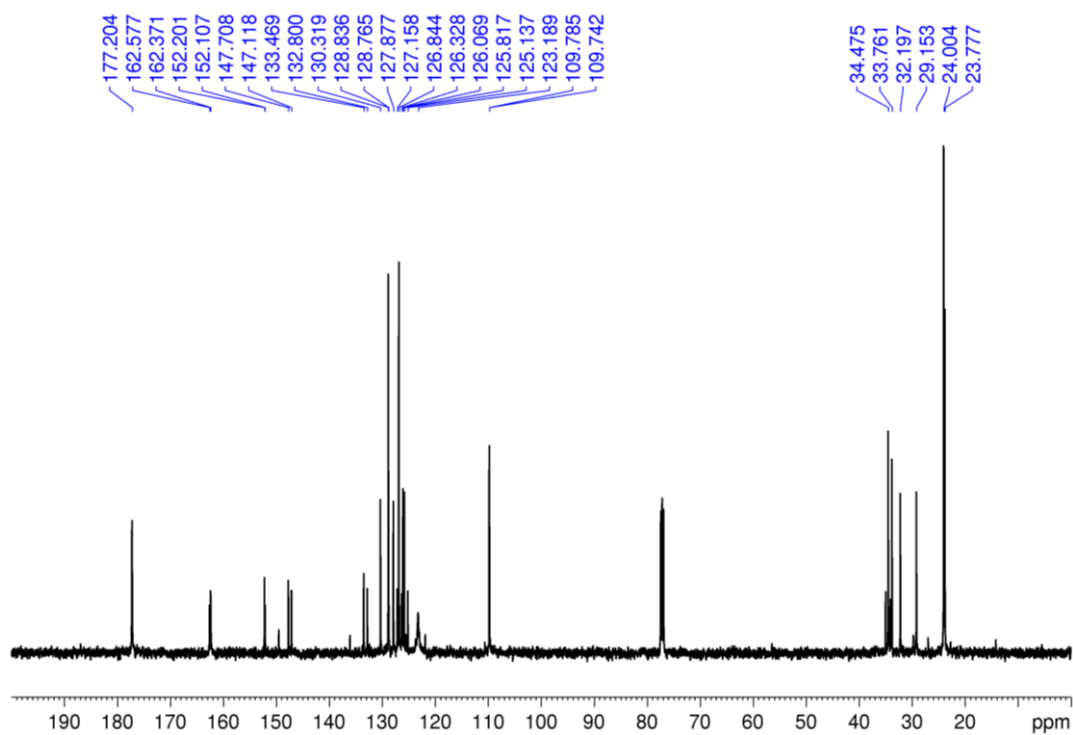


Figure S21. The ^{13}C -NMR spectrum of **1g**.

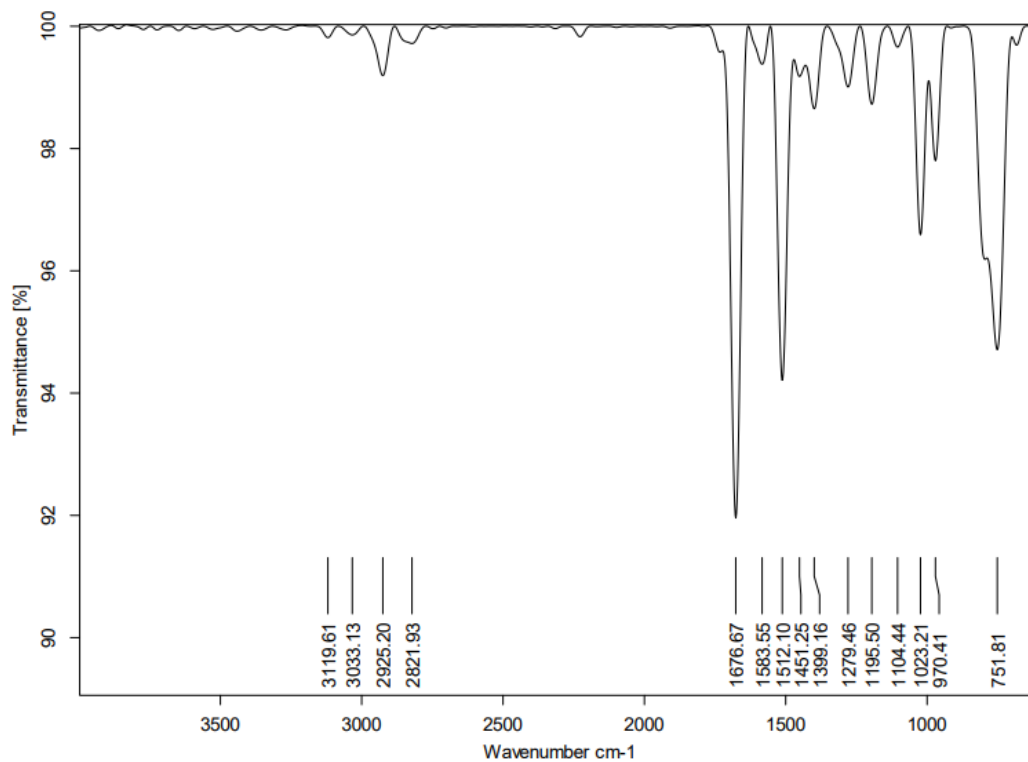


Figure S22. The FTIR Spectrum of **1h**.

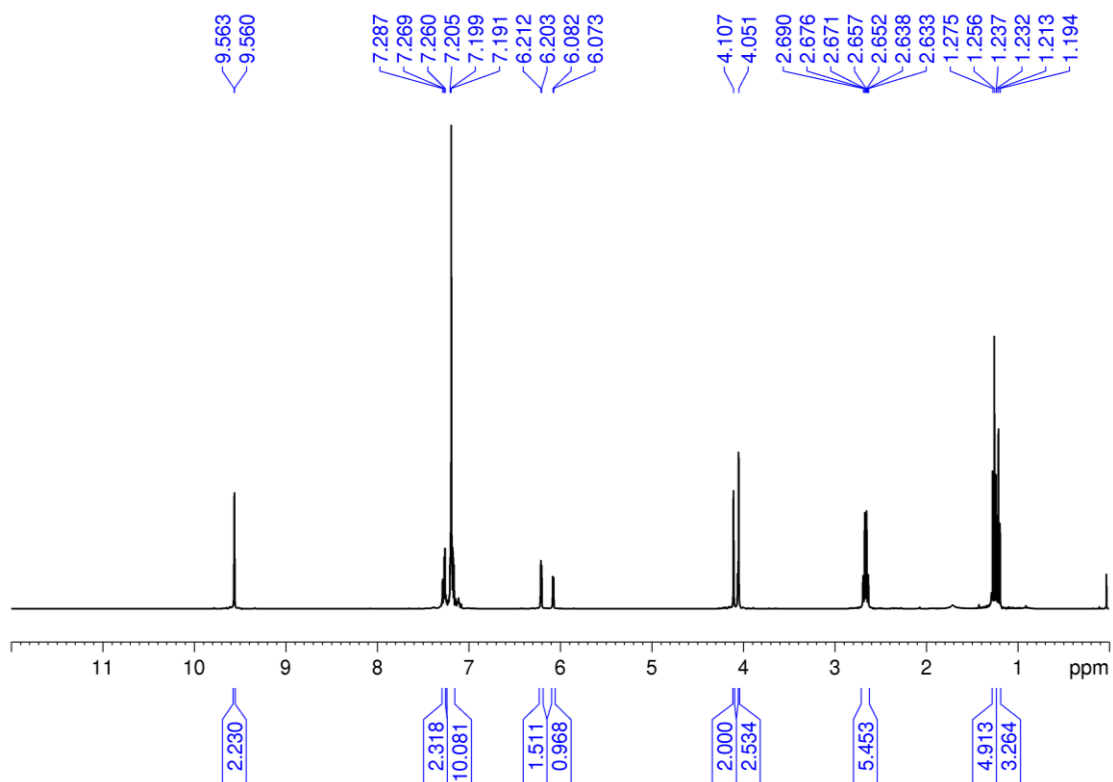


Figure S23. The ^1H -NMR spectrum of **1h**.

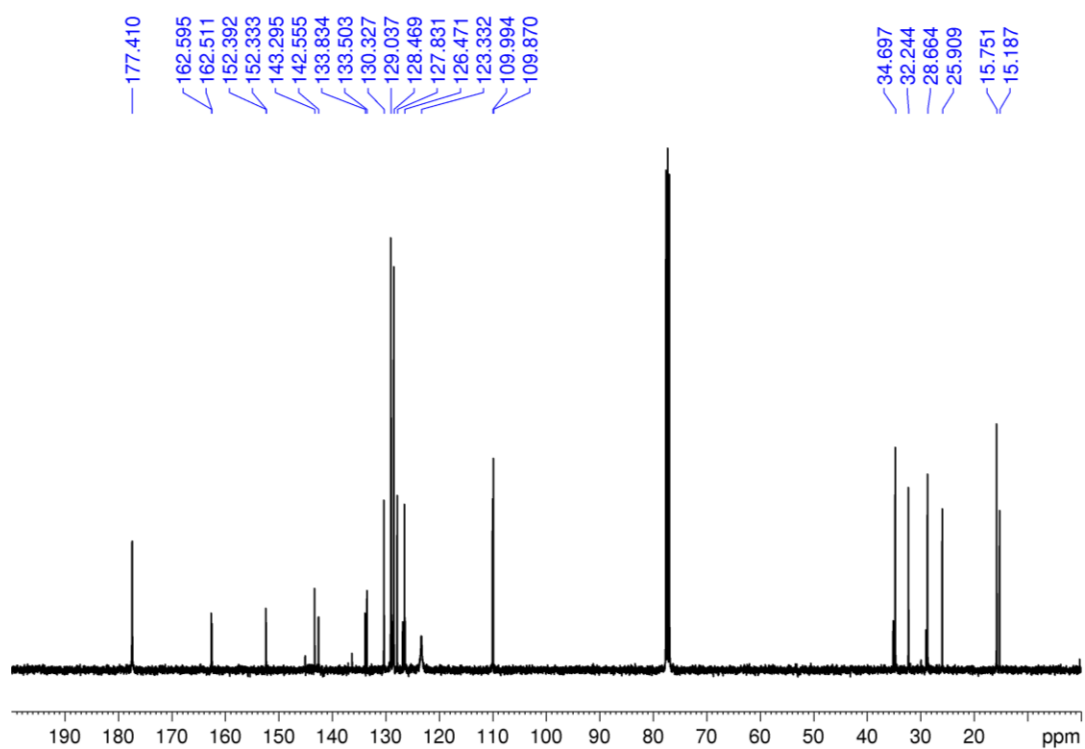


Figure S24. The ^{13}C -NMR spectrum of **1h**.

Characterization of 2-(arylmethyl)furans (AMFs)

2-(Benzenylmethyl)furan (**2a**): Transparent liquid (0.155 g, 73%), $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ (ppm): 3.98 (s, 2H), 6.01 (d, 1H, $J = 2.4$ Hz), 6.29 (dd, 1H), 7.24-7.34 (m, 6H). $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ (ppm): 34.6, 106.4, 110.4, 126.6, 128.6, 128.8, 138.3, 141.6, 154.7. FTIR (cm^{-1}): 3063, 3029, 2852, 1596, 1246, 1148, 1075, 1009.

2-(Toluenylmethyl)furan (**2b**): Transparent liquid (0.163 g, 76%), $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ (ppm): 2.36 (s, 3H), 2.37 (s, 3H), 3.98 (s, 2H), 4.00 (s, 2H), 5.95 (d, 1H, $J = 2.0$ Hz), 6.04 (d, 1H, $J = 2.4$ Hz), 6.32 (d, 2H, $J = 2.8$ Hz), 7.17-7.21 (m, 8H), 7.37 (s, 2H). $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ (ppm): 19.5, 21.2, 32.4, 34.2, 106.2, 106.3, 110.3, 110.4, 126.2, 126.9, 128.7, 129.3, 129.7, 130.4, 135.2, 136.1, 136.5, 136.6, 141.4, 141.5, 154.3, 155.0. FTIR (cm^{-1}): 3117, 2924, 2859, 1592, 1378, 1160, 1013.

2-(p-Xylenylmethyl)furan (**2c**): Transparent liquid (0.173 g, 80%), $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ (ppm): 2.28 (s, 3H), 2.31 (s, 3H), 3.93 (s, 2H), 5.92 (d, 1H, $J = 0.8$ Hz), 6.29 (dd, 1H), 6.97-7.07 (m, 3H), 7.33 (d, 1H, $J = 0.8$ Hz). $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ (ppm): 19.1, 21.2, 32.5, 106.3, 110.4, 127.7, 130.4, 130.5, 133.5, 135.7, 136.3, 141.5, 154.6. FTIR (cm^{-1}): 3118, 3007, 2857, 1593, 1215, 1154, 1073, 1009.

2-(m-Xylenylmethyl)furan (**2d**): Transparent liquid (0.169 g, 78%), $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ (ppm): 2.28 (s, 9H), 2.31 (s, 9H), 2.35 (s, 6H), 3.93 (s, 5H), 3.99 (s, 2H), 5.75 (dd, 1H), 5.90 (dd, 3H), 6.24 (dd, 1H), 6.27 (dd, 3H), 7.01 (m, 12H), 6.98-7.08 (m, 12H), 7.32 (t, 4H). $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ (ppm): 19.5, 20.2, 21.2, 28.8, 32.1, 105.7, 106.2, 110.3, 110.4, 126.8, 126.9, 128.4, 129.7, 131.3, 133.5, 136.5, 136.5, 137.2, 141.3, 141.5, 154.1, 154.7. FTIR (cm^{-1}): 3119, 2861, 2861, 1593, 1213, 1156, 1074, 1009.

2-(o-Xylenylmethyl)furan (**2e**): Transparent liquid (0.163 g, 80%), $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ (ppm): 2.24 (s, 3H), 2.27 (s, 9H), 2.33 (s, 3H), 3.94 (s, 3H), 4.01 (s, 2H), 5.91 (d, 1H, $J = 2.8$ Hz), 6.03 (d, 1H, $J = 3.2$ Hz), 6.30-6.32 (m, 2H), 7.00-7.10 (m, 8H), 7.35 (s, 2H). $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ (ppm): 15.4, 19.6, 20.0, 20.9, 33.1, 34.3, 106.2, 106.3, 110.4, 110.4, 125.7, 126.3, 127.8, 128.8, 130.0, 130.2, 134.9, 135.3, 135.7, 136.4, 136.9, 137.2, 141.4, 141.6, 154.8, 155.2. FTIR (cm^{-1}): 3007, 2922, 2856, 1211, 1074, 1008.

2-(Mesitylmethyl)furan (**2f**): Transparent liquid (0.176 g, 80%), $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ (ppm): 2.24 (s, 3H), 2.27 (s, 6H), 3.91 (s, 2H), 5.73 (s, 1H), 6.20 (s, 1H), 6.84 (s, 2H), 7.23

(s, 1H). ^{13}C -NMR (CDCl_3 , 100 MHz) δ (ppm): 20.1, 21.1, 28.4, 105.6, 110.3, 129.1, 131.7, 136.2, 137.1, 141.3, 154.3. FTIR (cm^{-1}): 3117, 3009, 2922, 2856, 1592, 1210, 1078, 1008.

2-(Cumenylmethyl)furan (**2g**): Transparent liquid (0.176 g, 81%), ^1H -NMR (CDCl_3 , 400 MHz) δ (ppm): 1.22 (d, 6H, $J = 6.8$ Hz), 1.26 (d, 6H, $J = 7.2$ Hz), 2.85-2.95 (m, 1H), 3.16-3.26 (m, 1H), 3.96 (s, 2H), 4.03 (s, 2H), 5.88 (d, 1H, $J = 3.2$ Hz), 6.02 (d, 1H, $J = 3.2$ Hz), 6.28-2.31 (m, 2H), 7.12-7.18 (m, 8H), 7.31-7.34 (m, 2H). ^{13}C -NMR (CDCl_3 , 100 MHz) δ (ppm): 24.0, 24.3, 29.1, 32.0, 34.0, 34.3, 106.3, 106.4, 110.4, 110.5, 124.7, 125.7, 126.0, 126.8, 127.4, 128.8, 130.3, 134.9, 135.7, 141.4, 141.6, 147.2, 147.3, 155.0, 155.1. FTIR (cm^{-1}): 2926, 2869, 1595, 1507, 1148, 1075, 1009. HRMS (ESI) calculated for $\text{C}_{14}\text{H}_{17}\text{O}$ $[\text{M}+\text{H}]^+$ 201.1274, found 201.1278.

2-(Ethylbenzyenylmethyl)furan (**2h**): Transparent liquid (0.162 g, 75%), ^1H -NMR (CDCl_3 , 400 MHz) δ (ppm): 1.18 (t, 3H, $J = 7.6$ Hz), 1.21 (t, 3H, $J = 7.6$ Hz), 2.62 (q, 2H, $J = 7.6$ Hz), 2.65 (q, 2H, $J = 7.6$ Hz), 3.92 (s, 2H), 3.97 (s, 2H), 5.88 (d, 1H, $J = 3.2$ Hz), 5.98 (d, 1H, $J = 3.2$ Hz), 6.26 (m, 2H), 7.13-7.19 (m, 8H), 7.30 (d, 2H). ^{13}C -NMR (CDCl_3 , 100 MHz) δ (ppm): 15.2, 15.8, 25.9, 28.7, 31.8, 34.3, 106.3, 106.4, 110.5, 110.5, 126.2, 127.2, 128.2, 128.7, 128.9, 130.1, 135.6, 135.8, 141.5, 141.6, 142.5, 142.6, 154.8, 155.1. FTIR (cm^{-1}): 3118, 3011, 2858, 1592, 1318, 1160, 1074, 1008.

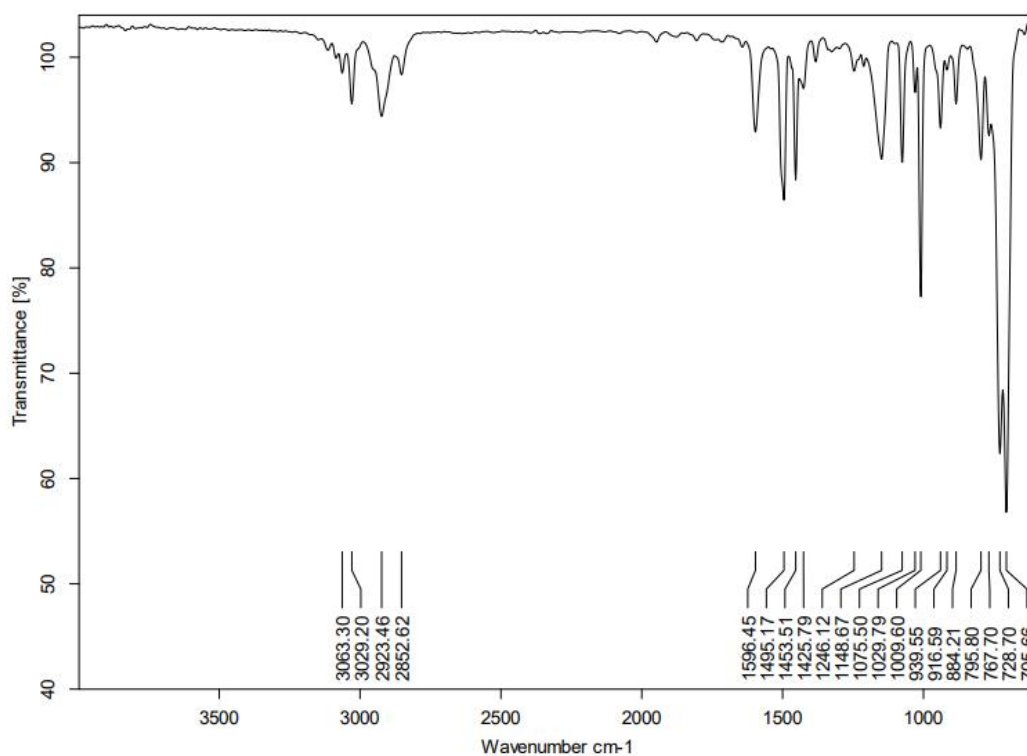


Figure S25. The FTIR spectrum of **2a**.

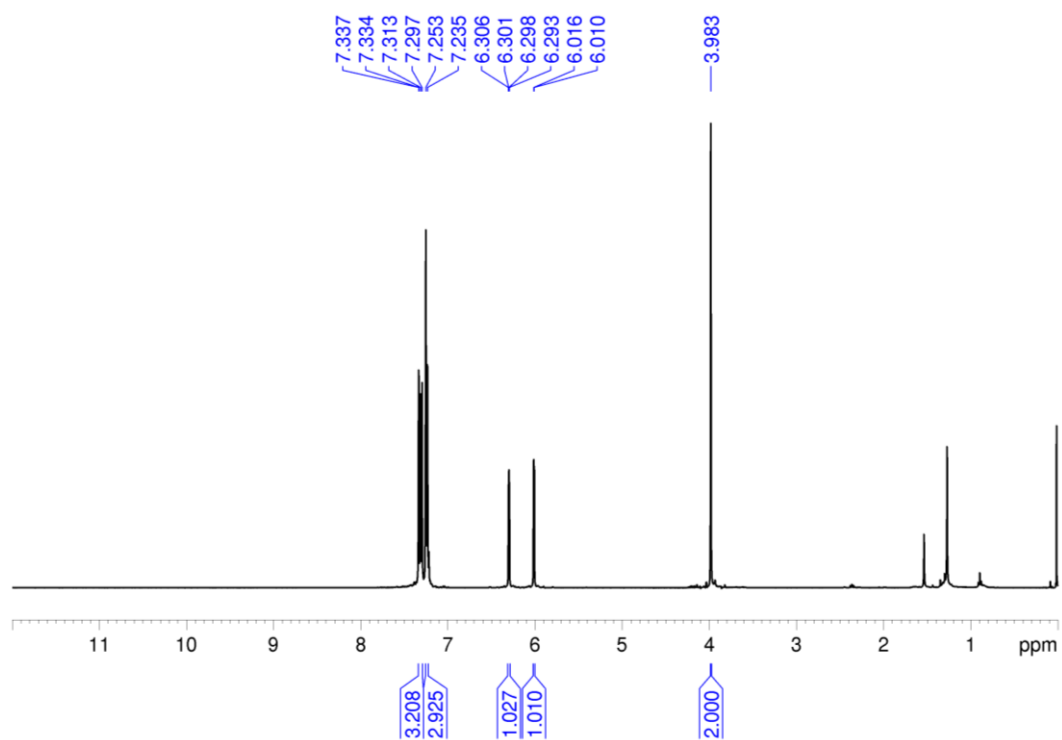


Figure S26. The ^1H -NMR spectrum of **2a**.

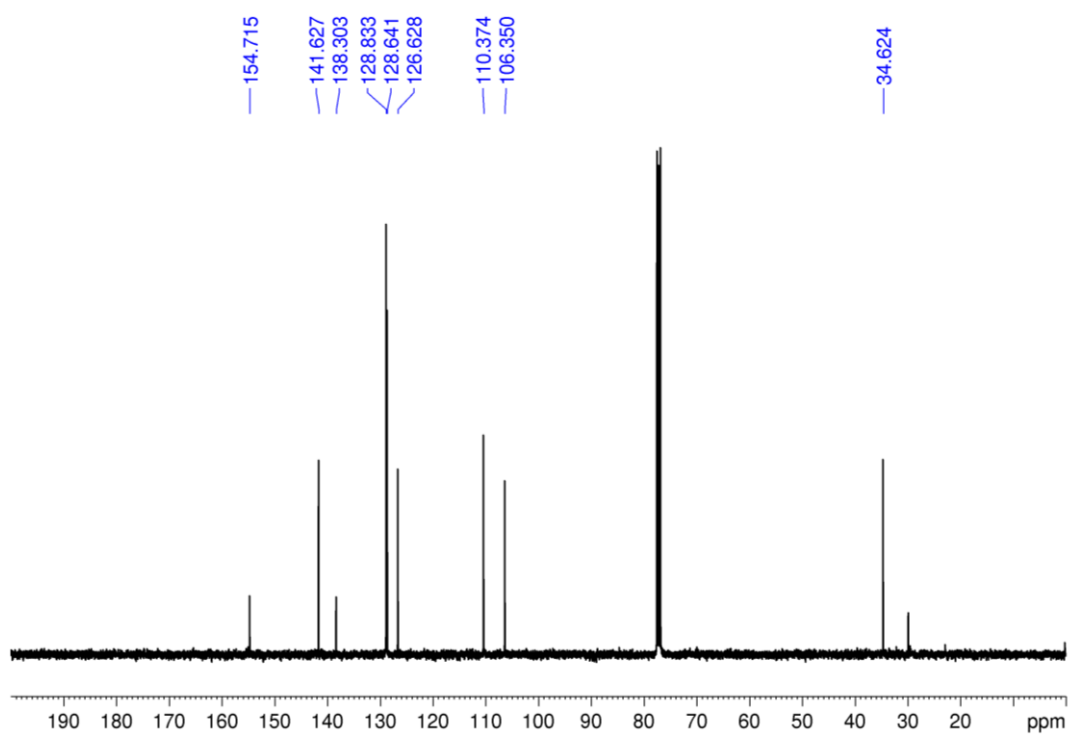


Figure S27. The ^{13}C -NMR spectrum of **2a**.

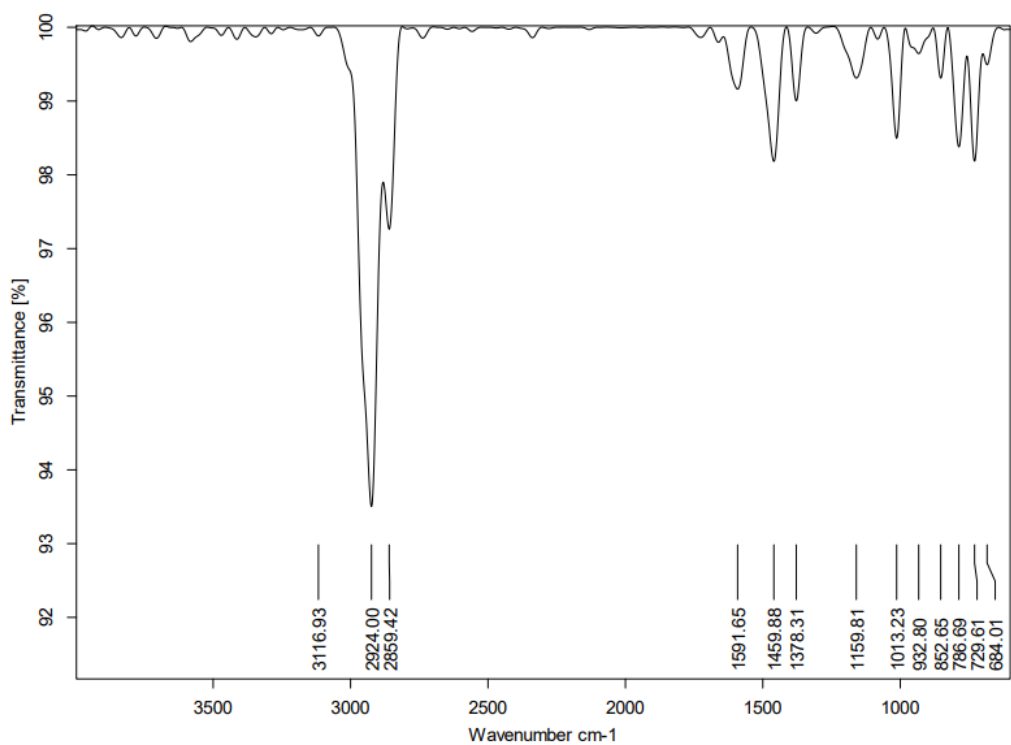


Figure S28. The FTIR spectrum of **2b**.

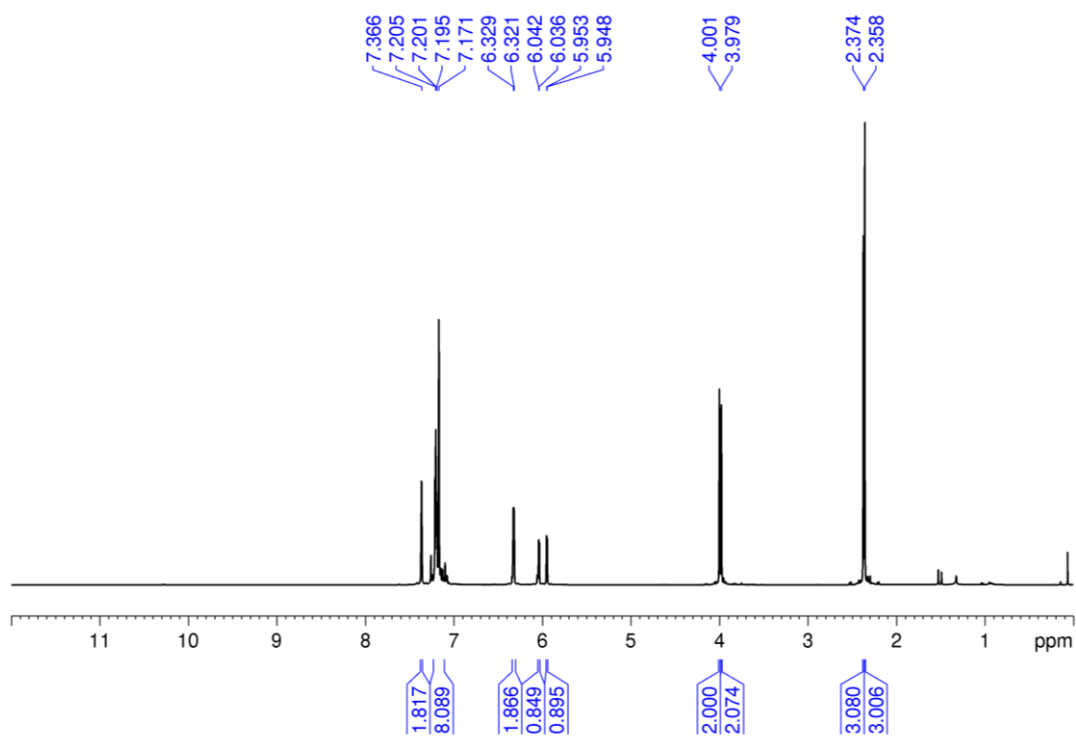


Figure S29. The ¹H-NMR spectrum of **2b**.

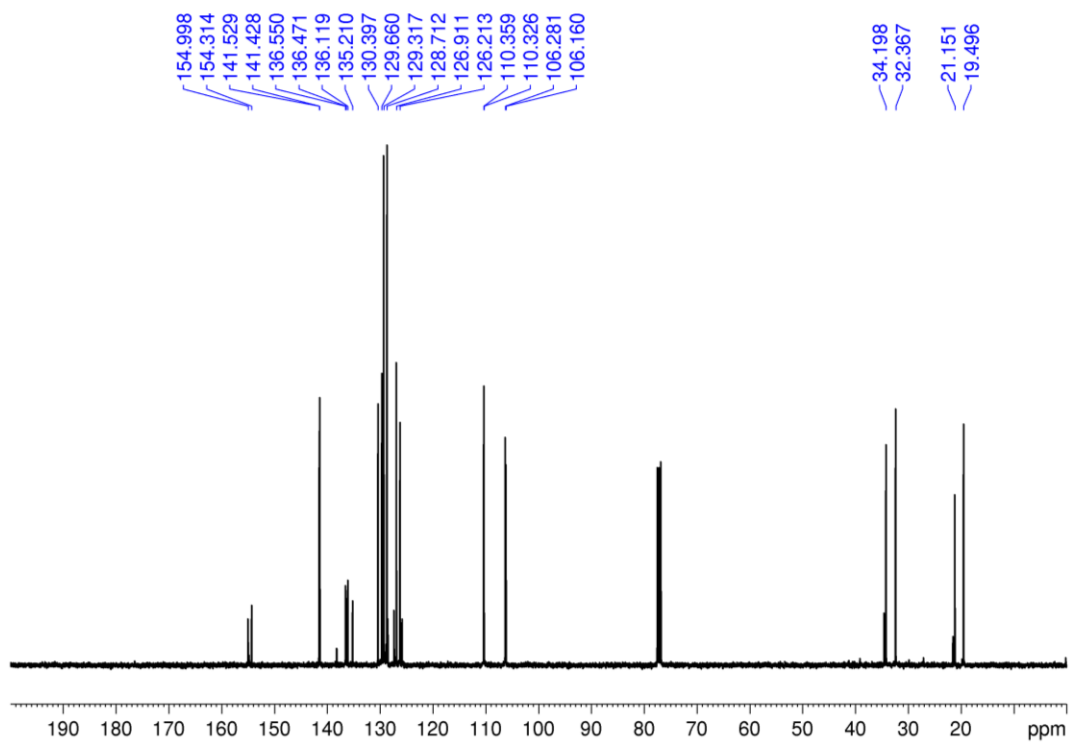


Figure S30. The ^{13}C -NMR spectrum of **2b**.

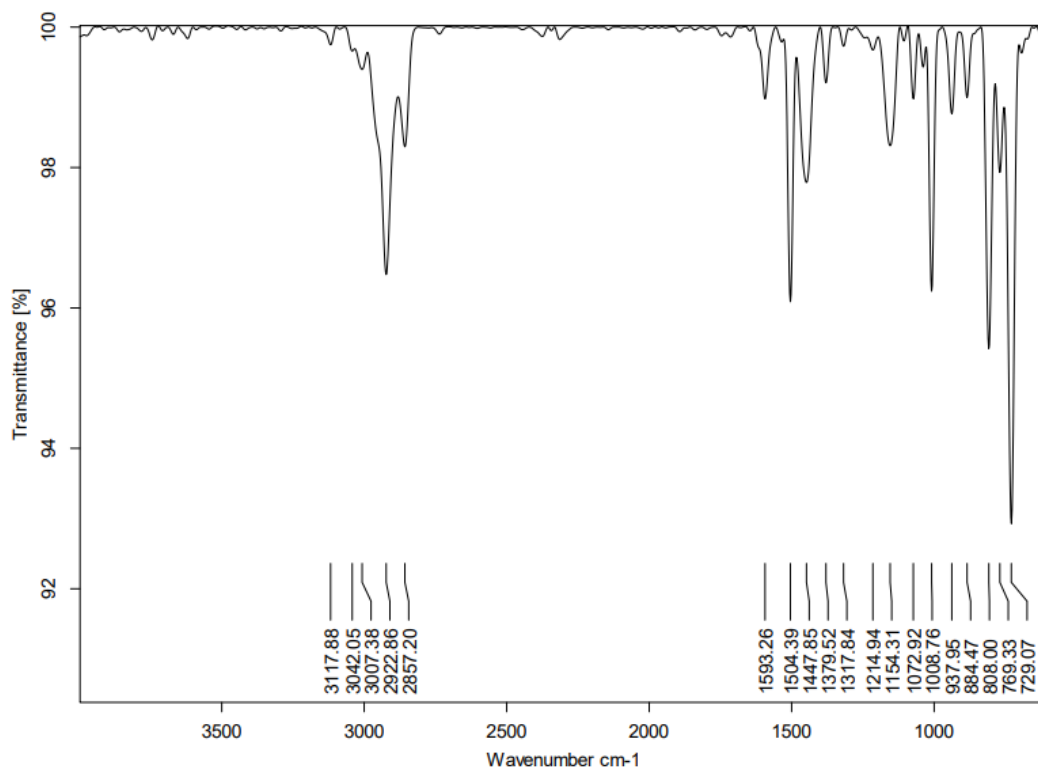


Figure S31. The FTIR spectrum of **2c**.

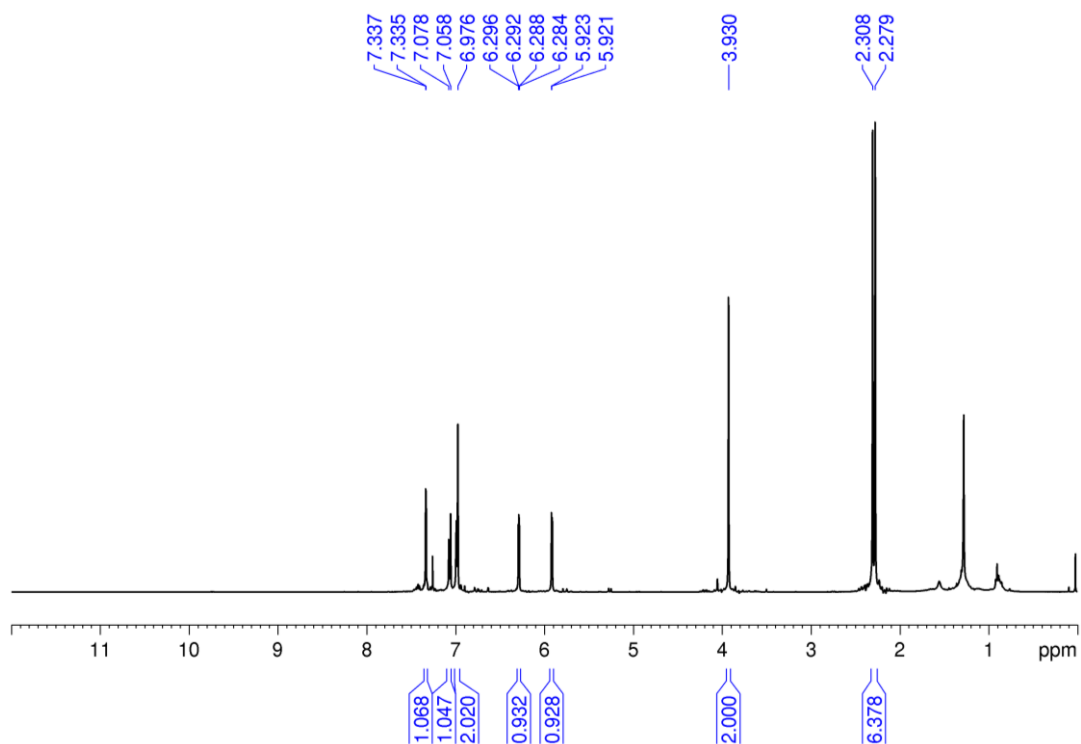


Figure S32. The ^1H -NMR spectrum of **2c**.

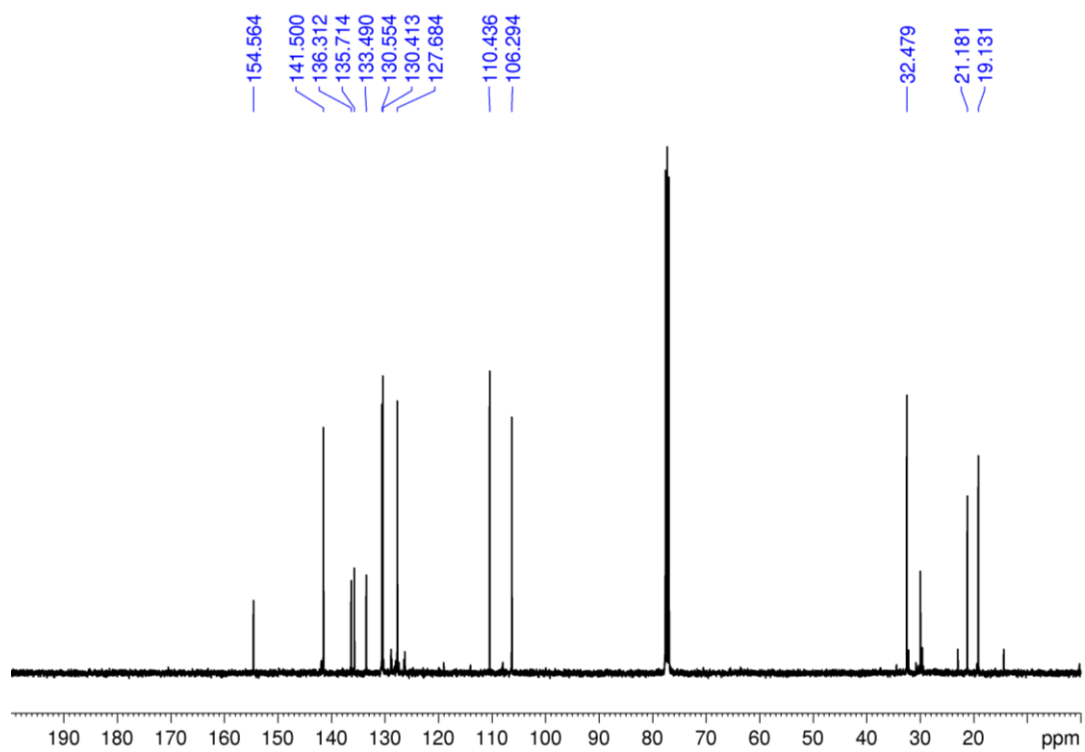


Figure S33. The ^{13}C -NMR spectrum of **2c**.

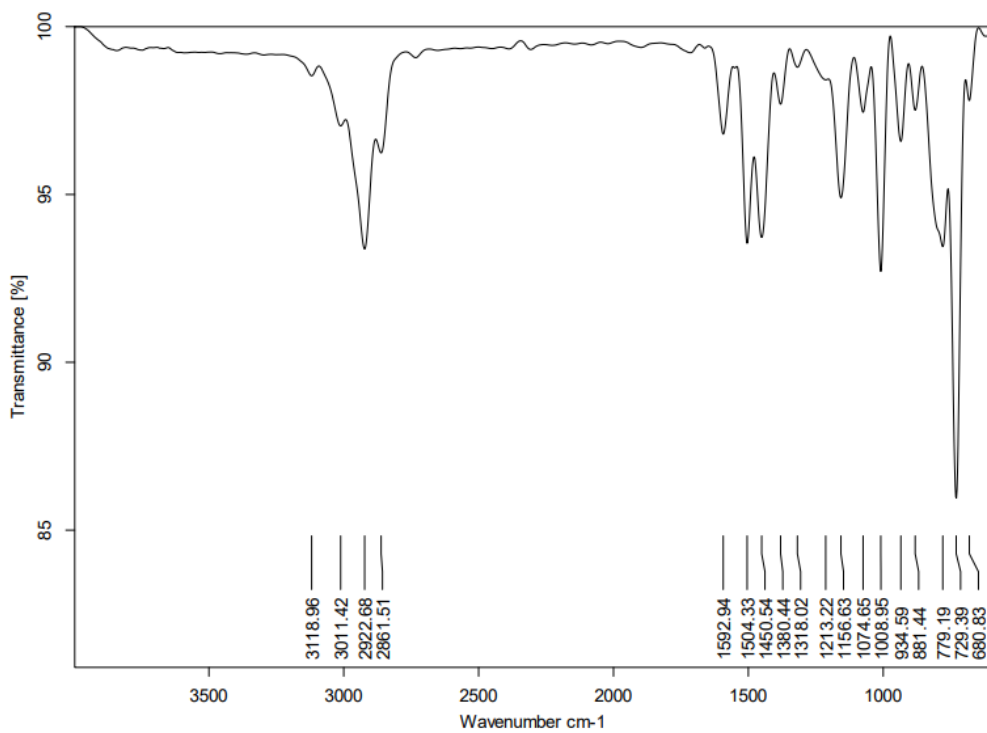


Figure S34. The FTIR spectrum of **2d**.

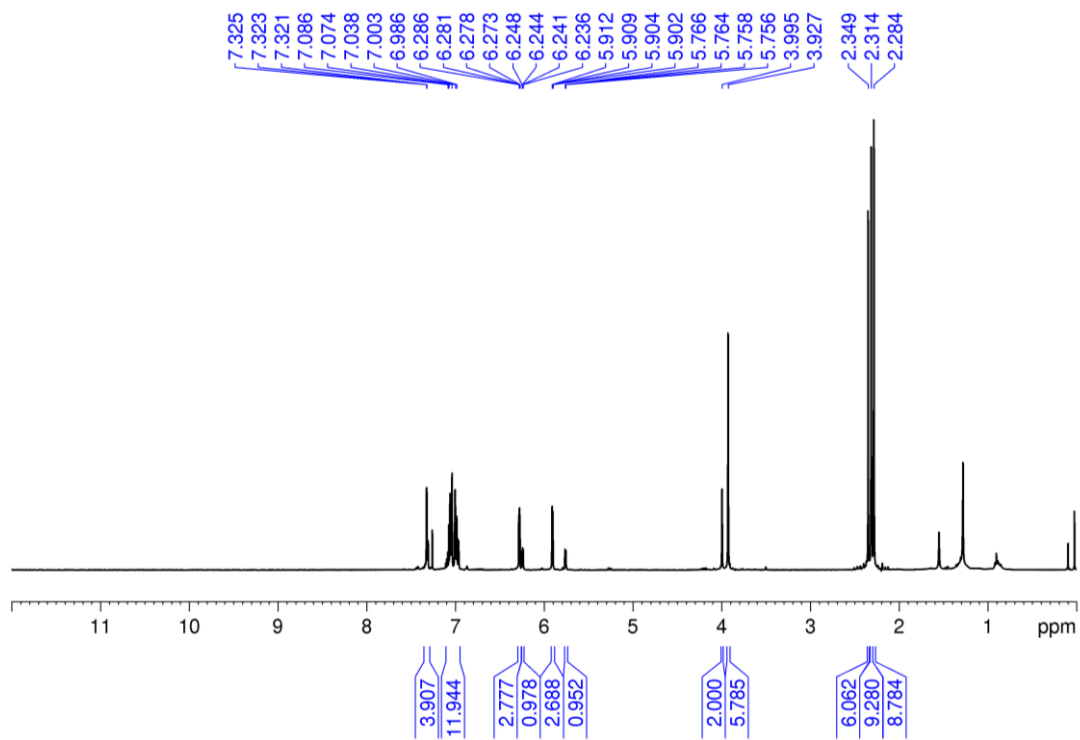


Figure S35. The ¹H-NMR spectrum of **2d**.

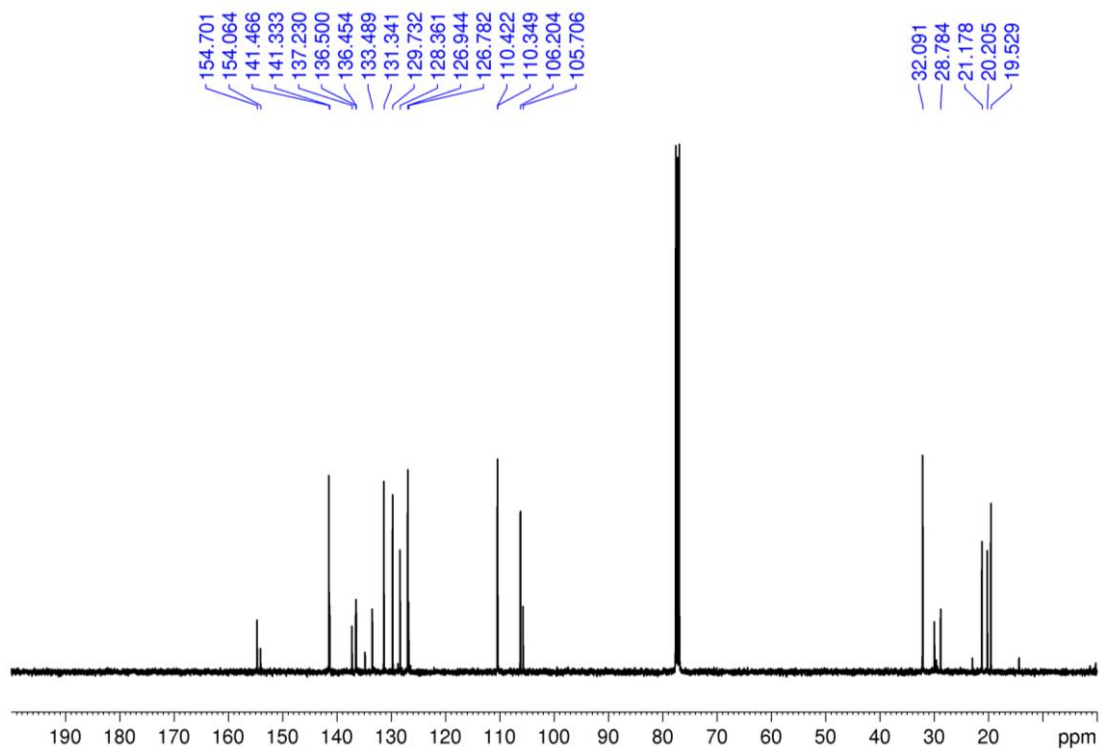


Figure S36. The ^{13}C -NMR spectrum of **2d**.

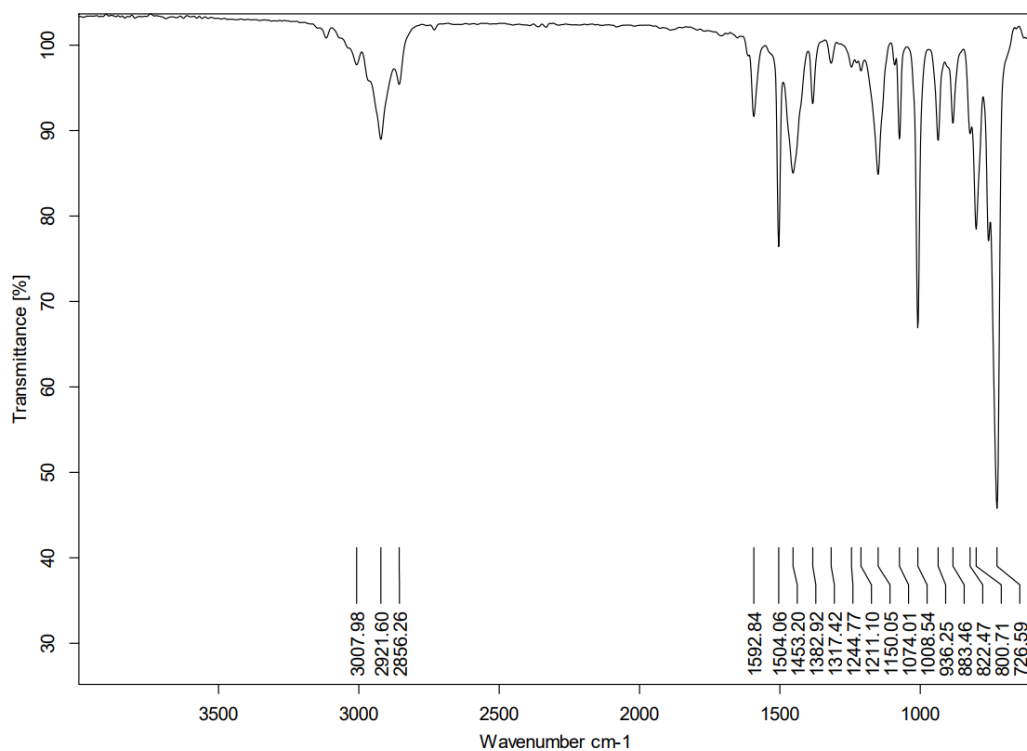


Figure S37. The FTIR spectrum of **2e**.

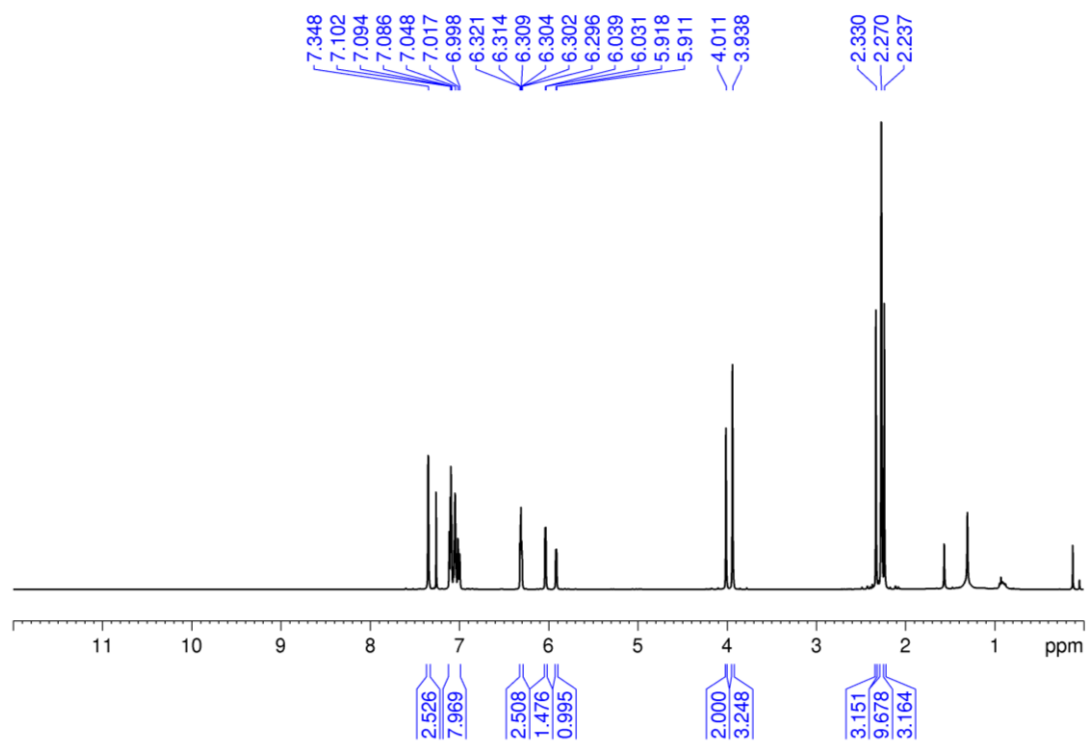


Figure S38. The ^1H -NMR spectrum of **2e**.

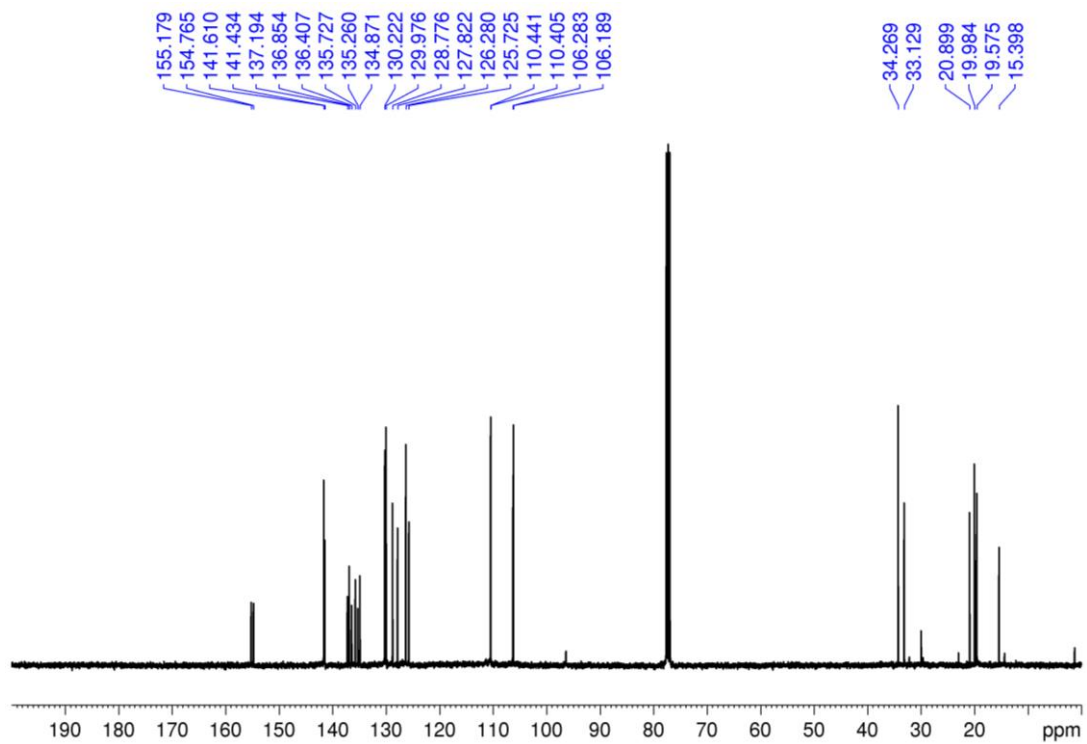


Figure S39. The ^{13}C -NMR spectrum of **2e**.

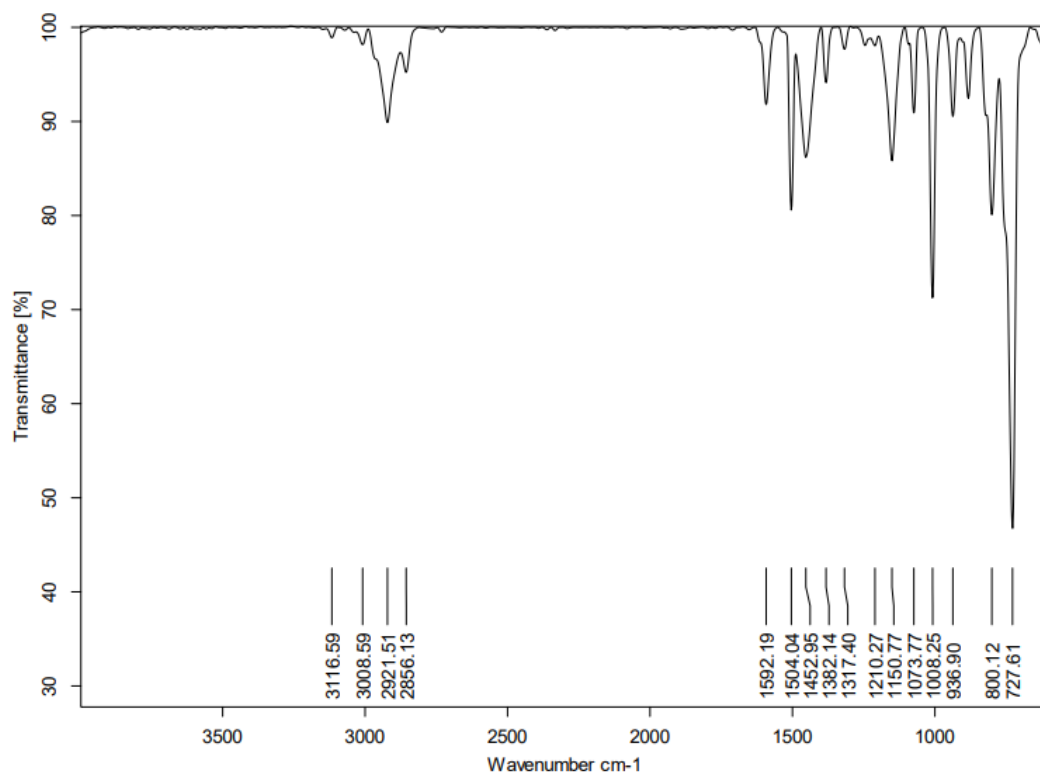


Figure S40. The FTIR spectrum of **2f**.

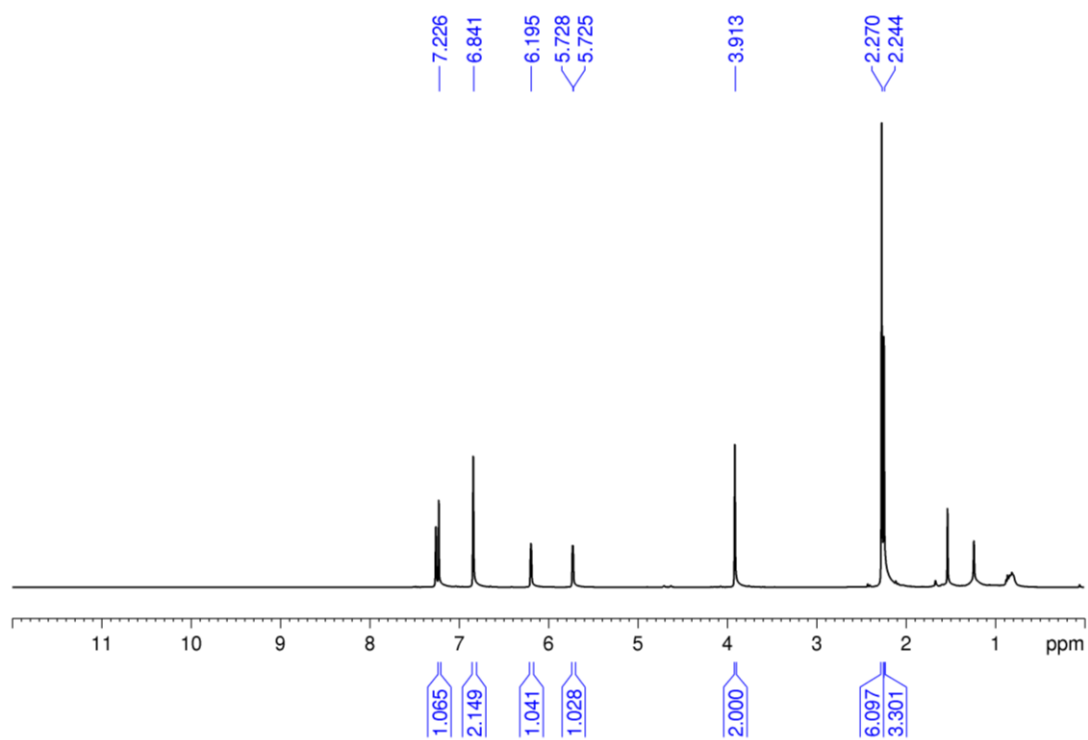


Figure S41. The ¹H-NMR spectrum of **2f**.

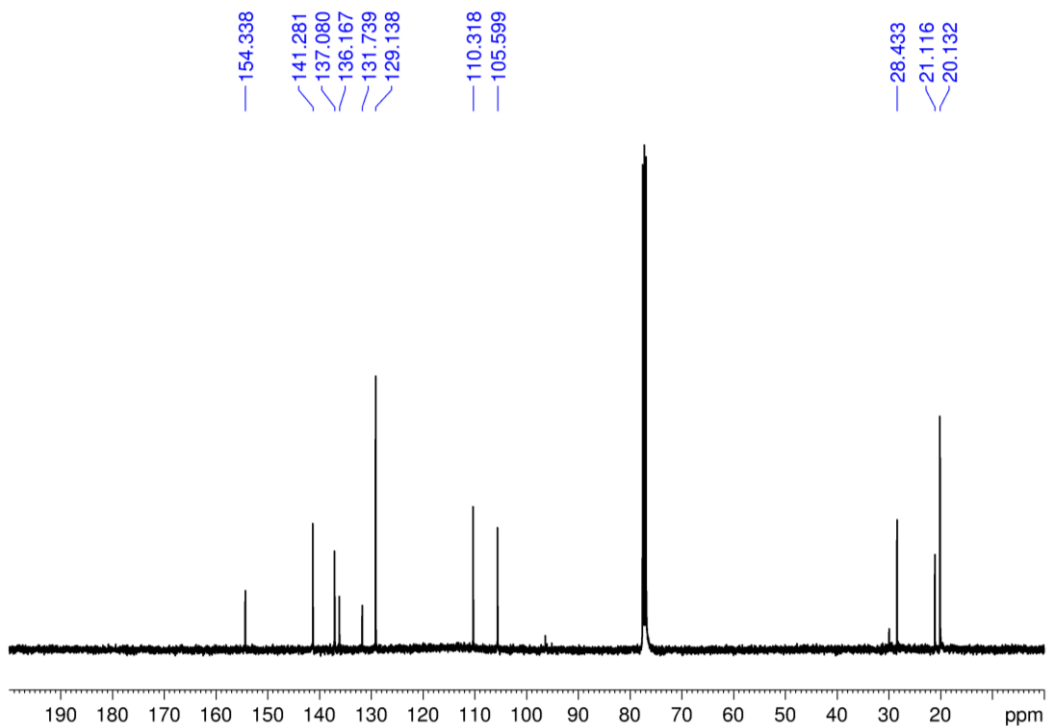


Figure S42. The ^{13}C -NMR spectrum of **2f**.

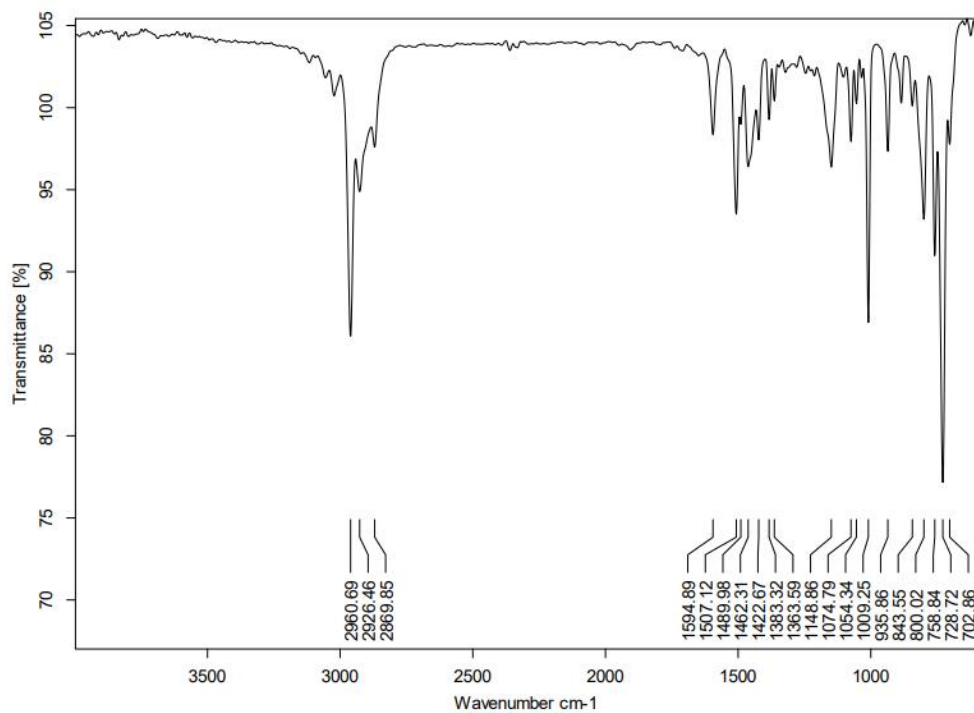


Figure S43. The FTIR spectrum of **2g**.

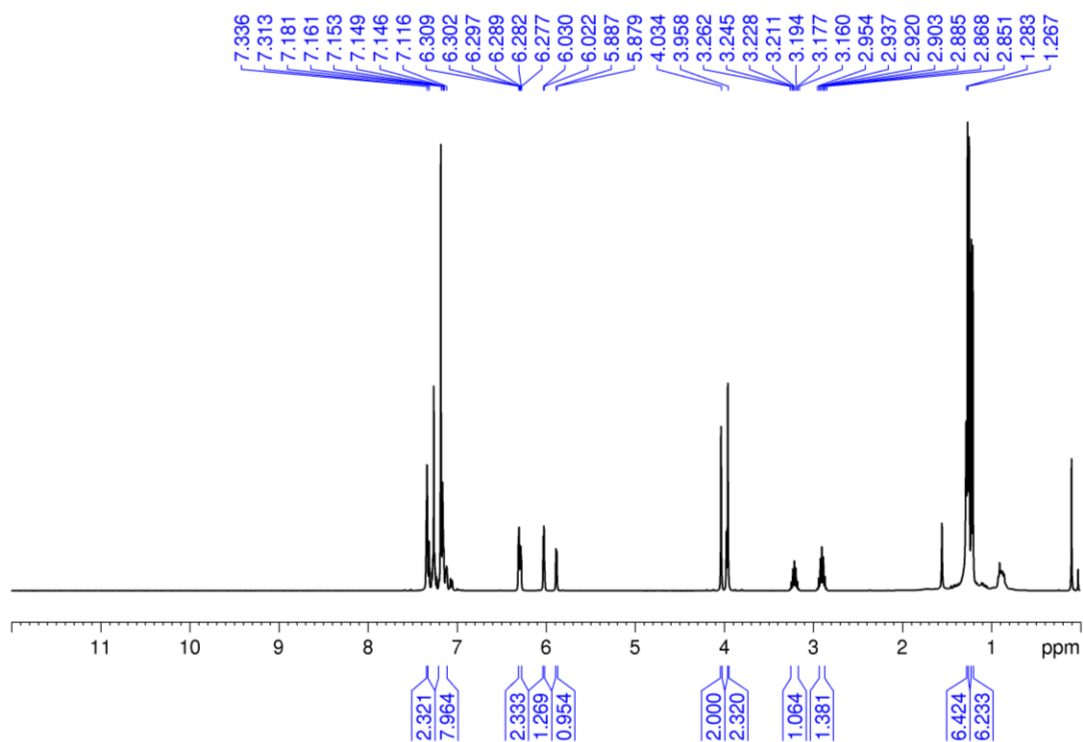


Figure S44. The ^1H -NMR spectrum of **2g**.

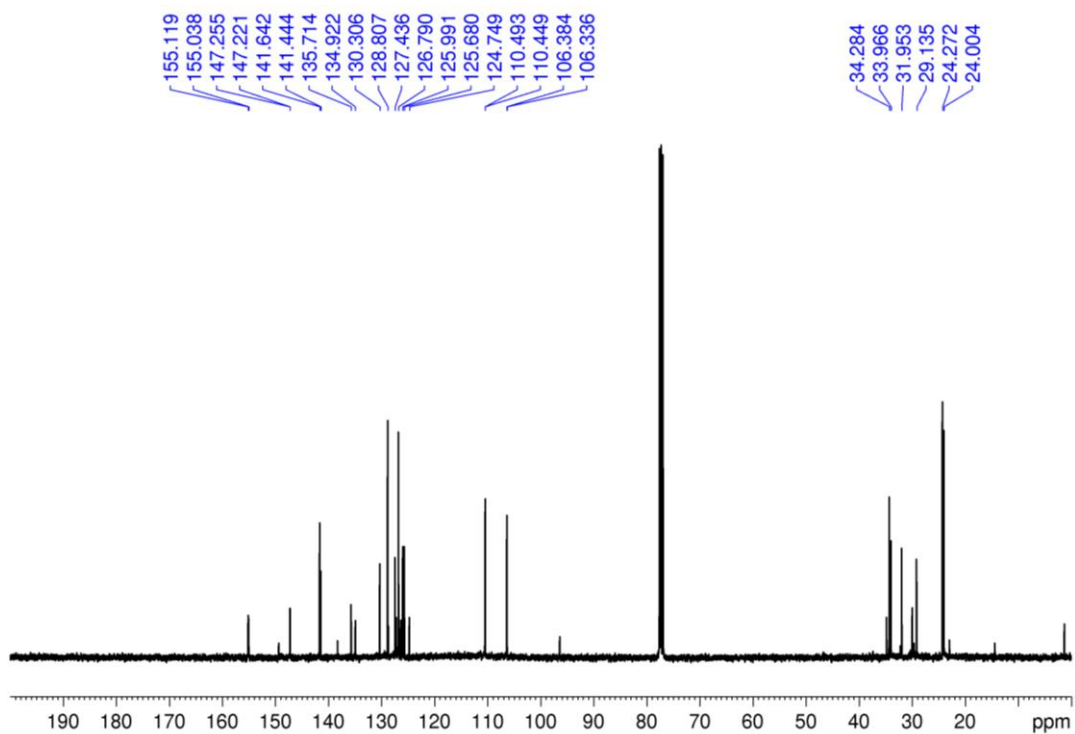


Figure S45. The ^{13}C -NMR spectrum of **2g**.

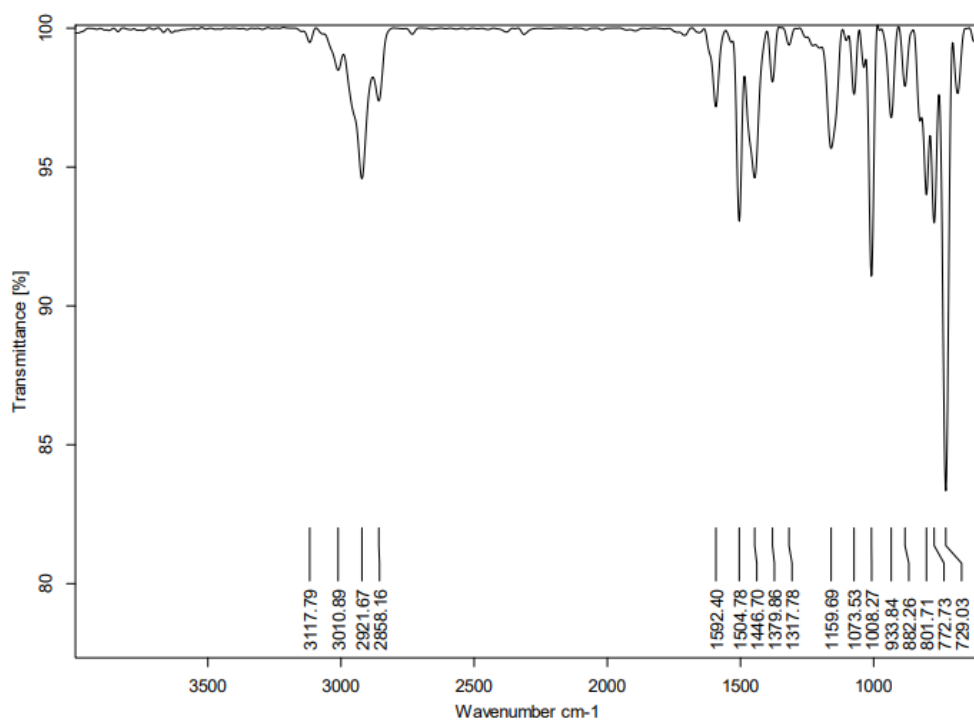


Figure S46. The FTIR spectrum of **2h**.

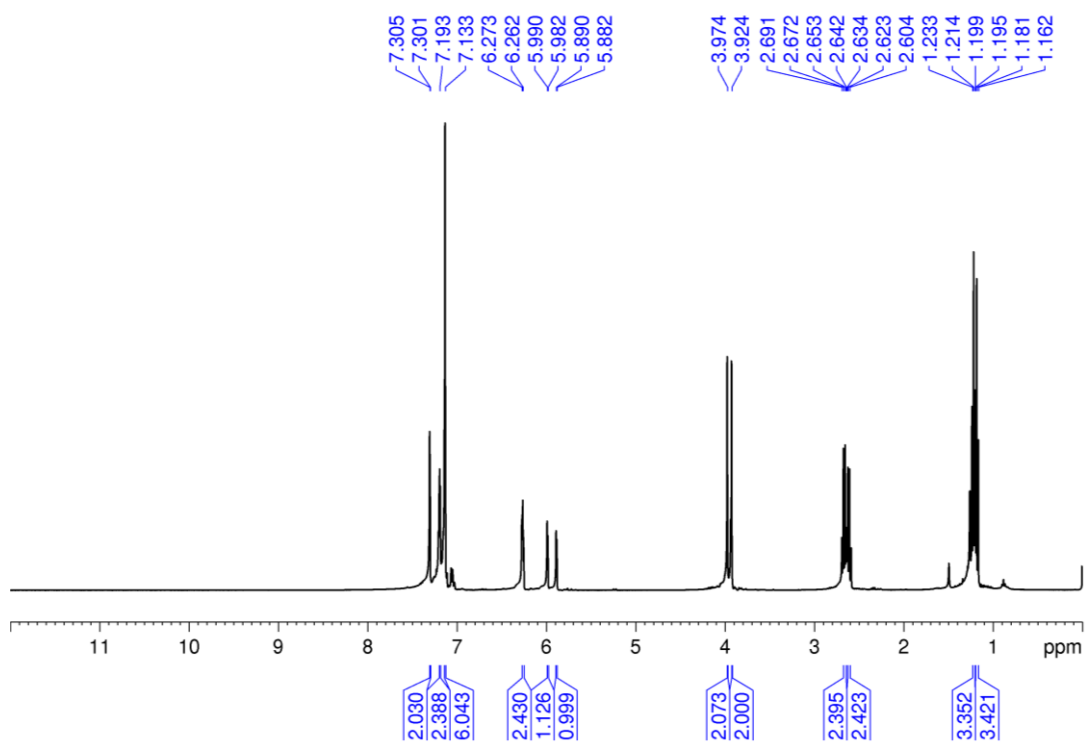


Figure S47. The ¹H-NMR spectrum of **2h**.

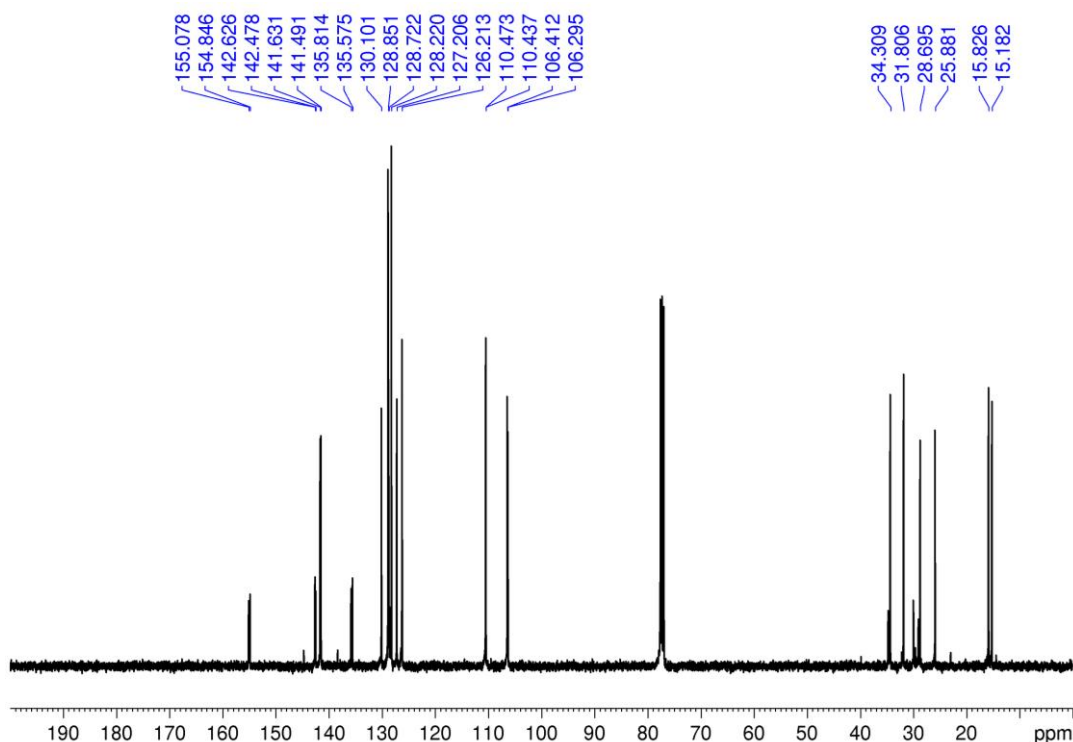


Figure S48. The ^{13}C -NMR spectrum of **2h**.

Characterization of 2-(arylmethyl)-5-methylfurans (AMMFs)

2-(Benzenylmethyl)-5-methylfuran (**3a**): Transparent liquid (0.194, 84%), ^1H -NMR (CDCl_3 , 400 MHz) δ (ppm): 2.30 (s, 3H), 3.97 (s, 2H), 5.91 (s, 2H), 7.27-7.35 (m, 5H). ^{13}C -NMR (CDCl_3 , 100 MHz) δ (ppm): 13.8, 34.8, 106.2, 107.1, 126.6, 128.7, 128.9, 138.7, 151.2, 152.9. FTIR (cm^{-1}): 3115, 3084, 2852, 1596, 1246, 1149, 1075, 1009.

2-(Toluenylmethyl)-5-methylfuran (**3b**): Transparent liquid (0.202 g, 87%), ^1H -NMR (CDCl_3 , 400 MHz) δ (ppm): 2.29 (d, 6H), 2.37 (d, 6H), 3.93 (s, 2H), 3.95 (s, 2H), 5.80 (d, 1H, $J = 2.8$ Hz) 5.90 (s, 3H), 7.17-7.21 (m, 8H). ^{13}C -NMR (CDCl_3 , 100 MHz) δ (ppm): 13.8, 19.6, 21.3, 32.5, 34.4, 34.7, 106.2, 106.2, 106.9, 107.1, 126.3, 126.9, 128.8, 129.3, 129.7, 130.4, 135.6, 136.1, 136.6, 136.8, 151.0, 151.1, 152.5, 153.2. FTIR (cm^{-1}): 3020, 2922, 2859, 1567, 1504, 1221, 1161, 1105, 1020.

2-(p-Xylenylmethyl)-5-methylfuran (**3c**): Transparent liquid (0.198 g, 85%), ^1H -NMR (CDCl_3 , 400 MHz) δ (ppm): 2.28 (s, 3H), 2.30 (s, 3H), 2.32 (s, 3H), 3.88 (s, 2H), 5.76 (d, 1H, $J = 2.8$ Hz), 5.86 (d, 1H, $J = 2.0$ Hz), 6.98 (d, 2H), 7.06 (d, 1H). ^{13}C -NMR (CDCl_3 , 100 MHz) δ (ppm): 13.8, 19.2, 21.2, 32.5, 106.2, 106.9, 127.5, 130.3, 130.5, 133.5, 135.6, 136.6, 150.9, 152.6. FTIR (cm^{-1}): 3005, 2922, 2859, 1613, 1592, 1167, 1075, 1007. HRMS (ESI) calculated for $\text{C}_{14}\text{H}_{17}\text{O} [\text{M}+\text{H}]^+$ 201.1274, found 201.1272.

2-(*m*-Xylenylmethyl)-5-methylfuran (**3d**): Transparent liquid (0.189 g, 81%), ¹H-NMR (CDCl₃, 400 MHz) δ (ppm): 2.26 (s, 6H), 2.29 (d, 10H), 2.35 (s, 6H), 3.87 (s, 3H), 3.94 (s, 2H), 5.58 (d, 1H, *J* = 2.8 Hz), 5.74 (d, 1H, *J* = 2.8 Hz), 5.80 (d, 1H, *J* = 2.0 Hz), 5.84 (d, 1H, *J* = 2.0 Hz), 7.04-7.08 (m, 8H). ¹³C-NMR (CDCl₃, 100 MHz) δ (ppm): 13.8, 19.6, 20.2, 21.2, 28.8, 32.1, 106.1, 106.2, 106.3, 106.9, 126.6, 126.9, 128.3, 129.7, 131.3, 133.8, 135.0, 136.3, 136.4, 137.2, 150.8, 150.9, 152.1, 152.8. FTIR (cm⁻¹): 2960, 2926, 1595, 1507, 1148, 1075, 1009. HRMS (ESI) calculated for C₁₄H₁₇O [M+H]⁺ 201.1274, found 201.1254.

2-(*o*-Xylenylmethyl)-5-methylfuran (**3e**): Transparent liquid (0.193 g, 83%), ¹H-NMR (CDCl₃, 400 MHz) δ (ppm): 2.21 (s, 3H), 2.24 (t, 16H), 2.30 (s, 3H), 3.85 (s, 3H), 3.92 (s, 2H), 5.72 (d, 1H, *J* = 2.8 Hz), 5.83 (d, 3H), 6.96-7.07 (m, 8H). ¹³C-NMR (CDCl₃, 100 MHz) δ (ppm): 13.8, 15.4, 19.6, 20.0, 20.9, 33.2, 34.3, 106.2, 106.2, 106.9, 107.0, 125.7, 126.3, 127.8, 128.6, 129.9, 130.2, 134.7, 135.3, 136.1, 136.7, 136.8, 137.1, 150.9, 151.1, 152.9, 153.3. FTIR (cm⁻¹): 3118, 2921, 2856, 1595, 1151, 1074, 1009. HRMS (ESI) calculated for C₁₄H₁₇O [M+H]⁺ 201.1274, found 201.1246.

2-(Mesitylmethyl)-5-methylfuran (**3f**): Transparent liquid (0.197 g, 84%), ¹H-NMR (CDCl₃, 400 MHz) δ (ppm): 2.27 (s, 3H), 2.30 (s, 6H), 2.33 (s, 3H), 3.92 (s, 2H), 5.61 (s, 1H), 5.81 (s, 1H), 6.89 (s, 2H). ¹³C-NMR (CDCl₃, 100 MHz) δ (ppm): 13.8, 20.1, 21.1, 28.5, 106.1, 106.2, 129.1, 132.0, 136.0, 137.1, 150.7, 152.4. FTIR (cm⁻¹): 3003, 2920, 2860, 1614, 1566, 1166, 1018.

2-(Cumenylmethyl)-5-methylfuran (**3g**): Transparent liquid (0.196 g, 84%), ¹H-NMR (CDCl₃, 400 MHz) δ (ppm): 1.22 (d, 6H, *J* = 6.8 Hz), 1.25 (d, 6H, *J* = 6.8 Hz), 2.27 (s, 6H), 2.85-2.93 (m, 1H), 3.20-3.27 (m, 1H), 3.90 (s, 2H), 3.98 (s, 2H), 5.72 (d, 1H, *J* = 2.4 Hz), 5.84-5.87 (m, 3H), 7.12-7.18 (m, 6H), 7.30-7.33 (m, 2H). ¹³C-NMR (CDCl₃, 100 MHz) δ (ppm): 13.8, 15.2, 15.9, 25.9, 28.7, 30.0, 31.8, 34.4, 96.4, 106.2, 106.2, 107.0, 107.1, 126.2, 127.1, 128.2, 128.7, 128.9, 130.1, 135.9, 136.1, 142.5, 151.0, 151.1, 152.9, 153.2. FTIR (cm⁻¹): 3106, 2923, 2854, 1613, 1567, 1159, 1021. HRMS (ESI) calculated for C₁₅H₁₉O [M+H]⁺ 215.1430, found 215.1433.

2-(Ethylbenzyenylmethyl)-5-methylfuran (**3h**): Transparent liquid (0.194 g, 83%), ¹H-NMR (CDCl₃, 400 MHz) δ (ppm): 1.24 (t, 3H, *J* = 7.6 Hz), 1.27 (t, 3H, *J* = 7.6 Hz), 2.29 (s, 6H), 2.64-2.75 (m, 4H), 3.92 (s, 2H), 3.97 (s, 2H), 5.78 (d, 1H, *J* = 2.8 Hz), 5.89 (m, 3H), 7.18-7.24 (m, 8H). ¹³C-NMR (CDCl₃, 100 MHz) δ (ppm): 13.8, 15.2, 15.9, 25.9, 28.7, 31.8, 34.4, 106.2, 106.2, 107.0, 107.1, 126.2, 127.1, 128.2, 128.6, 128.8, 130.1, 135.9, 136.1, 142.5, 142.5, 151.0,

151.1, 152.9, 153.2. FTIR (cm^{-1}): 3116, 2923, 2860, 1612, 1589, 1167, 1008. HRMS (ESI) calculated for $\text{C}_{14}\text{H}_{17}\text{O}$ $[\text{M}+\text{H}]^+$ 201.1274, found 201.1261.

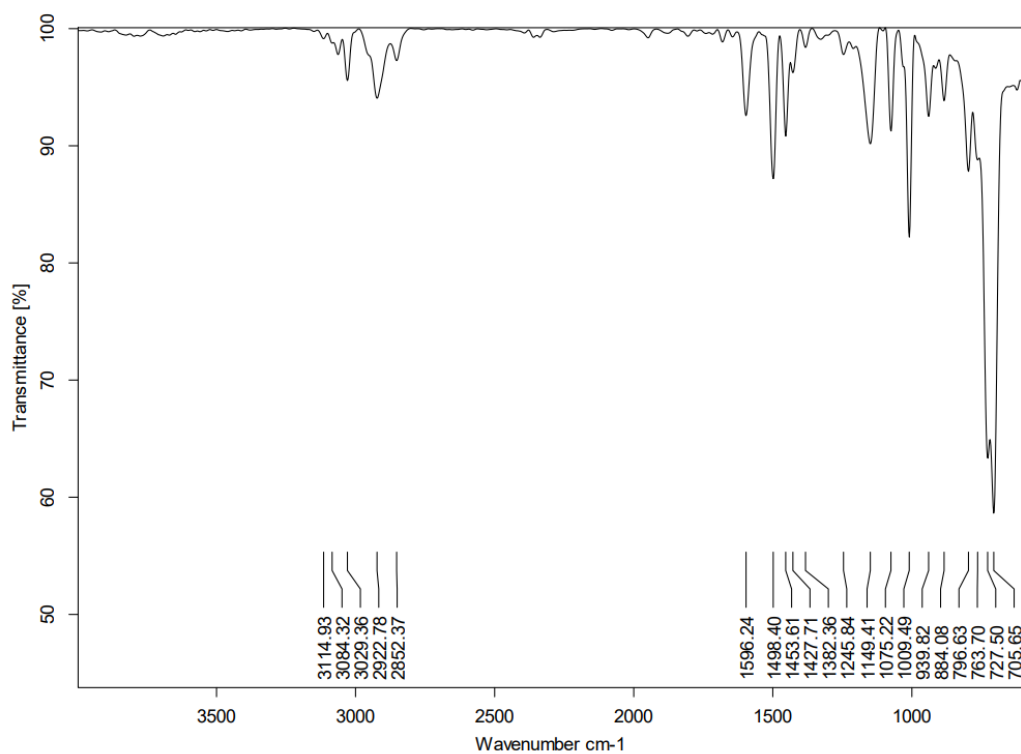


Figure S49. The FTIR Spectrum of 3a.

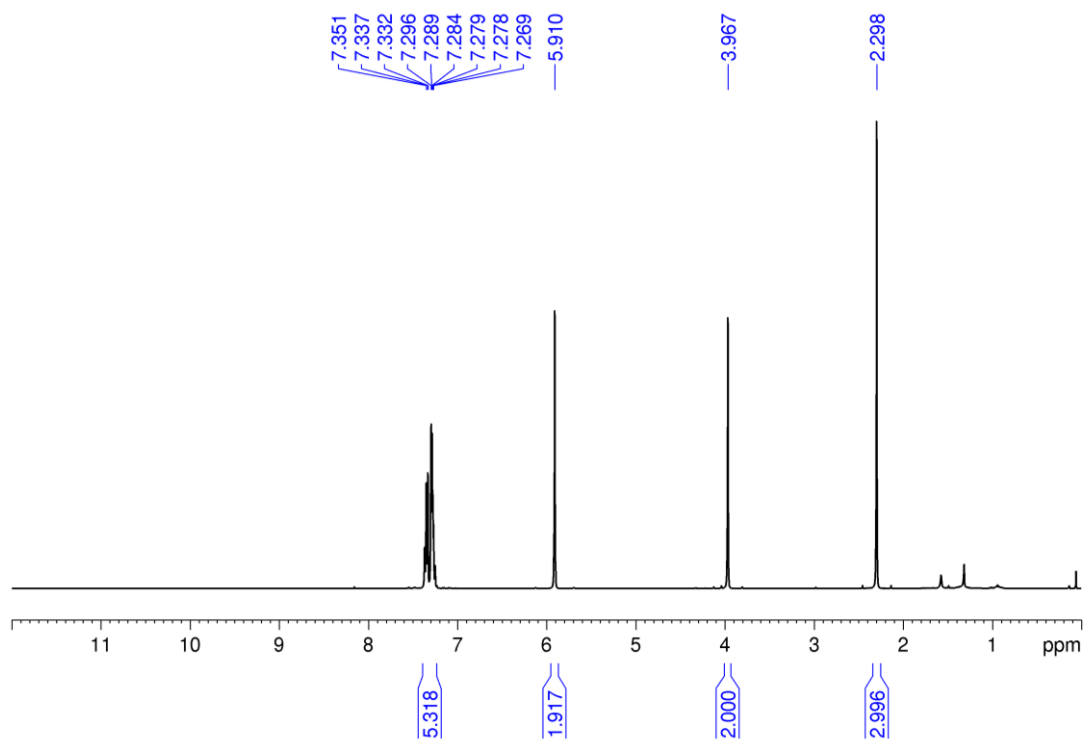


Figure S50. The ^1H -NMR spectrum of 3a.

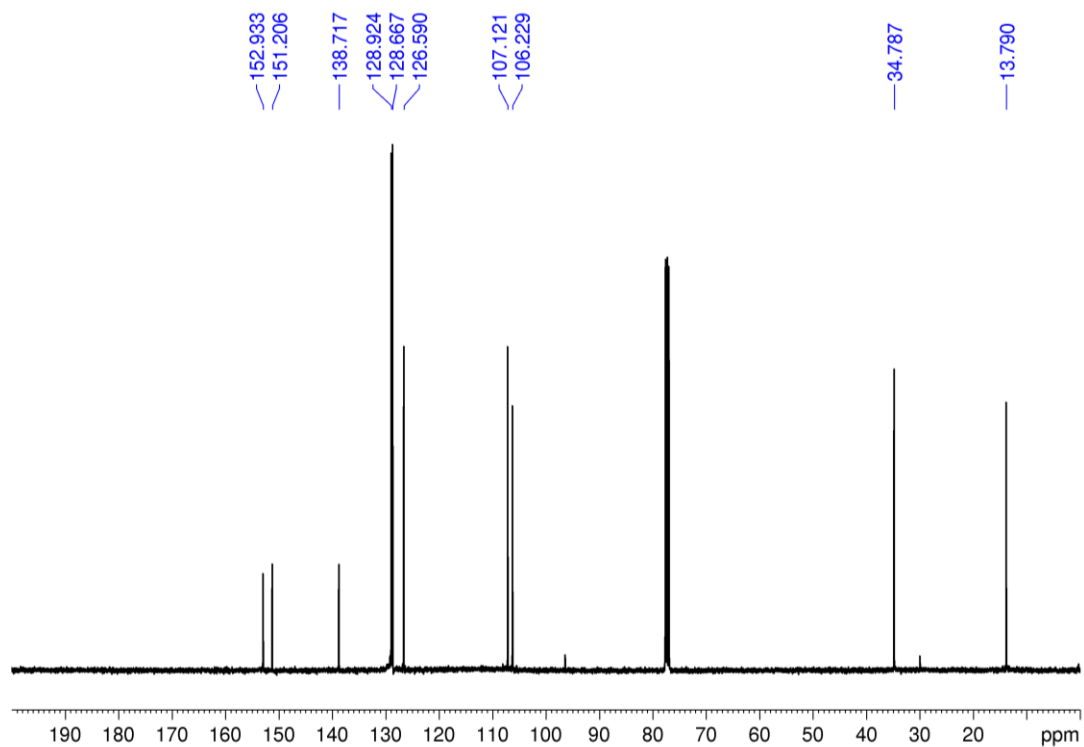


Figure S51. The ¹³C-NMR spectrum of **3a**.

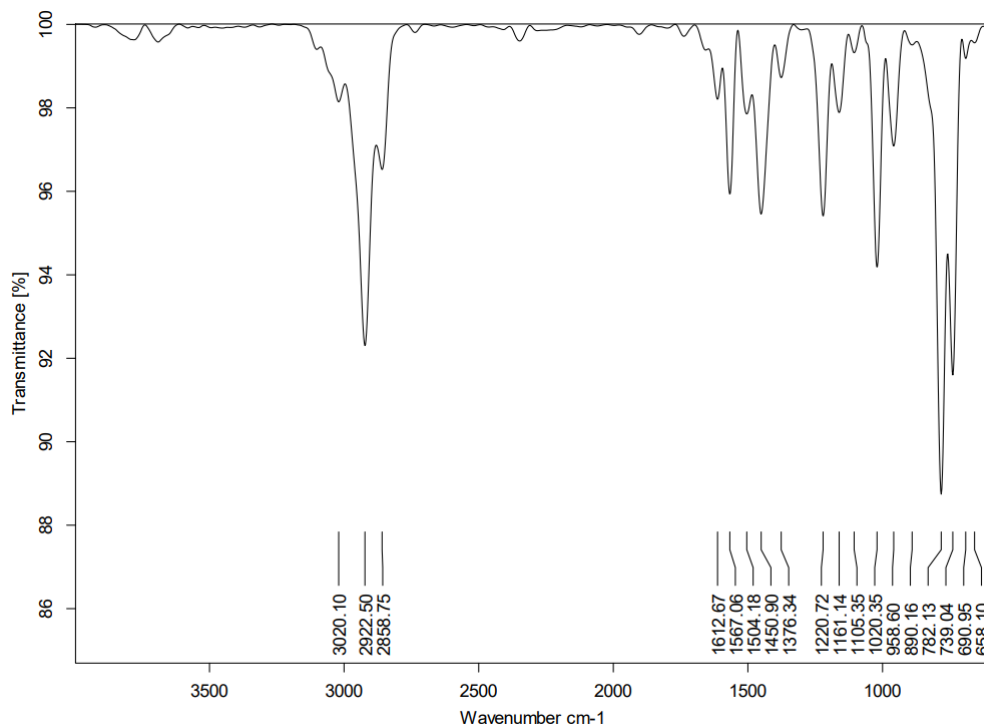


Figure S52. The FTIR Spectrum of **3b**.

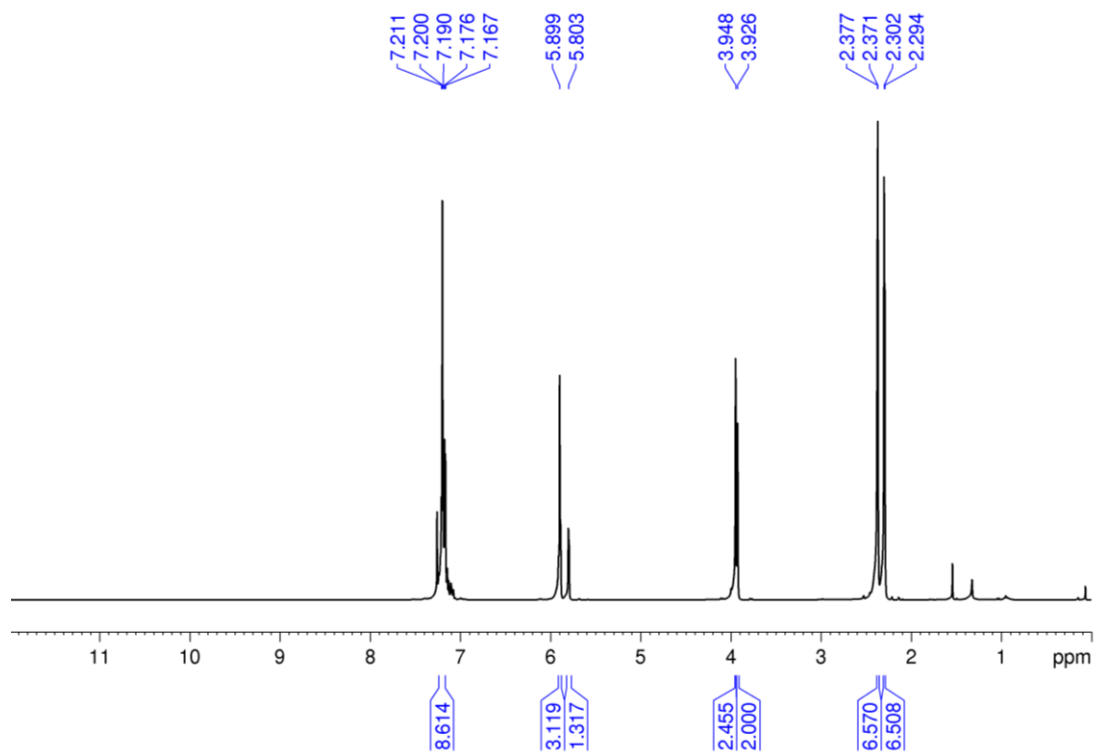


Figure S53. The ^1H -NMR spectrum of **3b**.

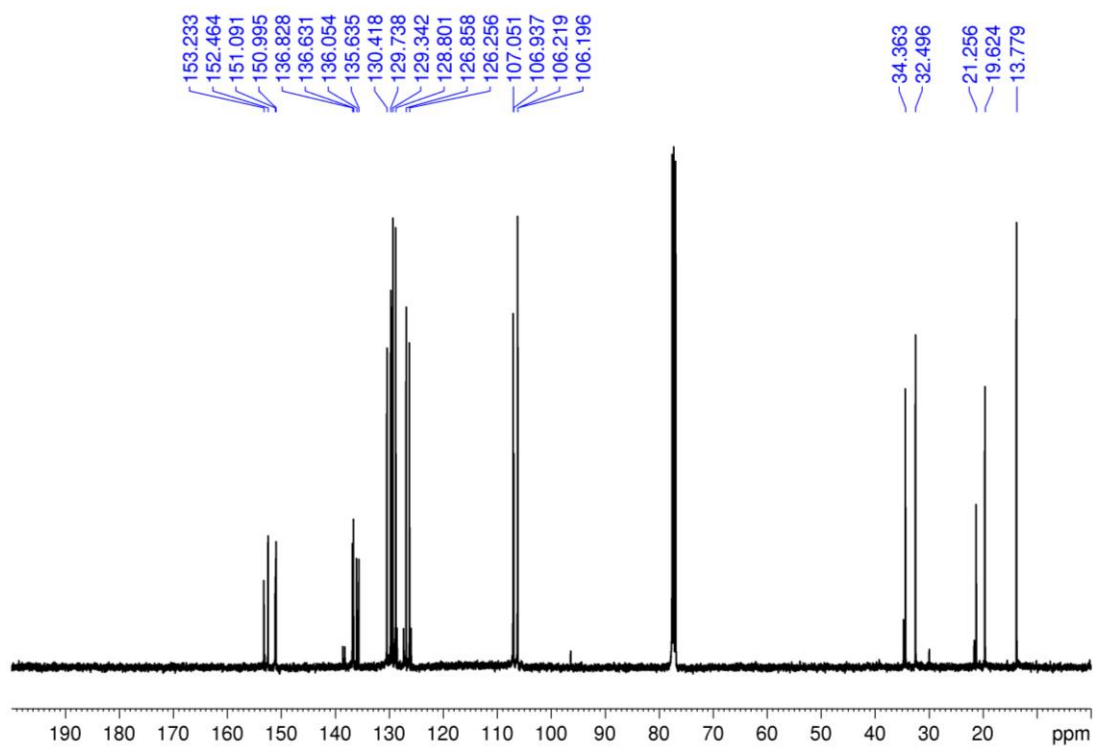


Figure S54. The ^{13}C -NMR spectrum of **3b**.

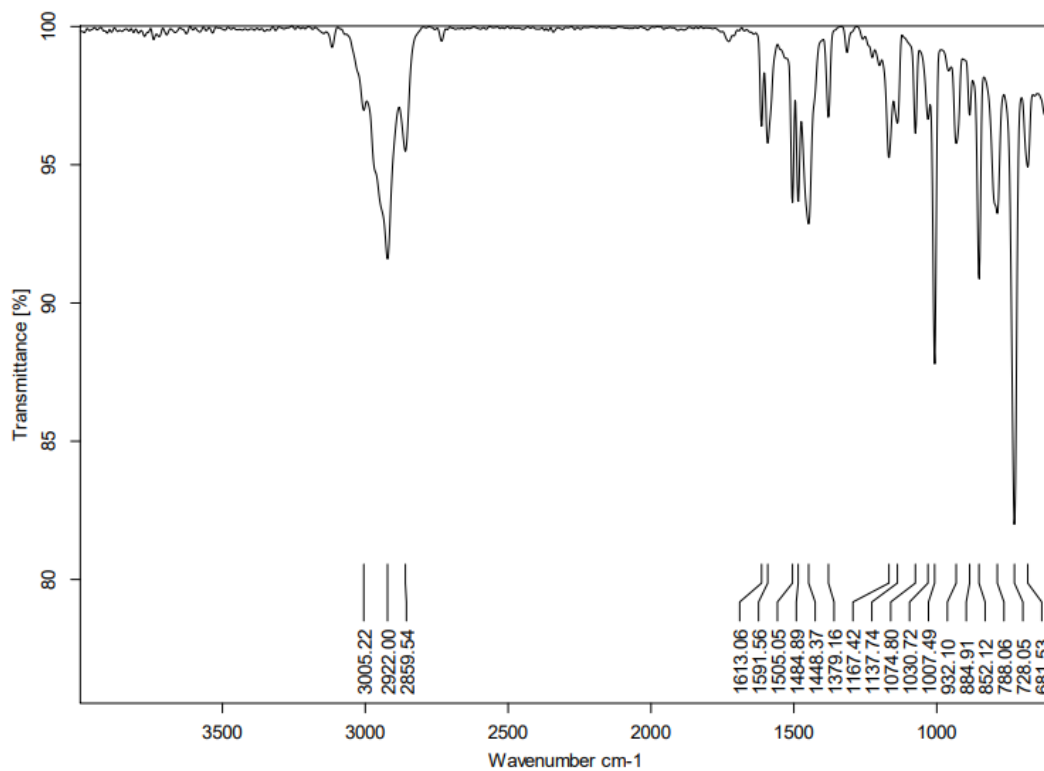


Figure S55. The FTIR Spectrum of 3c.

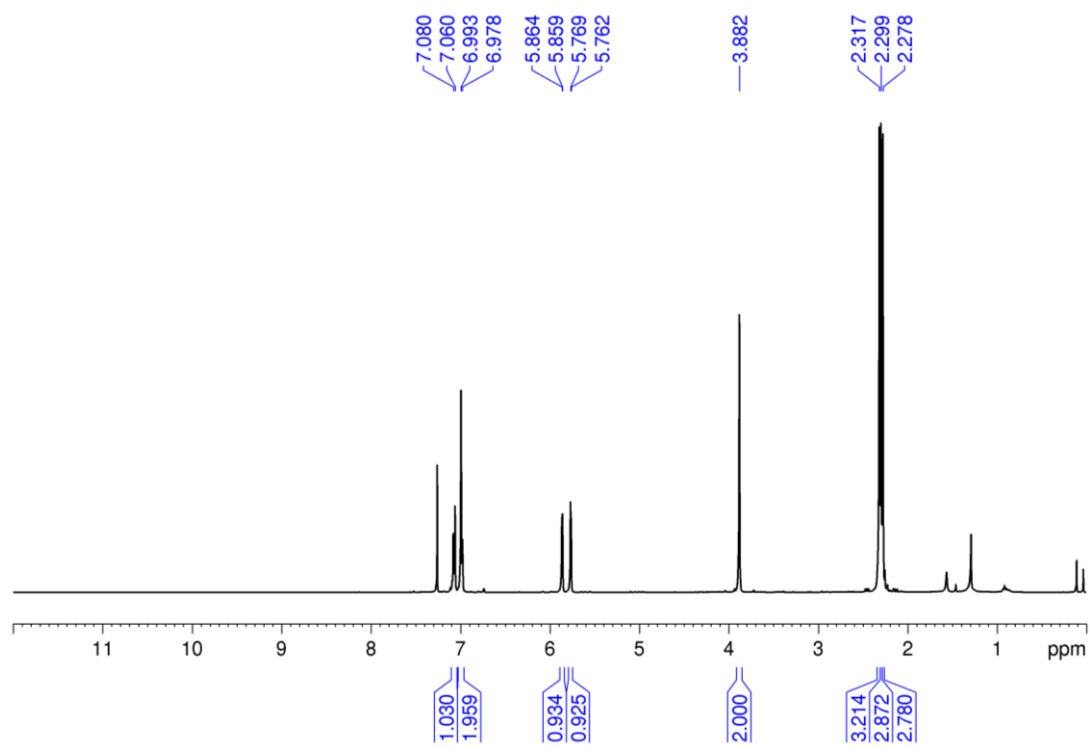


Figure S56. The ¹H-NMR spectrum of 3c.

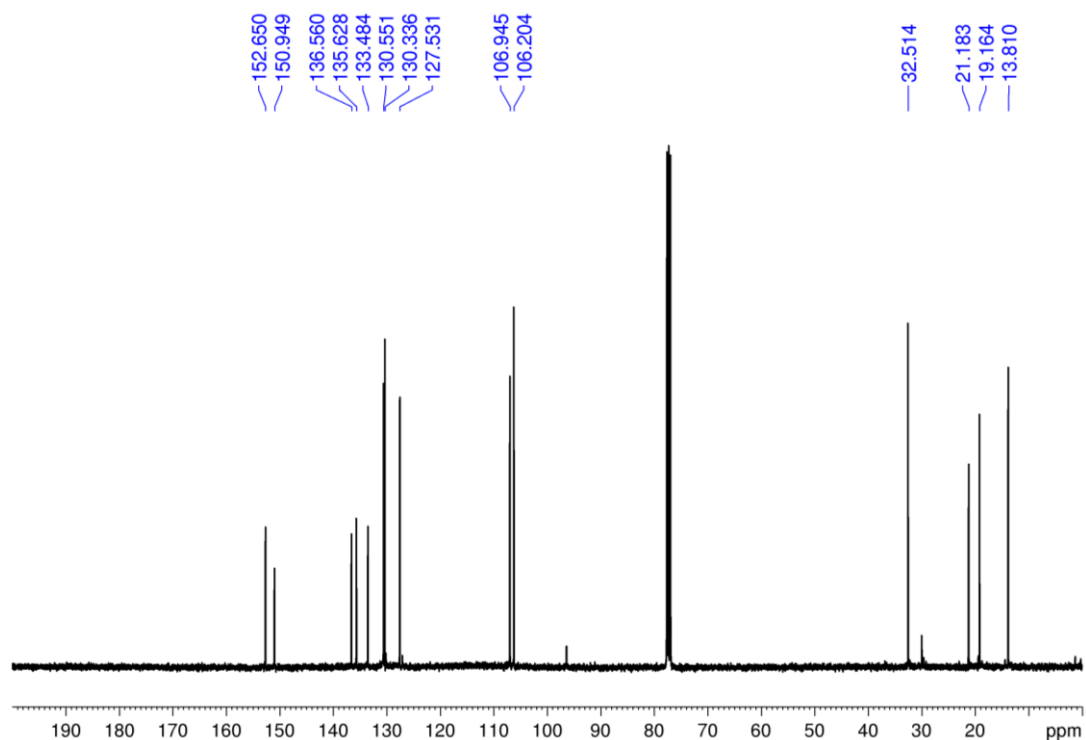


Figure S57. The ^{13}C -NMR spectrum of **3c**.

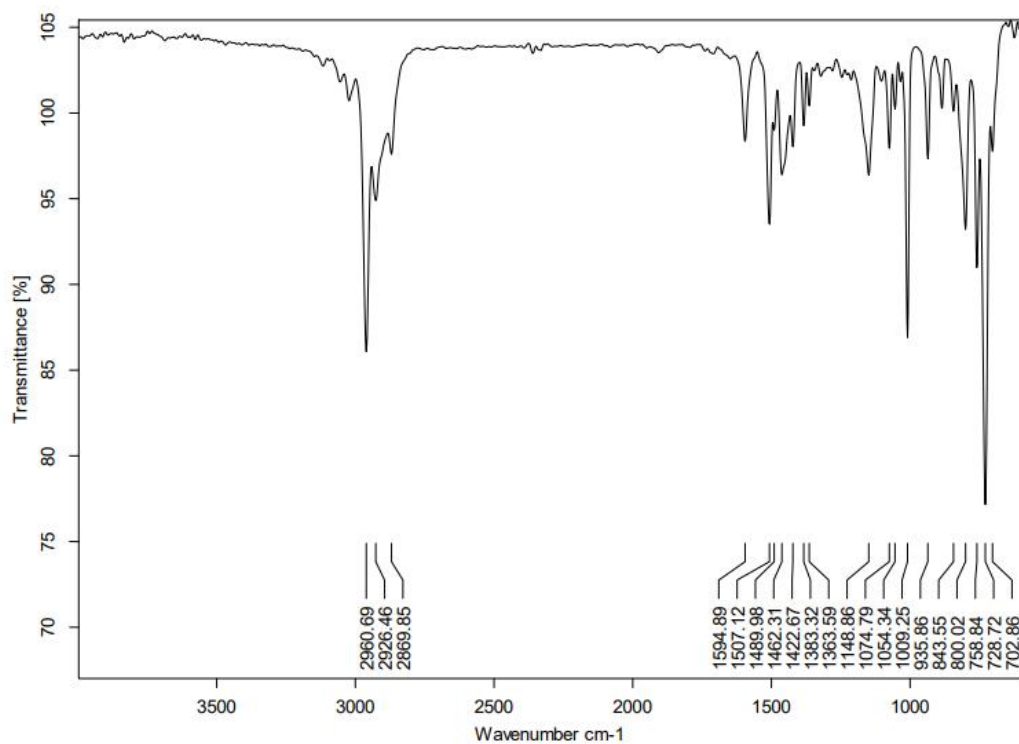


Figure S58. The FTIR Spectrum of **3d**.

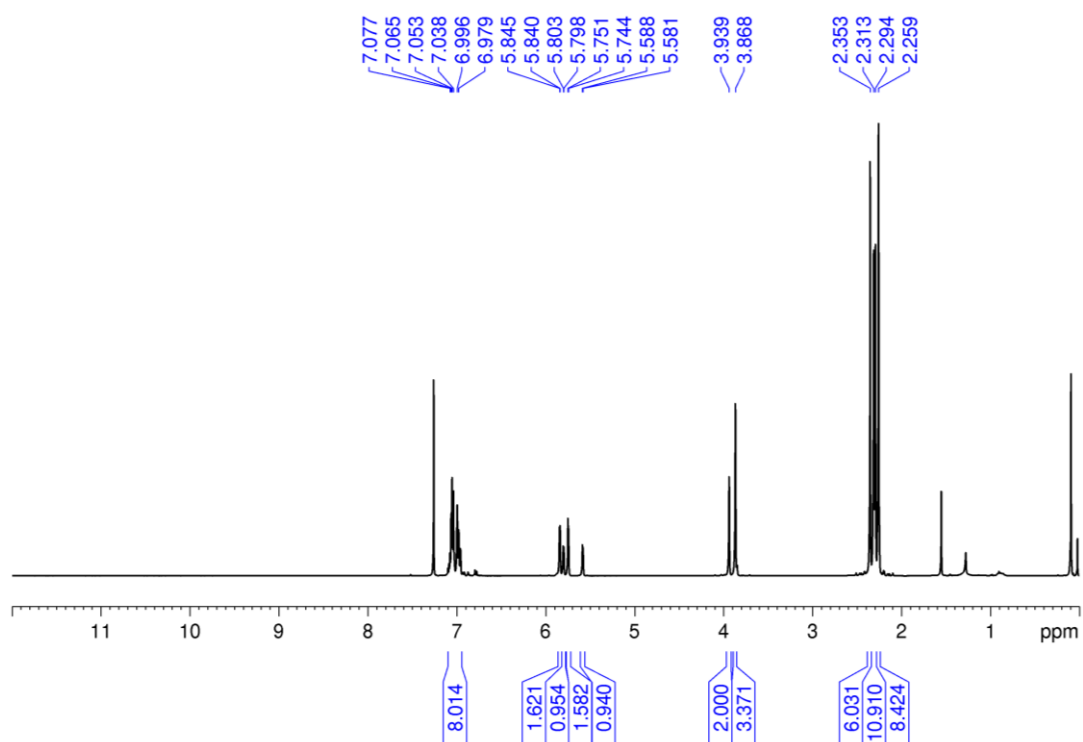


Figure S59. The ¹H-NMR spectrum of 3d.

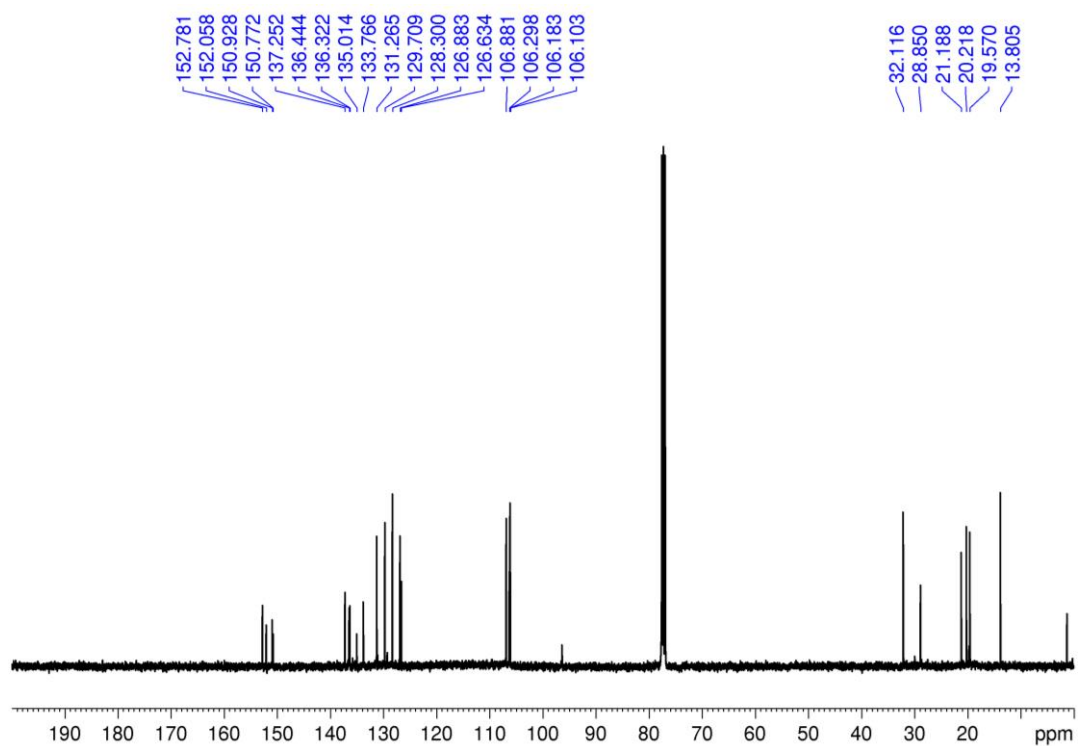


Figure S60. The ¹³C-NMR spectrum of 3d.

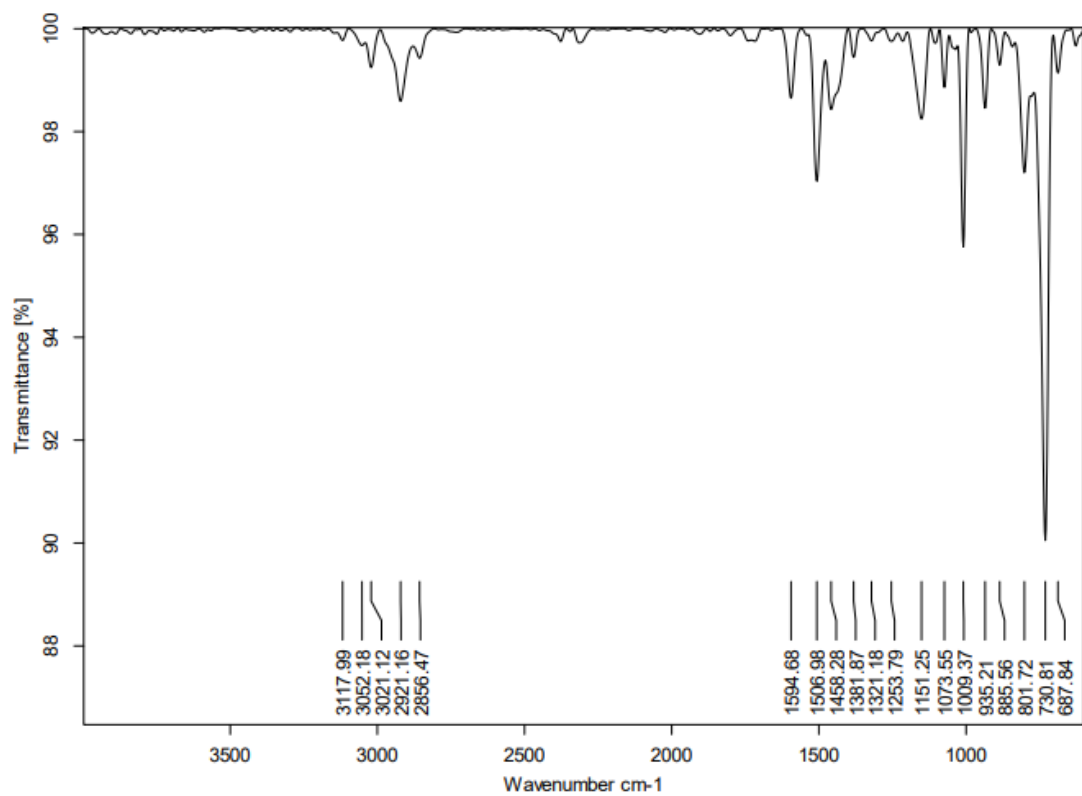


Figure S61. The FTIR Spectrum of 3e.

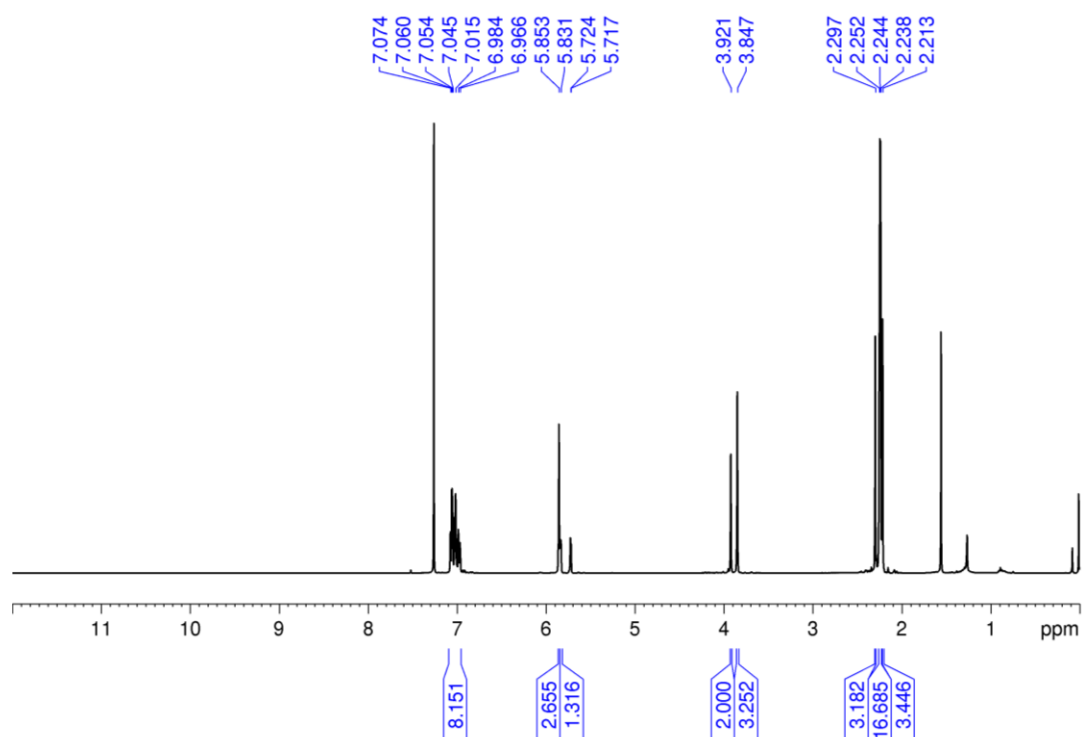


Figure S62. The ¹H-NMR spectrum of 3e.

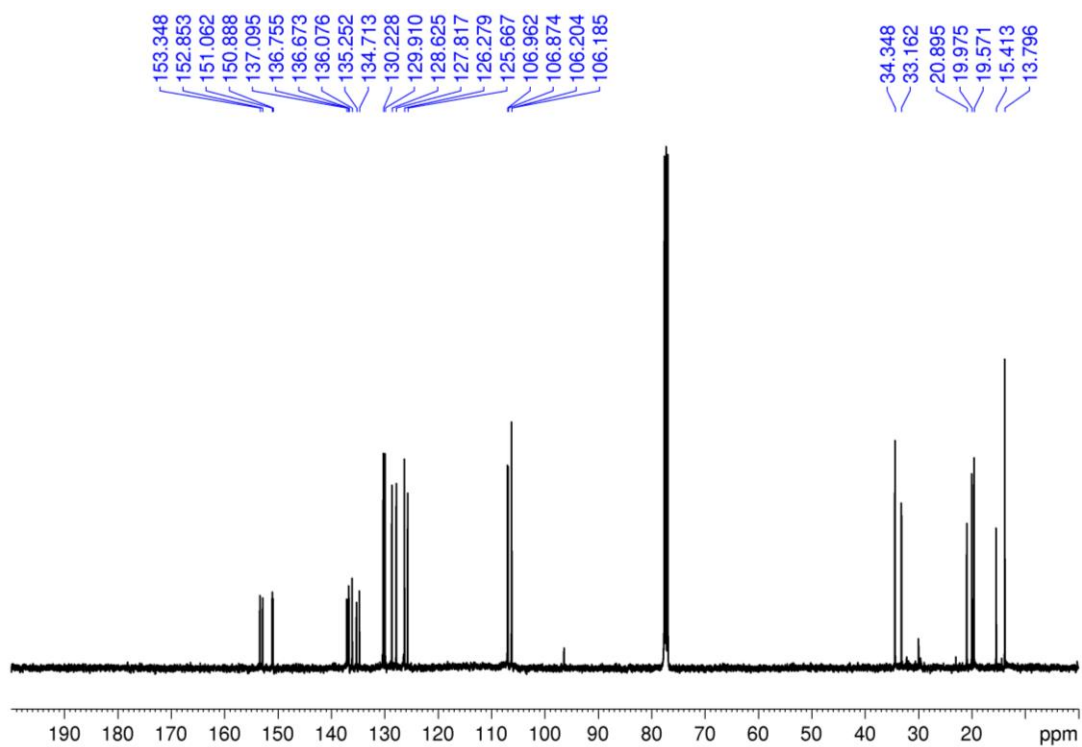


Figure S63. The ^{13}C -NMR spectrum of **3e**.

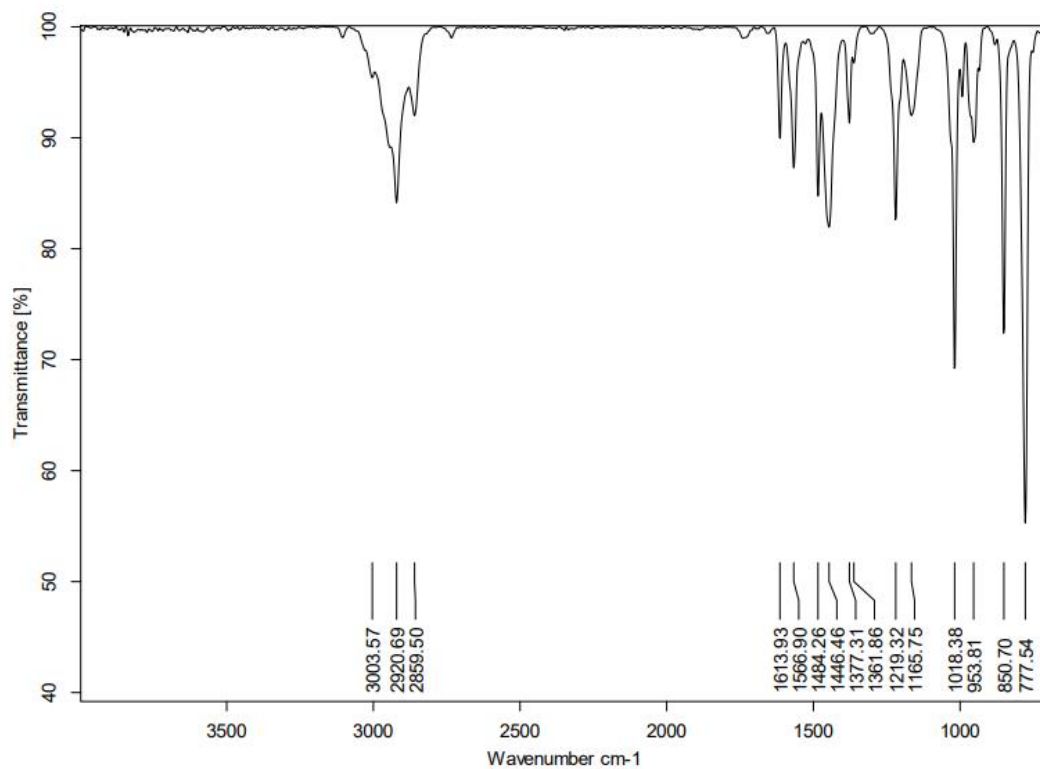


Figure S64. The FTIR Spectrum of **3f**.

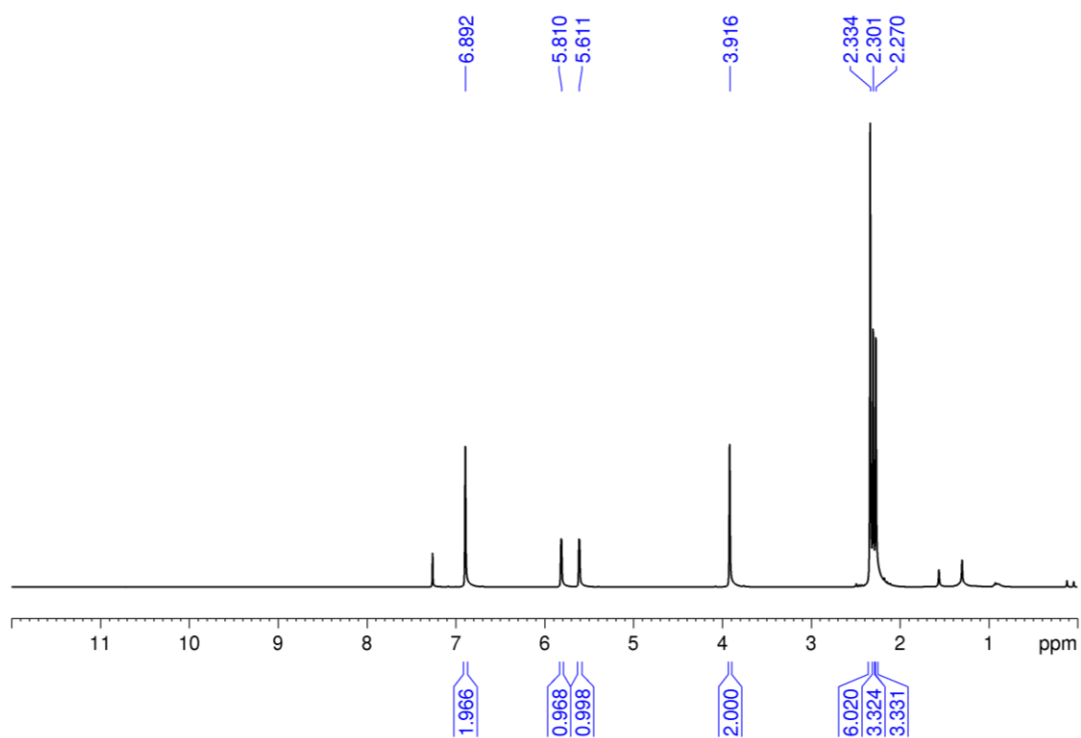


Figure S65. The ^1H -NMR spectrum of **3f**.

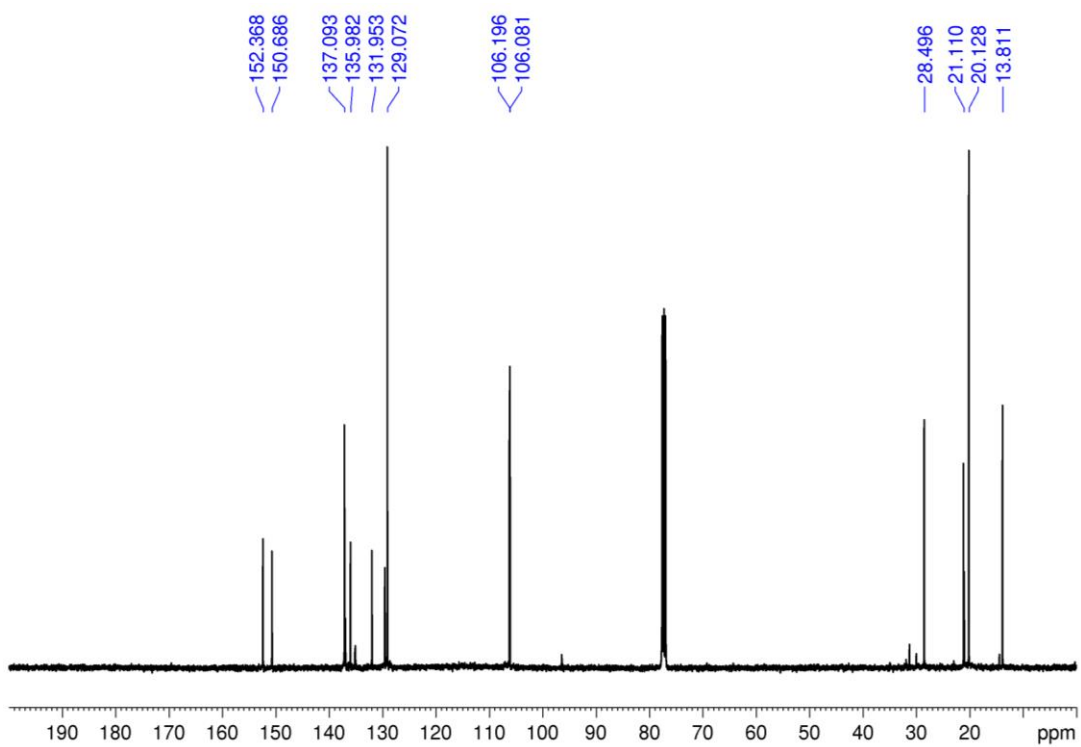


Figure S66. The ^{13}C -NMR spectrum of **3f**.

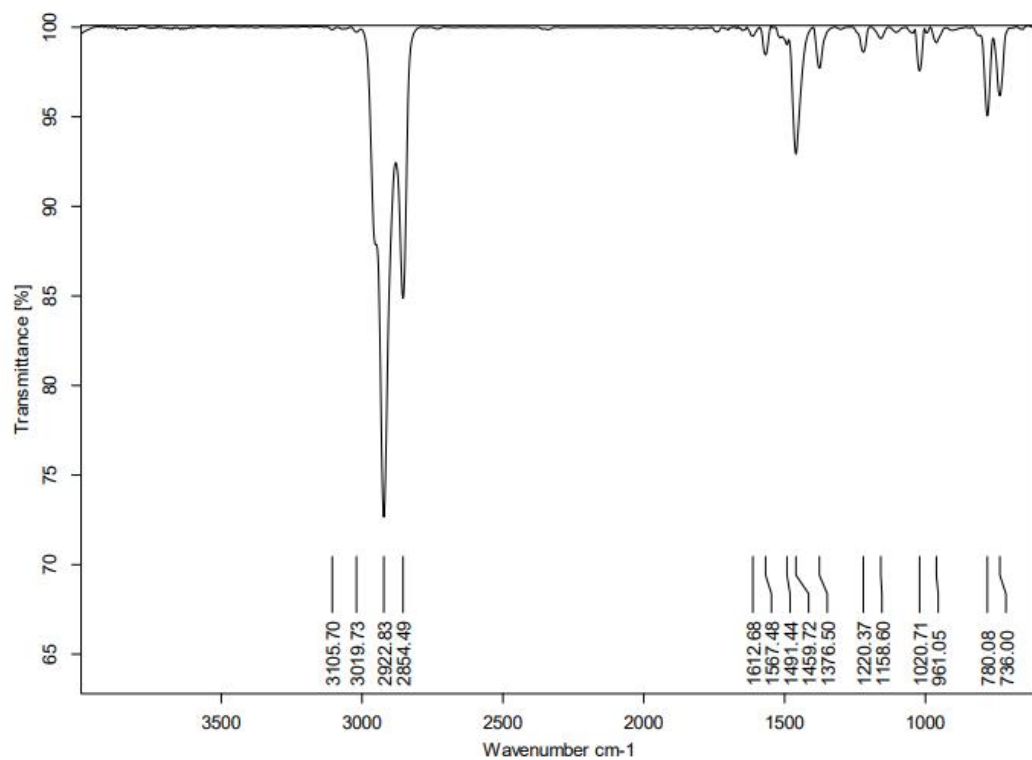


Figure S67. The FTIR Spectrum of **3g**.

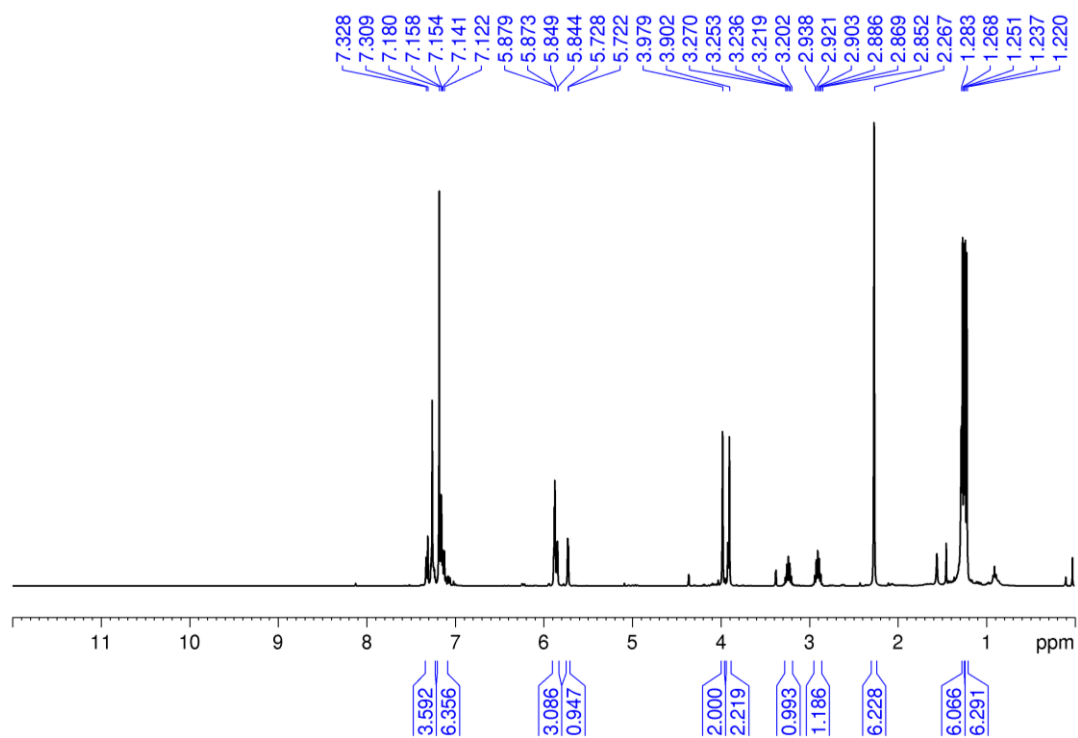


Figure S68. The ¹H-NMR spectrum of **3g**.

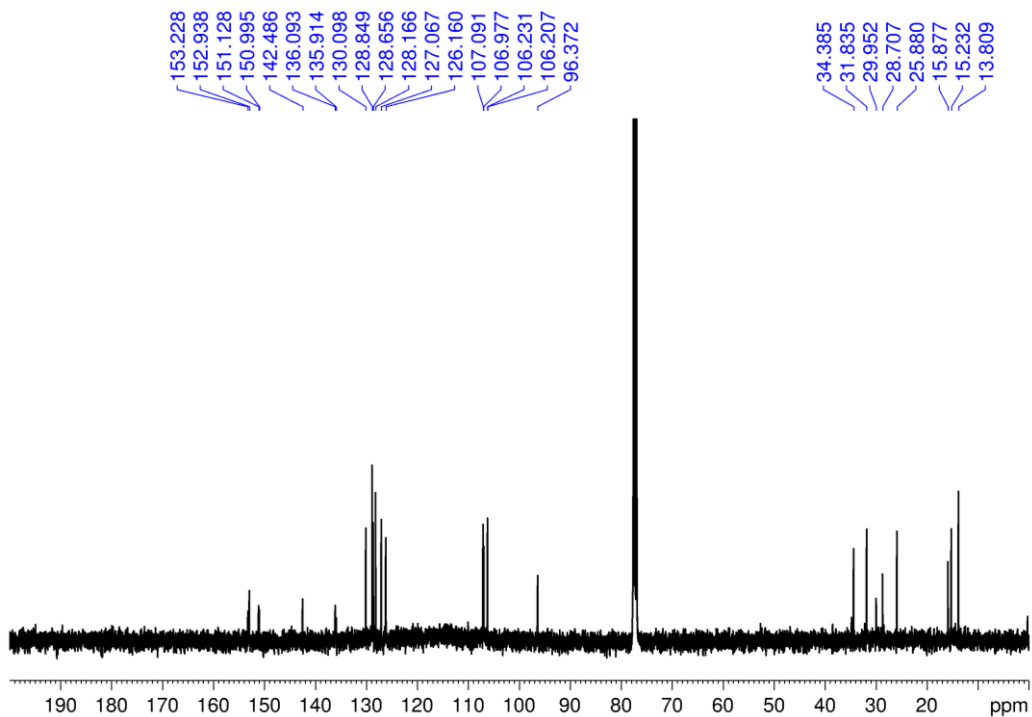


Figure S69. The ^{13}C -NMR spectrum of **3g**.

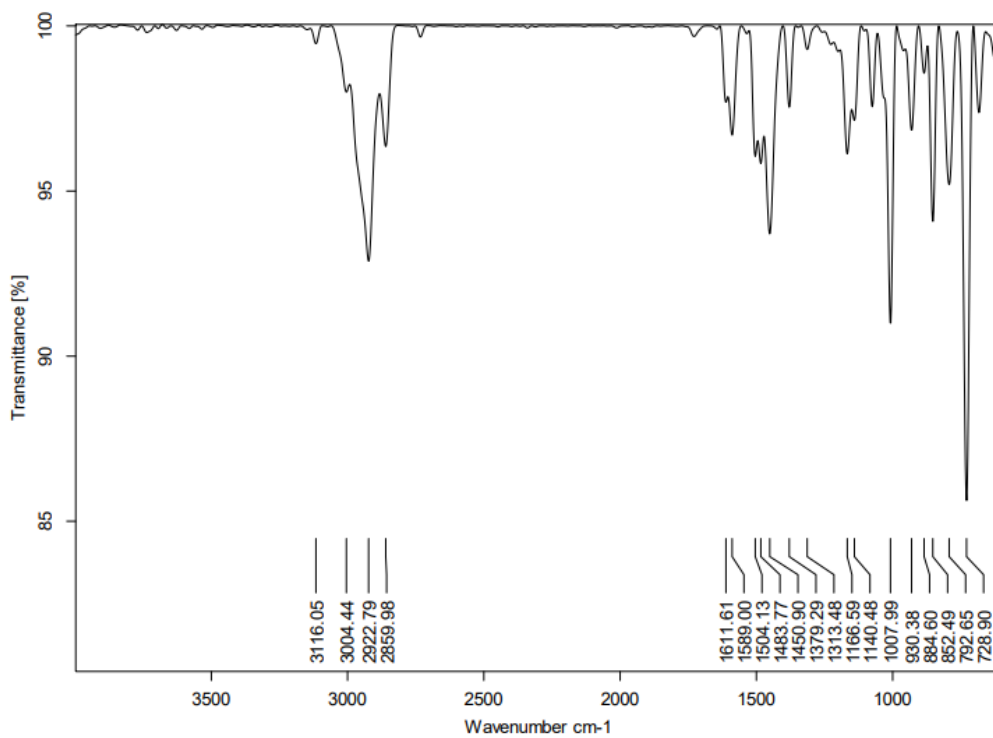


Figure S70. The FTIR Spectrum of **3h**.

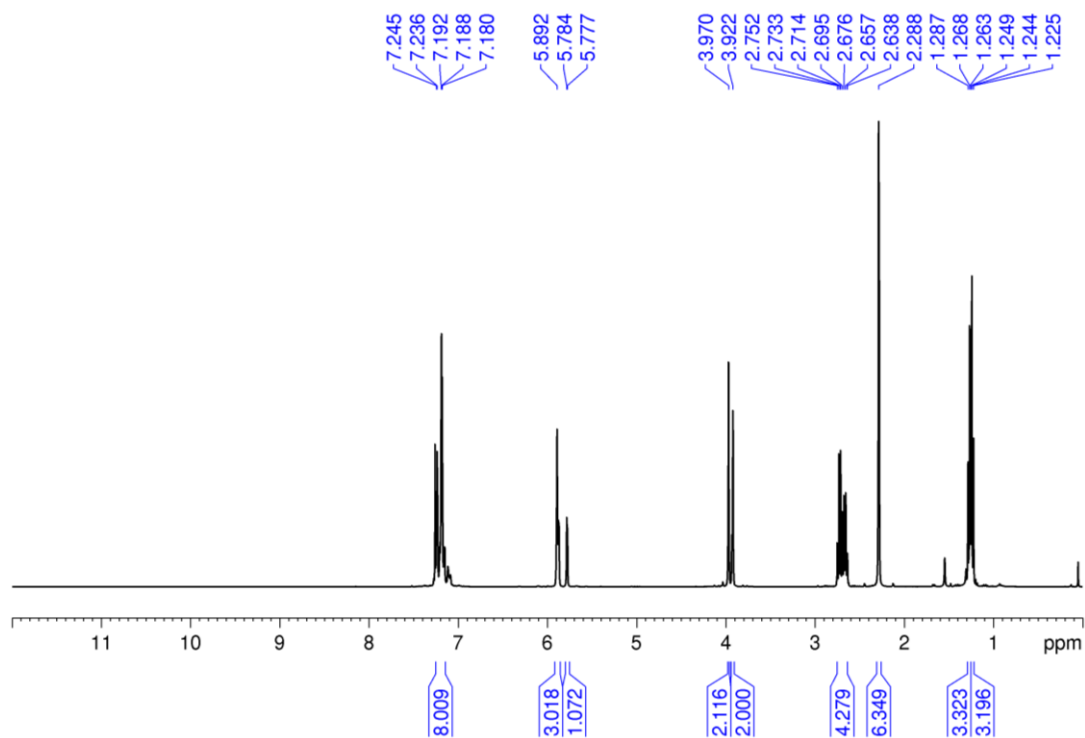


Figure S71. The ^1H -NMR spectrum of **3h**.

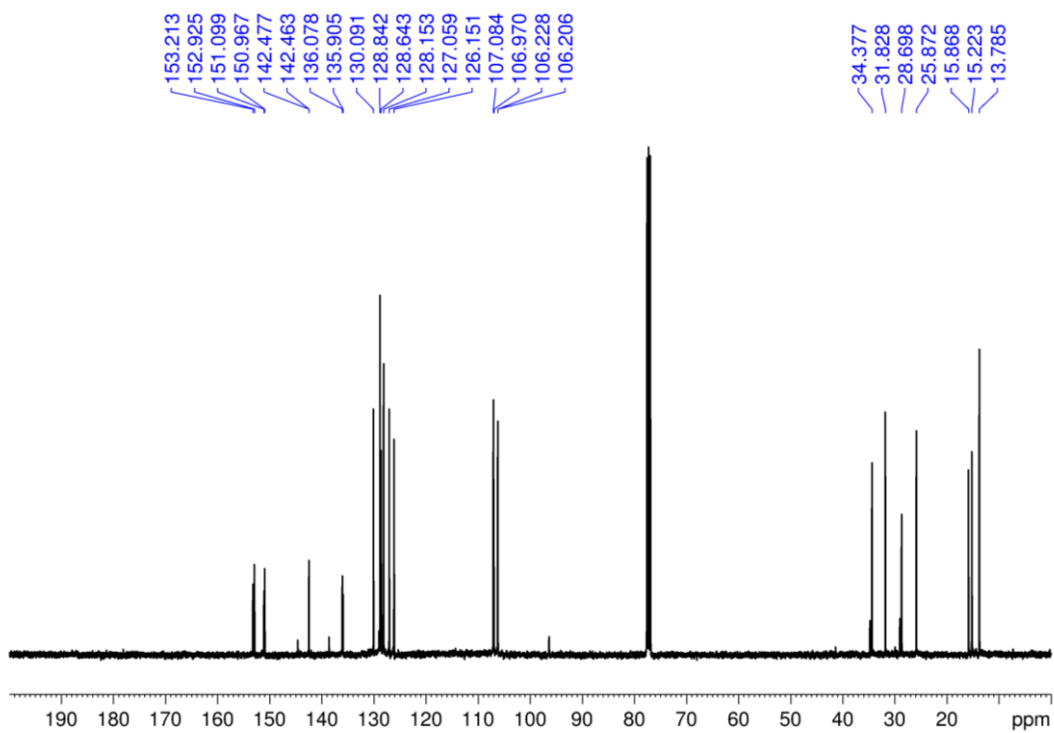


Figure S72. The ^{13}C -NMR spectrum of **3h**.

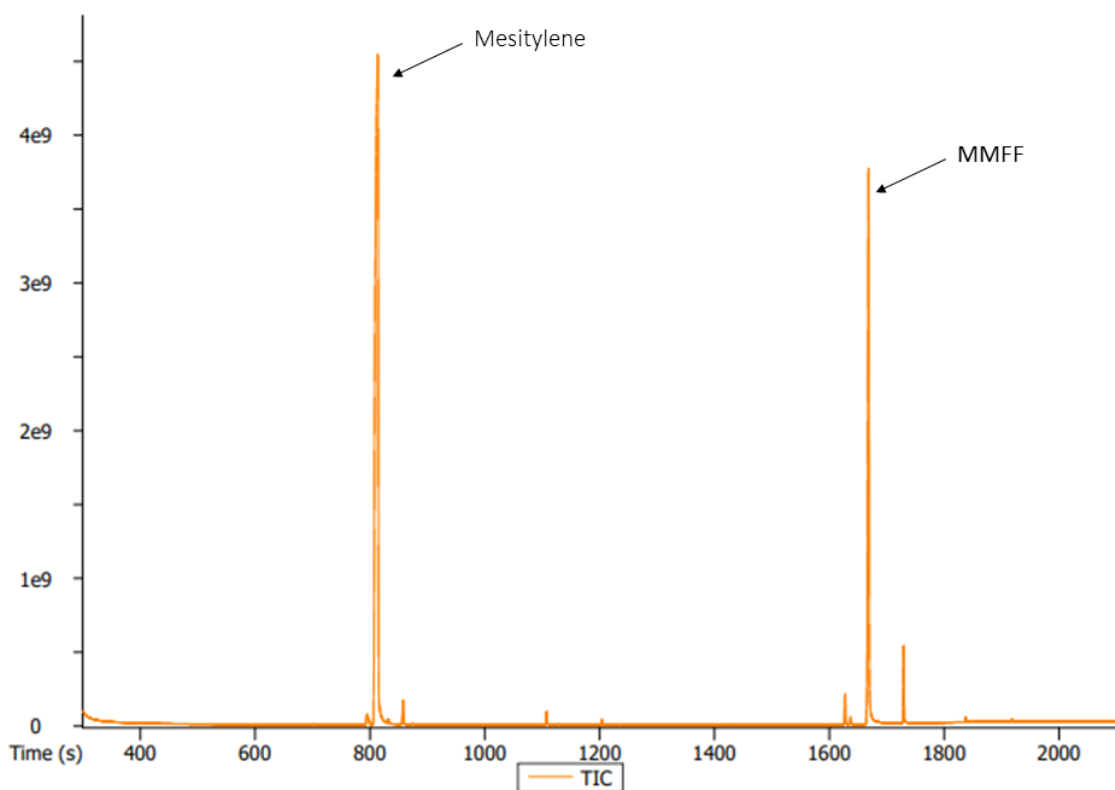


Figure S73. The GC chromatogram of Friedel-Crafts alkylation of AcMF using mesitylene reaction mixture at 120 °C for 3 h.