

Supporting Information

A Two Phase System for Clean and High Yield Synthesis of Furylmethane Derivatives over -SO₃H Functionalized Ionic Liquids

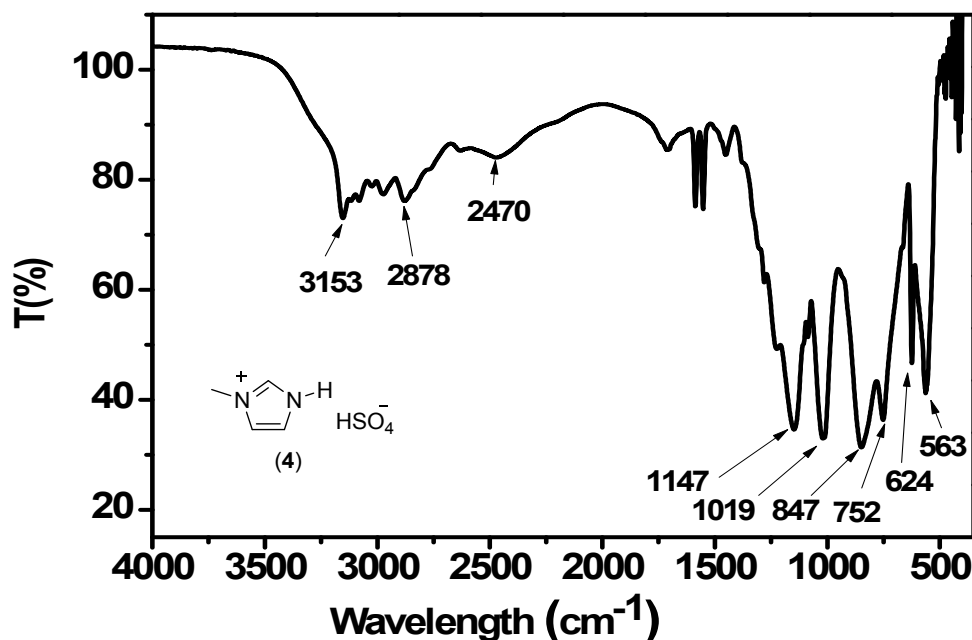
S. H. Shinde and C. V. Rode*

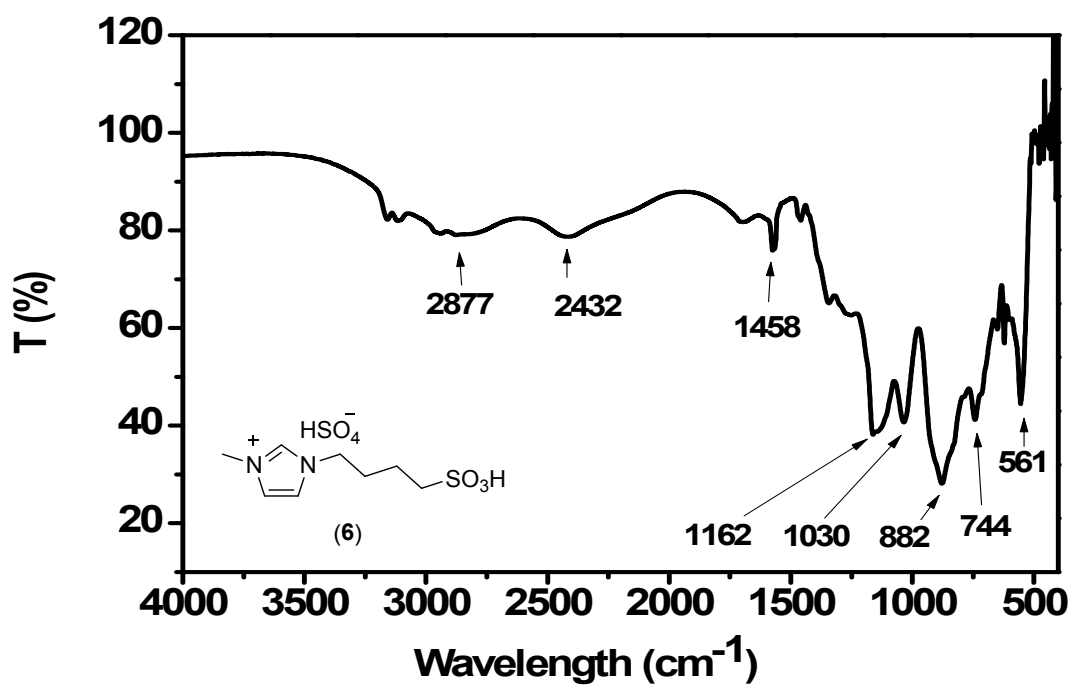
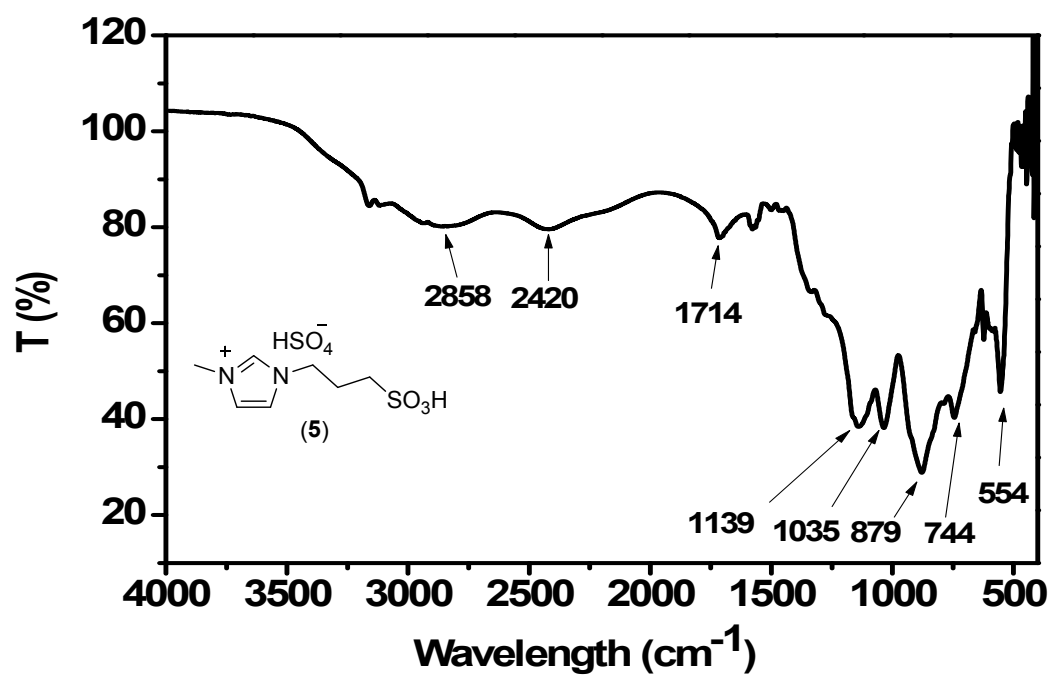
Chemical Engineering and Process Development Division, CSIR-National Chemical Laboratory,
Dr. Homi Bhabha road, Pune-411008

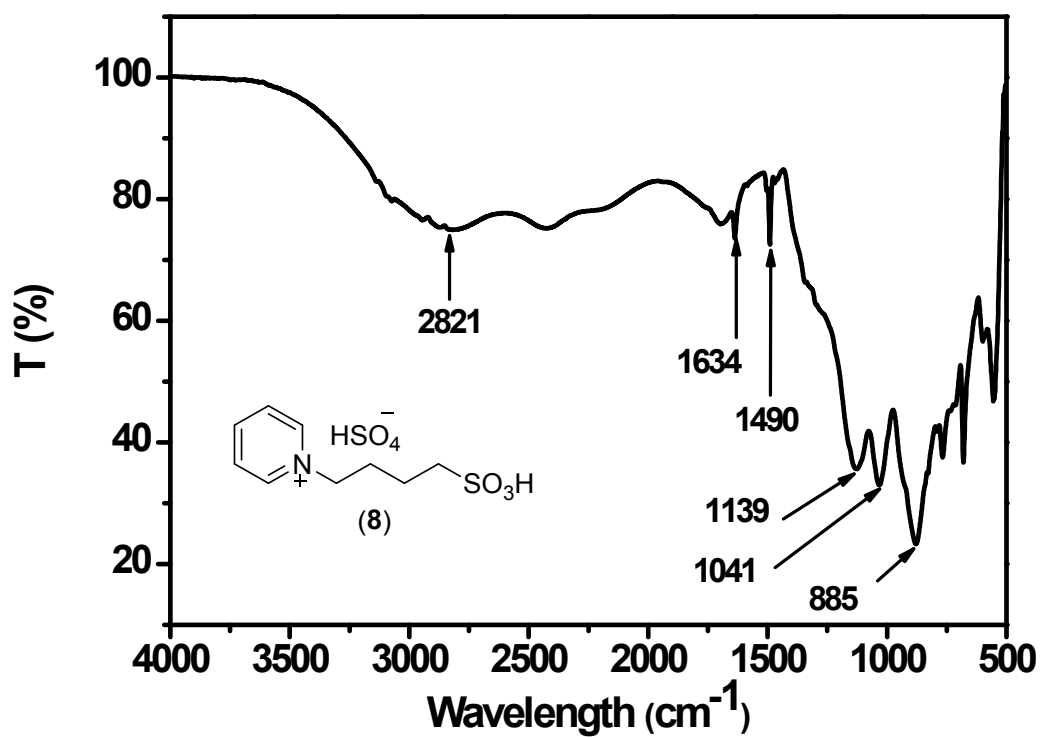
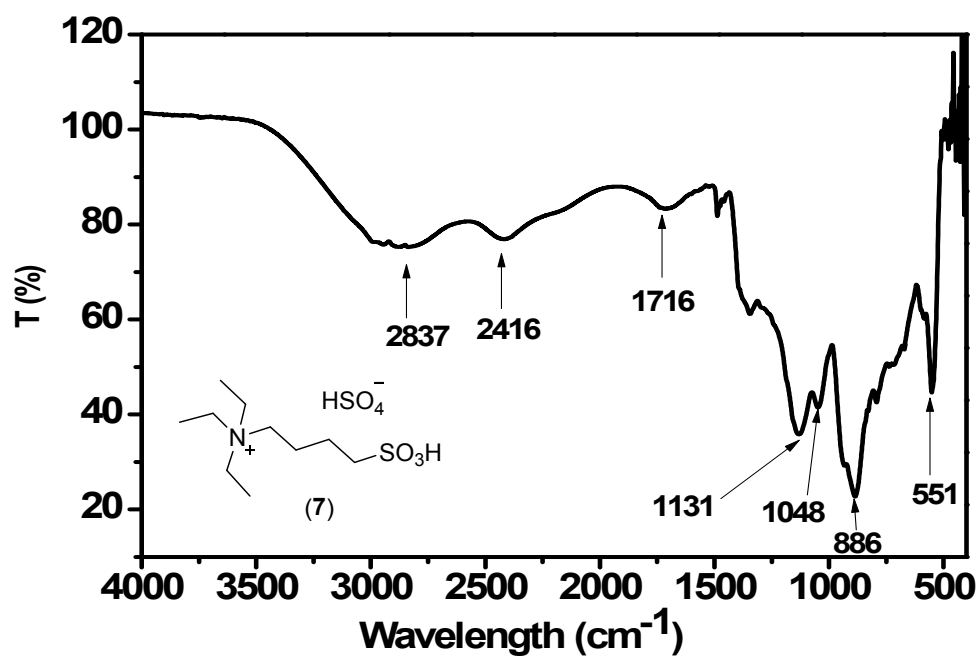
Corresponding author: Email: cv.rode@ncl.res.in, Fax: +91 20 2590 2621 Telephone: +91 20 2590 2349

Characterization of ionic liquids

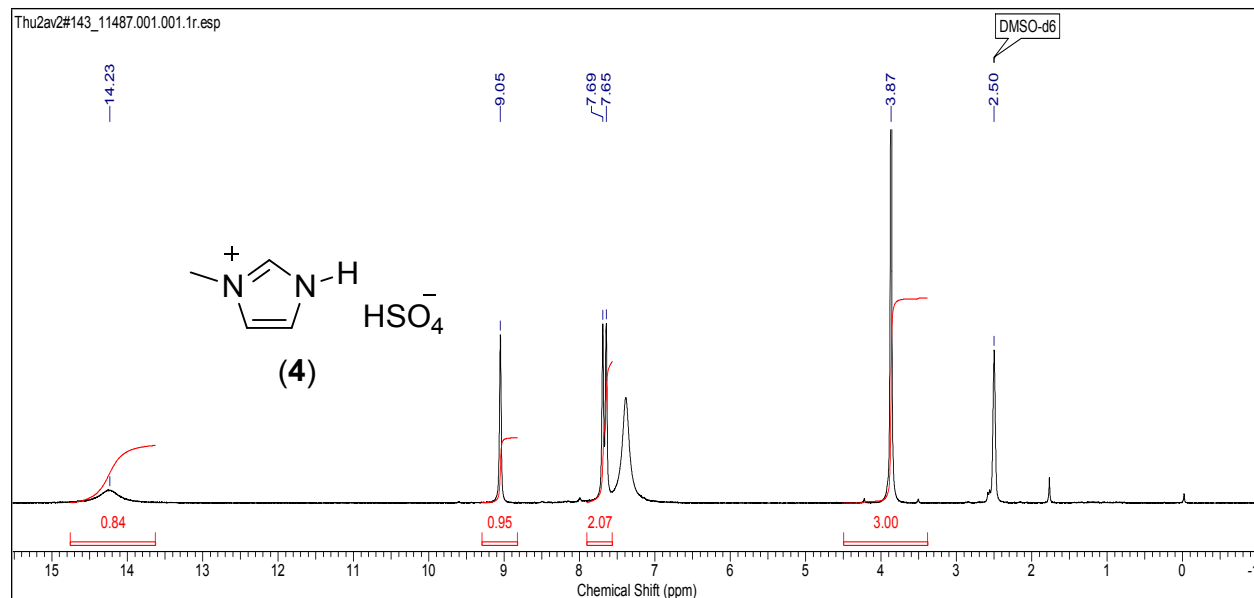
FTIR of ILs (4-8)



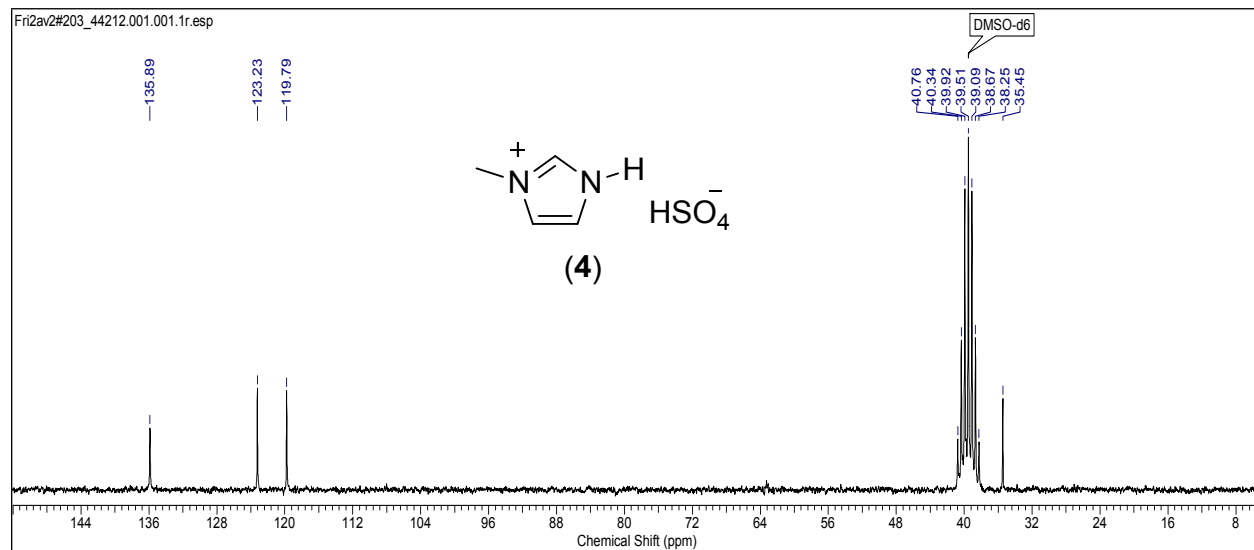




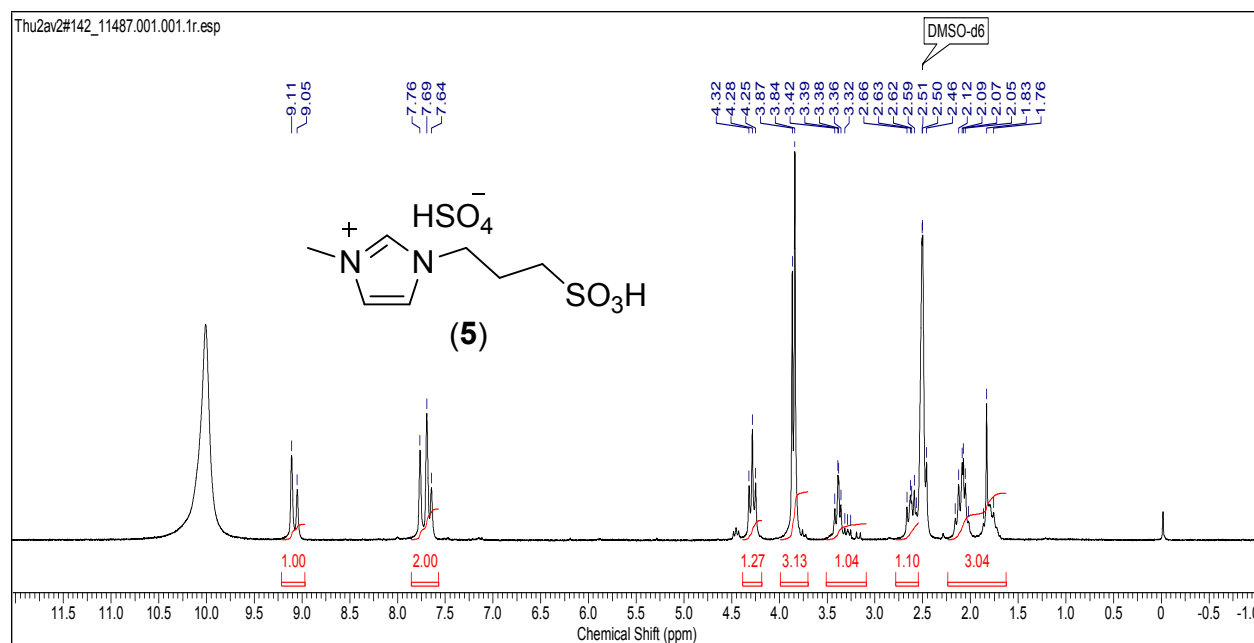
¹H and ¹³C NMR of (4-8)



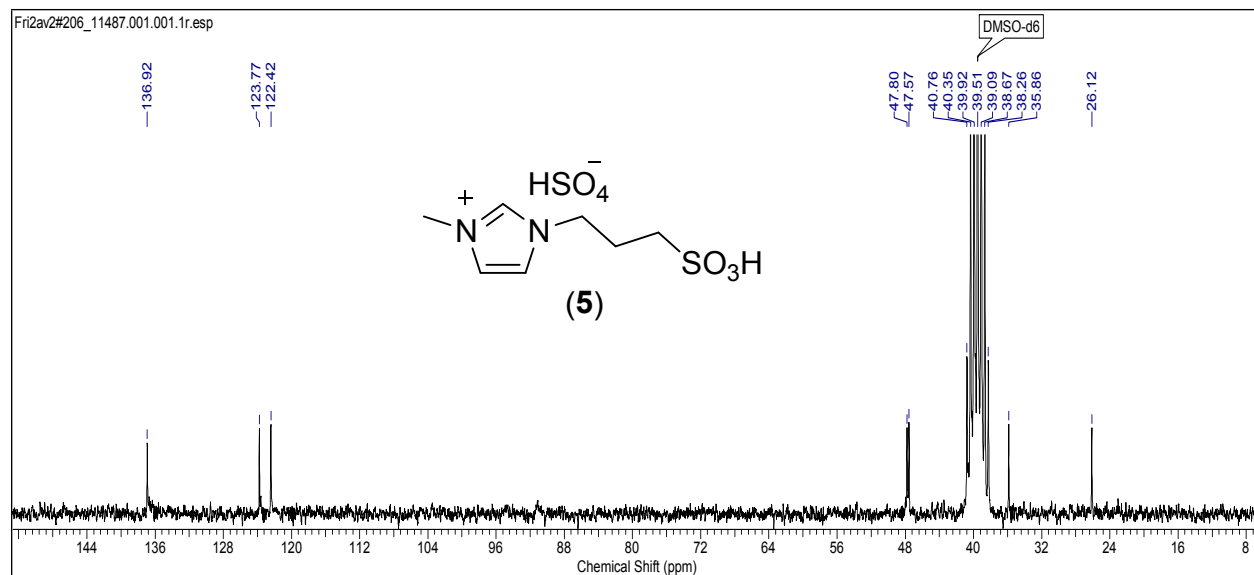
¹H NMR (200 MHz, DMSO-*d*₆) δ ppm 3.87 (s, 3 H) 7.67 (d, *J*=9.35 Hz, 2 H) 9.05 (s, 1 H) 14.23 (br. s., 1 H)



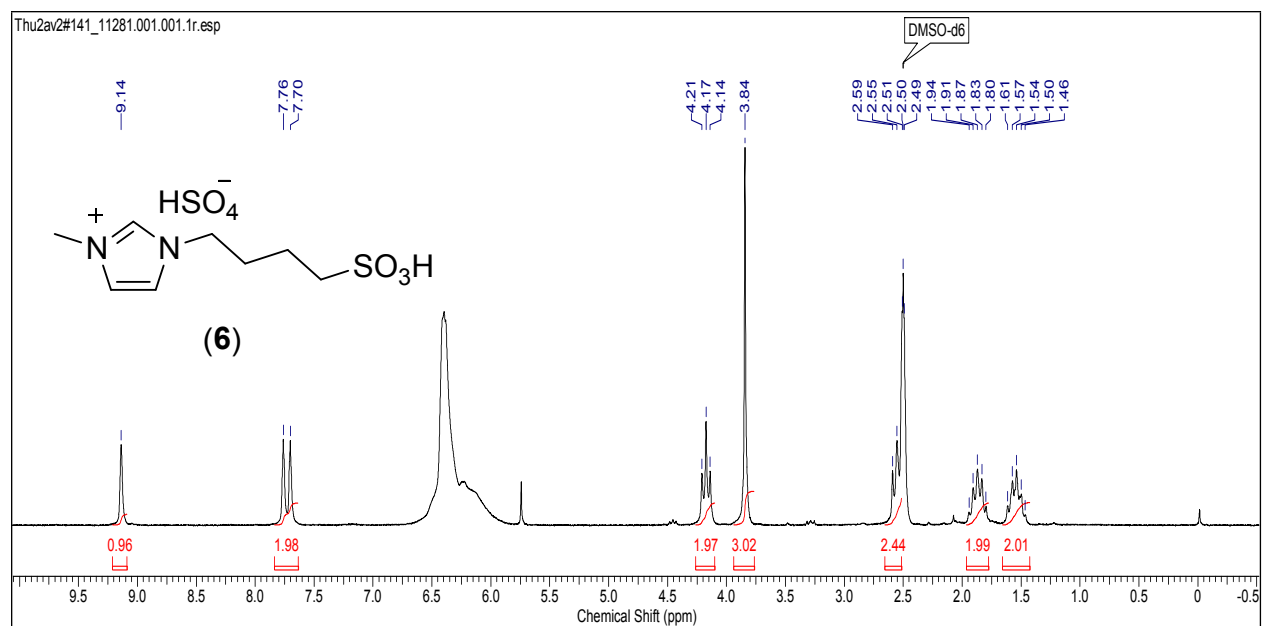
¹³C NMR (50 MHz, DMSO-*d*₆) δ ppm 35.45, 38.25, 38.67, 39.09, 39.92, 40.34, 40.76, 119.79, 123.23, 135.89.



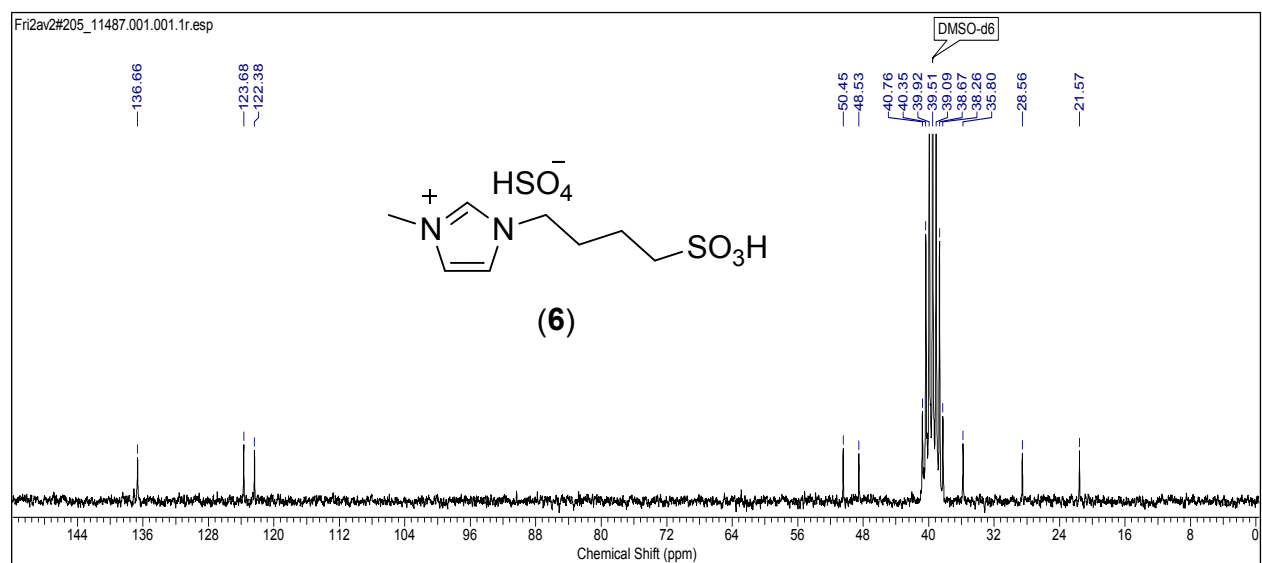
^1H NMR (200 MHz, $\text{DMSO}-d_6$) δ ppm 1.763 – 2.12 (m, 3 H) 2.55 - 2.78 (m, 1 H) 3.09 - 3.51 (m, 1 H) 3.85 (d, $J=5.56$ Hz, 3 H) 4.28 (t, $J=6.88$ Hz, 1 H) 7.57 - 7.85 (m, 2 H) 8.97 - 9.21 (m, 1 H)



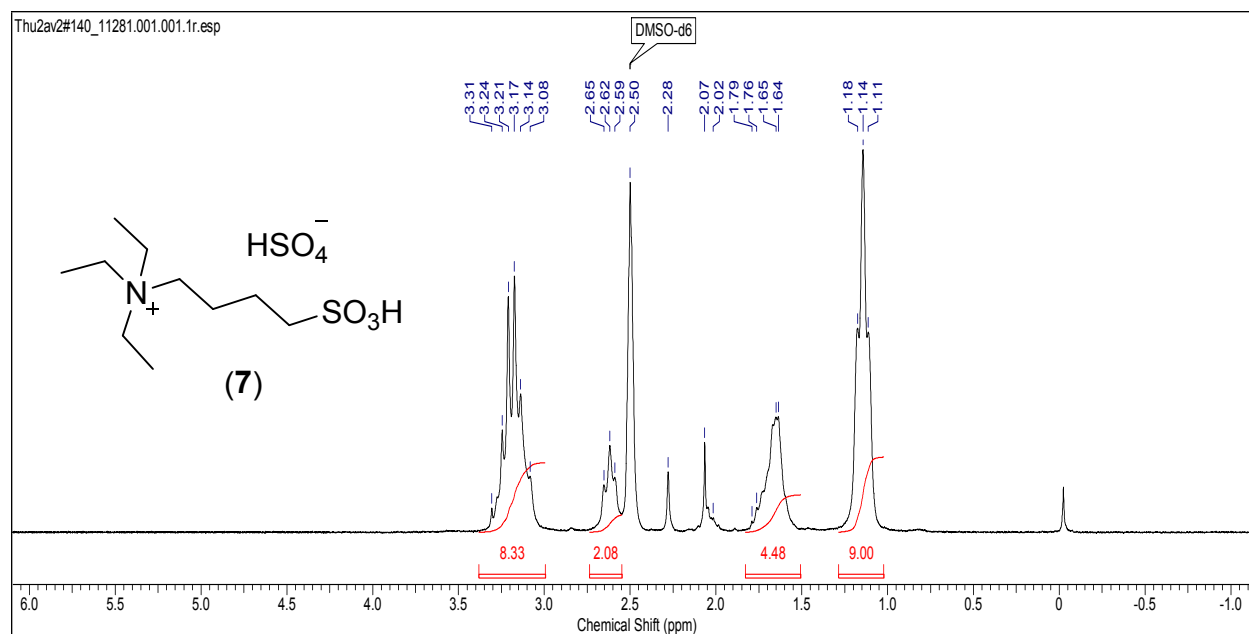
^{13}C NMR (50 MHz, $\text{DMSO}-d_6$) δ ppm 26.12, 35.86, 38.26, 38.67, 39.09, 39.92, 40.35, 40.76, 47.57, 47.80, 122.42, 123.77, 136.92.



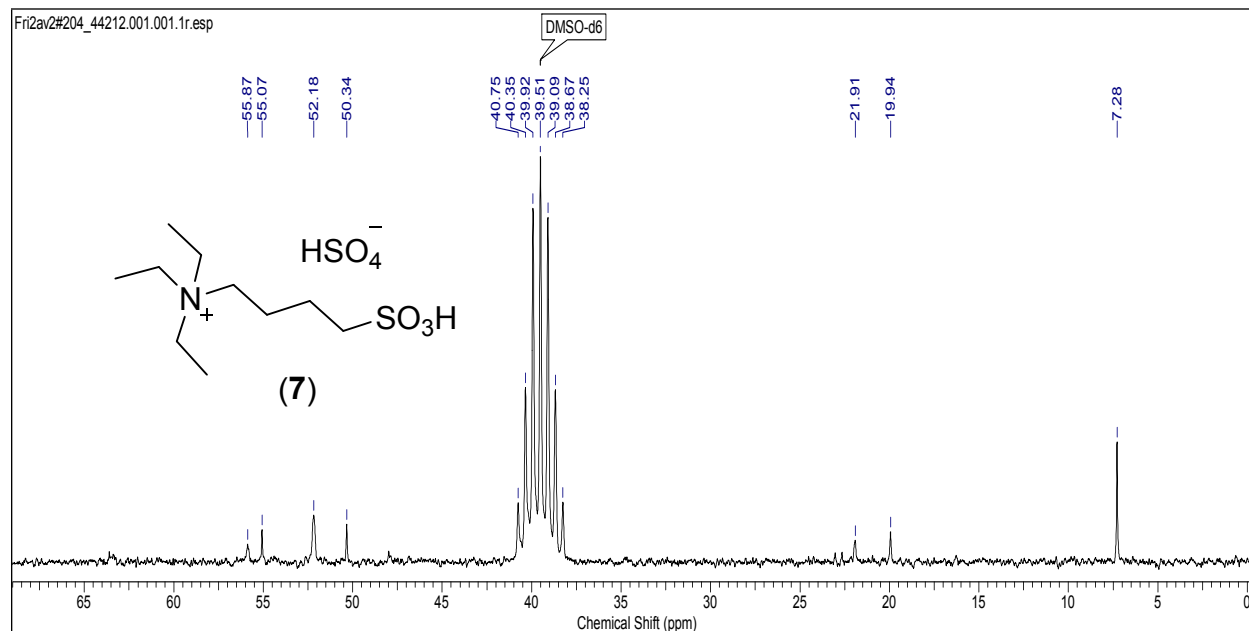
^1H NMR (200 MHz, $\text{DMSO}-d_6$) δ ppm 1.54 (quin, $J=7.42$ Hz, 2 H) 1.87 (quin, $J=7.20$ Hz, 2 H) 2.51 - 2.66 (m, 3 H) 3.84 (s, 3 H) 4.17 (t, $J=6.88$ Hz, 2 H) 7.76 (s, 1 H) 7.70 (s, 1 H) 9.14 (s, 1 H)



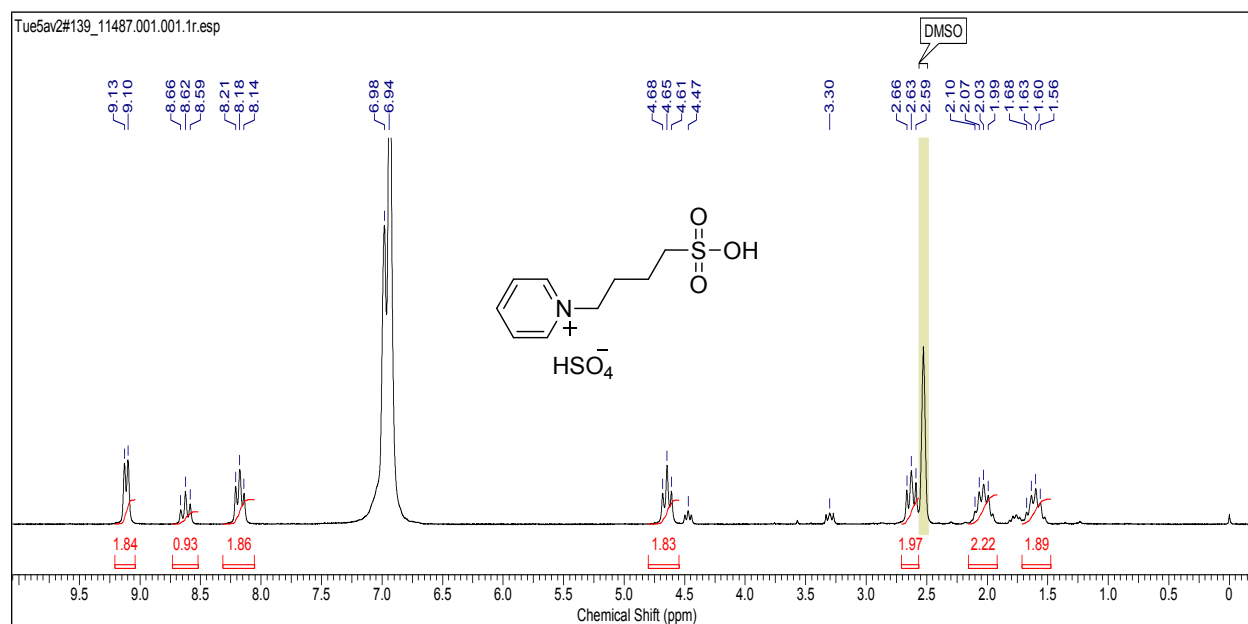
^{13}C NMR (50 MHz, $\text{DMSO}-d_6$) δ ppm 21.57, 28.56, 35.80, 38.26, 38.67, 39.09, 39.92, 40.35, 40.76, 48.53, 50.45, 122.38, 123.68, 136.66



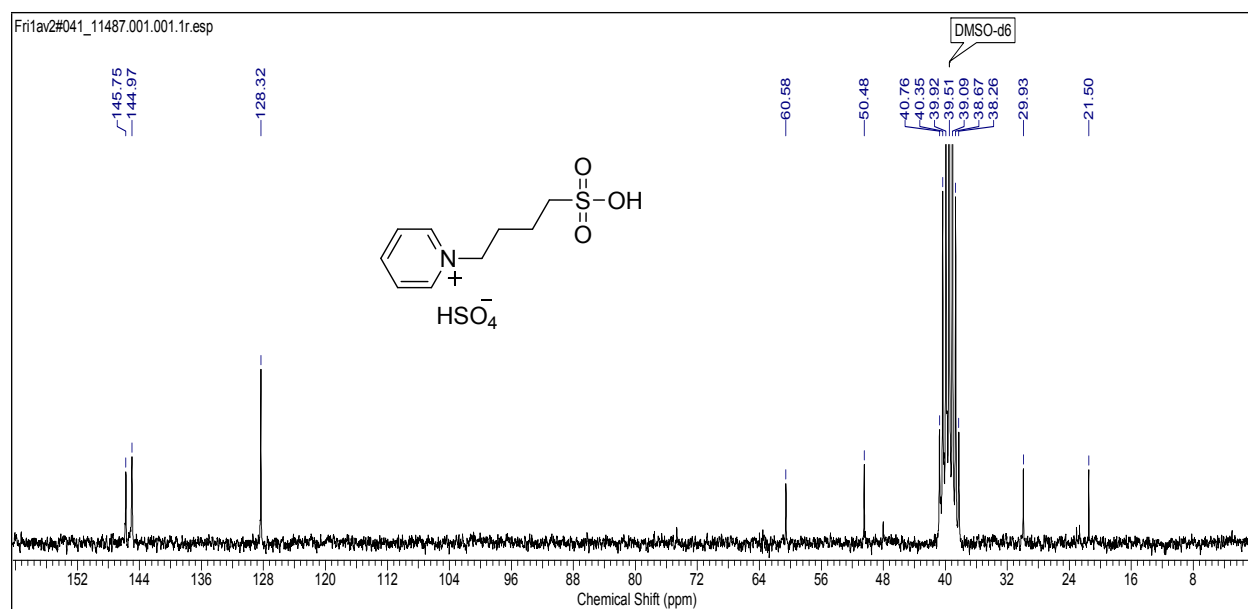
^1H NMR (200 MHz, $\text{DMSO}-d_6$) δ ppm 1.14 (t, $J=6.19$ Hz, 9 H) 1.51 - 1.71 (m, 4 H) 1.71 - 1.83 (m, 1 H) 2.62 (t, $J=6.32$ Hz, 2 H) 2.99 - 3.38 (m, 8 H)



^{13}C NMR (50 MHz, $\text{DMSO}-d_6$) δ ppm 7.28, 19.94, 21.91, 38.25, 38.67, 39.09, 39.92, 40.35, 40.75, 50.34, 52.18, 55.07, 55.87

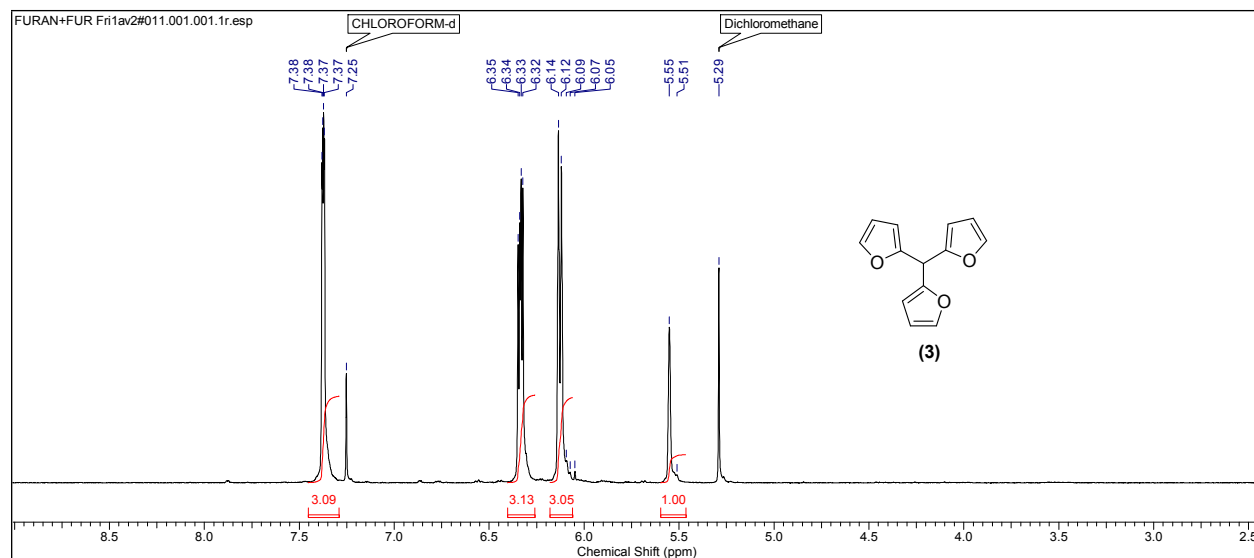


^1H NMR (200 MHz, $\text{DMSO}-d_6$) δ ppm 1.48 - 1.71 (m, 2 H) 1.92 - 2.16 (m, 2 H) 2.63 (t, $J=7.52$ Hz, 2 H) 4.65 (t, $J=7.26$ Hz, 2 H) 8.18 (t, $J=6.88$ Hz, 2 H) 8.52 - 8.73 (m, 1 H) 9.11 (d, $J=5.68$ Hz, 2 H)

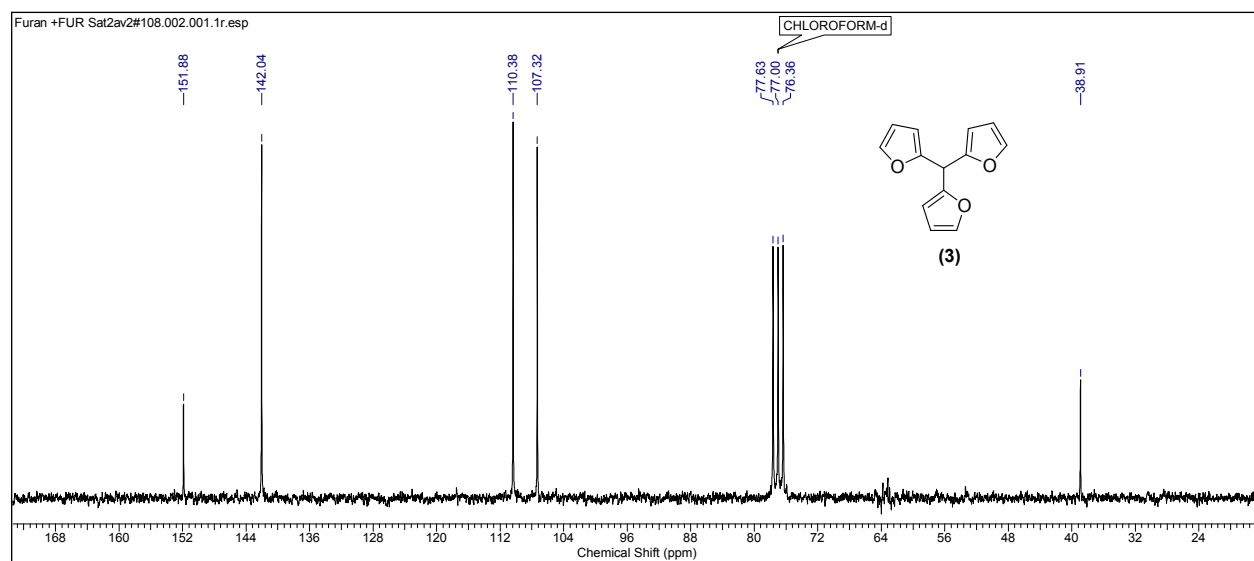


^{13}C NMR (50 MHz, $\text{DMSO}-d_6$) δ ppm 21.50, 29.93, 50.48, 60.58, 128.32, 144.97, 145.75.

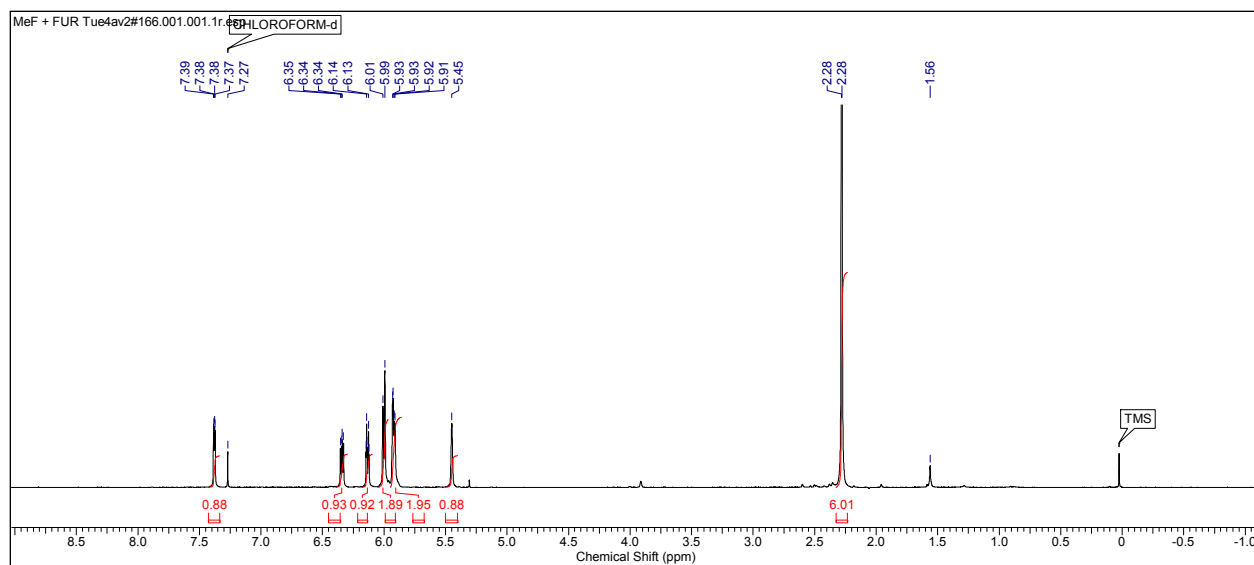
Characterization of compounds: ^1H and ^{13}C NMR



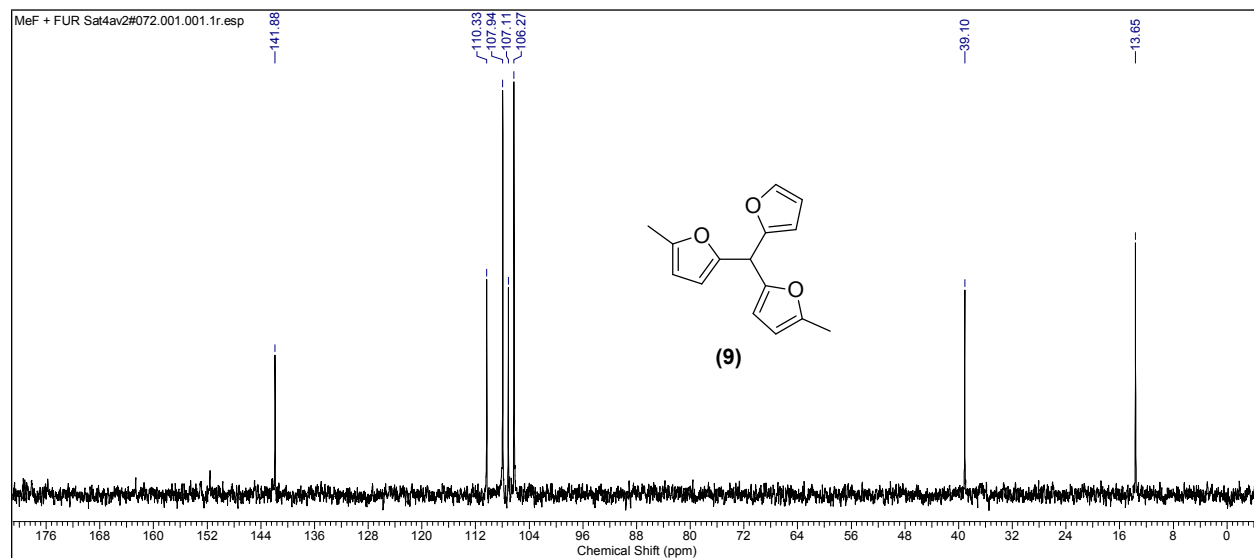
^1H NMR (200 MHz, CHLOROFORM-*d*) δ ppm 5.46 - 5.60 (m, 1 H) 6.06 - 6.18 (m, 3 H) 6.34 (dd, $J=3.16, 1.89$ Hz, 3 H) 7.37 (dd, $J=1.77, 0.76$ Hz, 3 H)



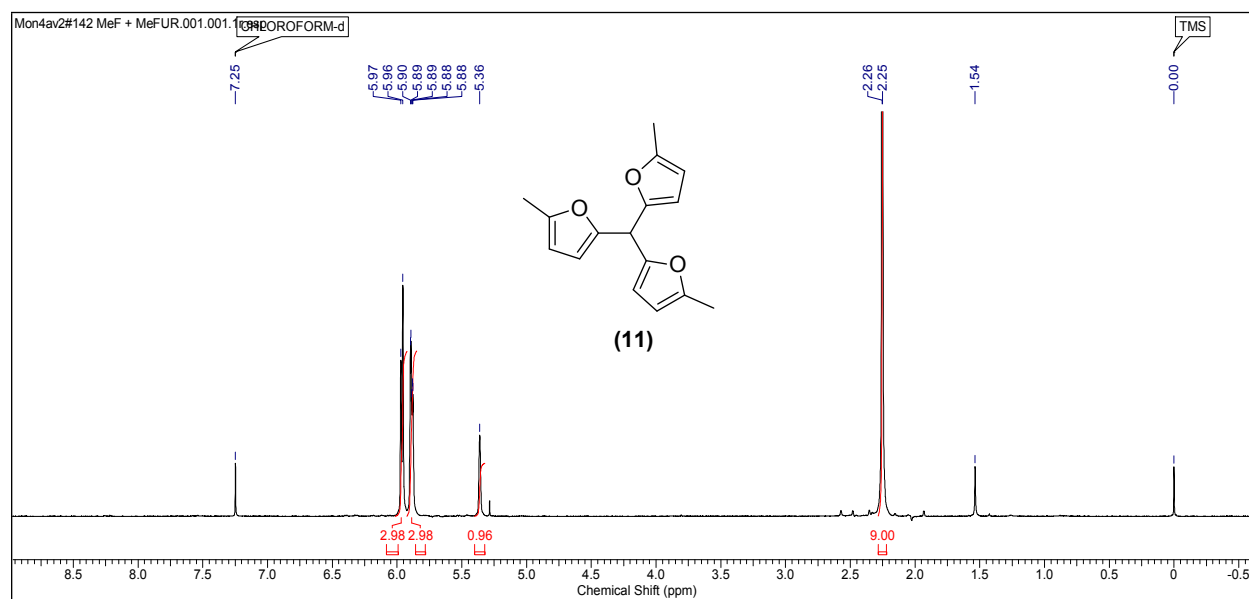
^{13}C NMR (50 MHz, CHLOROFORM-*d*) δ ppm 38.91, 107.32, 110.38, 142.04, 151.88



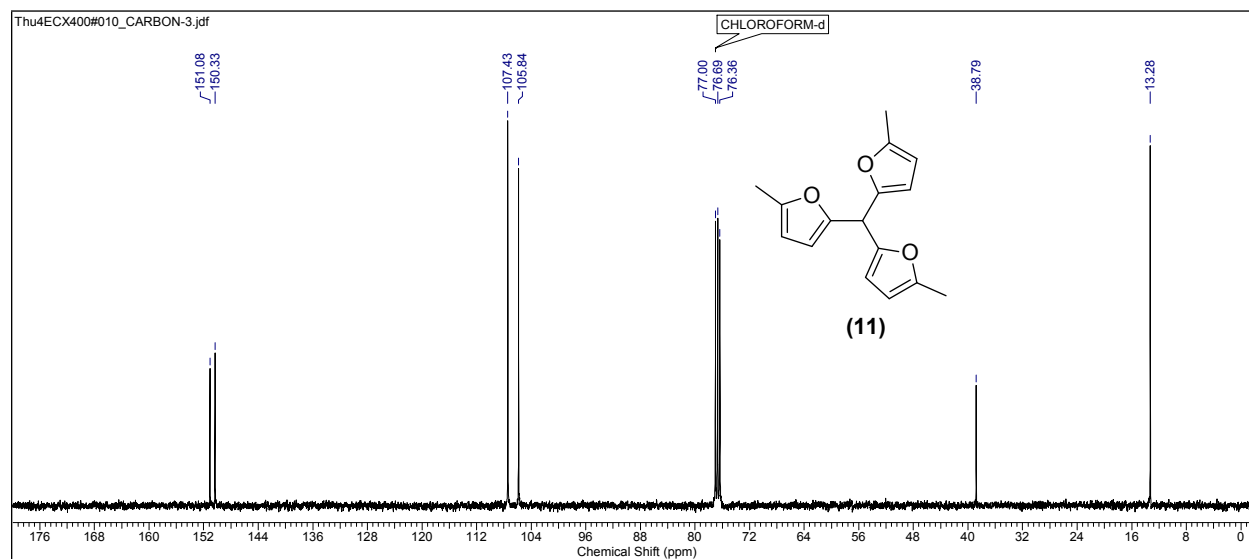
^1H NMR (200 MHz, CHLOROFORM- d) δ ppm 2.28 (s, 6 H) 5.45 (s, 1 H) 5.91 - 5.93 (m, 2 H) 5.99-6.01 (d, J =3.03 Hz, 2 H) 6.13-6.14 (dt, J =3.22, 0.79 Hz, 1 H) 6.34-6.35 (dd, J =3.03, 1.89 Hz, 1 H) 7.37-7.39 (dd, J =1.89, 0.88 Hz, 1 H)



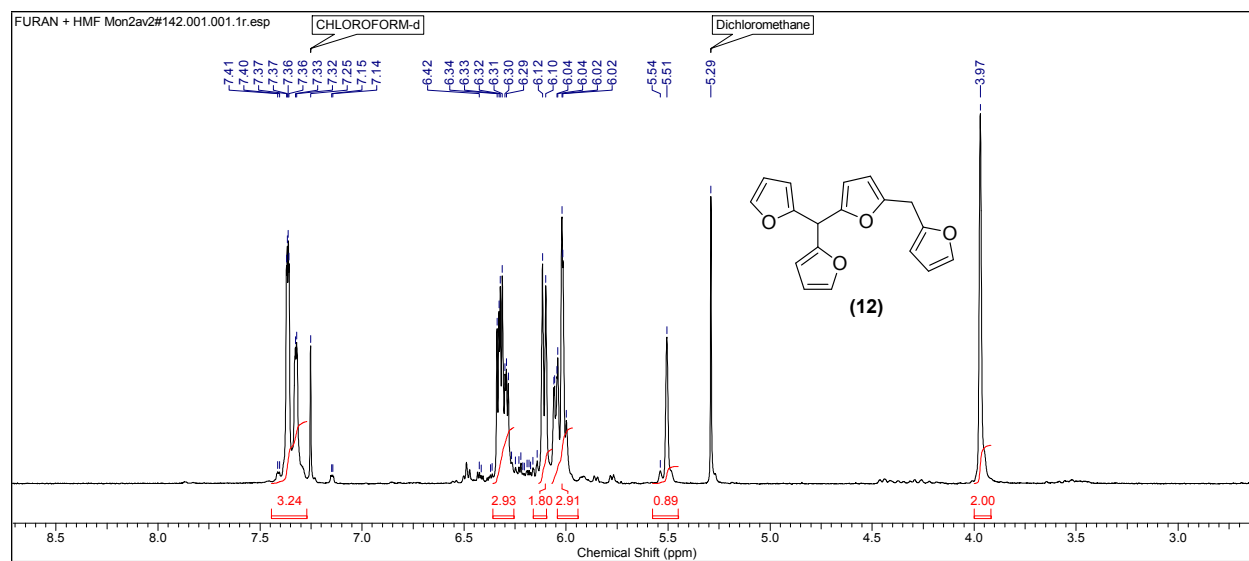
^{13}C NMR (50 MHz, CHLOROFORM- d) δ ppm 13.65, 39.10, 106.27, 107.11, 107.94, 110.33 and 141.88



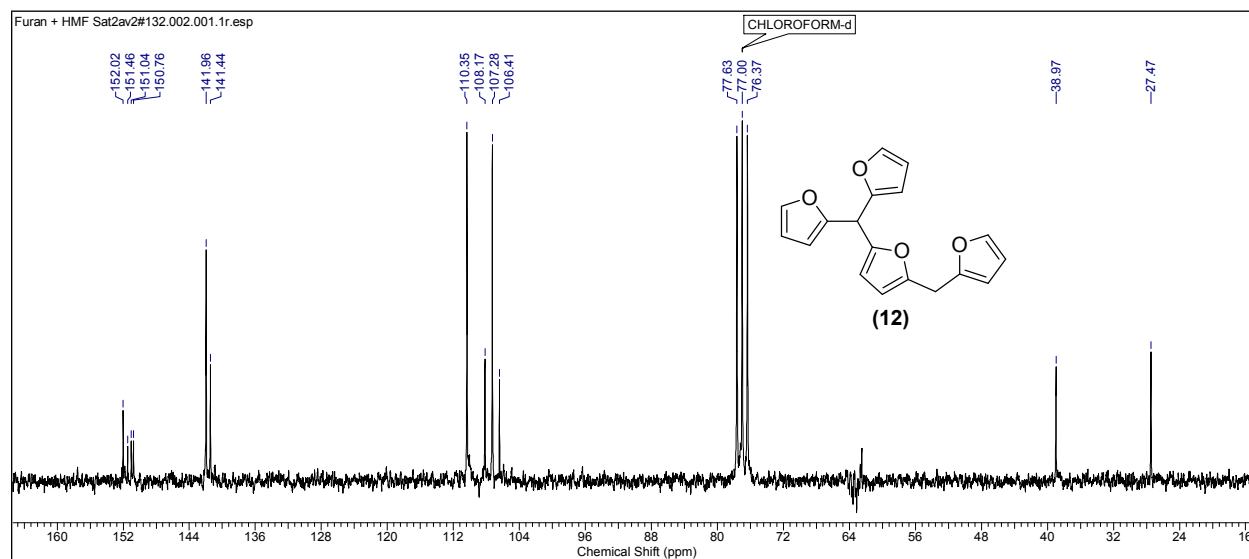
^1H NMR (200 MHz, CHLOROFORM-*d*) δ ppm 2.22 - 2.28 (m, 9 H) 5.36 (s, 1 H) 5.88 - 5.90 (m, 3 H) 5.96-5.97 (d, $J=3.03$ Hz, 3 H)



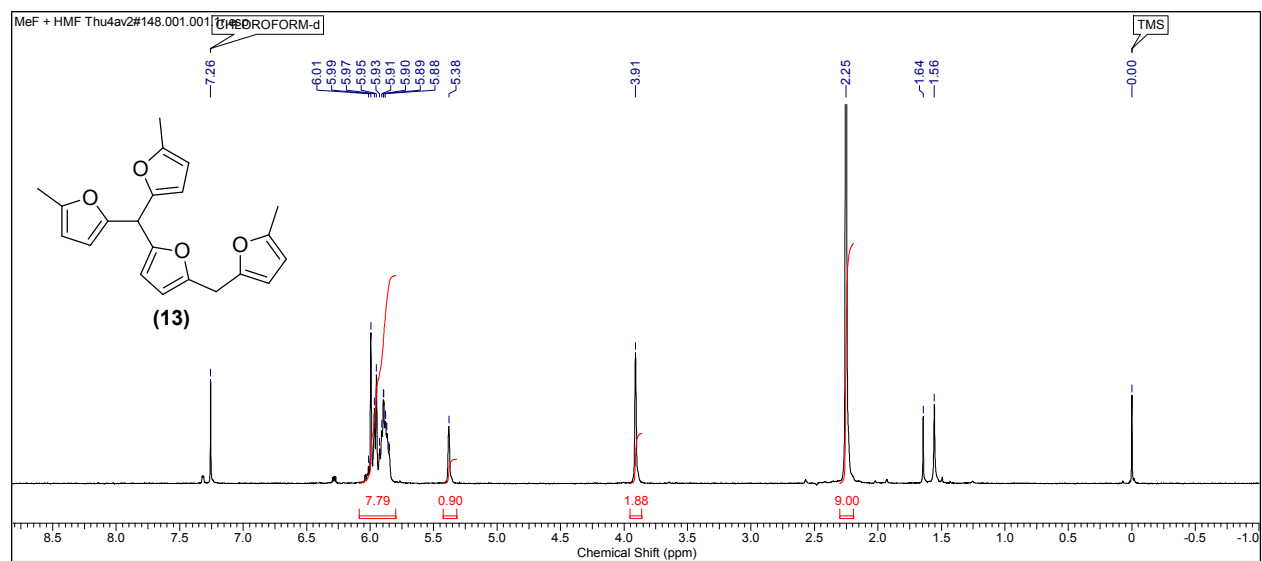
^{13}C NMR (101 MHz, CHLOROFORM-*d*) δ ppm 13.71, 39.22, 106.27, 107.86, 150.76 and 151.51



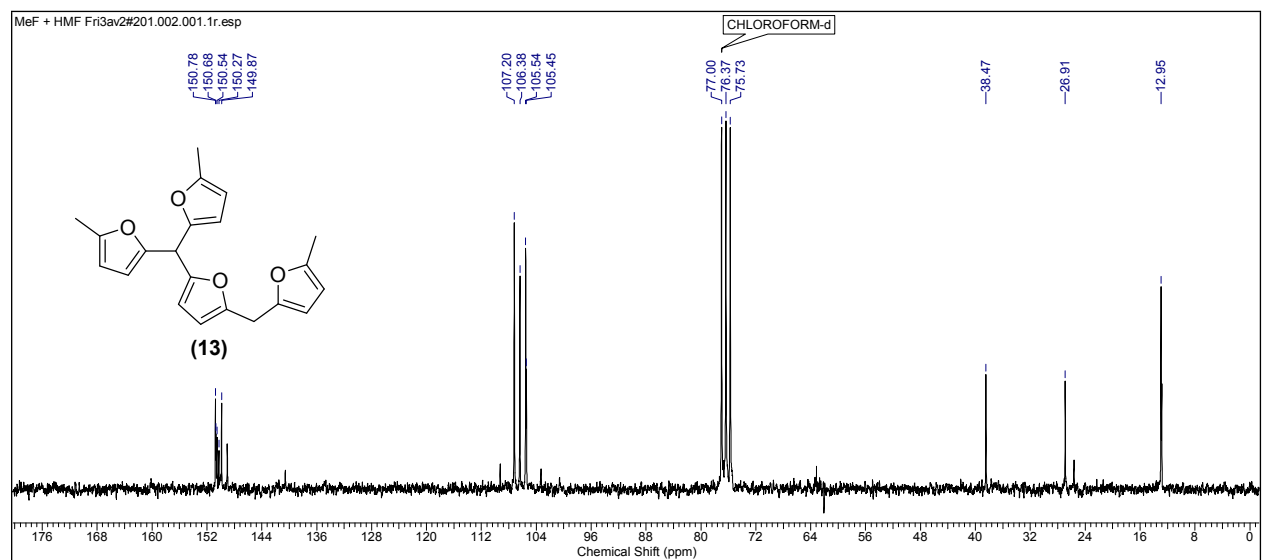
^1H NMR (200 MHz, CHLOROFORM-*d*) δ ppm 3.97 (s, 2 H) 5.51 - 5.54 (m, 1 H) 6.02 - 6.04 (m, 3 H) 6.10 - 6.12 (d, $J=3.16$ Hz, 2 H) 6.29 - 6.34 (m, 3 H) 7.25 - 7.41 (m, 3 H)



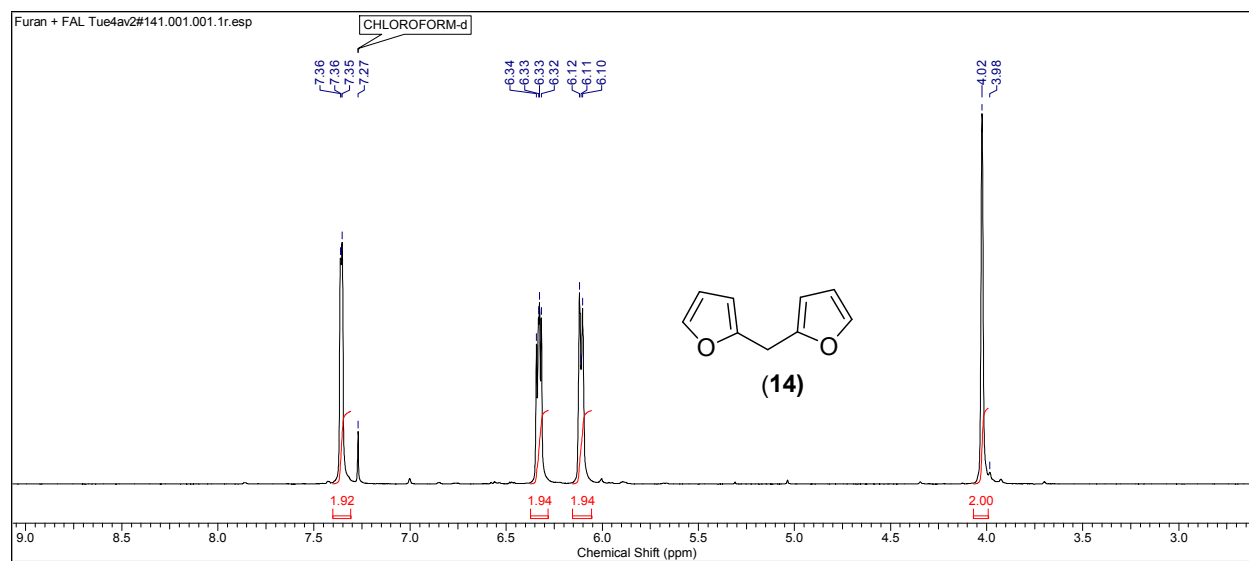
^{13}C NMR (50 MHz, CHLOROFORM-*d*) δ ppm 27.47, 38.97, 106.41, 107.28, 108.17, 110.35, 141.44, 141.96, 150.76, 151.04, 151.46, 152.02



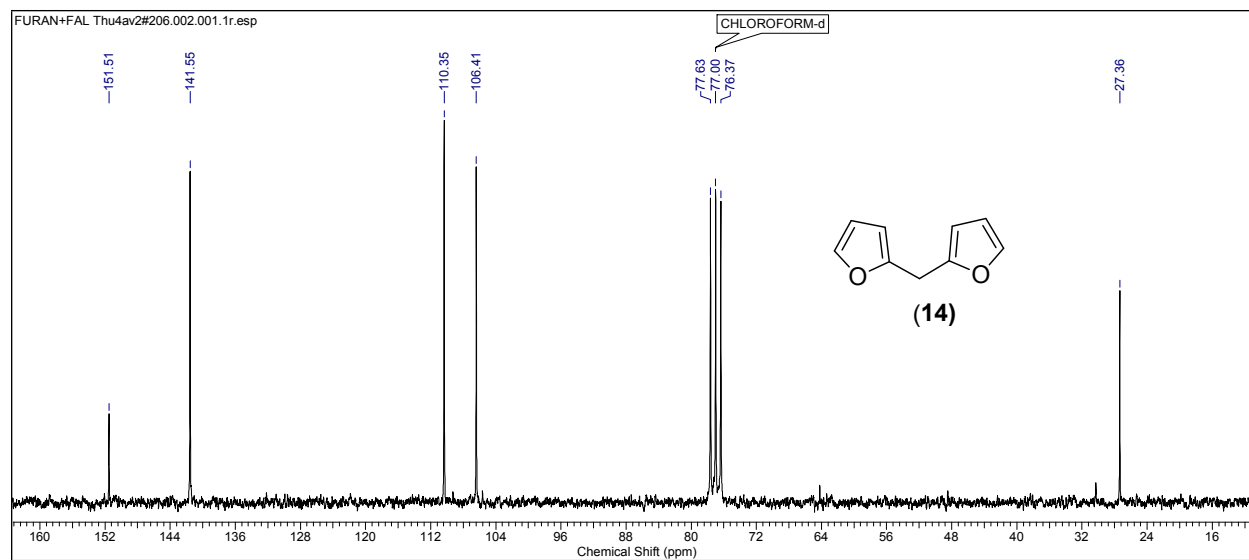
^1H NMR (200 MHz, CHLOROFORM-*d*) δ ppm 2.25 (s, 9 H) 3.91 (s, 2 H) 5.38 (s, 1 H) 5.88- 6.01 (m, 8 H)



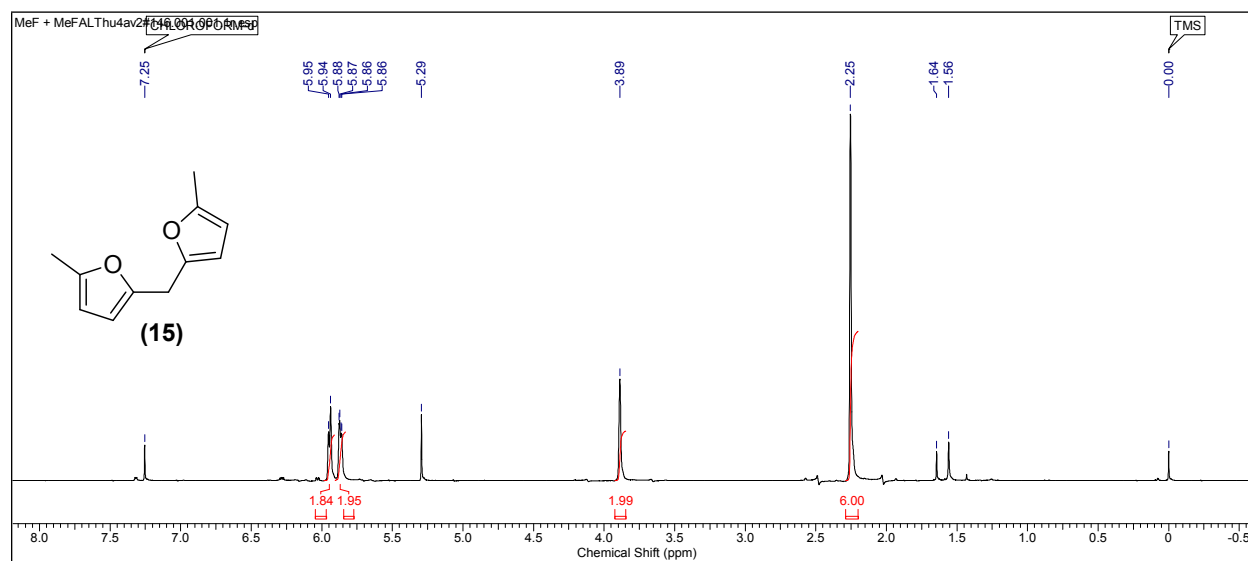
^{13}C NMR (50 MHz, CHLOROFORM-*d*) δ ppm 13.52, 13.62, 27.59, 39.15, 106.12, 106.22, 107.06, 107.88, 149.71, 150.55, 150.95, 151.21, 151.36, 151.45



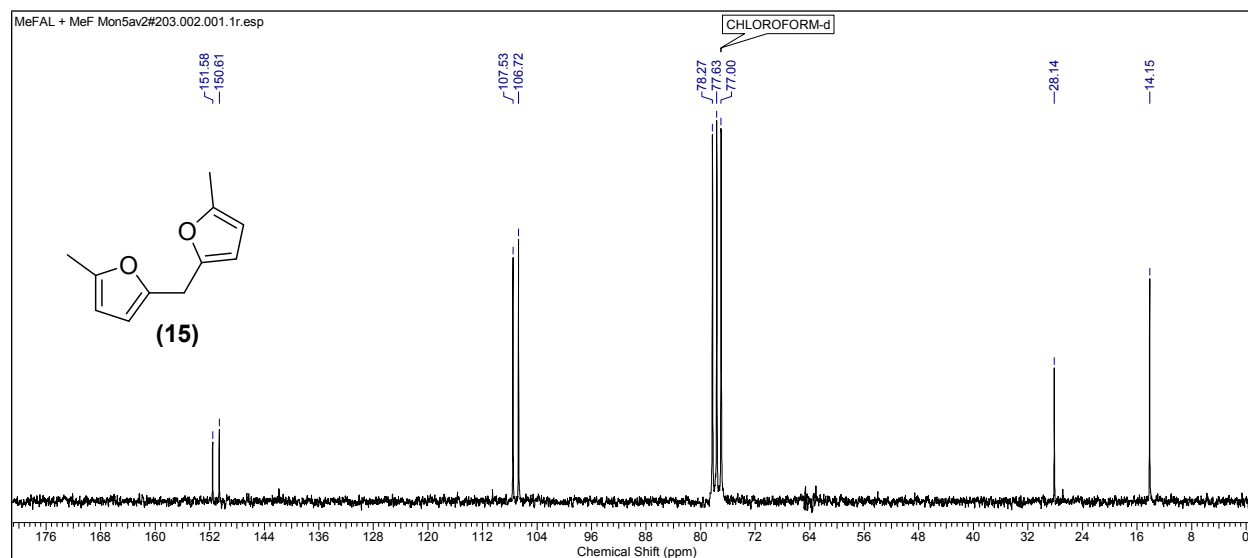
^1H NMR (200 MHz, CHLOROFORM- d) δ ppm 3.94 - 4.08 (m, 2 H) 6.05 - 6.17 (m, 2 H) 6.33 (dd, $J=3.03$, 2.02 Hz, 2 H) 7.29 - 7.41 (m, 2 H)



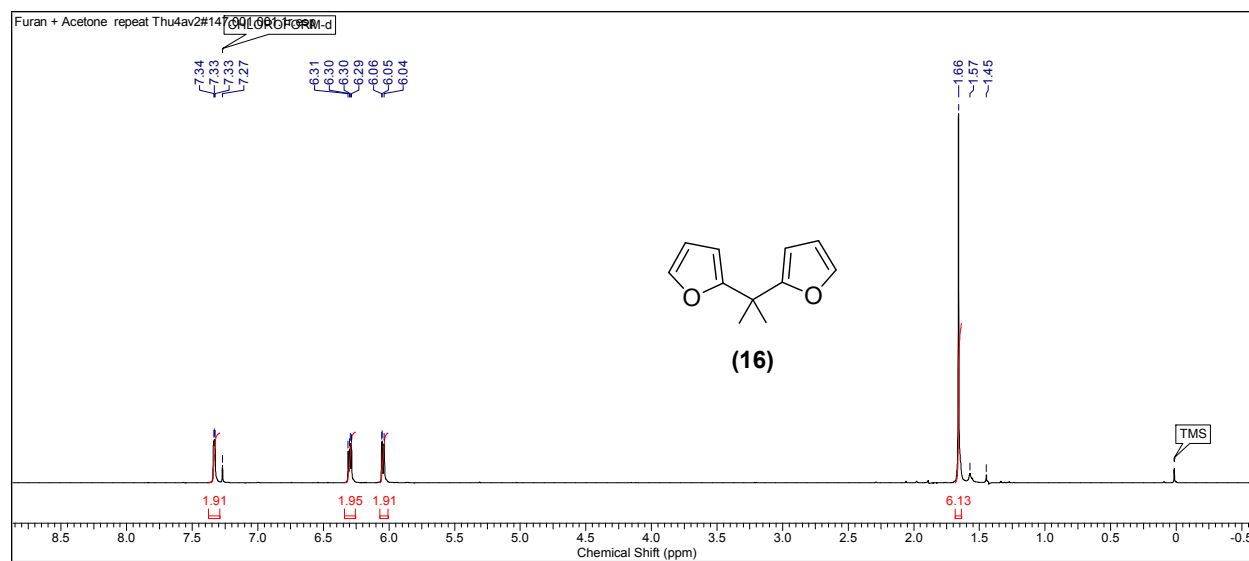
^{13}C NMR (50 MHz, CHLOROFORM- d) δ ppm 27.36, 106.41, 110.35, 141.55, 151.5



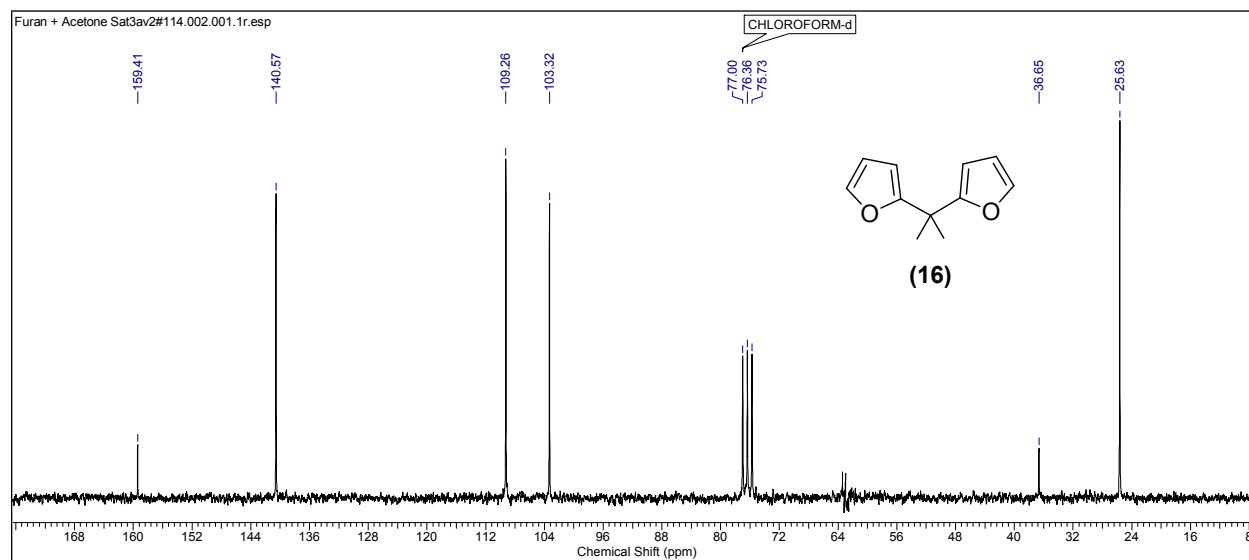
^1H NMR (200 MHz, CHLOROFORM-*d*) δ ppm 2.25 (s, 6 H) 3.89 (s, 2 H) 5.86 - 5.88 (dd, J = 2.91, 0.88 Hz, 2 H) 5.94 - 5.95 (d, J =3.03 Hz, 2 H)



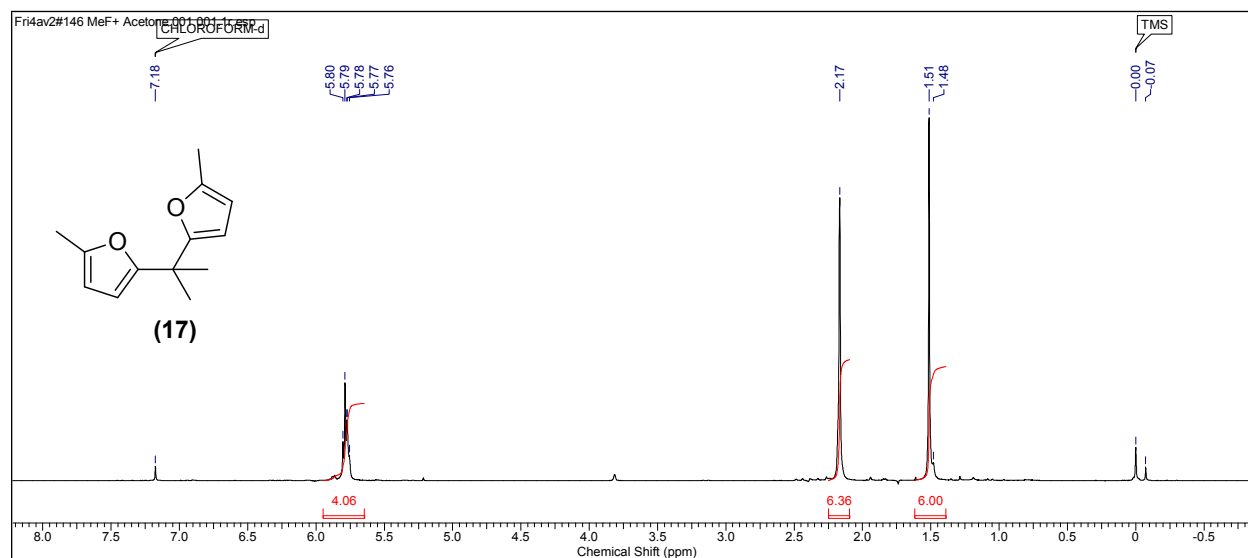
^{13}C NMR (50 MHz, CHLOROFORM-*d*) δ ppm 14.15, 28.14, 106.72, 107.53, 150.61, 151.58



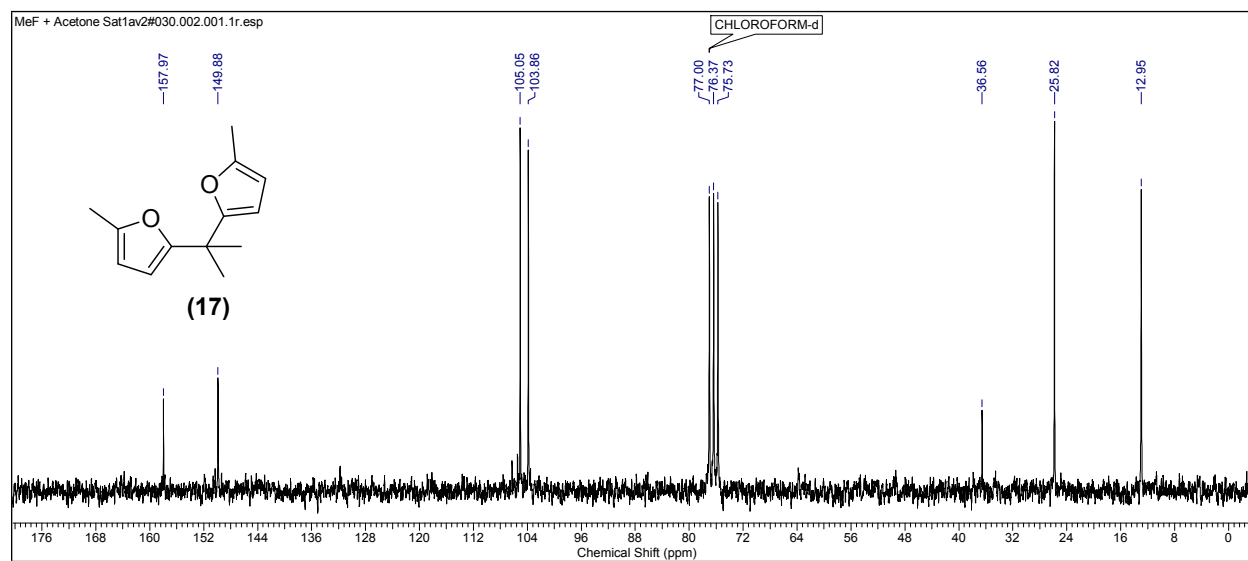
^1H NMR (200 MHz, CHLOROFORM-*d*) δ ppm 1.66 (s, 6 H) 6.01 - 6.07 (m, 2 H) 6.30 (dd, $J=3.22$, 1.83 Hz, 2 H) 7.29 - 7.38 (m, 2 H)



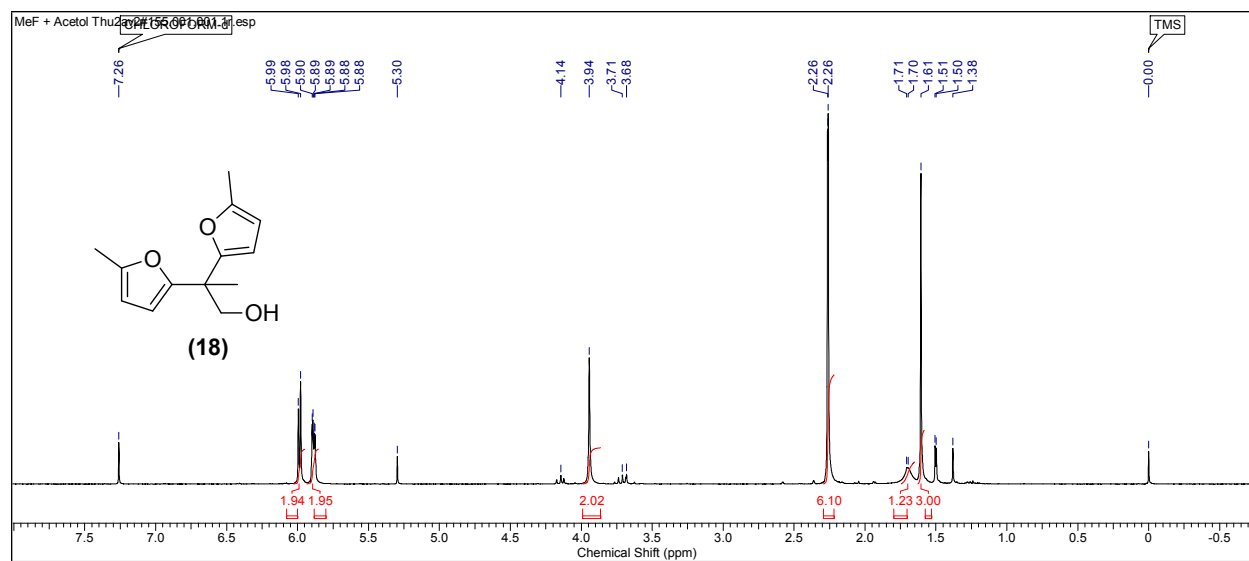
^{13}C NMR (50 MHz, CHLOROFORM-*d*) δ ppm 25.63, 36.65, 103.32, 109.26, 140.57, 159.41



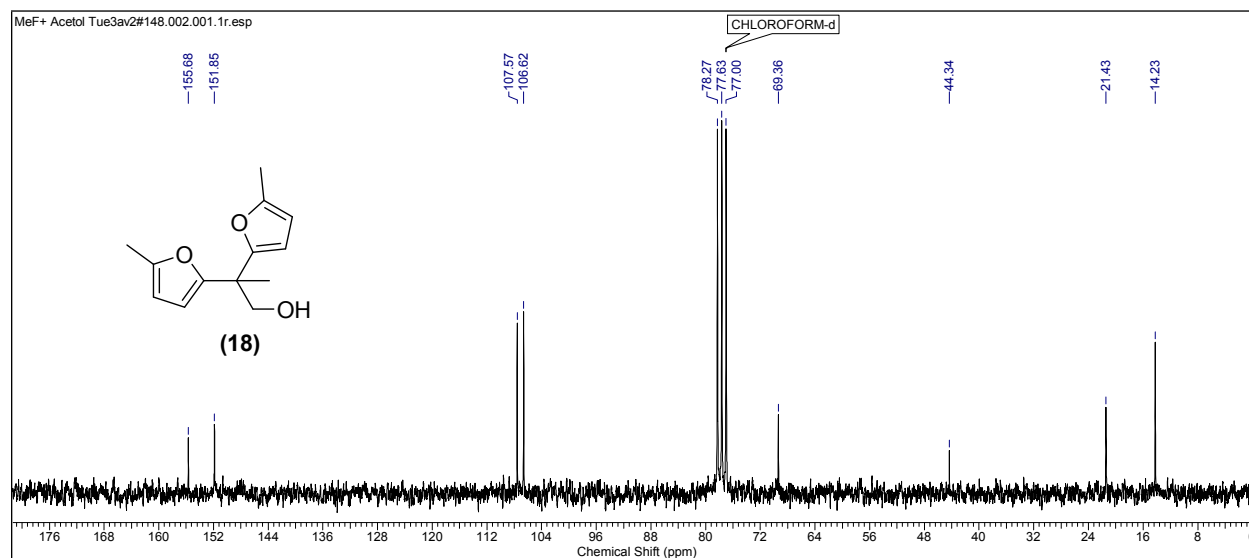
^1H NMR (200 MHz, CHLOROFORM-*d*) δ ppm 1.48 - 1.51 (m, 6 H) 2.17 (s, 7 H) 5.76 - 5.90 (m, 4 H)



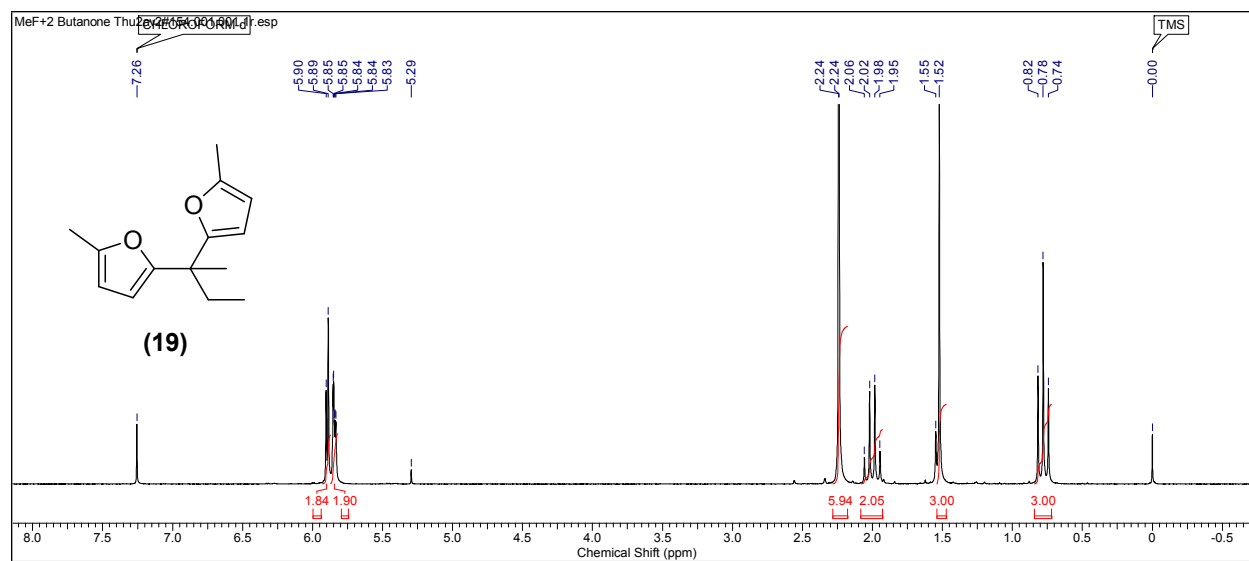
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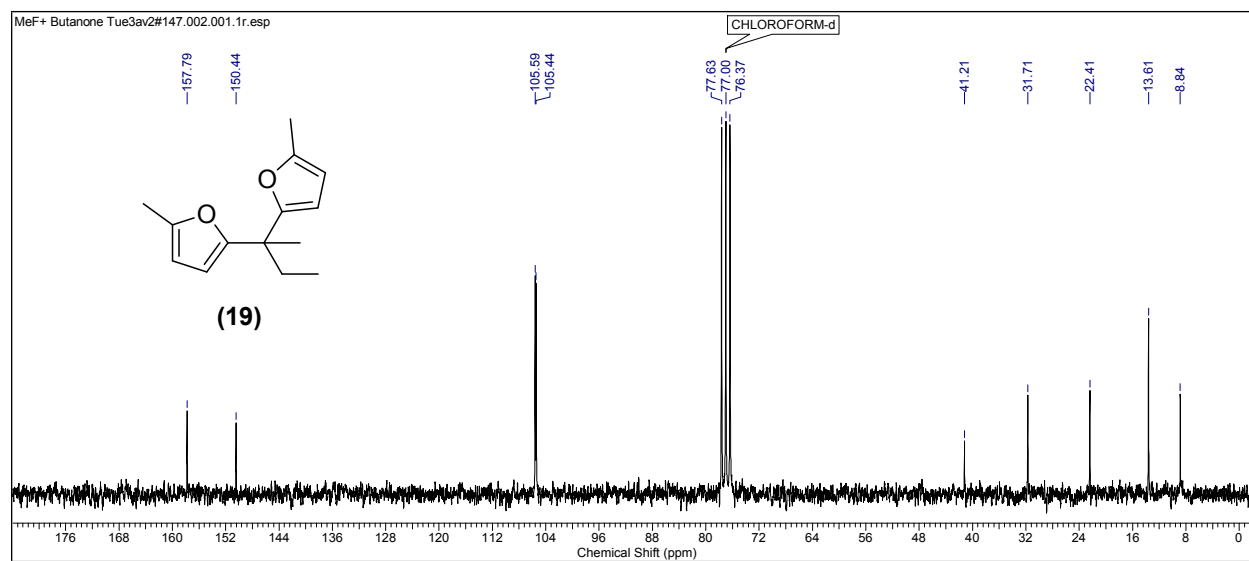
^1H NMR (200 MHz, CHLOROFORM-*d*) δ ppm 1.61 (s, 3 H) 1.65 - 1.74 (m, 1 H) 2.26 (d, $J=0.76$ Hz, 6 H) 3.94 (s, 2 H) 5.85 - 5.94 (m, 2 H) 5.99 (d, $J=3.16$ Hz, 2 H)



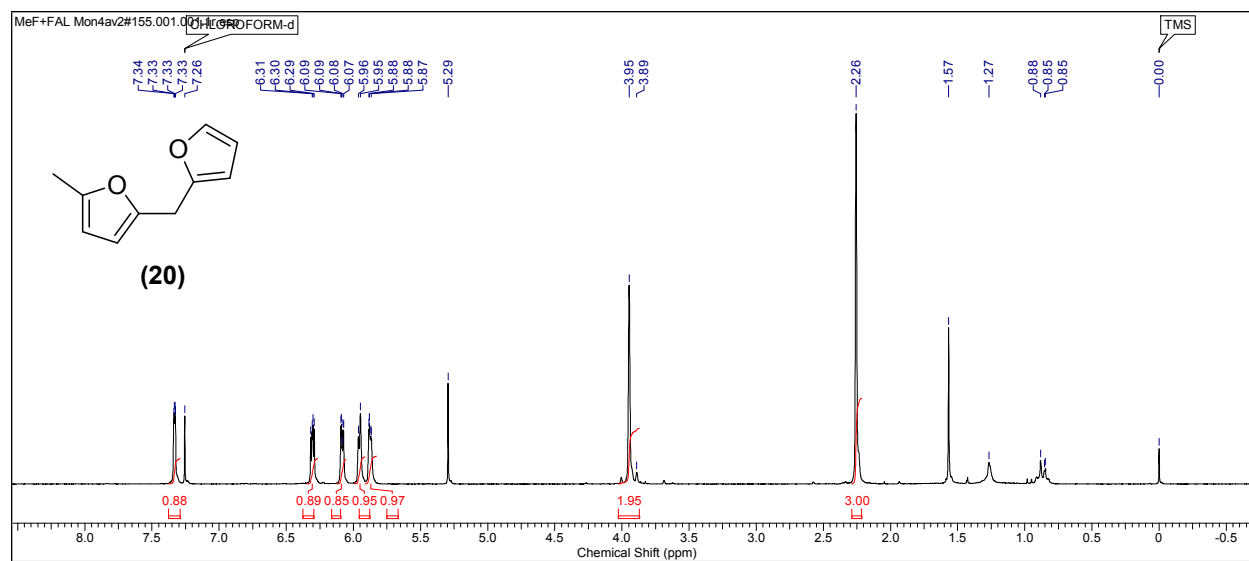
^{13}C NMR (50 MHz, CHLOROFORM-*d*) δ ppm 14.23, 21.43, 44.34, 69.36, 106.62, 107.57, 151.85 and 155.68



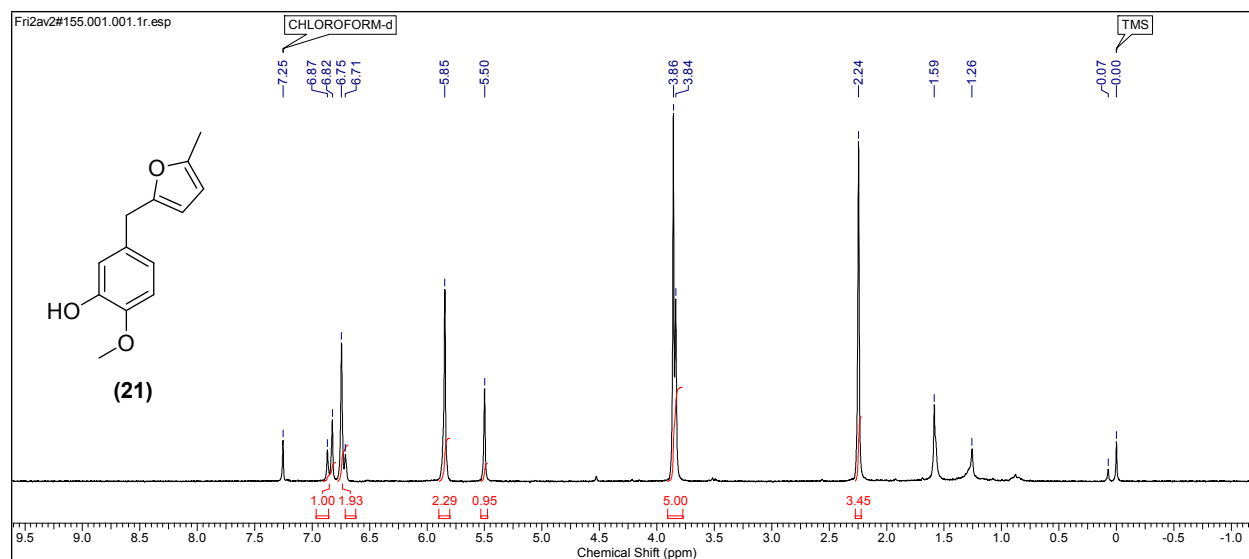
^1H NMR (200 MHz, CHLOROFORM-*d*) δ ppm 0.74-0.82 (t, $J=7.39$ Hz, 3 H) 1.52 (s, 3 H) 1.95-2.06 (q, $J=7.37$ Hz, 2 H) 2.24 (d, $J=0.88$ Hz, 6 H) 5.83 - 5.85 (m, 2 H) 5.89 - 5.90 (m, 2 H)



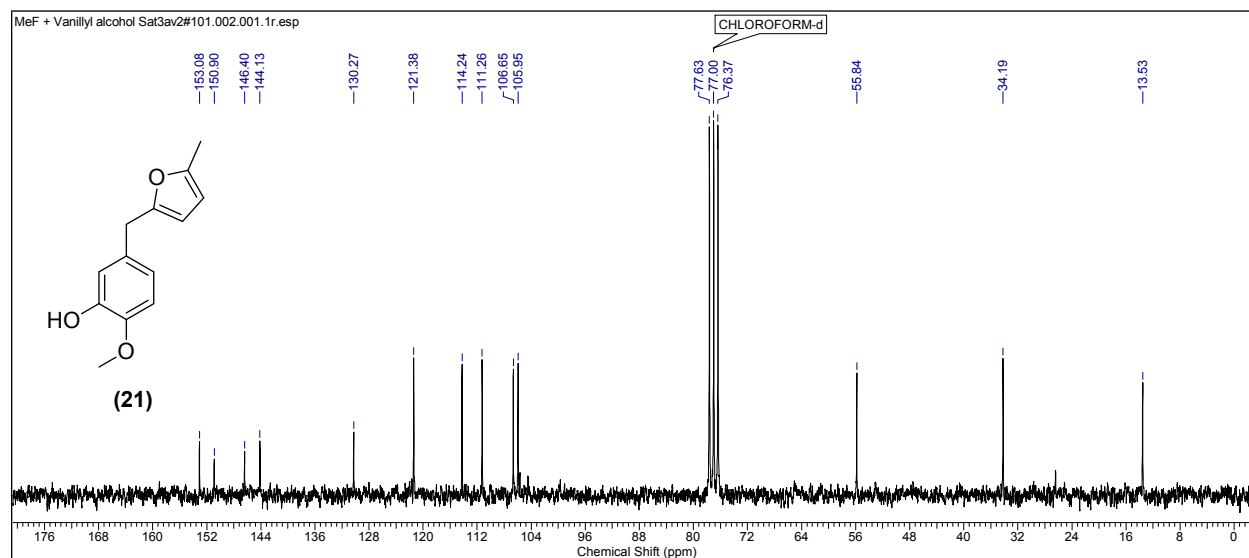
^{13}C NMR (50 MHz, CHLOROFORM-*d*) δ ppm 8.84, 13.61, 22.41, 31.71, 41.21, 105.44, 105.59, 150.44, 157.79



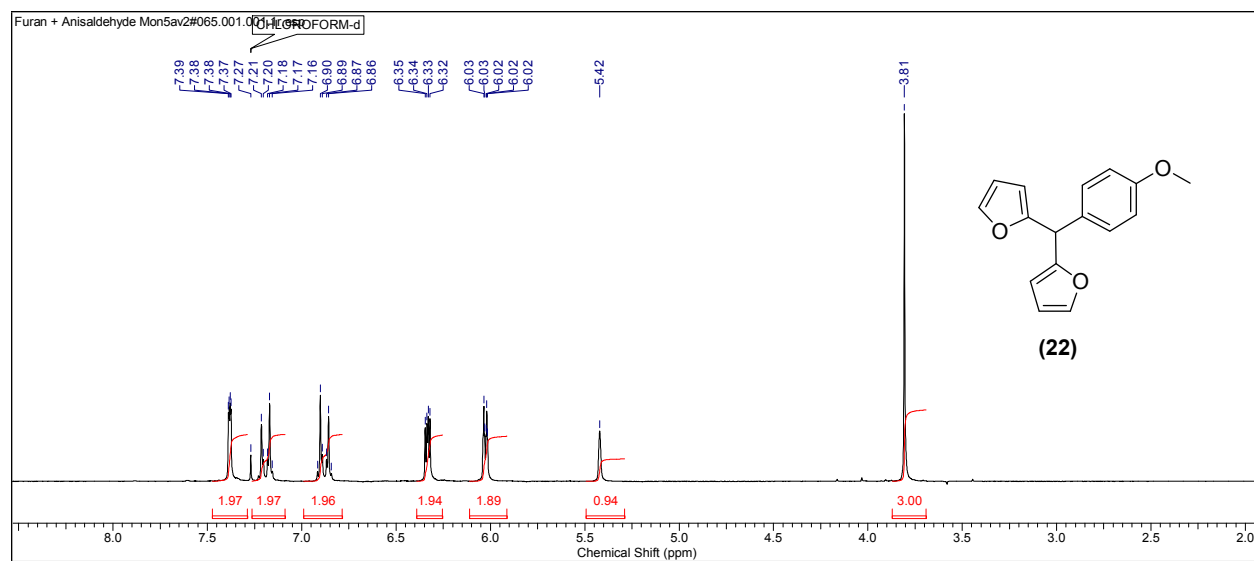
¹H NMR (200 MHz, CHLOROFORM-*d*) δ ppm 2.26 (s, 3 H) 3.89 - 4.01 (m, 2 H) 5.87 - 5.88 (m, 1 H) 5.95-5.96 (d, *J*=2.91 Hz, 1 H) 6.07 - 6.09 (m, 1 H) 6.29-6.32 (dd, *J*=3.16, 1.89 Hz, 1 H) 7.33-7.34 (dd, *J*=1.83, 0.69 Hz, 1 H)



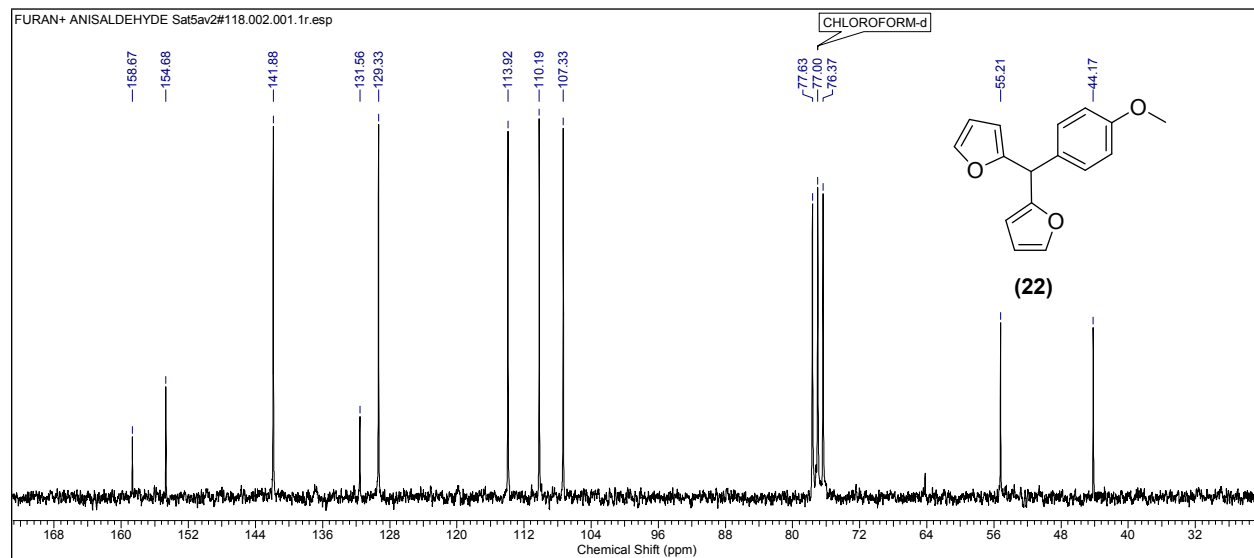
^1H NMR (200 MHz, CHLOROFORM-*d*) δ ppm 2.24 (s, 4 H) 3.84 - 3.86 (d, 5 H) 5.50 (s, 1 H) 5.85 (s, 2 H) 6.69 - 6.78 (d, J = 8.59 Hz, 2 H) 6.80 - 6.90 (d, J =6.82 Hz, 1 H)



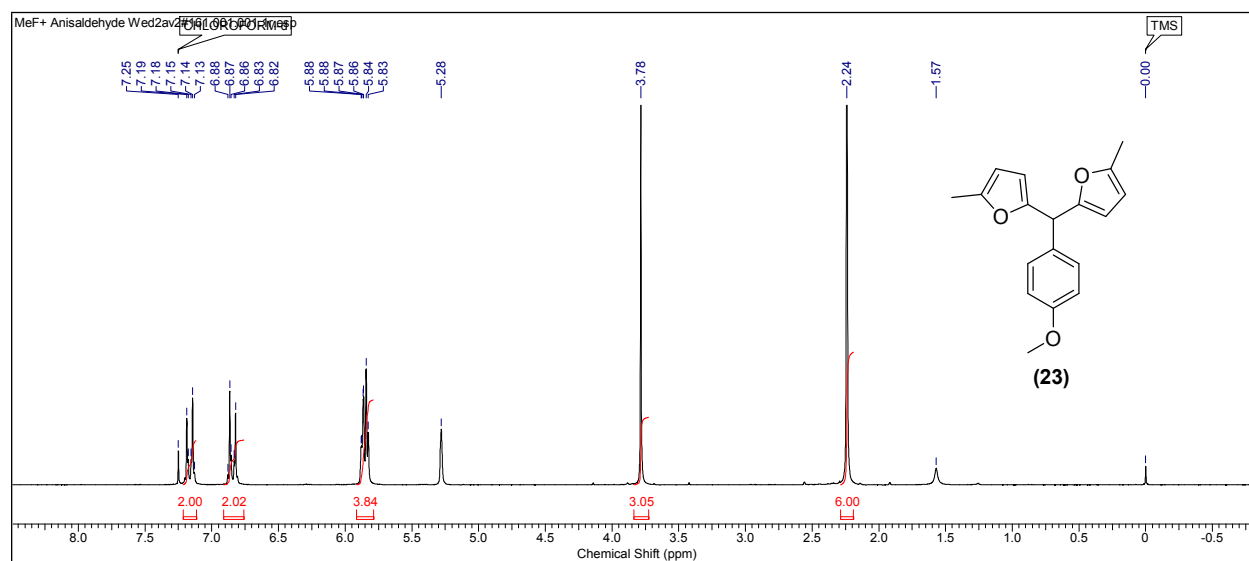
^{13}C NMR (50 MHz, CHLOROFORM-*d*) δ ppm 13.53, 34.19, 55.84, 105.95, 106.65, 111.26, 114.24, 121.38, 130.27, 144.13, 146.40, 150.90, 153.08



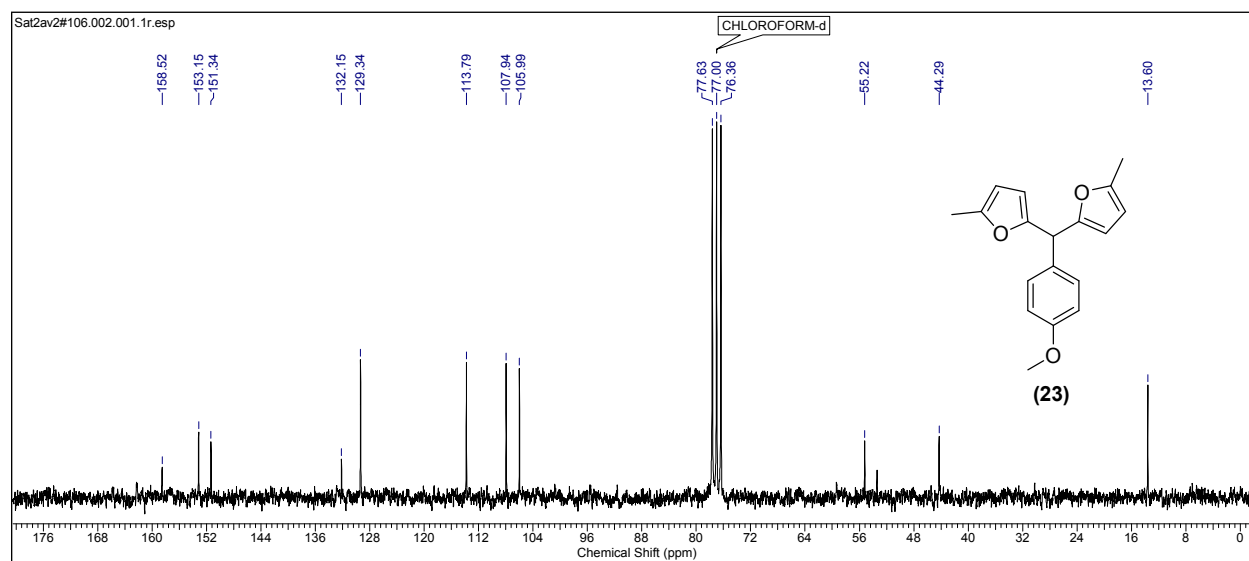
^1H NMR (200 MHz, CHLOROFORM-*d*) δ ppm 3.81 (s, 3 H) 5.42 (s, 1 H) 6.02 - 6.03 (m, 2 H) 6.32 -6.35 (dd, $J=3.22$, 1.83 Hz, 2 H) 6.86 - 6.90 (m, 2 H) 7.16 - 7.21 (m, 2 H) 7.37-7.39 (dd, $J=1.83$, 0.82 Hz, 2 H)



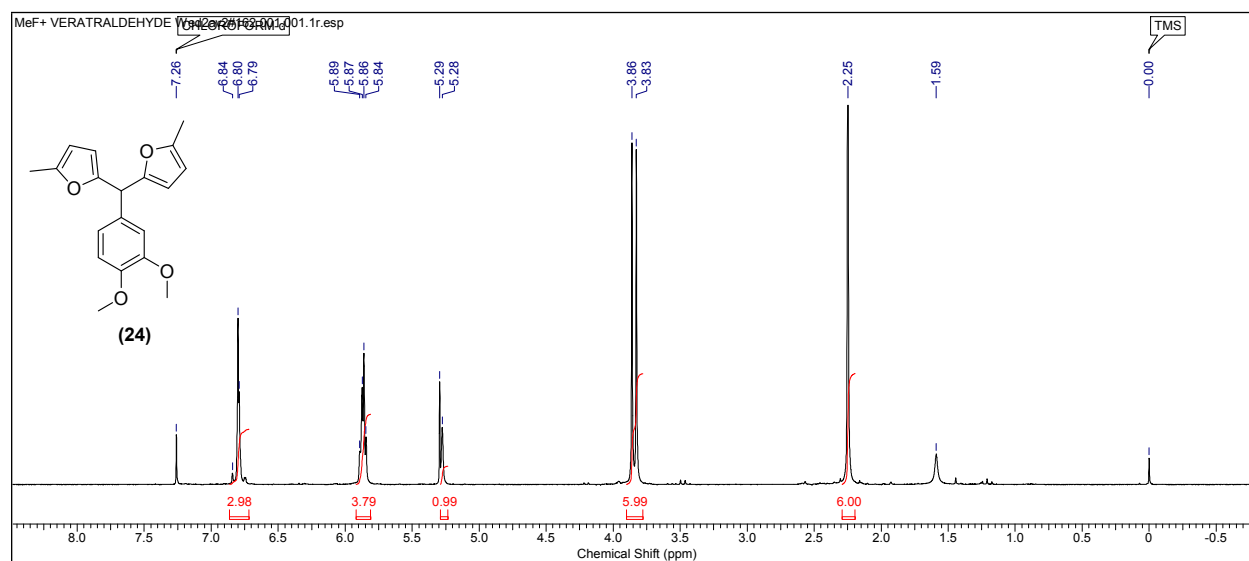
^{13}C NMR (50 MHz, CHLOROFORM-*d*) δ ppm 44.17, 55.21, 107.33, 110.19, 113.92, 129.33, 131.56, 141.88, 154.68, 158.67



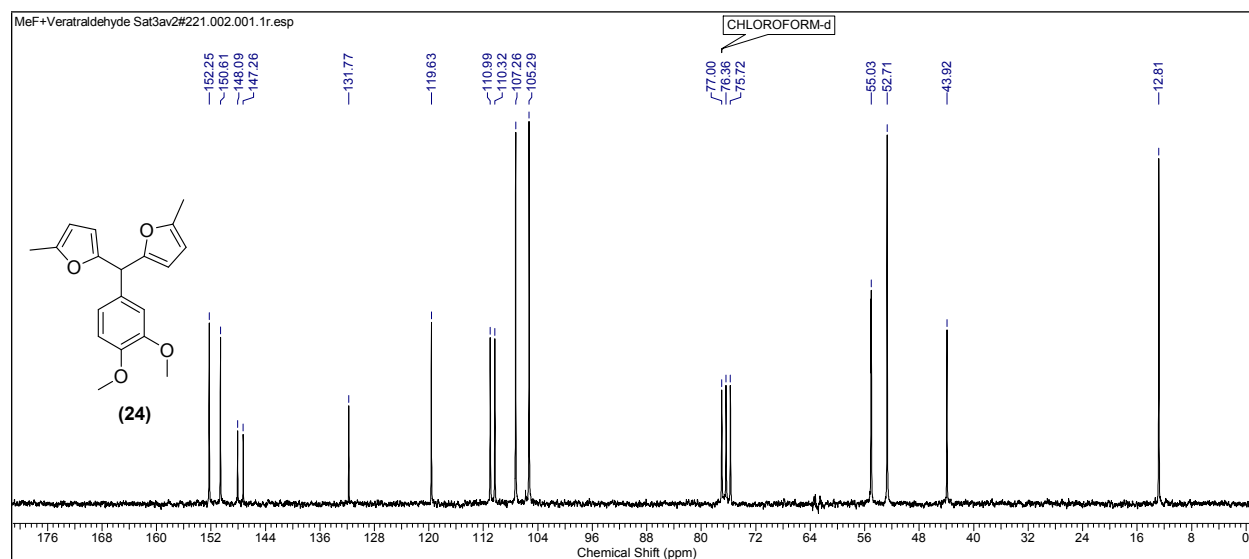
^1H NMR (200 MHz, CHLOROFORM-*d*) δ ppm 2.24 (s, 6 H) 3.78 (s, 3 H) 5.83 - 5.88 (m, 4 H) 6.82 - 6.88 (m, 2 H) 7.13 - 7.25 (m, 2 H)



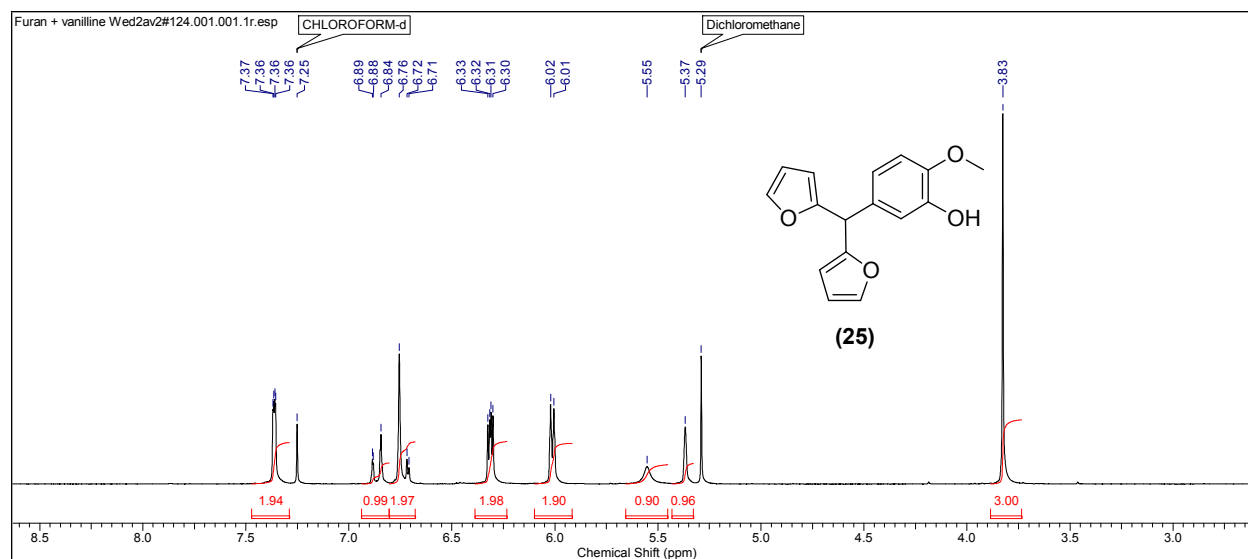
^{13}C NMR (50 MHz, CHLOROFORM-*d*) δ ppm 13.60, 44.29, 55.22, 105.99, 107.94, 113.79, 129.34, 132.15, 151.34, 153.15, 158.52.



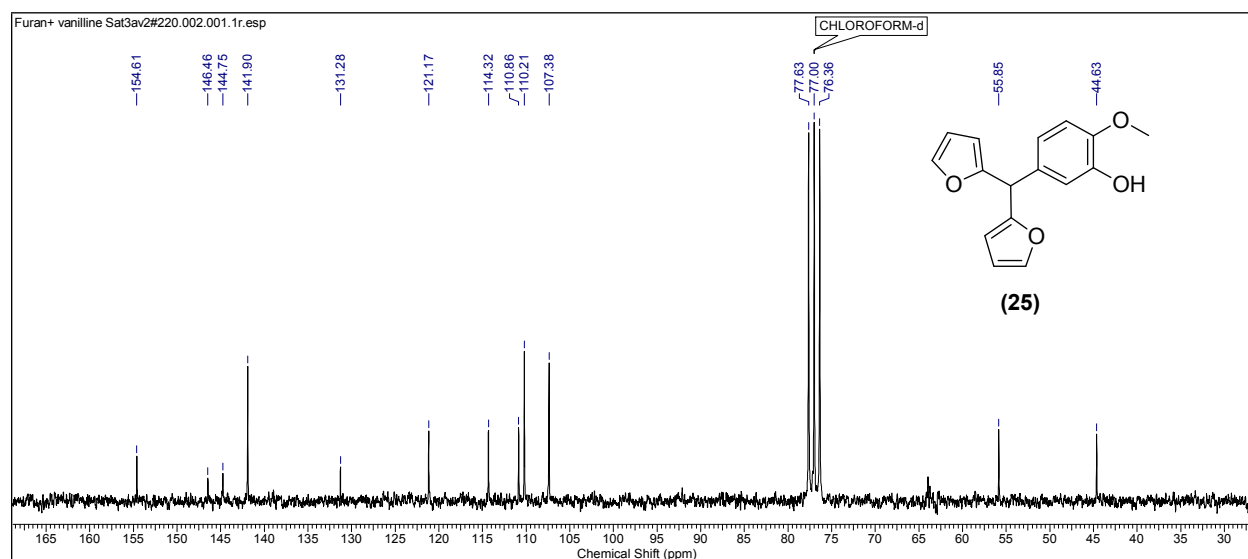
^1H NMR (200 MHz, CHLOROFORM-*d*) δ ppm 2.25 (s, 6 H) 3.86 (s, 3 H) 3.83 (s, 3 H) 5.28 (s, 1 H) 5.84 - 5.89 (m, 4 H) 6.79 - 6.84 (m, 3 H), (Singlet at 5.3 ppm is of residual dichloromethane).



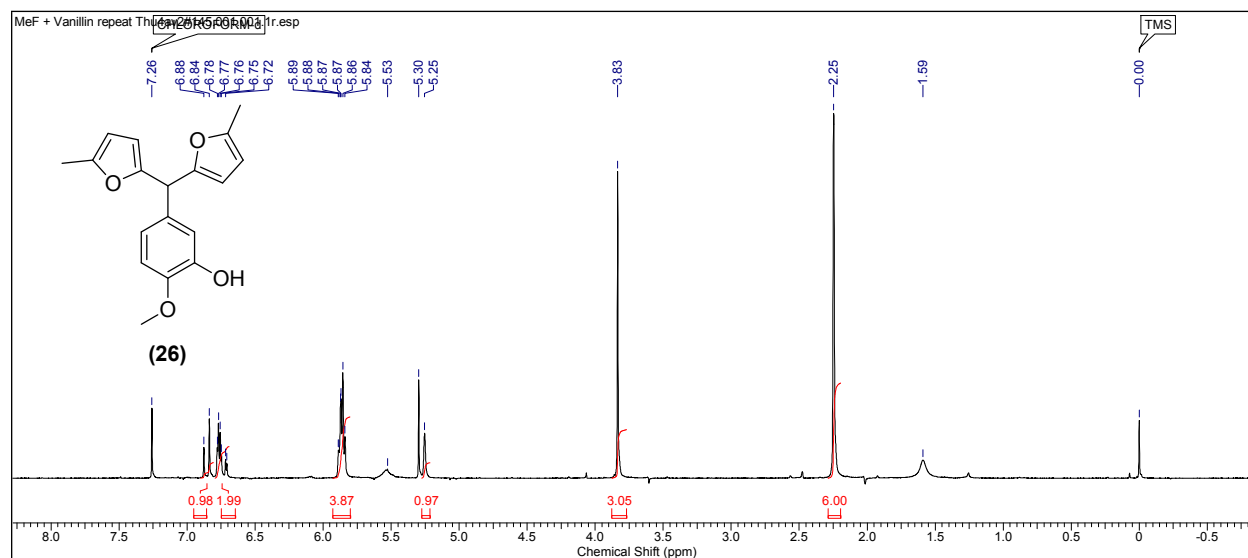
^{13}C NMR (50 MHz, CHLOROFORM-*d*) δ ppm 12.81, 43.92, 52.71, 55.03, 105.29, 107.26, 110.32, 110.99, 119.63, 131.77, 147.26, 148.09, 150.61 and 152.25



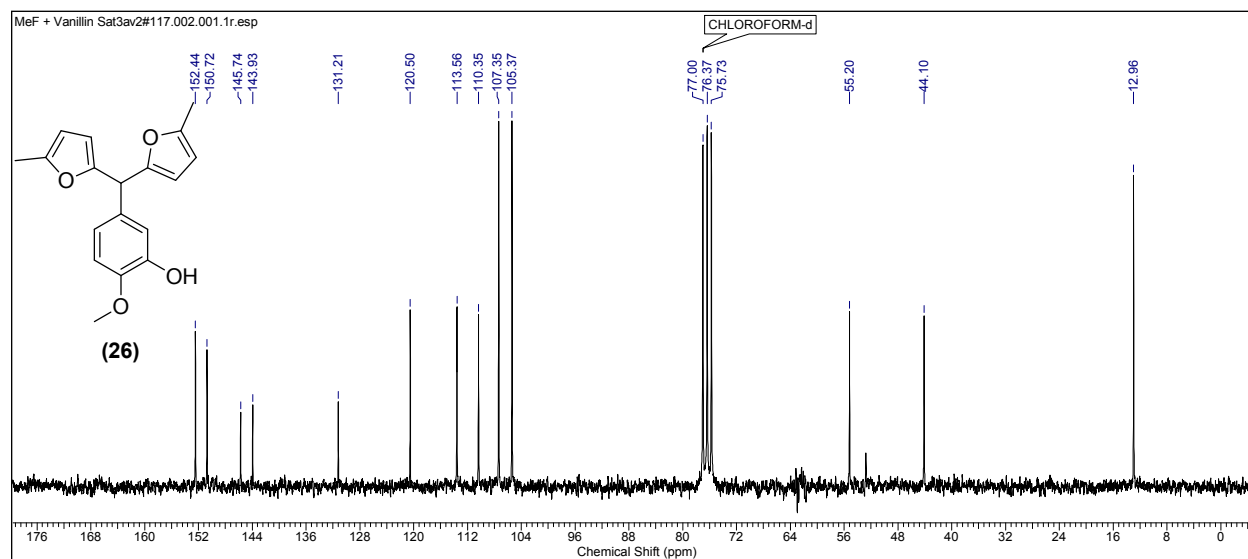
^1H NMR (200 MHz, CHLOROFORM-*d*) δ ppm 3.83 (s, 3 H) 5.37 (s, 1 H) 5.55 (br. s., 1 H) 6.01 - 6.02 (d, $J=3.16$ Hz, 2 H) 6.30 - 6.33 (dd, $J=3.16$, 1.89 Hz, 2 H) 6.71 - 6.76 (m, 2 H) 6.84 - 6.89 (m, 1 H) 7.36 - 7.37 (dd, $J=1.77$, 0.76 Hz, 2 H)



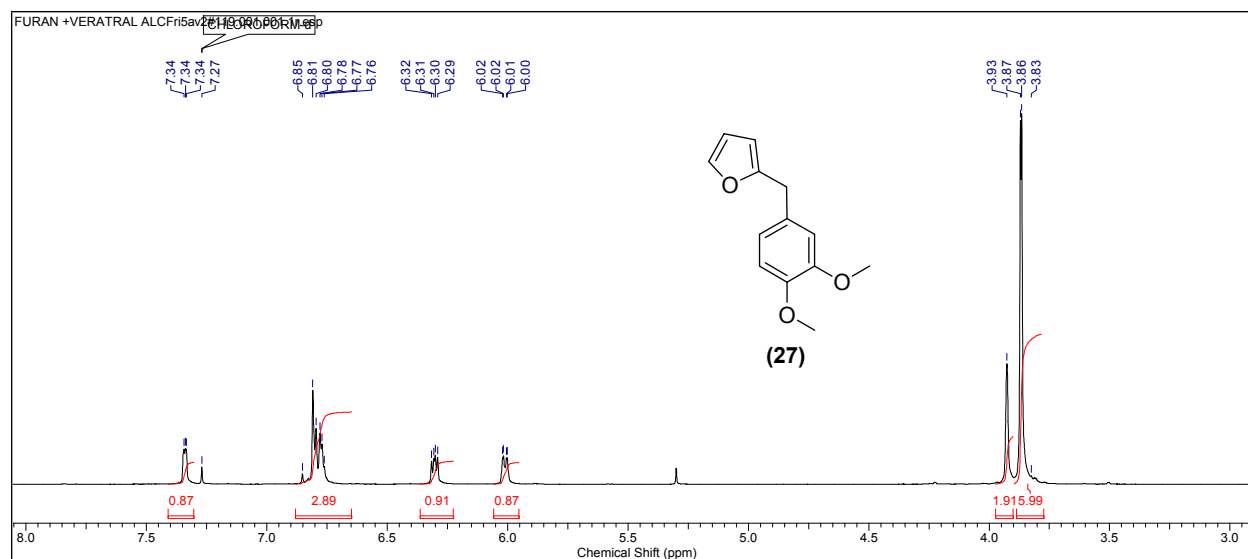
^{13}C NMR (50 MHz, CHLOROFORM-*d*) δ ppm 44.63, 55.85, 107.38, 110.21, 110.86, 114.32, 121.17, 131.28, 141.90, 144.75, 146.46, 154.61



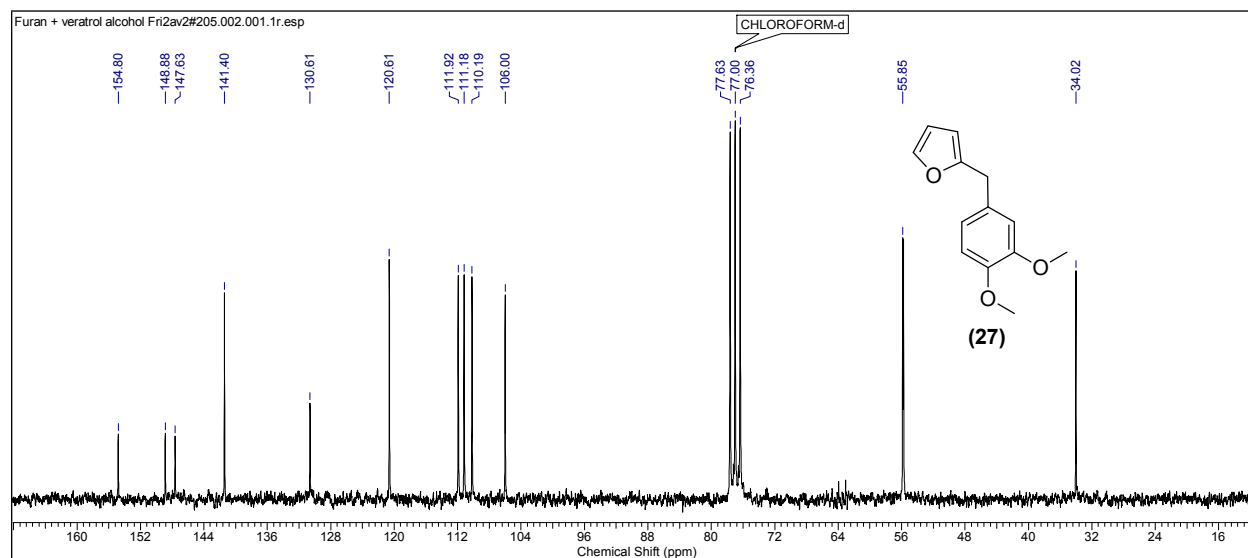
^1H NMR (200 MHz, CHLOROFORM-*d*) δ ppm 2.25 (s, 6 H) 3.83 (s, 3 H) 5.25 (s, 1 H) 5.84 - 5.89 (m, 4 H) 6.72 - 6.78 (m, 2 H) 6.84 - 6.88 (m, 1 H)



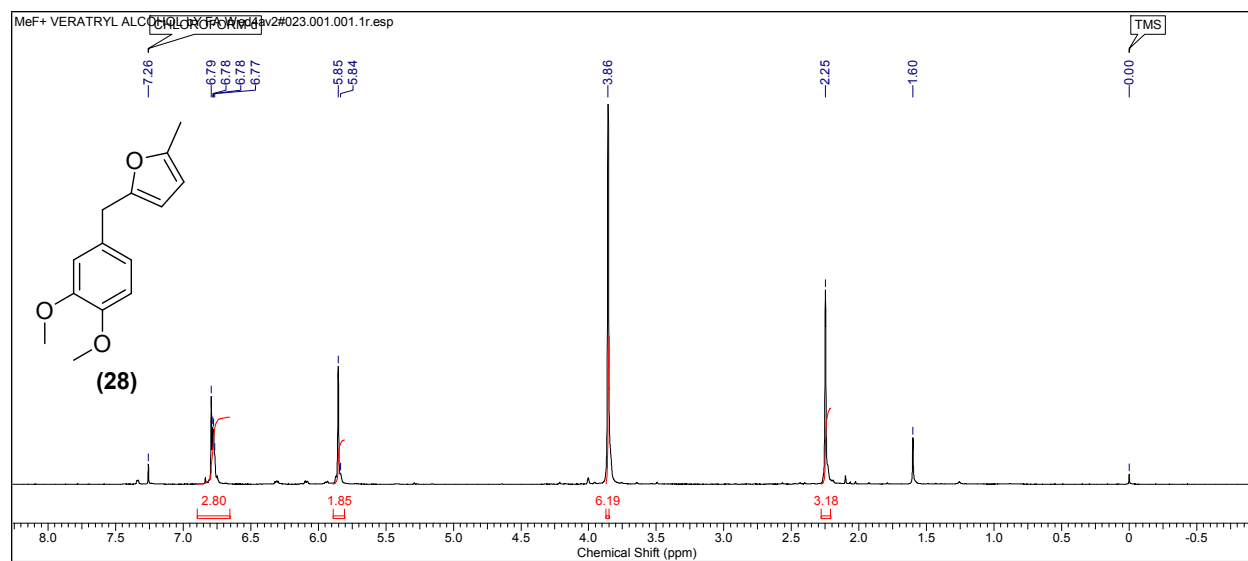
^{13}C NMR (50 MHz, CHLOROFORM-*d*) δ ppm 12.96, 44.10, 55.20, 105.37, 107.35, 110.35, 113.56, 120.50, 131.21, 143.93, 145.74, 150.72, 152.44



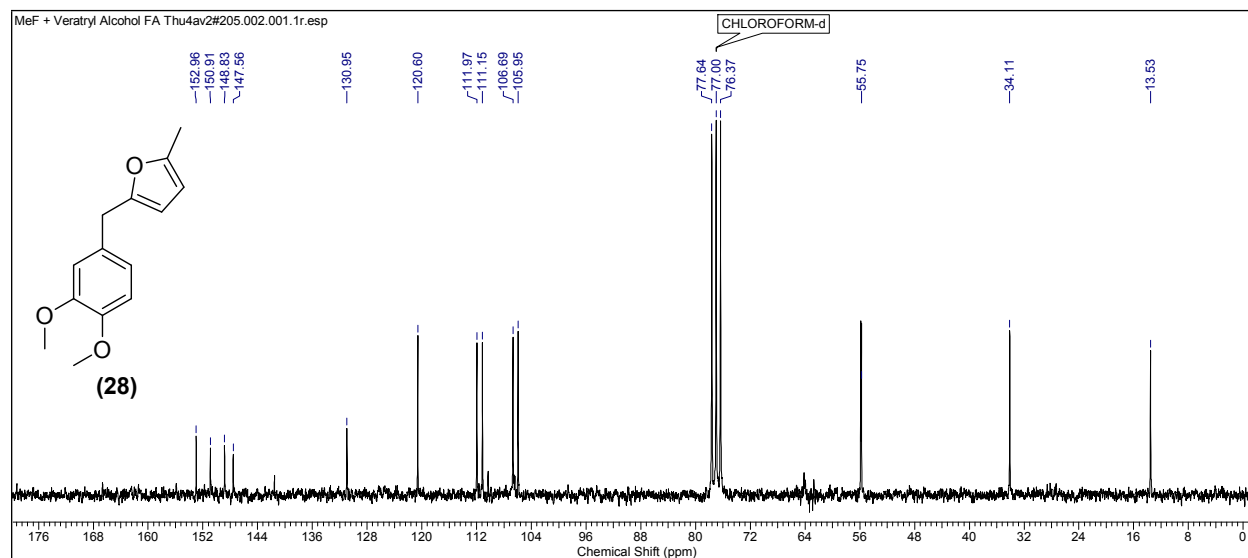
^1H NMR (200 MHz, CHLOROFORM-*d*) δ ppm 3.86 - 3.87 (d, 6 H) 3.93 (s, 2 H) 6.01 – 6.02 (dd, $J=3.16$, 0.76 Hz, 1 H) 6.29 - 6.32 (dd, $J=3.09$, 1.96 Hz, 1 H) 6.76 - 6.85 (m, 3 H) 7.34 (m, 1 H)



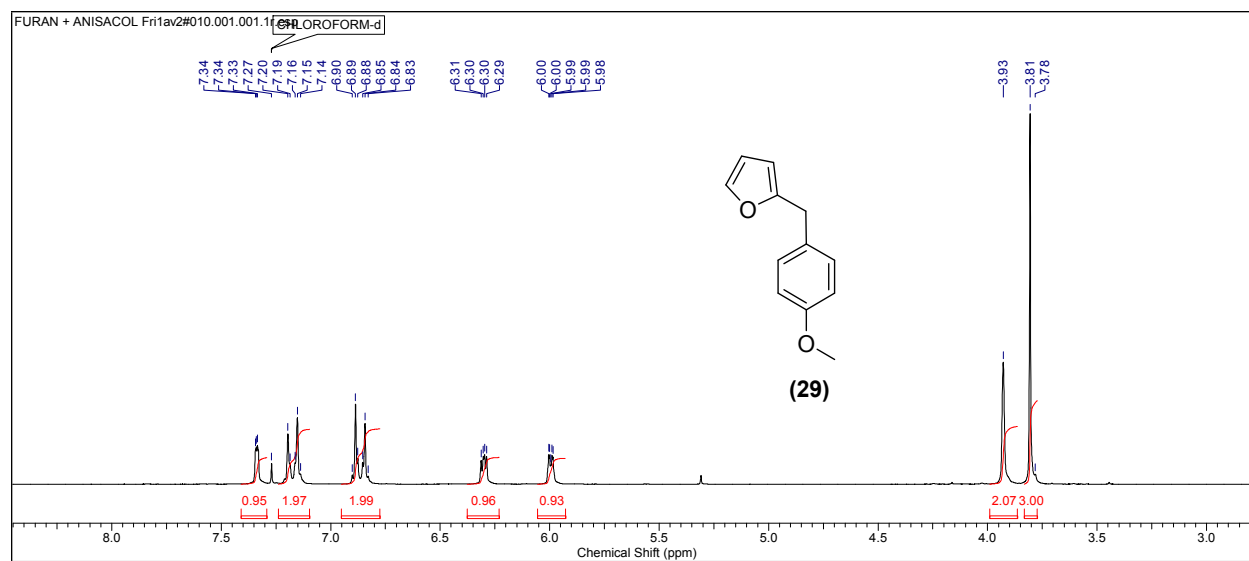
^{13}C NMR (50 MHz, CHLOROFORM-*d*) δ ppm 34.02, 55.85, 106.00, 110.19, 111.18, 111.92, 120.61, 130.61, 141.40, 147.63, 148.88, 154.80



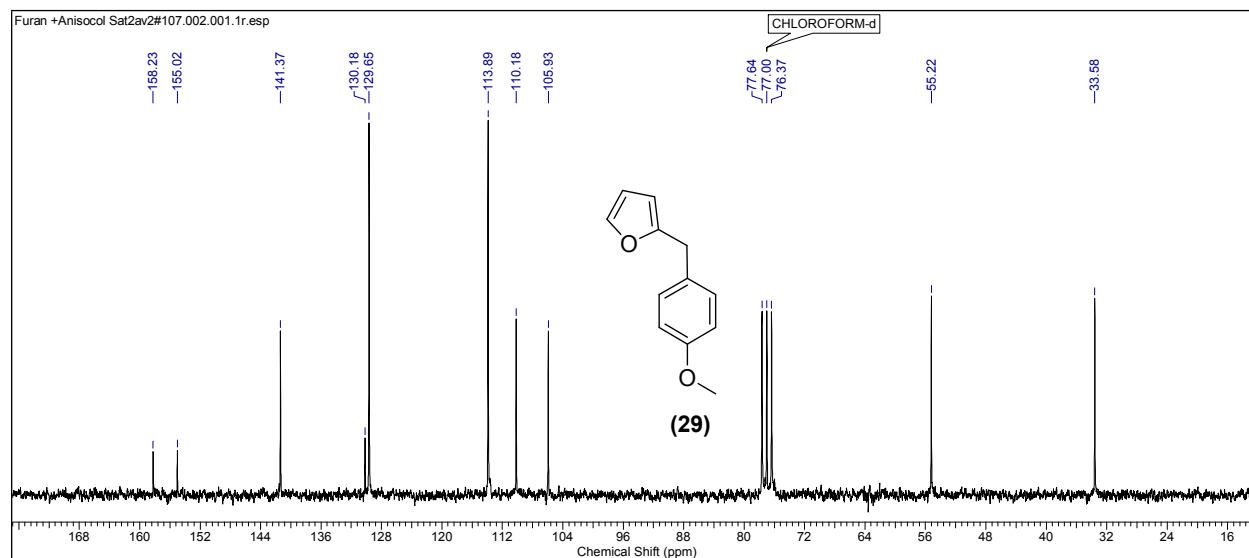
¹H NMR (200 MHz, CHLOROFORM-*d*) δ ppm 2.25 (s, 3 H) 3.86 (s, 6H) 5.84 - 5.85 (m, 2 H) 6.77 - 6.79 (m, 3 H)



¹³C NMR (50 MHz, CHLOROFORM-*d*) δ ppm 13.53, 34.11, 55.75, 105.95, 106.69, 111.15, 111.97, 120.60, 130.95, 147.56, 148.83, 150.91, 152.96



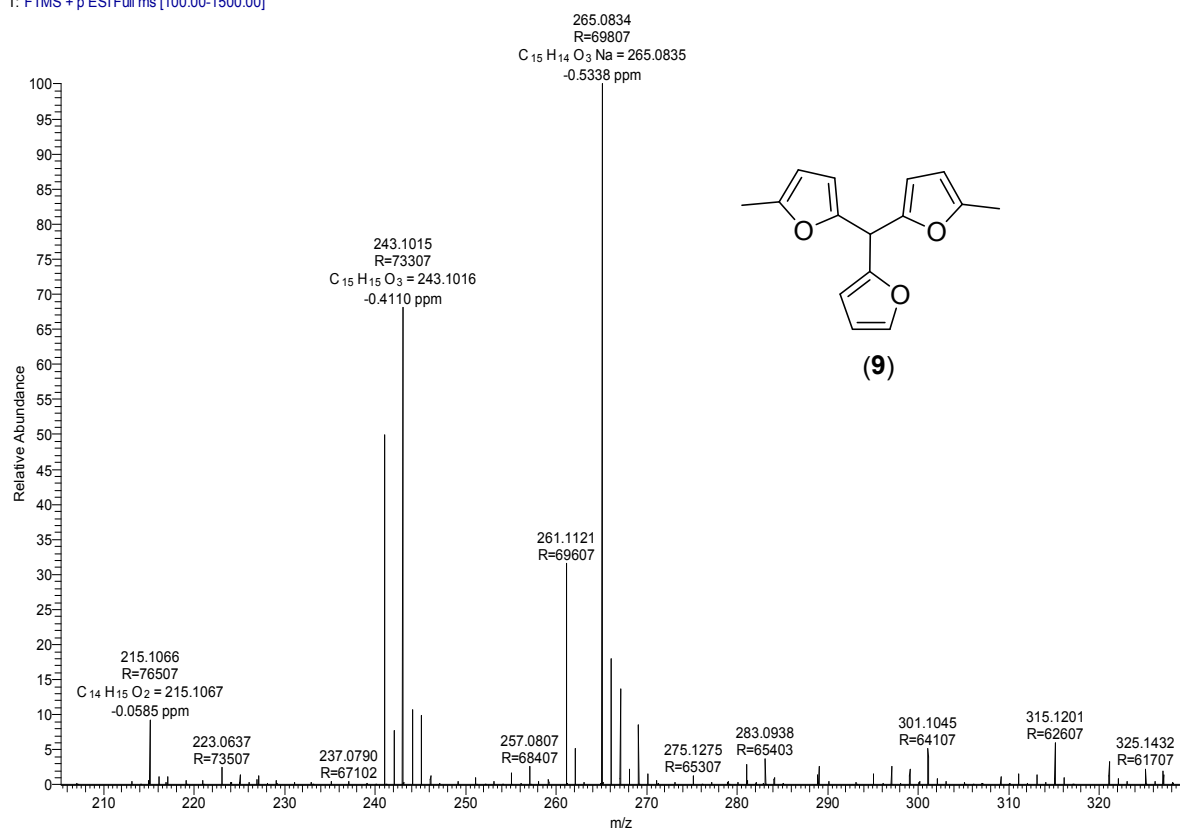
^1H NMR (200 MHz, CHLOROFORM-*d*) δ ppm 3.81 (s, 3 H) 3.93 (s, 2 H) 5.98 - 6.00 (m, 1 H) 6.29 - 6.31 (dd, $J=3.09$, 1.96 Hz, 1 H) 6.83 - 6.90 (m, 2 H) 7.14 - 7.20 (m, 2 H) 7.33 - 7.34 (m, 1 H)



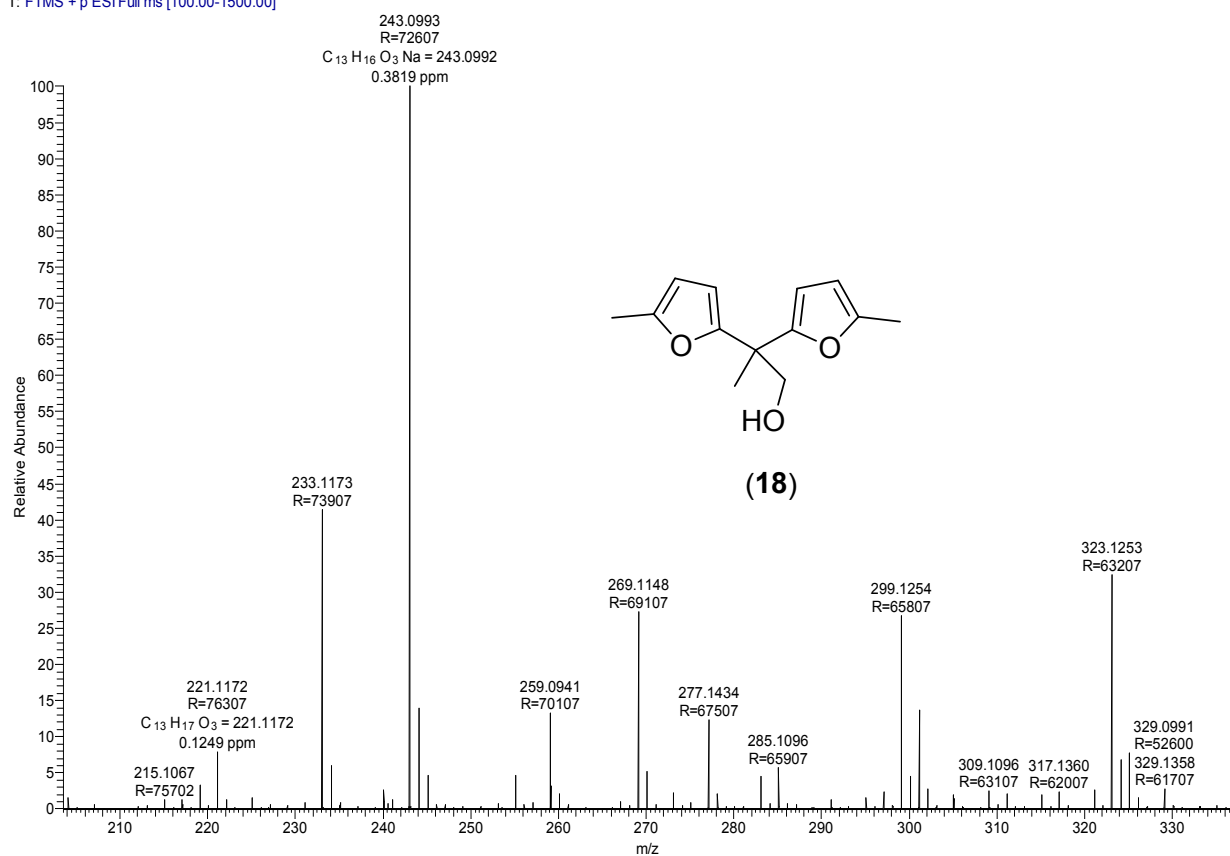
^{13}C NMR (50 MHz, CHLOROFORM-*d*) δ ppm 33.58, 55.22, 105.93, 110.18, 113.89, 129.65, 130.18, 141.37, 155.02, 158.23

HRMS analysis

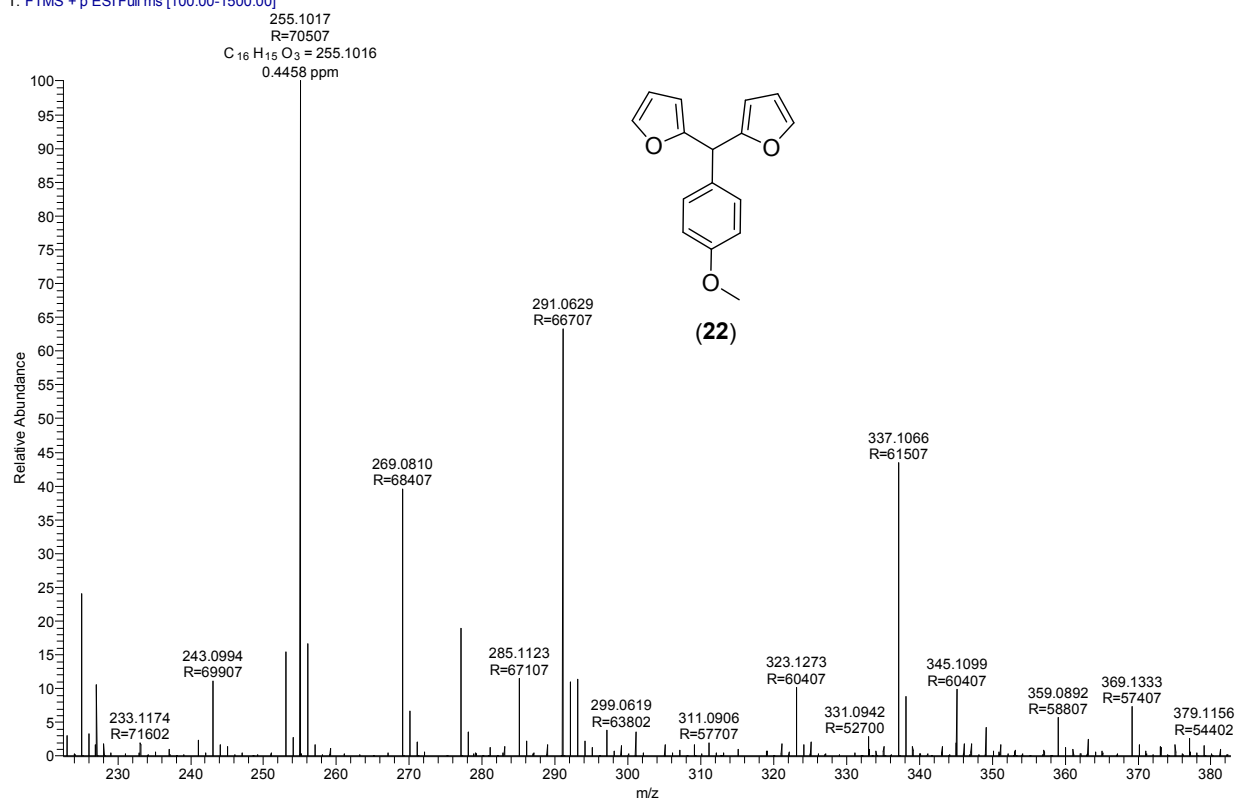
SHS-ALK-2 #127 RT: 0.57 AV: 1 NL: 2.14E8
T: FTMS + p ESI Full ms [100.00-1500.00]



SHS-ALK-4 #119 RT: 0.53 AV: 1 NL: 1.05E9
T: FTMS + p ESI Full ms [100.00-1500.00]



SHS-ALK-5 #128 RT: 0.57 AV: 1 NL: 1.79E8
T: FTMS + p ESI Full ms [100.00-1500.00]



SHS-ALK-1 #118 RT: 0.52 AV: 1 NL: 2.09E8
T: FTMS + p ESI Full ms [100.00-1500.00]

