Experimental Protocol for the preparation of a precursor of biofuel from Furfural and 2-methylfuran

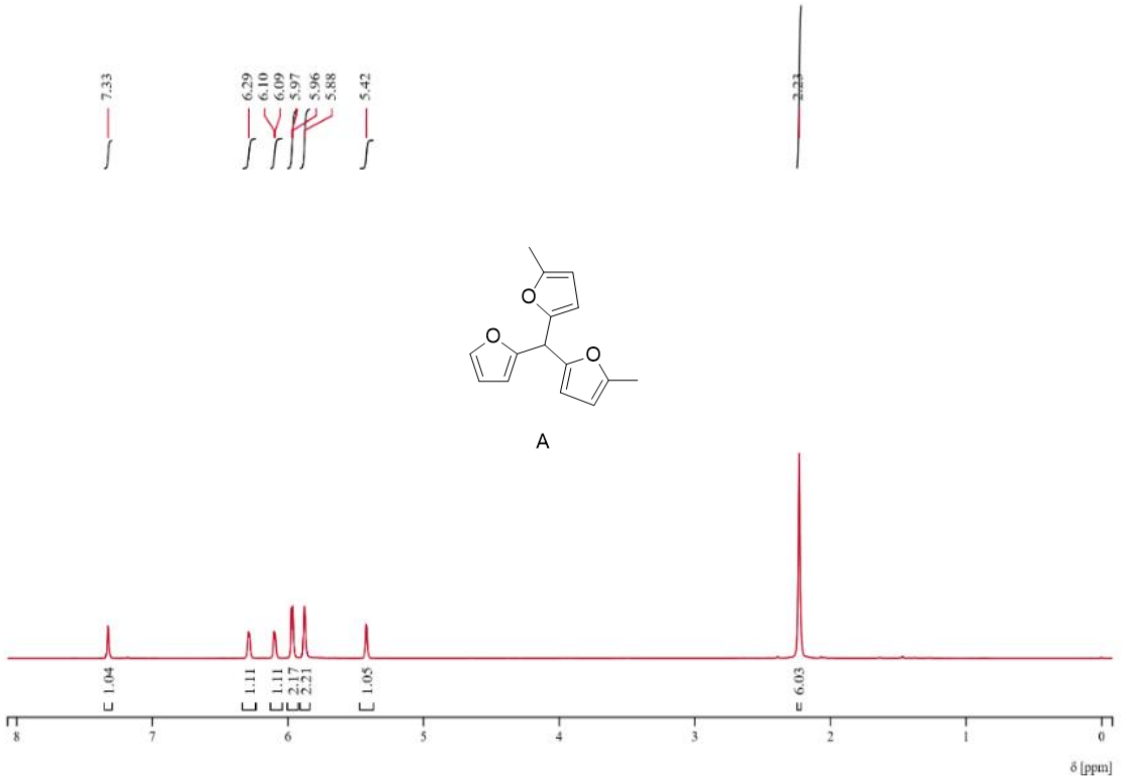
A 9 mL pressure tube was charged with 0.5 mL of 2-methylfuran (used as both solvent and reactant), furfural (41.4 µL, 0.5 mmol, 1 equiv.), and PSSA catalyst (4.93 mg, 0.025 mmol, 5 mol%). The reaction mixture was stirred at 70 °C for 2 hours and monitored by TLC (hexane: ethyl acetate, 4:1).

Upon completion, the reaction mixture was filtered under vacuum to recover the PSSA catalyst. The filtrate was transferred to a round-bottom flask, diluted with ethyl acetate, and concentrated using a rotary evaporator. The crude product was purified by column chromatography using a 19:1 mixture of hexane: ethyl acetate as the eluent.

Fractions containing the product were collected, transferred to a tared round-bottom flask, and the solvent was removed. The flask was then placed on a Schlenk line for further drying.

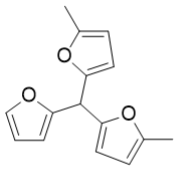
The product was obtained quantitatively as a light pink oil and subjected to IR and NMR analysis.

- **IR (oil) ν** 3107, 2981, 2951, 2922, 2883, 1451, 1195, 1073, 934, 886, 816 cm^{-1}
- **^{1}H NMR (400 MHz, $CDCl_3$) δ** 7.33 (d, 1H), 6.29 (d, *J*=3.13 Hz, 1H), 6.10 (m, 1H), 5.97 (s, *J*=3.13 Hz, 2H), 5.89-5.88 (m, 2H), 5.42(s, 1H), 2.23 (d, 6H)
- **^{13}C NMR (100 MHz, $CDCl_3$) δ** 152.6, 151.6, 150.4, 141.9, 110.4, 108.0, 107.2, 106.4, 39.1, 13.6.



A


152.68
151.57
150.45
141.92
110.40
108.02
107.18
106.37
39.18
13.63



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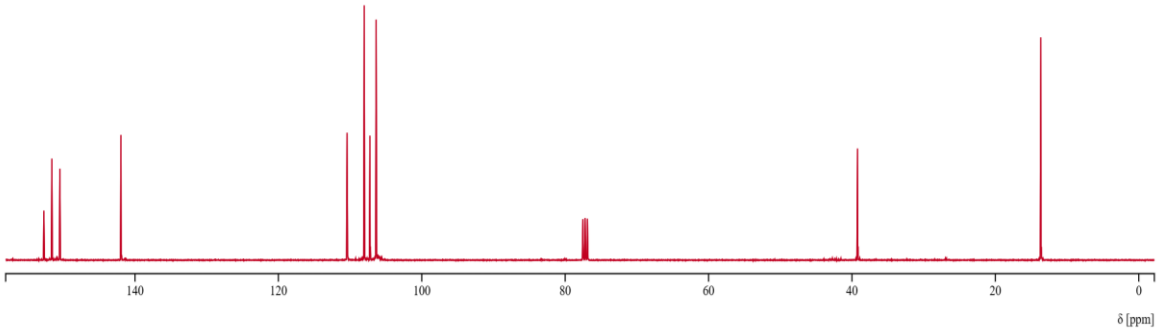
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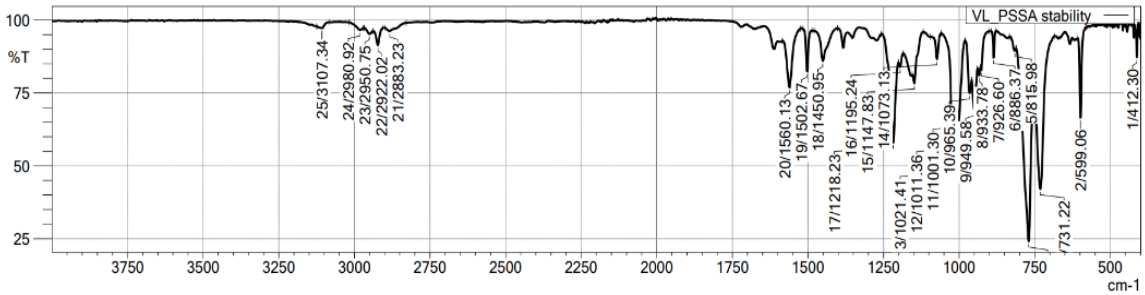
120
100
80
60
40
20
0
δ [ppm]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2250
2000
1750
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1000
750
500
cm-1