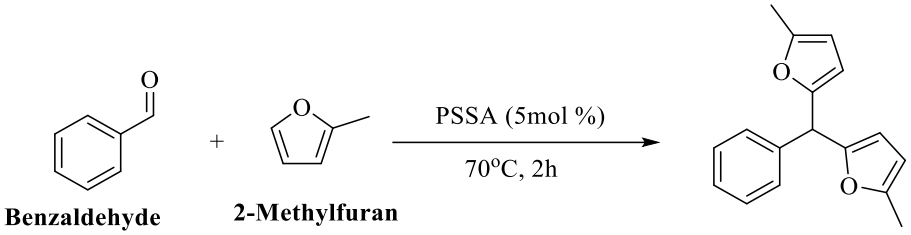
Experimental Protocol for the Friedel-Crafts reaction of benzaldehyde with 2-methylfuran



Reference: RSC Sustainability, 2026,4, 1986-1995. https://doi.org/10.1039/D5SU00927H

WATCH STEP-BY-STEP VIDEO OF A SIMILAR PROTOCOL

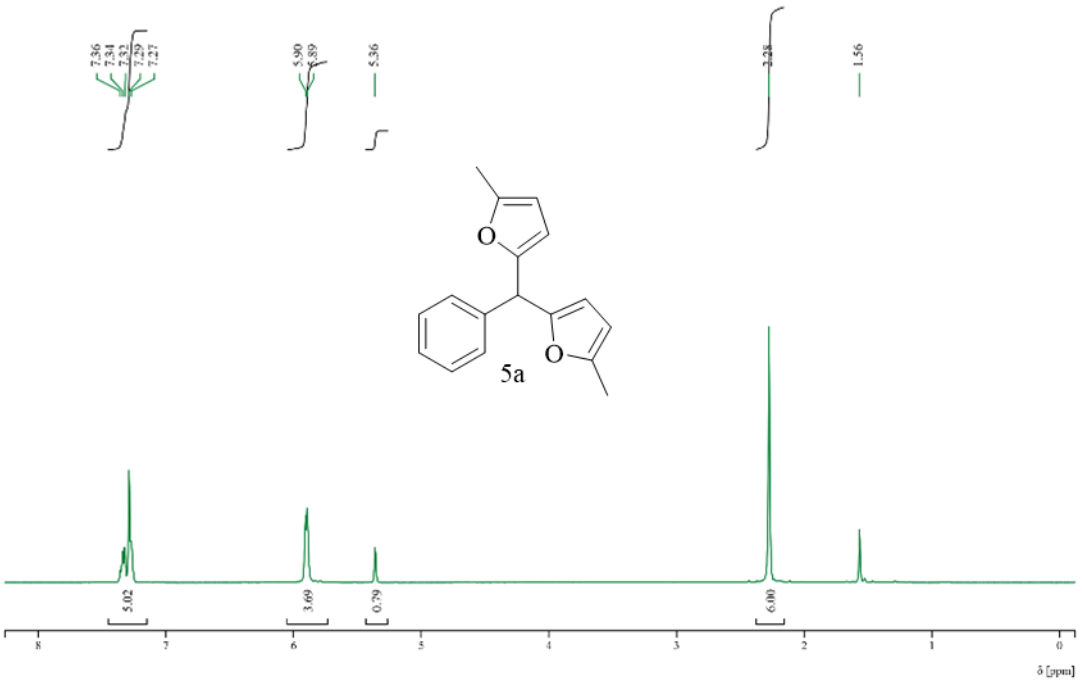
A 9 mL pressure tube was charged with 0.5 mL of 2-methylfuran (used as both solvent and reactant), benzaldehyde (50.5 µL, 0.5 mmol, 1 equiv.), and PSSA catalyst (4.93 mg, 0.025 mmol, 5 mol%). The reaction mixture was stirred at 70 °C for 2 hours and monitored by TLC (hexane: ethyl acetate, 4:1).

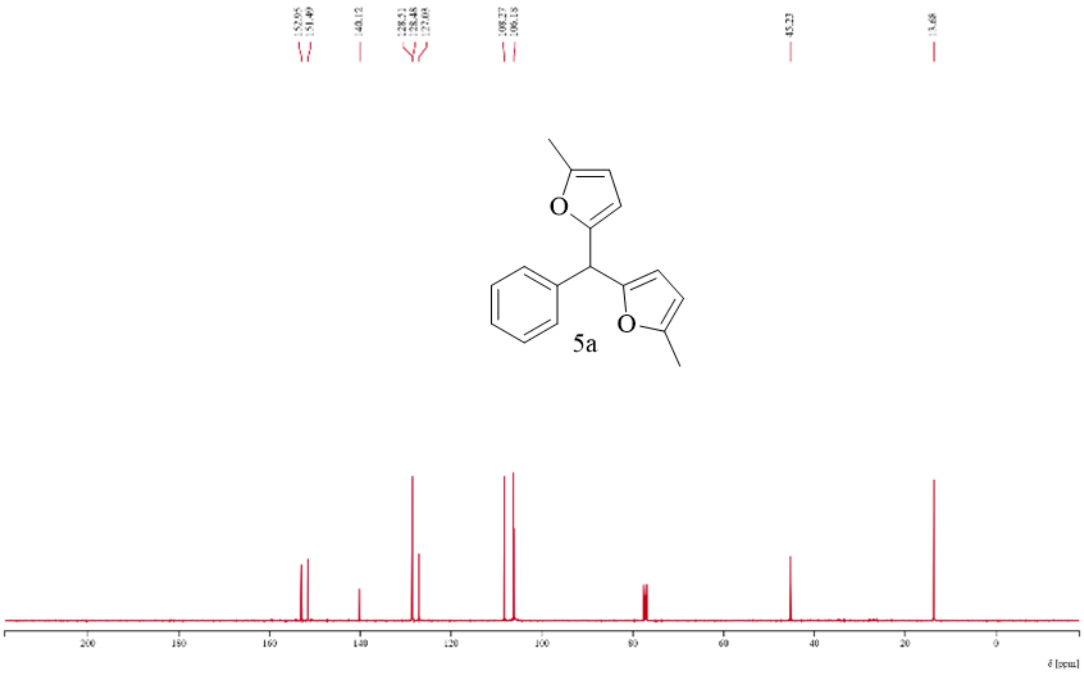
Upon completion, the reaction mixture was filtered under vacuum to recover the PSSA catalyst. The filtrate was transferred to a round-bottom flask, diluted with ethyl acetate, and concentrated using a rotary evaporator. The crude product was purified by column chromatography using a 24:1 mixture of hexane: 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-yellow oil (126 mg, 0.5 mmol) and subjected to IR and NMR analysis.

- **IR (oil) ν** 3104, 3064, 3028, 2978, 2949, 2922, 2883, 1603, 1495, 1452, 1185 cm^{-1}
- **^{1}H NMR (400 MHz, $CDCl_3$) δ** 7.34-7.21 (m, 5H), 5.87-5.85 (m, 4H), 5.36 (s, 1H), 2.28 (s, 6H).
- **^{13}C NMR (100 MHz, $CDCl_3$) δ** 152.9, 151.5, 140.1, 128.6, 128.4, 127.1, 108.3, 106.1, 45.2, 13.6


7.36
7.34
7.32
7.29
7.27
5.90
5.89
5.36
2.28
1.56
5a
5.02
3.69
0.79
6.00
δ [ppm]


152.95
151.49
140.12
128.51
128.48
127.69
108.27
106.15
45.23
13.68
5a
δ [ppm]