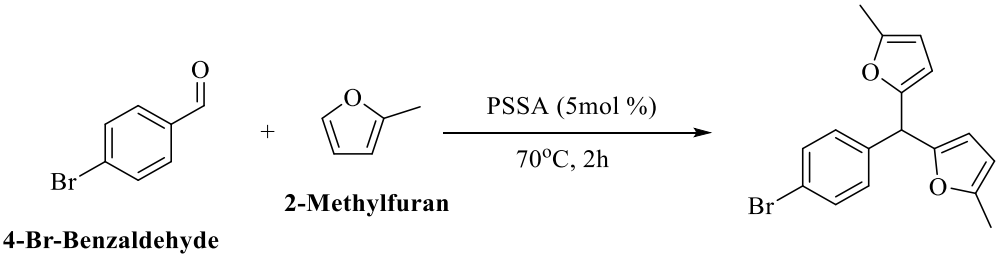
Experimental Protocol for the Friedel-Crafts reaction of 4-Br-benzaldehyde with 2-methylfuran



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

WATCH STEP-BY-STEP VIDEO OF A SIMILAR PROTOCOL

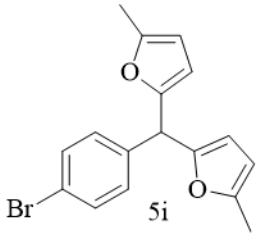
A 9 mL pressure tube was charged with 0.5 mL of 2-methylfuran (used as both solvent and reactant), 4-Bromobenzaldehyde (92.5 mg, 0.5 mmol, 1 equiv.), and PSSA catalyst (4.93 mg, 0.025 mmol, 5 mol%). The reaction mixture was stirred at 70 °C for 2 hours and monitored by TLC.

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 23:2 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 brown oil (165 mg, 0,5 mmol) and subjected to IR and NMR analysis.

- **IR (oil) ν** 3106, 2978, 2921, 2883, 1610, 1560, 1450, 1404, 1382, 1167, 963 cm^{-1}
- **^{1}H NMR (400 MHz, $CDCl_3$) δ** 7.32 (d, *J* = 8.00 Hz, 2H), 7.02 (d, *J* = 8.00 Hz, 2H), 5.80-5.76 (m, 4H), 5.19 (s, 1H), 2.14 (s, 6H).
- **^{13}C NMR (100 MHz, $CDCl_3$) δ** 152.2, 151.7, 139.2, 130.9, 121.0, 108.4, 106.2, 44.6, 13.6.



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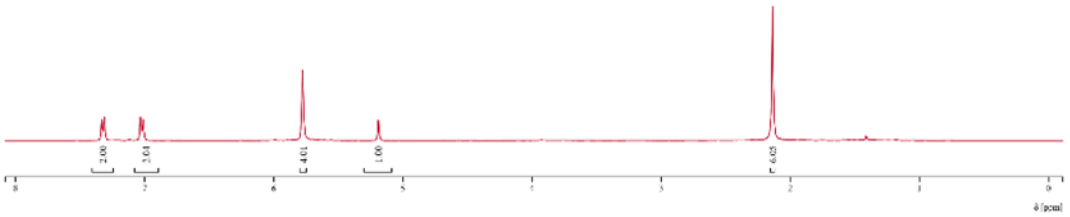

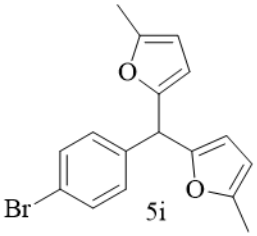

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