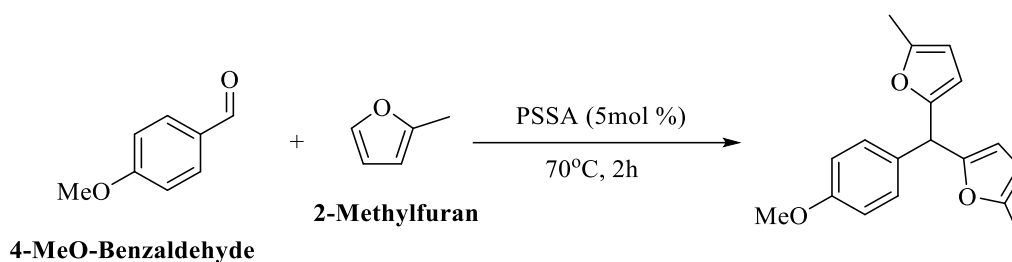


Experimental Protocol for the Friedel-Crafts reaction of 4-MeO-benzaldehyde with 2-methylfuran



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-Methoxybenzaldehyde (60.8 μL , 0.5 mmol, 1 equiv.), and PSSA catalyst (4.93 mg, 0.025 mmol, 5 mol%). The reaction mixture was stirred at 70 $^{\circ}\text{C}$ 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 with a 93% yield as a brown oil (131 mg, 0,46 mmol) and subjected to IR and NMR analysis.

- **IR (oil) ν** 3104, 2952, 2922, 2836, 1612, 1585, 1462, 1452, 1442, 1109, 856 cm^{-1}
- **^1H NMR (400 MHz, CDCl_3) δ** 7.07 (d, J = 8.25 Hz, 2H), 6.74 (d, J = 8.25 Hz, 2H), 5.76-5.75 (m, 4H), 5.19 (s, 1H), 3.65 (s, 3H), 2.13 (s, 6H).
- **^{13}C NMR (100 MHz, CDCl_3) δ** 158.6, 153.9, 151.4, 132.2, 129.4, 113.8, 108.1, 106.1, 55.2, 44.4, 13.6.

