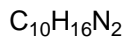


Chemical Formula:



Molecular Weight:

164.25

Preparation of 3-butyl-2,3-dimethylpyrazine (IV-JEH-018)

A dry 250 mL round-bottom flask fitted with a magnetic stir bar and a septum was flushed with N_2 gas and charged with 3-chloro-2,5-dimethylpyrazine (1.97 g, 13.8 mmol, 1 eq.). Column dried ether (60 mL) was added by syringe and the reaction mixture was cooled to $-78\text{ }^\circ\text{C}$ in a dry ice/acetone bath under an atmosphere of N_2 gas (balloon). Dimethylformamide (3.2 mL, 41.4 mmol, 3 eq) was added in one portion to the stirred reaction mixture. Using an oven-dried, glass syringe, *n*-BuLi (2 M in pentane, 7.3 mL, 1.45 mmol, 1.05 eq) was added in one portion and the reaction mixture immediately turned a deep red color. The red slurry was stirred at $-78\text{ }^\circ\text{C}$ for 1 h, and let warm to $0\text{ }^\circ\text{C}$ in an ice bath over 20 min. The reaction was quenched with saturated aqueous NH_4Cl (100 mL) and extracted with ether (3×70 mL). The combined organic layers were dried over MgSO_4 and the solvent was removed by careful fractional distillation. The residue was purified by flash chromatography (silica gel, 8% - 15% Et_2O in pentane) and the pure fractions (as judged by TLC) were pooled. The solvent was removed from the pooled fractions by fractional distillation giving the product as a yellow oil (832 mg, 37%).

^1H NMR (300 MHz, CDCl_3) δ 8.08 (s, 1H), 2.70 (t, $J = 7.8$ Hz, 2 H), 2.45 (s, 3H), 2.42 (s, 3 H), 1.66-1.52 (m, 2H), 1.36 (tq, $J = 7.6, 7.6$ Hz, 2H), 0.91 (t, $J = 7.5$ Hz, 3H)

^{13}C NMR (75 MHz, CDCl_3) δ 154.9, 150.0, 148.5, 140.6, 34.9, 30.8, 22.8, 21.2, 21.1, 13.9

IR (film, cm^{-1}) 3043 (aromatic C-H stretch), 2959 (aliphatic C-H stretch), 2930 (aliphatic C-H stretch), 2873 (aliphatic C-H stretch), 1453 (methyl C-H bend), 1374 (methyl C-H bend)