ORGANIC LETTERS

2012
Vol. 14, No. 14
3812–3813

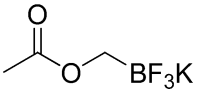
Additions and Corrections

Volume 14, 2012

Norio Murai, Masahiro Yonaga,* and Keigo Tanaka*

Palladium-Catalyzed Direct Hydroxymethylation of Aryl Halides and Triflates with Potassium Acetoxymethyltrifluoroborate

Page 1281. Page S2, Supporting Information. Preparation of potassium acetoxymethyltrifluoroborate was incorrectly reported. A corrected synthetic method is presented below.



Preparation of Potassium Acetoxymethyltrifluoroborate 1. To a mixture of 2-(bromomethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (16.6 g, 75.0 mmol) and tetrahydrofuran (150 mL) was added potassium acetate (8.83 g, 90.0 mmol) at 0 °C. Then, the reaction mixture was stirred at 50 °C for 14 h. After the reaction mixture was cooled at 0 °C, to the reaction mixture was added potassium hydrogen fluoride (14.6 g, 187 mmol). Distilled water (75.0 mL) was added dropwise to the stirring solution at the same temperature over 1.5 h, and then reaction mixture was stirred at room temperature for 30 min. The reaction mixture was concentrated under reduced pressure. Moreover residual water was removed by azeotroping with toluene before drying in vacuo over 5 h. To the resulting solid were added acetone (385 mL) and methanol (115 mL), and then the reaction mixture was stirred at 50 °C for 30 min, filtered, and concentrated under reduced pressure. To the resulting solid was added acetonitrile/methanol (30/1) solution, and then the mixture was stirred at 60 °C for 30 min and filtered. Ethyl acetate was added to the filtrate until the solid appeared. The resulting solid was filtered and dried under reduced pressure to obtain the potassium acetoxymethyltrifluoroborate **1** as a white solid (10.9 g, 60.6 mmol, 80.8%): ^{1}H NMR (400 MHz, DMSO-d_6) δ 3.00−3.12 (m, 2H),

1.87 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 171.6, 21.2; ^{19}F NMR (376.5 MHz, DMSO-d_6) δ 149.0; ^{11}B NMR (128.4 MHz, DMSO-d_6) δ 3.2; IR (ATR, cm^{-1}) 1725, 1381, 1335, 1272, 1103, 1005, 957, 882, 785; HRMS (ESI-) calcd for $C_3H_5BF_3O_2$ (M $-$ H) 141.0329, found 141.0334.

10.1021/ol3017512
Published on Web 07/10/2012