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| --- | --- | --- | --- | --- | --- | --- |
| **Reaction code** | | DM054 | | | **Date** | **2016-04-11** |
| **Reaction scheme** | | | | | | |
| |  |  |  |  |  |  |  |  |  | | --- | --- | --- | --- | --- | --- | --- | --- | --- | | **Compound** | w/% | c/mol L-1 | M/g mol-1 | eq | n/mmol | ρ/g mL-1 | m/g | V/mL | |  | - | - | 112.172 | 1.00 | 10.16 | - | 1.14 |  | | TMSOTf | - | - | 222.253 | 1.10 | 11.18 | - | 2.48 |  | | NEt3 | - | - | 101.193 | 1.10 | 11.18 | 0.700 | 1.13 | 1.62 | |  | - | - | 203.102 | 1.00 | 10.16 | - | 2.06 |  | |  | - | - | 179.204 | 3.00 | 30.49 | - | 5.46 |  | | HBr | 33.00 | - | 80.912 | 4.50 | 45.73 | 1.179 | 11.2 | 9.51 | | H2O | - | - | 18.015 | 10.0 | 101.6 | 0.998 | 1.83 | 1.83 | |  | - | - | 162.180 | 1.00 | 10.16 | - | 1.65 |  | |  | | | | | Yield: | | 1.05 | 63.6 % | | | | | | | |
| **Literature** | 10.1021/acs.orglett.5b00097 | | | | | |
| **Procedure** | | | | | | |
| According to literature (1, sup. info - general procedure 1). 9.49 mL 33 % HBr in AcOH was used. Reaction was quenched with saturated aqueous NaHCO3 (10 mL) and diluted with water. Mixture was extracted with hexane/Et2O (3x10 mL, 1:1, v/v), organics extract were dried over anhydrous MgSO4, filtered and solvent was evaporated at reduced pressure to yield dark orange liquid. Crude product was analyzed by GC/MS. Crude product was filtered through silica by rincing it with DCM/Hexane (50 mL, 1:1, v/v). Solvent was evaporated at reduced pressure. Yield 80 mg of almost colorless liquid with strong pleasant aroma. NMR, GC/MS - DM054A1. | | | | | | |
| **Chemical analysis** | NMR | | HRMS | GC/HPLC | Melting point | Other |
| 1H | 13C |
| DM054A1 |  |  |  |  |  |

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| **Reaction code** | | DMb075 | | | **Date** | **2017-05-19** |
| **Reaction scheme** | | | | | | |
| |  |  |  |  |  |  |  |  |  | | --- | --- | --- | --- | --- | --- | --- | --- | --- | | **Compound** | w/% | c/mol L-1 | M/g mol-1 | eq | n/mmol | ρ/g mL-1 | m/g | V/mL | |  | - | - | 178.038 | 2.00 | 13.43 | - | 2.39 |  | |  | - | - | 148.974 | 1.00 | 6.713 | - | 1.00 |  | | K2CO3 | - | - | 138.205 | 2.00 | 13.43 | - | 1.86 |  | | Pd(PPh3)2Cl2 | - | - | 701.904 | 0.00594 | 0.03989 | - | 0.0280 |  | |  | - | - | 344.502 | 1.00 | 6.713 | - | 2.31 |  | |  | | | | | Yield: | |  |  | | | | | | | |
| **Literature** | 10.1039/c5cc07523h, SI page S2 | | | | | |
| **Procedure** | | | | | | |
| The synthesis of the ligand was carried out by slight modification of a literature method.S1 2,6-Dichloropyrazine (1.0 eq, 1.00 g, 6.71 mmol), 4-t-butylphenylboronic acid (2.0 eq, 2.39 g, 13.4 mmol), K2CO3 (2.0 eq, 1.86 g, 13.4 mmol) and Pd(PPh3)2Cl2 (0.0059 eq, 28.0 mg, 39.9 µmol) were added to a round-bottomed flask. MeCN (40 mL) and H2O (40 mL) were added and N2 was bubbled through the mixture for 15 min, followed by refluxing under N2 for 60 h. The volume of the mixture was reduced to ca 30 mL. A brown solid precipitated which was filtered off, washed with H2O (3 × 20 mL) and EtOH (2 × 5 mL) and air dried. Residue was dissolved in chloroform (100 mL) and silica (10 g) was added under stirring. Solution was filtered and filtrate evaporated under reduced pressure. Yield yellowish crystaline solid. | | | | | | |
| **Chemical analysis** | NMR | | HRMS | GC/HPLC | Melting point | Other |
| 1H | 13C |
|  |  |  |  |  |  |