Supplementary Material for the Paper

Asymmetric Fluoro-alkynyl Mercurials: The Synthesis and Solid State Structures of RHgC≡CCF₃ (R = Ph, Fc)

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**Synthesis of MeHgC≡CCF₃.** In a typical procedure; with the exclusion of light, under N₂, a stirred ethereal solution (100 cm³) of HFC-245fa (0.45 cm³, 4.43 mmol) was treated with n-BuLi (4.80 cm³, 12.00 mmol), at −10°C, then after 20 min. MeHgCl (0.804 g, 3.20 mmol) in ether (20 cm³) was added. After stirring overnight at 0°C the reaction was allowed to attain ambient temperature then hexane (200 cm³) added to precipitate the inorganic salts; the settled mixture was filtered through Celite® and the solvent removed in vacuo. Yield 0.216 g, 22 %. δ_F −50.0 (^4J_{HgF} 23.4 Hz), δ_C 142.0 [q, 6.5, C≡CCF₃], 111.9 [q, 25.6, C≡CCF₃], 89.1 [q, 50.2, C≡CCF₃], 5.5 [S, CH₃, J_{HgC} 1189.8 Hz]. δ_H 0.8 (s, J_{HgH} 150.6 Hz).

**n-BuHgC≡CCF₃.** HFC-245fa (0.40 cm³, 3.94 mmol), n-BuLi (4.10 cm³, 10.25 mmol), n-BuHgCl (0.500 g, 1.71 mmol). Yield 0.451 g, 75 %. δ_F −49.9 (^4J_{HgF} 20.8 Hz), δ_C 144.0 [q, 7.0, C≡CCF₃], 111.8 [q, 257.0, C≡CCF₃], 89.2 [q, 49.0, C≡CCF₃], 29.3 [s, CH₂, J_{HgC} 55.2 Hz], 28.4 [s, CH₂, J_{HgC} 1225.1 Hz], 27.3 [s, CH₂, J_{HgC} 135.9 Hz], 12.7 [s, CH₂, J_{HgC} 26.1 Hz]. δ_H 2.0 – 0.0 (m).

**t-BuHgC≡CCF₃.** HFC-245fa (0.35 cm³, 3.45 mmol), n-BuLi (4.10 cm³, 10.25 mmol), n-BuHgCl (0.705 g, 2.40 mmol). δ_F −49.8 (^4J_{HgF} 18.2 Hz), δ_C 142.3 [q, 6.5, C≡CCF₃], 111.7 [q, 257.2, C≡CCF₃], 89.4 [q, 49.4, C≡CCF₃], 49.6 [s, C], 30.8 [s, CH₂], 27.3 [s, CH₃]. δ_H 1.9 (s, J_{HgH} 166.5 Hz).