Supporting Information

Reaction of Arenes with Iodine in the Presence of Potassium Peroxodisulfate in Trifluoroacetic Acid. Direct and Simple Synthesis of Diaryliodonium Triflates

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(1) Characterization Data for Diaryliodonium Salts

Diphenyliodonium triflate: 0.316 g (71%); mp 177-178 °C (lit.¹ mp 178-180 °C); ¹H NMR (300 MHz, CDCl₃) δ 8.17 (d, *J* = 7.5 Hz, 4 H, ArH), 7.70 (t, *J* = 7.5 Hz, 2 H, ArH), 7.51 (t, *J* = 7.5 Hz, 4 H, ArH); ¹³C NMR (75 MHz, CDCl₃) δ 136.4, 133.7, 133.2, 121.8 (q, *J*_{CF} = 315.9 Hz, SO₂CF₃), 115.9.

Bis(4-chlorophenyl)iodonium triflate:² 0.294 g (57%); mp 175-176 °C; ¹H NMR (300 MHz, CDCl₃) δ 8.05 (d, *J* = 8.1 Hz, 4 H, ArH), 7.58 (d, *J* = 8.1 Hz, 4 H, ArH); ¹³C NMR (75 MHz, CDCl₃) δ 140.6, 138.1, 133.3, 121.8 (q, *J*_{CF} = 316.4 Hz, SO₂CF₃), 113.6.

Bis(4-bromophenyl)iodonium triflate: 0.326 g (54%); mp 190-191 °C (lit.³ mp 198 °C); ¹H NMR (300 MHz, CDCl₃) δ 8.08 (d, *J* = 8.7 Hz, 4 H, ArH), 7.70 (d, *J* = 8.7 Hz, 4 H, ArH); ¹³C NMR (75 MHz, CDCl₃) δ 138.1, 136.3, 128.8, 121.8 (q, *J*_{CF} = 316.50 Hz, SO₂CF₃), 114.4.

Bis(4-fluorophenyl)iodonium triflate:⁴ 0.292 g (60%); mp 167-168 °C; ¹H NMR (300 MHz, CDCl₃) δ 8.25-8.21 (m, 4 H, ArH), 7.31 (t, *J* = 8.4 Hz, 4 H, ArH); ¹³C NMR (75 MHz, CDCl₃) δ 166.4 (d, *J*_{CF} = 252.22 Hz, CF), 139.3 (d, *J*_{CF} = 8.6 Hz, CCCC), 121.8 (q, *J*_{CF} = 316.5 Hz, SO₂CF₃), 120.6 (d, *J*_{CF} = 22.87 Hz, CCF), 110.2.

Bis(4-*tert*-butylphenyl)iodonium triflate:⁵ 0.311 g (55%); mp 164-165 °C; ¹H NMR (300 MHz, CDCl₃) δ 8.09 (d, *J* = 8.4 Hz, 4 H, ArH), 7.56 (d, *J* = 8.4 Hz, 4 H, ArH), 1.29 (s, 18 H, Me); ¹³C NMR (75 MHz, CDCl₃) δ 157.7, 136.1, 130.4, 121.8 (q, *J*_{CF} = 316.5 Hz, SO₂CF₃), 36.0, 31.3.

(4-Iodophenyl)(phenyl)iodonium triflate: 0.404 g (69%); mp 140-141 °C (lit.⁶ mp 144-148 °C); ¹H NMR (300 MHz, CDCl₃) δ 8.19 (d, *J* = 8.4 Hz, 2 H, ArH), 7.92 (d, *J* = 8.4 Hz, 2 H, ArH), 7.84 (d, *J* = 8.4 Hz, 2 H, ArH), 7.70 (t, *J* = 7.8 Hz, 1 H, ArH), 7.53 (t, *J* = 7.8 Hz, 2 H, ArH); ¹³C NMR (75 MHz, CDCl₃) δ 142.2, 137.8, 136.5, 133.7, 133.2, 121.7 (q, *J*_{CF} = 316.5 Hz, SO₂CF₃), 116.0, 115.1, 100.6.

Diphenyliodonium trifluoroacetate:⁷ 0.326 g (81%); mp 197-198 °C; IR (KBr, cm⁻¹) 3047, 2988, 1679 (br), 1565, 1470, 1438, 1208, 1132, 990, 839, 805, 740, 679; ¹H NMR (300 MHz, CDCl₃) δ 8.17 (d, *J* = 7.8 Hz, 4 H, ArH), 7.67 (t, *J* = 7.8 Hz, 2 H, ArH), 7.53 (t, *J* = 7.8 Hz, 4 H, ArH); ¹³C NMR (75 MHz, CDCl₃) δ 161.0 (q, *J*_{CO} = 36.52 Hz, COCF₃), 136.4, 133.5, 133.1, 121.4 (q, *J*_{CF} = 288.07 Hz, COCF₃), 116.1.

Bis(4-methylphenyl)iodonium triflate: 0.054 g (11%); mp 182-184 °C (lit.³ mp 185-187 °C); ¹H NMR (300 MHz, CDCl₃) δ 7.83 (d, *J* = 8.4 Hz, 4 H, ArH), 7.25 (d, *J* = 8.4 Hz, 4 H, ArH); ¹³C NMR (75 MHz, CDCl₃) δ 143.8, 136.4, 134.9, 133.1, 109.7, 121.6 (q, *J*_{CF} = 367.8 Hz, SO₂CF₃), 21.4.

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