

Effective and selective iodofunctionalisation of organic molecules in water using the iodine-hydrogen peroxide tandem

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Electronic supplementary information†

Starting substrates were purchased from Aldrich, Fluka and Merck, solvents (ACS grade) were purchased from Fluka and Merck and used as received. A commercially available 30% aqueous solution of hydrogen peroxide was used as received. Melting points were determined on a Büchi 535 apparatus and are uncorrected. Chemical shifts both ¹H NMR and ¹³C NMR are reported in ppm from TMS as internal standard. ¹H NMR spectra were recorded on a Varian Inova 300 spectrometer at 300 MHz and ¹³C NMR on the same instrument at 75 MHz. IR spectra were recorded on a Perkin-Elmer 1310 spectrometer. The standard KBr pellet procedure were used to obtain IR spectra of solids, while a film of neat material was used to obtain IR spectra of liquid products. Mass spectra were obtained on an Autospec Q instrument under impact (EI) conditions at 70 eV. Elemental analysis was carried out on a Perkin-Elmer 2400 CHN analyzer.

(±)-1-Oxo-2-iodo-2-carbethoxycyclopentane (4c); unstable oily compound (contains 10% of **3c**), conversion 90%, decomposed during TLC; ¹H NMR (300 MHz, CDCl₃) δ 1.31 (3H, t, *J*=7.2 Hz), 2.10-2.18 (2H, m), 2.32-2.56 (4H, m), 4.28 (2H, dq, *J*=7.1 Hz, *J*=0.6Hz); ¹³C NMR (75 MHz, CDCl₃) δ 13.9 (CH₃), 19.9 (C-4), 35.0 (C-3), 40.2 (C-5), 43.4 (C-2), 63.2 (CH₂), 168.0 (COOEt), 207.0 (CO); IR (liquid film); 2979, 1736 (br), 1443, 1367, 1250, 1236, 1019 cm⁻¹; MS (EI, 70eV) *m/z* 282 (M⁺, 14%), 254 (7), 237 (17), 227 (12), 209 (10), 199 (44), 181 (29), 167 (8), 155 (95), 127 (35), 109 (95), 99 (46), 85 (46), 81 (36), 55 (100); HRMS Calcd for C₈H₁₁IO₃: 281.9753, found: 281.9762.

3',5'-Dihydroxy-4'-iodoacetophenone (6c); white solid, mp=189.5-192.5°C (decomp), 85% from pentane/acetone; ¹H NMR (300 MHz, acetone-*d*₆-CDCl₃) δ 2.51 (3H, s), 7.06 (2H, s); ¹³C NMR (75 MHz, acetone-*d*₆-CDCl₃) δ 25.4 (CH₃), 92.8 (C-4), 104.8 (C-2,6), 137.9 (C-1), 157.0 (C-3,5), 195.9 (CO); IR (KBr); 3422, 1658, 1587, 1414, 1240, 1038, 849 cm⁻¹; MS (EI, 70eV) *m/z* 278 (M⁺, 71%), 263 (100), 235 (29), 108 (27). Anal. Calcd for C₈H₇IO₃: C 34.56, H 2.54. Found: C 34.40, H 2.26.