

Pyrene-4,5-dione (3). To a refluxing mixture of Na (3.80 g, 158 mmol) in THF (100 mL) was added diester **2** (12.0 g, 40.8 mmol) in THF (200 mL). The reaction progress was followed by NMR aliquots, and additional Na (2.81 g, 116 mmol) was added in small portions until the reaction was complete (3 h). The dark red solution was then cooled to room temperature, residual Na was carefully removed, the reaction mixture was poured into ethyl acetate (500 mL) and water (200 mL), and the resulting emulsion was left to stand overnight. The aqueous layer was extracted with ethyl acetate (1 L), combined with the organic layer, dried (Na₂SO₄), and concentrated. Column chromatography (4:1 hexanes/ethyl acetate) provided a bright orange solid (9.0 g, 95%): mp 302–304 °C (lit.^{3a} mp 304.5–306.4 °C);

¹H NMR (200 MHz, CDCl₃) δ 8.52 (dd, *J* = 7.8, 1.3 Hz, 2 H), 8.20 (dd, *J* = 7.8, 1.3 Hz, 2 H), 7.87 (s 2 H), 7.77 (t, *J* = 7.8 Hz, 2 H); ¹³C NMR (50 MHz, DMSO-*d*₆) δ 180.1, 135.4, 132.1, 131.0, 129.1, 128.4, 128.2, 127.6; MS (EI) *m/z* (relative intensity) 232 (40), 221 (24), 218 (11), 204 (94), 189 (16), 176 (55), 150 (11); exact mass calcd for C₁₆H₁₈O₂ 232.0524, found 232.0513.

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