

Short Note

# *N*-(9,10-Dioxo-9,10-dihydroanthracen-1-yl)-2-methylbenzamide

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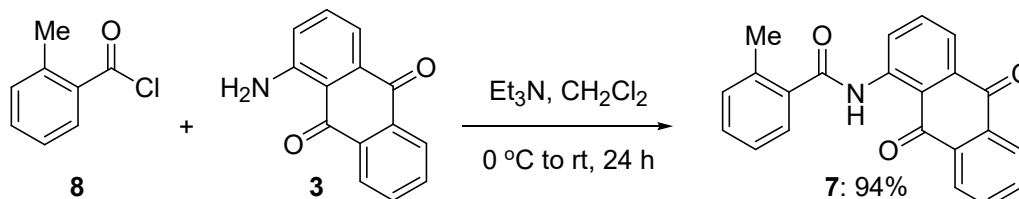
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## General Methods

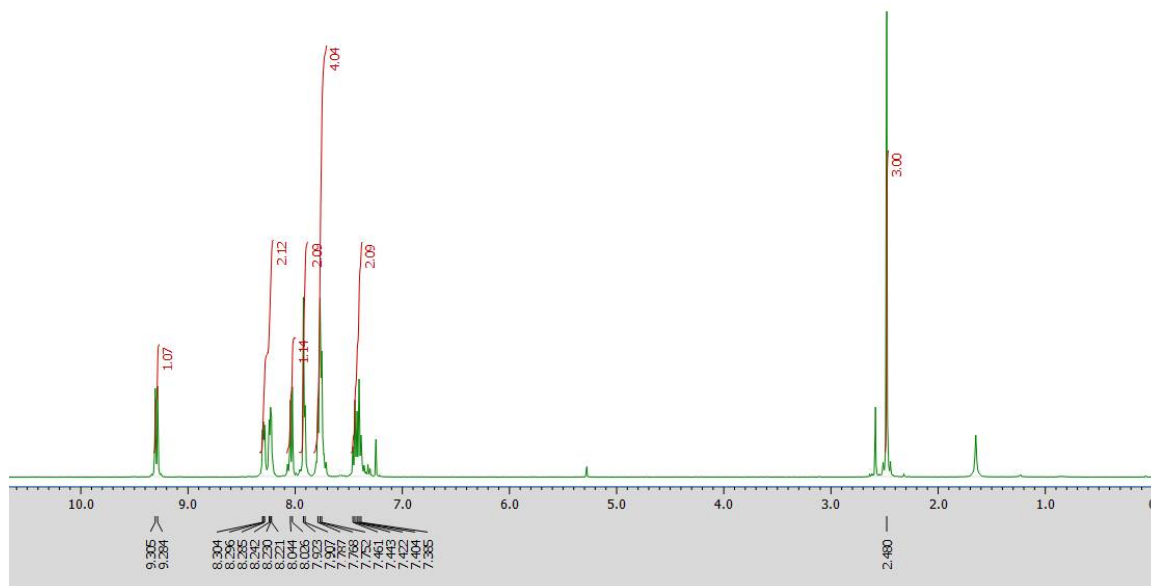
All chemicals, reagents and solvents were purchased from chemical companies (Sigma-Aldrich Chemie GmbH, Taufkirchen, Germany) and were used as received without prior purification. Reactions that required dry conditions were performed in an inert atmosphere with Ar gas. Syringes and needles for the transfer of reagents were oven dried and cooled in a desiccator over silica gel before use. The reaction's progress was monitored by thin-layer chromatography (TLC) on glass plates pre-coated with Merck silica gel. TLC plates were examined under UV lamplight (UVGL-58 Handheld 254/365 nm). Büchi-USA rotary evaporators were used to evaporate solvents using appropriate temperatures. Flash column chromatography was performed using silica gel (Kieselgel) (70–230) mesh as an adsorbent. The purified products were characterized using analyses NMR (<sup>1</sup>H-NMR, <sup>13</sup>C-NMR), IR, mass spectra and melting points. Melting points were recorded on the Gallenkamp-MPd350.bm2.5 melting point apparatus (Gallenkamp, Kent, UK). Attenuated total-reflectance IR spectra were recorded on pure samples on Agilent Technologies Cary 630 FTIR (Agilent, Santa Clara, CA, USA). <sup>1</sup>H-NMR spectra were recorded in CDCl<sub>3</sub> on JEOL ECX-400 spectrometers (JEOL Ltd, Tokyo, Japan). <sup>1</sup>H NMR chemical shifts (δ) were assigned in part per million (ppm) downfield using an internal standard trimethylsilane (TMS) and were referenced to CDCl<sub>3</sub>, δ = 7.24. Abbreviations s, d, t, q, quin, sept and m refer to singlet, doublet, triplet, quartet, quintet, septet and multiplet, respectively. Chemical shifts in <sup>13</sup>C spectra (175 MHz) were quoted in ppm and referenced to the central line of the CDCl<sub>3</sub> triplet, δ C 77.0. Coupling constants (J) were recorded in hertz (Hz). GC-MS spectra were obtained using an Agilent mass spectrometer (Agilent, Santa Clara, CA, USA).

## Synthesis of *N*-(9,10-Dioxo-9,10-dihydroanthracen-1-yl)-2-methylbenzamide (7)

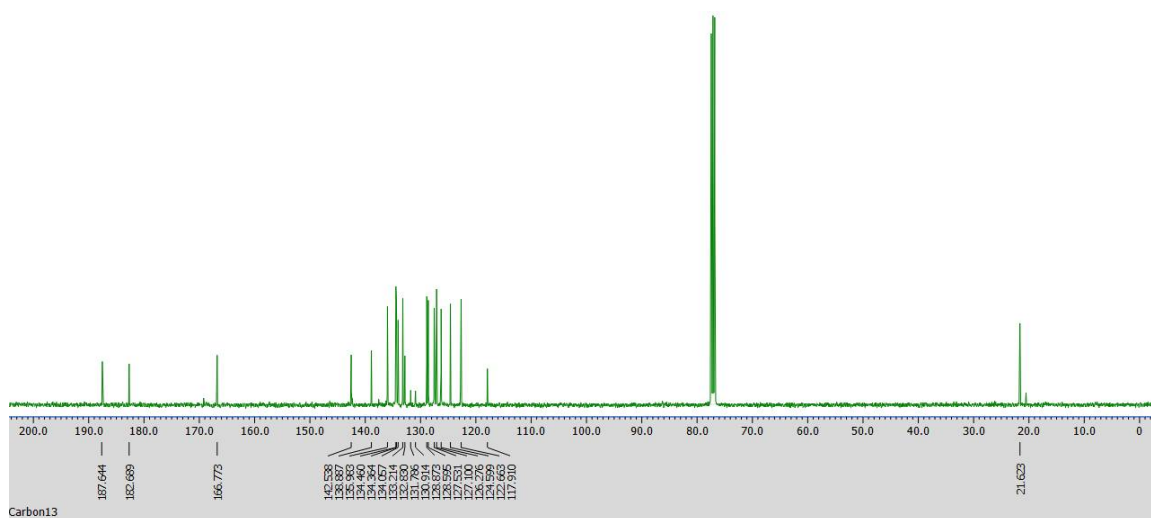


2-Methylbenzoyl chloride (8) (1.30 mL, 9.86 mmol) was added dropwise under an atmosphere of N<sub>2</sub> into a cold (0 °C ice-water bath) solution of 1-aminoanthraquinone (3) (1.10 g, 5.00 mmol), in CH<sub>2</sub>Cl<sub>2</sub> (20 mL). Et<sub>3</sub>N (1.40 mL, 10.0 mmol) was then added to the 0 °C mixture under N<sub>2</sub>. The mixture was stirred for 1 h at 0 °C, allowed to warm up to room temperature and then stirred for an additional 23 h. To the reaction mixture, water (30 mL) was added followed by aqueous saturated NaHCO<sub>3</sub> solution (30 mL) was added. The mixture was extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 × 30 mL). The combined organic extracts were washed with water (50 mL) and brine (50 mL) and then dried over anhydrous MgSO<sub>4</sub> and filtered. Recrystallization of the crude product from CH<sub>2</sub>Cl<sub>2</sub>:hexane, gave the title compound 7 as a yellow solid (1.60 g, 94%); R<sub>f</sub> = 0.37 (hexane/EtOAc, 1:2). Mp = 198–201 °C. <sup>1</sup>H-NMR

(400 MHz,  $\text{CDCl}_3$ )  $\delta$  9.29 (d,  $J = 8.7$  Hz, 1H), 8.30–8.22 (m, 2H), 8.04 (d,  $J = 7.3$  Hz, 1H), 7.92 (d,  $J = 6.4$  Hz, 2H), 7.77 (t,  $J = 7.1$  Hz, 4H), 7.46–7.39 (m, 2H), 2.48 (s, 3H);  $^{13}\text{C}$ -NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  187.6, 182.7, 166.8, 142.5, 138.9, 136.0, 134.5, 134.4, 134.1, 133.2, 132.8, 131.8, 130.9, 128.9, 128.6, 127.5, 127.1, 126.3, 124.6, 122.7, 117.9, 21.6; IR (film):  $\nu$  max/ $\text{cm}^{-1}$ : 3424, 3198, 1690, 1662, 1576. MS (EI)  $m/z$  (relative intensity): 341 (18), 281 (45), 262 (24), 207 (95), 191 (18), 147 (18), 119 (100), 91 (64). Elemental analysis, calculated: C (77.41), H (4.43), N (4.10), found: C (76.984), H (4.333), N (3.745).



**Figure S1.**  $^1\text{H}$ -NMR of *N*-(9,10-Dioxo-9,10-dihydroanthracen-1-yl)-2-methylbenzamide.



**Figure S2.**  $^{13}\text{C}$ -NMR of *N*-(9,10-Dioxo-9,10-dihydroanthracen-1-yl)-2-methylbenzamide.

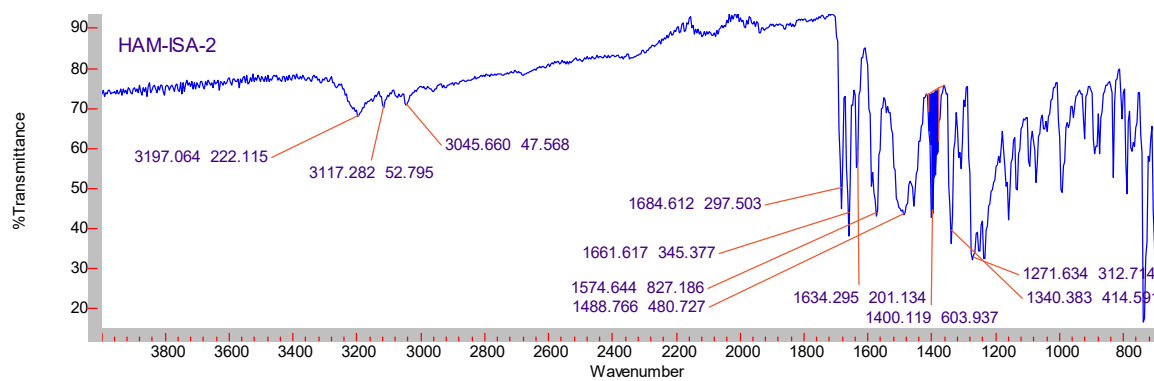


Figure S3. IR NMR of *N*-(9,10-Dioxo-9,10-dihydroanthracen-1-yl)-2-methylbenzamide.

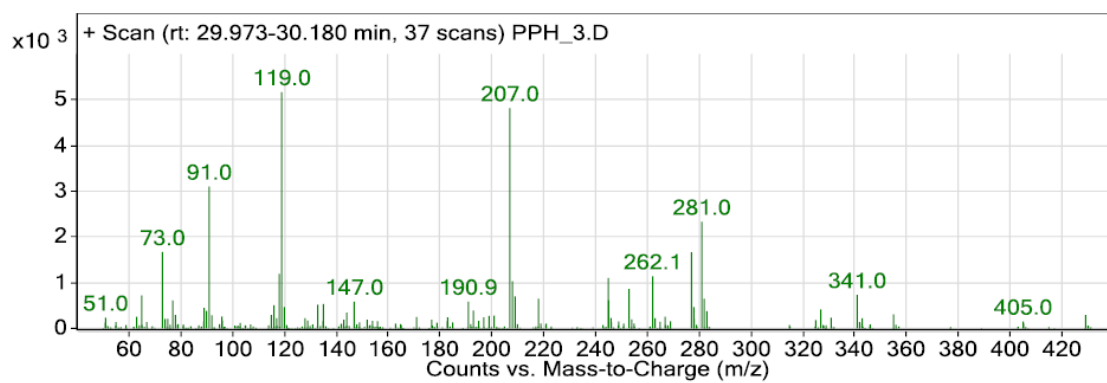


Figure S4. GC-MS of *N*-(9,10-Dioxo-9,10-dihydroanthracen-1-yl)-2-methylbenzamide.