

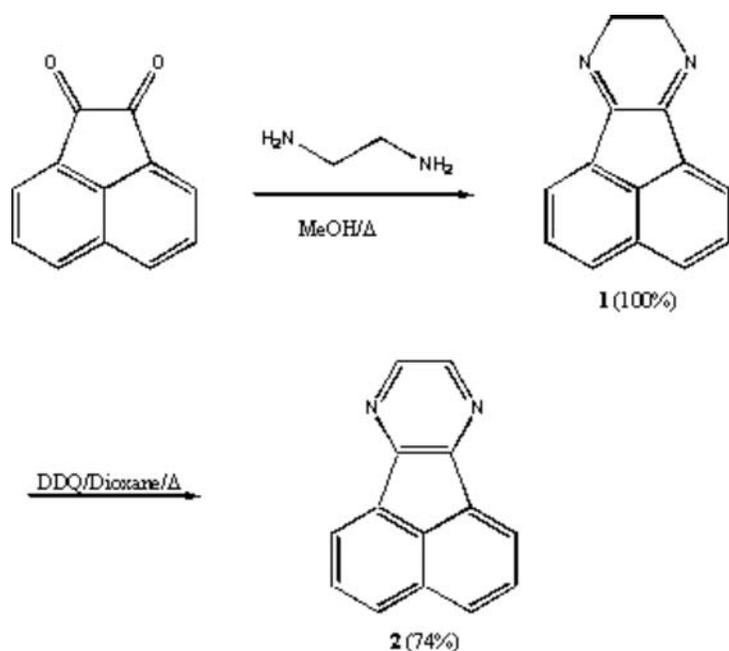
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<http://www.mdpi.net/molbank/>**7,10-Diazafluoranthene (Acenaphtho[1,2-*b*]pyrazine)****Jørgen Eskildsen, Jørn B. Christensen***

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7,10-Diazafluoranthene has been reported to be strongly mutagenic in an Ames-test [1], but no details regarding neither the synthesis nor other properties have been published.

7,10-Diaza-8,9-dihydrofluoranthene (1). (Compare to [2])

Acenaphthene quinone (4.1g; 23 mmol) was dissolved in refluxing MeOH (170 mL), a small portion of the quinone sometimes remained undissolved. Ethane-1,2-diamine (1.7 mL; 25 mmol) was slowly added. This mixture was refluxed for 1h, cooled to room temperature and evaporated to dryness. The dark brownish powder was dissolved in a small portion of MeOH and cooled in an ice bath. Water was slowly added until a fog of colloid precipitate became visible, which was removed by filtration. The filtrate was extracted twice with CH₂Cl₂ (1 ´ 70, 1 ´ 50 mL). The combined organic solutions were dried over MgSO₄, filtered and concentrated *in vacuo* to give almost pure **1** in quantitative yield. Yellow powder. Mp 94.4-96.0 °C.

M/z: M⁺ = 206 (70); 178 (90); 151 (20); 125 (5); 103 (3).

¹H NMR (400 MHz, CDCl₃, d, ppm): 7.96 (d, J = 8.4 Hz); 7.94 (d, J = 7.0 Hz); 7.69 (dd, J₁ = 7.2 Hz; J₂ = 7.0 Hz); 3.91 (s, 4 H).

^{13}C NMR (100 MHz, CDCl_3 , d, ppm): 158.6; 141.5; 131.7; 130.7; 128.5; 128.4; 118.7; 45.0.

7,10-Diazafluoranthene (2).

7,10-Diaza-8,9-dihydrofluoranthene (1) (5.35 g; 26 mmol) was dissolved in dioxane (270 mL). A solution of DDQ (6.0g; 26.5 mmol) in dioxane (150 mL) was added, and the mixture was refluxed for 30 min. After evaporation to dryness, the mixture was dissolved in CH_2Cl_2 (200 mL) and washed with a 0.3 M solution of Na_2SO_3 in water (40 mL). The solid material was removed by filtration, and dried *in vacuo*. The organic phase was evaporated to dryness, and combined with the material from the filtration. This mixture was subjected to Soxhlet-chromatography [3] on Aluminium oxide (Woelm, basic activity I) with CH_2Cl_2 as eluent to give 7,10-diazafluoranthene as yellow crystals in 74% yield (3.92 g) melting 145.0-147.1°C.

M/z: M^+ = 204 (100); 178 (74); 151 (24); 102 (9); 75 (16).

An analytical sample obtained by sublimation. (100 °C/ 0.01 mmHg) had:

Calcd. for $\text{C}_{14}\text{H}_8\text{N}_2$: 82.34% C; 3.95% H; 13.72% N

Found 82.36% C; 4.07% H; 13.47% N

Mp: 145.0-147.1 °C.

^1H NMR (400 MHz, CDCl_3 , d, ppm): 8.46 (s, 2 H); 8.28 (d, $J = 7.0$ Hz); 8.04 (d, $J = 8.2$ Hz); 7.78 (dd, $J_1 = 7.0$ Hz; $J_2 = 7.0$ Hz)

^{13}C NMR (100 MHz, CDCl_3 , d, ppm): 153.6; 141.1; 132.3; 131.4; 129.6; 129.4; 128.3; 122.7.

References and notes:

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Sample availability: Available from MDPI

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