

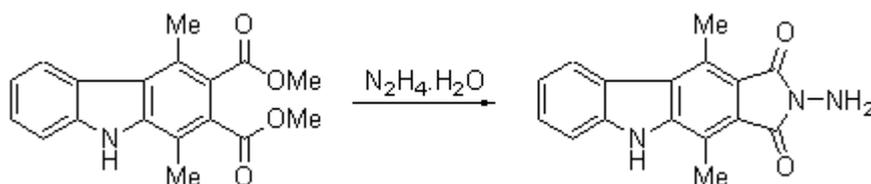
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2-Amino-4,10-dimethylpyrrolo[3,4-b]carbazole-1,3(2H,5H)-dione

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The general part of the experimental section [1] was presented elsewhere. In contrast to analogous cyclization reactions of 1-methyl-2,3-carbazoledicarboxylates which were reported to yield carbazole-fused pyridazinediones [1,2], condensation of dimethyl 1,4-dimethyl-2,3-carbazoledicarboxylate with hydrazine affords a five-membered cyclization product with an *N*-aminoimide structure. A mixture of dimethyl 1,4-dimethyl-2,3-carbazoledicarboxylate [3] (311 mg, 1 mmol) and 100% hydrazine hydrate (10 ml) was heated at reflux for 1.5 hr. After cooling, the solution was concentrated under reduced pressure to about half of its volume. The precipitate was collected by filtration and it was washed several times with warm ethanol to afford the title compound as fine, pale yellow needles (217 mg, 78%).

M.p. > 330 °C (ethanol)

¹H NMR (300 MHz, DMSO-*d*₆): 11.97 (s, 1H, NH), 8.24 (d, *J*₈₋₉ = 8.1 Hz, 1H, H-9, shows NOE on irradiation at 3.13 ppm), 7.64 (d, *J*₆₋₇ = 8.1 Hz, 1H, H-6), 7.54-7.47 (m, 1H, H-7), 7.32-7.25 (m, 1H, H-8), 4.78 (s, 2H, NNH₂), 3.13 (s, 3H, CH₃-10), 2.85 (s, 3H, CH₃-4).

IR (cm⁻¹, KBr): 3345 (N-H), 2949, 2920, 1757 (C=O), 1700 (C=O), 1623, 1608, 1521, 1408, 1365, 1330, 1233, 1125, 747, 720, 675, 626 cm⁻¹.

EI-MS: 280 (18%), 279 (100, M⁺), 263 (37), 234 (29), 218 (18), 206 (42), 205 (40), 193 (34), 192 (41), 191 (44), 190 (22), 165 (20), 140 (27), 96 (18), 83 (38), 63 (15)

Anal. calc. for C₁₆H₁₃N₃O₂ (279.3): C 68.81, H 4.69, N 15.04; found: C 68.60, H 4.78, N 15.06.

References

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Sample availability: available from the authors and from MDPI. MDPI ID 17923.

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