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SYNTHESIS OF 2-METHYL-BENZANTHRENO[2,3-*d*]IMIDAZOL-7-ONE

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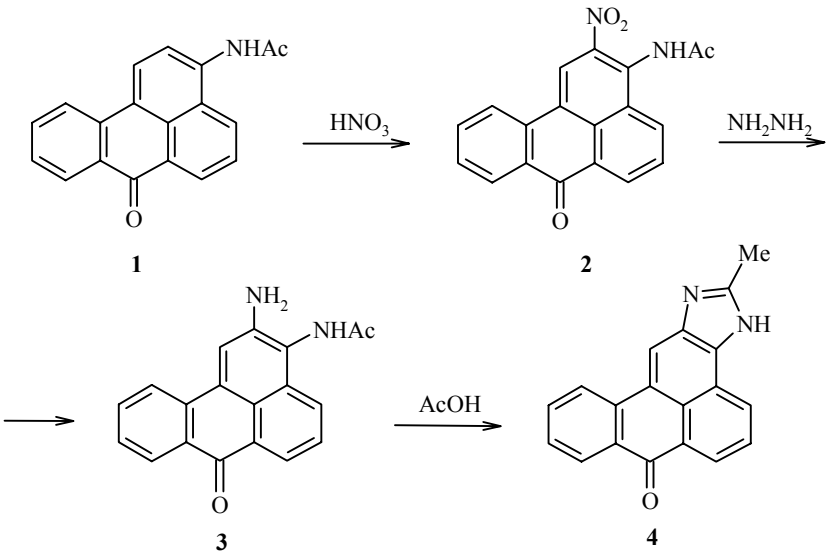
The synthesis of the benzanthrone based heterocyclic compound 2-methylbenzanthreno[2,3-d]imidazol-7-one has been achieved. It shows a strong luminescence intensity in the yellow-green region of the spectrum.

Keywords: 3-acetamido-2-nitrobenzanthrone, hydrazine hydrate, 2-methylbenzanthreno[2,3-*d*]imidazol-7-one.

This study is a continuation of our work on the chemistry of benzanthrone. Thanks to the wider distribution of methods for fluorescence analysis in biochemistry and medicine, benzanthrone derivatives have found use as fluorescence probes, e.g. 3-methoxybenzanthrone is used for studying conformational changes in proteins [1] and the compound ABM in the diagnosis of immune system disease [2].

We have previously reported the synthesis of 3-amino derivatives of benzanthrone and demonstrated their possible use as fluorescence probes [3, 4].



It was of interest to carry out an efficient synthesis of benzanthrone based condensed heterocyclic systems. A small number of similar systems have been described in the literature. For compounds containing a five membered heterocyclic fragment there are known pyrroles [5], indoles [6], and oxazoles [7] which are [5,6-*b*]-, [2,3-*b*]-, and [4,5-*d*]-annelated respectively to the benzanthrone.

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The chosen starting material was 3-acetamidobenzanthrone (**1**) which was nitrated as reported in [8] to give 3-acetamido-2-nitrobenzanthrone (**2**). The nitro derivative **2** was then reduced using hydrazine in the presence of Raney nickel to give compound **3**. Similarly to the method for the synthesis of benzimidazole [9], heating the 3-acetamido-2-aminobenzanthrone (**3**) in acetic acid at 90° for 3 h gave the 2-methylbenzanthreno[2,3-*d*]imidazol-7-one (**4**).

EXPERIMENTAL

^{1}H NMR spectra were recorded on a Bruker WH-90DS spectrometer (90 MHz) for DMSO-d_6 solutions with TMS as internal standard. IR spectra for the synthesized compounds were taken on a Specord M-80 instrument for KBr tablets. The course of the reaction was monitored using TLC on Silufol UV-254 plates with benzene–acetonitrile (6:1) as eluent.

3-Acetamido-2-nitrobenzanthrone (2). Mp 316-317°C (nitrobenzene). IR spectrum, ν, cm^{-1}: 1352, 1532 (NO_2); 1584 (C=C); 1640 (C=O); 3324, 3436 (N–H). ^{1}H NMR spectrum, δ, ppm: 3.44 (2H, br. s, NH_2); 7.38-8.87 (8H, m, CH_{arom}).

3-Acetamido-2-aminobenzanthrone (3). Hydrazine hydrate (0.5 ml) was added to a solution of compound **2** (0.5 g, 1.5 mmol) in DMF (40 ml). It was then heated to 60-70°C, Raney nickel was added, the product was heated at 50-60°C for 3 h, and left overnight. Raney nickel was added again, the product was heated for 2 h at 90-100°C, filtered hot, and water (100 ml) was added to the filtrate. The precipitate was filtered off and recrystallized from DMF. Yield 0.20 g (44%); mp 252-254°C. IR spectrum, ν, cm^{-1}: 1576 (C=C); 1636, 1656 (C=O); 3234, 3400 (N–H). ^{1}H NMR spectrum, δ, ppm: 2.16 (3H, s, CH_3); 5.49 (2H, br. s, NH_2); 7.58-8.82 (8H, m, CH_{arom}); 9.49 (1H, s, NH). Found, %: C 74.93; H 4.39; N 9.15. $C_{19}H_{14}N_2O_2$. Calculated, %: C 75.48; H 4.67; N 9.27.

2-Methylbenzanthreno[2,3-*d*]imidazol-7-one (4). Yield 84%; mp 292-294°C (benzene). IR spectrum, ν, cm^{-1}: 1576 (C=C); 1638 (C=O); 3228 (N–H). ^{1}H NMR spectrum, ν, ppm: 1;71 (3H, s, CH_3); 7.49-8.80 (9H, m, CH_{arom}, NH). Found, %: C 80.60; H 4.40; N 9.37. $C_{19}H_{12}N_2O$. Calculated, %: 80.27; H 4.25; N 9.65.

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