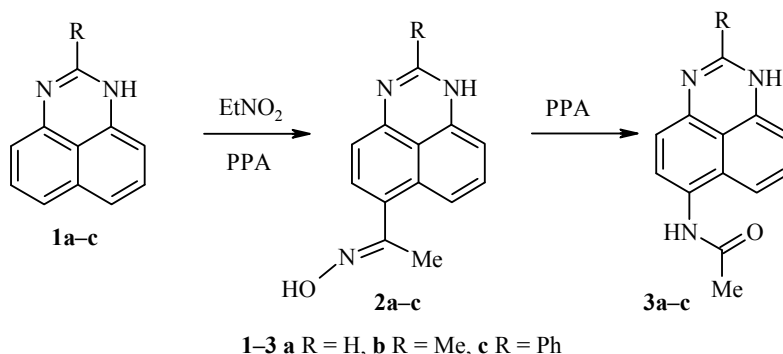


## NEW METHOD FOR THE ACETAMINATION OF PERIMIDINES

A. V. Aksenov<sup>1\*\*</sup>, N. A. Aksenov<sup>1</sup>, O. N. Nadein<sup>1</sup>, and A. E. Tsys<sup>1</sup>

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In previous work, we have developed a number of methods for the synthesis of acetaminoperimidines **3a-c** from perimidines **1a-c**. The first method involved consecutive acylation and the Schmidt reaction, while the second involved amination by sodium azide in PPA with subsequent acylation [1, 2]. In the present work, we report a one-step method for the synthesis of these compounds using a new reagent system containing nitroethane and PPA. Heating perimidines **1a-c** (1 mmol) and nitroethane (0.08 g, 1.07 mmol) in PPA\* (2-3 g) at 95-105°C for 3 h with monitoring by thin-layer chromatography gave 6(7)-acetaminoperimidines **3a-c** in 68-74% yield.



The reaction probably entails the intermediate formation of oximes **2a-c**, which undergo the Beckmann rearrangement to give amides **3a-c**.

The <sup>1</sup>H NMR spectra were taken on a Bruker WP-200 spectrometer at 200 MHz with TMS as the internal standard. The reaction course and purity of the products were monitored by thin-layer chromatography on Silufol UV-254 plates with 1:1 ethyl acetate-ethanol as the eluent.

The reaction mixture was treated with 50 ml water and brought to pH 8-9 by adding ammonium hydroxide. The precipitate formed was filtered off. The mother liquor was extracted with three 50-ml portions of hot benzene. The solvent was evaporated off and the residue was combined with the precipitate. The product was purified by recrystallization. The <sup>1</sup>H NMR spectra of **3a-c** were similar to those given in our previous work [1].

\* The PPA sample used contained 86% P<sub>2</sub>O<sub>5</sub> and was prepared according to Uhlig [3].

\*\* To whom correspondence should be addressed, e-mail: k-biochem-org@stavsu.ru.

<sup>1</sup>Stavropol State University, Stavropol 355009, Russia.

**6(7)-Acetaminoperimidine (3a)** was obtained in 68% yield; mp 225-226°C (ethyl acetate) (mp 225-226°C [1]).

**6(7)-Acetamino-2-methylperimidine (3b)** was obtained in 44% yield (0.091 g); mp 247-248°C (ethyl acetate) (mp 247-248°C [1]).

**6(7)-Acetamino-2-phenylperimidine (3c)** was obtained in 48% yield (0.106 g); mp 302-303°C (ethyl acetate) (mp 302-303°C [1]).

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