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LETTERS TO THE EDITOR

NOVEL METHOD FOR THE ACETAMINATION OF CROWN ETHERS

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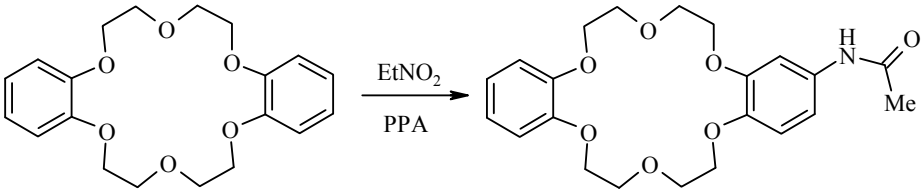
We have previously demonstrated the effectiveness of the novel system of reagents nitroethane–PPA for the preparation of acetaminoperimidines starting from perimidines [1]. Bearing in mind the current interest in developing methods for extending the functionalization of crown ethers [2, 3] we decided to use this reagent system for the acetamination of the dibenzo[18]crown-6 ether (**1**). It was found that reaction of ether **1** (0.36 g, 1 mmol) with nitroethane (0.083 g, 1.1 mmol) in PPA** (2-3 g) at 100-105ºC for 4 h (TLC monitoring) gave the 4'-acetaminodibenzo[18]crown-6 (**2**) in 54% yield.



1 **2**

Products of diacetamination were likely formed as side products but we were unable to obtain them in a pure state.

^{1}H and ^{13}C NMR spectra were recorded on a JNM-ECX 400 instrument (400 and 100 MHz respectively) with TMS as internal standard. Monitoring of the reaction course and the purity of the synthesized compounds was carried out by TLC on Silufol UV-254 plates in the system ethyl acetate–alcohol (1:1).

The reaction mixture was treated with water (50 ml) and basified with ammonia solution to pH 8-9. The precipitate formed was filtered off. The filtrate was extracted with methylene chloride (6×50 ml). Solvent was evaporated and the residue was combined with the precipitate. The material obtained was purified on L40/100 silica gel using ethyl acetate as eluent. Yield 0.226 g (54%); mp 185-186ºC (methylene chloride). According to the study [3] the mp is 185-186ºC. The ^{1}H NMR spectrum agrees with that given in [3].

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** The PPA used with an 87% P_2O_5 content was prepared by method [4].

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