

Publication is available for discussion in the framework of the on-line Internet conference “Butlerov readings”.

<http://butlerov.com/readings/>

Submitted on November 22, 2016.

Thematic course: The imides of maleic acid and derivatives. Part 5.

Synthesis of *N*-(2-butoxy-5-nitrophenyl)-1*H*-pyrrole-2,5-dione

© Oleg A. Kolyamshin,*⁺ Vladimir A. Danilov, Mikhail V. Kuzmin,
Valery A. Ignatiev, Lina G. Rogozhina, and Nikolay I. Koltsov

Department of Physical Chemistry and Macromolecular Compounds. Chuvash State University.

Moskovsky pr., 15. Cheboksary, 428015. Chuvash Republic. Russia.

Phone: +7 (8352) 45-24-68. E-mail: oleg.kolyamshin@yandex.ru

*Supervising author; ⁺Corresponding author

Keywords: 2-acetylamino-4-nitrophenol, alkylation, hydrolysis, cyclization, maleinimide, IR and ¹H NMR spectroscopy.

Abstract

Aminonitrofenols derivatives are widely used in the production of various dyes and their intermediates. Some of 2-acetylamino-4-nitrophenol are used as colorants for hair and wool. Derivatives having a sweet taste and are suitable as food additives have been synthesized. In turn the imide of maleic acid are important and are used as the main raw material in obtaining of thermoplastics and maleinimide resins. Some of maleinimide's derivatives show high pesticidal and pharmacological activity. The synthesis of new maleinimide – *N*-(2-butoxy-5-nitrophenyl)-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-1-yl has been described in this message.

Originally 2-acetylamino-4-nitro-1-butylsibenzol (**1**) has been obtained by boiling the solution of 2-acetylamino-4-nitrophenol and butyl bromide in DMF in the presence of potassium carbonate for 6 hours. Then 2-acetylamino-4-nitro-1-butoxybenzol has been boiled with aqueous solution of hydrochloric acid in isopropyl alcohol for 2 hours and as a result 2-amino-4-nitro-1-butoxybenzene (**2**) has been obtained. The corresponding amide of maleic acid **3** has been obtained by the interaction of synthesized amine with maleic anhydride in a solution of diethyl ether at room temperature. The corresponding amide of maleic acid has been cyclized with the help of acetic anhydride in the presence of sodium acetate in DMF solution at a temperature of 40-50 °C for 4 h as a result *N*-(2-butoxy-5-nitrophenyl)-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-1-yl (**4**) has been obtained.

The obtained compounds are crystalline substances, yellow (**2**, **3**) or light-gray (**1**, **4**). The structure of the synthesized compounds was characterized by IR and ¹H NMR spectra. The spectrum of maleic amide acid **3** contains of the amide group bands at 3280, 3245 and a carbonyl at 1680 cm⁻¹, respectively. In the IR spectrum of a maleinimide **4** there is a low intensity, but the characteristic band at 3100 cm⁻¹ corresponding oscillations to the double bond of maleinimide cycle, the vibrations at 1770, 1710 cm⁻¹ correspond to the carbonyl group of the maleinimide cycle. In the ¹H NMR spectrum of maleinimide **4** there is a characteristic singlet at 7.05 ppm corresponding to the fluctuations of protons of the double bond of the maleinimide cycle.

Thus in the course of the performed work *N*-(2-butoxy-5-nitrophenyl)-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-1-yl has been synthesized on the basis of 2-acetylamino-4-nitrophenol and its the physical and spectral properties have been studied.