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EFFICIENT SYNTHESIS AND DIRECTION OF METHYLATION OF 2-METHYLQUINAZOLINE-4-THIONE

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Abstract
The alkylation reaction of 2-Methylquinazolin-4-one with methyl iodide and dimethylsulfate in alcohol was studied. When aprotic dipolar DMFA is used as a solvent and used as a methylating agent, 1,3-dimethylquinazolin-4-one is also formed along with this compound.

Keywords: alkylation reaction, 2-Methylquinazolin-4-one, methyl iodide, dimethylsulfate, alcohol, dipolar DMFA, methylating agent, 1,3-dimethylquinazolin-4-one.

INTRODUCTION

Uzbekistan attaches great importance to agriculture, medicine and veterinary industry for its future development. A lot of attention is paid to livestock breeding and chemical treatment of land areas. In recent years, a number of measures have been developed on measures to increase the quality of continuous education and the effectiveness of science in the fields of chemistry and biology.

Our government has emphasized the need for extensive development of scientific research on the creation of herbicides, fungicides and bactericides, anthelmintics, means of combating plant weeds and pests in the cultivation of agricultural products, that is, the creation of effective new pesticides that quickly break down and do not accumulate in the environment, which are import substitutes and export oriented. attention is being paid to improving its chemical and biological properties[1-15].

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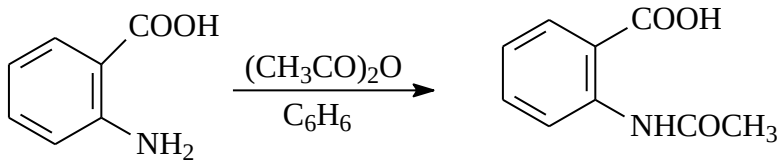
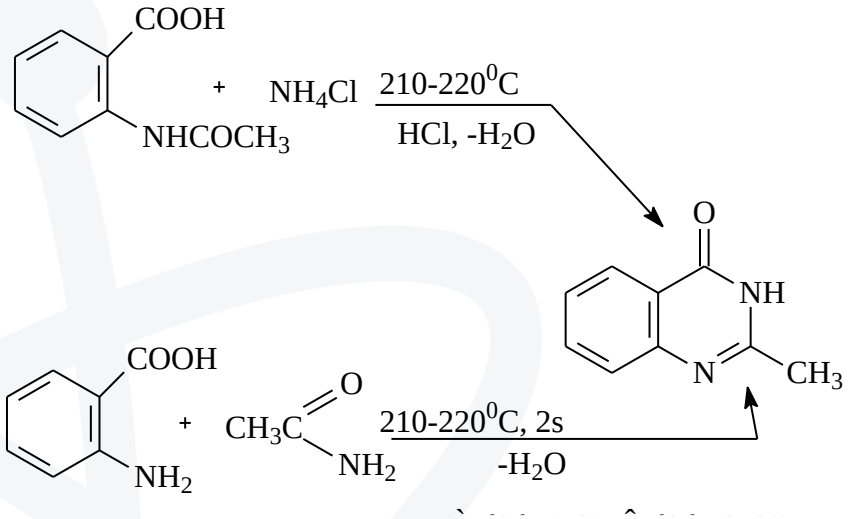
METHOD AND RESULTS

N-Acetylanthranilic acid is used as the main raw material in the synthesis of 2-methylquinazolin-4-ones. In the literature, this compound was synthesized by various methods and the product yield was 90-96%. An equivalent amount of anthranilic acid (AK) is first heated in benzene until boiling, so that all the anthranilic acid (AK) dissolves, then 1.75 equivalents of acetic anhydride are added and the mixture is allowed to cool, and white N-acetylanthranilic acid is formed:



N-acetylanthranilic acid is formed with a high yield (92%).

The synthesis of 2-methylquinazolin-4-one (30) required for research is carried out in two ways (A and B). According to method **A**: a mixture of initial N-acetylanthranilic acid and ammonium chloride reagents in a ratio of **N-acetylanthranilic acid :NH_4Cl** – 1:7 is heated at 210-220°C for 4 hours. The yield of 2-methylquinazolin-4-one (30) is 73%:



According to method B: anthranilic acid (AK) and acetamide were used as starting raw materials. In this case, a mixture of reagents - AK: acetamide - in a ratio of 1:2 is heated at 210-220°C for 2 hours. The yield of 2-methylquinazolin-4-one (2) obtained by this method is 92%. That is, the reaction in method B is carried out at a much lower temperature (compared to method A) and in a relatively short time, as well as obtaining the expected product in almost quantitative yields, means that method **B** is more efficient than method **A**.

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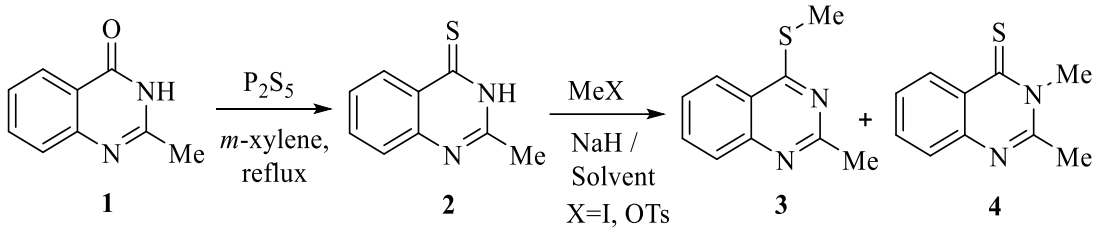
The reaction of 2-methylquinazolin-4(3H)-one (**1**) with P_2S_5 was carried out in boiling temperature of absolute m-xylene for 4 hours. The reaction mixture was filtered, the residue was washed with m-xylene, and the precipitate formed was treated with NaOH (10%), filtered, washed with water, and dried. As result we have obtained 2-methylquinazoline-4(3H)-thione (**2**) in good yield:



EXPERIMENTAL PART

Method A. 0.1 mol of N-acetylanthranilic acid, The mixture with 0.8 mol of NH_4Cl was heated and cooled at 210-220°C for 4-5 hours with the help of a reverse cooler. Then it was dissolved in parts with boiling water, the solutions were combined, neutralized with NH_4OH to pH-7.8, and cooled. The resulting precipitate was filtered and dried at room temperature. 15.48 g of 2-methylquinazolin-4-one was obtained with 73% yield. Ts=237-238°C R_f=0.134. System A

Method B. A mixture of 0.1 mol of anthranilic acid and 0.1 mol of acetamide was heated at 210-220°C for 2 hours using a reverse cooler, cooled, filtered, and dried. 15.48 g of 2-methylquinazolin-4-one was obtained with a yield of 92%. Ts=237-238°C R_f=0.134. System A

CONCLUSION

Methylation of 2-methylquinazoline-4(3H)-thione (**2**) with "soft" (methyl iodide) and "hard" (methyltosylate) alkylating agents in order to determine which direction the reaction takes place and the factors affecting were studied.

Methylation of thione (**2**) with alkylating agents in ethanol, acetonitrile, dioxane-1,4 at room temperature gives only 2-methyl-4-(methylthio)quinazoline (**3**). When the reaction temperature with methyltosylate increases, a mixture of 2-methyl-4-(methylthio)quinazoline (**3**) and 2,3-dimethylquinazoline-4(3H)-thione (**4**) are formed. Methylation of thione (**2**) with methyltosylate in DMFA also gives ccompounds **2,3**. If the temperature of this reaction increases, the yield of 2,3-dimethylquinazoline-4(3H)-thione (**4**) increases, which confirms the reaction proceeding through the " hard " center and the formation of a more stable product.

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