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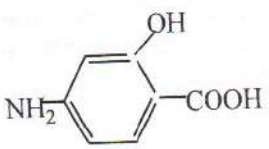
METHOD OF PRODUCING p-AMINOSALICYLIC ACID

Gaini Seitenova, Ainagul Kolpek, Gulnara Abdullina

The invention relates to the field of synthetic organic chemistry and can be used in the pharmaceutical industry for producing anti-tuberculosis drugs "p-Aminosalicylic acid" (PAS) (I), «The sodium salt of p-aminosalicylic acid" (PAS-Na) (II) and "Calcium salt of p-benzoylaminosalicylic acid "(BEPASK) (III).



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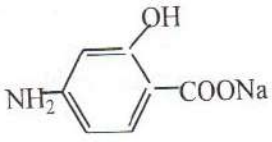

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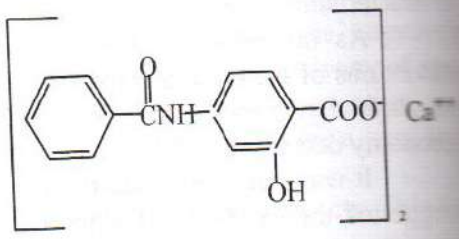


III

Despite the other, more effective antituberculosis drugs, p-aminosalicylic acid and its derivatives retain their meaning as one of the components of combination chemotherapy (simultaneous treatment of multiple drugs) [1]. According to the order of the Minister of Health of the Republic of Kazakhstan on December 22, 2004 № 83 "On approval of the List of basic (vital) drugs" p-aminosalicylic acid is a part of a list of essential (vital) drugs such as antituberculosis drug.

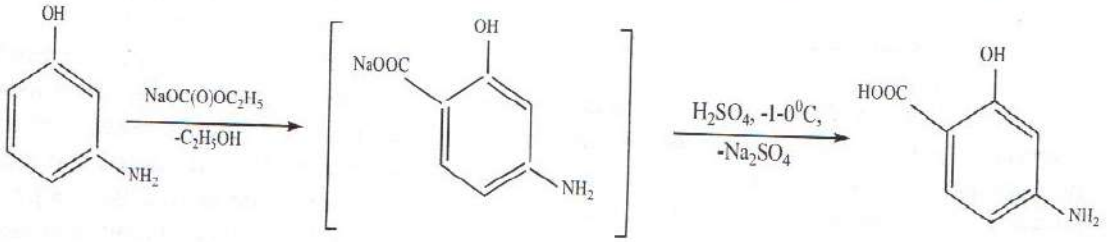
Carboxylation of m-aminophenol by the Kolbe-Schmidt in an aqueous medium under pressure of carbon dioxide is the main industrial way to obtain p-aminosalicylic acid [2]. The synthesis consists of two stages: 1. carboxylation of m-aminophenol pressurized carbon dioxide at a temperature of 103-105°C with the presence of potassium bicarbonate; 2. extraction of p-aminosalicylic acid by the action of 50% aqueous sulfuric acid upon the purified solution of potassium m-aminosalicylate. One of the biggest disadvantages of this method for producing p-aminosalicylic acid is a need for a process of carboxylation of m-aminophenol in the presence of more than a molar excess of potassium bicarbonate to shift the reaction equilibrium towards the formation of potassium p-aminosalicylate. In connection with the foregoing, other more environmentally friendly methods for producing p-aminosalicylic acid are of interest. One of these methods is the use of alkali salts of alkyl carbonic acids as carboxylation agent. Alkaline salts of alkylcarbonic acids are easily synthesized by reacting carbon dioxide with an alkali metal alkoxide or by reaction of carbon dioxide with alkali metal halides and alcohols in the presence of organic bases [3-4]. A simple, convenient and economical way to get sodium ethylate and potassium is to interact ethanol with sodium hydroxide and potassium [5-6].

Prepatents RK #15017 is only one Patent used in the literature about application of the sodium and potassium salts of ethyl carbonate as the agent of the carboxylation of m-aminophenol for the synthesis of p-amino salicylic acid [7]. However, we found that described in this prepatents conditions obtain not p-amino salicylic acid but its sodium salt-PAS-Na as the result. Obviously, the authors were misled by incorrect reference data on the physical constants of p-amino salicylic acid: melting temperature of p-amino salicylic acid 220°C and 219-220°C [8]. According to some refined data the melting point of p-amino salicylic acid is 146-147°C and 147°C [9-11].

Kolbe-Schmidt's method of producing p-aminosalicylic acid by carboxylation of m-aminophenol with carbon dioxide is the closest to the suggested among other described methods. Synthesis of p-aminosalicylic acid is carried out under pressure of carbon dioxide in the aqueous medium in the presence of a fivefold excess of potassium bicarbonate at 96°C, process duration is 90 hours.

The most significant disadvantages of this method are the complexity of the reaction (using a large excess of potassium bicarbonate, a large amount of wash water), small yields of the desired product (50-60%) and longer duration of the process (90 hours).

The object of the invention is a method for preparing p-aminosalicylic acid and eliminating the above drawbacks. This is achieved by the fact that in contrast to the known process carboxylation of m-aminophenol is carried out by sodium salt of ethyl carbonatcand heating the reactants at 150-170°C in carbon dioxide medium at a pressure of 6-10 bar for 4-6 hours.



Experiment.2.72g (0.025 mol) of m-aminophenol and 3.08 g (0.025 mol) sodium ethyl carbonate are charged into a glass reactor placed in a steel autoclave, the autoclave is sealed, purged twice with carbon dioxide to remove the air and then filled with carbon dioxide to 8 bar, stirring and heating are turned on. The temperature of the reaction mixture is raisedto 160°C during 4 hours, (temperature rise rate ≈35°C/h) and maintained at this temperature for 1 hour. Thereafter, stirring and heating are stopped, the autoclave is cooled to room temperature. The reaction mixture is treated with water. The resulting aqueous solution is extracted with ether to separate the unreacted m-aminophenol. After removing the solvent 1.0 g of the unreacted m-aminophenol is gained from the organic phase. The aqueous phase is cooled to -1 - 0°C. Reaction product (p-aminosalicylic acid) is separated by acidification at -1 - 0°C of aqueous sulfuric acid phase. 2.4 g (62.8%) of p-aminosalicylic acid is obtained, melting temperature = 144-145°C. After recrystallization (ethanol) the melting temperature is 146°C. Yield of p-aminosalicylicacidreacted with m-aminophenol is 99.5%. The melting temperature is 146-147 °C. From subsequent experiments it is shown that the most optimal reaction conditions are: temperature 160°C, pressure 8 bar, the duration of 5 hours.

Under these conditions the yield of the target product reaches 62,8% (or 99.5% for reacted with m-aminophenol).In comparing to the known method, using the proposed method for producing p-aminosalicylic acid provides the following advantages:

1. Eliminating the use of water as solvent;
2. Eliminating the use of a large excess of potassium bicarbonate;
3. Process duration is reduced from 90 hours to 5 hours.

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NEW STANDART – NEW QUALITY

Andiya Serikbayeva

1. Introduction

Over two years ago Kazakhstan launched development, improvement and methods of introduction of the Malaysian halal standard MS 1500:2004, which fully complies with the Islamic canons and requirements. Currently, halal standard products, in particular, meat products are already widely popular. And this is not surprising: because halal means food products produced in accordance with the Muslim traditions, which are consumed by people of any nationalities and confessions. The Hadji Association of Kazakhstan even established the special technical committee for issues of an international standard MS 1500 – Halal Food.

Moreover, "halal" labeling is perceived to be additional quality guarantee and product safety, the mark meaning that is contains no harmful chemicals.

Animals killing is performed in accordance with Islamic norms. The livestock the day before killing shall be healthy. It is killed through intersection of cervical arteries, in a single step, without delay and interruption, with the name of Allah on the lips simultaneously with intersection of main cervical arteries and gullet, better closer to the head. Carotid artery of every animal is intersected so as blood may completely discharge, that cannot be achieved in existing ways of killing: nervous breakdown with electrical shock, brain affection using mechanical attack, anesthetizing using carbon dioxide and any other chemicals, coagulated blood of the animal stays in the meat.

In this case cortisol, stress hormones start discharging into the animal blood, which further on start entering the human body harmfully affecting the human health. This also immobilizes it during killing, that in turn results in significant or complete disturbance of blood extraction. It is of crucial importance that during killing of one animal, the others do not see this so as not to be scared and avoid stress and stress hormones release.

In accordance with religious norms it is extremely important that blood flows from killed animal under influence of "natural convulsion".

Basic principles of the Islamic "Halal" mean humane treatment of animals and birds before killing, an animal should be alive and healthy, careful avascularization of animals with its heart functioning.

In II century of the last millenium Ibn Sina (Avicenna) was the first to demonstrate fear impact by the example of a lamb placed in cage located in close proximity to wolf's cage so that animals were within visual range of each other. The lamb relatively quickly died from developed

CONTENTS

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