

Batch and flow injection spectrophotometric methods for the determination of barbituric acid in aqueous samples via oxidative coupling with 4-aminoantipyrine

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Abstract

A batch and flow injection (FI) spectrophotometric methods are described for the determination of barbituric acid in aqueous and urine samples. The method is based on the oxidative coupling reaction of barbituric acid with 4-aminoantipyrine and potassium iodate to form purple water soluble stable product at λ 510 nm. Good linearity for both methods was obtained ranging from 2 to 60 $\mu\text{g mL}^{-1}$, 5–100 $\mu\text{g mL}^{-1}$ for batch and FI techniques, respectively. The limit of detection (signal/noise = 3) of 0.45 $\mu\text{g mL}^{-1}$ for batch method and 0.48 $\mu\text{g mL}^{-1}$ for FI analysis was obtained. The proposed methods were applied successfully for the determination of barbituric acid in tap water, river water, and urine samples with good recoveries of 99.92% and 100.67% for batch and FI method, respectively.

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1. Introduction

Barbituric acid {2,4,6(1H,3H,5H)-pyrimidine-trione} (BA) or 6-hydroxyuracil is an organic compound based on a pyrimidine heterocyclic skeleton. Preparation of barbiturates, dyes and polymerization catalysts, and pharmaceutical preparation has been depend mainly on association of BA. Moreover, it was identified as an intermediate in many processes and

there is no effect on the central nervous system by BA itself. However, it is used as a precursor to medical barbiturates which can be lethal in excessive amounts [1].

Thus, for the reasons above and for biological and industrial processes, the development of a simple, rapid, sensitive, and selective method for determination of trace amounts of barbituric acid is an important issue.

Different approaches have been developed for the determination of BA in various matrices such as pharmaceutical formulation, blood, urine and aqueous solutions included spectrophotometry [2–5], high

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performance liquid chromatography [6–8], capillary electrophoresis [9], electrochemical [10–12], chemiluminescence [13], a narrow-bore porous graphitized carbon column using water–dioxane mixtures as mobile phases for simultaneous effect of organic modifier and physicochemical parameters of barbiturates [14], reversed-phase thin-layer chromatography using methanol:water mixtures as a mobile phase for the determination of lipophilicity of thiobarbituric acid derivatives assessed [15], little attention have been paid to develop a new method for the spectrophotometric determination of barbituric acid.

The purpose of this work is to determine barbituric acid using batch and flow injection (FI) spectrophotometric methods since FI become a popular method utilized in the analysis and application in various samples. Thus, the proposed study describes new, fast, low cost, simple, sensitive batch and FI spectrophotometric methods for the determination of barbituric acid via oxidative coupling reaction with 4-aminoantipyrine in the presence of potassium iodate forming purple colored product that has a maximum absorption at λ_{\max} at 510 nm.

2. Experimental

2.1. Apparatus and chemical materials

A Shimadzu 1650 PC UV-VIS double beam spectrophotometer was used for λ_{\max} determination. A two-channel manifold (Fig. 1) was employed for the flow injection spectrophotometric determination of barbituric acid. A peristaltic pump (Gilsasonminipuls (2))

was used to transport the carrier solution equipped with flexible polyvinyl chloride tubes of 0.8 mm internal diameter. Injection valve (Rheodyne-USA) was employed to provide appropriate injection volumes of standard solutions and samples. Potassium iodate was supplied by BDH company and 4-aminoantipyrine obtained from Fluka while barbituric acid was purchased from Sigma Aldrich.

2.2. Preparation of solutions

4-Aminoantipyrine (0.5 M) was prepared by dissolving 10.162 g in 100 mL of distilled water in a volumetric flask of 100 mL.

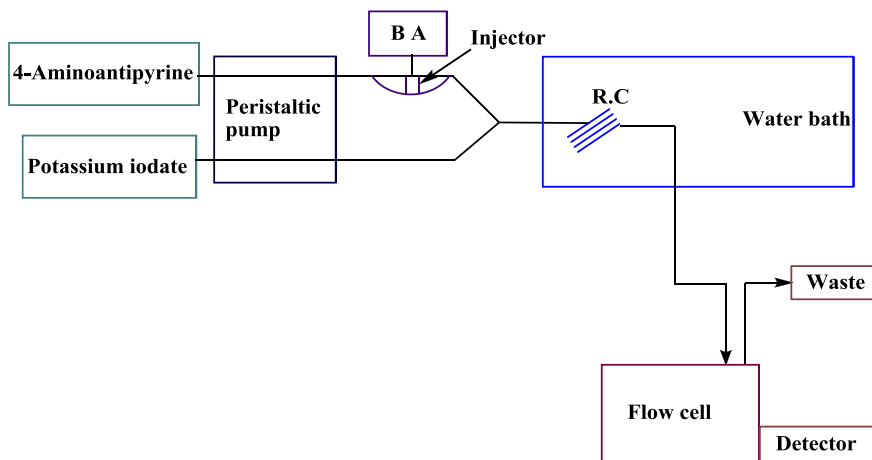
Barbituric acid standard solutions ($500 \mu\text{g mL}^{-1}$) were prepared by dissolving 0.05 g of barbituric acid in distilled water the solution was made up to 100 mL with distilled water.

Potassium iodate (0.5 M) was prepared by dissolving 10.7 g of potassium iodate in distilled water the solution was made up to 100 mL with distilled water.

Water and urine samples: all water samples were filtrated before analysis whereas urine samples were diluted 1:4 with distilled water and then filtering using a No. 4 Whatman filter paper.

2.2.1. Samples

All urine samples were collected from healthy individuals volunteers living in Baghdad, Iraq (3 males and 2 females; age 18 to 24). All volunteers were asked to complete a rapid questionnaire regarding their sex and age and gave written consent before being enrolled in the study.



BA= barbituric acid, RC= reaction coil

Fig. 1. Manifold employed for FI spectrophotometric determination of BA with 4-aminoantipyrine.

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