KINETIC SPECTROPHOTOMETRIC DETERMINATION OF ACETYLSALICYLIC ACID IN DOSAGE FORM "ACELYSIN-KMP"

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Research purpose. To develop a new kinetic spectrophotometric method of quantitative determination of acetylsalicylic acid (ASA) in the selected drug using the indicator reaction of catalytic *p*-phenetidine (*p*-Ph) oxidation by hydrogen peroxide.

Materials and methods. "ACELYSIN-KMP", Kyivmedpreparat – lysini acetylsalicylas is a mixture of DL-lysine acetylsalicylate and glycine with a ratio of 9:1. It has pharmacological properties of ASA, but unlike the latter is easily soluble in water. Our method is based on the system of two coupled reaction: ASA perhydrolysis (reaction with excess of H_2O_2 in a weak alkaline medium with peracetic acid (PA) formation) and following *p*-Ph oxidation by newly generate PA to azodue (λ_{max}=358nm). Its increasing absorbance allows to determine ASA. Kinetic spectrophotometric initial rate method was used for computing.

Results. Optimal conditions for ASA perhydrolysis and thus for indicator reaction was determined, including order of mixing, reagent concentration and pH. In the pH range 8.2–8.5 rate of 4.4'-azoxyphenetol formation directly proportional to the concentration of ASA. It is shown experimentally that the perhydrolysis reaction is the limitative stage of *p*-Ph oxidation in *p*-Ph–H_2O_2–ASA system. Stated kinetic feature of the passing reactions and sufficiently high selectivity of indicator reaction of *p*-Ph oxidation by newly generate PA in the presence of relatively large excess of H_2O_2 is the basis of new developed procedure of quantitative assay of ASA in dosage form "ACELYSIN-KMP". Kinetic curve of 4.4'-azoxyphenetol accumulation in *p*-Ph–H_2O_2–ASA system under optimized reaction conditions was obtained. Sites from 5 to 10min has linear dependence and specify initial reaction rate (A vs. τ dependence tangent angle). The Beer's law was verified from the calibration curve by plotting a graph of concentration vs. increasing of absorbance from the series of ASA concentrations ranging from 22-180μmol L^{-1}. Calibration graph for ASA was obtained: tgα=325.5c–0.0004, (r=0.9987), where *c* is the concentration of analyte, mol L^{-1}, and tgα is the initial conditional reaction rate, min^{-1}. Standart deviation for the slope (S_b=21.386) and intercept (S_a=0.0023) was calculated. The limit of quantitation is 12μmol L^{-1}. The relative standard deviation is less than 1.2%.

Conclusion. Thus, a highly selective and sensitive kinetic spectrophotometric method has been developed for the determination of ASA in dosage form "ACELYSIN-KMP". The proposed procedure proved to be selective, simple and rapid (single analysis time does not exceed 10 min) for the quantitative determination of ASA in the selected dosage form in the presence of it hydrolytic cleavage products and compresent components. For five determinations of 44μmol L^{-1}, 88μmol L^{-1} and 130μmol L^{-1} ASA the reproducibility has a RSD of 1.18, 1.06 and 0.76% respectively. Hence the proposed method is more sensitive, simple and express in comparance with the well-known one. Dosage form "ACELYSIN-KMP" recovery is 98.95±1.32% of ASA.