COMBINATION OF HIGH-ALKALINE SAMPLE PREPARATION OF SAMPLES WITH ORGANIC MATRIX,

FOLLOWED BY CATALYTIC KINETIC DETERMINATION OF TOTAL IODINE IN THE FORM OF IODIDE BY FERUM(III)-NITRITO-THIOCYANATE REACTION

Alkaline high-temperature sample preparation of samples with the organic matrix and the subsequent determination of total iodine in the form of iodide was studied by iron(III)-nitrite-thiocvanate reaction. By using titrimetric method was found residual content of alkali in solution samples after mineralization using KOH and ZnSO4, as ashing reagent. In constructing the calibration curve and in determining iodide maintained in solution the same acidity. To do this: a) in the case of building a calibration curve was added in each solution a quantity of KOH contained in the aliquot of the sample solution, and b) when determining iodide was added to each solution a volume of water equivalent to the maximum amount of the standard iodide solution when of building calibration curve. The influence of various factors (pH, reagent concentration, the order of addition of reagents, temperature, presence of other components) was investigated on the course of iron(III)-nitrite-thiocyanate reaction. The concentrations of reactants chosen such, that the value of the optical density of the solution was about 0.8 in the absence of catalyst (iodide), and at its maximum content is not less than 0.1. Based on these conditions optimal for the studied reactions were the following concentrations of reagents in solution: $C_{HNO3}=0.5$ M; $C_{KSCN}=2.4 \cdot 10^{-3}$ M; $C_{Fe(III)}=0.035$ M; C_{NaNO2} =0.05 M, 30°C while observing the reaction 20 min. Optimal order of mixing of reagents are as follows: to a solution of iodide, thiocyanate solution is added, then add the acid solution of iron (III) (simultaneous creation of optimal acidity) and recently added a solution of nitrite that starts the catalytic reaction. Conditions of alkaline hightemperature sample preparation of samples with organic matrix combined with conditions the following kinetic catalytic determination of total iodine in the form of iodide by iron(III)-nitrite-thiocyanate reaction. The optimized method used for the determination of total iodine in samples of fresh and powdered milk. Metrological characteristics of the techniques tested method additions and analysis of standard sample of milk powder. The relative standard deviation did not exceed 0.15 in the case of the analysis of these objects.

Key words: sample preparation, total iodine, kinetic methods of analysis.