

SAMPLE PREPARATION TECHNIQUES IN TRACE ELEMENT ANALYSIS OF WATER

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Sample preparation techniques for the analysis of water for trace elements using X-ray emission spectroscopy are described.

Fresh water samples for the analysis of transition metals were prepared by complexation with ammonium-pyrrolidone-dithiocarbamate (APDC) and filtering through a membrane filter. Analyses of water samples for halogens was done on samples prepared by precipitation with AgNO_3 and subsequent filtration. Two techniques for seawater preparation for cesium determination are described, viz. precipitation with APDC in the presence of iron (III) as a carrier and complexation with APDC followed with adsorption on activated carbon.

In all cases trace element levels at 10^{-3} $\mu\text{g/g}$ were measured.

1. Introduction

X-ray emission spectroscopy offers a simple and fast method for trace element analysis of water. In this report results of trace element analysis of different water samples obtained by tube excited X-ray fluorescence (XRF) are compared to results obtained by proton induced X-ray emission (PIXE). Three preconcentration procedures were used: precipitation, coprecipitation and complexation with subsequent adsorption on activated carbon. The choice of the preconcentration method depends on the elements of interest, the available equipment, required accuracy, etc.

The sample preparation techniques used are characterized by simplicity and the short time required for target preparation, which reduces the possibility of contamination. In addition, surfaces of deposits are homogeneous and can be directly examined.

Nonspecific multielement reagent ammonium-pyrrolidone-dithiocarbamate (APDC) was used as the precipitating reagent because of its high stability and the low aqueous solubility of its complexes with transition metals. In addition, the advantage of this chelating agent is that elements such as Ca, K, Na and other cations present in percent quantities in natural water do not precipitate, which eliminates their interference and unnecessary increase in the detector counting rate.

In this report we present details of target preparation techniques and some of the experimental results obtained.

2. Experimental

In order to obtain accurate quantitative analysis, attention should be paid to the problems of sampling,

sample storage and transport treatment. Water samples were collected with a special sampler made of teflon to avoid contamination. Samples were transported to the laboratory in polyethylene containers, and immediately prepared to prevent losses by adsorption to the vessel walls.

2.1. Precipitation procedure

A small amount of hydrochloric acid was added to 100 ml of water and the pH was adjusted to 3. Two ml of 1% APDC solution (freshly prepared daily) was added, mixed and allowed to stand at room temperature to permit complexation. The suspension was filtered through a membrane filter. The precipitates on the filter were air dried before being analyzed. The time required for sample preparation did not exceed 30 min.

The equipment for sample preparation consisted of a Pyrex filter holder and included a reservoir, a 17 mm circular fritted glass support and a metal clamp.

2.2. Sample preparation procedure for halogenous elements

The standard calibration solution (100 ml) contained 0.5-4 mg of iodine and bromine. The pH value of the solution was adjusted with a few drops of nitric acid and 20 ml and 1000 mg/l silver standard was added. The solution was mixed and allowed to stand over night in a dark place. Next day the suspension was filtered through a membrane filter. The precipitates on the filter were then suitable for analysis. The whole procedure was done in the presence of red light to prevent decay of halogenous compounds.

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