



Development of a new aerosol phase extraction method for metal determination through inductively coupled plasma atomic emission spectrometry

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ABSTRACT

In this work, a new extraction method termed aerosol phase extraction has been developed for the first time. The new method was based on the nebulisation of the sample onto the extracting solution to maximize the contact surface. The influential parameters are: agitation time, chelating agent concentration and inorganic acid concentration. The method has been applied to the extraction of molybdenum with organophosphorus chelating agents such as tributyl phosphate (TBP) and bis(2-Ethylhexyl) phosphoric acid (D2EHPA) dissolved in *n*-hexane from aqueous hydrochloric and phosphoric acid solutions. In order to test the efficiency of the method, the aqueous phase has been analyzed by means of Inductively Coupled Plasma Atomic Emission Spectrometry. The extraction of molybdenum under aerosol phase was found to be faster than the conventional extraction method. Equilibrium time was shortened under aerosol phase extraction and molybdenum extraction yields were comparable, or better as compared to the conventional method.

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1. Introduction

The solvent extraction is one of the most versatile and widely used procedures for the removal, separation and concentration of various metals [1,2]. In classical liquid–liquid extraction, small droplets are generated by agitation, which increases the contact surface area and improves the extraction efficiency. However, the classical procedure has not proved to be effective enough in some cases and presents some disadvantages (e.g. laborious manipulation, large volumes of solvents, high operation costs, possible formation of emulsions, large equipments and long analysis time, it is often time-consuming and equilibrium cannot be attained after shaking for a long time).

In the last few years, research activities are oriented towards the development of more efficient, economical, and miniaturized sample preparation methods in order to increase the extraction yields obtained under conventional solvent extraction. The advantages of miniaturization are the reduction of the sample size. In addition, transport phenomena are faster in microsystems than in ordinary systems, and therefore, one may expect that liquid extraction takes less time to be achieved in miniaturized

devices. This feature was demonstrated recently with many configurations [2,3].

One of the means for increasing the extraction efficiency is to disperse the extracting solvent into the liquid sample. For example, Rezaee et al. [4] developed a dispersive liquid–liquid microextraction (DLLME) method which is a miniaturized green sample preparation method. In their method a dispersing agent (acetone) was added to the extracting one (carbon tetrachloride) to extract organic compounds in water. A cloudy solution was then formed. Then this solution was centrifuged and the two phases were separated.

Molybdenum acidified aqueous solutions were chosen as the model metallic cation and organophosphorous extractants, such as tributyl phosphate (TBP) and bis(2-Ethylhexyl) phosphoric acid (D2EHPA) were employed to extract this element. This choice was based on the fact that molybdenum is considered as a bio-essential trace nutrition element for both plants and animals, including humans where it plays an important role [5–7]. Very low concentrations of molybdenum can be found in plants, natural water and seawater, and other aqueous matrices. For example, the assay of molybdenum in seawater is very important, since this metal is part of biochemical phenomena in most marine flora and fauna. Under normal conditions the reported concentrations are included within the 6–20 $\mu\text{g L}^{-1}$ range [2,3]. The complexity of the sample matrix makes advisable the development of

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