



An electrothermal vaporization unit with axially focusing convection upstream and influence of modifiers. Part I: Experimental [☆]

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Abstract

A novel, further improved construction of an electrothermal vaporization (ETV) unit is described, and new measurements are presented. Its main feature is an upstream within a vertical tube creating an axially focusing convection zone, where the condensation occurs in a laminar flow predominantly apart from the colder walls. The upstream is shielded against incandescent emission of the graphite furnace (GF) and surrounded with different cooling gas flows, that have additional focusing effect. The transported ETV generated aerosol is electrostatically precipitated onto a secondary platform that is subsequently analyzed for the transported analytes. Transport efficiencies (TEs) for Cu, Fe, Mn, Ni, and Pb are studied using a continuum source coherent forward scattering multi-element spectrometer coupled to the ETV. Increasing TE is observed with the ageing of the GF. The effect is presumably ascribed to the growing content of carbon particulates released during the vaporization. A positive impact of K and Pd modifiers in microgram amounts foremost on the TEs of more volatile species is observed and discussed.

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1. Introduction

Various constructions of electrothermal vaporization (ETV) cells have been used as sample introduction tools for inductively coupled plasma optical emission and mass spectrometry (ICP-OES/MS) [1–5] as well as for graphite furnace (GF) analytical techniques [6–9]. Advantages of the ETV dosing are the capability for solid, liquid, and slurry sampling, reduction of sample preparation time with reduced risk of sample contamination and of analyte loss prior to analysis. In addition, the ETV offers the possibility to separate the analytes

from matrix components by external thermal pretreatment and vaporization steps.

Up to now, the ETV devices are based on the following designs: (1) cup vaporizers, (2) end-on flow-through GFs with vapor outlet through one of the ends of the graphite tube [2–8,10–12], and (3) upstream configurations with the gas entering the GF through its ends and released upwards through a hole in the tube center [1,9,12,13]. The comparison shows the superiority of the upstream configuration [12]. Nevertheless, for both constructions an evident dependence of analyte transport efficiency (TE) and analyte losses on element volatility, sample mass, ETV operation conditions and other parameters is revealed.

The application of modifiers such as NaCl [10], Mg/Pd(NO₃)₂ [14,15], citric acid [14], or components of diluted NASS-3 seawater [16] can support the formation of transportable aerosol and increase the TE that allows to reduce the above mentioned dependencies. Generally, the modifier may improve the formation of carriers and the co-vaporization of sample analytes with carriers. Gaseous additives such as carbon hydrates, halide carbon hydrates [5,12,17,18], and carbon particle aerosols [19] are used

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