

# Multielement determination of heavy metals in water samples by continuous powder introduction microwave-induced plasma atomic emission spectrometry after preconcentration on activated carbon<sup>☆</sup>

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## Abstract

A novel continuous powder introduction microwave-induced plasma atomic emission spectrometry method (CPI-MIP-AES) has been developed for trace determination of metals in ground and tap water samples after preconcentration on activated carbon. The experimental setup consisted of integrated rectangular cavity TE<sub>101</sub> and vertically positioned plasma torch. The technical arrangement of the sample introduction system has been designed based on the fluidized bed concept. The satisfactory signal stability required for sequential analysis was attained owing to the vertical plasma configuration, as well as the plasma gas flow rate compatibility with sample introduction flow rate. The elements of interest (Cd, Cu, Cr, Fe, Mn, Pb, Zn) were preconcentrated in a batch procedure at pH 8–8.5 after addition of activated carbon and then, after filtering and drying of the activated carbon suspension, introduced to the MIP by the CPI system. An enrichment factor of about 1000-fold for a sample volume of 1 l was obtained. The detection limit values for the proposed method were 17–250 ng l<sup>-1</sup>. The proposed method was validated by analyzing the certified reference materials: SRW “Warta” Synthetic River Water and BCR CRM 399 major elements in freshwater. The method was successfully applied to the determination of the heavy metals in tap water samples.

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## 1. Introduction

Direct determination of trace elements in water samples sometimes meets difficulties due to the insufficient sensitivity of the detection system. Various preconcentration procedures have been proposed to improve the analytical performance of the instrumental method used. Among them, those with activated carbon (AC) as a collecting material for trace elements are widely used. Maximum sorption efficiencies were obtained in the presence of complexing agents

[1–4]. However, the use of activated carbon alone assures almost quantitative sorption of the elements after a suitable pH adjustment [5–9].

The aqueous sample solution usually passes the solid-phase extraction column, and the analytes are retained by the sorbent to be subsequently eluted with a suitable small volume of aqueous or organic solution before determination [1–9]. However, the elution of a group of analytes could be sometimes troublesome owing to irreversible binding of some elements. In addition, the desorption step leads to sample dilution and causes decrease of the analyte enrichment factor. In all of the abovementioned applications, the elements of interest were determined in eluates using different analytical techniques. The new approach, successfully applied in this work, is to carry out the direct multielement analysis of metals sorbed on activated carbon using microwave-induced plasma atomic emission spec-

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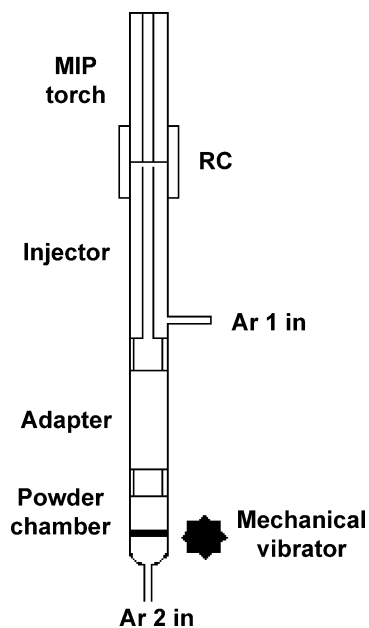


Fig. 1. Schematic diagram of the continuous powder introduction system (Ar1 in plasma gas inlet, Ar 2 in injection gas inlet, RC—rubber connector).

trometry with continuous powder introduction system (CPI-MIP-AES). An important advantage of MIP-AES is its compatibility with various sample introduction techniques.

Microwave-induced plasma atomic emission spectrometry proved to be the suitable analytical tool for trace element analysis and speciation studies [9–16]. The method exhibits maximum analytical potential when coupled to sample introduction techniques, which assure separation of the analyte from the matrix, i.e., gas chromatography, hydride generation and electrothermal vaporization. MIP-AES coupled with ETV is a useful and sensitive method for the analysis of microsamples, e.g., determination of less than  $0.1 \mu\text{g g}^{-1}$  of heavy metals in powdered 2-mg samples of various certified biological materials by Aziz et al. [13] and Yang et al. [14]. Kawaguchi and Vallee [15] determined picogram amounts of zinc by vaporizing microgram amounts of enzyme samples. The possible application is represented by the elemental analysis of coal samples using direct powder injection into the MIP by Gehlhausen and Carnahan [16]. However, the direct determination of ultratrace amounts of elements by MIP-AES is usually difficult owing to an insufficient detection power. In the last years, on-line separation and preconcentration techniques based on flow injection have been shown to be effective in enhancing the sensitivity of MIP-AES [9,17].

A direct introduction of sorbent materials (with analytes trapped) into inductively coupled plasma has been previously studied by Guevremont and De Silva [18] for silica immobilized 8-hydroxyquinoline and Chelex-100, by Rattray and Salin [19] for Chelex-100 and by Broekaert et al. [20] for modified cellulose. However, the use of activated carbon offers some unique advantages in combination with powder introduction technique. The sorbent is easily decomposed in

the plasma environment in comparison with chelating resins. Secondly, as a conducting medium, it improves the plasma excitation conditions and gives relatively low spectral interferences during the excitation.

In this work, we developed the solid sampling technique based on continuous activated carbon powder introduction into the Ar-MIP for multielement determination of heavy metals preconcentrated on the sorbent. The method was applied to ground and tap water analysis.

## 2. Experimental

### 2.1. Apparatus

The experimental setup used in this study consists of a TE<sub>101</sub> integrated microwave resonator with a microwave power supply MIP-2 and a vertically positioned aerosol cooled plasma torch (Plazmatronika-Service, Wrocław, Poland), a MIP-750 MV sequential spectrometer (Analab, Warsaw, Poland) [21] and a laboratory-made continuous powder introduction system. The borosilicate glass-made sample introduction system based on fluidized bed concept consists of a powder chamber in the form of fluidized column, a glass tube adapter and a sample injector device directly attached to the MIP torch with a silicon rubber connector. The total height of the CPI system was about 50 cm. A continuous argon flow is traveling up the outer tube of the sample injector to maintain the plasma. The injection gas flow rate transports the analyte material through the inner tube of the sample injector device to the MIP torch. The mechanical vibrator was used for sample agitation to improve the formation of particle aerosol throughout the measurements. A diagram of the device is shown in Fig. 1. Pure argon (99.999%) was used as the plasma gas. The instrument settings are detailed in Table 1.

### 2.2. Reagents

Activated carbon Darco G-60, -100 mesh (Aldrich, Steinheim, Germany) was used after pretreatment with hydrochloric acid. A suitable portion of AC was boiled two times with  $3 \text{ mol l}^{-1}$  of hydrochloric acid of GR grade for 10 min, filtered and washed with deionized water.

Standard stock solutions of individual metals were prepared from oxides of spectrographic grade (Johnson

Table 1  
Instrumental details and operating conditions for CPI-MIP-AES system

Microwave frequency [MHz]	2450
Microwave power [W]	180
Plasma torch, vertical position	Quartz, 4 mm i.d.
Plasma-viewing mode	Axial
Plasma gas flow rate [ $\text{ml min}^{-1}$ ]	100
Injection gas flow rate [ $\text{ml min}^{-1}$ ]	150
Integration time [s]	0.5

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