



Development of interface for online coupling of micro-fluidic chip-based photo-micro-reactor/ultrasonic nebulization with microwave induced plasma spectrometry and its application in simultaneous determination of inorganic trace elements in biological materials



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ABSTRACT

A novel analysis online system based on direct coupling of micro-fluidic chip-based photo-micro-reactor/ultrasonic nebulization (μ chip-UV/MR/USN) to microwave induced plasma-optical emission spectrometry (MIP OES) for simultaneous determination of inorganic trace elements in human body fluids and water samples was developed. A univariate approach and the simplex optimization procedure were used to achieve optimized conditions and derive analytical figures of merit. Minimum dead volume, high photo-decomposition and nebulization efficiency and high sample transport efficiency were obtained by using this interface. The experimental concentration detection limits for simultaneous determination, calculated as the concentration giving a signal equal to three times the standard deviation of the blank (LOD, $3\sigma_{\text{blank}}$ criterion, peak height) were 30, 6, 1, 2, 10, 2, 7, 5, 8, 10, 9 and $30 \mu\text{g L}^{-1}$ for Ba, Ca, Cd, Cu, Fe, Li, Mg, Mn, Pb, Sr, Zn and C, respectively. Repeatability expressed as the relative standard deviations (RSD, $n = 6$) for the target elements ranged from 4% to 8%. Quantitative data obtained for Seronorm urine and water reference materials were in good agreement with the corresponding reference values, proving the potential of this analysis system for multi-element trace analysis *via* MIP OES. The proposed method had been successfully applied for the determination of inorganic trace elements in urine, mineral water and implant fluid samples. The developed method is quite suitable for routine analysis of inorganic trace elements in biological and environmental fields and precious biological samples such as implant fluids.

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1. Introduction

Sample preparation is often the most critical step in analytical procedures [1]. The decomposition of organic matter is generally carried out by supplying energy to break chemical bonds and ultraviolet radiation can be employed to this end [2]. Despite the common use in view of simplicity, sample preparation in open or closed vessels, in macro-scale, requires large amounts of reagents, being also time consuming and susceptible to contamination and analyte loss, thus affecting accuracy and precision. Additionally, safety of the analyst may be hindered depending on the process and sample composition. Performing sample preparations in a micro-scale format has significant advantages for many applications and show characteristics that overcome most of these difficulties [3]. Flow-based procedures, in which sample preparation is carried out in micro-fluidic chip-based micro-reactor, provide the ability to perform sample preparations in extremely small total volumes [4].

Miniaturization is an important trend in biomedical and environmental monitoring, allowing reductions in cost, efficient sample preparation within small volumes, lower sample and reagent consumption and increased analytical speed [5]. Microfluidic technology is emerging as a new platform which is able to manipulate small volumes of liquid (μL – nL) in a micro-channel network with micro-scale dimensions. It demonstrates the capability to integrate different elements on a single chip in order to realize complex functionality.

The development of suitable multi-element methodologies for the determination of trace levels in biological and environmental samples is still a challenge for analytical chemistry, as in environmental context, especially when high sample throughput is required. Different element-specific detection techniques, such as atomic absorption spectrometry (AAS), optical emission spectrometry (OES), atomic fluorescence spectrometry (AFS), spectrophotometry and mass spectrometry (MS), have been used for the determination of inorganic trace elements in biological and environmental samples. Among them, microwave-induced plasma optical emission spectrometry (MIP OES) has some attractive features: low detection limits, wide linearity, multi-element capability and amenability for coupling with various online methodologies [6]. However, using these techniques, the procedures usually include extensive sample

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Table 1
Equipment and operating conditions of MIP OES.

Microwave plasma spectrometer	
Mounting	Czerny-Turner in tetrahedral setup
Focal length (mm)	500
Spectral range (nm)	193–852
Order lines	28th–123rd
MIP-OES parameters	
Microwave frequency (MHz)	2450
Microwave forward power (W)	100–200, variable
Microwave cavity	TE ₁₀₁ rectangular, water cooled
Microwave generator	700 W, MPC-01 (Plazmatronika Ltd., Wrocław, Poland)
Plasma viewing mode	Axial
Plasma form	Annular
Plasma torch	Quartz tube, 3.0 mm i.d., air cooled
Carrier argon flow rate (mL min ⁻¹)	200–1000, variable
Plasma supporting helium flow rate (mL min ⁻¹)	100–300, variable
OES data acquisition	
Read	On-peak
Integration time (s)	0.1
Background correction	Fixed point
Measurement mode	Peak height
Determination	Simultaneous
Analytical wavelength/nm (line type)	Ba 455.403 (II), Ca 393.366 (II), Cd 226.502 (II), Cu 324.754 (I), Fe 238.204 (II), Li 670.776 (I), Mg 285.213 (I), Mn 257.611 (II), Pb 405.783 (I), Sr 407.771 (II), Zn 213.857 (I), C 247.856 (I)

preparation procedures prior to instrumental analysis. The conventional wet digestion procedures require large quantities of high-purity reagents, which generate a considerable amount of hazardous waste. They also might include intensive manipulation, besides being time

Table 2
Operating experimental conditions for the developed μ chip-UV/MR-USN system.

Micro-reactor chip	
Material	Quartz
Chip size (mm)	90 × 28 × 4.5
Fabrication process	HF etching and thermal bonding
Reaction volume (μ L)	250
Mixing channel size (depth × width)(μ m)	250 × 300
Mixing channel length (mm)	532
Reaction channel size (depth × width)(μ m)	250 × 400
Reaction channel length (mm)	2509
UV-assisted sample mineralization	
UV light source pen lamp power (W)	6
Sample solution flow rate (μ L min ⁻¹)	9
Nitric acid concentration (% v/v)	10
Nitric acid solution flow rate (μ L min ⁻¹)	9
Mineralization time (min)	~15
Ultrasonic nebulization parameters	
NOVA-DUO	Dual-mode ultrasonic nebulizer without desolvation system
Solution flow mode	Continuous
Sample solution flow rate (μ L min ⁻¹)	18
Transducer frequency (MHz)	1.65
Acoustic power (W)	45
Transducer type (W)	Piezoelectric quartz plate, water cooled
Spray chamber	Cyclonic
Nebulizer gas (Ar) flow (mL min ⁻¹)	600

consuming and potentially introducing systematic errors due to incomplete analyte extraction.

Since atomic spectrometers typically need at least a few hundreds or thousands of micro-liters for analysis, relatively small number of works can be found in literature about the element-selective detectors hyphenated with microchips. Wang *et al.* [7] developed a chip-based liquid phase micro-extraction device to combine with electrothermal vaporization–inductively coupled plasma mass spectrometry (ETV-ICP-MS) for the determination of trace Cu, Zn, Cd, Hg, Pb and Bi in cell samples and human serum samples. Recently, micro-fluidic chip-based reaction chamber (30 μ L volume) was applied before the determination of chromium species using ICP-MS in aqueous environmental samples [8]. However, no combination of micro-fluidic chip-based photo-micro-reactor with the traditional and often used MIP spectrometer was found. Therefore, the combination of μ chip-UV/MR/USN with MIP OES could provide a powerful technique for the simultaneous determination of inorganic trace elements.

The objective of this work is to develop a simple and efficient interface for coupling of chip-based photo-micro-reactor (μ chip-UV/MR) with MIP OES by using high-efficiency micro-ultrasonic nebulizer (USN) and to establish a method for simultaneous determination of several trace elements in biological and environmental samples using the online hyphenated μ chip-UV/MR/USN-MIP OES system with the developed interface. Analytical conditions of the coupled μ chip-UV/MR/USN-MIP OES system by the proposed interface were optimized and discussed. A micro-reactor chip was proposed for sample micro-decomposition under mild conditions, aiming minimizing analysis time, reagent consumption, waste generation and risk of sample contamination. Furthermore, the method developed in this study was applied for the determination of target metal ions in a human body fluid and a water samples.

2. Experimental

2.1. Apparatus

2.1.1. MIP OES system

A Carl Zeiss Echelle spectrometer (Model PLASMAQUANT 100) using fiber-optical light-guides and photomultiplier tubes (PMT) and the TE₁₀₁ microwave plasma cavity assembly was used and it was essentially the same as previously described [9].

The MIP resonant cavity as an excitation source, specified previously [9] was used. The plasma is viewed axially with the axis of the plasma perpendicular to the plane of the entrance slit. Since the plasma torch, cavity, generator and gas flow have been described in detail in a previous paper [10], they will not be discussed again here but briefly summarized only. The microwave generator is connected by means of a flexible cable to the rectangular resonant cavity of the TE₁₀₁ design. The torch is a quartz capillary made of Suprasil (Heraeus, Hanau, Germany). Instrument settings and operational parameters used for the experimental MIP OES system are summarized in Tables 1 and 2. A schematic illustration of the system; diagram of the entire experimental setup (i.e., sample decomposition and introduction system-MIP OES) is shown in Fig. 1.

2.2. General experimental setup

2.2.1. Microfluidic chip-based UV/MR/USN system

The chip/photo-micro-reactor was interfaced to the MIP OES via a commercially available, low flow USN, as shown in Fig. 1.

The quartz micro-reactor chip of 250 μ L working volume (The Dolomite Centre Ltd., United Kingdom) for UV-assisted mineralization of environmental and biological samples was used. Sample solution and nitric acid solutions were introduced to the chip by two separate PTFE tubings (0.5 mm i.d., 20 cm length) using two-channel syringe pump (Asia, Syrris Ltd., United Kingdom) with flow of 9 μ L min⁻¹.

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