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Low gas flow inductively coupled plasma optical emission spectrometry for the analysis of food samples after microwave digestion



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ABSTRACT

In this work, the recently introduced low flow inductively coupled plasma optical emission spectrometry (ICP-OES) with a total argon consumption below 0.7 L/min is applied for the first time to the field of food analysis. One goal is the investigation of the performance of this low flow plasma compared to a conventional ICP-OES system when non-aqueous samples with a certain matrix are introduced into the system. For this purpose, arsenic is determined in three different kinds of fish samples. In addition several nutrients (K, Na, Mg, Ca) and trace metals (Co, Cu, Mn, Cd, Pb, Zn, Fe, and Ni) are determined in honey samples (acacia) after microwave digestion. The precision of the measurements is characterized by relative standard deviations (RSD) and compared to the corresponding precision values achieved using the conventional Fassel-type torch of the ICP. To prove the accuracy of the low flow ICP-OES method, the obtained data from honey samples are validated by a conventional ICP-OES. For the measurements concerning arsenic in fish, the low flow ICP-OES values are validated by conventional Fassel-type ICP-OES. Furthermore, a certified reference material was investigated with the low gas flow setup. Limits of detection (LOD), according to the 3σ criterion, were determined to be in the low microgram per liter range for all analytes. Recovery rates in the range of 96–106% were observed for the determined trace metal elements. It was proven that the low gas flow ICP-OES leads to results that are comparable with those obtained with the Fassel-type torch for the analysis of food samples.

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1. Introduction

Inductively coupled plasma-optical emission spectroscopy (ICP-OES) has proven to be one of the most powerful tools for trace metal analysis in recent decades.

Unfortunately a total argon consumption of 14–20 L/min is normal when a conventional Fassel-type torch is used. This high argon consumption of the ICP-OES system currently is the main driving force for the development of alternative excitation sources. The major part of the argon consumption is reserved for the cooling of the torch, so alternatives were sought for already more than thirty years ago [1]. The miniaturization of the conventional torch as a means to minimize the argon consumption has been described by different working groups [2–4]. Another approach was to change the cooling technique from internal to external by

using cheaper cooling media such as air [5,6] or water [7,8]. These investigations have led to the conclusion, that air was better suited than water as cooling media [9,10]. Dedicated assemblies for optimized gas flow lines were also one part of the investigations to reduce the argon consumption [11,12], as an alternative torch designs, which worked with reduced rf powers and lower argon flow rates [13]. The operation of an ICP-OES system below atmospheric pressure was another attempt to reduce the argon consumption [14,15]. One of the most recent constructive attempts was using a double featured gas inlet for argon and helium ICPs [16]. A new inductively coupled plasma torch was presented in 2005 with the Static High-Sensitivity ICP (SHIP) [17]. The analytical performance of this system with respect to its detection power is in general comparable with the state-of-the-art performance of the conventional setup in aqueous solution [18,19] and the system was successfully applied to the determination of rare earth elements in luminescent nanoparticles after microwave digestion and matched the state of the art of the conventional setup [20]. However, the system is less well characterized with respect to its

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application for samples with decisive matrix contents. The much lower argon consumption of the low-flow system leads to a higher sample and matrix concentration within the plasma. Also, the much lower argon flow rate and the bulb shaped design of the low flow torch lead to a higher residence time of the samples within the plasma compared to a Fassel-type torch, which can lead to devitrification and a rapid deterioration of the torch. Therefore it was our aim to investigate other matrices than aqueous samples with the low flow ICP-OES and to compare the results to those observed with the conventional setup. In order to investigate the robustness of the low-flow torch for the analysis of real-world samples with decisive matrix content we selected food samples. Therefore, arsenic was determined in plaice, haddock und catfish and several elements like potassium, sodium, magnesium, calcium, cobalt, copper, manganese, cadmium, lead, zinc, iron and nickel were determined in honey samples (acacia) after microwave digestion with the low flow ICP-OES.

2. Experimental

2.1. Instrumentation

2.1.1. ICP-OES/Low flow ICP-OES

For multielement determination, a Spectro CIROS^{CCD} ICP-OES (Spectro Analytical Instruments, Kleve, Germany) instrument with axial plasma viewing was used in two different operation modes. The gas flows were controlled by internal mass flow controllers. The free-running generator was operated at 27.12 MHz with a maximum output of 2 kW and the inner diameter of the load coil was 25 mm. In low flow ICP-OES mode, the conventional Fassel type torch was replaced by an externally air-cooled torch and the ICP was operated at significantly lower argon flow rates (< 0.7 L/min). The torch design was preliminary characterized in detail [20,21]. For sample introduction of the low flow ICP-OES, a self aspirating PFA nebulizer with a double pass spray chamber (Scott type) was used (Fig. 1). For the Fassel mode (ca. 14 L/min argon consumption), a standard Fassel-type torch (No. 75160526, Spectro Analytical Instruments) was employed and the system's peristaltic pump with a cross flow nebulizer and a double-pass spray chamber (Scott type) was used for sample introduction. The wavelengths for potassium, cobalt, lead, calcium, sodium, iron, copper, magnesium, nickel, cadmium, arsenic, zinc and manganese were selected based on their highest sensitivity.

The operating conditions for both ICP-OES measurements are shown in Table 1.

2.1.2. Microwave

Microwave sample digestion was carried out with a CEM Mars 5 (CEM Corporation, Mathews, NC, USA) instrument. Operational parameters for microwave digestion are shown in Table 2.

2.2. Reagents/Standards

2.2.1. Chemicals

Standards of arsenic, cadmium, calcium, cobalt, copper, iron, lead, magnesium, manganese, nickel, potassium sodium and zinc (1.000 mg/L for each element), nitric acid (suprapure grade) cesium chloride (per analysis) and lanthanum chloride (per analysis) were obtained from Merck KGaA (Darmstadt, Germany). Hydrogen peroxide, (per analysis) was purchased from ACROS Organics (Geel, Belgium). All chemicals were used in the highest quality available. Purified water (18.2 M Ω cm, TOC < 4 ppb, Milli-Q Gradient, Millipore GmbH, Schwalbach, Germany) was used for all experiments.

2.2.2. Standard and stock solutions

Volumetric flasks for ICP-OES measurements were pretreated with 2% suprapure HNO₃ and purified water to minimize adsorption effects. Calibration solutions (from 1.000 mg/L single element standard solutions) for ICP-OES and FAAS were prepared by diluting the

Table 1
Instrument parameters (Spectro CIROS^{CCD} ICP-OES, axial plasma viewing).

ICP-OES system	Low-flow ICP-OES	Conv. ICP-OES
Rf power (W)	1100	1400
Total argon flow (L/min)	0.66	14.05
Sample carrier gas (L/min)	0.36	1.05
Auxiliary plasma gas (L/min)	0.3	1.00
Outer plasma gas (L/min)	–	12.0
External air cooling (m/s)	60	–
Torch geometry	SHIP type ^a	Fassel type ^b
Torch injector tube i.d. (mm)	1	2.5
Nebulizer	PFA	CrossFlow
Sample uptake rate (mL/min)	0.27	2.00

^a 24 mm o.d. SHIP-II torch²⁰.

^b 19.5 mm o.d. conventional Fassel type torch.

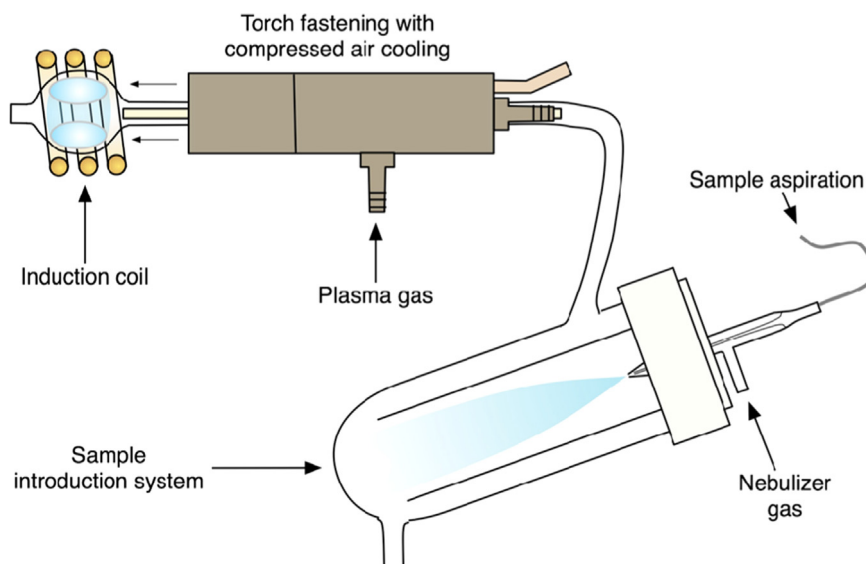


Fig. 1. Schematic sketch for the low flow ICP-OES.

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