



Comparison of a high temperature torch integrated sample introduction system with a desolvation system for the analysis of microsamples through inductively coupled plasma mass spectrometry



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ABSTRACT

This work describes for the first time the comparison of the analytical performances obtained with a high temperature torch integrated sample introduction system (hTISIS) against those found with a commercially available desolvation system (APEX) associated with inductively coupled plasma mass spectrometry (ICP-MS). A double pass spray chamber was taken as the reference system. Similar detection limits and sensitivities were obtained in continuous injection mode at low liquid flow rates for the APEX and hTISIS operating at high temperatures. In contrast, in the air-segmented injection mode, the detection limits obtained with hTISIS at high temperatures were up to 12 times lower than those found for the APEX. Regarding memory effects, wash out times were shorter in continuous mode and peaks were narrower in air segmented mode for the hTISIS as compared to the APEX. Non spectral interferences (matrix effects) were studied with 10% nitric acid, 2% methanol, for an ICP multielemental solution and a hydro-organic matrix containing 70% (v/v) acetonitrile in water, 15 mmol L⁻¹ ammonium acetate and 0.5% formic acid containing lanthanide complexes. In all the cases, matrix effects were less severe for the hTISIS operating at 200 °C and the APEX than for the double pass spray chamber. Finally, two spiked reference materials (sea water and Antarctic krill) were analyzed. The hTISIS operating at 200 °C gave the best results compared to those obtained with the APEX and the double pass spray chamber. In conclusion, despite the simplicity of the hTISIS, it provided, at low liquid flow rates, results similar to or better than those obtained with the by other sample introduction systems.

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1. Introduction

Inductively coupled plasma mass spectrometry (ICP-MS) has become a widely used analytical technique across many industrial and research applications [1]. However, sample matrix can lead to spectral [2,3] as well as non spectral interferences [4] and/or nebulizer blockage because of the high solids content of the solution. Matrix can also affect the aerosol characteristics thus modifying the total mass of analyte transported towards the plasma and the analytical performances [5–9]. It is widely known that this kind of interferences can be mitigated by careful selection of the operating conditions, mainly the nebulizer gas flow rate and the generator RF power. Additional alternatives to increase the plasma robustness in ICP-MS, thus reducing the extent of matrix effects, include the addition of an auxiliary nitrogen gas stream [10]

or to employ a modified torch with improved gas dynamics. Obviously, these solutions do not correct for matrix effects originated from the sample introduction system. These phenomena can be thus overcome by employing an appropriate sample introduction system. On this subject, it is recognized that the spray chamber is a key component of the system in terms of matrix effects [11].

For the analysis of certain biological, nuclear, toxic or forensic samples, whose available volume or analyte mass are small, a high efficiency sample introduction system is required for trace elements determination [12,13]. The use of low sample consumption devices makes it possible to simplify the spray chamber design, thus minimizing the matrix effects. Nevertheless, even for these systems, matrix effects can still be observed, especially if plasma conditions are not robust as they depend on the liquid flow rate, matrix composition and characteristics of the sample introduction device. Note that with these devices, the analyte mass transport efficiency ranges from roughly 10 to 60%, although the actual value strongly depends on variables such as the liquid flow rate,

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spray chamber design and aerosol temperature. Therefore, the plasma may degrade, thus giving rise to a change in the sensitivity and/or promotion of spectral and non-spectral interferences. One of the ways employed to lower the solvent plasma load is to decrease the temperature of the spray chamber walls. Spray chambers thermostated at low temperatures show several advantages regarding the reduction of the extent of polyatomic spectral interferences in ICP-MS [14–17]. However, the solvent evaporation inside the spray chamber is dampened what lowers the solvent plasma load. Unfortunately, the use of such devices also lowers the analyte mass transported towards the plasma [18]. A desolvation system operating in two separate steps can also be proposed; the wet aerosol is first heated so as to promote the solvent evaporation, hence increasing the transport efficiency, then a significant fraction of the generated solvent vapor is removed by either lowering the temperature or using a membrane [19–21]. There are several commercially available designs, among them is the so-called APEX [22]. Desolvation systems afford lower limits of detection with respect to conventional sample introduction systems [9,23–26]. This is due to two main reasons: on the one hand, lower background noise levels are achieved and, on the other hand, the mass of analyte delivered to the plasma is increased. Previous studies have demonstrated that the so-called torch integrated sample introduction system working at high temperature (hTISIS) mitigated the extent of the interferences caused by organic as well as inorganic concomitants both in ICP-OES and ICP-MS [27–29]. In order to further remove the interferences and, simultaneously, lower the solvent plasma load, a segmented-flow injection mode can be used, while increasing the chamber temperature up to 350 °C [28,29]. Under these conditions, the analyte transport efficiency is virtually 100% regardless the sample matrix.

To the best of our knowledge, there is not any previous study aimed at comparing the performances obtained in ICP-MS through two main approaches: removal of the solvent by means of a desolvation system and promotion of the aerosol evaporation by increasing the chamber temperature. The goal of the present work was thus to compare the analytical performances of the hTISIS and the APEX desolvation system against those obtained with a double pass spray chamber in ICP-MS working both in continuous sample aspiration mode at low liquid flow rates (*c.a.*, 20 $\mu\text{L min}^{-1}$) and segmented-flow injection mode. A further goal was to evaluate the sensitivity obtained with the hTISIS and the APEX sample introduction systems for a wide range of elements in various inorganic (nitric, sodium) and/or organic (methanol, acetonitrile) matrices either alone or mixed as well as their application to the analysis of environmental reference materials (*i.e.*, sea water, krill). Besides, it was also interesting to evaluate whether the hTISIS was able to provide similar or better performances than those obtained with a more complex system such as the APEX.

2. Experimental

2.1. Chemicals and samples

Ultrapure water was supplied by a three-step ion-exchange system Milli-Q, fed by reverse osmosis, Elix 3, both from Millipore (El Paso, TX, USA). An ICP multielement standard solution (Merck IV, Merck KGaA, Darmstadt, Germany) containing 1000 mg element L^{-1} was used to prepare the standards (100 $\mu\text{g L}^{-1}$) by a proper dilution into the corresponding matrix. 65% HNO_3 (Suprapur®, Merck, Darmstadt, Germany), methanol (HPLC grade, Panreac, Barcelona, Spain) and acetonitrile (UHPLC grade, Panreac, Barcelona, Spain) were also employed. 65% HClO_4 and 30% H_2O_2 were provided by, Panreac, Barcelona, Spain. Ammonium acetate and formic acid were obtained from Panreac (Barcelona, Spain) and Merck KGaA (Darmstadt, Germany), respectively. Diethylenetriaminepentaacetic acid (DTPA) ($\text{C}_{14}\text{H}_{23}\text{N}_3\text{O}_{10}$; purity $\geq 98\%$) and ethylenediaminetetraacetic (EDTA) tetrasodium salt dihydrate ($\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}_8\text{Na}_4 \cdot 2\text{H}_2\text{O}$; purity $\geq 99.5\%$) were purchased from Sigma

Aldrich (Saint-Quentin Fallavier, France). 25% (w/w) Ammonia solution was employed to adjust the solution pH.

Two reference materials (RMs) were analyzed in the present work. The first one corresponded to a NASS-6 seawater certified reference material provided by NRC (National Research Council Canada, Canada). The second RM (ISS-A2) corresponded to an Antarctic krill (National Institute of Health, Italy). In order to carry out the analysis through ICP-MS, this sample was prepared by microwave-assisted acid digestion. Approximately 250 mg of ISS-A2, weighed with a precision of ± 0.1 mg, were transferred to a microwave digestion vessel and then 6 mL of 65% nitric acid, 1 mL of 65% HClO_4 and 1 mL of 30% H_2O_2 were added. The sample was digested at 200 °C for 30 min, using the microwave digestion system Start D (Milestone, Sorisole, Italy). The digestates were transferred to graduated glass flasks and diluted to 25 mL with Milli-Q water.

Neodymium, europium, gadolinium and erbium elemental standard solutions (SPEX Certiprep Group, Longjumeau, France) containing 1000 mg element L^{-1} in HNO_3 1.5%, w/w, were used for the preparation of an additional hydro-organic matrix. Lanthanide (Ln) stock solutions were obtained by diluting each lanthanide standard solution in ultrapure water, to obtain a concentration of 5 mmol L^{-1} . Ligand stock solutions (5 mmol L^{-1}) were prepared by dissolving appropriate amount of DTPA and EDTA in ultrapure water. These complexing agents are used in advanced spent nuclear fuels treatment processes [30]. Lanthanide complexes of EDTA and DTPA, representative of species found in aqueous back extraction phases encountered in actinides/lanthanides partitioning from spent nuclear fuel, were prepared in water, by mixing suited volumes of ligand and elemental stock solutions, to reach the Ln:EDTA:DTPA ratio of 2:1.75:0.75 with Ln concentration at 1 mmol L^{-1} each. The pH was further adjusted to 3.2 with concentrated ammonia. The final samples were obtained by dilution of that solution in the hydro-organic matrix containing acetonitrile/water 70/30 (v/v), 15 mmol L^{-1} ammonium acetate and 0.5% formic acid.

2.2. Instrumentation

A 7700× Agilent ICP-MS spectrometer (Agilent, Santa Clara, USA) equipped with a 12 MHz octopole ORS [3] cell was used. Table 1 summarizes the main instrumental conditions employed. The ORS [3] collision cell mode with helium as gas was employed with the aim of removing potential polyatomic spectral interferences in the ICP (*e.g.*, $^{40}\text{Ar}^{12}\text{C}^+$ on $^{52}\text{Cr}^+$ or $^{40}\text{Ar}^{15}\text{N}^+$ on $^{55}\text{Mn}^{55}$).

A High efficiency nebulizer (HEN, Meinhard®, Colorado, USA) was used throughout the study. The reference spray chamber was a double pass Scott-type chamber set at 2 °C. A hydrofluoric acid resistant desolvation system (APEX HF, ACM, ESI, Omaha, NE, USA) was also employed. In this case, a heated cyclonic spray chamber is adapted to a first air cooled condenser at room temperature followed by a second Peltier-cooled multi-pass condenser. This second condenser holds a N_2 port to decrease the oxide interferences and to improve the sensitivity,

Table 1
ICP-MS operating conditions.

Nebulizer gas flow rate (Q_g)	0.65 L min^{-1}
Sample flow rate (Q_s)	20–90 $\mu\text{L min}^{-1}$
Dilution gas (High Matrix Introduction, HMI)	0.56 L min^{-1}
ICP-MS Agilent 7700×	
RF Power	1600 W
Plasma gas flow rate	18 L min^{-1}
Auxiliary gas flow rate	1.2 L min^{-1}
Collision cell (He)	4.3 mL min^{-1} (recommended by the manufacturer)
Isotopes measured	^{53}Cr , ^{55}Mn , ^{59}Co , ^{60}Ni , ^{63}Cu , ^{88}Sr , ^{111}Cd , ^{137}Ba , ^{140}Ce , ^{146}Nd , ^{153}Eu , ^{157}Gd , ^{166}Er , ^{208}Pb

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