

Technical note

# A new HF-resistant tandem spray chamber for improved determination of trace elements and Pb isotopes using inductively coupled plasma-mass spectrometry

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Received 20 January 2005; accepted 19 April 2005

Available online 17 May 2005

## Abstract

The use of a new HF-resistant tandem spray chamber arrangement consisting of a cyclonic spray chamber and a Scott-type spray chamber made from PFA and PEEK provides a straightforward approach for improving the performance of inductively coupled-mass spectrometry (ICP-MS). The characteristics of the tandem spray chamber were critically evaluated against a PEEK cyclonic and a PFA Scott-type spray chamber, respectively. Sensitivity across the entire mass range was increased by about three times compared to the conventional setup utilizing only one spray chamber. Precision of the results, especially at low signal intensities, improved by 160% and 31% compared to the cyclonic and Scott-type spray chamber, respectively. Using the tandem spray chamber, the oxide formation rate was lowered by about 50%. Signals as low as 30 counts could be determined under routine measurement conditions with a RSD of 2.4% thus allowing to precisely quantify small concentration differences at the  $\text{ng l}^{-1}$  concentration level. The excellent precision (0.02–0.07%) of  $^{206}\text{Pb}/^{207}\text{Pb}$  and  $^{206}\text{Pb}/^{208}\text{Pb}$  ratios determined in pore water samples was rather limited by the instrumental capabilities of the single collector ICP-MS instrument than by the performance of the tandem spray chamber.

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**Keywords:** ICP-MS; Tandem spray chamber; Lead isotope ratios; Trace elements; Pore waters

## 1. Introduction

The stability and efficiency of the sample introduction system is one of the limiting factors for the precision achievable using ICP-MS. The smoother the sample is introduced into the mass spectrometer, i.e. the more homogeneous the droplet size distribution of the sample aerosol, the better the precision of the analytical results.

In this respect, the two most frequently employed types of spray chambers used in ICP-MS, cyclonic and Scott-type spray chambers, respectively, offer different performance characteristics. Cyclonic spray chambers provide

higher sensitivity, whereas Scott-type spray chambers generally help to produce a more stable sample aerosol. Assembling both spray chamber designs in series yielding a single tandem spray chamber arrangement can potentially combine the advantages of both ways of aerosol processing. A similar tandem spray chamber made from quartz glass and introduced several years ago already demonstrated improved performance, but suffers from some drawbacks which have restricted its use so far [1–4]. For example, additional heating of the cyclonic spray chamber with an IR lamp and cooling the Scott-type spray chamber was required to improve sensitivities by about three times [1] compared to one spray chamber. Moreover, this tandem spray chamber is made from quartz glass and thus has another two major disadvantages: 1) analyte solutions containing HF or  $\text{HBF}_4$  cannot be analyzed and 2) even though quartz glass will provide blank contribu-

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tions that are acceptably low for many applications, contaminations originating from chemically inert materials such as PFA and PEEK are much smaller and thus these materials are nowadays preferred for low level ( $\text{ng l}^{-1}$  or below) determinations.

The aim of this study is to critically evaluate the performance characteristics of the prototype of a new HF-resistant tandem spray chamber by the determination of 24 trace elements (Ag, Al, Ba, Bi, Ca, Cd, Co, Cr, Cs, Cu, Fe, Mg, Mn, Ni, Pb, Rb, Sb, Sc, Sr, Tl, Ti, U, V, Zn) and Pb isotope ratios in small volumes of humic-rich pore water samples from peat bogs using clean room facilities and an inductively coupled plasma-sector field mass spectrometer (ICP-SMS).

## 2. Experimental

### 2.1. Laboratories and instrumentation

All sample handling in the laboratory and the preparation of all standards were performed in clean rooms under laminar flow clean air benches of class 100 to minimize the potential risk of contamination.

The ICP-MS used in this study was an Element2 ICP-SMS (Thermo Electron, Bremen, Germany) equipped with a guard electrode to eliminate secondary discharge in the plasma and to enhance overall sensitivity. A micro volume autosampler (ASX 100, Cetac Technologies, Omaha, NE, USA) and a HF-resistant sample introduction kit consisting of a microflow PFA nebulizer (Elemental Scientific Inc., Omaha, NE, USA), a PEEK cyclonic spray chamber (internal volume: 40 ml) or a PFA double pass Scott-type spray chamber (internal volume: 100 ml) and a sapphire injector tube served as the standard configuration. In the tandem spray chamber arrangement, a prototype consisting of the PEEK cyclonic spray chamber mentioned above was directly connected to a Scott-type spray chamber made from PFA (internal volume: 100 ml) (electronic supplementary information—ESI, Fig. 1). Both spray chambers were drained using a peristaltic pump operated at 2 rpm. At the low sample uptake used in this study ( $<100 \mu\text{l min}^{-1}$ ), however, no liquid was drained from the Scott-type spray chamber.

The PFA nebulizer was operated in the self-aspirating mode ( $\sim 75 \mu\text{l min}^{-1}$ ) to avoid potential contamination from the peristaltic pump tubing and to allow a smooth introduction of the sample aerosol into the ICP-SMS. The optimum nebulizer gas flow was slightly higher for the tandem spray chamber compared to that needed when the ICP-SMS was operated with only one spray chamber. Further details of the ICP-SMS operating conditions and the data acquisition parameters for both concentration and Pb isotope determinations have been reported previously [5–8]. The entire sample introduction system was hosted in a class 100 laminar flow bench.

### 2.2. Reagents and standards

For the preparation of all solutions, high purity water ( $18.2 \text{ M}\Omega \text{ cm}$ ) from a MilliQ-Element system designed for ultra trace analysis (Millipore, Milford, MA, USA) was used. Nitric acid (65%, analytical-reagent grade, Merck, Darmstadt, Germany) was further purified twice by distillation, using a high purity quartz unit for sub-boiling of acids (MLS GmbH, Leutkirch, Germany). Both the water purification system and the sub-boiling distillation unit were operated in clean rooms.

Calibration solutions for the 24 elements considered (Ag, Al, Ba, Bi, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Ni, Pb, Rb, Sr, Tl, Ti, U, V, Zn) were prepared daily by appropriate dilution of a multi element stock standard solution (Merck) and single element standard solutions (Cs, Sb, Sc; Merck) with 1% (v/v) high purity nitric acid. Quantification of trace element concentrations was performed establishing calibration curves by linear regression. For daily mass calibration of the ICP-SMS, a multi element solution containing  $10 \text{ mg l}^{-1}$  of each Ba, B, Co, Fe, Ga, In, K, Li, Lu, Na, Rh, Sc, Tl, U and Y was diluted with 1%  $\text{HNO}_3$  to a concentration level of  $1 \mu\text{g l}^{-1}$ . Indium at a concentration of  $1 \mu\text{g l}^{-1}$  was used for internal standardization of the measurements. A 0.05% detergent (Klarin®, Reinelt, Friedberg, Germany) solution, used for rinsing in between the analyte solutions, was prepared by diluting appropriate amounts of the detergent with 1% high purity nitric acid.

### 2.3. Sample collection and processing

Samples (volume: 10 ml) were collected from specific depths using a pre-cleaned, home-made pore water extraction device constructed from plexiglass. Immediately after collection, the pore water samples were filtered on-site through  $0.45 \mu\text{m}$  high purity filters (GD/XP, Whatman) into 15 ml-Falcon® tubes. Following US EPA Method 1669 [9], pore water samples were acidified with double sub boiled nitric acid to 1% (v/v) in a class 100 clean bench upon arrival in the laboratory. After some days of storage at  $4^\circ\text{C}$  in the refrigerator, a precipitate of humic acids was visible in the pore water solutions. This precipitate, developing at low pH values, was subjected to centrifugation at 3000 rpm for 10 min. The supernatant was analyzed for trace elements and Pb isotopes in this study. Detailed investigations on the trace element content of the precipitate revealed that generally the precipitate contains only a very small amount (from 2% to 4% of the total metal concentration of the filtered pore water sample) of trace elements with a maximum of 10% for Cu and Cr in the worst case. Thus it is well justified to consider only the supernatant for analyses.

All pore water samples were collected in triplicate at each specific depth. The results presented for Pb isotope ratios are the mean of the analyses of the three pore water samples.

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