

***Magnetic Sector ICP Mass Spectrometry - Short Course
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COURSE TITLE

APPLICATION OF MAGNETIC SECTOR INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY IN GEOSCIENCES - FUNDAMENTALS, INSTRUMENTATION AND APPLICATIONS

Course Abstract

Although Inductively Coupled Plasma Quadrupole Mass Spectrometry (ICP-QMS) is a well established technique for multitrace element determinations, it has limitations due to plasma and matrix derived spectroscopic interferences that result in degradation of detection limits and incorrect analytical results. Therefore unambiguous separation of analyte ions from spectroscopic interferences is a prerequisite of accurate and precise elemental analysis.

Several strategies have been employed to reduce these interference effects: Correction equations, front-end sample introduction, plasma optimization, matrix separation, collision and reaction cells and high resolution magnetic sector ICP-MS (HR-ICP-MS). However, because preconcentration procedures are tedious and contamination prone and cell conditions (gases, and flow rates) are highly variable, a high resolution ICP-MS is needed in order to mass-separate the numerous interferences and allow reliable quantification of the isotopes of interest. (The more complex the matrix, the more interferences are to be expected).

In this short course we will briefly describe fundamental principles of HR-ICP-MS and focus on spectroscopic interferences derived from the matrix, the plasma and

entrained gases. The current instrument configurations will be described followed by an examination of analytical figures of merit and a critical comparison of magnetic sector ICP-MS with alternate collision and reaction cells using conventional figures of merit such as *REAL* limits of detection, sample throughput, sample preparation requirements, ease of use and cost per analysis. In making this comparison the following pragmatic questions will be addressed: In what way is Magnetic Sector ICP-MS better than collision and reaction cells? Is high mass resolution the universal means for reducing spectroscopic interferences and for taking full benefit of the multielement capability for determination of low analyte concentrations in geological and related samples such as brines, complex geological samples using conventional sample and advanced sample introduction strategies

Multicollector ICP-MS instrumentation will be described and its performance reviewed for accurate isotopic determination for solving geological and environmental problems. This strategy will be compared with TIMS.

Course outline

1. INTRODUCTION

- 1.1. Introduction to high resolution magnetic sector ICP-Mass Spectrometry (HR-ICP-MS).
- 1.2. Historical aspects
- 1.3. Why do we need 'High Resolution'

2. TYPES OF SPECTROSCOPIC INTERFERENCES

- 2.1. Polyatomic ions, multiply charged ions and isobaric isotopes. Interferences include species derived from the plasma (Ar), entrained gases (C, O, N, H) and matrix.
- 2.2. Can we predict-Identify interferences on analyte isotopes of interest?

3. FUNDAMENTALS

- 3.1. What is a magnetic sector?
- 3.2. Ion separation mechanisms – magnetic fields, kinetic energy
- 3.3. 'Double focusing' (angular and energy focusing).
- 3.4. What are the differences between HR-ICP-MS and quadrupole ICP-MS, sensitivity, resolution, precision, background noise
- 3.5. Peak shape
- 3.6. Why magnetic sector MS is more sensitive than quadrupole ICP-MS

4. INSTRUMENTATION

- 4.1. Instrument configurations and principles of operation. Single focusing vs. Nier-Johnson double-focusing designs.
- 4.2. Ion sampling from the plasma – plasma shields
- 4.3. Description of the mass analyzer
- 4.4. Ion and energy focusing:
- 4.5. *Electromagnet - ion focusing*
- 4.6. *Electrostatic analyzer (ESA) – ion energy focusing.* In standard design, ESA is located in front of magnet. In reversed design – it is situated behind the magnet.
- 4.7. Collector slits. High resolution is achieved by making slits very narrow so that the beam reaching the detector only has a very narrow band width of mass.
- 4.8. Detector - Pulse counting electron multiplier and analog detectors to ensure a wide linear dynamic detection range

5. ANALYTICAL FIGURES OF MERIT

- 5.1. Relation between mass resolution and sensitivity
- 5.2. Limits of detection at high resolution – Alice in Wonderland
- 5.3. Does the reduction in sensitivity at high resolution compromise limits of detection
- 5.4. Analytical power of the system – superior limits of detection and accurate fit-for-purpose data
- 5.5. Limitations of resolution – isobars
- 5.6. Procedure for determining resolution

- 5.7. Space charge and matrix effects – salt barrier
- 5.8. Mass bias – Origin of mass discrimination. Plasma vs. mass spectrometer

5. OVERVIEW OF MULTICOLLECTOR ICP-MS

- 1.1. Relationship between Multicollector (MC) ICP-MS and TIMS
- 2.2. Instrument configuration, zooming and rpq.
- 3.3. Detectors

6. APPLICATIONS

- 6.1. Direct analysis of seawater, industrial brines, complex geological samples hazardous wastes, using conventional and advanced sample introduction procedures.
- 6.2. Focus on the determination of “difficult” transition elements
- 6.3. Speciation using GC and LC interfaces
- 6.4. High precision isotope ratios determination using ICP-MC-MS
- 6.5. Flat peaks, mass discrimination correction laws
- 6.6. Application of multicollectors in petrogenesis, age determinations and as environmental markers
- 6.7. Laser Ablation for local analysis and isotope ratios

7. Participant Discussions

- 7.1. Is larger operator competence required in high resolution ICP-MS and MC-MS. Because of smaller interferences, is HR-ICP-MS simpler?
- 7.2. Weighing the merits for user applications – resolution vs. sensitivity
- 7.3. Precision of isotope ratio determinations – Multicollectors vs. TIMS

Biographical sketch of the instructor

Dr. Isaac (Joe) Brenner is as a senior consulting scientist in Brenner Scientific that specializes in compliant routines in environmental and nutrition technologies. He obtained his Ph.D. in Geochemistry from the Hebrew University, Jerusalem, Israel in 1980. Dr. Brenner was a guest professor in the Earth Dynamics Science Center in the National Cheng Kung University in Taiwan. In the past, Joe Brenner was a guest scientist in the Laboratoire de Chimie Analytique Bio Inorganique et Environnement, (LCABIE) CNRS, Pau, France and the Chuo University, Tokyo, Japan

He was head of the application labs in Jobin Yvon in Longjumeau, France; a senior scientist in the Varian Research Center, Palo Alto, California, USA In these positions, he developed plasma-based analytical methodologies for environmental, high technology, clinical and nutrition samples. Brenner is an independent consulting scientist for the application, marketing and sales for environmental, nutrition and clinical analysis. Brenner specializes in preparing laboratories for ISO 22000 and ISO/EC 17025 accreditation using AOAC, Standard Methods and US EPA procedures. This includes sampling strategies, preparation of SOPs, instruction, QA/QC and internal audits world-wide.

Dr. Joe Brenner has delivered more than 500 oral presentations, short courses, round table discussions, at universities, international symposiums, research institutes, and instrument manufacturers; he has 120 peer-reviewed scientific publications.