



Application Note # CA-270109

High Matrix Sample Analysis with the Bruker 820-MS using the Collision Reaction Interface

Introduction

Inductively Coupled Plasma Mass Spectrometry is a powerful tool for a wide range of applications. The excellent sensitivity, multi-element capability and the wide analytical range give this technique the ability to analyze trace levels in a variety of environmental samples.

The unique design of the Bruker 810-MS and 820-MS features a patented 90-degree ion mirror [1] that significantly reduces the background signals. It allows photons and neutrals to pass directly through the hollow structure of the mirror while analyte ions are directed into the mass analyzer. The amount of analyte passing through the skimmer cone to the quadrupole is over 80%, which provides excellent ion transmission efficiency [2].

The new Bruker 820-MS, in addition to the benefits of the ion mirror, has also been designed to reduce/eliminate interferences. The unique Collision Reaction Interface (CRI) introduces a new era of interference management for ICP-MS systems.

This application note demonstrates the capability of the Bruker 820-MS system to determine sub- $\mu\text{g/L}$ concentrations of a large number of elements in solutions containing high amounts of total dissolved solids (often defined as "high matrix" samples) as waste extracts or digests.

Instrumentation

All measurements were carried out using the Bruker 820-MS equipped with Collision Reaction Interface (CRI) and a Sample Preparation System, SPS3. The system operations of the Bruker 820-MS are fully controlled by Bruker's ICP-MS Expert software. The software provides one-step instrument setup, optimization, and method development. By using the auto-optimization routine supplied in the ICP-MS Expert software, the instrument was automatically tuned to chosen CRI conditions.

Materials and reagents

All calibration standards were prepared by diluting multi-element stock standards (Var-Cal-1 and Var-Cal-2, Inorganic Ventures, Inc., Lakewood, NJ, USA) with 1% v/v nitric acid (Ultrapur[®] HNO₃ 60%, Merck, Kilsyth, Victoria, Australia). Working standards below 10 $\mu\text{g/L}$ were prepared immediately before the measurement. An internal standard solution, containing 100 $\mu\text{g/L}$ of ⁶Li, ⁴⁵Sc, ¹¹⁵In, ⁸⁹Y, ¹⁵⁹Tb, and ²⁰⁹Bi, was prepared by diluting a 100 mg/L of internal standard stock (Var-IS-1 Inorganic Ventures, Inc., Lakewood, NJ, USA).

Sample preparation

High matrix samples were prepared to match the EPA 6020 interference check sample matrix (ICS A) [3]. Samples and spikes were prepared by dilution of a stock solution (6020ICS-A, Inorganic Ventures, Inc., Lakewood, NJ, USA) and then spiked to a final concentration of 0.5 $\mu\text{g/L}$ with the multi-element stock standard (Var-Cal-1 and Var-Cal-2, Inorganic Ventures, Inc., Lakewood, NJ, USA). Table 1 lists the elemental concentrations of the high matrix sample (ICS A) and the spiked solution (ICS AB).

The EPA 6020 method requires MDLs to be determined using a spiked, fortified blank solution. That is, a sample at low levels with no matrix. By using ICS-AB to determine the MDLs, the data below shows that even with a highly demanding sample (with a high matrix) the Bruker 820-MS can easily meet the required control limits.

Table 1: Composition of high matrix sample and spike.

Analytes	Concentrations in mg/L	
	ICS A	ICS AB
Al	100,000	100,000
Ca	100,000	100,000
Fe	100,000	100,000
Mg	100,000	100,000
K	100,000	100,000
Na	100,000	100,000
C	200,000	200,000
Cl	1,000,000	1,000,000
P	100,000	100,000
S	100,000	100,000
Mo	2,000	2,000
Ti	2,000	2,000
As		0.5
Cd		0.5
Co		0.5
Cu		0.5
Mn		0.5
Ni		0.5
Ag		0.5
Zn		0.5
Sb		0.5
Ba		0.5
Pb		0.5
Se		0.5
Tl		0.5
V		0.5

Table 2: ICP-MS conditions for analyses of interference check samples.

Parameters	CRI 1	CRI 2
Skimmer gas	H ₂	He
Sampler gas	none	none
Skimmer flow (mL/min)	90	180
Sampler flow	0	0
Outer flow	16.5	16.5
Intermediate flow	1.65	1.65
Sheath gas	0.2	0.2
Nebulizer flow	0.95	0.98
RF power (kW)	1.3	1.3
Sampling depth (mm)	6.5	6.5
Pump rate (rpm)	3	3
Stabilization delay (s)	60	60
Spray chamber (°C)	3	3
First extraction lens (V)	-1	-1
Second extraction lens (V)	-21	-75
Third extraction lens (V)	-195	-235
Corner lens (V)	-177	-197
Mirror lens left (V)	39	38
Mirror lens right (V)	35	39
Mirror lens bottom (V)	23	24
Entrance lens (V)	0	-2
Fringe bias (V)	-2.5	-3.5
Entrance plate (V)	-29	-28
Pole bias (V)	0	0
Scan mode	Peak hopping	Peak hopping
Dwell time (ms)	20	20
Points per peak	1	1
Scans/Replicate	20	20
Replicates/Sample	5	5

Conditions

For the analysis of all samples in this work, normal sensitivity mode was used. Two condition sets were used in this study. Both sets used CRI conditions. The method parameters used for the two conditions sets are summarized in Table 2.

Discussion

In order to verify corrections for elemental and polyatomic isobaric interferences EPA Method 6020 requires the analysis of two interference check samples, ICS A and ICS AB, at the beginning of the analysis run.

In this work, the ICS AB solution (0.5 µg/L) was analyzed to evaluate a lower working range in the presence of a high

matrix. Table 3 summarizes the found results for the ICS A and ICS AB solutions and recoveries of the 0.5 µg/L spike for the different CRI conditions.

This spike level of 0.5 µg/L is 40 times lower than EPA 6020 requires (20 µg/L). This was done to make the analysis more demanding.

The Method Detection Limits (MDLs) and Method 6020 control limits are also listed. Seven subsequent reading of the ICS AB solution were used to calculate MDLs for each of the selected isotopes.

Table 3. Results summary for Interference check samples spike recoveries.

Element	Isotope (m/Z)	Spike level		With CRI		
		ICS AB (µg/L)	Recovery %	MDL* (µg/L)	CRDL (µg/L)	CRI condition
V	51	0.50	94	0.183	5	[2]
Cr	53	0.50	110	0.259	5	[2]
Mn	55	0.50	101	0.103	5	[2]
Co	59	0.50	98	0.032	5	[2]
Ni	60	0.50	103	0.089	5	[2]
Cu	63	0.50	107	0.297	10	[1]
Zn	66	0.50	145	0.253	10	[1]
As	75	0.50	101	0.292	1	[1]
Se	78	0.50	105	0.257	5	[1]
Ag	107	0.50	99	0.050	5	[2]
Cd	111	0.50	102	0.107	1	[2]
Sn	118	0.50	99	0.100	1	[2]
Sb	121	0.50	102	0.027	-	[2]
Ba	137	0.50	102	0.031	5	[1]
Tl	205	0.50	94	0.038	5	[2]
Pb	206+7+8	0.50	97	0.036	10	[1]

[1] Skimmer H₂, [2] Skimmer He

* MDL were calculated using SD of seven subsequent readings of interference check sample spike (ICS AB) multiplied by 3.14

Conclusion

This work has successfully demonstrated that the Bruker 820-MS is an excellent analytical instrument for routine determination of trace elements in high matrix solutions. All MDLs obtained in this study were below the control limits required by Method 6020 when the CRI conditions were used. The MDL study showed good precision for a low concentration solution in the presence of high matrix. Spike recovery values demonstrated accuracy at the lower end of the calibration and illustrated the stability of the Bruker CRI-ICP-MS for environmental standard analysis.

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References

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Keywords

ICP-MS
Collision Reaction Interface
High Matrix
Multiple Condition Sets

Instrumentation & Software

Bruker 820-MS
ICP-MS Expert Software

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