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Attom Note **NA09**

Quantitative Analysis of Portuguese Mineral Water Using the Unique Extended Dynamic Range Detector System

Introduction

Quadrupole ICP-MS is a widely used and well accepted technique for the rapid quantification of a large number of elements in a single sample. However, high resolution ICP-MS (HR-ICP-MS) instruments also have a role to play due to their superior detection limits, better sensitivity and ability to resolve isobaric interferences.

For HR-ICP-MS instruments, it is important to have a detector system with as wide a dynamic range as possible to allow major and ultra trace elements to be analysed without resorting to sample dilution. The Attom from Nu Instruments has a unique, patented detector system with three detector modes, consisting of pulse counting, 'attenuated' pulse counting, and Faraday modes. The detector system is described in more detail in Notes NA07 & NA08. A simplified graph showing the detector stages versus analyte signal intensity is depicted in Figure 1.

The switchover between different detector modes is fast and automatic. Furthermore, the crossover calibration is mass independent and very stable over the course of analytical sessions (NA07). The linearity of the detector system has been described in NA18.

In this note we show quantitative data acquired for three different Portuguese mineral water samples for a range of elements with varying concentrations.

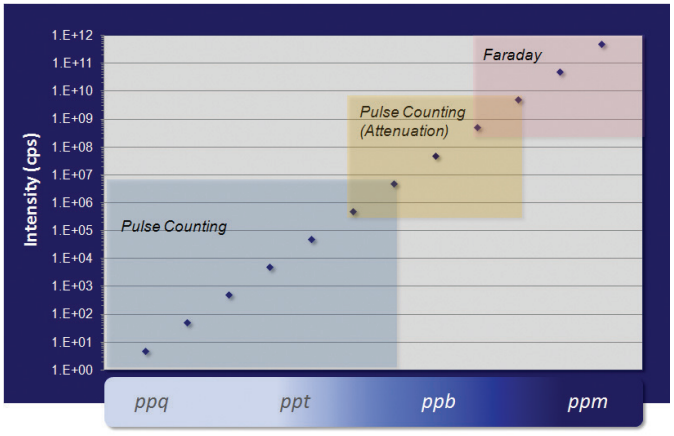

Figure 1: Detector modes for the Attom HR-ICP-MS

Instrumentation

The Nu Attom is a double-focusing, high-resolution magnetic sector mass spectrometer. The instrument is entirely purpose designed and built to provide the best performance and reliability coupled with flexibility and ease-of-use for precise and accurate elemental and isotope ratio analysis. A unique detector system gives the Nu Attom a large dynamic range, and its electrostatic scanning capability has the widest range in its class (40%). Furthermore, the continuously variable high resolution means that sufficient resolution for isobaric separation can be selected with minimum compromise in sensitivity.

Experiment

The first step in this experiment was to generate an external calibration curve for the elements of interest.

Three different multi-elemental standards were used (SPEX CertiPrep Group, USA; 10 mg/L stock concentration, CLMS-1 for rare earth elements (REE), CLMS-2 and CLMS-4 for the rest). A 2 mg/L mix solution of CLMS-2 and CLMS-4 was prepared using ultrapure 2% HNO_3 and then used subsequently to prepare a dilution series of 1, 5, 50, 100 and 500 ng/g. The blanks (one for the standards and one for the samples), standards and samples to be analysed in conjunction with this mix standard series were all spiked with 1 ng/g Tm for internal normalisation purposes.

For the determination of the REE, a separate standard series was prepared in the way described above using the CLMS-1 standard. In this case, all blanks, standards and samples were spiked with 1 ng/g Rh for internal normalisation purposes.

Internal standard normalisation and blank correction were applied prior to calculating the calibration curves. No mathematical corrections for isobaric overlap (e.g. Sn on Cd) or spectral interferences (e.g. BaO on Eu) were made. All calibration curves yielded R^2 >0.999.

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In a second step, three mineral water samples as well as the corresponding 2% HNO_3 blank were kindly provided by R. Santos of the Laboratório Nacional de Energia e Geologia (LNEG, Portugal). The samples were measured as provided by LNEG. Analysis was performed using the standard wet plasma sample introduction system, 100 µl/min uptake rate, peak scanning mode at resolutions of 300, 4,500 and 10,000 RP using 1,000 ms dwell time per isotope.

Discussion

The data was acquired using the full extended dynamic range of the detector system without any user intervention. Raw data from the different detector modes were automatically recalculated using the crossover calibration factors.

The results are reported in Table 1 and consist of 6 repeat analyses of each sample and the blank in each resolution setting. Data are shown along with concentrations previously detected by Q-ICPMS wherever available.

The results show that the HR-ICP-MS data is in good agreement with the quadrupole ICP-MS results. The determination of the REE in the samples proved to be difficult as most of the concentrations are extremely low. Therefore the concentration data for these elements can be used as a guide only.

Conclusion

The Nu Attom is a high resolution ICP-MS that is ideal for the most demanding ICP-MS requirements where sensitivity, precision, and speed of analysis are paramount. The results of this experiment highlight that the instrument is suitable for the quantitative analysis of samples with varying concentrations in a range of materials, in this case mineral water samples.

Acknowledgement

Nu Instruments would like to gratefully acknowledge Rui Santos of the Laboratório Nacional de Energia e Geologia (LNEG, Portugal) for providing the mineral water samples.

	Values in µg/L (ppb)								
	Sample1	RSD%	Known Values S1	Sample 2	RSD%	Known Values S2	Sample 3	RSD%	Known Values S3
Li	841	1.2	872	1.3	3.1	1.4	679	2.0	693
Be	0.11	52.6	0.24	n.d.	<0.04	0.07	32.7	<0.67	146823
B	2491	1.9	2128	3.5	3.2	2.8	165	2.9	156
Al	3.7	3.3	2.2	10.9	4.2	9.8	3.1	4.0	6.1
Sc	2.3	4.4	0.4	2.4	0.9	1.8	1285179	1136599	1554863
Mn	17.0	2.3	14.6	2.6	3.0	2.2	431	1.9	400
Fe(56)	3.2	2.6	<48.8	0.5	6.2	<80.6	6486	2.1	7200
Fe(57)	3.3	10.8	<48.8	0.6	20.7	<80.6	6680	2.3	7200
Co	0.0	28.9	<0.04	0.5	5.3	0.37	0.0	23.3	<0.06
Ni	0.2	12.5	<2.5	2.4	4.4	1.6	2.2	5.5	<1.3
Cu(63)	0.2	16.4	<0.31	1.6	4.0	1.1	5.3	2.4	5
Cu(65)	0.2	12.9	<0.31	1.6	4.7	1.1	5.4	3.4	5
Zn(66)	0.4	10.8	0.38	36.9	2.8	25	11.8	2.2	12.2
Zn(67)	0.4	33.5	0.38	36.9	3.4	25	11.9	5.7	12.2
Ga	0.5	4.9	0.45	0.002	78.5	<0.01	0.02	33.2	<0.18
Ge	20.7	2.1	19.9	0.02	46.6	<0.31	3.8	3.5	3.9
As	11.2	8.6	10	0.6	44.4	<0.56	3.7	10.0	2.7
Se	2.5	107.6	2.9	0.73	78.0	<0.56	3.3	143.3	<3.6
Rb	120	4.1	116	0.6	0.0	0.53	83.6	5.0	77.6
Sr	344	2.3	330	9.8	1.2	11.5	1463	1.0	1326
Y	0.002	24.9	<0.04	0.2	4.0	0.17	0.2	0.9	0.17
Zr	0.02	20.3	<0.65	n.d.	<0.20	1.7	1.1	1.7	
Mo	0.1	63.8	0.24	n.d.	0.01	n.d.	<0.41		
Ag	n.d.	<0.20	n.d.	n.d.	<0.39				
Cd(111)	1.0	4.2	0.93	0.1	3.4	0.08	0.0	768.9	0.03
Cd(114)	1.0	2.2	0.93	0.1	2.3	0.08	0.01	89.6	0.03
Sn	0.003	161.6	<0.24	0.001	44.2	<0.04	0.13	181.8	<0.18
Sb	0.004	38.3	<0.09	0.195	0.8	0.16	0.057	8.9	<0.11
Te	n.d.	<0.19	n.d.	<0.05	n.d.	<0.23			

Table 1: Concentration data in µg/L for the three different mineral water samples. Where available, concentrations derived from quadrupole ICP-MS analyses are shown for comparison (n.d. = not detected).

	Values in µg/L (ppb)								
	Sample1	RSD%	Known Values S1	Sample 2	RSD%	Known Values S2	Sample 3	RSD%	Known Values S3
Cs	198.4	2.1	196	0.04	4.8	0.07	131	2.0	127
Ba	n.d.	3.1	0.7	6.0	1.9	284	3.1	286	
La	0.09	6.7	0.032	1.9	0.01	5.3			
Ce	0.003	24.9	0.002	5.5	0.012	5.7			
Pr	n.d.	n.d.	n.d.						
Nd	n.d.	n.d.	n.d.						
Sm	n.d.	0.016	7.1	0.002	24.4				
Eu(151)	n.d.	0.003	18.0	0.007	8.5				
Eu(153)	n.d.	0.003	12.3	0.014	3.8				
Gd	n.d.	0.021	9.2	0.005	21.0				
Tb	n.d.	0.001	13.5	n.d.					
Dy	n.d.	0.005	16.8	0.0005	172.7				
Ho	0.0003	168.0	0.004	8.0	0.0033	6.1			
Er	n.d.	0.011	4.2	0.0120	5.2				
Tm	n.d.	0.001	6.3	0.0014	10.1				
Yb	n.d.	0.007	4.5	0.0126	7.5				
Lu	n.d.	0.001	17.8	0.0019	12.7				
Ta	0.028	21.1	<0.04	n.d.	<0.01	n.d.	<0.04		
W	370	0.5	361	n.d.	<0.22	1.9	2.8	2.1	
Hg	n.d.	0.46	n.d.	<0.13	n.d.	<0.64			
Tl	n.d.	<0.04	n.d.	<0.01	n.d.	<0.17			
Pb	0.05	40.6	<0.11	0.001	100.7	<0.08	0.097	4.5	0.08
Bi	n.d.	<0.06	n.d.	<0.02	n.d.	<0.05			
Th	0.003	32.3	n.d.	0.0009	22.0				
U	0.01	6.2	<0.07	0.004	7.1	<0.07	0.0193	6.1	0.02

Table 1 (continued): Concentration data in µg/L for the three different mineral water samples. Where available, concentrations derived from quadrupole ICP-MS analyses are shown for comparison (n.d. = not detected).