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Improving accuracy and uncertainty in inductively coupled plasma optical emission spectrometry (ICP-OES) when a sequential spectrometer is used

Ramiro PÉREZ-ZAMBRA^a, Daniel CAIMI^a, Pablo CONSTANTINO^b and Elizabeth FERREIRA^a

^aDepartamento de Metrología Química, Laboratorio Tecnológico del Uruguay, ^bDepartamento de Metrología Física, Laboratorio Tecnológico del Uruguay; rperez@latu.org.uy

INTRODUCTION

ICP-OES is a widely applied technique for the certification of monoelemental solutions along with gravimetry and titration. Therefore, it is essential to ensure traceability of measurements to the International System of Units (SI). In particular, an ICP-OES method developed by the National Institute of Standards and Technology (NIST)[1,2] is used by National Metrology Institutes and reference material producers due to its high accuracy and low uncertainty, typically in the order of 0,1% (relative expanded uncertainty). This method requires the determination of the instrumental response drift pattern and its modeling by an equation. However, in sequential ICP-OES instruments this approach cannot be applied because analyte and internal standard signals are not measured simultaneously and consequently a drift pattern cannot be determined.

OBJETIVE

The aim of this work was to develop a highly accurate and low uncertainty method for the determination of elements in monoelemental solutions to be used as certified reference materials (CRM) by sequential ICP-OES.



EXPERIMENTAL

Instrumental operating conditions		
	CADMIUM	SODIUM
ICP-OES	PerkinElmer Optima 7000 DV	
Nebulizer	Concentric	
Spray chamber	Cyclonic	
Injector	Alumina, 2 mm	
Analyte wavelength (nm)	226,502	589,592
Internal standard wavelength (nm)	Sc 361,383	In 325,609
RF power (W)	1300	1450
Plasma gas flow (l min ⁻¹)		15
Auxiliary gas flow (l min ⁻¹)		0,2
Nebulizer gas flow (l min ⁻¹)	0,8	0,7
Sample flow rate (ml min ⁻¹)		1,5
Plasma viewing	Axial	Radial
Processing mode	Peak area	
Background correction	2-point, manual	
Integration time (s)	0,05	
Read time (s)	3	
Number of replicates	5	

Proposed method

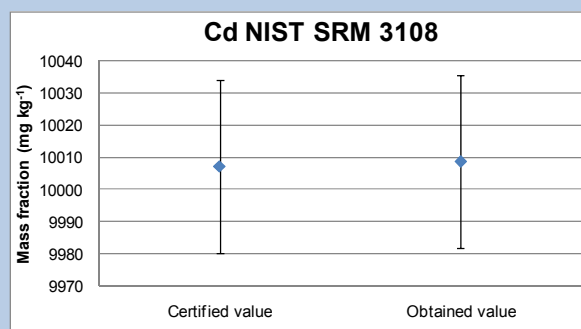
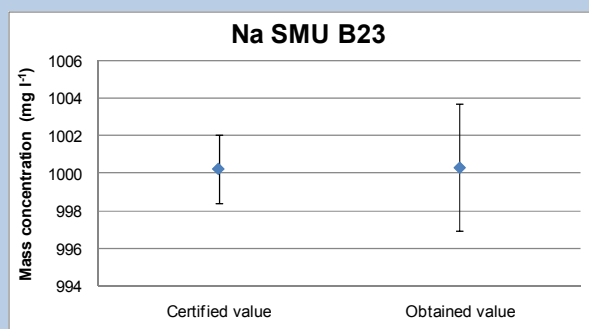
Firstly, the instrumental response factor for analyte and internal standard were determined. Both sample and calibrant were diluted with the internal standard solution six times to yield a signal ratio close to 1. Each replicate had approximately the same concentration of analyte and internal standard. After a comprehensive study, the instrumental parameters were optimized. Samples were measured between two calibrants which were randomly selected and quantified using the average slope of each calibrant. This approach presents the benefits of both exact-matching and bracketing calibration given that the most accurate estimation of the response factor at that level is achieved and instrumental drift contributions are minimized.

A sodium CRM from NIST 3152a and a cadmium CRM from SMU (Slovak Metrology Institute, Slovakia) B08 were used as calibrants. To study the method accuracy a sodium monoelemental solution CRM from SMU and a cadmium monoelemental solution CRM from NIST were used.

Uncertainty estimation

A probability density function was assigned to each parameter of the equation, enabling an uncertainty estimation through Monte Carlo[3] method using MCM Alchimia[4] software.

RESULTS



For both analytes an excellent level of agreement was achieved. Having used calibrants with a $U_{rel}=0,20\%$, the estimated expanded relative uncertainty for sodium was 0,34% and for cadmium it was 0,27%.

CONCLUSIONS

Due to its high accuracy and low uncertainty, the proposed method is suitable for conducting homogeneity and stability studies as well as assign values for sodium, cadmium monoelemental solution CRMs. This method will be further tested with other analytes.

REFERENCES

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