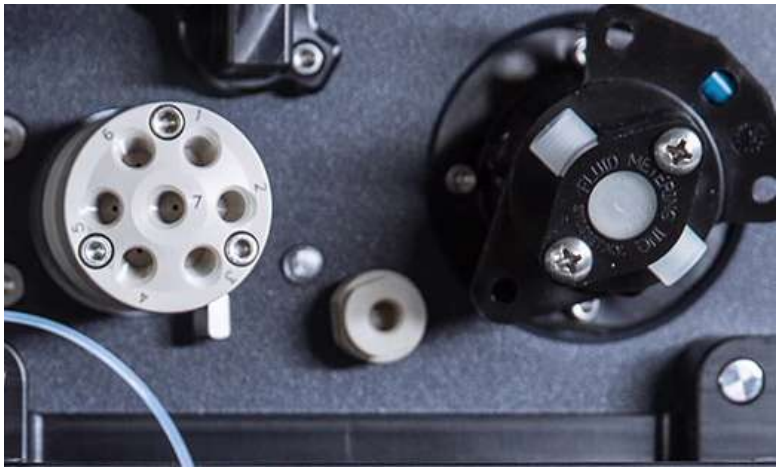
Ingeniatrics
Nebulizers and Nozzles

Application Note

Advantages in the use of the OneNeb® nebulizer with Agilent AVS valve systems and SPS4 autosampler. ICP-OES



21/11/2022

Authors

Miguel Ángel García Sevillano, PhD.

MG Analytical Chemistry
Instruments Consulting

1. Introduction

It is well known that the AVS06-AVS07 valve reduces considerably analysis times (~ 1 min/sample) and cost, having a favorable impact on instrument and accessories life, but also on plasma argon consumption. The AVS (Advanced Valves System) operates by internally switching vias allowing a sequential loading of a sample loop between the peristaltic pump and the nebulizer on the ICP-OES instrument models 5100/5110/5800/5900 (Agilent Technologies).

The high-efficiency OneNeb® universal nebulizer (Part No. G8010-60293, Agilent Technologies) allows accurate, reproducible (< 1% RSD) quantitation and high signal stability over long run times or batches of numerous samples and that without the need of an internal standard or a humidifier.

This nebulizer has been designed for all types of matrices, offering exceptional tolerance to high levels of dissolved solids (TDS) and is suitable for all types of solutions, including aggressive acids, hydrofluoric acid digestions and organic solvents.

CMG
CONSULTOR EN QUÍMICA ANALÍTICA INSTRUMENTAL

2. Experimental

Reagents and solutions

Aqueous calibration standards of 1, 5, 10, 50, 100 and 250 µg g^{-1} were prepared by appropriate dilution of a mono-elemental stock solution of 10000 mg L^{-1} of each Ag, As, Cu, Pb, Fe and Zn (ICP CetriPUR, Merck, Darmstadt, Germany) being these the analytes of interest to be quantified in the present application. Dilutions were performed using 18 MΩ cm deionized water. All aqueous solutions are acidified by adding up to 5% nitric acid and 15% high purity 30% hydrochloric acid (Merck, Darmstadt, Germany). An aqueous calibration blank was also prepared containing HNO3 and HCl in the same proportions, like the matrix samples, once mineralized.

Instrumentation

All measurements were carried out using a 5110 (Agilent Technologies) inductively coupled plasma-optical emission spectrometer (ICP-OES), equipped with an AVS06 switching valve (Agilent Technologies, Part No.: G8494-60000) and a SPS4 autosampler (Agilent Technologies) with 0.5 mm ID sampling probe (Agilent Technologies, Part No.: G8410-80101). The length of the capillary between the sampler probe and AVS valve was 1.0 m (0.5 mm ID), and the sample loop volume was 2.0 mL (0.5 mm ID). 5110 ICP-OES offers detection of wavelengths in both axial and radial acquisition modes, allowing accurate quantification of major and minor elements in all types of matrices and in a single run for a wide linear range of quantification.

To adapt OneNeb® nebulizer to the AVS06 valve, it is necessary to cut the inlet capillary to 7 cm and seal it with a 1/8 mm OD fitting as shown in Figure 1 and Figure 2.

Figure 1. Adaptation of OneNeb to the Agilent AVS06 valve.

Figure 2. OneNeb connected to the Agilent AVS06 valve.

For the described instrumental configuration, the optimized conditions are shown at Table I.

ICP-OES		
RF Power (kW)	1.5	
Plasma gas flow (L min^{-1})	15.0	
Auxiliary gas flow (L min^{-1})	1.0	
Nebulization gas flow (L min^{-1})	0.60 (150-200 kPa)	
Peristaltic pump rate (rpm)	10	
Replicates	3	
Nebulizer	OneNeb®	
Spray chamber	double pass	
AVS 06		
Pump rate Uptake (mL min^{-1})	35.0	
Pump rate Injection (mL min^{-1})	5.0	
Valve uptake delay (s)	8.0	
Bubble injection times (s)	Not connected	
Preemptive rinse time (s)	2.0	
Rinse time (s) (rate 30 rpm)	5.0	
Acquisition mode	*Axial*	*Radial*
Analysis time (s)	3.0	3.0
Stabilization time (s)	5.0	3.0
Wavelengths monitored (nm)	As 188.980 Ag 328.068	Cu 324.754 Fe 261.187 Pb 220.354 Zn 206.200

Table I. Operational conditions using ICP-OES 5110 (AVS06), Agilent Technologies.

3. Results and discussion

Sensitivity and signal stability

The OneNeb® nebulizer uses Flow Blurring nebulization technology instead of the traditional Venturi effect. This allows the generation of a very fine droplet aerosol with a narrow size distribution (most droplets are smaller than 10 µm), which improves efficiency over a wide range of nebulization gas flow rates, especially 0.60-0.75 L min^{-1} (150-250 kPa nebulization pressure).

The optimal aerosol generated by the OneNeb® nebulizer is also more efficiently desolvated and excited in plasma, helping to improve precision values, typically less than 1% RSD between replicates of the same run analysis, even at low sample flow rates, which also explains why it is much more sensitive than conventional nebulizers.

In order to find the best nebulization conditions for optimum sensitivity, a nebulization flow curve was made using a solution of 5 µg kg^{-1} of Mn, monitoring the wavelength 293.305 nm, and obtaining the best performance for 0.6 L min^{-1} of nebulization gas, at nebulization pressures of 150±10 kPa.

For signal stability and plasma drift assessment, without internal standard for the analytes of interest, a monitoring standard solution containing 25 µg g^{-1} of each element was prepared.

This solution was analyzed once every 5 mineralized CRM samples, in order to evaluate the stability of the signal, obtaining acceptable results for all the elements in a confidence interval of 97-103%.

Precision and reproducibility evaluation

Precision values were evaluated using geological matrix certified reference materials (CRM) after aqua regia mineralization using a microwave-assisted oven. Precision is expressed as the relative standard deviation percentage (RSD%).

Table II summarizes the certified and the experimental registered values for each monitored wavelength, as well as the RSD obtained for 10 replicates of each of the CRMs used in the experimental development of this application note.

Digested with aqua regia, CRM samples and the monitoring controls were analyzed in the same analytical batch of samples.

A total of 60 determinations were analyzed in 75 minutes, approximately 1 minute of analysis time per sample.

Certified Reference Materials (CRM)	Emission lines		Certified values (Aqua regia digestion)	Experimental values	
			Concentration	Concentration	RSD (%) (10 replicates)
OREAS 621	Ag 328.068 nm	µg kg^{-1}	68	69	1,37
	As 188.980 nm	µg kg^{-1}	75	74	1,45
	Cu 324.754 nm	mg kg^{-1}	0,366	0,362	1,01
	Fe 261.187 nm	mg kg^{-1}	3,43	3,41	1,82
	Pb 220.354 nm	mg kg^{-1}	1,36	1,37	1,50
	Zn 206.200 nm	mg kg^{-1}	5,17	5,14	1,61
OREAS 622	Ag 328.068 nm	µg kg^{-1}	101	99	1,25
	As 188.980 nm	µg kg^{-1}	106	106	1,30
	Cu 324.754 nm	mg kg^{-1}	0,484	0,482	1,57
	Fe 261.187 nm	mg kg^{-1}	4,05	4,07	1,61
	Pb 220.354 nm	mg kg^{-1}	2,19	2,20	1,36
	Zn 206.200 nm	mg kg^{-1}	10,01	10,09	2,50
OREAS 623	Ag 328.068 nm	µg kg^{-1}	20,4	20,1	1,48
	As 188.980 nm	µg kg^{-1}	76	77	1,56
	Cu 324.754 nm	mg kg^{-1}	1,72	1,71	1,49
	Fe 261.187 nm	mg kg^{-1}	13,01	13,12	2,61
	Pb 220.354 nm	mg kg^{-1}	0,252	0,250	1,45
	Zn 206.200 nm	mg kg^{-1}	1,01	1,02	1,26
OREAS 624	Ag 328.068 nm	µg kg^{-1}	45	46	1,30
	As 188.980 nm	µg kg^{-1}	108	107	0,62
	Cu 324.754 nm	mg kg^{-1}	3,09	3,10	1,51
	Fe 261.187 nm	mg kg^{-1}	16,14	16,21	2,57
	Pb 220.354 nm	mg kg^{-1}	0,629	0,623	1,74
	Zn 206.200 nm	mg kg^{-1}	2,40	2,37	1,59
OREAS 134b	Ag 328.068 nm	µg kg^{-1}	204	202	1,57
	As 188.980 nm	µg kg^{-1}	221	219	1,63
	Cu 324.754 nm	µg kg^{-1}	1363	1368	1,35
	Fe 261.187 nm	mg kg^{-1}	12,25	12,18	1,85
	Pb 220.354 nm	mg kg^{-1}	13,31	13,36	2,38
	Zn 206.200 nm	mg kg^{-1}	17,70	17,67	2,15

Table II. Experimental and certified values for each monitored wavelength, as well as the RSD obtained for 10 replicates of the different CRM used in this application note.

4. Conclusions

When used together with the Agilent 5110 ICP-OES and AVS 06 valve system, the OneNeb® nebulizer provides excellent values of sensitivity, stability and precision under the conditions described in this application note, offering an amazing testing speed and a great reproducibility for analytical batches of 60 samples without the need of an online internal standard.

The presented configuration also allows the analysis of majority and minority elements in the same analytical batch, just taking approximately 1 minute per sample, and maximizing, therefore, the advantage of axial and radial acquisition modes provided by the Agilent ICP-OES 5110 instrument.

The conditions and configuration optimized in this study can also be extrapolated to the ICP-OES 5100, 5800 and 5900 models from Agilent Technologies.

Ingeniatrics
Nebulizers and Nozzles

P.I. Parque Plata
C/ Camino Mozárabe, 41
41900 Camas, Seville
(Spain)

(+34) 954 08 1214
info@ingeniatrics.com

www.Ingeniatrics.com