Ultra-high speed analysis of soil extracts using an Advanced Valve System installed on an Agilent 5110 SVDV ICP-OES

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Introduction

Laboratories are constantly looking to improve productivity and reduce operating costs by increasing sample throughput and minimizing overheads. Agricultural labs typically deal with high quantities of samples, however, higher analysis speeds normally create some sort of analytical compromise, such as reduced precision.

The Agilent 5110's Synchronous Vertical Dual View (SVDV) capability and Vista chip II detector covering all wavelengths in a single reading, combined with the fully integrated Advanced Valve System (AVS), allows the analysis of samples at ultra-high speeds, without compromising performance. This high speed sample throughput equates to a low cost-per-analysis, as shorter analysis times per sample results in less argon usage.

The Agilent Advanced Valve System (AVS 6/7) provides:

- **Ease-of-use:** the AVS is easy to setup and use as it is fully integrated within the ICP-OES hardware and controlled through the ICP Expert software. The ICP Expert software incorporates an AVS parameter calculator to assist with setup and method development. The AVS is designed for simple assembly and disassembly, facilitating simple routine maintenance, maximizing instrument up time.

- **Uncompromising performance:** Controlled bubble injection reduces sample uptake, stabilization times, and rinse delays to deliver the highest analytical precision.

- **Speed:** The robust high speed positive displacement pump also decreases uptake time, ensuring the sample loop can be filled quickly and effectively.
A pre-emptive rinse means the autosampler probe moves while the loop is still filling. This starts autosampler probe rinse and valve rinse before the sample is even injected and decreases the total run time.

This application note describes the ultra-high speed analysis of micronutrients Cu, Fe, Mn, Zn, Co, Ni and heavy metals Cd and Pb in a DTPA extracted soil sample using the Agilent 5110 Synchronous Vertical Dual View (SVDV) ICP-OES fitted with an integrated Advanced Valve System (AVS 6) six port switching valve.

The Agilent 5110 SVDV ICP-OES fitted with Advanced Valve System (AVS) six port valve

Experimental

Instrumentation

All measurements were performed using an Agilent 5110 SVDV ICP-OES fitted with an AVS 6 port valve and configured with an SPS 4 autosampler. The sample introduction system consisted of a SeaSpray nebulizer, double-pass cyclonic spray chamber and a 1.8 mm i.d injector torch. Tables 1 and 2 list the operating conditions used for the ICP-OES and the AVS 6.

Table 1. Agilent 5110 RV ICP-OES operating parameters

Parameter	Setting
Read time (s)	1
Replicates	1
Sample uptake delay (s)	0
Stabilization time (s)	2
Rinse time (s)	0
Pump Speed (rpm)	25
RF power (kW)	1.20
Viewing mode	SVDV
Viewing height (mm)	8
Aux flow (L/min)	1.0
Plasma flow (L/min)	12.0
Nebulizer flow (L/min)	0.70
SPS 4 autosampler rinse pump control speed	Fast

Table 2. Agilent 6 port Advanced Valve System (AVS 6) settings

Parameter	Setting
Sample loop volume (mL)	0.25
Pump rate: Valve uptake (mL/min)	40
Pump rate: Inject (mL/min)	28
Valve uptake delay (s)	2.6
Bubble injection time (s)	2.6
Pre-emptive rinse time (s)	1.0

Standard and sample preparation

Soil samples were supplied dried and ground.
DTPA extraction solution: the extraction solution comprised 0.005 M diethylenetriaminepentaacetic acid (DTPA), 0.01 M calcium chloride dihydrate ($CaCl_2.2H_2O$) and 0.1 M triethanolamine (TEA). 1.97 g of DTPA, 1.47 g $CaCl_2.2H_2O$ and 14.92 g TEA were dissolved separately in deionized water and then combined. The pH was adjusted to 7.3 using concentrated HCl and the volume made up to 1 L with distilled water.

Sample extraction: 10 g of soil was weighed and 20 mL of DTPA extraction solution added. After shaking for 120 minutes, the sample was filtered through with 110 mm diameter filter paper.

Multi-element calibration standards: 0.01, 0.05, 0.1, 0.5 and 1.0 µg/mL of Cd; 0.05, 0.25, 0.5, 2.5 and 5 µg/mL of Co and Ni; 0.1, 0.5, 1.0, 5.0 and 10.0 µg/mL of Cu, Zn and Pb; 0.5, 2.5, 5.0, 25.0 and 50.0 µg/mL of Mn; 1.0, 5.0, 10.0, 50.0 and 100.0 µg/mL of Fe. All calibration blanks and standards were prepared in the DTPA extraction solution.

Results and Discussion

Long Term Stability

The stability of the Agilent 5110 SVDV ICP-OES with integrated AVS 6 and SPS 4 autosampler was evaluated by analyzing a DTPA soil extract solution 120 times.
Good precision was obtained, with all elements achieving < 3.4 %RSD over the duration of the run (Table 3 and Figure 1).

Results and Discussion

Table 3. Precision data for the repeated (n=120) measurement of 8 elements in a soil extract

Element & line	Mean concentration (mg/L)	%RSD
Cd 214.439 nm	0.021	3.06
Co 228.615 nm	0.135	2.35
Cu 324.754 nm	1.29	2.27
Fe 234.350 nm	18.6	1.70
Mn 293.305 nm	1.42	2.03
Ni 231.604 nm	0.175	3.36
Pb 220.353 nm	0.811	3.06
Zn 213.857 nm	0.254	1.94

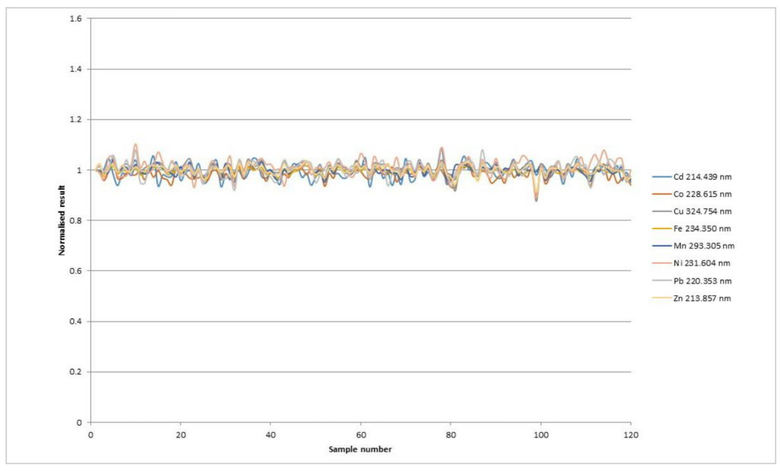
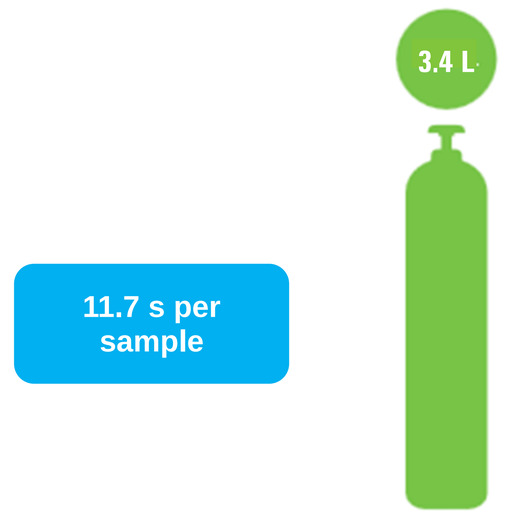


Figure 1. Stability plot from the repeated (n=120) measurement of 8 elements in a DTPA soil extract using a 5110 SVDV ICP-OES with AVS 6

Sample time and argon usage

The 5110 with AVS 6 analyzed 120 samples and 6 standards in under 25 minutes.
This equates to an average sample-to-sample run time of 11.7 seconds and an Ar consumption of only 3.4 L.

Furthermore, such efficient analysis of complex matrices extends the life of consumable items, further reducing operating costs.



Conclusions

Agilent's 5110 SVDV ICP-OES with its vertical torch not only provides the robustness required for the determination of DTPA extracted soil samples over long sampling periods but delivers exceptionally fast analysis times, without any compromise in analytical performance.

When fitted with a fully integrated AVS and the use of an SPS 4 autosampler the 5110 was able to achieve:

- Very fast analysis times with an average of 11.7 seconds per sample.

- Excellent precision with < 3.4 % RSD for all elements over the duration of a 120 sample analytical run.

- Very low Ar consumption of < 3.4 L/sample.

By significantly improving sample analysis times, the 5110 SVDV ICP-OES fitted with AVS and SPS 4 autosampler cuts the cost-per-analysis which is an important consideration of high throughput labs.