

Dramatically improved sample throughput using a new sample introduction technique with ICP-OES

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Abstract

Introduction

Improving productivity is a key issue for many laboratories that require fast, accurate and precise determinations of major, minor and trace elements in a variety of sample matrices. Achieving reliable improvements in sample throughput with ICP-OES can result in greater sample capacity, improved laboratory responsiveness, or provide cost savings through reductions in argon consumption, labor, electricity and reagent consumption.

We will evaluate a new sample introduction accessory for ICP-OES that uses flow-injection technology which is able to provide up to twice the sample throughput without compromising analytical accuracy or reproducibility. By eliminating the “dead time” in the sample introduction stages such as sample uptake, stabilization time and washout, it is possible to make significant gains in productivity without impinging on the actual measurement time.

In this work we will validate the system performance using a soil and plant tissue reference materials. This analysis is typical of those done in food and agriculture laboratories around the world.

This work describes the coupling of the SVS2, a new and innovative sample introduction system, with an Agilent 720 Series Inductively Coupled Plasma - Optical Emission Spectrometer (ICP-OES) to improve sample throughput. The SVS2 is comprised of a triple stacked 4-way diagonal flow switching valve, sample loop and a high speed, positive displacement pump. The SVS2 uses flow injection technology to virtually eliminate delays associated with sample uptake and plasma stabilization.

A typical sample analysis cycle using and ICP-OES involves sample uptake at high pump rates from the autosampler to the plasma, followed by a stabilization delay to allow the plasma to come back into equilibrium, followed by a measurement of the sample before a rinse of the sample from the system. A high-throughput contract lab measuring large numbers of samples on a daily basis using a conventional sample introduction system would typically measure a solution about 1 minute. This equates to approximately 1440 solutions measured per day.

This SVS2 improves the efficiency of sample introduction by greatly reducing sample uptake and washout times. This can result in more than a doubling of the sample throughput, and significant reductions in operating costs.

As a flow of solution to the plasma is constantly maintained, there is an improvement in plasma stability and reduction in stabilization times.

Experimental

Instrumentation

An Agilent 720 Series simultaneous ICP-OES with axially viewed plasma and SPS3 autosampler was used for this work.

The Agilent 720 features a custom designed CCD detector, which provides true simultaneous measurement and full wavelength coverage from 167 to 785 nm. The patented CCD detector contains continuous angled arrays that are matched exactly to the two-dimensional image from the echelle optics. The thermally stabilized optical system contains no moving parts, ensuring excellent long-term stability.

The SVS2 has two valve positions controlled in software. The first position (Figure 1) allows the sample to be quickly loaded into a sample loop using a positive displacement pump operating at up to 500rpm. The size of the sample loop is dependant on the method, although a 0.5mL can be filled in under 5 seconds. The sample is loaded and ready to be aspirated into the plasma for measurement.

When all of the sample is loaded in the injection loop, the controlling software triggers the valves to switch and inject the sample into the ICP-OES (figure 2.). In a typical analysis without the SVS2, sample is fast-pumped into the plasma destabilizing it, which requires a stabilization time of 10 to 15 seconds to settle prior to measurement. With the SVS2, solution is never directly injected into the plasma at high pump speeds ensuring much shorter stabilization delays can be used. In fact, an uptake delay isn’t required at all and a stabilization of <10s is sufficient to load the sample loop, inject the sample into the plasma to attain a stable signal. A system without SVS2 would typically require 25 seconds to perform the same function.

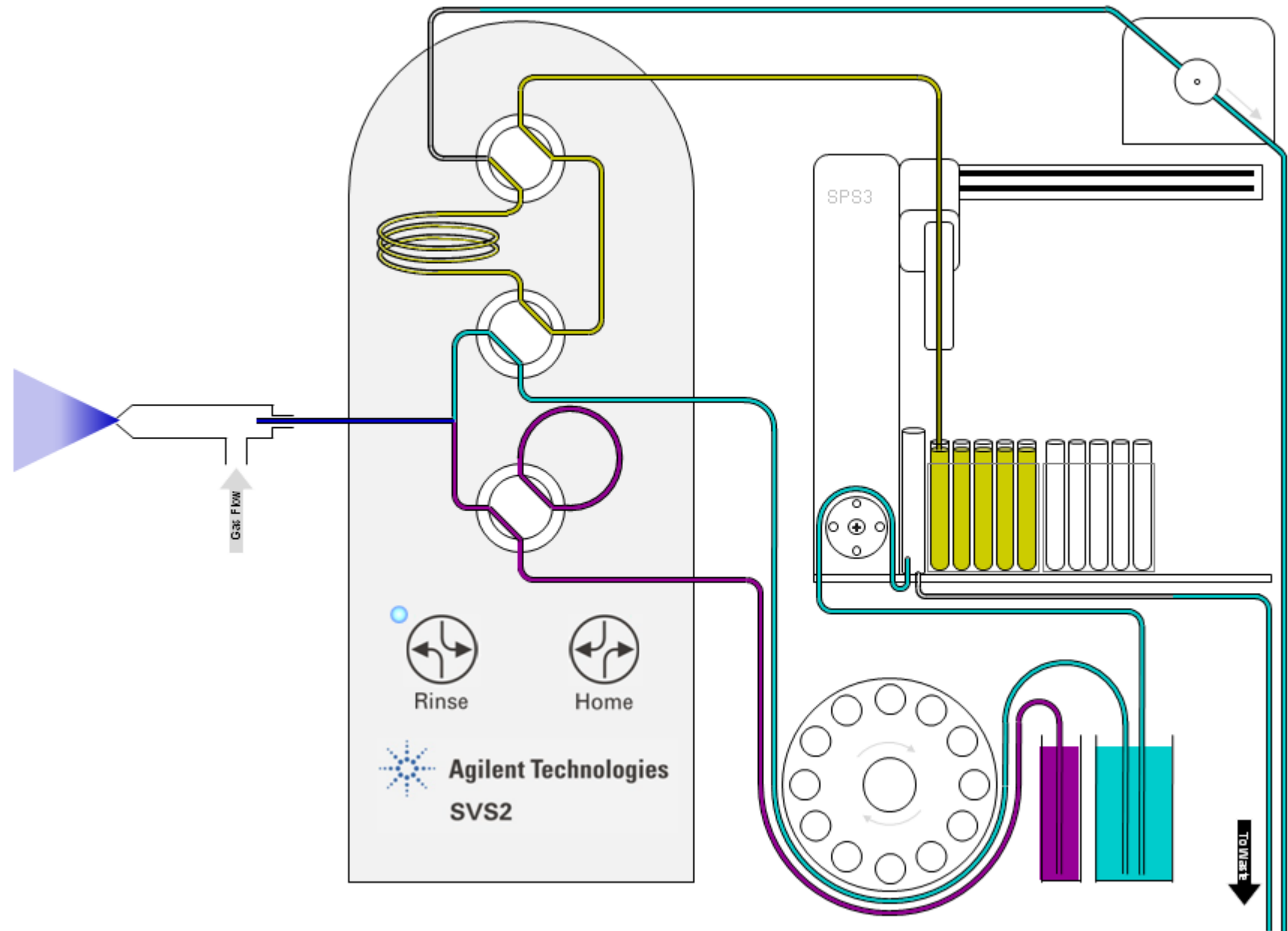


Figure 1 SVS2 in sample load position

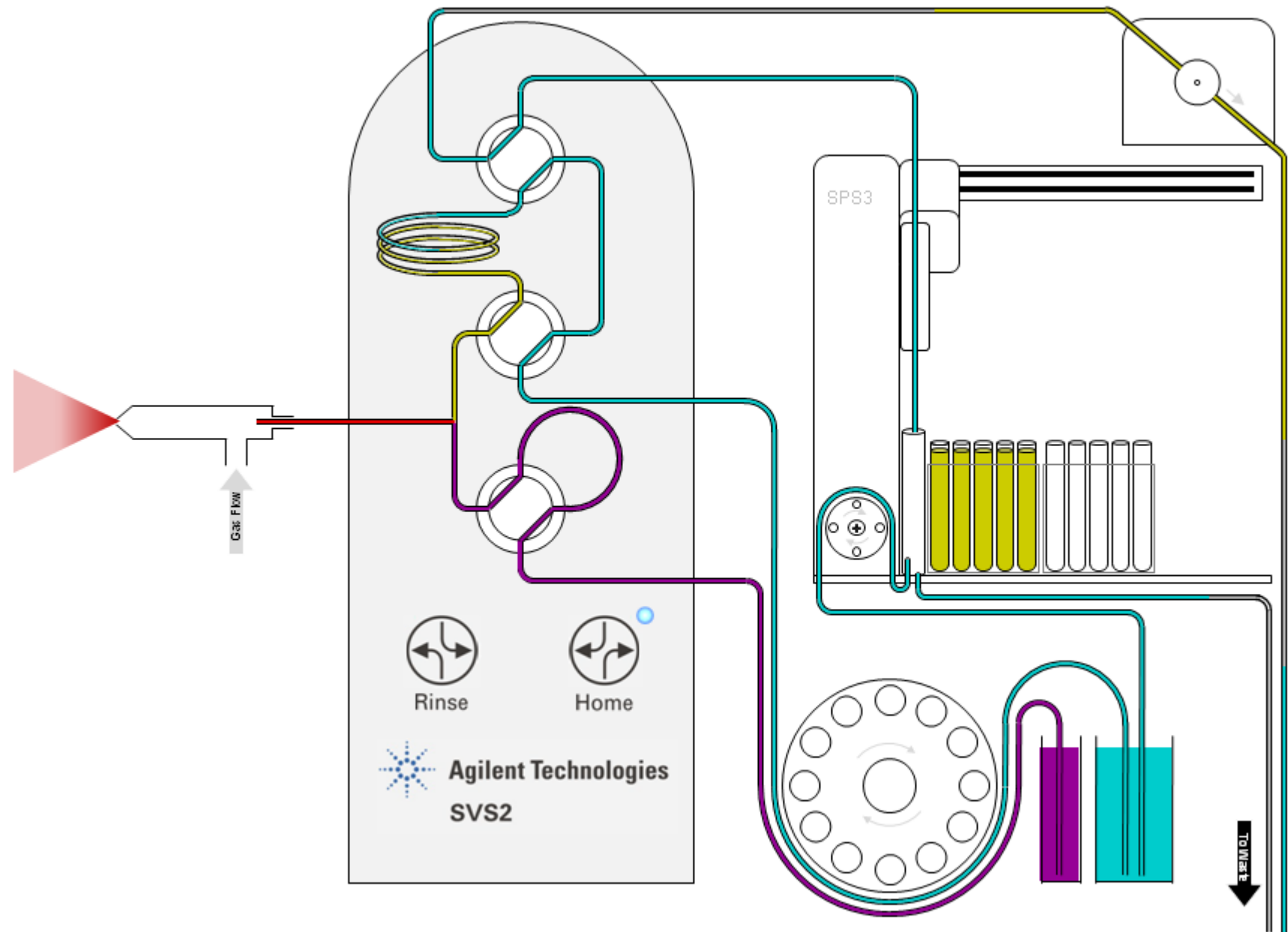


Figure 2 SVS2 in sample inject position

During the measurement phase the entire system is continuously flushed with rinse solution. Again, a conventional system without SVS2 would require a rinse time of at least 20 seconds to washout the system sufficiently to reduce the analyte signal to at least 4 orders of magnitude. With SVS2, the same level of washout is achievable without a rinse time saving a further 20 seconds.

A CEM Mars 5 closed-vessel microwave-assisted acid digestion system was used to extract the elements from the soil and leaf samples following USEPA method 3051A guidelines³. This method is designed to mimic extraction using conventional heating with nitric acid (HNO₃) and hydrochloric acid (HCl) and does not accomplish total decomposition and sample matrix compounds such as quartz, silicates, titanium dioxide, alumina and other oxides are not easily dissolved. Therefore, the extracted analyte concentrations may not reflect the total content in the sample.

Internal standardization and ionization buffering was used throughout the analysis. A solution of 2 mg/L Yttrium (reference element) and 1% CsNO₃ (ionization buffer) in a 5% HNO₃ sample matched matrix was added online to the sample stream, using the third channel of the peristaltic pump, keeping sample preparation to a minimum.

Operating Parameters

- 1.3 kW RF Power
- 15 L/min Plasma gas flow
- 1.5 L/min Auxiliary gas flow
- Spray chamber type single pass glass cyclonic
- Torch Standard one-piece quartz torch
- Nebulizer type SeaSpray Concentric Glass Nebulizer
- Nebulizer flow = 0.7 L/min

Pump tubing

- Rinse/Instrument pump: white/white (1.02 mm ID)
- Waste: blue-blue (1.65 mm ID)
- Buffer/Internal Standard: black/black (0.76 mm ID)
- Pump speed = 12 rpm
- Total sample usage = 1 mL
- Replicate read time = 5 s
- Number of replicates = 2
- Sample delay time = 0 s
- Stabilization time = 7 s
- Rinse time 0 s
- Fast pump Off
- Fitted Background correction

SVS2 operating parameters

- 6 s Loop uptake delay
- 500 rpm uptake pump refill speed
- 100 rpm uptake pump inject speed
- 5 s Time in Sample
- 5 s Bubble Inject Time

Results and Discussion

Table 1 Results for NIST SRM 2710 Montana soil digestion

Element	Unit	Specified Mean	Specified Range	Measured	Recovery
As 188.980	mg/kg	590	490-600	532 ± 30	90%
Ba 455.403	mg/kg	360	300-400	311.5 ± 16.1	87%
Co 228.615	mg/kg	8.2	6.3-12	9.66 ± 0.79	118%
Cr 267.716	mg/kg	19	15-23	18.7 ± 1.5	99%
Cu 324.754	mg/kg	2700	2400-3400	2484 ± 109	92%
Mn 257.610	mg/kg	7700	6200-9000	6845 ± 404	89%
Ni 231.604	mg/kg	10.1	8.8-15	10.0 ± 1.2	99%
Pb 220.353	mg/kg	5100	4300-7000	4523 ± 302	89%
Zn 206.200	mg/kg	5900	5200-6900	5382 ± 378	91%
Al 396.125	Wt %	1.8	1.2-2.6	1.87 ± 0.18	104%
Ca 315.887	Wt %	0.41	0.38-0.48	0.380 ± 0.013	93%
Fe 234.350	Wt %	2.7	2.2-3.2	3.14 ± 0.34	117%
K 766.491	Wt %	0.45	0.37-0.50	0.502 ± 0.016	112%
Mg 285.213	Wt %	0.57	0.43-0.60	0.503 ± 0.020	88%
Na 588.995	Wt %	0.054	0.049-0.062	0.069 ± 0.003	128%

Table 2 Results for the NIST SRM 1571 orchard leaves

Element	Unit	Specified Mean	Specified Range	Measured	Recovery
As 188.980	mg/kg	590	490-600	532 ± 30	90%
Co 228.615	mg/kg	8.2	6.3-12	9.66 ± 0.79	118%
Cr 267.716	mg/kg	19	15-23	18.7 ± 1.5	99%
Cu 324.754	mg/kg	12	11-13	12.0 ± 0.7	100%
Fe 234.350	mg/kg	-	-	285.4 ± 16.7	-
Mn 257.610	mg/kg	91	87-95	87 ± 6	95%
Na 588.995	mg/kg	82	76-88	64.9 ± 2.9	79%
Ni 231.604	mg/kg	10.1	8.8-15	10.0 ± 1.2	99%
Pb 220.353	mg/kg	45	42-48	42.1 ± 1.8	94%
Zn 206.200	mg/kg	25	22-28	27.5 ± 1.6	110%
Al 396.125	Wt %	NA	-	275 ± 15	-
Ca 315.887	Wt %	2.09	2.06-2.12	2.28±0.11	109%
K 766.491	Wt %	1.47	1.44-1.50	1.453 ± 0.096	99%
Mg 285.213	Wt %	0.62	0.60-0.64	0.59 ± 0.02	95%

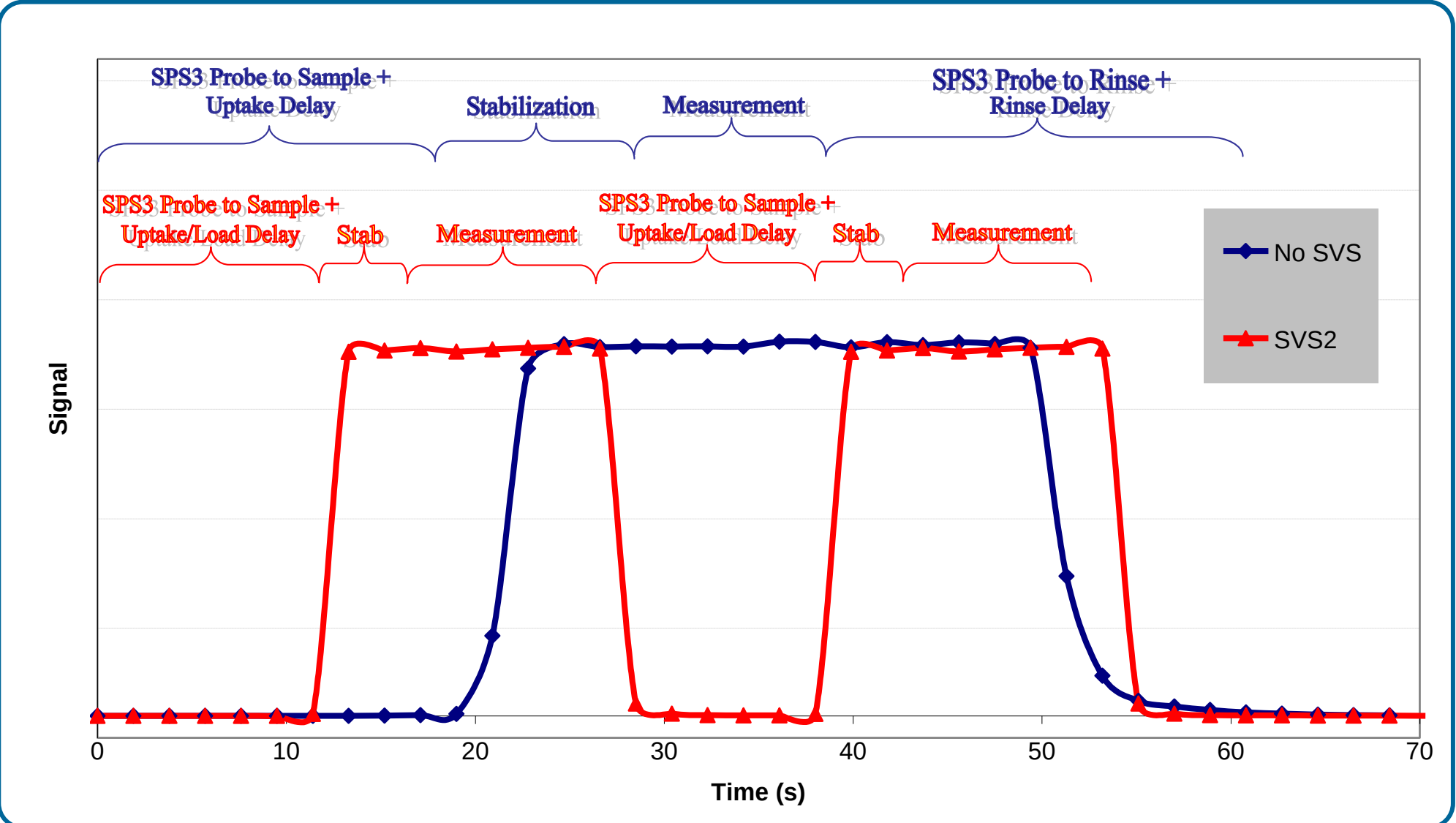


Figure 3 1000 ppm Mn washout with and without SVS2

Conclusion

Using the SVS2 with an Agilent 720 Series axially viewed ICP-OES the sample to sample cycle time is reduced from 60 seconds^{1,2} to less than 30 seconds while maintaining analytical accuracy and reducing cost of argon gas., standards and other reagents and longer lifetime of torches and pump tubing.

Washout between samples resulted in about 4 orders of magnitude in signal reduction.

Analytical stability was excellent.

With the swept volume of the SVS2 components minimized, sample throughput has been optimized and carryover virtually eliminated.

As the sample does not make contact with peristaltic pump tubing prior to being aspirated into the plasma, the inert sample path results in reduced sample carry-over.

References

1. Calderon. V., Varian Application Note 35. Rapid measurement of major, minor and trace levels in soils using the Varian 730-ES
2. Ryan. A., Varian Application Note 33. Rapid measurement of major, minor and trace elements in plant and food material using the Varian 730-ES
3. USPEPA Methods 3051A “Microwave assisted acid digestion of sediments, sludge and oils” Revision 1, January 1998