

## Addressing Today's Sample Throughput Limitations for ICP-AES Soils Analysis with Modern Sample Introduction Technology

### INTRODUCTION

Soil samples are routinely analyzed by atomic spectroscopic techniques such as Inductively Coupled Plasma – Atomic Emission Spectrometry (ICP-AES) for trace and minor elements, as agricultural soil elemental makeup can have a direct impact on crop yields and quality. However, today's soils analysis has become more challenging than ever. With few application-specific solutions available to laboratory management, finding off-the-shelf solutions to common problems can be a difficult, if not impossible task (*many soil lab tools/devices are devised and hand-made by the lab employees themselves*). Soils laboratory management has historically been "on their own" to find solutions to their everyday problems.

### PROBLEM

Extreme seasonal fluctuations in sample throughput complicate matters further, making efficient scheduling and operation a truly daunting task, affecting profitability and customer retention. The ability to accommodate high sample analysis loads in peak demand periods (which may be as high as 10,000+ samples per day, in many cases; for instance, one lab in central Nebraska will analyze approximately 165,000 soils samples in 2009, and expects 200,000 samples in 2010) becomes the soils laboratory manager's highest priority. It is not uncommon for soils labs to see approximately 80% of their annual business/throughput in the two busy seasons (spring and fall), a time period which amounts to approximately 4 to 5 months of the year. The reliability of the laboratory equipment during these periods is a key factor in assuring the aforementioned profitability and related customer retention. Soils lab customers expect and receive an approximate 24 hour turnaround time on the processing of their samples. Delays of any kind, including equipment downtime, are detrimental to the lab's objective of meeting their customer's needs.

Laboratory costs associated with such high seasonal sample throughput can be considerable, including expenses associated with excessive consumption of ICP peristaltic pump tubing, Argon gas and electrical power. Additional costs associated with long term wear of ICP-AES hardware such as nebulizers, spray chambers, torches and viewing optics are also considerable.

### REQUIREMENTS

The characteristics of a properly applied technique to address high demand sample throughput effectively and efficiently are as follows:

- Significant throughput gain
- Reliable operation
- Robust construction
- Minimal required maintenance
- Minimal method manipulation/validation
- Simple interface with existing, proven autosampling equipment and nebulizer system
- Software package requiring minimal host computer resources



Figure 1. DSA-7 Discrete Sampling Accessory

## SOLUTION

While the capacity to accommodate high sample throughput is of prime consideration to the lab manager, the nature of soils analysis requires that any solution be extremely robust and durable. The equipment used in this application sees daily, continuous exposure to dust particles and corrosive acid vapors. The damage that can be caused by this exposure is exacerbated by the minimal available time during the busy seasons to perform service and maintenance functions.

The solution that addresses this throughput fluctuation and need for “workhorse” rated equipment has been developed by a company whose success has been built upon solving customers’ problems. This “new class” of solution-based technology has revolutionized the manner in which the soils industry addresses these daily challenges. CETAC Technologies, a world leader in automated sample handling and introduction systems for atomic spectrometry, introduces the *DSA-7* Discrete Sampling Accessory (Figure 1). The *DSA-7* enables higher sample throughput by reducing time required to perform several required process steps:

1. Sample loading time
2. Sample stabilization /read delay time
3. Sample washout time

Note that the actual time spent measuring analyte signals from the ICP-AES will remain unchanged.

The main components of the *DSA-7* are an inert high-speed vacuum pump and inert, 6-port injection valve. The sample probe (from the autosampler) is connected to the injection valve. The high-speed vacuum pump rapidly loads a fixed-volume sample loop connected to the valve. Once the sample loop is loaded, the valve mode is switched from “load” to “inject”, thereby transporting the loop contents into a pumped stream (via the standard ICP-AES peristaltic pump) of carrier liquid to the standard ICP-AES nebulizer/spray chamber system. Note that the flow rate of the carrier stream can remain unchanged (no “fast-pumping” is necessary) throughout the entire sample sequence, and that *the analyte never contacts the peristaltic pump tubing* (Figures 3a and 3b).

The vacuum pump loop loading function greatly reduces the typically much longer (ex. 30 seconds) step of increasing the speed of the peristaltic pump to transport sample solution through the sample probe, peristaltic pump tubing, and transfer tubing to the nebulizer. In addition, since samples do not come into contact with the peristaltic tubing, sample memory effects from this tubing are eliminated.

Since the flow rate of the carrier stream that pushes the fixed-volume sample to the nebulizer now remains constant, sample stabilization times are also reduced. A conventional analysis will usually allow at least 15 seconds for the ICP nebulizer system to stabilize after the peristaltic pump speed is lowered from sample load speed to sample analysis speed.

The sample outlet line of the injection valve can be placed in very close proximity to the nebulizer inlet, facilitating very rapid sample input and shorter read delay times. Once the measurement of the required analytes is complete, the injection valve switches mode from “inject” to “load” to begin the next sample analysis, thus allowing the carrier stream to quickly washout the nebulizer/spray chamber.

Please note Figures 2, 3a, and 3b, which depict sample solution flow paths for both conventional and *DSA-7*-equipped sample introduction systems. Also note that the same autosampler and peristaltic pump systems are used, with the addition only of the *DSA-7* pump/valve module and a simple software user interface (see Figure 4).

This Discrete Sampling Accessory reduces sample delivery times to the instrument significantly. Sample consumption is minimal, typically 2 mL or less, allowing adequate sample volume for multiple soils analysis runs, if desired.

This system delivers the performance features required by the most demanding soils analysis laboratory. As the *DSA-7* is coupled with the “industry standard” CETAC autosampler, (ASX-260 two rack capacity, ASX-520/520HS four rack capacity, or EXR-8 eight rack capacity) it is designed and built with the same performance driven, reliable and robust functionality that soils labs have come to trust for many years. The *DSA-7* uses CETAC design and technology to solve the seasonal throughput variance problems that soils labs commonly encounter.

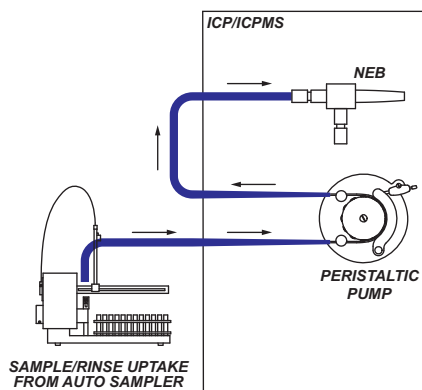


Figure 2. Conventional Sample Introduction

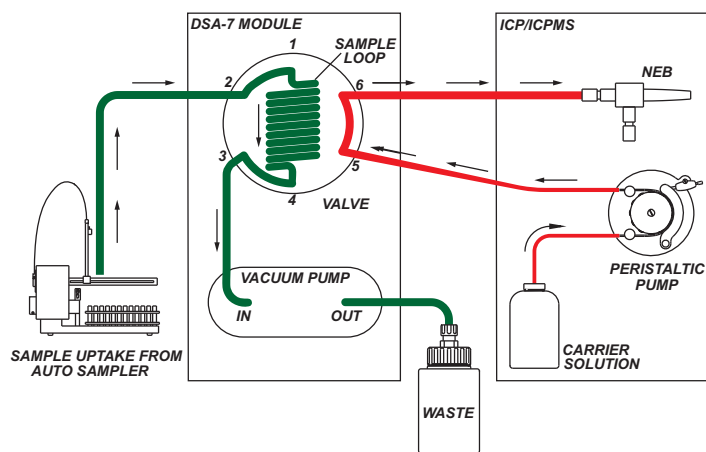


Figure 3a. *DSA-7* Sample Introduction “Load” Diagram

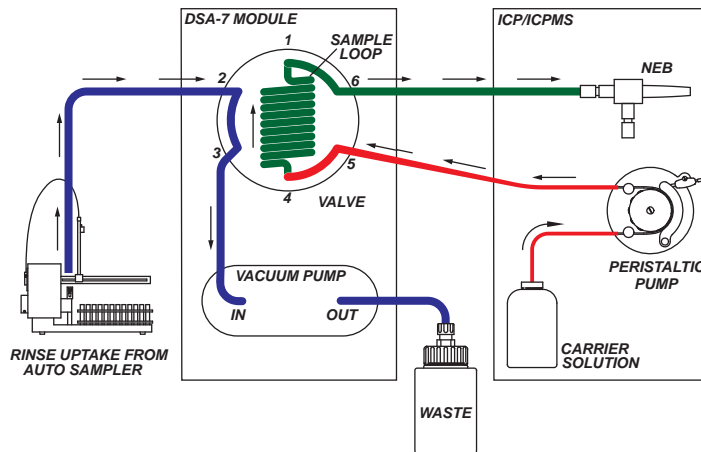


Figure 3b. *DSA-7* Sample Introduction “Inject” Diagram

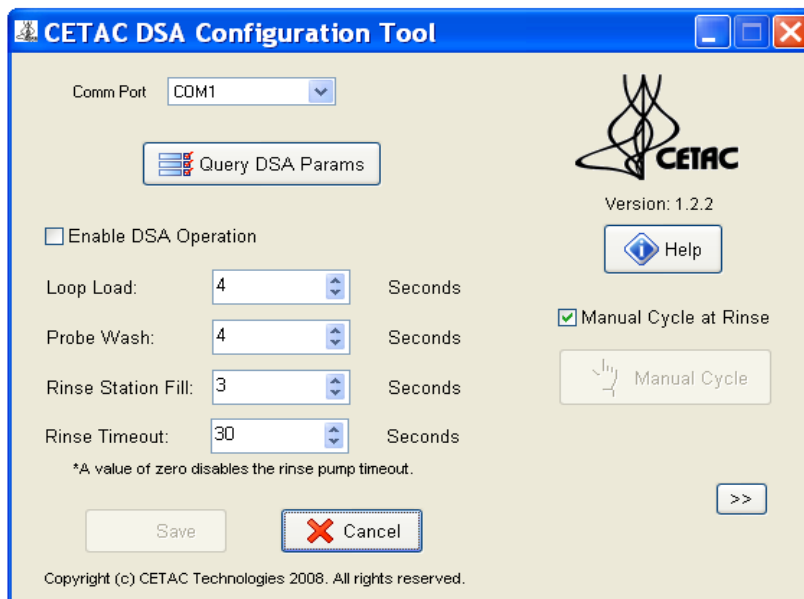


Figure 4. DSA-7 Configuration Tool

## SAMPLE THROUGHPUT

In an experiment conducted on a 240 sample run on an ICP-AES instrument, flush and rinse times were compared between the *DSA-7* sample introduction system and a standard sample introduction system. Note the improvement (Table 1) in times to complete the flush (20 sec. improvement) and rinse (30 sec. improvement) process steps when using the *DSA-7*. When accounting for loop load time (using 1.25mL loop), a 45 second per sample improvement is realized. Note the overall analysis (240 samples) time improvement (3 hours, 3 seconds) as is noted in Table 2. The *DSA-7* Discrete Sampling Accessory achieved a 40% reduction in overall analysis time.

Setup	Flush Time (s)	Rinse Time (s)	Loop Load Time (s)
<i>DSA-7</i> Introduction System	5	5	5
Standard Introduction System	25	35	0

Table 1. Flush and Rinse Time Comparison

Setup	Total Sample Run Analysis Time
240 Samples with <i>DSA-7</i> Introduction System	4 hours, 35 minutes
240 Samples with Standard Introduction system	7 hours, 38 minutes

Table 2. Total Analysis Time Comparison

## WASHOUT IMPACT ON SAMPLE THROUGHPUT

The *DSA-7* sample flow path washout is exceptionally rapid and effective. The high flow rate vacuum pump provides ample flow and mechanical cleaning action of the sample uptake stream. The line from the multi-port valve to the nebulizer is the shortest possible length to ensure rapid and complete washout via the analyzer's peristaltic pump.

Note the graph below (Figure 5) which depicts the washout characteristic and comparative throughput comparison between the *DSA-7* Discrete Sampling Accessory and a standard sample introduction system. For this depiction, timing values from the same 240 sample run mentioned above (see Table 1 for timing values) have been represented graphically to compare the speeds at which flush and rinse operations can be performed. These improvements in time values contribute to the overall throughput gains made possible by the *DSA-7*.

The intensity values noted in the graph are normalized; that is, they have been assigned an arbitrary value at steady state and a value of zero before the stabilization time begins and after washout has completed. By graphically representing the data in this manner, the resulting graph clearly illustrates the throughput comparison between the *DSA-7* Discrete Sampling Accessory and a standard sample introduction system with emphasis on sample-to-sample timing only. Note that the *DSA-7* flush and rinse times are significantly shorter than the standard system times. Also note that the *DSA-7* equipped analysis has reached steady state of the second sample by the time the standard introduction-equipped analysis is beginning the washout phase of the first sample.

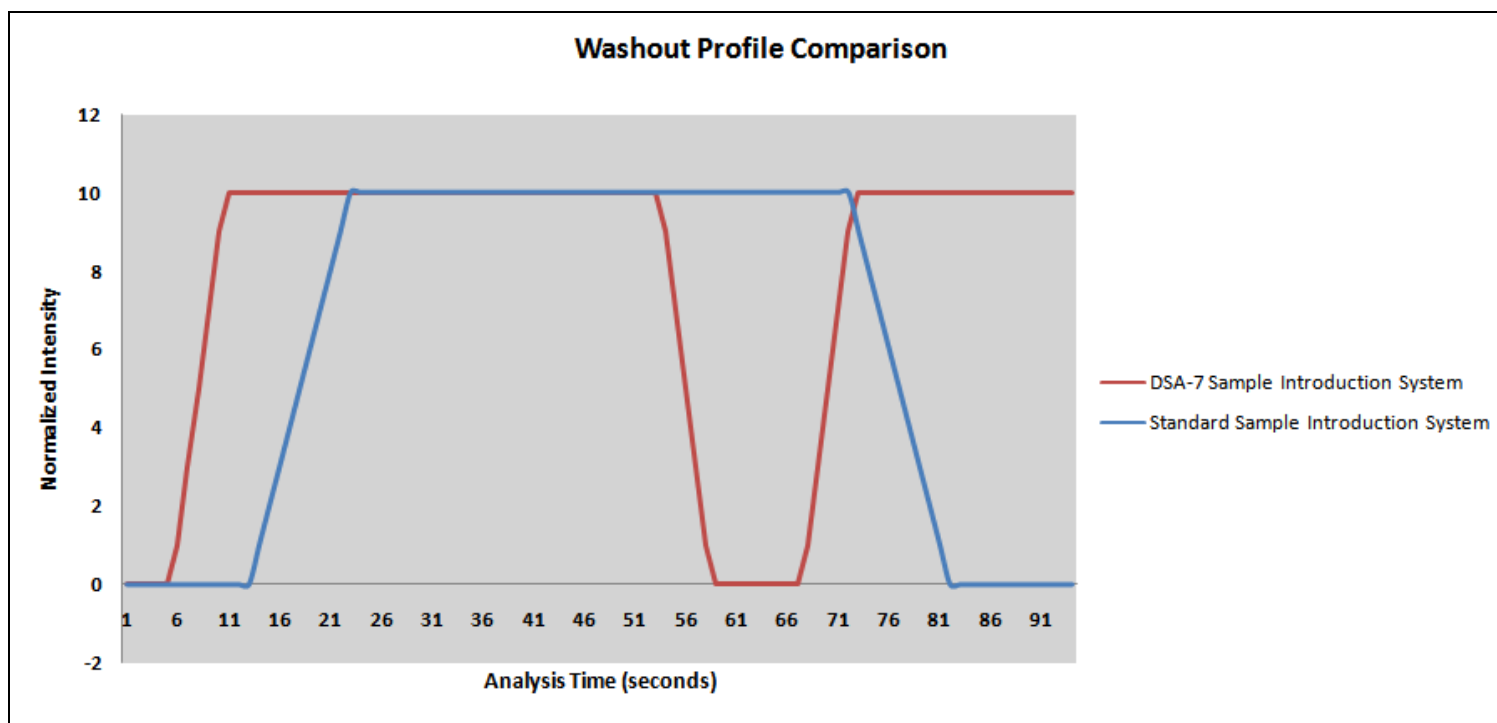


Figure 5. Washout Impact on Throughput

## STABILITY

While increasing sample throughput is extremely beneficial to the soils laboratory, any measure incorporated to expedite the analytical process must preserve the stability of the analyte signal. Note the graph below depicting the signal stability (Figure 6a and 6b) as noted with and without the *DSA-7* on the above-mentioned 240 sample run analyzed on an ICP-AES instrument. Not only does the *DSA-7* preserve the stability of the existing process, it actually provides improvement over the length of the run. This improvement is realized by virtue of the speed of throughput afforded by the *DSA-7*, which

virtually eliminates signal drift commonly encountered in standard sample introduction analysis runs. This drift is noted in the steady decline in concentration level shown in the standard introduction system graph in figure 6a below. Note the absence of this drift when using the *DSA-7* sample introduction system, as is depicted in the graph in figure 6b below.

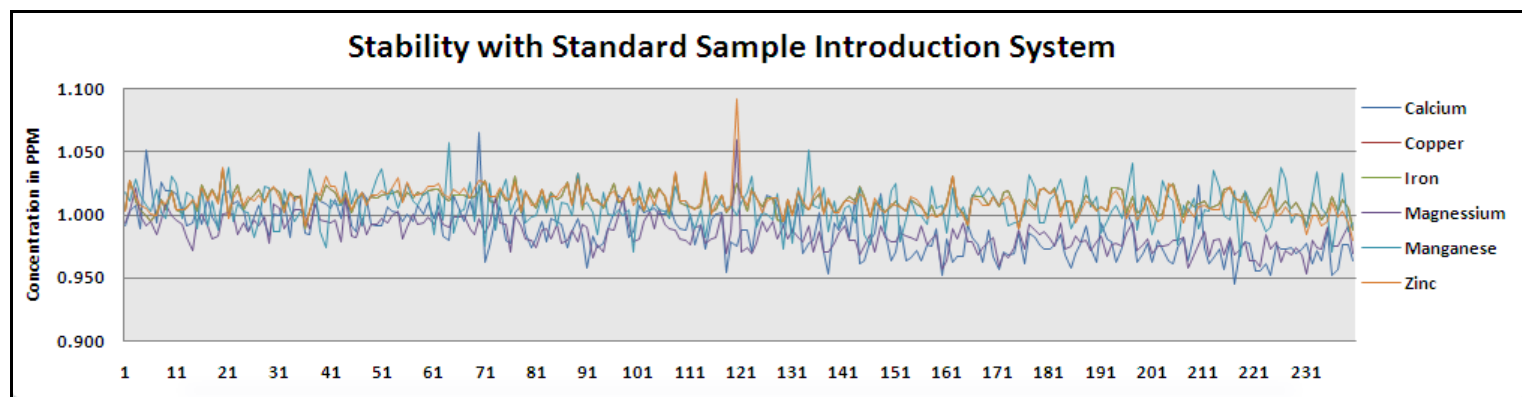


Figure 6a. Standard Sample Introduction System Stability Graph

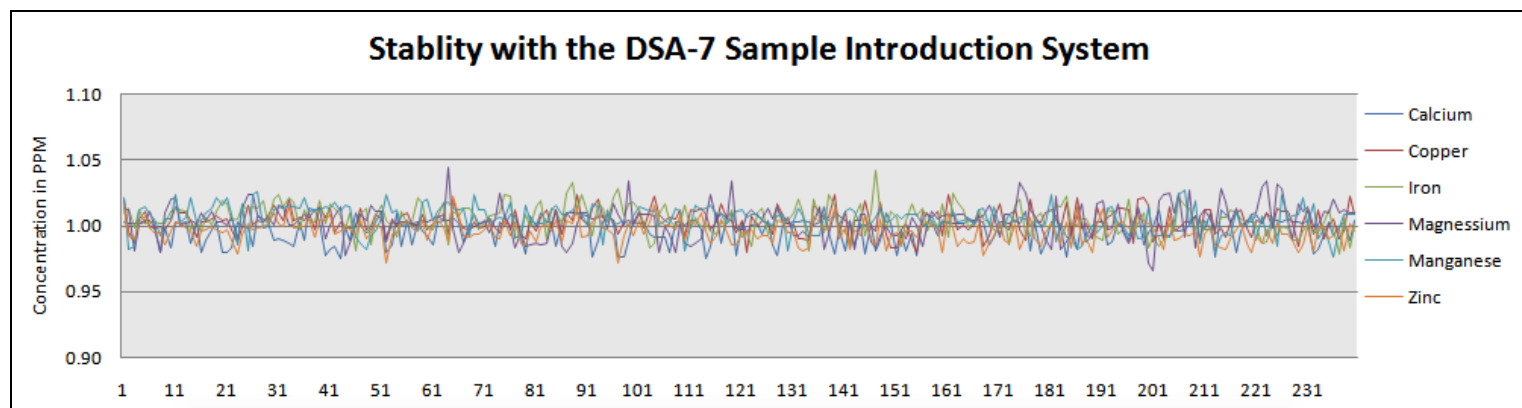


Figure 6b. *DSA-7* Sample Introduction System Stability Graph

## MAINTENANCE

Operating and maintenance costs can be a major expense consideration for any soils laboratory operation. The *DSA-7* can significantly reduce these costs. Increased throughput/reduced sample run times equate to reduced run time on the instrument and increased instrument (and instrument hardware) lifespan. Additional *DSA-7* benefits include lower costs associated with Argon gas and electrical power consumption, as well as for peristaltic pump tubing replacement (The *DSA-7* functionality eliminates sample contact with the peristaltic pump tubing).

A reliable and low maintenance system, the *DSA-7* Discrete Sampling Accessory employs a multi-port injection valve with large 0.060" ID ports (Figure 7) which are clog resistant and virtually self cleaning, a valuable low maintenance product feature providing great benefit to the soils laboratory operation by greatly extending service interval time. With very few sample flow path components, mistake proof connection cabling and minimal software requirements, the *DSA-7* provides reliable, simple and low maintenance operation in a compact package.



**Figure 7. Large Injection Valve Port Size**

## **CONCLUSION**

By listening to our customers, CETAC Technologies is able to provide solutions that solve today's real problems experienced in the laboratory. With the *DSA-7* Discrete Sampling Accessory, the soils laboratory can be relieved of the once customary chore of creating their own application-specific equipment solution to the variable throughput dilemma, allowing personnel to focus their resources on other laboratory operations.

The *DSA-7* solves variable sample throughput problems, while delivering the robust, performance-oriented reliability soils laboratories have come to expect from CETAC Technologies. While reducing ICP-AES sample run times significantly, the *DSA-7* virtually eliminates the drift commonly associated with standard introduction-equipped sample runs. In addition, operating and maintenance costs are greatly reduced while increasing productivity and ensuring reliability. This robust, reliable, simple-to-use system delivers bottom-line-boosting value, increasing the soils laboratory's profitability while providing the highest level of performance and dependability which laboratories worldwide have come to expect from CETAC Technologies.