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Application Note

Rapid, Stable Analysis of Mehlich-3 Extracts by ICP-OES

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Introduction

Soil is a complicated matrix with many different nutrients that can aid or deter the growth of desired crops. Knowing the levels of these nutrients helps farmers properly assess the health of their fields and fertilize or change crops accordingly for the next planting season. Given the critical nature of this information and the dependency of fertilizer application on weather conditions, labs are under constant pressure to improve sample analysis times.

Advancements in ICP-OES technology have greatly shortened the time required for analysis such that the majority of the time per sample is allotted to sample uptake and washout. The *ASXPRESS PLUS* Rapid Sample Introduction System greatly reduces sample uptake times by using a vacuum pump to rapidly pull the sample to the nebulizer. The same pump rinses the sample probe while the ICP is analyzing the sample. By reducing sample uptake and eliminating rinse times, the total sample time for soil extract analysis can be reduced significantly. In this application note, the *ASXPRESS PLUS* will be shown to decrease analysis time by 30% while maintaining the accuracy and precision expected of ICP-OES.

Sample Preparation

Given the variable nature of soil, proper sample preparation is of utmost importance. Methods vary regionally as they may work more or less efficiently for different types of soil. Mehlich-3 extraction is one of the most common techniques used in the analysis of soil nutrients as it is applicable for several elements and has been shown to be advantageous for analyzing phosphorous, magnesium and potassium when compared with other extracting techniques.

For this application note, two different solutions were prepared. First, a purchased Mehlich-3 extractant solution was spiked at a level equal to half of the highest calibration standard. The solution was poured into 240 – 14 mL autosampler vials. Next, a dried and sieved sediment was extracted. To 5 g of sediment, 50 mL of Mehlich-3 extract solution was added. The sample was agitated for 5 min, centrifuged, and filtered. Then, it was spiked at a level equal to that of the highest calibration standard.

Instrument Conditions

Samples were analyzed using a Perkin Elmer Avio 500 ICP-OES in conjunction with an ASX-560 and *ASXPRESS PLUS*. A GemCone nebulizer was used. Conditions are listed in Table 1 and Table 2. For the ASX-560, pump speed was set to 100% while the axis speed for both axes was set to 5.

The instrument was calibrated using a blank and 2 standards prepared in Mehlich-3 extract solution. Correlation coefficients for the calibration were 0.9999 or better. Yttrium was added to all samples and standards for a final concentration of 1mg/L. Wavelengths and calibration standards used for analysis are listed in Table 3. All elements were analyzed radially.

Table 1: Avio Settings

Parameter	Setting Standard Run	Setting *XPRESS* Run
Pump Speed	3 mL/min	2.5 mL/min
Read Delay	20 s	8 s
Rinse Time	0 s	0 s
Power	1500 W	
Nebulizer	0.7 L/m	
Auxiliary Gas	0.4 L/m	
Plasma	10 L/m	
Replicates	2	

Table 2: *XPRESS* Settings

Parameter	Setting
Loop Size	0.7 mL
Loop Evacuation Delay	0.1 s
Loop Load	0.8 s
Equalization Delay	1.0 s
Time to Evacuate Probe	1.0 s
Probe Rinse	1.0 s
Rinse Station Fill	2.8 s

Table 3: Wavelengths and Calibration Standards

Element	Wavelength	Standard 1 (mg/L)	Standard 2 (mg/L)
Aluminum	308.215	10	100
Boron	249.677	1	10
Calcium	317.933	10	100
Copper	327.393	0.1	1
Iron	238.204	1	10
Potassium	766.490	4	40
Magnesium	285.213	10	100
Manganese	257.610	0.4	4
Sodium	589.592	4	40
Phosphorus	213.617	1	10
Sulfur	180.669	0.4	4
Zinc	206.200	0.4	4
Yttrium	371.029	Internal standard	

Results

Fast

To demonstrate the reliability and speed of the *ASXPRESS PLUS*, a full tray of 240 spiked aliquots were analyzed. A blank was analyzed every 10 samples to verify no carry-over occurred during the analysis. None of the blanks had detects higher than 10% of the low standard. No rinse time was included in the standard method, as rinsing is often skipped in soils analysis to save time. With the *ASXPRESS PLUS*, the system is rinsed during analysis, so no additional rinse time is needed.

The time required to analyze 24 blanks and 240 samples was 1:31:03 for an average time of 20.7 seconds per sample, roughly 3 per minute. This allows an additional 431 samples to be run in an 8-hour shift (see Table 4) as compared with the standard method.

Laboratories analyzing Mehlich extracted samples often use only 1 replicate for analysis. In our test, using 1 replicate saved about 2 seconds of analysis time resulting in sample analysis of less than 20 seconds per sample with the *ASXPRESS PLUS*.

Table 4: Sample Analysis Times

	Analysis Time Per Sample	Time Savings	Samples run during 8 hr Shift	Samples run during 24 hr Day
Std Method	30 s	-	960	2880
ASXPRESS PLUS	20.7 s	30%	1391	4174
Additional Samples			431	1294

Precise

The relative standard deviation (RSD) was calculated from the analysis of the 240 spiked extracts. The results are in Figure 1. All RSDs are less than 5% indicating excellent precision. The average % RSD for all elements is 1.0%.

Accurate

The % recovery for each element of the spiked extracts is shown in Figure 2. All recoveries pass within ±10% of the true value. Average recovery for all elements is 99%.

Figure 1: Relative Standard Deviation for 240 Mehlich-3 standards

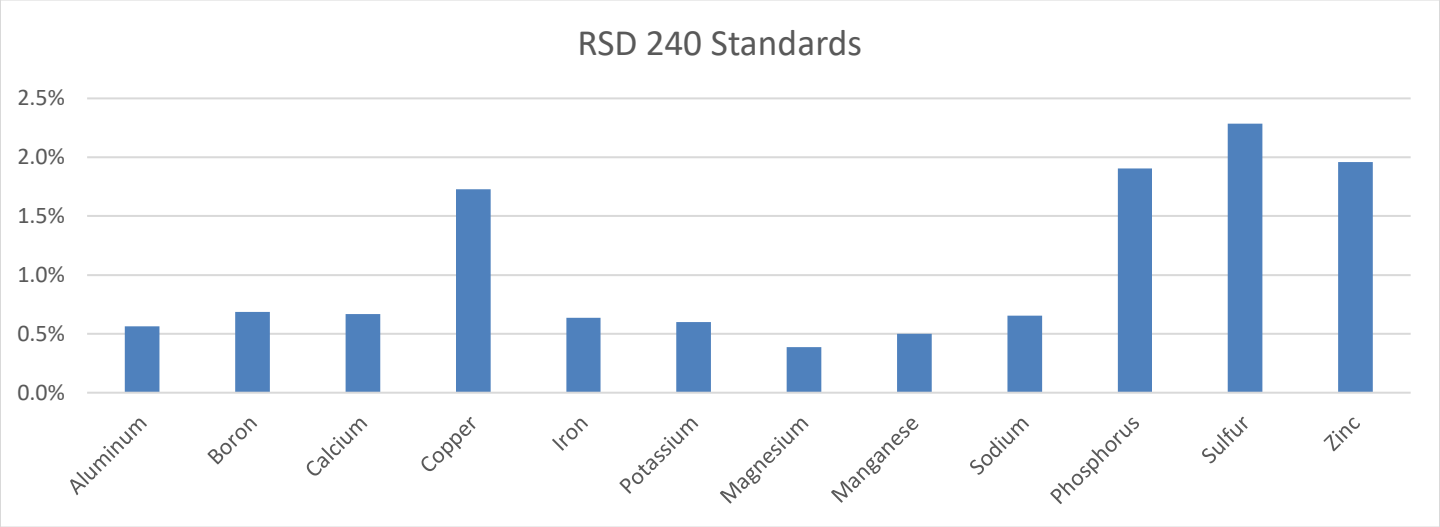
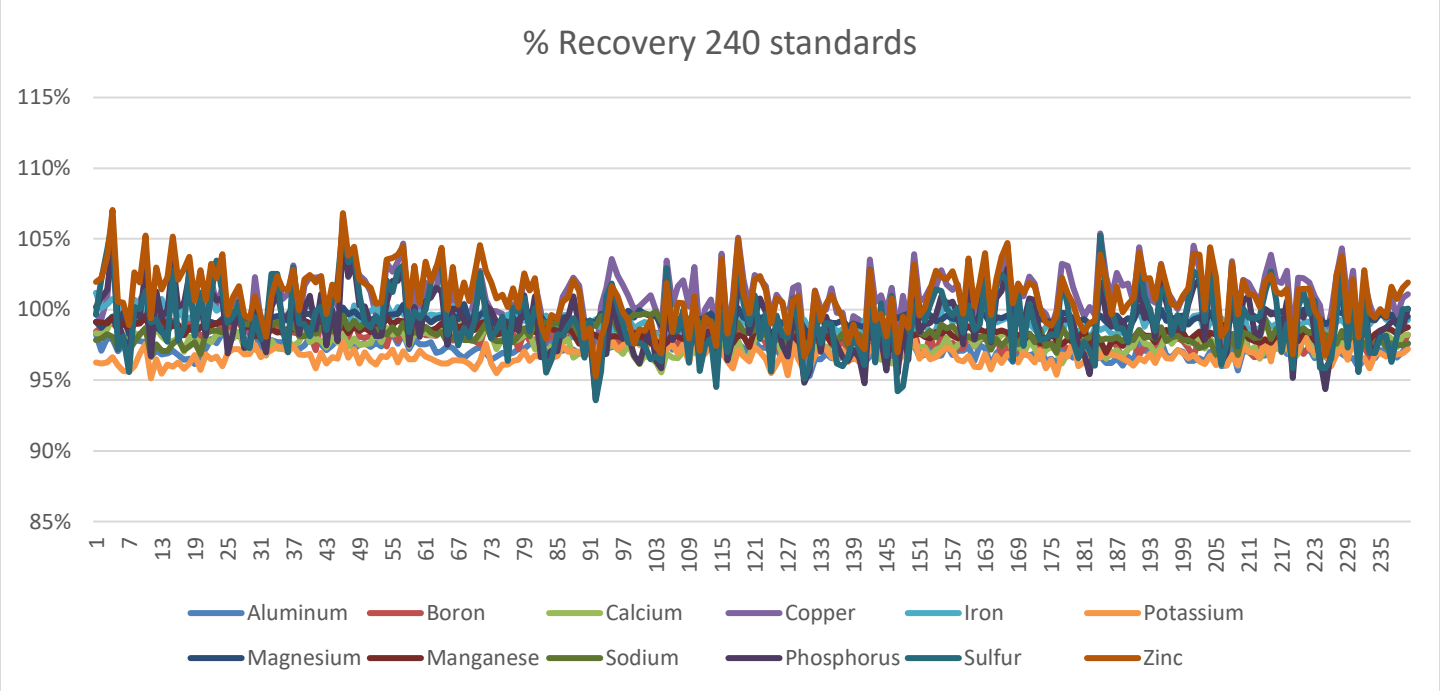


Figure 2: Recovery of Each Mehlich Standard



Precise Matrix Spike Results

A soil sample was extracted, spiked, and analyzed 10 times. The average resulting concentrations are listed in Table 5. The relative standard deviation from the 10 analyses was calculated. The results are in Figure 3. Despite the influence of the sample matrix, RSDs are passing at less than 5%. The average % RSD for all elements is 1.5%.

Table 5: NIST SRM Results

Element	Result (mg/L)
Aluminum	180
Boron	9.2
Calcium	320
Copper	1.1
Iron	63
Potassium	51
Magnesium	110
Manganese	90
Sodium	41
Phosphorus	11
Sulfur	23
Zinc	5.6

Reduced Carry-over

To determine carry-over, the same blank was analyzed before and after the 10 matrix spike soil samples. The standard run did not have a rinse step that is typical for laboratories analyzing Mehlich samples. In contrast to the standard run, the *ASXPRESS PLUS* features a segmented washout that cleans liquid flows paths more completely and in less time. The benefit is demonstrated by calculating the difference between the initial blank result and the final blank result (Figure 4). With the addition of the *ASXPRESS PLUS*, carry-over is reduced without adding additional time to sample analysis. Carry-over is important as it can impact both sample and QC results. For example, the Manganese result for a mid-level standard analyzed before and after the 10 samples increased by 12%. Without the *ASXPRESS PLUS,* a standard analyzed after the 10 samples was barely passing at 109.45% recovery. With the *ASXPRESS PLUS*, sample lines are always rinsed, reducing potential QC failures due to carryover.

Figure 3: Relative Standard Deviation for 10 analyses of spiked soil extract

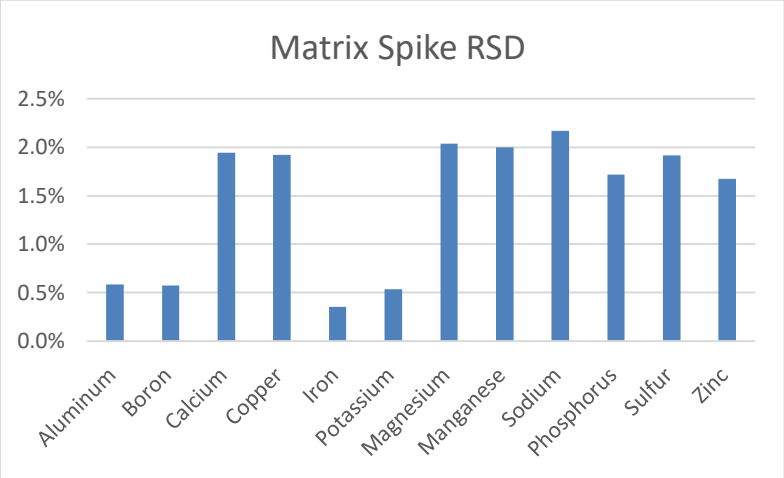
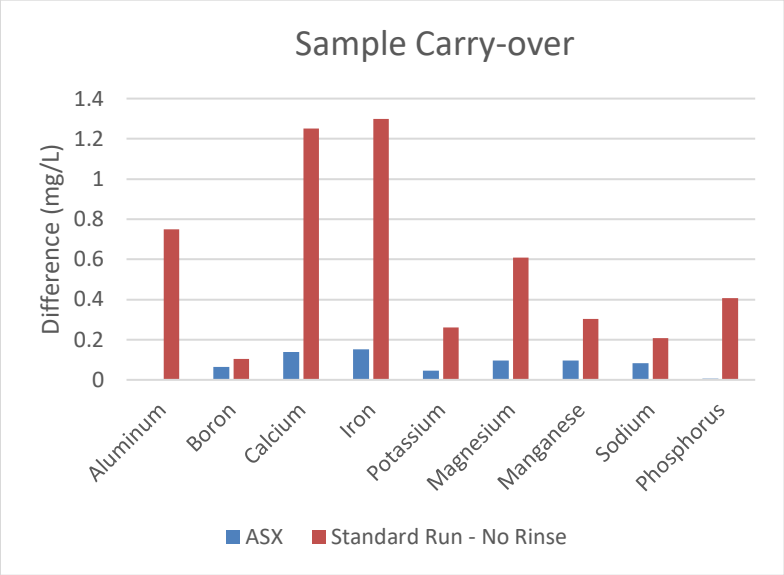


Figure 4: Change in blank results after 10 sample



Conclusion

The *ASXPRESS PLUS* accessory shortens sample analysis time by 30% while maintaining excellent precision and accuracy. This savings represents a potential gain of 431 samples across an 8-hour shift or nearly 1300 samples in the course of a full day. In addition, sample rinse can be accomplished efficiently, reducing carryover without increasing analysis time. The *ASXPRESS PLUS* is a simple and reliable accessory ideal for increasing throughput and improving data quality in the rapid analysis of soil samples.

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