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Application Note

Improving Sample Throughput in the Mining Industry with the *ASXPRESS PLUS*

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Introduction

Mining companies routinely analyze collected sediments to determine the mineral content for exploration or market value. Accurate and rapid analysis of these samples is important as erroneous or late results can impact the price of the specimen and cause lost productivity. The addition of ICP-OES to the analysis of geochemical samples has greatly reduced analysis time and improved efficiency. However, the speed of analysis is limited by long sample uptake and rinse times. Using the *ASXPRESS PLUS* Rapid Sample Introduction System, sample uptake times are significantly shorter and rinse times are eliminated, resulting in substantial throughput improvement. In this application note, the *ASXPRESS PLUS* will be shown to reduce sample analysis time by 70% without impacting the precision and accuracy of the results.

Sample Preparation

A geological certified reference material (CRM), OREAS 45f (Ore Research & Exploration P/L, Victoria, Australia), was digested using a 2-acid aqua regia (AR) digest. To each digestion vessel, 0.5 g of sample was added followed by 9 mL of HCl and 3 mL of HNO_3. Samples were digested in a graphite block digestor at 95 °C for 2 hours. After the digestion was complete, the samples were filled to a final volume of 50 mL with laboratory grade water. Final AR acid concentration was 24%.

Instrument Conditions

Samples were analyzed using a PerkinElmer Avio 500 ICP-OES in conjunction with a Teledyne CETAC ASX-560 autosampler and *ASXPRESS PLUS*. A Meinhard glass nebulizer aspirated the samples. Operating conditions are shown in Tables 1 and 2. Due to the high concentrations of several analytes of interest, the extra loop rinse option was used in this method. The extra rinse feature cleans the probe line and sample loop prior to the uptake of the sample aliquot, reducing carryover.

The instrument was calibrated using a blank and 6 standards. Due to the interference of Iron with other analytes, it was calibrated separately. Calibration standards were prepared in 24% AR. All elements were analyzed radially. Element wavelengths and calibration standards are listed in Table 3. The elements analyzed in this method as well as elements present at concentrations above 1 mg/L in the OREAS 45f were analyzed individually to evaluate interferences. Whenever possible, an emission line free of interferences was selected for analysis. If an interferent free line could not be selected, an inter-element correction (IEC) was created. An IEC check solution containing the interfering elements was analyzed with each run to verify the accuracy of the IEC.

Two internal standards (IS), Yttrium and Terbium, were added to samples and standards at a final concentration of 10 mg/L and 1 mg/L, respectively. The internal standards were added using the IS port of the 7-port valve for the *ASXPRESS PLUS* and a mixing tee for the standard sample introduction.

Table 1: Avio Settings

Parameter	Setting Standard Run	Setting Xpress Run
Pump Speed	1.5 mL/min	2.5 mL/min
Read Delay	30 s	14 s
Flush Speed	3.0 mL/min	-
Flush Time	30 s	-
Rinse Time	30 s	-
Power	1500W	
Nebulizer	0.7 L/m	
Auxiliary Gas	0.4 L/m	
Plasma	10 L/m	
Replicates	3	

Table 2: Xpress Settings

Parameter	Setting
Loop Size	2.0 mL
Loop Evacuation Delay	0.1 s
Loop Load	3.0 s
Equalization Delay	1.0 s
Time to Evacuate Probe	1.0 s
Probe Rinse	2.0 s
Rinse Station Fill	6 s
Extra Loop Rinse	Yes
Loop Rinse Delay	3.0 s

Table 3: Element Wavelengths, IS, and Calibration Standards

Element	Wavelength	Internal Standard	Calibration Standards
Barium	233.527	Y 371.029	0, 0.1, 0.5, 1.0, 10 mg/L
Cobalt	228.616		
Chromium	267.716		
Copper	213.597	Tb 350.917	
Manganese	260.568	Y 371.029	
Nickel	231.604	Tb 350.917	
Scandium	361.383	Y 371.029	
Strontium	421.552		
Vanadium	311.071		
Zinc	202.548		
Sulfur	180.669	Tb 350.917	0, 0.5, 1.0, 10 mg/L
Iron	234.349	Y 360.073	0, 100, 1000 mg/L

Both the *ASXPRESS PLUS* and standard runs had excellent curve linearity. As an example, the calibration curve for Manganese is in Figure 1. The correlation coefficient for all elements was greater than 0.9999.

Results

Fast

The greatest advantage of using the *ASXPRESS PLUS* is the reduced sample time. Ten samples were analyzed with and without the *ASXPRESS PLUS*. Results are in Table 4. The *ASXPRESS PLUS* reduced the time per sample by 70%. These time savings are due to faster sample uptake and elimination of the rinse time. With these savings, a laboratory can analyze 615 additional samples during an 8-hour shift.

Precise

The decrease in sample time does not impact the precision of the instrument. The relative standard deviation (RSD) was calculated for the 10 samples analyzed with and without the *ASXPRESS PLUS*. As shown in Figure 2, the RSD is less than 2% for all elements and is consistent with both sample introduction systems. RSDs will vary with instrumentation and methodology; however, the data demonstrates that adding an *ASXPRESS PLUS* valve system will not increase sample RSDs relative to the standard sample introduction system.

Accurate

Before and after analyzing the 10 RSD samples, a second source standard at 1 mg/L was analyzed for all elements except Scandium and Iron. Results are in Figure 3. Using the *ASXPRESS PLUS*, the second source had recoveries of ± 5% while the standard run recoveries were within 10%. Moreover, the valve system also resulted in better CRM recovery than the standard run with the recovery of all elements except Manganese and Nickel within 5%. The average recovery for all elements in the OREAS 45f for the *ASXPRESS PLUS* was 102% versus 105% for standard sample introduction.

Figure 1: Calibration Curve with The *ASXPRESS PLUS* vs The Standard Run

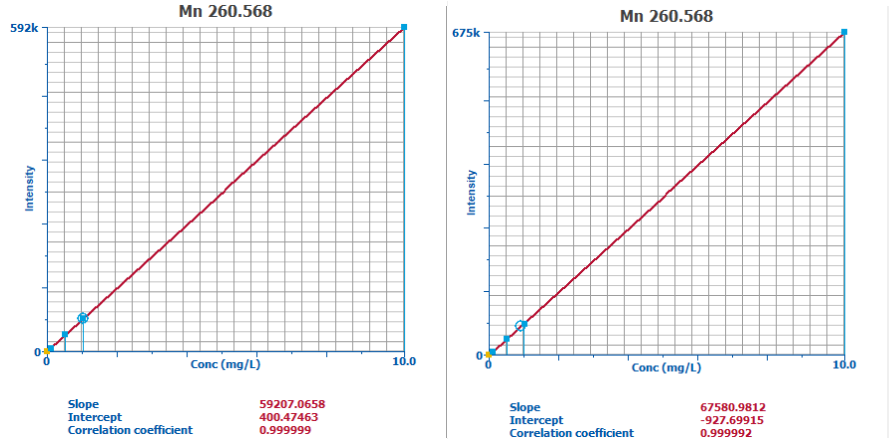


Table 4: Analysis Time Standard ICP Set-Up vs *ASXPRESS PLUS*

	Total Analysis Time 10 samples	Time per sample	Samples per 8 hrs
Standard Run	18 min 40 s	112 s	257
ASXPRESS PLUS	5 min 29 s	33 s	872

Figure 2: RSD Comparison Standard Sample Introduction System vs *ASXPRESS PLUS*

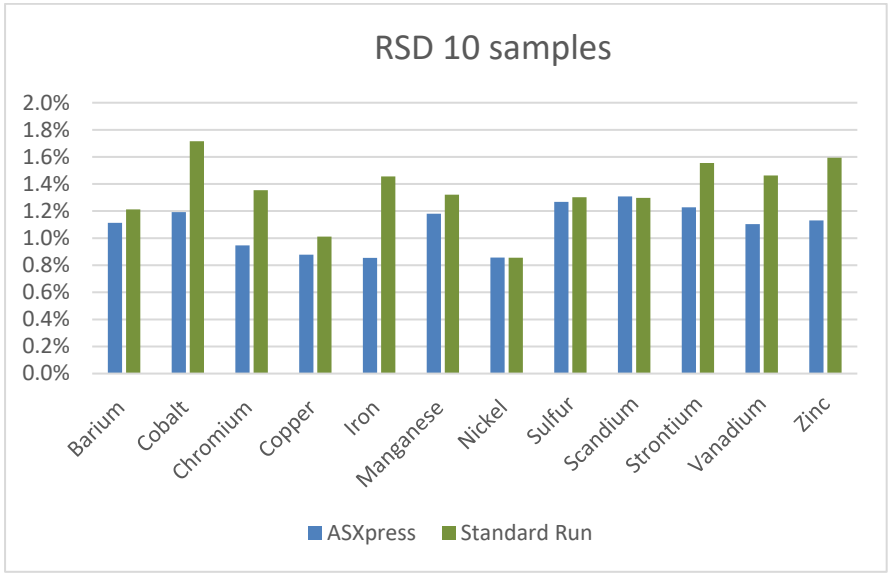


Figure 3: Initial Calibration Verification (ICV) before and after sample analysis

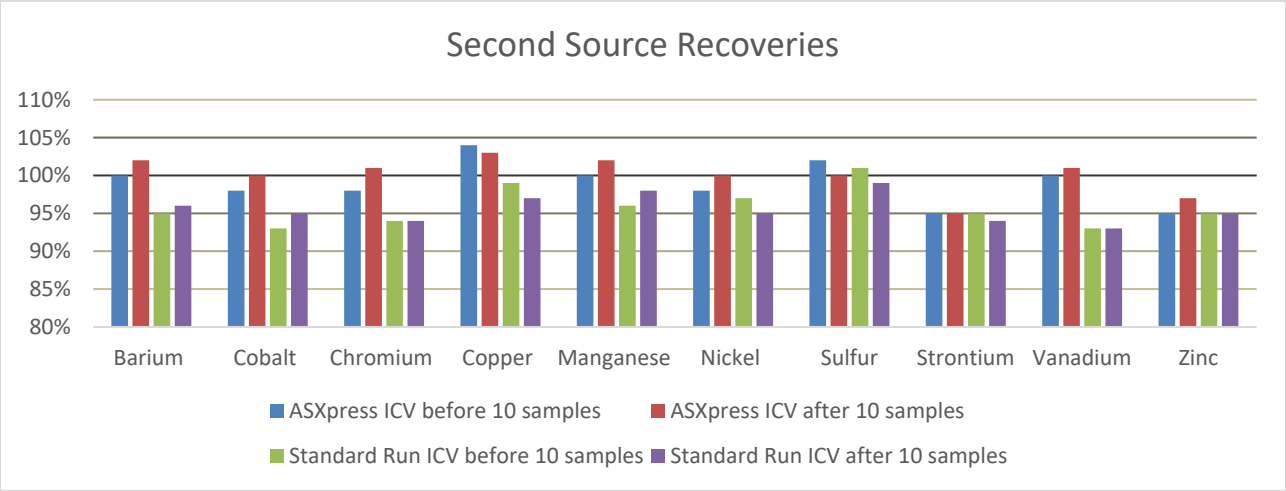
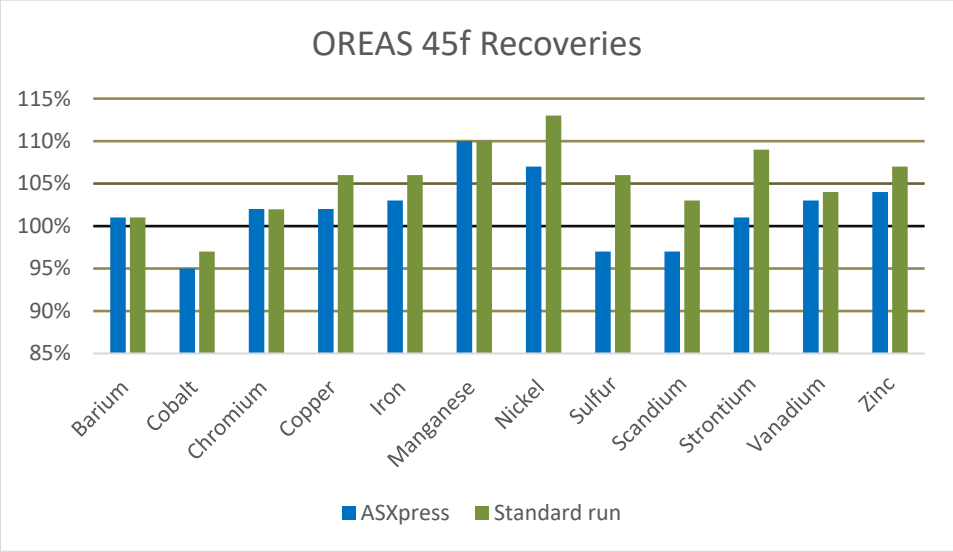


Figure 4: OREAS 45f Average Recovery 10 Sample Analyses



Conclusion

Method requirements vary considerably among laboratories testing mining samples, however, this application note has demonstrated that the *ASXPRESS PLUS* can meet or exceed results obtained with a standard sample introduction system. The *ASXPRESS PLUS* reduced the time per sample by 70% in a complex matrix of 24% AR while maintaining or improving the precision, accuracy and repeatability of the analysis.

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