

ICP-Optical Emission Spectroscopy

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Stable Analysis of Lithium Metaborate Fusion Samples with the Avio 500 ICP-OES

Introduction

The analysis of geological materials poses a challenge due to their matrix composition and the sample preparation process required to convert the samples into solution. Sample preparations vary

depending on the sample type and elements of interest, but a commonly used sample preparation technique for geological samples is lithium fusion. The fusion process involves mixing the sample with excess lithium borate and heating until the lithium borate melts and dissolves the sample to form a homogenous mass. The resulting solid is dissolved in acid for analysis.

Fusion samples are some of the most punishing samples for an ICP-OES sample introduction system, as they contain high concentrations of Group I elements, such as lithium (Li), sodium (Na), and potassium (K). The high salt concentrations from the fusion preparation process can result in deposits on the nebulizer and injector, resulting in signal drift. In addition, the high concentrations of Group I elements may cause rapid devitrification of the quartz torch. Obtaining accurate results with good precision over longer time intervals is very challenging. However, with the proper choice of sample introduction components, these challenges can be overcome.

This work describes the analysis of fusion samples leveraging the PerkinElmer Avio® 500 ICP-OES, with a focus on long-term stability.

Experimental

Samples

Ore samples, containing high levels of silicon dioxide, aluminum oxide, calcium oxide, magnesium oxide, and manganese (Mn), were prepared by a standard lithium metaborate fusion procedure. After the fusion was complete, the resulting mass was dissolved in 5% nitric acid (v/v) for analysis. The dissolved solid content of the samples is 3.5 weight percent.

All measurements were made against calibration curves of two known in-house reference materials which underwent lithium fusion sample preparation. Yttrium (Y) was added to all standards and samples as an internal standard.

Instrumental Conditions

All analyses were performed on an Avio 500 ICP-OES (PerkinElmer Inc., Shelton, Connecticut, USA) using the conditions and parameters in Table 1 and the analytes and wavelengths in Table 2. Due to the high dissolved solid content of the samples, a SeaSpray™ nebulizer with an Elegra™ argon humidifier was used to prevent nebulizer clogging and minimize deposit formation on the injector tip. While the SeaSpray™ nebulizer itself is highly resistant to clogging, the Elegra™ argon humidifier was also used to further minimize any signal drift that occurs from matrix deposition in the nebulizer and on the injector. The high dissolved solid content can also cause deposits on the torch, which leads to devitrification and instability. To alleviate devitrification, a ceramic torch was used, while the 1.2 mm injector reduced plasma loading. Because of the Avio 500's high sensitivity, the reduced amount of sample introduced to the plasma did not affect the data quality.

Table 1. Avio 500 ICP-OES Conditions and Parameters.

Component/Parameter	Description/Value
Nebulizer	SeaSpray™
Argon Humidifier	Elegra™
Spray Chamber	Twister baffled cyclonic
Torch	D-Torch, ceramic
Injector	1.2 mm id, tapered, alumina
RF Power	1500 W
Torch Position	-4.5
Sample Uptake Rate	0.7 mL/min
Read Time	1 sec/analyte

Table 2. Analytes and Wavelengths.

Element	Wavelength (nm)	Plasma View
Si	251.612	Radial
Al	308.211	Radial
Ca	317.925	Radial
Mg	285.212	Radial
Mn	257.608	Radial
Y (internal standard)	371.029	Radial

Results and Discussion

To determine the stability of the methodology, the internal standard signal was monitored in all fusion samples over a 12.5-hour run and normalized to the first sample. The resulting plot (Figure 1) shows exceptional stability, with 92% of the readings being within 2% of the initial reading; the remaining 8% of the recoveries are within 5% of the initial reading. These results demonstrate the exceptional long-term stability of the system in the presence of high total dissolved solid (TDS) samples.

To further confirm the stability of the system, the results for the two in-house QC samples from the same run are shown in Figures 2 and 3. These QC samples were analyzed every six samples during the analytical run. For the QC standards, all recoveries for all analytes are within + 3% of their true value, with the exception of Al in QC Standard 2, which consistently recovers around 110%. These results demonstrate the excellent accuracy of the methodology and show that Al contamination is present in QC Standard 2. The relative standard deviations (RSDs) for the results over the 12.5-hour analysis are also shown in both figures. With RSDs less than 1% for all elements, the high salt content of the samples does not affect the stability of the system.

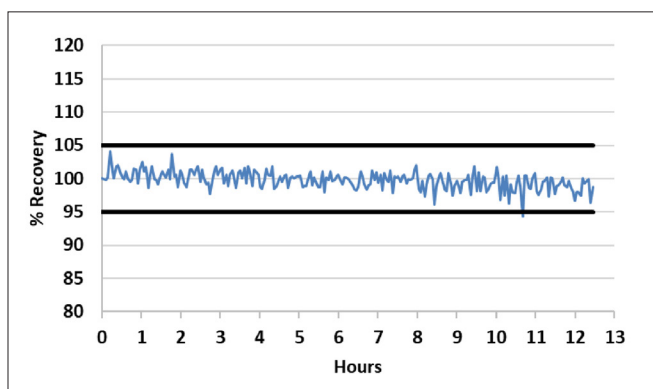


Figure 1. Internal standard stability over a 12.5-hour run of fusion samples. All results are normalized to the first sample. The black bars represent 95% and 105% recoveries.

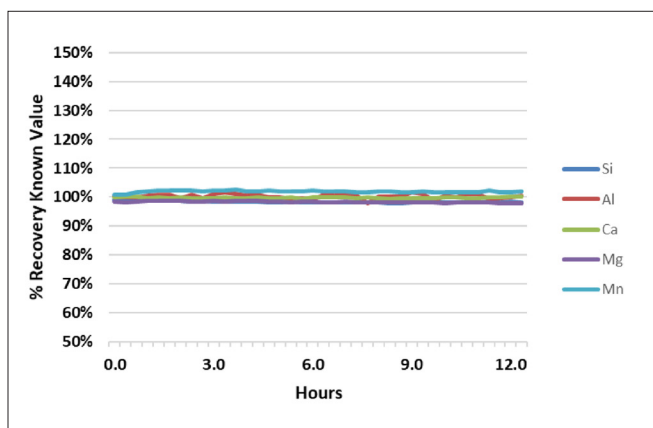


Figure 2. Analyte recovery in QC Standard 1 during a 12.5-hour analysis of fusion samples.

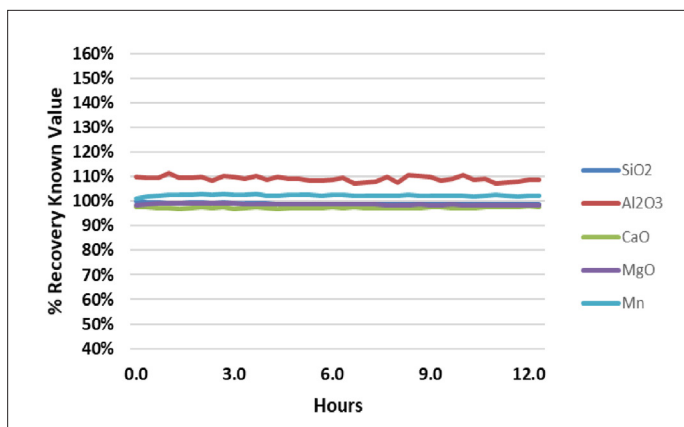


Figure 3. Analyte recovery in QC Standard 2 during a 12.5-hour analysis of fusion samples.

Working together, the Elegra™ argon humidifier in conjunction with a SeaSpray™ nebulizer, a baffled cyclonic spray chamber, a narrow bore injector, and ceramic torch minimize salt deposition in the sample introduction components, allowing high TDS samples to be analyzed over 12.5 hours without a problem. Also contributing to this stability is the Avio 500's PlasmaShear™ technology, which generates a thin stream of air that cuts off the top of the plasma (as shown in Figure 4), preventing matrix deposition on the entrance to the axial sampling window of the spectrometer. Although all measurements were done in radial mode, deposition on the axial window could affect stability if matrix components became dislodged and fell into the torch during analysis.

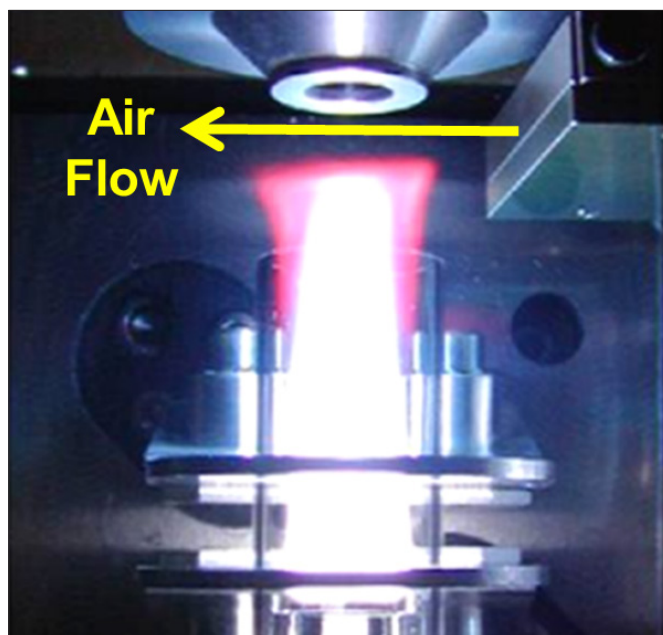


Figure 4. PlasmaShear technology in the Avio 500 ICP-OES cutting off the top of the plasma, preventing deposition on the interface window.

Conclusion

This work has demonstrated the ability of the Avio 500 ICP-OES to provide accurate, reproducible results of fusion samples over an extended period of time. Although the high TDS content of fusion samples can cause problems during extended analyses, the Avio's PlasmaShear system – in conjunction with the use of an Elegra™ argon humidifier, SeaSpray™ nebulizer, baffled cyclonic spray chamber, narrow bore injector, and ceramic torch – mitigates against the effect of high TDS, allowing accurate, stable results when analyzing high-TDS samples.

Consumables Used

Component	Part Number
Sample Uptake Tubing, Black/Black (0.76 mm id), PVC	N0777043 (flared) 09908587 (non-flared)
Drain Tubing, Red/Red (1.14 mm id), PVC	09908585
SeaSpray™ Nebulizer	N0811307
Elegra™ Argon Humidifier	N0781598
Ceramic D-Torch	N0811912
1.2 mm Tapered Alumina Injector	N0791181
Autosampler Tubes	B0193233 (15 mL, box of 500) B0193234 (50 mL, box of 500)