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Enhanced Automation of Lithium Iron Phosphate Cathode Material Preparation Using prep*FAST* MX

Introduction

Mitra Chem

Mitra Chem is developing and commercializing iron-based cathode materials to enable mass-market electrification in transportation and energy storage. Lithium Iron Phosphate ($LiFePO_4$) offers substantial improvements over traditional cathode materials in consumer safety, manufacturing cost, and supply chain risk. Mitra Chem is built to shorten the lab-to-production timeline through accelerated process iteration and metrology. Mitra Chem's mission to rapidly create world-class material at scale must be met by rapid, world-class analysis.

Controlling elemental composition of $LiFePO_4$ is crucial for battery performance. Imbalances or unwanted elements can lead to impurity phases that are electrochemically inactive or cause side reactions that reduce performance. This requires accurate measurements of stoichiometry in both $LiFePO_4$ and precursor materials.

While traditional ICP analytical methods often focus on trace analysis, Mitra Chem's unique needs require robust bulk or 'matrix' analysis, focusing on the main elemental components of cathode material. To meet these demands, Mitra Chem entered a collaborative effort with ESI, leveraging their industrial expertise and dedication to excellence, to push ICP-OES to unprecedented levels of precision and accuracy. This achievement was catalyzed by the prep*FAST* MX, which improves both the consistency and speed of measurements. Stable sample introduction with precise internal standard addition gives more stable results over time. Accurate automatic dilution of samples and calibration standards prevents cascading error and minimizes result drift. The prep*FAST* MX delivers increased productivity by facilitating efficient sample and standard preparation, ensuring stable analytical conditions, and automating repetitive dilution tasks. Ultimately, complex analysis of advanced materials becomes an automated and unattended task, liberating valuable operator time and enabling savings on consumables.

**Mitra Chem*

Introduction *(Continued)*

Through rigorous method development, Mitra Chem's ICP-OES reliably attains less than 0.30 %RSE across lithium, iron, manganese, and phosphorus bulk analysis calibration lines. For trace analytes such as salt or metal impurities below the ppm level, <1.0% RSE is routinely achieved. To robustly quantify result uncertainty, Mitra Chem averages five intensity readings per acquisition instead of the usual three, with less than 0.45 %RSD on average. For a true challenge of the prep*FAST* MX's dilution repeatability, 8 hours of repeated dilutions and acquisitions were run, in which a relative total difference in intensity of only 1.3% was observed. Mitra Chem and ESI's collaboration resulted in highly-repeatable stoichiometric analysis method with world-class precision down to the single-digit millimole level.

prep*FAST* MX:

The prep*FAST* MX from Elemental Scientific is an advanced, fully automated sample introduction system engineered to enhance the performance and efficiency of ICPMS and ICP-OES analytical workflows. This application note details the system's technical capabilities, precision fluidics, and modular automation platform, providing clear evidence of its impact on laboratory throughput, data quality, and sample integrity.

Designed to perform automated inline dilution and calibration, the prep*FAST* MX supports real-time method flexibility while eliminating the variability and labor associated with manual sample prep. The system enables unattended operation of large sample batches—with built-in capabilities for:

- Inline auto-dilution of over-range samples with closed-loop feedback
- Dynamic standard preparation for multi-point calibrations
- High-precision reagent delivery across five channels with microliter resolution

With intelligent software control and seamless integration into existing ICPMS and ICP-OES systems, the prep*FAST* MX reduces sample turnaround time, increases reproducibility, and minimizes cross-contamination. Its compact footprint and modular design make it ideal for high-throughput laboratories in environmental testing, geochemistry, semiconductor QC, nuclear safeguards, and clinical trace metal analysis.

This note presents comparative performance data on detection limits, linearity, and long-term stability, demonstrating how the prep*FAST* MX significantly improves method robustness and operational efficiency in a demanding analytical environment.

Table 1. Instrument analysis settings

Baseline Method
4-rack autosampler with peripump driven sample introduction
Manual preparation of calibration standards gravimetrically
Manual dilution of the samples
prep*FAST* MX
ESI Black/Black PVC and Blue/Blue Santoprene Peristaltic Pump Tubing
ESI 1.8 mm ZipTorch quartz glass torch
ESI PFA MicroFlow ST-3 Nebulizer
ESI DXCi Autoampler with prep*FAST* MX
ESI PressFit double pass quartz glass baffled spray chamber
ESI *pergo* 500 Argon Nebulizer Gas Humidifier

pergo:

The *pergo* from Elemental Scientific Inc. (ESI) is an innovative argon gas humidification system designed to continuously humidify the interior of the nebulizer to prevent sample and crystal buildup and improve plasma stability in ICPMS and ICP-OES analyses.

The *pergo* 500 is crucial for nebulizer protection and sustained precision during long analytical runs with advanced materials. Achieving the necessary performance in this application is impossible with even minor disruptions to sample flow – battery material analysis already faces risks high total dissolved solids (TDS), high acid, and high alkaline content in samples. Despite having a Total Dissolved Solids (TDS) level below the recommendation for flow gas humidification, Mitra Chem's cathode samples failed to meet the strict performance criteria without the *pergo* 500.

The *pergo* system uses a high-efficiency membrane to introduce controlled moisture into the argon carrier gas stream before nebulization. This humidified argon mitigates common issues such as:

- Salt crystallization at the nebulizer tip
- Plasma instability during extended sample runs
- Signal drift and memory effects in high-TDS matrices
- Unscheduled downtime due to clogging or cleaning requirements

The *pergo* improves long-term signal consistency, reduces maintenance frequency, and enhances method robustness.

It is especially advantageous in environmental, clinical, pharmaceutical, and geochemical laboratories, where continuous, high-precision elemental analysis is critical.

ESI One-piece Torch

The One-piece Torch from Elemental Scientific Inc is a robust, high-performance alternative to traditional multi-component torch assemblies used with commercially available ICP-OES systems. Designed to optimize both ease of use and analytical performance, these precision-manufactured torches integrate the outer, intermediate, and injector components into a single fused body, eliminating the need for complex assembly, alignment, or maintenance.

This application note details the performance and operational advantages of ESI's one-piece torches when used in conjunction with Agilent's 5000 series ICP-OES instruments. Key benefits include:

- Simplified installation and consistent alignment – reducing setup time and operator error
- Improved robustness and durability – with fewer joints and seals, the torch resists wear from thermal cycling and aggressive matrices
- Enhanced analytical stability – precision-engineered injector geometry provides stable plasma conditions and improved signal reproducibility
- Reduced contamination risk – single-piece design minimizes crevices where residues can accumulate

Performance data presented demonstrates improved signal stability and lower background.

Compatible with axial and radial configurations, ESI's One-Piece Torches provide a cost-effective solution for laboratories seeking high reliability, reproducible performance, and operational efficiency in routine or high-throughput ICP-OES workflows.

Experimental

To evaluate the prep*FAST* MX we compared method quality to the Mitra Chem baseline method across six months of production data on the same Agilent ICP-OES 5800 with dual view. As a test of the autodilutor's repeatability, an 8-hour continuous analysis of an iron solution was undertaken. Operator labor and consumable savings were also scrutinized.

Mitra Chem's baseline method is highly customized to have minimal interference from sample introduction and an alternative method of internal standard correction with standard Agilent hardware. The prep*FAST* MX method is a prep*FAST* MX unit direct from ESI in a "high-volume" configuration, with a full ESI sample introduction system: torch, spray chamber, and nebulizer.

Trace Element Data

Intensity %RSD is the relative standard deviation of five ICP-OES intensity readings of a given element from the same solution. (Also called CV or Coefficient of Variation.) It is a good metric of overall sample introduction stability and is commonly used in assessing instrument hardware and the stability of sample matrix chemistry. Low %RSD is a major factor in overall instrument precision, influencing both calibration linearity and sensitivity, and ultimately, accuracy. The prep*FAST* MX's syringe driven introduction system has significantly less variation than the more common "y-junction" tubing and peristaltic pump. Most labs aim for between 5-10% RSD. The prep*FAST* MX offered radically improved sample introduction stability across the entire trace element(>1 ppm) analytical suite, except Lithium, with absolute gains on average greater than 0.5%. This uncommonly stable sample introduction is a serious contributor to world-class elemental analysis.

Calibration linearity is assessed with %RSE. Though correlation coefficient (R^2) is common, %RSE provides a more direct expression of calibration error because it considers the squared difference between the predicted probability and the observed frequency, thus revealing systematic sources of error more transparently. Because of this, it is a good metric for comparing hundreds of analytical batches across different methods. This calibration uses four points forced through zero; all standards are at or below 1 ppm. An excellent %RSE value is 5% or less.

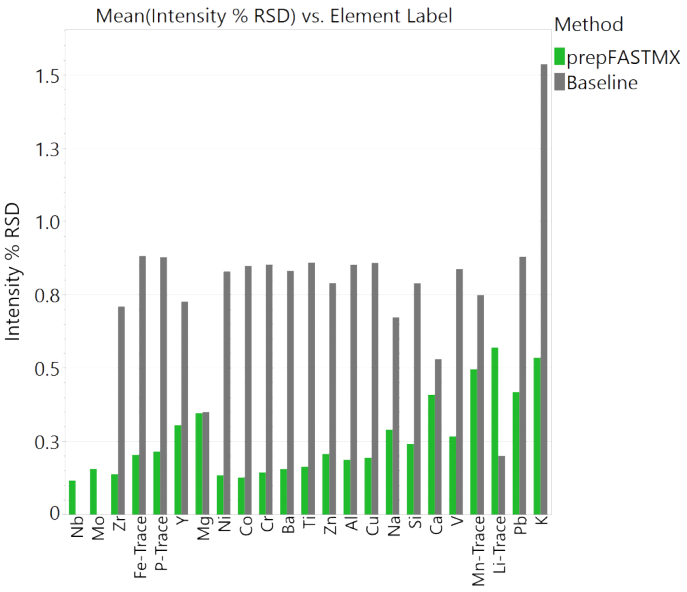

Figure 1. Comparison of average measurement stability (Intensity %RSD) of each trace element in calibration solutions across the baseline and prep*FAST* MX methods.

Trace Element Data *(Continued)*

%RSE also greatly improved with the prep*FAST* MX, likely due to improved sample introduction and accurate autodilution. Most elements are below 2 %RSE on average – a very strong benchmark for analytical accuracy. A key benefit of an autodilutor is far more repeatable standard creation than can be achieved by a human hand. This leads to more consistent results and minimization of cascading errors as a result of poor calibration standard creation.

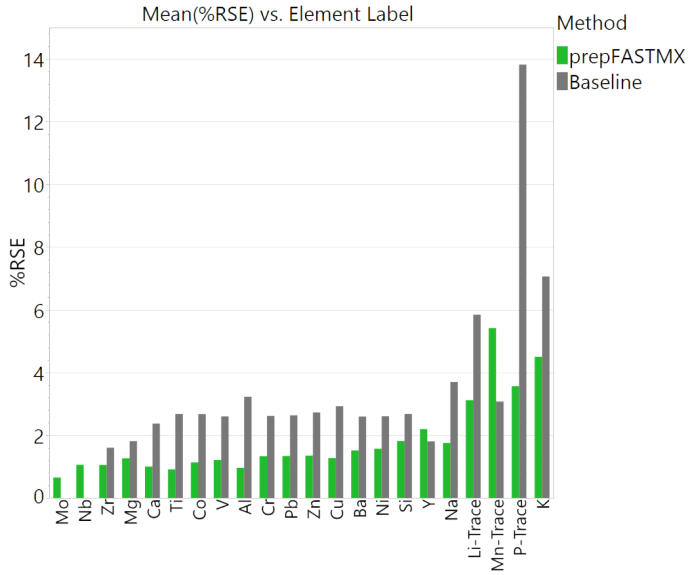

Figure 2. Comparison of average calibration linearity (%RSE) of each trace element across the baseline and prep*FAST* MX methods.

Stoichiometric / Bulk Element Data

Mitra Chem's baseline method was optimized for bulk elemental analysis for the determination of stoichiometric ratios of cathode materials, which requires maintaining very low % RSD of signal intensity. This precision was achieved in the baseline method by manual gravimetric sample dilution which was too slow to meet growing production demands.

While it was clear that prep*FAST* MX could dramatically improve throughput by replacing the manual sample preparation, Mitra Chem needed to ensure that it could also meet the demanding precision requirement for reliable determination of stoichiometric ratios.

Data generated with prep*FAST* MX showed slightly larger variation (%RSD) of the concentration of individual bulk element. However, prep*FAST* MX offered comparable precision as the baseline method for the normalized molar ratios of those three bulk elements (Figure 3) which is the most important for our bulk analysis.

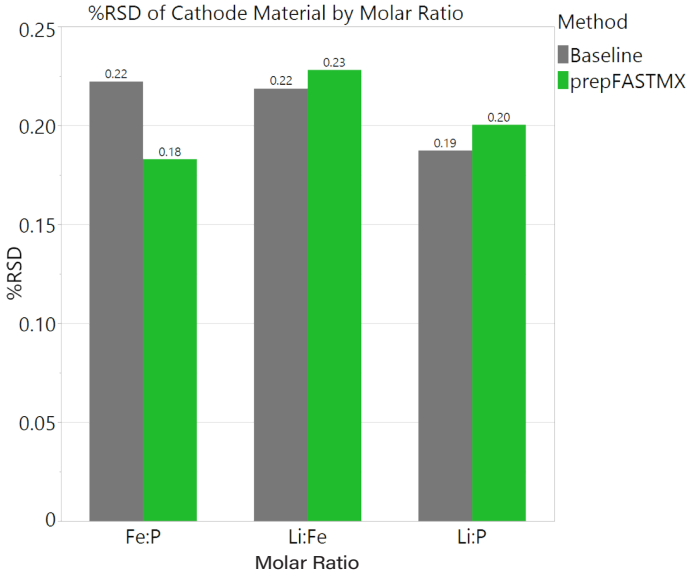

Figure 3. Comparison of average measurement stability (Intensity %RSD) of stoichiometric ratios in cathode material samples across the baseline and prep*FAST* MX methods.

Instrument Stability

As an assessment of the autodilutor's long-term analytical precision, an experiment with repeated dilutions and acquisitions of the same Fe solution was executed. The intensity increased monotonically at the beginning. The maximum variation (range/mean) of signal intensity containing the data of such period is about 1.3%, which includes instrumental drift. However, the signal stabilized after 4 hours (Figure 4) and the variation of the signal does not exceed 0.5% for more than three hours. For comparison, a competitive autodilutor running with the same ICP was unable to achieve less than 5% in signal variation.

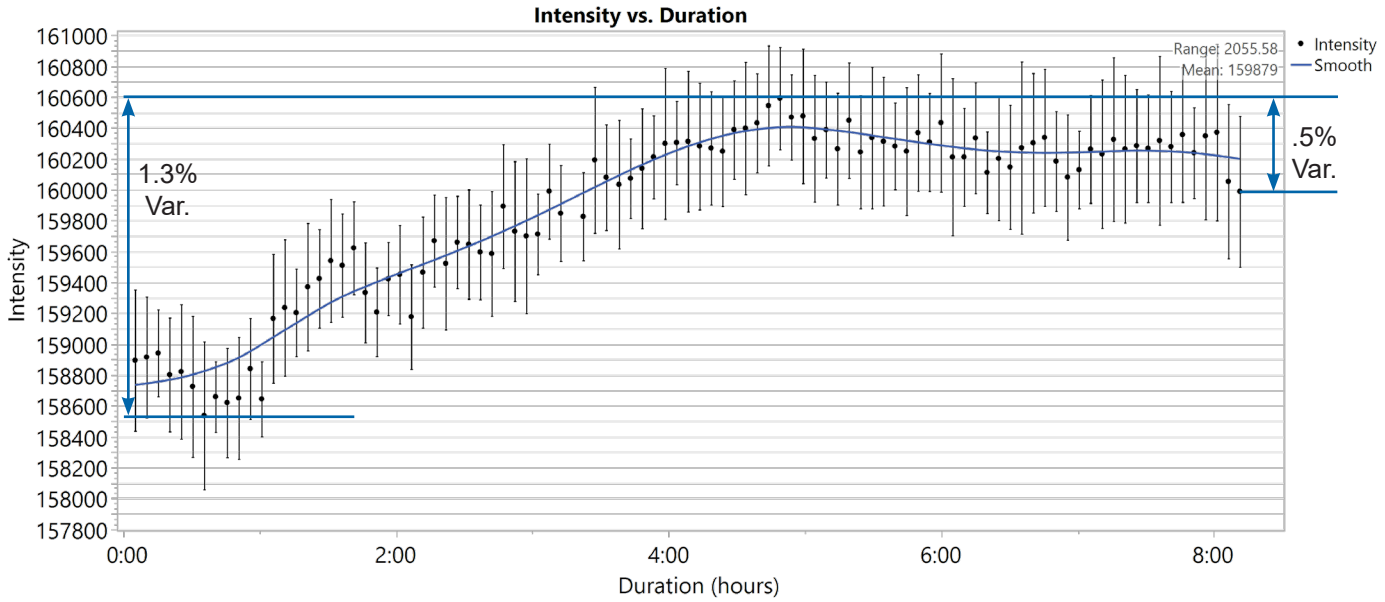

Figure 4. Stability results from a 225 ppm Fe acidic solution repeatedly acquired and diluted 15 times over 8 hours by a prep*FAST* MX.

Labor and Consumable Savings

Mitra Chem was a particularly good fit for the benefits offered by a reliable autodilutor – the "baseline method" required meticulously prepared calibration standards and samples. The prep*FAST* MX was able to halve the preparation steps, the sample vials, and the attending generated waste. Additionally, the prep*FAST* MX enables Mitra Chem to analyze the bulk and trace analytes in the same instrument run, instead of separate runs with separate instrument configurations. The prep*FAST* MX eliminates an arduous second dilution during sample preparation, and completely supplants weekly calibration standard preparation. No more pipetting or pipette tips.

The time savings are the most important - the prep*FAST* MX offers complete elemental analysis of four cathode materials prepared in triplicate in just 210 minutes, 69% faster than the baseline method. The old saying "time is money" maps well to ICP: time is Argon. Simplified sample preparation and instrument operation makes training operators for this high-level analytical technique easier, frees up valuable engineer time, and offers substantial long-term savings on consumable costs.

Conclusion

The prep*FAST* MX demonstrated a radical improvement in trace element analysis, superior sample introduction stability (Intensity %RSD) with average absolute gains exceeding 0.5% and improved calibration linearity (%RSE), with most elements averaging below 2%. These gains are attributed to the prep*FAST* MX's syringe-driven introduction system and accurate autodilution capabilities, which contribute to world-class elemental analysis and highly repeatable calibration standard creation.

For stoichiometric/bulk element analysis, there was no statistically significant difference in measurement precision of real samples between the two methods, allowing Mitra Chem to maintain current quality control standards while benefits of the autodilutor, which include 69% faster analysis time and consumable savings.

prep*FAST* MX offers substantial labor and consumable savings. It eliminates the need for meticulously hand diluted calibration standards, halves preparation steps, reduces reliance on expensive consumables, and minimizes sample waste compared to the "baseline method." Furthermore, the prep*FAST* MX consolidates bulk and trace analysis into a single instrument run. The prep*FAST* MX saves hours of precious operator time and hundreds of dollars of consumables every week.