

Direct Elemental Analysis of Battery Materials Using ICP-OES with ElectroThermal Vaporization (ETV)

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■ Background and Challenges

The battery industry requires accurate and rapid elemental analysis of raw materials such as graphite, carbon black, and cathodic materials to ensure product quality and performance. Trace metal impurities in these materials can significantly affect battery efficiency, stability, and lifetime. In addition to performance considerations, increasingly stringent environmental and safety regulations have reinforced the need for precise trace element monitoring. For example, the EU Battery Regulation (EU) 2023/1542 restricts the use of hazardous elements such as cadmium (Cd), mercury (Hg), and lead (Pb), while the Chinese standard GB/T 24533-2019 imposes strict limits on Cd and Pb content in battery materials. These regulatory frameworks require manufacturers to implement reliable analytical methods capable of detecting trace-level impurities with high accuracy.

Therefore, analytical techniques that enable fast, accurate, and direct analysis with minimal sample preparation are becoming essential.

Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) combined with ElectroThermal Vaporization (ETV) offers a powerful solution for direct solid sample analysis. This technique enables rapid and accurate determination of trace elements without complex digestion procedures, making it particularly suitable for battery material analysis and regulatory compliance. This application note demonstrates the performance of ICP-OES equipped with an ETV option for the analysis of reference materials and carbon black samples commonly used in battery applications.

■ ElectroThermal Vaporization (ETV)

ElectroThermal Vaporization (ETV) is a sample introduction technique that enables direct analysis of solid samples without acid digestion.

The main advantages of ETV include:

- Direct analysis of solid samples
- No complicated sample digestion
- Ideal for small sample amounts (0.1–10 mg, up to 500 mg for carbon samples)
- Reduced preparation errors
- Faster analysis time (around 2 minutes)

A suitable quantity of dry and homogeneous sample is weighed into graphite boats and introduced into the furnace, where a temperature program is applied consisting of an ashing/pyrolysis step at approximately 200-500°C followed by a vaporization step between 1700 and 2500°C depending on the matrix.

The total analysis time is approximately 2 minutes. During this time, spectral signals are continuously collected, allowing accurate quantification through signal integration.

Figure 1 shows the ICP-OES instrument equipped with the ETV module positioned in front of the ICP torch, enabling efficient vaporization and transport of solid samples into the plasma.



Figure 1. ICP-OES instrument configured with an ETV (Electrothermal Vaporization) module positioned in front of the ICP torch, enabling efficient vaporization and introduction of solid and difficult matrices into the plasma.

■ Sample Introduction

The sample preparation and measurement procedure is simple and fast:

1. Weigh 1–20 mg of sample into a graphite boat using a precision balance
2. Place the graphite boat into the furnace
3. Run the vaporization program with ashing (Preheating) at 200-500°C and vaporization at 700-2500°C
4. Measure the emission signal profile of each element

The ICP-NEO software records emission signals during vaporization and cooling of the sample.

As shown in Figure 2, the emission signal of Ba at 233.528 nm obtained from the reference material (BAM-S009) illustrates the vaporization peak and baseline region used for quantification. The software allows the selection of signal integration range, baseline correction, and automatic concentration calculation. This ensures accurate and reproducible quantitative results.

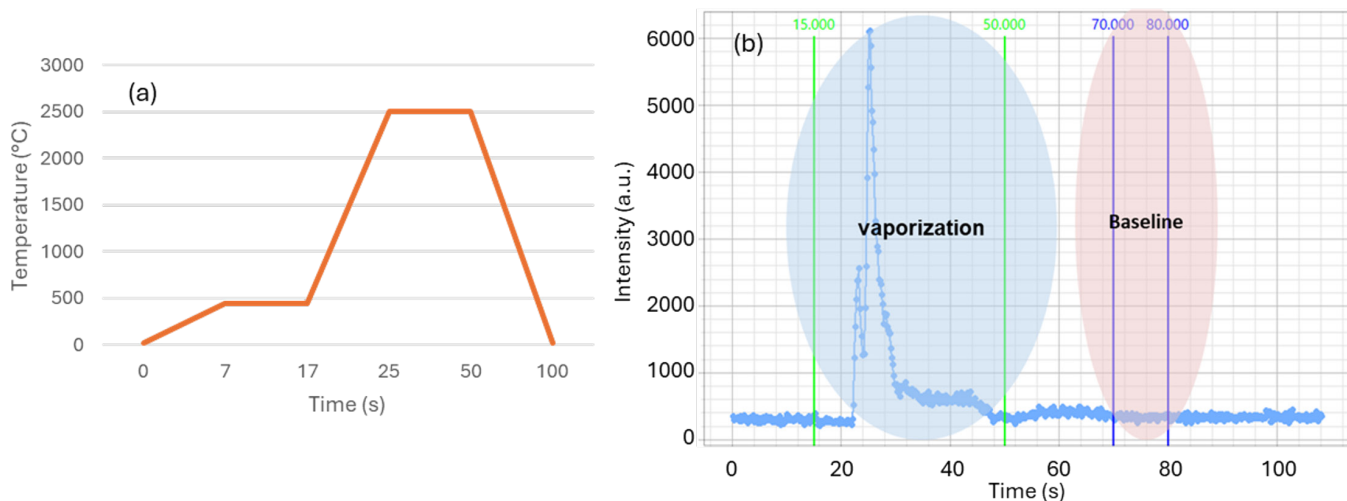


Figure 2. (a) Temperature ramping for ETV measurement with ashing (Preheating) at 480°C and vaporization at 2500°C, (b) ICP-OES emission signal of Ba at 233.528 nm obtained from the reference material (BAM-S009), showing the ETV vaporization peak and baseline measurement region used for signal evaluation and quantification.

■ Analysis of Reference Graphite Materials

Reference graphite material BAM-S009¹ was analyzed to evaluate the accuracy of the ICP-OES with ETV option. Table 1 presents the measured concentrations compared with certified reference values. The results show good agreement between measured and certified values. Most elements fall within the uncertainty range of reference material. Only Sr shows a slight deviation, while all other elements demonstrate satisfactory accuracy.

These results confirm the reliability of ICP-ETV for trace element analysis in battery-related materials.

Table 1. Concentration of reference materials (BAM-S009)

Element	Wavelength (nm)	Concentration measured by ICP-ETV (ppm)	Concentration of Reference (ppm)
S	180.668	11.18	10.7±1.8
Ba	233.528	0.89	0.80±0.09
Mn	257.613	0.08	0.094±0.015
Fe	259.94	27.16	28±4
Mg	279.078	0.13	0.135±0.030
Ti	334.944	9.84	8.6±1.6
Sr	407.775	0.46	0.32±0.05

■ Analysis of carbon black materials

Carbon black is widely used in battery electrodes as a conductive additive. The presence of metallic impurities can influence electrical conductivity and battery performance. Therefore, accurate determination of trace elements in carbon black is essential.

Figure 3 shows the preparation of the carbon black sample in a graphite boat for ETV measurement. The ICP-ETV method enables direct analysis of carbon black without digestion, significantly reducing preparation time and contamination risk.

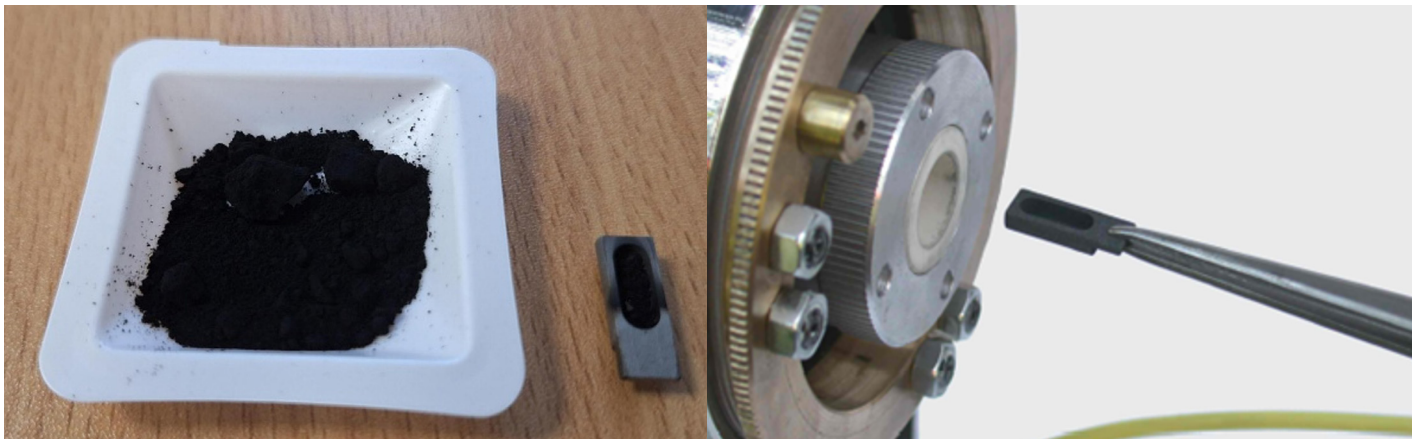


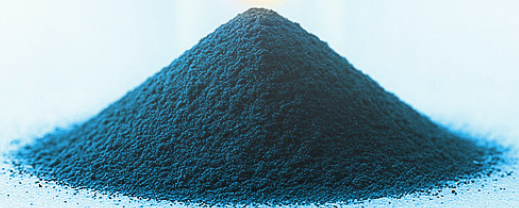
Figure 3. (left) Photo of carbon black sample and sample in graphite boat prepared for ETV measurement, (right) putting graphite boat into the furnace.

Table 2. Concentration of carbon black materials measured by ICP-ETV

Element	Wavelength (nm)	Concentration measured by ICP-ETV (ppm)
Pb	220.353	Under LOD (0.1 ppm)
Ba	233.528	0.60
Mn	257.613	0.42
Fe	259.94	21.3
Mg	279.078	2.4
Ti	334.944	0.02
Sr	407.775	0.05

Table 2 presents the concentration of elements measured in the carbon black sample. The results show the presence of Fe as the dominant impurity, while Ba, Mn, Mg, Ti, and Sr are present at low concentration levels. The low detection limits of ICP-ETV allow reliable measurement of trace elements in carbon-based materials, including Pb at trace levels.

This demonstrates that ICP-OES with ETV is well suited for the analysis of carbon black used in battery applications.



■ Conclusion

ICP-OES equipped with ElectroThermal Vaporization (ETV) provides a fast and reliable solution for the direct analysis of battery materials such as reference materials and carbon black. The method enables direct solid analysis without digestion, offering short analysis time, low detection limits, reduced contamination risk, and accurate and reproducible results, making it particularly suitable for carbon and graphite-based battery materials.

The analysis of reference materials confirms the accuracy of the method, while carbon black measurements demonstrate its applicability to real battery materials. ICP-OES with ETV therefore represents an efficient and practical tool for elemental analysis in the battery industry, supporting quality control and material development.

■ References

1. [Certified Graphite Powder Reference Materials, Bundesanstalt für Materialforschung und -prüfung \(BAM\). https://webshop.bam.de/webshop_en/reference-material/special-materials/graphite-and-battery-materials/bam-s009.html](https://webshop.bam.de/webshop_en/reference-material/special-materials/graphite-and-battery-materials/bam-s009.html)



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