IRON BASED PARTICLE CONTAMINANTS ANALYTICS C-NERGY™ L-GRADE GRAPHITES



ASSURING HIGH PRODUCT QUALITY – THROUGH IMPROVED DETECTION OF PARTICLE CONTAMINANTS

WHY A NEW ANALYTICAL METHOD?

The evolution and sophistication of customers' needs over the years necessitated a subsequent refinement of our analytical methods for the characterization of particle contaminants in our products.

Our R&D team has developed a high degree of expertise in particle contaminants quantification.

Counting methods have limitations for the quantification of particle contaminants in graphite powder. Limitations related to an insufficient degree of precision in the detection of contaminants can translate into safety-related risks affecting the final product.

The ICP-MS method was developed to achieve higher precision and control for processes and products not feasible using other analytical methods.







ANALYTICAL METHOD ICP-MS

OBJECT OF THE MEASUREMENT

Solution This method measures only the iron present as foreign particles, or contaminants. It does not measure the iron present within the graphite particles. The iron present as a contaminant is a fraction of the total Fe (internally measured by SD-OES).

MEASUREMENT PROCEDURE

ICP-MS for contaminant Fe particles.

- A sufficiently large graphite powder sample is dispersed in water with the aid of a wetting agent.
 A 0.5 T strong magnet with a protective coating is placed into the dispersion.
- Solution While gently stirring the dispersion the magnet collects all iron based, and magnetic particles.
- 🔗 After this process the contaminant particles stick to the magnet surface. These are then leached out with a proper acid mixture.
- Solution is then analyzed with ICP-MS to quantify the element concentration it contains and is therefore in the graphite sample.
- States of the method: "Talanta, Volume 224, 1 March 2021, 121827" https://doi.org/10.1016/j.talanta.2020.121827

CONCLUSION

- Solution The working range of the "ICP-MS" method is more suitable to investigate the level of Fe-based contaminants in graphite powder. It has a significantly lower LoQ (limit of quantification) than other counting methods.
- Solution The significantly larger sample allows for both an increased method and sampling precision and a reduced sampling bias and cross-contamination effects.
- So The LoQ of "counting methods" is often too high for a proper characterization of L-grade graphites.
- Solution by Due to its higher precision this method is validated and implemented in the Quality Control laboratory for process control of L-grade graphites.



METHOD PERFORMANCE

METHODS	ICP-MS >BEST SOLUTION FOR GRAPHITE	H-XRF	SEM-EDX	OPTICAL MICROSCOPES
Category	Total contaminant determination.	Particle "counting" method.	Particle "counting" method.	Particle "counting" method.
How to extract particle contaminants from the sample	Through collection via a magnet in liquid phase. Extracted contaminants are digested by acids. Procedure is done following best practices for trace element quantification.	Typically not done. The powder sample is used as is.	Typically through collection via a magnet in liquid phase. Afterwards, contaminants are transferred onto an SEM holder for analysis.	Typically through collection via a magnet in liquid phase. Subsequent cleaning steps are required to prepare the contaminants for optical imaging analysis.
Representativeness of the sample	High Large sample size is used.	Low The sample size is relatively small.	Very low Typically only a few grams of material can be analyzed.	Medium Medium sample size is used.
Method capability to characterize product	High The large sample size is highly representative of the material, leading to high precision and accuracy. Working range of the method is adequate.	Medium Small sample size and lack of a concentration step leads to high cross-contamination risk. The ability of this method is limited due to the graphite properties. Working range of the method might be inadequate. Method can identify non magnetic contaminants.	Medium The small sample size might not be representative for the material. Working range of the method might be inadequate.	Low Typically the sample preparation is complex, chance of losing contaminants is high, leading to a poor method performance. Working range of the method might be inadequate.
Easiness of implementation	High Sample preparation is straight forward. Cross contamination is low.	Medium Careful sample preparation required. Cross contamination risk is high.	Medium Careful sample preparation required. Cross contamination risk is high.	Medium Sample preparation is complex, requiring several intermediate steps. Cross contamination risk is high.



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