

APPLICATION NOTE

Graphite – Thermogravimetry – Mass Spectrometry

Detection and Identification of Impurities in Ultra-High Purity Graphite Samples by STA Measurements

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Introduction

In the graphite industry, high-purity graphite generally refers to graphite containing more than 99.99% carbon. At present, the application of high-purity graphite in the photovoltaic industry is relatively large. Graphite is also the most commonly used anode material in lithium-ion battery manufacturing due to its relatively low cost, high energy density, and high conductivity. Graphite's hexagonal layered structure allows lithium to intercalate. It ensures that the battery remains stable during charge and discharge cycles. Its structural stability results in longer battery life. For high-performance batteries, a purity higher than 99.95% and a particle size between 10 and 30 μm is required.

Measurement Conditions

The STA *Jupiter*[®] series coupled to the NETZSCH *Aëolos*[®] mass spectrometer is well suited for determination of even the tiniest impurities. High sample loads can be achieved even with low-density powders using the available STA crucibles for possible large sample volumes (up to 10 ml). This, combined with the high-end MS coupling system (transfer temperatures up to 300°C), allows for the transfer and identification of low levels of even high-boiling materials.

In table 1, the measurement conditions are summarized.



1 STA *Jupiter*[®], coupled to the NETZSCH *Aëolos*[®] mass spectrometer

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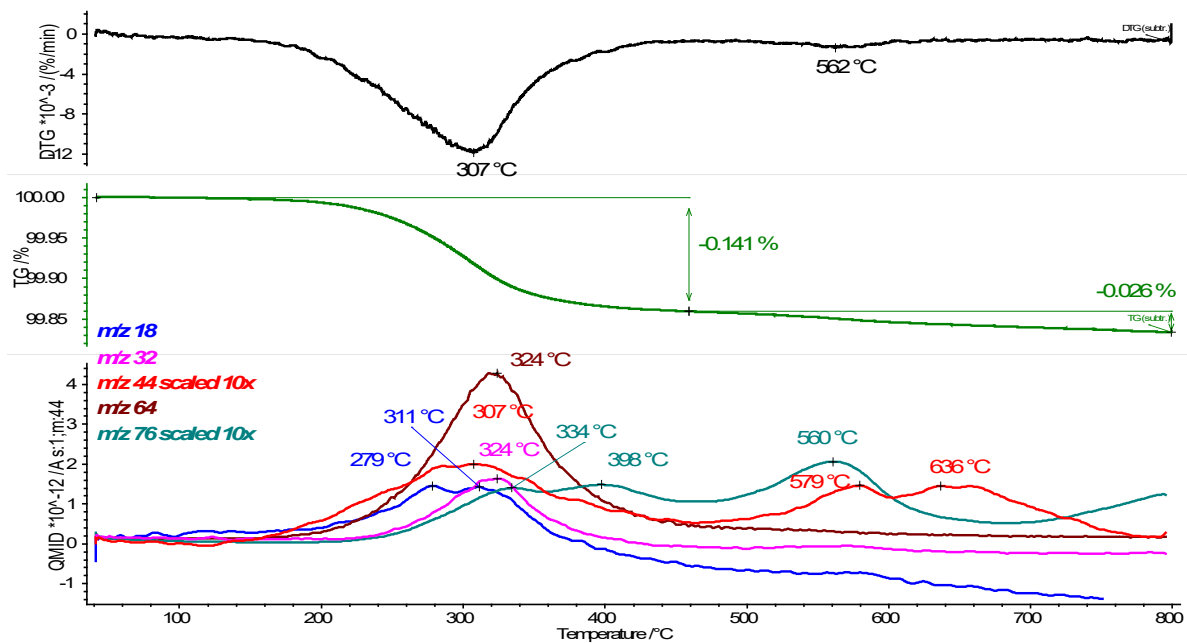
Table 1 Measurement parameters for TGA-MS

Furnace	SiC
Sample carrier	TGA pin with OTS® (Oxygen Trap System)
Crucible	Al ₂ O ₃ , 5 ml, open
Sample thermocouple	Type S
Purge gas	Ar, 50 ml/min
Protective gas	Ar, 20 ml/min
Temperature program	RT - 800°C, 10 K/min
MS parameter	Scan mode in the range 1-300 amu, Integration time per amu 20 ms
Sample mass	3226.33 mg

Measurement Results and Discussion

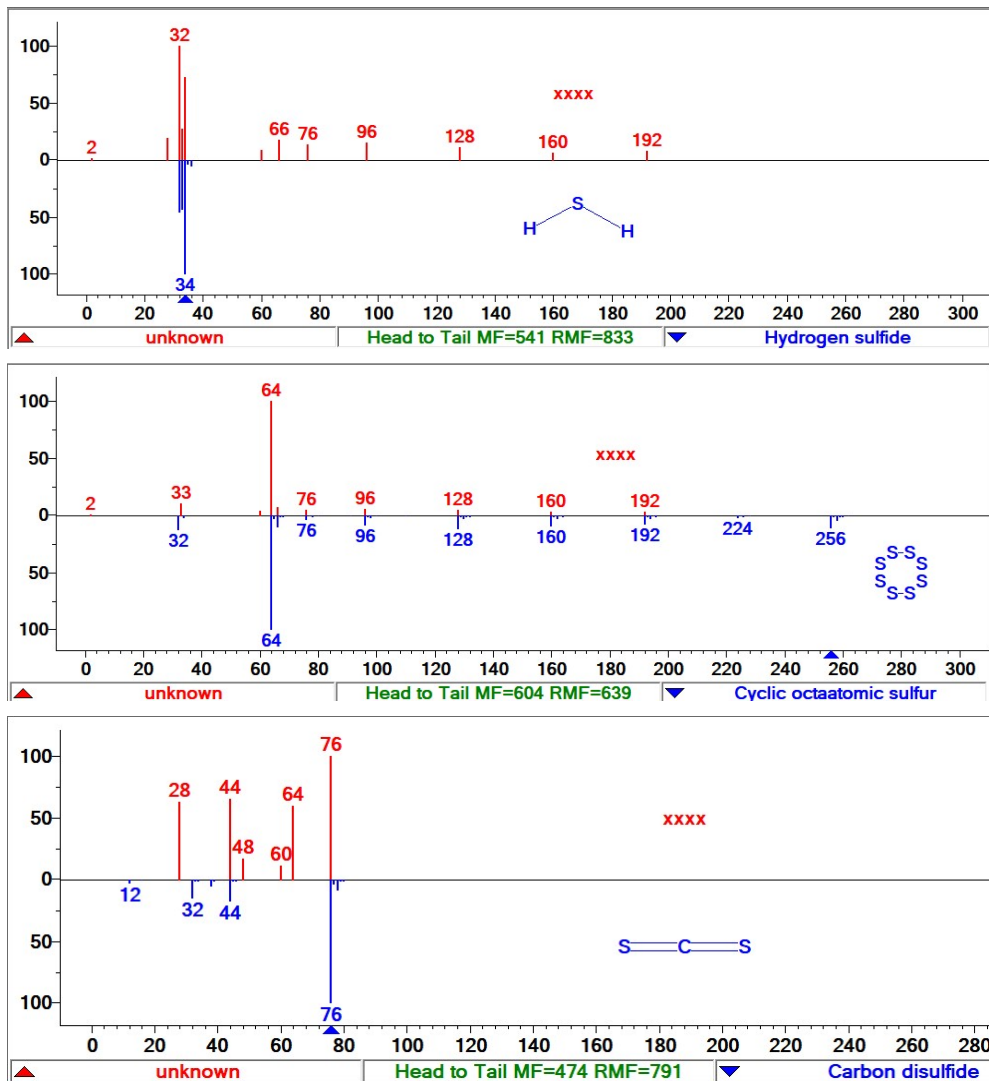
The graphite sample was heated in an inert atmosphere to 800°C, during which it exhibited two mass-loss steps of 0.14% and 0.026% with DTG peaks at 307°C and 562°C. The mass spectrometer detected the release of water (m/z 18), carbon dioxide (m/z 44) and sulfur (S₈= m/z 64). The release of m/z 32 and m/z 34 can be associated with the release of H₂S at 324°C. Mass number 76 indicates the release of CS₂ at 334°C, 398°C and 560°C. The release of sulfur was detected with an MS peak at 324°C.

Figure 2 shows the mass-loss curves in green with the corresponding traces of mass numbers m/z 18, 32, 44, 64 und 76.



2 Temperature-dependent mass change (TGA, green), rate of mass change (DTG, black) and mass numbers m/z 18, 32, 44, 64 and 76 (individually scaled) of the ultra-high-purity graphite sample

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- 3 (a) Mass spectra of the ultra-high-purity graphite sample at 243°C compared to the library spectrum of hydrogen sulfide
 (b) Mass spectra of the ultra-high-purity graphite sample at 300°C compared to the library spectrum of sulfur.
 (c) Mass spectra of the ultra-high-purity graphite sample at 567°C compared to the library spectrum of carbon disulfide.

Comparison of the spectra measured at different temperatures with the NIST library proves the release of the various compounds; see figure 3.

Summary

In conclusion, STA-MS coupling with measurements in TGA mode is a suitable method for detecting and

identifying impurities in ultra-high-purity graphite samples. It was possible to identify the simultaneous release of different sulfur compounds and to relate these to the mass-loss curve. With the help of this highly sensitive analytical method, the purity of different graphite grades can be studied and controlled, especially in applications such as batteries, where high purity is mandatory.