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Instrumental Gas Analysis (IGA) Services

IGA is an industry-standard technique for determining gas-forming elements (H, O, C, N, and S) in steels, metals, and alloys. IGA can also be a complementary technique for bulk and trace elemental analysis, and evolved gas analysis.

Instrumental gas analysis (IGA) measures mass fractions of gas-forming elements (C, H, O, N and S) present in samples from ppm to weight percentage levels. A high-temperature furnace is used to rapidly heat the sample under a flowing gaseous stream and releasing above elements from the sample for detection on a variety of detectors.

Carbon and sulfur are measured by combustion (Figure 1) and infrared detection. The analytical method is based on the complete oxidation of the solid sample by combustion in an oxygen plasma. The sample is placed in a ceramic crucible, along with high purity suppoort materials to promote complete oxidation, in a high frequency induction furnace where it is combusted. The combustion of the sample releases various gases, which are measured by infrared detectors. The sulfur content of the sample is determined by analyzing the SO_2. The carbon content is evaluated from analyzing CO and CO_2 gases released from the sample.

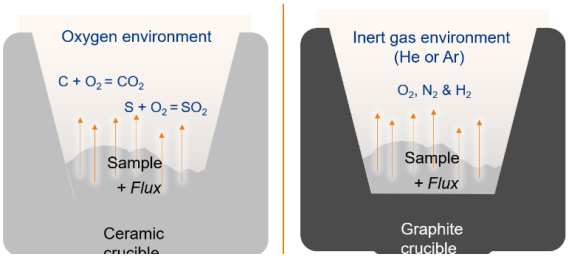


Figure 1: *Carbon and sulfur are measured based on combustion method for gas extraction (left). Oxygen, nitrogen, and hydrogen are measured using inert gas fusion gas extraction method (right).*

Nitrogen, oxygen and hydrogen are measured using inert gas fusion or hot extraction techniques (Figure 1). The sample is placed in a graphite crucible, along with high purity support materials to optimize sample heating/melting, and inserted into a furnace, where it is held between the electrodes. After purging with an inert gas (He or Ar), a high current is passed through the crucible, increasing the temperature in the crucible above 2000°C. Any gases generated in the furnace (CO, CO_2, N_2, H_2, or H_2O) are released into a flowing inert gas stream, which is directed to the appropriate detector: infrared or Thermal Conductivity Detector (TCD). Instrument calibration is performed using known reference materials.

Multiphase carbon and water determination is performed by programming multiple furnace steps to quantify the amount of carbon and water present in a sample. The furnace is purged with oxygen or nitrogen to create oxidizing or inert conditions during temperature programmed sample heating. In oxidizing conditions, all forms of carbon (except some carbides) are converted to CO_2. In inert conditions, a sample is combusted in a nitrogen environment to detect water and carbonate. A standard, slow temperature ramping is used to differentiate carbon and water release from different phases in unknown samples and can be further optimized for best results. Multiphase carbon determination is used, as an example, to verify the quality of the carbonaceous electrode, commonly known as the anode, in lithium-ion batteries

IGA Ideal Uses

- Accurate C, H, O, N and S measurements from ppm to weight% in solids,
- Fractional gas analysis to quantify H, O, and N released from various bonding chemistries and chemical environments.
- Surface, free, organic, inorganic carbon differentiation in unknown samples.
- Differentiate surface and bulk concentrations of H, O, and N

IGA Strengths

- Mature technique for high precision measurement of H
- Less complex and minimal sample preparation
- High throughput (20-40 mins/sample)
- Well-established technique for steel industry
- Can provide H and O environment information (surface or bulk) using temperature programs and reaction agents

IGA Limitations

- Destructive Analysis
- Larger sample mass required for trace level detection (0.5g to 1g)

Case Study: Carbon Fiber Reinforced Carbon Composites (CFRC)

Carbon Fiber Reinforced Carbon Composites (CFRCs) are also referred to as carbon/carbon composites. They consist of a carbon matrix holding together carbon or graphitic fibers. These high-performance materials find applications in aerospace and fusion technologies. This composite can have a range of physical properties based on various designing aspects that include the type of individual components used, the layering of the components, the orientation of the fibers within these components, and the way all these components are put together as a product (Figure 2). It is essential to have a survey of chemical purity to achieve desirable mechanical properties and reliability of the complex architecture of the product. Among the critical information required is carbon, hydrogen, oxygen and nitrogen composition and their outgassing at a high temperature. This example, demonstrates how the IGA technique can be used to derive such information.

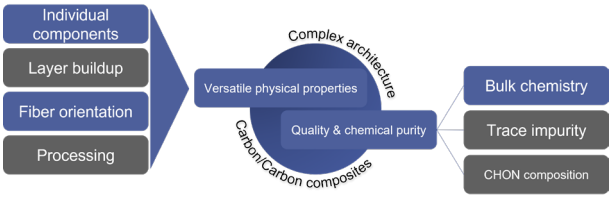
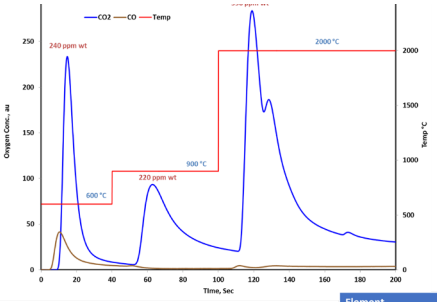


Figure 2: *Example of how the IGA technique can be used to derive the carbon, hydrogen, oxygen, and nitrogen composition and their outgassing at a high temperature.*



Total amount of outgassing H, O and N using well-established ASTM standards

Element	C	H	O	N
Average, wt%	≥99.5	0.029	0.11	0.065
%RSD (n = 3-5)	NA	4	10	12

Figure 3: *The plot shows oxygen concentration in composite is compared with heating time and temperature. The table shows average Concentration and RSD values of carbon, hydrogen, oxygen, and nitrogen.*

Carbon, hydrogen, oxygen, and nitrogen measurements are determined using a calibration performed by analyzing NIST traceable standards and standardized ASTM test methods. Figure 3 shows oxygen concentration in a composite material measured as a function of heating time and temperature. The distinct outgassing profiles observed for CO and CO_2 and two main oxygen-containing species show the presence of different oxygen chemistries and provide insight into how deoxygenation occurs for this composite. The table shows the average concentration of carbon, hydrogen, oxygen, and nitrogen measured following standardized ASTM test methods, using calibration performed by analyzing NIST traceable standards. Improved precision values were achieved enabling additional evaluation on this composite by using a

hydrogen to carbon ratio which is essential to determine the aromaticity of components.

Case Study: High-Precision Carbon Analysis for Carbon Fibers and Powder

Carbon fibers are made from acrylic fiber, which contains approximately 68% carbon. After carbonization and graphitization, carbon fibers are made with high elasticity. Therefore, the elasticity of carbon fiber depends on the carbon percentage in the carbon fiber. For example, fibers with standard elasticity have 90% or more carbon with nitrogen as the secondary element. Determination of carbon weight% therefore becomes essential to verify the quality of carbon fiber and a high precision method is used for measurement as opposed to the standard method.

In a standard carbon measurement blank, certified reference material, and samples are analyzed in sequence. However, for high precision measurements, a stricter statistical approach is adopted. After analyzing blanks, two reference materials are also analyzed: one with a higher and one with a lower carbon concentration than the expected concentration in the sample. More replicates of the sample are analyzed as and a reference material with the carbon concentration close to the measured concentration in the sample is analyzed to verify instrument drift. If instrument drift is detected, the entire method is repeated

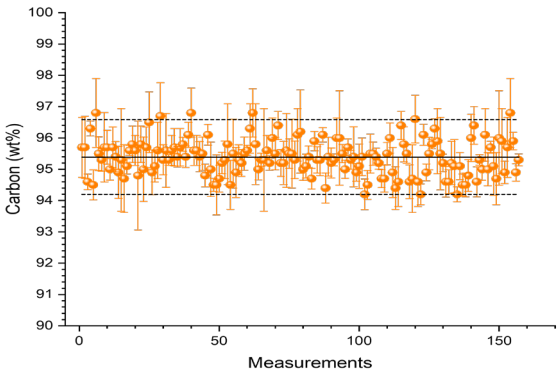


Figure 4: *The x-axis shows the number of measurements while the y-axis shows the carbon weight%.*

In this example, actual sample measurements using the high precision method over a three-year period were plotted (Figure 4). The data show high consistency in the measurements, verifying the method for determining > 90 weight% of carbon.

While the high precision method can take more than two hours to analyze one sample, as opposed to 30-40 minutes for the standard method, its consistency is essential to verify the quality of the material.

IGA Services at Eurofins EAG

IGA testing can be divided into two major categories: the well-established testing where standard ASTM test methods are used to analyze conventional samples (e.g., steel, titanium alloys, and refractory metals) and client-specific testing methods for unconventional samples (e.g., non-porous samples, battery electrodes) developed by highly experienced scientists going beyond standardized ASTM test methods. At Eurofins EAG, four state-of-the-art laboratories provide fast, accurate IGA services worldwide. Our Syracuse, NY, Toulouse, France, Eindhoven, Netherlands, and Shanghai, China facilities are operating daily under our well monitored quality systems.

EAG is a global leader for materials testing services with a broad range of modern instruments and expertise to take on most challenging materials and engineering related issues. We have an extensive knowledge base and skilled expertise that enables us to offer multi-disciplinary approaches that can be scaled to client-specific needs.