

Project 18

Thermogravimetric analysis of carbon nanotube materials

Objectives

1. Evaluate a proposed protocol for thermogravimetric analysis (TGA) of carbon nanotube populations, including for usability, reproducibility, and uncertainty.

2. Demonstrate use of the protocol to establish homogeneity of three different carbon nanotube samples, and determination of the primary oxidation temperature, residual mass, oxidation temperature of each peak(s).

Background

Thermogravimetric analysis (TGA) is an important measurement technique for carbon nanotube (CNT) containing samples, providing a qualitative assessment of thermal stability and homogeneity. TGA can give quantitative measure of the types of carbon species present as well as non-carbon impurities in a material.

This effort will support transition of the current documentary standard for TGA, ISO/TS 11308:2020 “Nanotechnologies - Characterization of carbon nanotube samples using thermogravimetric analysis”, from a technical specification to an international standard (IS) in ISO. This standard specifies how to prepare carbon nanotube materials for thermogravimetric

analysis, how to perform the measurement, and steps in the data interpretation. An interlaboratory comparison (ILC) study is needed to validate the measurement protocol as presented in this document.

Standardization needs

There is a need for standardized measurements of CNTs by TGA. Validation of published international standards has not been established; this measurement is an essential measurement in the manufacturing environment.

Work Programme

Initial work will be development and evolution of a specific measurement protocol based on ISO/TS 11308 for TGA of CNT materials applicable to multiple sample formats. Experimental ILC round(s) of TGA on provided CNT materials using the supplied measurement protocol will follow.

Provision of three (3) samples, including single-wall and multi-wall carbon nanotube samples is anticipated, along with several material format types, e.g., powder or fibers, to represent a variety of real-world materials. Measurands will include primary oxidation temperature, residual mass, oxidation temperature of other peaks, along with collection of

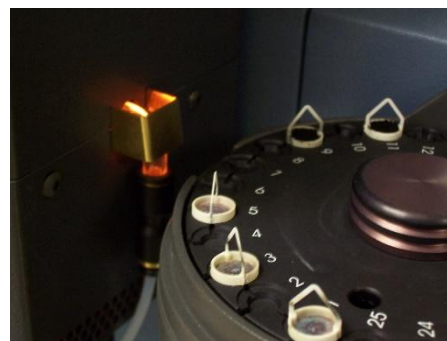


Figure: Picture of a TGA instrument

instrumental details, and any variances from the specified procedure. Reported values will be returned for central analysis in a provided spreadsheet. Statistical analysis of the results from participants will be conducted as consistent with ISO/TS 11308.

Deliverables and Dissemination

This ILC study will support revision of ISO/TS 11308 to an IS level document. A Peer Reviewed publication of ILC results with statistical analysis of the sample results will also be pursued. Summary of ILC results will also be included in ISO/IS 11308 revision (in progress as of 11/2023) and VAMAS and NIST reports.

Funding: Participants fund their own involvement in the project.

Reference: ISO/TS 11308:2020 “Nanotechnologies— Characterization of carbon nanotube samples using thermogravimetric analysis”

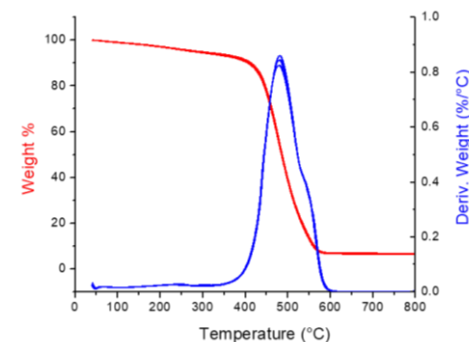


Figure: Example TGA of a single-wall carbon nanotube population

Status: Distribution of methods for comments, samples in Feb. 2025, additional participants welcomed.

For more information:

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