



BSI Standards Publication

Nanotechnologies — Determination of elemental impurities in samples of carbon nanotubes using inductively coupled plasma mass spectrometry

National foreword

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**Nanotechnologies — Determination
of elemental impurities in samples of
carbon nanotubes using inductively
coupled plasma mass spectrometry**

*Nanotechnologies — Dosage des impuretés dans les nanotubes en
carbone (CNTs) par spectrométrie de masse à plasma induit (ICP-MS)*





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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 229, *Nanotechnologies*.

This second edition cancels and replaces the first edition (ISO/TS 13278:2011), which has been technically revised.

Introduction

Metal particle catalysts are essential in the mass production of nanotubes by chemical vapour deposition (CVD).^{[1][2][3]} Removal of these residual catalysts (typically Fe, Co, and/or Ni) after CNT production is one of the key challenges for the application of CNTs in many fields.^[4] After complicated purification steps, the concentration of such catalysts is measured. It is of great concern that the results of toxicological and ecological impact studies of carbon nanotubes could be misinterpreted due to the presence of impurities in the test materials^{[5][6][7]} and that the metals could be released into the environment during disposal of the product by means of combustion or other ways. Additionally, the actual desired performance of nanotube materials might depend on these impurities, which is the reason why it is so crucial to use reliable techniques to determine their content in these materials.

Currently available methods for analysis of the purity of CNTs include neutron activation analysis (NAA), transmission electron microscopy (TEM) with electron energy loss spectroscopy (EELS), scanning electron microscopy (SEM) with energy dispersive X-ray analysis (EDX), Raman spectroscopy, X-ray photoelectron spectroscopy (XPS), thermogravimetric analysis (TGA), and X-ray fluorescence (XRF) spectrometry^{[8][9][10][11][12]}. A number of these techniques for the characterization of single-wall and/or multiwall carbon nanotubes are the subject of standardization within ISO/TC 229, including SEM (see ISO/TS 10798), TEM (see ISO/TS 10797), and measurement methods for the characterization of multiwall carbon nanotubes (see ISO/TR 10929).

However, each method has its limitations for determination of elemental impurities. ICP-MS is capable of providing highly accurate and precise results, while being widely available in most commercial laboratories. However, using conventional solution sample introduction to ICP-MS, the sample has to be completely solubilized. Digestion of some types of samples requires thorough pretreatment schemes. Standard sample preparation procedures are available for routine matrix types, including soils, rocks and biological specimens. In the case of carbon nanotubes, because of their extremely stable structure and possible encapsulation of metals in structural defects, it is necessary that the materials go through special destructive pretreatments before analysis by ICP-MS^{[1][16][17][18]}.

The purpose of this document is to provide guidelines for optimized sample pretreatment methods for single-wall carbon nanotubes (SWCNTs) and multiwall carbon nanotubes (MWCNTs) to enable accurate and quantitative determinations of elemental impurities using ICP-MS. An example of the determination of elemental impurities in commercially produced carbon nanotubes, using the methods described, is given in [Annex A](#).

Nanotechnologies — Determination of elemental impurities in samples of carbon nanotubes using inductively coupled plasma mass spectrometry

1 Scope

This document provides methods for the determination of residual elements other than carbon in samples of single-wall carbon nanotubes (SWCNTs) and multiwall carbon nanotubes (MWCNTs) using inductively coupled plasma mass spectrometry (ICP-MS).

The purpose of this document is to provide optimized digestion and preparation procedures for SWCNT and MWCNT samples in order to enable accurate and quantitative determinations of elemental impurities using ICP-MS.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO/TS 80004-3, *Nanotechnologies — Vocabulary — Part 3: Carbon nano-objects*

ISO/TS 80004-6, *Nanotechnologies — Vocabulary — Part 6: Nano-object characterization*

3 Terms, definitions, symbols and abbreviations

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TS 80004-3, ISO/TS 80004-6 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

3.1.1

ICP-MS

inductively coupled plasma mass spectrometry method in which a high-temperature discharge generated in flowing argon by an alternating magnetic field induced by a radio-frequency (RF) load coil that surrounds the tube carrying the gas is detected using a mass spectrometer

Note 1 to entry: ICP-MS permits quantitative determinations of trace, minor and major elements in samples pertaining to almost every field of application of analytical chemistry.

3.1.2

elemental impurity

element other than carbon that is present in a carbon nanotube sample

Note 1 to entry: Such impurities are primarily remnants of metal catalysts used during large-scale production of CNTs.