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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 spectroscopie de masse à plasma induit (ICP-MS)*



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Contents

	Page
Foreword	v
Introduction	vi
1 Scope	1
2 Normative references	1
3 Terms, definitions, symbols and abbreviations	1
3.1 Terms and definitions.....	1
3.2 Symbols and abbreviations.....	2
4 Samples and reagents	3
4.1 General.....	3
4.2 Samples.....	3
4.3 Reagents.....	3
4.3.1 General.....	3
4.3.2 Purity of acids.....	3
4.3.3 Purity of reagents.....	3
4.3.4 Purity of water.....	3
4.4 Stock solutions.....	3
4.4.1 General.....	3
4.4.2 ICP-MS calibration standard stock solution No. 1.....	3
4.4.3 ICP-MS calibration standard stock solution No. 2.....	4
4.4.4 ICP-MS calibration standard stock solution No. 3.....	4
4.5 Stock spike solutions.....	4
4.5.1 General.....	4
4.5.2 Stock spike solution No. 1.....	4
4.5.3 Stock spike solution No. 2.....	4
4.6 Stock internal standard solutions.....	4
4.6.1 General.....	4
4.6.2 Internal standard No. 1.....	4
4.6.3 Internal standard No. 2.....	4
4.6.4 Internal standard No. 3.....	4
4.6.5 Internal standard No. 4.....	4
4.6.6 Internal standard No. 5.....	5
4.7 Stock standard tuning solutions.....	5
4.7.1 General.....	5
4.7.2 Standard tuning solution No. 1.....	5
4.7.3 Standard tuning solution No. 2.....	5
4.7.4 Standard tuning solution No. 3.....	5
4.7.5 Standard tuning solution No. 4.....	5
4.7.6 Standard tuning solution No. 5.....	5
5 Apparatus	5
6 Sample pretreatment	5
6.1 Sample preparation for ICP-MS analysis.....	5
6.2 Wet digestion under high pressure.....	6
6.3 Combined dry ashing and acid digestion.....	7
6.4 Microwave-assisted digestion.....	7
7 Experimental procedures	8
7.1 Instrumental settings for ICP-MS.....	8
7.2 Interferences in ICP-MS.....	8
7.3 Isotope selection.....	9
7.4 Standard calibration curve.....	9
7.5 Method recovery evaluation using standard addition.....	9
7.6 The use of internal standards in ICP-MS.....	9

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8	Data analysis and interpretation of results	10
	8.1 Calculation of elemental impurity mass fraction in test sample.....	10
	8.2 Calculation of the spike (method) recovery.....	10
9	Uncertainty estimation	11
10	Test report	11
Annex A (informative) Example of determination of elemental impurities in carbon nanotubes ...		13
Bibliography		19

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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).

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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 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^{[12][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](#).