

ICS 71.040

CCS A 40



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 35418-2017

**Nanotechnologies -- Determination of elemental impurities in
samples of carbon nanotubes -- Inductively coupled plasma
mass spectrometry**

纳米技术 碳纳米管中杂质元素的测定 电感耦合等离子体质谱法

(English Translation)

Issued on: December 29, 2017

Implemented on: April 1, 2018

**Issued by: General Administration of Quality Supervision, Inspection and
Quarantine of the People's Republic of China, Standardization
Administration of the People's Republic of China**

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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard uses the translation method identical with ISO/T S13278.2011 "Nanotechnology Applications Inductively Coupled Plasma-Mass Spectrometry Determination Impurity elements in carbon nanotubes".

This standard made the following editorial changes.

--- Modify the standard name.

This standard proposed by the Chinese Academy of Sciences.

This standard by the National Institute of Technology Standardization Technical Committee (SAC/TC279) centralized.

The main drafting units of this standard. National Nano Science Center, Institute of High Energy Physics, Chinese Academy of Sciences.

Introduction

Inductively Coupled Plasma Mass Spectrometry (ICP-MS) is a widely accepted multi-element analytical technique for rapid and accurate detection of samples Trace elements. Compared with atomic absorption spectrometry and inductively coupled plasma atomic emission spectrometry (ICP-AES), inductively coupled plasma Mass spectrometry has many advantages, such as. matrix interference, high sensitivity, low detection limit.

Carbon nanotubes (CNTs) have unique physical and chemical properties that have potential applications in many fields and have attracted a great deal of research interest.

The use of chemical vapor deposition mass production of carbon nanotubes, the need to use metal particles as a catalyst [1-3]. Remove residual catalyst (eg Fe, Co, Ni) for the application of carbon nanotubes is of great significance [4]. After complicated purification steps, the catalyst content was determined. Due to gold the presence of a catalyst can be released with the use of carbon nanotubes, resulting in toxicological and ecological consequences of carbon nanotubes Ring [5-7]. In addition, the function of carbon nanotubes may depend on these catalyst impurities, so reliable techniques must be used to detect the impurity content.

Current methods of analyzing the purity of carbon nanotubes include neutron activation analysis (NAA), transmission electron transport loss (EELS) (TEM), scanning electron microscopy (SEM) with energy dispersive X-ray spectroscopy (EDX), Raman spectroscopy, X-ray photoelectron XPS, TGA and XRF spectroscopy [8-12]. ISO/TC229 enacted some characterization of single-walled carbon Technical method standards for nanotubes and multi-walled carbon nanotubes, such as SEM (ISO/T S10798), TEM (ISO/T S10797), multi-Metric tube characterization method (ISO/T R10929).

However, each detection method has its limitations. TGA can only reflect the change of mass under heated conditions, the impact of factors, including equipment, experimental conditions and sample factors, so the reliability of the test results is not high; neutron activation analysis is based on nuclear anti-Quantitative and qualitative methods should be used. The method has the advantages of high sensitivity, accuracy and precision, and can simultaneously measure trace, trace and main elements with sensitivity up to 10^{-6} ~ 10^{-9} , therefore, often used as a standard reference formulation. However, due to the need for large nuclear reactor irradiation samples, neutron activation analysis Applications have been greatly limited, in addition, high cost and radiation hazards, longer analysis cycles, complex operating techniques, poor practicality, the application of neutron activation analysis method. Inductively coupled plasma mass spectrometry (ICP-MS) also has high sensitivity, accuracy and precision Degree, widely used in laboratory testing. It requires samples to be completely digested, and traditional digestion methods are suitable for soil, rock and biological sample analysis.

Carbon nanotubes have an extremely stable structure, and metal residues may be

encapsulated in the carbon shell structure, therefore, it is necessary to use a specific sample pre-Treatment [12-15]. Inductively coupled plasma mass spectrometry is better than graphite furnace atomic absorption spectrometry and inductively coupled plasma atomic emission spectrometry Has a higher sensitivity.

This standard is the application of inductively coupled plasma mass spectrometry accurate detection of single-walled carbon nanotubes and multi-walled carbon nanotubes residual impurity elements, Provides the best sample preparation method. Appendix A is an example of method application.

Determination of impurity elements in nanotechnology carbon nanotubes Inductively coupled plasma mass spectrometry

1 Scope

This standard specifies the use of inductively coupled plasma mass spectrometry (ICP-MS) determination of single-walled carbon nanotubes (SWCNT) and multi-walled carbon nano Method for remaining impurity elements in a tube (MWCNT).

This standard provides optimized digestion and pretreatment methods for single-walled carbon nanotubes and multi-walled carbon nanotube samples to apply inductive coupling Plasma mass spectrometry accurate quantitative determination of impurity elements.

2 Normative references

The following documents for the application of this document is essential. For dated references, only the dated version applies to this article Pieces. For undated references, the latest edition (including all amendments) applies to this document.

Nanotechnology - Part 3. Carbon nanostructures (ISO/T S80004-3.2010, IDT) GB/T 30544.3-2015

3.1 Terms and definitions

GB/T 30544.3-2015 as defined in the following terms and definitions apply to this document.

3.1.1

Inductively coupled plasma source Atmospheric pressure generated by the RF electromagnetic field in argon plasma components.

3.1.2

Inductively coupled plasma mass spectrometry inductively coupled mass spectrometry ICP-MS by the injection system, inductively coupled plasma source, plasma/vacuum interface, mass spectrometer (including ion focusing, separation and detection system)

constitute.

Note: Inductively coupled plasma mass spectrometry can quantify trace, trace and major elements in a sample and is suitable for analytical chemistry.

3.1.3

Impurity element elemental impurity Non-carbon elements in the sample, elements that appear in the sample and do not exist as carbon nanotubes.

Note 1: These impurities are mainly the residues of metal catalysts used in mass production of carbon nanotubes.

Note 2: Amorphous carbon can be used as another impurity in carbon nanotubes and is outside the scope of this document.

3.2 Symbols and abbreviations

The following symbols and abbreviations apply to this document.

CCT Collision Cell Technology (collision cell technology) ϕ_i injection volume sensitivity coefficient, ϕ_i refers to $df/d\phi_i$ (sensitivity coefficient for input quantity, ϕ_i , defined as $df/d\phi_i$) CNT carbon nanotube (carbon nanotube)