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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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# Introduction

Inductively coupled plasma mass spectrometry (ICP-MS) is a well-established multi-element analytical technique used for fast, precise and accurate determinations of trace elements. ICP-MS has many advantages over other elemental analysis techniques such as atomic absorption and ICP atomic emission spectrometry (ICP-AES). The ability to handle both simple and complex matrices with a minimum of matrix interferences is due to the high temperature of the ICP source. ICP-MS also has high sensitivity and superior detection capability.

Owing to their unusual physical and chemical properties, and potential applications in a number of areas, interest in carbon nanotubes (CNTs) has shown tremendous growth in the past decade. 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 (ISO/TS 10798), TEM (ISO/TS 10797[1)]), and measurement methods for the characterization of multiwall carbon nanotubes (ISO/TR 10929[2)]).

However, each method has its limitations for determination of elemental impurities. TGA can only provide a gross estimation of metal content. NAA is a quantitative and qualitative method based on nuclear reactions between neutrons and target nuclei. This method provides high efficiency for the precise and simultaneous determination of a number of major, minor and trace elements in different types of samples in the parts per billion ($10^{-9}$) to parts per million ($10^{-6}$) range. Moreover, due to the superior figures of merit, including high accuracy, good precision and no matrix blank requirement, NAA is widely used in the certification of reference materials. NAA is, however, not a technique that is readily available, being not only a highly specialised field of analysis, but also requiring access to a nuclear reactor. ICP-MS, on the other hand, is also 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 solubilised. 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][13][14][15]. ICP-MS offers better sensitivity than graphite furnace atomic absorption spectrometry with the multi-element speed of ICP-AES.

The purpose of this Technical Specification 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.

1) Under preparation.

2) Under preparation.