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Nanotechnologies — Measurement technique matrix for the characterization of nano-objects

Nanotechnologies — Matrice de méthodes de mesure pour les nano-objets manufacturés

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Foreword

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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 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.

The committee responsible for this document is ISO/TC 229, *Nanotechnologies*.

Introduction

This document connects the nano-object parameters that most commonly need to be measured with corresponding measurement techniques. This document will be a useful tool for nanotechnology interested parties to rapidly identify relevant information for measuring nano-objects. The common nano-object parameters are listed along the top row of the Quick-Use-Matrix (see Table 1). If a measurement technique listed in the first column of the matrix is applicable, the box in the matrix will be marked. Once a measurement technique of interest is identified, it is recommended that the reader then enter this document's body of text (see Clause 5), where you will find an alphabetical listing of the measurement techniques and descriptions of the advantages, limitations, relevant standards, measurand(s), and applicable nano-object parameters of each technique.

As scientific advances are made and additional commercial measurement techniques become available, this document will be periodically reviewed and updated to maintain its relevance.

Many of the techniques listed in this document have not been validated through round-robin testing or any other means for the measurement of nano-objects. This document is intended as a starting point and resource to help identify potentially useful and relevant techniques; it is not an exhaustive or primary source. It is recommended that once a technique has been identified, the reader refers to relevant international standards and conducts a literature search for similar or comparable applications. Other sources of information include instrument manufacturer's applications notes and technical literature.

Nanotechnologies — Measurement technique matrix for the characterization of nano-objects

1 Scope

This document provides a matrix that guides users to commercially available techniques relevant to the measurements of common physiochemical parameters for nano-objects. Some techniques are also applicable to nanostructured materials.

NOTE Guidance on sample separation and preparation is given in Annex A.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TS 80004-1, 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 http://www.iso.org/obp

3.1 General terms

3.1.1
dispersion
heterogeneous system in which a finely divided material is distributed in another material

[SOURCE: ISO 472:2013, 2.288]

3.1.2
measurand
quantity intended to be measured

[SOURCE: ISO Guide 99:2007, 2.3]

3.1.3
nano-object
discrete piece of material with one, two or three external dimensions in the nanoscale

Note 1 to entry: The second and third external dimensions are orthogonal to the first dimension and to each other.

[SOURCE: ISO/TS 80004-2:2015, 2.2]

3.1.4
nanostructured material
material having internal or surface structure in the nanoscale

Note 1 to entry: If external dimensions are in the nanoscale, the term *nano-object* (3.1.3) is recommended.

[SOURCE: ISO/TS 80004-4:2011, 2.11]

3.2 Nano-object parameters

3.2.1
chemical composition
identity and possible quantification of a material's composite parts and its impurities

3.2.2
concentration
content of nano-objects in a sample, quantified as number, area, volume or mass content

Note 1 to entry: Transformation calculation from one type of quantity to another (e.g. number to volume) requires the knowledge of the complete particle size distribution and the assumption of a size-independent shape, porosity or density[6].

3.2.3
crystal property
effect due to the presence of three-dimensional order at the level of molecular dimensions

Note 1 to entry: See Reference [1].

3.2.4
electrokinetic potential
difference in electric potential between that at the slipping plane and that of the bulk liquid

Note 1 to entry: See Reference [7].

Note 2 to entry: Electrokinetic potential is expressed in volts.

Note 3 to entry: Often called "zeta potential".

3.2.5
shape
external geometric form of a particle

Note 1 to entry: See Reference [8].

Note 2 to entry: For quantitative description by macroshape, mesoshape or microshape descriptors, see Reference [9].

3.2.6
size
linear dimensions of a particle determined by a specified measurement method and under specified measurement conditions

Note 1 to entry: See Reference [10].

Note 2 to entry: Different methods of analysis are based on the measurement of different physical properties. Independent of the particle property actually measured, the particle size is reported as a linear dimension, e.g. as the equivalent spherical diameter.

Note 3 to entry: Examples of size descriptors are those based at the opening of a sieve or a statistical diameter, e.g. the Feret diameter, measured by image analysis.

Note 4 to entry: In ISO 9276-1, the symbol x is used to denote the particle size. However, it is recognized that the symbol d is also widely used to designate these values. Therefore, the symbol x can be replaced by d.

3.2.7
particle size distribution
cumulative distribution of particle concentration as a function of particle size

[SOURCE: ISO 14644-1:2015, 3.2.4]

Note 1 to entry: Particle size distribution is of statistical nature so it can be expressed as a function of particle size[11].

Note 2 to entry: The type of quantity (number, area, volume or mass) as well as the measurand, the measured equivalent spherical diameter, shall be indicated.

Note 3 to entry: Particle size distribution can be expressed as cumulative distribution or a distribution density (distribution of the fraction of material in a size class, divided by the width of that class).

3.2.8
surface area
extent of available surface area as determined by given method under stated conditions

Note 1 to entry: See Reference [12].

Note 2 to entry: Surface area is the quantity of accessible surface of a sample when exposed to either gaseous or liquid adsorbate phase. Surface area is conventionally expressed as a mass-specific surface area or as volume-specific surface area where the total quantity of area has been normalized either to the sample's mass or volume[13].

4 Parameters included in the matrix

Several comprehensive literature searches, input from ISO/TC 229 experts, and a review of current ISO work have identified frequently sought nano-object parameters that are listed across the top of the Quick-Use-Matrix (see Table 1). The parameters listed from left to right are in alphabetical order. A single technique alone often cannot provide sufficient information about all parameters of interest for a particular nano-object under study, nor is one technique likely to fully capture relevant information for a single parameter. Therefore, when available, it is recommended that more than one technique should be used for any parameter investigation.

Table 1 — Quick-Use-Matrix[4]

Technique									
	Acronym	Chemical composition	Concentration	Crystal properties	Electrokinetic potential	Shape	Size	Size distribution	Surface area
Acoustic spectroscopy			+				+	+	
Analytical centrifugation	AC							+	
Aerosol particle mass analyser	AMS		+				+		
Auger electron spectroscopy (scanning)	AES	+	+			+	+		
Brunauer-Emmett-Teller	BET								+
Condensation particle counter	CPC		+						
Differential mobility analysis system[a]	DMAS		+				+	+	
Differential scanning calorimetry	DSC	+							
Dynamic light scattering[b]	DLS						+	+	
Electroacoustic spectroscopy					+				

a Requires a special detector or additional instrumentation to obtain the desired parameter.

b Light scattering technique.

Table 1 *(continued)*

Technique	Acronym	Chemical composition	Concentration	Crystal properties	Electrokinetic potential	Shape	Size	Size distribution	Surface area
Electron energy loss spectroscopy[a]	EELS	+							
Electrophoresis/ capillary electrophoresis					+				
EM based X-ray spectrometry[a]	EDX/ EDS/WDS	+							
Field flow fractionation[a]	FFF	+	+				+	+	
Fluorescence spectroscopy	FL		+						
Fourier transform infrared spectroscopy/ imaging	FTIR	+							
Induced grating method	IG						+	+	
Inductively coupled plasma–mass spectrometry and single particle ICP-MS	ICP-MS	+	+				+	+	
Laser diffraction[b]							+	+	
Liquid chromatography–mass spectrometry	LC-MS	+	+						
Particle tracking analysis	PTA				+		+	+	
Optical absorption spectroscopy	UV/Vis/ NIR	+	+				+		
Quartz microbalances	QCM						+		
Raman spectroscopy/ imaging		+		+					
Resonant mass measurement	RMM						+	+	
Scanning electron microscopy[a]	SEM	+		+		+	+	+	
Scanning probe microscopy	SPM/AFM					+	+	+	
Secondary ion mass spectrometry	SIMS		+						
Small angle X-ray scattering	SAXS					+	+	+	

[a] Requires a special detector or additional instrumentation to obtain the desired parameter.

[b] Light scattering technique.

Table 1 *(continued)*

Technique	Acronym	Chemical composition	Concentration	Crystal properties	Electrokinetic potential	Shape	Size	Size distribution	Surface area
Static light scattering[b]	SLS/SMLS		+				+		
Single particle light interaction methods[b]			+				+	+	
Thermogravimetric analysis	TGA	+							
Transmission electron microscopy[a]	TEM	+		+		+	+	+	
X-ray diffraction	XRD			+			+		
X-ray photoelectron spectroscopy	XPS	+	+						

[a] Requires a special detector or additional instrumentation to obtain the desired parameter.

[b] Light scattering technique.

5 Measurement techniques included in the matrix

5.1 General

In the following subclauses, measurement techniques are listed in alphabetical order (with some exceptions where similar techniques are grouped together). Included under each technique are the nano-object parameters that can be measured by the technique and a listing of the technique's advantages, limitations, measurands, and relevant standards. Generally, a single technique cannot provide characterization of all nano-object parameters of interest. Also, a technique may be useful to determine other parameters not listed. For comprehensive analysis of a single parameter, use of multiple techniques, where applicable, is highly recommended.

In some cases, there are multiple techniques that share a similar underlying theory of operation or physical phenomenon, but which have differences that make them uniquely applicable to different types of materials and measurement conditions. For example, different electromagnetic scattering techniques are better suited for the analysis of different materials (e.g. metals, polymers or refractories) or materials in different states (e.g. suspended in a liquid or incorporated into a solid matrix).

5.2 Acoustic spectroscopy

5.2.1 Description

The basic concept is to measure the frequency-dependent attenuation or velocity of ultrasound as it propagates through a heterogeneous sample, e.g. colloids, dispersions and emulsions. There are two main types of vibrations: free and forced. Free vibrations are the natural or normal modes of vibration for a substance. Forced vibrations are caused by some sort of excitation to make the analyte resonate beyond its normal modes.

Both acoustic and electroacoustic spectroscopies (see 5.4) are linked to a sound propagation through a heterogeneous system such as a suspension or an emulsion. An acoustic spectrometer measures only the changes in the properties of the sound wave, whereas an electroacoustic spectrometer deals with connection between electrodynamic phenomena and the sound wave pressure field. Acoustic and electroacoustic spectroscopies are independent methods because the attenuation has little effect

