PD ISO/TR 18196:2016

BSI Standards Publication

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

Documents
Document Sharing Hub

Full document details: PD ISO/TR 18196:2016

National foreword

This Published Document is the UK implementation of ISO/TR 18196:2016.

The UK participation in its preparation was entrusted to Technical Committee NTI/1, Nanotechnologies.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

© The British Standards Institution 2017.
Published by BSI Standards Limited 2017

ISBN 978 0 580 82364 0
ICS 07.120

Compliance with a British Standard cannot confer immunity from legal obligations.

This Published Document was published under the authority of the Standards Policy and Strategy Committee on 28 February 2017.

Amendments/corrigenda issued since publication

Date	Text affected

Documents
Document Sharing Hub

Full document details: PD ISO/TR 18196:2016

TECHNICAL REPORT

ISO/TR 18196

First edition
2016-11-15

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

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

Reference number
ISO/TR 18196:2016(E)

Documents
Document Sharing Hub

© ISO 2016

Full document details: PD ISO/TR 18196:2016

COPYRIGHT PROTECTED DOCUMENT

© ISO 2016, Published in Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub

Full document details: PD ISO/TR 18196:2016

Contents

Page

Foreword ... **viii**
Introduction ... **ix**
1 Scope ... **1**
2 Normative references ... **1**
3 Terms and definitions ... **1**
3.1 General terms ... 1
3.2 Nano-object parameters ... 2
4 Parameters included in the matrix ... **3**
5 Measurement techniques included in the matrix ... **5**
5.1 General ... 5
5.2 Acoustic spectroscopy ... 5
5.2.1 Description ... 5
5.2.2 Nano-object parameters ... 6
5.2.3 Advantages ... 6
5.2.4 Limitations ... 6
5.2.5 Measurand ... 6
5.2.6 Relevant standards ... 6
5.3 Analytical centrifugation (AC) ... 6
5.3.1 Description ... 6
5.3.2 Nano-object parameters ... 7
5.3.3 Advantages ... 7
5.3.4 Limitations ... 7
5.3.5 Measurand ... 7
5.3.6 Relevant standards ... 7
5.4 Electroacoustic spectroscopy ... 7
5.4.1 Description ... 7
5.4.2 Nano-object parameters ... 7
5.4.3 Advantages ... 8
5.4.4 Limitations ... 8
5.4.5 Measurand ... 8
5.4.6 Relevant standards ... 8
5.5 Aerosol particle mass analyser (AMS) ... 8
5.5.1 Description ... 8
5.5.2 Nano-object parameters ... 9
5.5.3 Advantages ... 9
5.5.4 Limitations ... 9
5.5.5 Measurand ... 9
5.5.6 Relevant standards ... 9
5.6 Auger electron spectroscopy (AES) ... 9
5.6.1 Description ... 9
5.6.2 Nano-object parameters ... 9
5.6.3 Advantages ... 10
5.6.4 Limitations ... 10
5.6.5 Measurand ... 10
5.6.6 Relevant standards ... 10
5.7 Brunauer-Emmett-Teller (BET) method for physical adsorption — Surface area determination ... 10
5.7.1 Description ... 10
5.7.2 Nano-object parameters ... 11
5.7.3 Advantages ... 11
5.7.4 Limitations ... 11
5.7.5 Measurand ... 11

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD ISO/TR 18196:2016

5.7.6 Relevant standards ... 11
5.8 Condensation particle counter (CPC) ... 11
5.8.1 Description ... 11
5.8.2 Nano-object parameters ... 12
5.8.3 Advantages ... 12
5.8.4 Limitations ... 12
5.8.5 Measurand ... 12
5.8.6 Relevant standards ... 12
5.9 Differential mobility analysis system (DMAS) ... 12
5.9.1 Description ... 12
5.9.2 Nano-object parameters ... 13
5.9.3 Advantages ... 13
5.9.4 Limitations ... 13
5.9.5 Measurand ... 13
5.9.6 Relevant standards ... 13
5.10 Differential scanning calorimetry (DSC) ... 13
5.10.1 Description ... 13
5.10.2 Nano-object parameters ... 13
5.10.3 Advantages ... 13
5.10.4 Limitations ... 14
5.10.5 Measurand ... 14
5.10.6 Relevant standards ... 14
5.11 Dynamic light scattering (DLS) ... 14
5.11.1 Description ... 14
5.11.2 Nano-object parameters ... 15
5.11.3 Advantages ... 15
5.11.4 Limitations ... 15
5.11.5 Measurand ... 15
5.11.6 Relevant standards ... 15
5.12 Electron energy loss spectroscopy (transmission EELS) ... 16
5.12.1 Description ... 16
5.12.2 Nano-object parameters ... 16
5.12.3 Advantages ... 16
5.12.4 Limitations ... 16
5.12.5 Measurand ... 16
5.12.6 Relevant standards ... 16
5.13 Electrophoresis/capillary electrophoresis ... 16
5.13.1 Description ... 16
5.13.2 Nano-object parameters ... 18
5.13.3 Advantages ... 18
5.13.4 Limitations ... 18
5.13.5 Measurands ... 18
5.13.6 Relevant standards ... 18
5.14 Energy dispersive X-ray spectrometry (EDS/EDX and WDS) ... 18
5.14.1 Description ... 18
5.14.2 Nano-object parameters ... 18
5.14.3 Advantages ... 19
5.14.4 Limitations ... 19
5.14.5 Measurand ... 19
5.14.6 Relevant standards ... 19
5.15 Field flow fractionation (FFF) ... 19
5.15.1 Description ... 19
5.15.2 Nano-object parameters ... 19
5.15.3 Advantages ... 20
5.15.4 Limitations ... 20
5.15.5 Measurand ... 20
5.15.6 Relevant standards ... 20
5.16 Fluorescence spectroscopy ... 20

© ISO 2016 – All rights reserved
Documents
Document Sharing Hub
Full document details: PD ISO/TR 18196:2016

5.16.1 Description 20
5.16.2 Nano-object parameters 20
5.16.3 Advantages 20
5.16.4 Limitations 21
5.16.5 Measurand 21
5.16.6 Relevant standards 21
5.17 Fourier transform infrared (FT-IR) spectroscopy and FT-IR imaging 21
5.17.1 Description 21
5.17.2 Nano-object parameters 21
5.17.3 Advantages 21
5.17.4 Limitations 22
5.17.5 Measurand 22
5.17.6 Relevant standards for FT-IR 22
5.18 Induced grating method (IG) 22
5.18.1 Description 22
5.18.2 Nano-object parameters 22
5.18.3 Advantages 22
5.18.4 Limitations 22
5.18.5 Measurand 23
5.18.6 Relevant standards 23
5.19 Inductively coupled plasma–mass spectrometry (ICP-MS) and single particle inductively coupled plasma–mass spectrometry (SP-ICP-MS) 23
5.19.1 Description 23
5.19.2 Nano-object parameters 23
5.19.3 Advantages 23
5.19.4 Limitations 23
5.19.5 Measurand 24
5.19.6 Relevant standards 24
5.19.7 Nano-hyphenated ICP/MS techniques 24
5.20 Laser diffraction 25
5.20.1 Description 25
5.20.2 Nano-object parameters 25
5.20.3 Advantages 25
5.20.4 Limitations 25
5.20.5 Measurand 25
5.20.6 Relevant standards 25
5.21 Liquid chromatography–mass spectrometry (LC-MS) 26
5.21.1 Description 26
5.21.2 Nano-object parameters 26
5.21.3 Advantages 26
5.21.4 Limitations 26
5.21.5 Measurand 26
5.21.6 Relevant standards 26
5.22 Particle tracking analysis (PTA) 26
5.22.1 Description 26
5.22.2 Nano-object parameters 27
5.22.3 Advantages 27
5.22.4 Limitations 27
5.22.5 Measurand 27
5.22.6 Relevant standards 28
5.23 Optical absorption spectroscopy (UV/Vis/NIR) 28
5.23.1 Description 28
5.23.2 Nano-object parameters 28
5.23.3 Advantages 28
5.23.4 Limitations 28
5.23.5 Measurand 29
5.23.6 Relevant standards for 29
5.24 Quartz crystal microbalance (QCM) 29

AVA Documents
Document Sharing Hub

© ISO 2016 – All rights reserved

Full document details: PD ISO/TR 18196:2016

5.24.1 Description 29
5.24.2 Nano-object parameters 29
5.24.3 Advantages 29
5.24.4 Limitations 29
5.24.5 Measurand 29
5.24.6 Relevant standards 29
5.25 Raman spectroscopy/Raman imaging 30
5.25.1 Description 30
5.25.2 Nano-object parameters 30
5.25.3 Advantages 30
5.25.4 Limitations 30
5.25.5 Measurand 30
5.25.6 Relevant standards for Raman 30
5.26 Resonant mass measurement (RMM) 31
5.26.1 Description 31
5.26.2 Nano-object parameters 31
5.26.3 Advantages 31
5.26.4 Limitations 31
5.26.5 Measurand 31
5.26.6 Relevant standards 31
5.27 Scanning electron microscopy (SEM) 31
5.27.1 Description 31
5.27.2 Nano-objects parameters 32
5.27.3 Advantages 32
5.27.4 Limitations 32
5.27.5 Measurand 33
5.27.6 Relevant standards 33
5.28 Scanning probe microscopy (SPM) 33
5.28.1 Description 33
5.28.2 Nano-object parameters 34
5.28.3 Advantages 34
5.28.4 Limitations 34
5.28.5 Measurand(s) 35
5.28.6 Relevant standards 35
5.29 Secondary ion mass spectrometry (SIMS) and Time of Flight SIMS (TOF-SIMS) 35
5.29.1 Description 35
5.29.2 Nano-object parameters 35
5.29.3 Advantages 35
5.29.4 Limitations 36
5.29.5 Measurand 36
5.29.6 Relevant standards 36
5.30 Small angle X-ray scattering (SAXS) 36
5.30.1 Description 36
5.30.2 Nano-object parameters 36
5.30.3 Advantages 37
5.30.4 Limitations 37
5.30.5 Measurand 37
5.30.6 Relevant standards 38
5.31 Static light scattering (SLS) and static multiple light scattering (SMLS) 38
5.31.1 Description 38
5.31.2 Nano-object parameters 38
5.31.3 Advantages 38
5.31.4 Limitations 39
5.31.5 Measurands (SLS) 39
5.31.6 Measurands (SMLS) 39
5.31.7 Relevant standards 39
5.32 Single particle light interaction methods 39
5.32.1 Description 39

© ISO 2016 – All rights reserved
Documents
Document Sharing Hub
Full document details: PD ISO/TR 18196:2016

5.32.2 Nano-object parameters ... 40
5.32.3 Advantages ... 40
5.32.4 Limitations ... 40
5.32.5 Measurand ... 40
5.32.6 Relevant standards ... 40
5.33 Thermogravimetric analysis (TGA) ... 40
5.33.1 Description ... 40
5.33.2 Nano-object parameters ... 40
5.33.3 Advantages ... 40
5.33.4 Limitations ... 41
5.33.5 Measurand ... 41
5.33.6 Relevant standards ... 41
5.33.7 Hyphenated TGA techniques ... 41
5.34 Transmission electron microscopy (TEM) ... 41
5.34.1 Description ... 41
5.34.2 Nano-object parameters ... 41
5.34.3 Advantages ... 42
5.34.4 Limitations ... 42
5.34.5 Measurand ... 42
5.34.6 Relevant standards ... 42
5.35 X-ray diffraction (XRD) ... 43
5.35.1 Description ... 43
5.35.2 Nano-object parameters ... 43
5.35.3 Advantages ... 43
5.35.4 Limitations ... 43
5.35.5 Measurand ... 43
5.35.6 Relevant standards ... 43
5.36 X-ray photoelectron spectroscopy (XPS) ... 44
5.36.1 Description ... 44
5.36.2 Nano-object parameters ... 44
5.36.3 Advantages ... 44
5.36.4 Limitations ... 44
5.36.5 Measurand ... 44
5.36.6 Relevant standards ... 44

Annex A (informative) **Sample separation/preparation** ... **46**

Bibliography ... **49**

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD ISO/TR 18196:2016

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 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*.

© ISO 2016 – All rights reserved
Documents
Document Sharing Hub
Full document details: PD ISO/TR 18196:2016

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.

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD ISO/TR 18196:2016

Documents
Document Sharing Hub

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]

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub

Full document details: PD ISO/TR 18196:2016

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].

© ISO 2016 – All rights reserved
Documents
Document Sharing Hub
Full document details: PD ISO/TR 18196:2016

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.

Documents
Document Sharing Hub

© ISO 2016 – All rights reserved

Full document details: PD ISO/TR 18196:2016

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.

© ISO 2016 – All rights reserved
Documents
Document Sharing Hub
Full document details: PD ISO/TR 18196:2016

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

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD ISO/TR 18196:2016

on the electroacoustic spectra and conversely, electrokinetic phenomena have negligible effect on the attenuation spectra.

5.2.2 Nano-object parameters

Concentration, size, and size distribution.

5.2.3 Advantages

5.2.3.1 Does not require sample dilution; applicable for concentrated samples.

5.2.3.2 Method is suitable for characterizing aggregated and structured systems.

5.2.3.3 Measurement is not affected by stirring and/or pumping of the sample.

5.2.3.4 Absolute method; does not require size calibration.

5.2.3.5 Instrument verification can be conducted by measurement of water with known acoustic properties.

5.2.3.6 Solvent-based samples (non-aqueous) can be analysed.

5.2.4 Limitations

5.2.4.1 Applicable only to liquid-borne particles.

5.2.4.2 Lower size limit ≈10 nm; upper size limit ≈3 mm.

5.2.4.3 Lower volume fraction limit is ≈0,1 %; upper volume fraction limit is ≈50 %.

5.2.5 Measurand

Equivalent spherical particle size distribution on a volume basis calculated from the ultrasound attenuation frequency spectrum.

5.2.6 Relevant standards

— ISO 20998-1, *Measurement and characterization of particles by acoustic methods — Part 1: Concepts and procedures in ultrasonic attenuation spectroscopy*

— ISO 20998-2, *Measurement and characterization of particles by acoustic methods — Part 2: Guidelines for linear theory*

5.3 Analytical centrifugation (AC)

5.3.1 Description

Analytical centrifugation (AC) is chosen here as the generic term for a number of centrifugal sedimentation techniques, (some of which are) also known as centrifugal liquid sedimentation (CLS), analytical ultracentrifugation (AUC) or differential centrifugal sedimentation (DCS). Together, this is a versatile family of methods with broad diameter range (1 nm to 5 000 nm) and high resolution. The nanoparticles are forced to move through a predominantly aqueous medium inside an AC measuring cell under a centrifugal force of 500 to 500 000 × g (rotor speed 100 r/min to 60 000 r/min). Sedimentation causes fractionation to take place, because the larger particles move faster. The sedimentation

© ISO 2016 – All rights reserved
Documents
Document Sharing Hub
Full document details: PD ISO/TR 18196:2016

velocities of the different fractions are measured with refractive index, ultraviolet (UV)-absorption or light scattering/turbidity detectors. The diameters of the different fractions can be calculated from the difference in sedimentation velocity, i.e. the complete particle size distribution, according to Stokes' law. The resolution of the AC method is so high because, according to Stokes, velocity is proportional to the square of particle diameter.

5.3.2 Nano-object parameters

Size distribution.

5.3.3 Advantages

5.3.3.1 High diameter resolution.

5.3.3.2 Broad diameter range (1 nm to 5 000 nm).

5.3.4 Limitations

The AUC particle size distribution method can give spurious results if particle size is too high, i.e. if the particles form a sediment too quickly or if they display a broad chemical heterogeneity (i.e. no uniform particle density).

5.3.5 Measurand

5.3.5.1 Settling velocity in centrifugal field.

5.3.5.2 Refractive index, ultraviolet (UV)-absorption or light scattering/turbidity.

5.3.6 Relevant standards

— ISO 13318-1, *Determination of particle size distribution by centrifugal liquid sedimentation methods — Part 1: General principles and guidelines*

— ISO 13318-2, *Determination of particle size distribution by centrifugal liquid sedimentation methods — Part 2: Photocentrifuge method*

— ISO 13318-3, *Determination of particle size distribution by centrifugal liquid sedimentation methods — Part 3: Centrifugal X-ray method*

5.4 Electroacoustic spectroscopy

5.4.1 Description

Electroacoustic phenomena arise from the coupling between the ultrasound field and electric field in a liquid that contains ions. Either of these fields can be a primary driving force. The liquid might be a simple Newtonian liquid or a complex heterogeneous dispersion, emulsion or even a porous body. There are several different electroacoustic effects, depending on the nature of the liquid and the type of driving force. Electroacoustic instruments are built as a probe, which includes elements for generating (measuring) ultrasound field and elements for generating (measuring) electric field (current).

5.4.2 Nano-object parameters

Electrokinetic potential.

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD ISO/TR 18196:2016

5.4.3 Advantages

5.4.3.1 Does not require sample dilution; applicable for concentrated samples.

5.4.3.2 Method is suitable for characterizing aggregated and structured systems.

5.4.3.3 Mixing and pumping of the sample are allowed.

5.4.3.4 Sensor design is a probe that can be inserted into the static or into the mixed or pumped sample.

5.4.3.5 Measurement time is under 1 min.

5.4.3.6 Sample volume can be as little as 0,1 ml.

5.4.3.7 Applicable for high ionic strength up to several mole per litre.

5.4.3.8 Solvent-based samples (non-aqueous) are allowed.

5.4.3.9 Well suited for pH and surfactant titrations of the intact concentrated samples.

5.4.4 Limitations

5.4.4.1 Applicable only to liquid-borne nanoparticles.

5.4.4.2 There is no restriction on the lower size limit; upper size limit ≈0,1 mm.

5.4.4.3 Lower volume fraction limit is ≈1 %; upper volume fraction limit is ≈50 %.

5.4.5 Measurand

Electrokinetic potential.

5.4.6 Relevant standards

— ISO 13099-1, *Colloidal systems — Methods for zeta potential determination — Part 1: Electroacoustic and electrokinetic phenomena*

— ISO 13099-3, *Colloidal systems — Methods for zeta potential determination — Part 3: Acoustic methods*

5.5 Aerosol particle mass analyser (AMS)

5.5.1 Description

This technique classifies aerosol particles into fractions based on the mass-to-charge ratio. The basic concept is to flow aerosol with a Boltzmann charge distribution through an annulus in a cylindrical centrifuge with a DC voltage applied. It is the principle of the force equation where the centrifugal acceleration of the particles is counterbalanced by the electrostatic force on a particle.

The aerosol shall first pass through a neutralizing section. At the outlet of the device, an aerosol detection system is required to determine the particle number size distribution. The applied voltage and rotational velocity can be varied to obtain size fractions selected by particle mass.

© ISO 2016 – All rights reserved
Documents
Document Sharing Hub
Full document details: PD ISO/TR 18196:2016

5.5.2 Nano-object parameters

Concentration and size.

NOTE Size is calculated from particle mass.

5.5.3 Advantages

5.5.3.1 High mass resolution.

5.5.3.2 No mass distribution assumptions required.

5.5.3.3 Near real-time measurement.

5.5.3.4 Effective density information when coupled to differential mobility analysis (see 5.9).

5.5.3.5 Direct measurement of particle mass.

5.5.3.6 Determination of number concentration by mass basis.

5.5.4 Limitations

5.5.4.1 Applicable only to airborne nanoparticles (suspensions or powders are required to be aerosolized).

5.5.4.2 Lower mass limit about 0,01 fg; upper limit about 100 fg (from about 30 nm to 500 nm in size assuming that the shape of particles is spherical with density of 1 g/cm^3).

5.5.4.3 Requires an aerosol particle charger and a radioactive charge neutralizer (beta or alpha emitter).

5.5.5 Measurand

Particle number concentration as function of mass-to-charge ratio of the particles.

5.5.6 Relevant standards

No standards were identified; however, there are papers describing the technique[14][15][16].

5.6 Auger electron spectroscopy (AES)

5.6.1 Description

Auger electron spectroscopy (AES) is an electron beam technique that is commonly used to determine the chemical composition of surfaces. Excitation of the sample with an energetic electron beam results in the generation of a core hole. Relaxation from this excited state occurs when this core hole is filled by an electron from a higher energy state with the simultaneous emission of either an X-ray photon or an Auger electron. Due to the relatively low energies (<3 keV) of the emitted Auger electrons, its mean free path within the sample is limited to less than 10 nm, making AES inherently surface sensitive.

5.6.2 Nano-object parameters

Concentration (secondary electron imaging or elemental mapping), size (secondary electron imaging or elemental mapping), chemical composition and shape.

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD ISO/TR 18196:2016