STN P	**Nanotechnológie** **Odporúčania na detekciu a identifikáciu nanoobjektov v komplexných matriciach**	**STN P** **CEN/TS 17273** 60 3031

Nanotechnologies - Guidance on detection and identification of nano-objects in complex matrices

Táto norma obsahuje anglickú verziu európskej normy.
This standard includes the English version of the European Standard.

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Nanotechnologies - Guidance on detection and identification of nano-objects in complex matrices

Nanotechnologies - Document d'orientation pour la détection et l'identification des nano-objets dans les matrices complexes

Nanotechnologien - Leitfaden für die Detektion und Identifizierung von Nanoobjekten in komplexen Matrizen

This Technical Specification (CEN/TS) was approved by CEN on 28 September 2018 for provisional application.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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European foreword

This document (CEN/TS 17273:2018) has been prepared by Technical Committee CEN/TC 352 "Nanotechnologies", the secretariat of which is held by AFNOR.

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Introduction

Nanotechnology is a rapidly developing field of science and technology that focuses on processes and materials at the nanoscale size (particle dimensions that are approximately 1 nm to 100 nm). It is a highly multidisciplinary field with a wide range of materials and applications, e.g. health care, information and communication technologies, energy production and storage, materials science/chemical engineering, manufacturing, environmental protection, consumer products (e.g. food, cosmetics, etc.). Therefore, the resulting products containing nanoscaled materials are very diverse and different in their properties.

CEN/TS 17010 provides guidelines for the identification of measurands to characterize nano-objects, and their agglomerates and aggregates and to assess specific properties relevant to the performance of materials that contain them. This document describes the measurands for characterizing nano-objects based on popular current techniques for characterizing nano-objects. Due to variable matrix interferences, a method-specific sample preparation protocol to separate particles of interest from their respective matrices is mandatory.

The production and use of nanomaterials may lead, among others, to an increasing release of nano-objects into the environment e.g. by liquid waste and production streams. To ensure sustainable use and development of nanotechnology there is a need for control and monitoring of nanomaterial systems according to their application (e.g. risk assessment). For that reason, it is essential to identify useful measurement techniques for the detection and characterization of nano-objects in so-called complex matrices, such as natural liquids, waste water, food and cosmetics [1]. It is important that specific characteristics of the nano-objects are known to be able to identify them.

There are numerous techniques for fractionation of nano-objects based on, e.g. Centrifugal Liquid Sedimentation (CLS) or flow based separation methods, such as Field-Flow Fractionation (FFF), hydrodynamic chromatography (HDC) and size exclusion chromatography (SEC). Generally, particle size distributions are obtained by the measurement of the particle concentration from the different size fractions.

Imaging techniques such as Electron Microscopy (EM) after appropriate sample preparation allow the detection/imaging of single particles according to several features, e.g. projection area, longest or shortest external dimension.

In case of counting techniques, after a high and known dilution of a particle stream only single particles are present in the detection zone and, e.g. particle volume or particle projection area dependent signals can be measured and related to the particle numbers by light scattering counting methods.

When many different particles are present in the detection zone, ensemble techniques such as static or dynamic scattering techniques can be used, provided that the size polydispersity of the particles is limited. Dynamic light scattering (DLS) analyses, for example, generate signal spectra with size dependent components. DLS can deconvolute these spectra into primary intensity-weighted particle size distributions, but only for relatively simple sample systems, with well-separated particle size modes and with the help of advanced algorithms.

The well-established particle size analysis techniques mentioned so far do not cover the chemical identification of the nano-objects. This document addresses the detection of nano-objects in complex liquid matrices which might contain an elevated level of inorganic salts, organic contaminants and larger organic and inorganic particles as well as natural background nano-objects. Therefore, it is important that for each particle two measurands are combined: not only the size is needed (for classification as a nano-object) but also the elemental composition (to discriminate the target particles with an a priori known elemental composition or morphology, from the matrix and background particles). The aim of this document is to guide the users how to combine size measurement with chemical identification for each particle.

The document proposes the usage of 3 main characterization methods:

— Field Flow Fractionation combined with multiple detection systems delivering size related information and additionally material identification;

— Electron Microscopy equipped with Energy Dispersive X-ray Spectroscopy (EDX) to determine the elemental composition of the particles, additionally to their geometrical measures;

— Single particle Inductively Coupled Plasma – Mass Spectrometry as an elemental specific detection system gives as well size related information.

For the identification of nano-objects, this document requires a priori knowledge of their nature, e.g. their elemental composition.

All proposed methods currently do not allow *in situ* but only *ex situ* characterization.

1 Scope

This document sets requirements for sampling and treatment of the complex matrices in order to obtain a liquid dispersion with sufficiently high concentration of the nano-objects of interest.

This document provides guidelines for detection and identification of specific nano-objects in complex matrices, such as liquid environmental compartments, waste water and consumer products (e.g. food, cosmetics). This document requires for the identification a priori knowledge of the nature of the nano-objects like their chemical composition. The selected detection and identification methods are based on a combination of size classification and chemical composition analysis. Identification can also be supported, e.g. by additional morphology characterization. Currently only Field Flow Fractionation, Electron Microscopy and single particle Inductively Coupled Plasma – Mass Spectrometry fulfil this combination condition.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

CEN/TS 17010:2016, *Nanotechnologies — Guidance on measurands for characterising nano-objects and materials that contain them*

EN ISO/IEC 17025:2017, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:2017)*

CEN ISO/TS 80004-1:2015, *Nanotechnologies — Vocabulary — Part 1: Core terms (ISO/TS 80004-1:2015)*

CEN ISO/TS 80004-2:2017, *Nanotechnologies — Vocabulary — Part 2: Nano-objects (ISO/TS 80004-2:2015)*

ISO 9276-2, *Representation of results of particle size analysis — Part 2: Calculation of average particle sizes/diameters and moments from particle size distributions*

ISO 9276-3, *Representation of results of particle size analysis — Part 3: Adjustment of an experimental curve to a reference model*

ISO 9276-4, *Representation of results of particle size analysis — Part 4: Characterization of a classification process*

ISO 9276-5, *Representation of results of particle size analysis — Part 5: Methods of calculation relating to particle size analyses using logarithmic normal probability distribution*

ISO 9276-6, *Representation of results of particle size analysis — Part 6: Descriptive and quantitative representation of particle shape and morphology*

ISO 13322-1, *Particle size analysis — Image analysis methods — Part 1: Static image analysis methods*

ISO 14488, *Particulate materials — Sampling and sample splitting for the determination of particulate properties*

ISO/DIS 19749, *Nanotechnologies — Measurements of particle size and shape distributions by scanning electron microscopy*

ISO/TS 21362, *Nanotechnologies — Analysis of nano-objects using asymmetrical-flow and centrifugal field-flow fractionation*

ISO/DIS 21363, *Nanotechnologies — Measurements of particle size and shape distributions by transmission electron microscopy*