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Determination of particle size distribution and number concentration by particle tracking analysis (PTA)

Détermination de la distribution granulométrique et de la concentration en nombre par l'analyse de suivi de particule (PTA)

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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 document 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).

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For an explanation of the voluntary nature of standards, 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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 24, *Particle characterization including sieving*, Subcommittee SC 4, *Particle characterization*.

This second edition cancels and replaces the first edition (ISO 19430:2016), which has been technically revised.

The main changes are as follows:

— Inclusion of particle counting and number concentration measurements.

— Inclusion of information on gravitational motion tracking.

— Inclusion of information on simultaneous multispectral detection.

— Inclusion of particle number concentration comparison to other methods.

— Inclusion of information on serial dilution for PTA.

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Introduction

Regulatory, scientific and commercial requirements for nanomaterial characterization or characterization of particulate suspensions where particle sizing and counting is used provide a strong case for further development of techniques such as particle tracking analysis (PTA), also known as nanoparticle tracking analysis (NTA).[1] Due to the fact that the term PTA covers a larger size range and is more generic,[1)] the term PTA is used throughout this document to refer to NTA and PTA.

PTA is based on measuring the diffusion movement of objects (particles, droplets or bubbles) in a dispersion, but can also be used to undertake gravitational migration tracking by means of laser illumination, imaging of scattered light, particle identification and localization, and individual particle tracking.[2)] This document covers two tracking regimes.

— Brownian motion tracking for smaller particles.

— Gravitational fall tracking for larger particles.

In both cases, the suspension is an even dispersion of particles, gas bubbles or other liquid droplets. The hydrodynamic diameter of the individual particles, droplets or bubbles is related to Brownian motion parameters via the Einstein equation and via Stokes law for gravitational migration dynamics.

In recent years, the academic community working in fields such as liposomes and other drug delivery vehicles, nanotoxicology, viruses, exosomes, protein aggregation, inkjet inks, pigment particles, cosmetics, foodstuffs, fuel additives and ultrafine bubbles began using the PTA technology for characterization. ASTM E2834-12 was developed to give guidance to the measurement of particle size distribution by means of NTA. This document aims to broaden the scope of the specification and to introduce system tests for PTA operation as well as to extend the particle size range from nanoscale to microscale sizes. One way to do this is to combine Brownian motion tracking with gravitational migration tracking in the same device on the same sample.

For a number of years, the stakeholders working with nanomaterials safety, regulation, compliance and fundamental research into applications such as biomedicine, catalysis, fuel additives and others were looking for a method (or a combination of methods) for counting and sizing particles in a wide size range (larger than 1 nm to 100 nm). Particle size distributions are often used to evaluate nanomaterials for regulatory purposes (see Reference [41] on the definition of nanomaterial) or for material specification compliance. A number of techniques are available for such characterization, but samples need to be monodisperse. A bigger challenge is to provide an accurate particle count. Techniques such as PTA, electron microscopy, spICP-MS or electrical sensing zone (see ISO 13319-1) allow particle count but have method-specific issues.

One of the key aspects of PTA is the interpretation of data. The key measurand obtained from PTA measurement is the number-based particle size distribution where the size is taken to mean the hydrodynamic diameter of the particles in the sample. The hydrodynamic particle diameters measured with PTA can be different from equivalent particle diameters obtained with different techniques[3] such as dynamic light scattering (DLS) (see ISO 22412:2017) or electron microscopy (see ISO 21363 and ISO 19749).

1) NTA is the most recognised abbreviation for the technique described in this document. However, PTA includes NTA in its size range of measurements.

2) For the purpose of this document, “tracking” is intended to mean “following in terms of particle’s x and y position”; “track” is defined in 3.32.

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Determination of particle size distribution and number concentration by particle tracking analysis (PTA)

1 Scope

This document specifies the particle tracking analysis (PTA) method under static (no flow) conditions for the determination of the number–based particle size distribution and the number concentration in liquid dispersions (solid particles, liquid droplets or bubbles suspended in liquids).

This document covers two tracking regimes.

— Brownian motion tracking for smaller particles.

— Gravitational fall tracking for larger particles.

This document outlines the theory and basic principles of the PTA method along with its limitations and advantages for both size evaluation and number concentration measurements. It also describes commonly used instrument configurations and measurement procedures as well as system qualifications and data reporting.

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.

ISO 13322-1:2014, *Particle size analysis — Image analysis methods — Part 1: Static image analysis methods*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
nanoscale
length range approximately from 1 nm to 100 nm

[SOURCE: ISO 80004-1:2023, 2.1]

3.2
nanoparticle
discrete piece of material with all external dimensions in the *nanoscale* (3.1)

Note 1 to entry: If the dimensions differ significantly (typically by more than three times), terms such as "nanofibre" or "nanoplate" are preferred to the term nanoparticle.

[SOURCE: ISO 80004-1:2023, 3.3.4]

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