PD CEN ISO/TS 19590:2019

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# Nanotechnologies – Size distribution and concentration of inorganic nanoparticles in aqueous media via single particle inductively coupled plasma mass spectrometry (ISO/TS 19590:2017)

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# National foreword

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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.

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# CEN ISO/TS 19590

February 2019

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# Nanotechnologies - Size distribution and concentration of inorganic nanoparticles in aqueous media via single particle inductively coupled plasma mass spectrometry (ISO/TS 19590:2017)

Nanotechnologies - Distribution granulométrique et concentration de nanoparticules inorganiques en milieu aqueux par spectrométrie de masse à plasma induit en mode particule unique (ISO/TS 19590:2017)

Nanotechnologien - Größenverteilung und Konzentration anorganischer Nanopartikel in wässrigen Medien durch Massenspektrometrie an Einzelpartikeln mit induktiktiv gekoppeltem Plasma (ISO/TS 19590:2017)

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## European foreword

The text of ISO/TS 19590:2017 has been prepared by Technical Committee ISO/TC 229 "Nanotechnologies" of the International Organization for Standardization (ISO) and has been taken over as CEN ISO/TS 19590:2019 by Technical Committee CEN/TC 352 "Nanotechnologies" the secretariat of which is held by AFNOR.

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## Endorsement notice

The text of ISO/TS 19590:2017 has been approved by CEN as CEN ISO/TS 19590:2019 without any modification.



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Document Sharing Hub
Full document details: PD CEN ISO/TS 19590:2019

# Contents

Page

**Foreword** ... **iv**
**Introduction** ... **v**
**1 Scope** ... **1**
**2 Normative references** ... **1**
**3 Terms and definitions** ... **1**
**4 Abbreviated terms** ... **2**
**5 Conformance** ... **2**
**6 Procedure** ... **3**
6.1 Principle ... 3
6.2 Apparatus and equipment ... 3
6.3 Chemicals, reference materials and reagents ... 3
6.3.1 Chemicals ... 3
6.3.2 Reference materials ... 3
6.3.3 Reagents ... 4
6.4 Samples ... 4
6.4.1 Amount of sample ... 4
6.4.2 Sample dilution ... 5
6.5 Instrumental settings and performance check ... 5
6.5.1 Settings of the ICP-MS system ... 5
6.5.2 Checking the performance of the ICP-MS system ... 5
6.6 Determination of the transport efficiency ... 6
6.6.1 Determination of transport efficiency based on measured particle frequency ... 6
6.6.2 Determination of transport efficiency based on measured particle size ... 7
6.7 Determination of the linearity of response ... 8
6.8 Determination of the blank level ... 8
6.9 Analysis of aqueous suspension ... 8
6.10 Data conversion ... 9
**7 Results** ... **9**
7.1 Calculations ... 9
7.1.1 Calculation of the transport efficiency ... 10
7.1.2 Calculation of the ICP-MS response ... 10
7.1.3 Calculation of particle concentration and size ... 10
7.1.4 Calculation of the particle concentration detection limit ... 11
7.1.5 Calculation of the particle size detection limit ... 12
7.1.6 Calculation of ionic concentration ... 13
7.2 Performance criteria ... 13
7.2.1 Transport efficiency ... 13
7.2.2 Linearity of the calibration curve ... 13
7.2.3 Blank samples ... 13
7.2.4 Number of detected particles ... 13
**8 Test report** ... **13**
**Annex A** (informative) **Calculation spreadsheet** ... **15**
**Bibliography** ... **19**

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# 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.

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# Introduction

This document was developed in response to the worldwide demand of suitable methods for the detection and characterization of nanoparticles in food and consumer products. Products based on nanotechnology or containing engineered nanoparticles are already in use and beginning to impact the food-associated industries and markets. As a consequence, direct and indirect consumer exposure to engineered nanoparticles (in addition to natural nanoparticles) becomes more likely. The detection of engineered nanoparticles in food, in samples from toxicology and in exposure studies therefore becomes an essential part in understanding the potential benefits, as well as the potential risks, of the application of nanoparticles.

Single particle inductively coupled plasma mass spectrometry (spICP-MS) is a method capable of detecting single nanoparticles at very low concentrations. The aqueous sample is introduced continuously into a standard ICP-MS system that is set to acquire data with a high time resolution (i.e. a short dwell time). Following nebulization, a fraction of the nanoparticles enters the plasma where they are atomized and the individual atoms ionized. For every particle atomized, a cloud of ions results. This cloud of ions is sampled by the mass spectrometer and since the ion density in this cloud is high, the signal pulse is high compared to the background (or baseline) signal if a high time resolution is used. A typical run time is 30 s to 200 s and is called a “time scan.” The mass spectrometer can be tuned to measure any specific element, but due to the high time resolution, typically only one m/z value will be monitored during a run (with the current instruments).

The number of pulses detected per second is directly proportional to the number of nanoparticles in the aqueous suspension that is being measured. To calculate concentrations, the transport efficiency has to be determined first using a reference nanoparticle. The intensity of the pulse and the pulse area are directly proportional to the mass of the measured element in a nanoparticle, and thereby to the nanoparticle’s diameter to the third power (i.e. assuming a spherical geometry for the nanoparticle). This means that for any increase of a particle’s diameter, the response will increase to the third power and therefore a proper validation of the response for each size range of each composition of nanoparticle is required. Calibration is best performed using a reference nanoparticle material; however, such materials are often not available. Therefore, calibration in this procedure is performed using ionic standard solutions of the measured element under the same analytical condition.

The data can be processed by commercially available software or it can be imported in a custom spreadsheet program to calculate the number and mass concentration, the size (the spherical equivalent diameter) and the corresponding number-based size distribution of the nanoparticles. In addition, mass concentrations of ions present in the same sample can be determined from the same data.

The interested reader can consult References [1] to [4] for further information.

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# Nanotechnologies — Size distribution and concentration of inorganic nanoparticles in aqueous media via single particle inductively coupled plasma mass spectrometry

## 1 Scope

This document specifies a method for the detection of nanoparticles in aqueous suspensions and characterization of the particle number and particle mass concentration and the number-based size distribution using ICP-MS in a time-resolved mode to determine the mass of individual nanoparticles and ionic concentrations.

The method is applicable for the determination of the size of inorganic nanoparticles (e.g. metal and metal oxides like Au, Ag, $TiO_2$, $BVO_4$, etc.), with size ranges of 10 nm to 100 nm (and larger particles up to 1 000 nm to 2 000 nm) in aqueous suspensions. Metal compounds other than oxides (e.g. sulfides, etc.), metal composites or coated particles with a metal core can be determined if the chemical composition and density are known. Particle number concentrations that can be determined in aqueous suspensions range from $10^6$ particles/L to $10^9$ particles/L which corresponds to mass concentrations in the range of approximately 1 ng/L to 1 000 ng/L (for 60 nm Au particles). Actual numbers depend on the type of mass spectrometer used and the type of nanoparticle analysed.

In addition to the particle concentrations, ionic concentrations in the suspension can also be determined. Limits of detection are comparable with standard ICP-MS measurements. Note that nanoparticles with sizes smaller than the particle size detection limit of the spICP-MS method may be quantified as ionic.

The method proposed in this document is not applicable for the detection and characterization of organic or carbon-based nanoparticles like encapsulates, fullerenes and carbon nanotubes (CNT). In addition, it is not applicable for elements other than carbon and that are difficult to determine with ICP-MS. Reference [5] gives an overview of elements that can be detected and the minimum particle sizes that can be determined with spICP-MS.

## 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/TS 80004-1, *Nanotechnologies — Vocabulary — Part 1: Core terms*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TS 80004-1 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**
**nanoparticle**
nano-object with all three external dimensions in the nanoscale

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

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