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# Pigments and extenders — Determination of experimentally simulated nano-object release from paints, varnishes and pigmented plastics

*Pigments et matières de charge — Détermination de la libération simulée de nanoobjets présents dans des peintures, des vernis et des plastiques pigmentés*



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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).

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This document was prepared by Technical Committee ISO/TC 256, *Pigments, dyestuffs and extenders*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

 

# Introduction

The possible release of nano-objects (nanoscale pigments and extenders) from paints, varnishes and pigmented plastics into surrounding air or liquid is an important consideration in health and safety, for the end user and the environment. Therefore, it is important to obtain data about the propensity of pigmented paints and plastics to release nano-objects, thereby allowing exposure to be evaluated[10], controlled and minimized. This property will likely depend on both the physico-chemical properties of the nano-objects and the matrix containing the nano-objects.

The currently available methods to assess the propensity of pigmented paints, varnishes and plastics to release nano-objects into the air require energy to be applied to a sample to induce abrasion, erosion or comminution, which cause dissemination of the particles into the gaseous phase, i.e. generation of aerosols.

Due to their higher sensitivity, the particle number concentration and the number-weighted particle size distribution are necessary for the quantification of the release of nano-objects since the particle mass depends on the cubed particle diameter and the mass concentrations of nano-objects are too low in order to detect them with currently commercially available instruments. Further measurements, such as the total particle surface concentration, e.g. References [11] and [12], can be helpful for the interpretation e.g. in regard to health aspects. If the shape, morphology, porosity, and density of the particle material are known, an exact conversion into the different quantity types is possible by measuring the total particle size distribution.

Beside the selection of appropriate measurement instrumentation, a quantitative assessment of process-induced particle release requires furthermore detailed information on the samples, the introduced stress and the kind of interconnection with the instruments. Figure 1 shows for example the single stages, which have to be considered for the quantitative characterization of airborne particulate release.



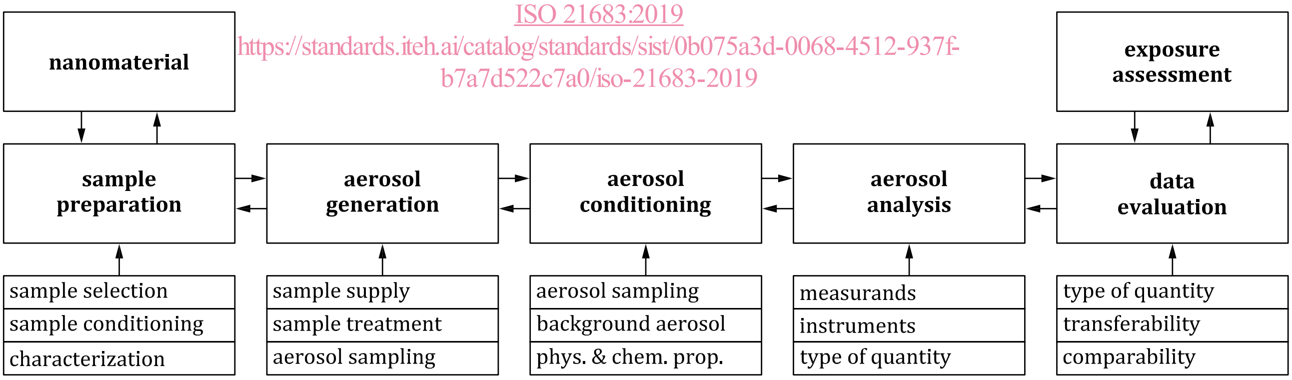


**Figure 1 — Stages for the characterization of process-induced airborne particulate release[5]**

# Pigments and extenders — Determination of experimentally simulated nano-object release from paints, varnishes and pigmented plastics

## 1 Scope

This document specifies a method for experimental determination of the release of nanoscale pigments and extenders into the environment following a mechanical stress of paints, varnishes and pigmented plastics.

The method is used to evaluate if and how many particles of defined size and distribution under stress (type and height of applied energy) are released from surfaces and emitted into the environment.

The samples are aged, weathered or otherwise conditioned to simulate the whole lifecycle.

## 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 9276-1, *Representation of results of particle size analysis — Part 1: Graphical representation*

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



ISO/TS 80004-2, *Nanotechnologies — Vocabulary — Part 2: Nano-objects*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TS 80004-1, ISO/TS 80004-2 and the following apply.

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

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

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

### 3.1 General terms and definitions

**3.1.1**
**aerosol**
system of solid or liquid particles suspended in gas

[SOURCE: ISO 15900:2009, 2.1]

**3.1.2**
**nanoscale**
length range approximately from 1 nm to 100 nm

Note 1 to entry: Properties that are not extrapolations from a larger size are predominantly exhibited in this length range.

[SOURCE: ISO/TS 80004-1:2015, 2.1]

**3.1.3**
**nanoparticle**
*nano-object* (3.1.4) with all external dimensions in the *nanoscale* (3.1.2) where the lengths of the longest and the shortest axes of the nano-object do not differ significantly

Note 1 to entry: If the dimensions differ significantly (typically by more than 3 times), terms such as nanofibre or nanoplate may be preferred to the term nanoparticle.

[SOURCE: ISO/TS 80004-2:2015, 4.4]

**3.1.4**
**nano-object**
discrete piece of material with one, two or three external dimensions in the *nanoscale* (3.1.2)

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

[SOURCE: ISO/TS 80004-1:2015, 2.5]

**3.1.5**
**paint**
pigmented coating material which, when applied to a substrate, forms an opaque dried film having protective, decorative or specific technical properties

[SOURCE: ISO 4618:2014, 2.184]

**3.1.6**
**equivalent spherical diameter**
x
diameter of a sphere having the same physical properties as the particle in the measurement



Note 1 to entry: Physical properties are for instance the same settling velocity or electrolyte solution displacing volume or projection area under a microscope.



Note 2 to entry: The physical property to which the equivalent diameter refers shall be indicated using a suitable subscript, for example $x_S$ for equivalent surface area diameter or $x_V$ for equivalent volume diameter.

[SOURCE: ISO 26824:2013, 1.6]

**3.1.7**
**particle size distribution**
**PSD**
cumulative distribution of the fraction of material smaller (undersize) than given particle sizes, represented by equivalent spherical diameters or other linear dimensions or distribution density of the fraction of material in a size class, divided by the width of that class

Note 1 to entry: Particle size distributions are described in ISO 9276-1.

**3.1.8**
**condensation particle counter**
**CPC**
instrument that measures the particle number concentration of an *aerosol* (3.1.1)

Note 1 to entry: The sizes of particles detected is usually smaller than several hundred nanometres and larger than a few nanometres.

Note 2 to entry: A CPC is one possible detector for use with a DEMC.

Note 3 to entry: In some cases, a condensation particle counter may be called a condensation nucleus counter (CNC).

[SOURCE: ISO 15900:2009, 2.5]

 

**3.1.9**
**differential electrical mobility classifier**
**DEMC**
classifier that is able to select *aerosol* (3.1.1) particles according to their electrical mobility and pass them to its exit

Note 1 to entry: A DEMC classifies aerosol particle sizes by balancing the electrical force on each particle with its aerodynamic drag force in an electrical field. Classified particles are in a narrow range of electrical mobility determined by the operating conditions and physical dimensions of the DEMC, while they can have different sizes due to difference in the number of charges that they have.

[SOURCE: ISO 15900:2009, 2.7]

**3.1.10**
**differential mobility analysing system**
**DMAS**
system to measure the size distribution of submicrometre *aerosol* (3.1.1) particles consisting of a DEMC, flow metres, a particle detector, interconnecting plumbing, a computer and suitable software

[SOURCE: ISO 15900:2009, 2.8]

## 3.2 Specific terms and definitions

**3.2.1**
**particle release from paints, varnishes and plastics**
transfer of material from paints, varnishes and plastics to a liquid or gas as a consequence of mechanical stress



**3.2.2**
**particle number release**
$n$
total number of particles in a specified size range, released from a test specimen as a consequence of mechanical stress



**3.2.3**
**area-specific particle number release**
$n_A$
*particle number release* (3.2.2), divided by the stressed surface area of the test specimen

**3.2.4**
**mass-specific particle number release**
$n_m$
*particle number release* (3.2.2), divided by the mass of removed material

**3.2.5**
**total volume flow rate**
$V_t$
volume flow rate, which takes up all air-transported emissions at the particle source and transfers them

**3.2.6**
**particle number concentration**
$n_V$
number of particles per volume of air

**3.2.7**
**process concentration**
*particle number concentration* (3.2.6), which results from the *total volume flow rate* (3.2.5) and the *particle number release* (3.2.2) as a consequence of mechanical stress on the test specimens

**3.2.8**
**measuring concentration**
*particle number concentration* (3.2.6), which is calibrated by defined dilution of the *process concentration* (3.2.7), in order to establish optimal conditions for the aerosol analysis

**3.2.9**
**model room concentration**
*particle number concentration* (3.2.6), which results from the *area-specific particle number release* (3.2.3) under optimal mixing conditions for a defined room height

Note 1 to entry: The model room concentration is independent of the selected test conditions and represents a reference concentration for real particle number concentrations (e.g. particle pollution in the laboratory) when the height of the model room has been selected carefully.

## 4 Symbols and abbreviated terms

For the purposes of this document, the following symbols (see Table 1) and abbreviated terms (see Table 2) apply.

**Table 1 — Symbols**

| Symbol | Dimension | SI unit |
|---|---|---|
| $n$ | particle number release | Without dimension |
| $n_V$ | particle number concentration | $m^{-3}$ |
| $n_A$ | area-specific particle number release | $m^{-2}$ |
| $n_m$ | mass-specific particle number release | $kg^{-1}$ |
| $V_t$ | total volume flow | $m^3 s^{-1}$ |



**Table 2 — Abbreviated terms**

| Abbreviation | Meaning |
|---|---|
| APS | aerodynamic particle sizer |
| CPC | condensation particle counter |
| DEMAS | differential electrical mobility analysing system |
| DEMC | differential electrical mobility classifier |
| EAD | electrical aerosol detector |
| EDX | energy dispersive X-ray spectroscopy |
| EEPS | engine exhaust particle sizer |
| ELPI | electrical low pressure impactor |
| ESP | electrostatic precipitator |
| FAPES | fast aerosol particle emission spectrometer |
| FMPS | fast mobility particle sizer |
| HEPA | high efficiency particulate air filter |
| ICP-MS | inductively coupled plasma mass spectrometry |
| ICP-OES | inductively coupled plasma optical emission spectrometry |
| LAS | laser aerosol spectrometer |
| NSAM | nanoparticle surface area monitor |
| OPC | optical particle counter |
| OPS | optical particle sizer |
| PM | particulate matter |
| PSD | particle size distribution |

 

**Table 2** *(continued)*

| Abbreviation | Meaning |
|---|---|
| SEM | scanning electron microscopy |
| SMPS | scanning mobility particle sizer |
| TEM | transmission electron microscopy |
| TP | thermal precipitator |
| WRAS | wide range aerosol sampler |

# 5 Methods of stress

## 5.1 Test specimens requirements

Coatings applied on respective substrates or solid materials are suitable test specimens. For good reproducibility the test specimens should be plane and a homogenous distribution of the pigments or extenders in the matrix material should be given.

For interpretation of the measuring results reference test specimens shall be prepared in addition to the actual test specimens. Unpigmented or unfilled test specimens can give information on the influence of these in regard to particle release. For analysing aged or weathered test specimens, unaged or unweathered equivalent test specimens shall be consulted for data interpretation.

An important aspect is the given condition of the test specimen. Detailed information on preparation of test specimens, used pigments and extenders, on pre-conditioning and treatment (ageing, exposure) shall be documented.



Contaminations of the test specimens during preparation, pre-conditioning, pre-treatment, transport, and storage shall be reduced to a minimum. Finished test specimens shall be analysed promptly in order to avoid changes of the physico-chemical properties (e.g. hardness, elasticity) of the test specimens due to impacts of external influences (e.g. temperature variation, UV radiation).

When transporting the test specimens, it shall be observed that the test specimens are not contaminated due to contact with the container used for transport or other test specimens. The duration of contact with ambient aerosol shall be minimized as far as possible.

## 5.2 Test apparatus requirements

### 5.2.1 General

The test apparatus shall cover the aspects of introduction of the test specimens, the stress application on the test specimens, and the sampling.

For the verification of systematic analysis, i.e. for obtaining reproducible results, the test specimens shall be introduced so that the stress is applied only once in order to avoid interferences of repeating applications of energy and constant changes of the stress intensity of the test specimen under test.

Particle number release quantification requires a test apparatus for the simulation of mechanical stress. The intensity of mechanical should be adjustable to the physico-chemical properties of the test specimen. The test apparatus should be described carefully, and appropriate test parameter should be identified before testing. For testing, the test parameter shall be adjusted, checked, and documented.

In order to enable quantification of particle number release, all of the particles released as consequence of mechanical stress shall be measured as close as possible to the location of their formation.

NOTE Mechanical stress application on test specimens can lead to thermal particle generation, which could lead to an overestimation of the particle number release.