SLOVENSKI STANDARD
oSIST prEN ISO 20427:2024

01-junij-2024

Pigmenti in polnila - Postopek disperzije za določanje porazdelitve velikosti delcev na podlagi sedimentacije suspendiranih pigmentov ali polnil v tekoči fazi (ISO 20427:2023)

Pigments and extenders - Dispersion procedure for sedimentation-based particle sizing of suspended pigment or extender with liquid sedimentation methods (ISO 20427:2023)

Pigmente und Füllstoffe - Dispergierverfahren zur sedimentativen Teilchengrößenbestimmung von suspendierten Pigmenten oder Füllstoffen mit Flüssigsedimentationsverfahren (ISO 20427:2023)

Pigments et matières de charge - Procédure de dispersion pour la granulométrie par sédimentation d'un pigment ou d'une charge en suspension à l'aide de méthodes de sédimentation en milieu liquide (ISO 20427:2023)

Ta slovenski standard je istoveten z: **prEN ISO 20427**

ICS:

87.060.10	Pigmenti in polnila	Pigments and extenders

oSIST prEN ISO 20427:2024 **en,fr,de**

INTERNATIONAL STANDARD

ISO 20427

First edition
2023-11

Pigments and extenders — Dispersion procedure for sedimentation-based particle sizing of suspended pigment or extender with liquid sedimentation methods

Reference number
ISO 20427:2023(E)

COPYRIGHT PROTECTED DOCUMENT

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

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 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).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 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.

Pigments and extenders — Dispersion procedure for sedimentation-based particle sizing of suspended pigment or extender with liquid sedimentation methods

1 Scope

This document specifies sample preparation methods to determine the size distribution of separate particles of a single pigment or extender, which is dispersed in a liquid by application of a standardized dispersion procedure, using an ultrasonic device, shaker device or wet jet mill.

The sample preparation methods described are optimized for measurements carried out with a particle sizing technique based on sedimentation. This technique relies on particle migration due to gravitation or centrifugal forces and requires a density contrast between the particles and the liquid phase.

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 3696, *Water for analytical laboratory use — Specification and test methods*

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

ISO 13317-1, *Determination of particle size distribution by gravitational liquid sedimentation methods — Part 1: General principles and guidelines*

ISO 13317-2, *Determination of particle size distribution by gravitational liquid sedimentation methods — Part 2: Fixed pipette method*

ISO 13317-3, *Determination of particle size distribution by gravitational liquid sedimentation methods — Part 3: X-ray gravitational technique*

ISO 13317-4, *Determination of particle size distribution by gravitational liquid sedimentation methods — Part 4: Balance method*

ISO 13318-1:2001, *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*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

ASTM D5965, *Standard Test Methods for Density of Coating Powders*

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 from approximately 1 nm to 100 nm

Note 1 to entry: Properties that are not extrapolations from a larger size are predominantly exhibited in this size range. For such properties, the size limits are considered approximate.

Note 2 to entry: The lower limit in this definition (approximately 1 nm) is introduced to avoid single and small groups of atoms from being designated as nano-objects or elements of nanostructures, which can be implied by the absence of a lower limit.

[SOURCE: ISO 80004-1:2023, 3.1.1 — modified, notes 1 and 2 to entry have been added.]

3.2
nanoparticle
nano-object with all external dimensions in the *nanoscale* (3.1) 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 three times), terms such as nanofibre or nanoplate may are preferred to the term nanoparticle.

[SOURCE: ISO 80004-1:2023, 3.3.4, modified — "where the lengths of the longest and the shortest axes of the nano-object do not differ significantly" has been added to the definition.]

3.3
agglomerate
collection of weakly or medium strongly bound particles where the resulting external surface area is similar to the sum of the surface areas of the individual components

Note 1 to entry: The forces holding an agglomerate together are weak forces, for example van der Waals or simple physical entanglement.

Note 2 to entry: Agglomerates are also termed secondary particles and the original source particles are termed *primary particles* (3.5).

[SOURCE: ISO 80004-1:2023, 3.2.4]

3.4
aggregate
particle comprising strongly bonded or fused particles where the resulting external surface area is significantly smaller than the sum of surface areas of the individual components

Note 1 to entry: The forces holding an aggregate together are strong forces, for example covalent or ionic bonds, or those resulting from sintering or complex physical entanglement, or otherwise combined former *primary particles* (3.5).

Note 2 to entry: Aggregates are also termed secondary particles and the original source particles are termed *primary particles* (3.5).

[SOURCE: ISO 80004-1:2023, 3.2.5, modified — "or otherwise combined former primary particles" has been added to the end of note 1 to entry.]

3.5
primary particle
single nano-object with at least one of three external dimensions at the nanoscale

Note 1 to entry: Sometimes, if the primary particle is present in crystalline form, it also contains twinning boundaries.

3.6
spin fluid
inert liquid which is injected into the disc of a disc centrifuge photosedimentometer prior to the sample to define a certain radius dependent gradient of viscosity for sedimentation

Note 1 to entry: Alkaline conditions minimize agglomeration of dispersed aggregates in most cases.

3.7
wet jet milling
dispersing method of particles in liquid phase using the complex shear force arising from turbulent flow in the channel and cavitation from the abrupt pressure change

Note 1 to entry: This method is also called high pressure homogenizer method.

4 Principles of dispersion

4.1 Principles of ultrasonic dispersion

A piezo electrical ceramic material is driven by an applied alternating current electrical field to expand and shrink periodically at an ultrasonic frequency in the range of 15 kHz up to 80 kHz and more. This movement creates acoustic waves moving through the dispersion, which produce cavitation bubbles. The collapse of these cavitation bubbles leads locally to strong thermal effects and shear-stress, which are responsible for the destruction of agglomerates and even aggregates.

Energy density of sonication, temperature and particle volume concentration of the dispersion are critical parameters of sonication and should be held at recipe values strictly.

In addition to probe-type sonicators ultra sonic (US) baths, inverted cup-horn sonicators and so-called vial-tweeters also exist. US baths, cup-horn dispersers and vial-tweeters are known as indirect dispersers, where sound energy is inserted via the wall of the container. Determining the energy input of these dispersers is much more difficult than for probe sonication, but contamination is reduced[9].

4.2 Principle of wet jet mill dispersion

The wet jet milling method is a wet-type milling to disintegrate agglomerates of powder samples in liquid. In this method, particles suspended in a liquid medium are passed through a narrow channel at high pressure. Then, the suspension of the particles is enhanced by the complex shear force arising from turbulent flow in the channel. In addition, the high pressure in the narrow channel induces the cavitation bubbles from the abrupt pressure change. The burst of the cavitation bubbles then work to disperse powder samples in the liquid phase, as in the ultra-sonication method. The advantage of this dispersion technique is that it yields suspensions with low contamination, unlike the ultra-sonic homogenizer method. The pressure range is the important factor to disperse the powder samples in the liquid phase. Typically, the pressure range is from 80 MPa to 245 MPa[10][11].

4.3 Principle of shaker-based dispersion

The shaker device should be built like a plate with holders for the high-density polyethylene (HDPE) bottles (see Annex B). A successful dispersion is achieved when the plate is shaking vertically from back to front with a vibration amplitude of minimum 32 mm and a frequency of 660 Hz.

Important aspects are:

— inclusion of grinding beads, high loading;

— particle dispersion limitations: agglomerates/aggregates <100 µm in a liquid (viscous medium);

— grinding beads are agitated by rotary, tumbling and/or 2D-vibratory motion of the container/vessel;

— shear and elongational stress on agglomerates at squeezing of liquid between colliding grinding beads and impulse exchange from collisions of agglomerates with grinding beads[12][13].

5 Principles of sedimentation-based techniques for particle size analysis

5.1 Stokesian sedimentation analysis

For all sedimentation-based procedures for particle sizing which are cited in this document, Stokesian sedimentation analysis of dispersions is used. ISO 13318-1:2001, 4.1 describes in detail the general procedure and calculations used to approach a particle size distribution of dispersed particles.

5.2 Disk-type centrifuges

The particles settle within an optically clear, rotating disc. When particles approach the outside edge of the rotating disc, they block/scatter a portion of a light beam or X-ray beam that passes through the disc. The change in light intensity shall be continuously recorded, and converted by the operating software into a particle size distribution, in accordance with ISO 13318-1.

Instead of detecting the local particle concentration with optical turbidity, X-ray absorption shall be used in certain instruments with the advantage of direct particle mass dependency, in accordance with ISO 13318-3.

5.3 Cuvette-type centrifuges

The cuvette-type centrifuge is a special analytical centrifuge that instantaneously measures the particle concentration at one or more radial positions within the rotating sedimentation cuvette.

For instance, space- and time-resolved extinction of the transmitted light across the entire length of the sample allows the analysis of particle and droplet velocity distributions for creaming and sedimentation phenomena without the need of any material data. This process additionally performs particle sizing according to ISO 13318-2.

The centrifugal speed of these instruments is typically between 50 min^{-1} and 60 000 min^{-1}. Instruments with a centrifugal speed below 10 000 min^{-1} are typically called cuvette centrifuges. Devices which can rotate above 10 000 min^{-1} rotation are called ultracentrifuge. For centrifugal speeds greater than 6 000 min^{-1}, the detection of particle sizes is limited to 1 µm or below.

5.4 Gravitation-based sedimentation methods

The gravitation-based liquid sedimentation shall be executed using four different techniques: the fixed pipette method in accordance with ISO 13317-2, the X-ray gravitation-based technique in accordance with ISO 13317-3, the balance method in accordance with ISO 13317-4 and gravitation-based photo sedimentation.

With the balance method as well as with the pipette method in accordance with ISO 13317-2, a resolution below 1 µm is critical because of the limitations of the used detection mechanisms. The X-ray sedimentation on the other hand depends on vibration isolation and detector quality. It can resolve 100 nm, similar to the photo sedimentation.

Therefore, only the liquid X-ray sedimentation in accordance with ISO 13317-1 and ISO 13317-3 is included in this document.

The concentration of a dispersed sample is measured by the attenuation of an X-ray beam. A stable, narrow, monochromatic collimated beam of X-rays passes through a suspension of the sample and is detected at a known distance from the top of the sample cell. The sample cell is filled completely with the sample suspension for the duration of the analysis. The settling height at which the particle concentration is determined may be reduced during the analysis for the purpose of obtaining a more rapid analysis compared to an analysis where all measurements are made at the same height value.