International Standard

ISO 20427

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

Pigments et matières de charge — Mode opératoire de dispersion pour la détermination granulométrique basée sur la sédimentation des pigments ou matières de charge en suspension par des méthodes de sédimentation dans un liquide

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Foreword

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This document was prepared by Technical Committee ISO/TC 256, *Pigments, dyestuffs and extenders*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 298, *Pigments and extenders,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 20427:2023), which has been technically revised.

The main changes are as follows:

— in 5.5, a note has been added with additional information on the effective particle density;

— in 6.9 the original Tables 1 and Table 2 have been combined in one single table;

— in 6.9, Table 1, row 7, columns 6 to 8, a table footnote has been added explaining the density dependency of these values;

— in 6.9, Table 1, row 10, column 7, “class cylinder beaker” has been changed to “sedimentation bath”;

— in Clause 10, a note has been added with a mathematical definition of precision;

— in the second list item of C.1 and Annex E, the distance between the beaker bottom and the ultrasonic probe has been changed from 5 mm to 10 mm;

— in F.2 and F.3, notes have been added to explain why a temperature of 40 °C is important, and to explain the influence of the cooling bath on the sonication power;

— the normative references have been updated.

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.

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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, requirements and guidance*

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 13317-5, *Determination of particle size distribution by gravitational liquid sedimentation methods — Part 5: Photosedimentation techniques*

ISO 13318-1, *Determination of particle size distribution by centrifugal liquid sedimentation methods — Part 1: General principles, requirements and guidance*

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 18451 (all parts), *Pigments, dyestuffs and extenders — Terminology*

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

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

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3 Terms and definitions

For the purposes of this document, the terms and definitions given in the ISO 18451 series and the following 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 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.

[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.]

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3.5
primary particle
single nano-object with at least one of three external dimensions at the *nanoscale* (3.1)

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 to 80 kHz or 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 shall 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 Principles 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 works 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 Principles of shaker-based dispersion

The shaker device shall be built like a plate with holders for the high-density polyethylene (HDPE) bottles in accordance with A.2. 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.

The following aspects should be considered:

— inclusion of grinding beads, high loading;

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