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

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Foreword

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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.htmlwww.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 ~~methods for~~ 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 ~~described~~ 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*

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ISO/TS 21362, *Nanotechnologies — Analysis of nano-objects using asymmetrical flow and centrifugal field flow fractionation*

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/obphttps://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/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/TS 80004-1:2015, 2.1 + 2.1 — modified, an extra sentence hasnotes 1 and 2 to entry have been added to Note 1 to entry. Note 2 to entry has 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 3three times), terms such as nanofibre or nanoplate may beare preferred to the term nanoparticle.

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

[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/TS 80004-2:20151:2023, 3.2.4]

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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/TS 80004-2:2015:2023, 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.

BesidesIn addition to probe-type sonicators also ultra sonic (US) baths, inverted cup-horn sonicators and so-called vial-tweeters also exist. US bathbaths, cup-horn dispersers and vial-tweeters are so calledknown as indirect dispersers, where sound energy is inserted via the wall of the container; the determination of. Determining the energy input of these dispersers is much more difficult than for probe sonication, but contamination is reduced[10]. [9]

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4.2 Principle of wet jet mill dispersion

WetThe 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. 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; then, the. The burst of the cavitation bubbles then work to disperse powder samples in the liquid phase such, 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][12].

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][14].

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

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phenomena, without the need of any material data, and. 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 photosedimentationphoto 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 is able tocan 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. The cumulative mass percentage of the sample present at a given sedimentation height is continuously determined. The X-ray signal attenuation at the known height is compared to the attenuation in the suspending liquid and also to the attenuation in the homogeneously dispersed sample present in the liquid. The attenuation of the emergent X-ray beam is proportional to the mass of the powder in the beam.

5.5 Centrifugal field-flow fractionation method

Field-flow fractionation is a flow-based separation methodology. Centrifugal field-flow fractionation (CF3) is a separation technique that uses a centrifugal field applied perpendicular to a circular channel that spins around its axis to achieve size separation. of particles between the limits of 10 nm and 50 µm. In this method, separation is governed by a combination of size and effective particle density, indicating that applicable size range is dependent on and limited by the effective particle density. In CF3, the mobile phase and analyte flow longitudinally through the channel. The channel is designed to separate the sample components along its length, resulting in the elution of constituents at different times. The channel and its large aspect ratio are designed to promote parabolic or near-parabolic laminar flow between two infinite planes under normal operational conditions. Fractionation is achieved during passage through the channel, based on the velocity flow profile, after which the mobile phase containing separated constituents exits to online detectors and/or a fraction collector for off-line analysis. Common detectors used for analysis of pigment and extender include ultraviolet-visible (UV-Vis) absorbance, fluorescence, multi-angle light scattering (MALS), dynamic light scattering (DLS) and element detectors such as the inductively coupled plasma mass spectrometer (ICP-MS). Combinational analysis of the sizing and concentration evaluation detectors, as well as the size distribution analysis have been performed using this method according to ISO/TS 21362.

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