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Determination of particle size distribution by centrifugal liquid sedimentation methods —

Part 1: General principles, requirements and guidance

Détermination de la distribution granulométrique par les méthodes de sédimentation centrifuge dans un liquide —

Partie 1: Principes généraux, exigences et orientation

Reference number
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Foreword

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This document was prepared by Technical Committee ISO/TC 24, *Particle characterization including sieving*, Subcommittee SC 4, *Particle characterization*.

This second edition cancels and replaces the first edition (ISO 13318-1:2001), which has been technically revised.

The main changes are as follows:

— revision of core terms (3.3, 3.5, 3.10, 3.11, 3.16, 3.14) and inclusion of further terms;

— revised and expanded explanation of measurement principle and techniques (Clauses 5 and 6);

— inclusion of the terminal sedimentation velocity as a measurand;

— introduction of informative annexes that provide:

  — an overview of particle sizing techniques based on centrifugal liquid sedimentation (Annex A);

  — remarks on particle density (Annex B);

  — information regarding sedimentation beyond the validity of Stokes' law (Annex C);

  — trueness, reproducibility and uncertainty determination for velocity and particle size (Annex D);

  — an explanation on the multiwavelength approach (Annex E);

  — a description of the spatial distribution of particles in centrifugal fields (Annex F);

  — the use of CLS for particle characterization beyond the particle size and sedimentation velocity (Annex G).

A list of all parts in the ISO 13318 series can be found on the ISO website.

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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Introduction

Particle size analysis by centrifugal liquid sedimentation (CLS) methods has been performed for several decades, and there are numerous national and international standards employed in various academic and industrial applications. Along with the development of manifold new measurement techniques during the last two decades, sedimentation methods are currently enjoying a renaissance due to technical progress (e.g. multiwavelength features) and the fact that most sedimentation techniques are based on the first-principle measurement of the directed motion (migration) of particles in gravitational or centrifugal fields. The rate of this motion is the particle sedimentation velocity, its acceleration-specific value is called "sedimentation coefficient". Both are individual particle properties, which are related to the particles' external dimensions, and can be considered as primary measurands of liquid sedimentation methods.

Analytical centrifuges determine distributions of these primary measurands from the variation of concentration-dependent signals over time and/or along the radial coordinate of the centrifugal field. This step does not require any pre-knowledge of the dispersed or liquid phase. Further, data processing allows for particle size distributions to be derived from sedimentation velocity and sedimentation coefficient. For this purpose, spherical particles and the applicability of Stokes' law are assumed; the equivalent particle diameter is called the Stokes diameter. The conversion requires knowledge of the relevant properties of the particles and liquids (e.g. particle shape, density or refractive index). In this regard, CLS resembles gravitational liquid sedimentation (see ISO 13317-1 for further information).

The ISO 13318 series covers methods for characterizing dispersed materials in liquids by centrifugation with respect to the particle size distribution and the related distributions of sedimentation velocity and sedimentation coefficient. Their common principle is allocating a particle quantity to the rate of migratory motion in the centrifugal field. They differ with respect to particle quantification, mode of operation and data analysis.

The measurement techniques described in the ISO 13318 series are applicable to liquid dispersions, like suspensions and emulsions, with the continuous phase being a Newtonian liquid. Particles and liquid should not undergo any interactions, which cause significant changes of the dispersed phase in the course of the measurement, such as swelling, shrinking, and dissolution. For some instrumentation, the density difference (also called density contrast) between the dispersed and continuous phase should be limited to positive values.

The measurable particle size range depends on the properties of the materials and typically reaches from a few nm to 100 µm for aqueous samples, whereas the migration velocity can be quantified for the range of 10 nm/s to 1 mm/s. Sedimentation analysis is conducted for low particle concentrations. The permissible range of concentration depends on the measurement technique and theory of analysis; with no correction, it is typically no higher than 0,5 vol%.

As a fractionating technique, sedimentation analysis allows for distinguishing between particle fractions of close sedimentation velocity and the corresponding equivalent Stokes diameter. Accordingly, particle size distributions can be finely resolved, which is an advantage compared to spectroscopic ensemble techniques.

Finally, CLS techniques principally offer the chance to characterize liquid dispersions beyond sedimentational particle properties (see Annex G). For instance, some CLS techniques facilitate the quantification of the total and fractional particle concentration. Moreover, if particles are very fine, i.e. in the case of nanoparticles such as protein aggregates or quantum dots, the diffusional flux of particles can be in the order of the sedimentation flux. Such a situation allows a multidimensional characterization of particle systems, i.e. the simultaneous determination of more than one distributed quantity (e.g. particle size and density or shape factor).

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Determination of particle size distribution by centrifugal liquid sedimentation methods —

Part 1: General principles, requirements and guidance

1 Scope

This document specifies the principles of particle size analysis by centrifugal liquid sedimentation (CLS). It also:

— defines the relevant terms;

— describes the various measurement techniques;

— gives guidance for sample preparation, conducting measurements as well as data analysis;

— establishes rules for method validation, determination of the uncertainty budget as well as representation of results.

An important part of this document deals with the derivation of particle size distributions from CLS data, including discussions on:

— the impact of Brownian motion;

— the parallel determination of particle concentrations;

— the working range with respect to size and concentration;

—the handling of non-spherical and porous particles.

This document applies to samples composed of dispersions of low particle concentration, so that the particles' motion can be considered as that of fully isolated particles. This document does not cover particle migration by gravity, electric or magnetic forces. It also does not cover deriving particle properties other than size, sedimentation velocity and sedimentation coefficient.

NOTE This document can involve hazardous materials, operations and equipment. It does not purport to address all the safety problems associated with its use. Regulations regarding explosion-proof analysers can apply when examining volatile liquids with a low flash point. It is the responsibility of the user of this document to establish appropriate safety and health practices and to determine the applicability of the regulatory limitations prior to its use.

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*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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