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Determination of particle size distribution by gravitational liquid sedimentation methods —

Part 3: X-ray gravitational technique

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

Partie 3: Méthode aux rayons X par gravité

Reference number
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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this part of ISO 13317 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 13317-3 was prepared by Technical Committee ISO/TC 24, *Sieves, sieving and other sizing methods*, Subcommittee SC 4, *Sizing by methods other than sieving*.

ISO 13317 consists of the following parts, under the general title *Determination of particle size distribution by gravitational liquid sedimentation methods*:

— *Part 1: General principles and guidelines*

— *Part 2: Fixed pipette method*

— *Part 3: X-ray gravitational technique*

Annex A of this part of ISO 13317 is for information only.

Determination of particle size distribution by gravitational liquid sedimentation methods —

Part 3:
X-ray gravitational technique

SAFETY PRECAUTIONS — This part of ISO 13317 does not purport to address all of the safety considerations associated with its use. It is the responsibility of the user of this part of ISO 13317 to establish appropriate safety and health practices and determine the applicability of the regulatory limitations prior to its use.

1 Scope

This part of ISO 13317 describes a method for the determination of the particle size distribution of a powder dispersed in a liquid using gravity sedimentation. The measurement of the concentration of solids settling in a liquid suspension is achieved by monitoring the incremental signal absorption from a beam of X-rays.

The method of determining the particle size distribution described in this part of ISO 13317 is applicable to powders which can be dispersed in liquids or powders which are present in slurry form. The typical particle size range for analysis is from about 0,5 μm to about 100 μm. The method is used for materials containing particles of the same chemical composition which produce adequate X-ray opacity.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 13317. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 13317 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 787-10, *General methods of test for pigments and extenders — Part 10: Determination of density — Pyknometer method*.

ISO 8213, *Chemical products for industrial use — Sampling techniques — Solid chemical products in the form of particles varying from powders to coarse lumps*.

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 14887, *Sample preparation — Dispersing procedures for powders in liquids*.

3 Terms and definitions

For the purposes of this part of ISO 13317, the terms and definitions given in ISO 13317-1 apply.

4 Symbols

For the purpose of this part of ISO 13317, the symbols given in ISO 13317-1 and the following apply.

x_{St} Stokes equivalent spherical diameter (m) (practical unit: micrometres, μm)

η Suspending liquid viscosity (Pa·s), (practical unit: mPa·s)

h Sedimentation height (m)

ρ_s Sample density [effective particle density] (kg·m^{-3})

ρ_l Liquid density (kg·m^{-3})

g Acceleration due to gravity (9,807 m·s^{-2})

t Sedimentation time, (seconds, s)

B Function of atomic number of sample elements in beam

C Concentration of sample in beam

I_0 Attenuation of emergent X-ray beam passing through the suspending fluid

I Attenuation of emergent X-ray beam through suspension at settling height h, at time t

D X-ray density [lg$_{10}$ (I_0/I)]

5 Sampling

The sampling procedure according to ISO 13317-1 shall be followed.

6 Representation of results

The results shall be represented according to ISO 9276-1.

7 Principle

The concentration of dispersed sample is measured by attenuation of an X-ray beam. A stable, narrow, collimated beam of X-rays passes through a suspension of the sample: it is detected at a known height from the top of the sample cell. The sample cell is completely filled with sample suspension for the duration of the analysis. The settling height, h, at which the particle concentration, C, is determined, may be reduced during the analysis for the purpose of obtaining a more rapid analysis than would be possible if all measurements were made at the same value of, h. The cumulative mass percent of sample present at a given sedimentation height is continuously determined. The X-ray signal attenuation at the known height is compared to the attenuation with suspending liquid and also to the attenuation with the homogeneously dispersed sample present in the liquid. The attenuation of the emergent X-ray beam is proportional to the mass of powder in the beam, and is expressed by the following formula:

$$I = I_0 \exp(-BC) \quad (1)$$

or

$$C = (-1/B) \ln(I/I_0) \quad (2)$$

The X-ray density, D, is expressed as follows:

$$D = -BC\lg e \tag{3}$$

also

$$D = \lg\left(I_0/I\right) \tag{4}$$

thus

$$D = -BC\lg e = \lg\left(I_0/I\right) \tag{5}$$

The Stokes diameter x_{St}, corresponding to the X-ray density at settling height h and time t, is given by:

$$x_{st} = \left[\frac{18\eta h}{\left(\rho_s - \rho_l\right) gt}\right]^{1/2} \tag{6}$$

The X-ray density D is proportional to the concentration C, and thus mass, of sample in the beam. A plot of the X-ray density D, taken as a function of time t and h, versus

$$\sqrt{18\eta h/\left(\rho_s - \rho_l\right) gt}$$

provides the cumulative mass distribution versus equivalent spherical diameter.

8 Apparatus

8.1 Main apparatus

The main apparatus (see Figure 1) typically consists of a temperature-controlled analysis compartment and mixing chamber; a plumbing system for circulation of suspending liquid or sample suspension; an X-ray source/detector system and a control module for apparatus control, data acquisition and reduction. The sedimentation cell within the temperature-regulated analysis compartment may be repositioned throughout the analysis relative to the signal source and detector to reduce analysis time. Alternative designs, such as one in which the X-ray source and detector move while the sedimentation cell remains stationary, may also be used.

The use of a magnetic stirrer should be avoided if magnetically susceptible particles are to be tested. The dispersion may then be maintained by means of a mechanical stirrer.

8.2 Ancillary apparatus

Ultrasonic bath, probe or high-speed mechanical stirrer capable of 500 revolutions to 1 000 revolutions per minute.

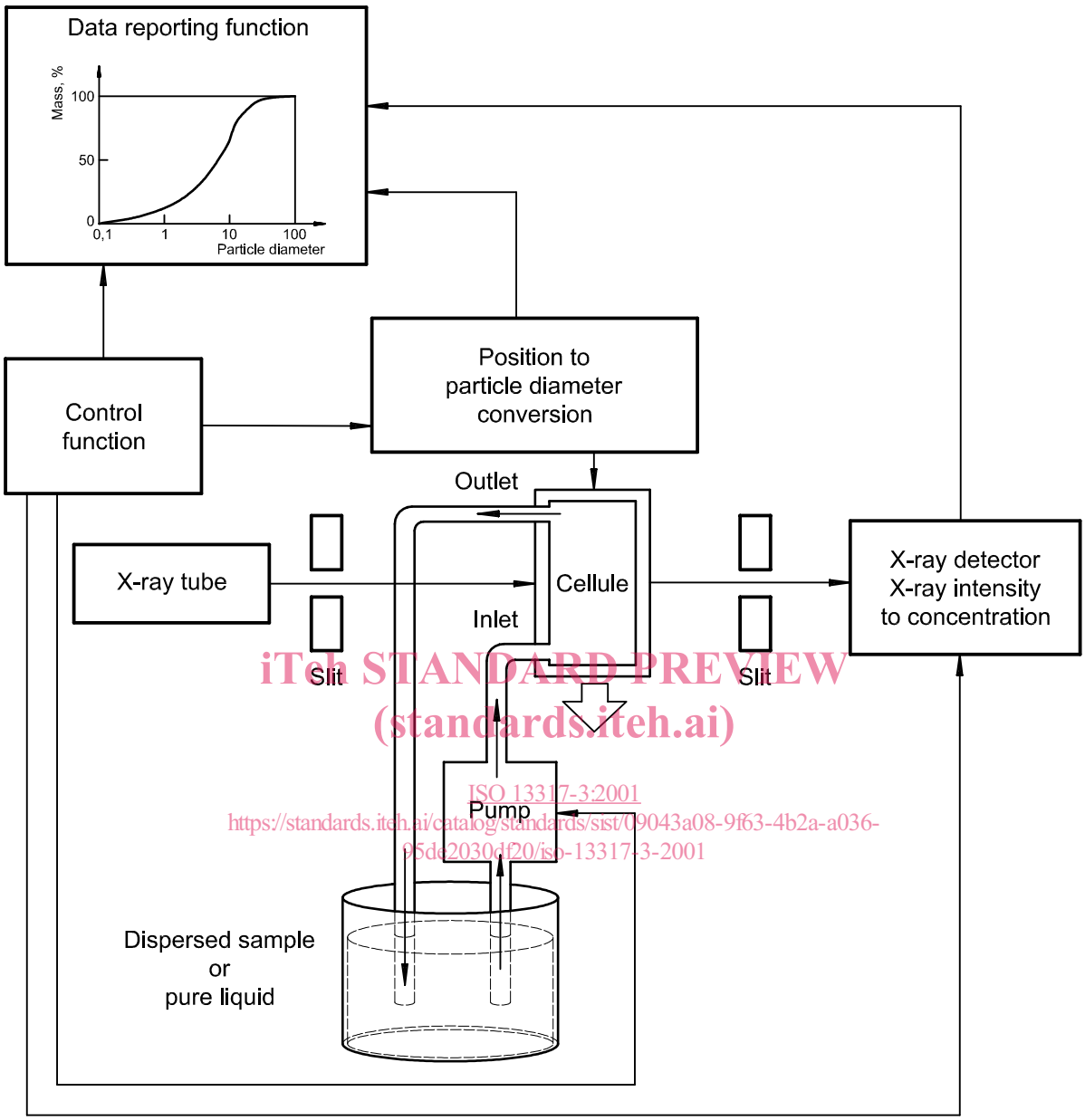

Figure 1 — Schematic of typical apparatus using the X-ray gravitational technique

9 Sample preparation

A representative sample for analysis shall be taken according to ISO 8213. It shall be dispersed according to ISO 14887 in a suspending medium of adequate viscosity and X-ray transparency. The use of dispersing agents and ultrasonics or mechanical stirring to aid dispersion will be recorded. Typically 50 ml of dispersed sample is required for the analysis. The sample concentration will be prepared in accordance with the manufacturers' instructions. Higher sample concentrations may be required with powders having a low X-ray absorptivity coefficient. Knowledge of the exact sample concentration is not required, but the influence of concentration should be checked. The minimum concentration of suspension compatible with the measuring method is preferred (see ISO 13317-1). The required sample concentration will typically produce a reduction in the beam signal of 10 % to 30 % relative to the signal observed with the suspending liquid. The sample may be dispersed in either an aqueous or an organic medium. Any liquid compatible with sample cell materials and having low absorptivity for X-rays may be used. Typical liquids are water, glycols, kerosene, mineral oils, alcohols and mineral spirits. It is recommended that the viscosity of the suspending medium should have a value such that the largest particle to be measured has a Reynolds number of $\leqslant$ 0,25 (see ISO 13317-1). Any temperature change of the suspension should be minimized during a measurement (see 10.3).

The viscosity and density of the suspending liquid at the temperature of the analysis will be reported. The effective particle density of the sample is also reported. The suspending liquid, including dispersants at the analysis concentration, may be used as the displacement fluid to experimentally determine the effective particle density.

10 Procedure

10.1 General

See ISO 13317-1 for guidance on restrictions of upper size limit and lower size limit, and on test conditions. The validity of the measurement will be dependent on the Stokes equation applying to creeping flow for the suspension under study. The lower size limitation will in part be a function of the ability of the system to minimize thermal convection and mechanical effects. Additionally, for the fine size fraction, Brownian motion effects may contribute to the vertical spread of particles of the same size originating from the same initial settling height.

10.2 Base-line determination

Determine the attenuation of the X-ray signal with only suspending liquid in the sample cell (0 % solids). Where possible, a base-line scan of the entire measuring portion of the cell is advised for subsequent correction of the corresponding sample suspension data. The base-line scan of the entire measuring portion of the cell is recommended as this will provide optimum correction for variability in the cell window thickness, or non-parallel alignment of the cell windows.

10.3 Temperature equilibration

Place the dispersed sample suspension in the mixing chamber, and circulate the suspension through the sample cell for typically 60 s to 90 s. Confirmation is obtained that the analysis compartment is operating within the specified temperature band, preferably within $\pm$ 1 K of the temperature set point.

10.4 Bubble elimination

Check for the presence of air bubbles in the sample cell either manually or automatically. If bubbles are detected, remove them by manual or automatic operation. If air bubbles are detected, make a further check prior to commencing the analysis. Repeat the bubble detection and elimination steps until confirmation is obtained that no bubbles are detected.