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ISO 13317-2

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Determination of particle size distribution by gravitational liquid sedimentation methods —

Part 2: Fixed pipette method

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

Partie 2: Méthode de la pipette fixe

Reference number
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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

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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 13317-2 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.

Introduction

This part of ISO 13317 describes a method to determine particle size distribution using a fixed position pipette apparatus commonly referred to as the Andreasen pipette. The Andreasen pipette employs an incremental method of analysis which gives the mass distribution directly. In incremental methods, the solids concentration at the measurement level determines directly the proportion by mass of the analysis sample that consists of particles having a diameter less than that corresponding to the velocity of fall at the time of sampling.

Determination of particle size distribution by gravitational liquid sedimentation methods —

Part 2:
Fixed pipette method

1 Scope

This part of ISO 13317 describes a method using a pipette to determine particle size distribution, typically in the size range 1 µm to 100 µm, by gravitational sedimentation in a liquid.

NOTE This part of ISO 13317 may involve hazardous materials operations and equipment. This part of ISO 13317 does not purport to address all the safety problems 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 to determine the applicability of the regulatory limitations prior to its use.

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 method is applicable to powders made up of particles having the same density and of comparable shape. Particles should not undergo any chemical or physical change in the suspension liquid. It is necessary that the particles have a density higher than that of the liquid.

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 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, definitions and symbols

3.1 Terms and definitions

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

3.2 Symbols

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

Size	Symbol	Unit	Derivative unit
Calibrated volume of the sedimentation vessel	V	l	ml
Volume of the pipette to the graduation mark	V_p	l	ml
Mass of sample solids in 10 ml at time t_0	W_0	kg	g
Mass of sample solids in 10 ml at time t_n	W_n	kg	g
Pipette sampling height (or drop height)	h_n	m	cm
Sample withdrawal time	t_n	s	–
Stokes diameter corresponding to withdrawal time t_0	x_n	m	µm
Cumulative frequency by mass at withdrawal time t_n; it is equal to W_n / W_0	F_n	Dimensionless	Dimensionless

4 Sampling

The sampling method given in ISO 13317-1 applies.

5 The fixed position pipette (Andreasen) method

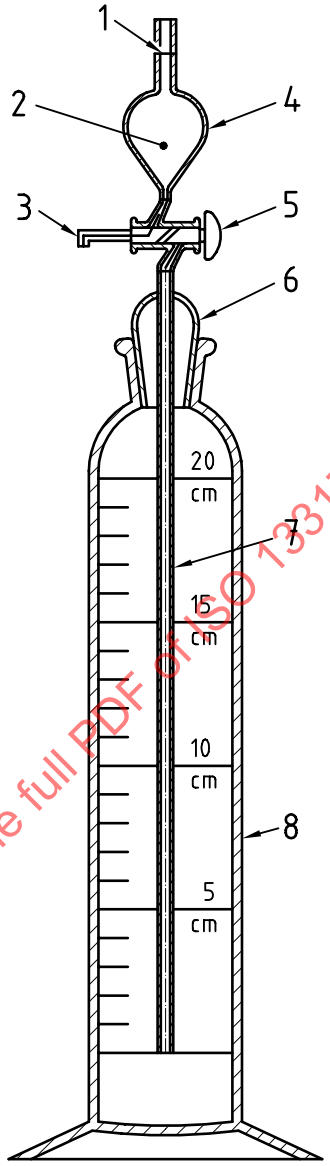
5.1 Principle

Samples are withdrawn from a suspension during sedimentation by means of a calibrated pipette at a series of known times after initial agitation with the tip of the pipette being at a known depth h below the surface. After time, t, the sample withdrawn contains only those particles with Stokes diameters less than that of particles settling at rate h/t, since all particles larger than this will have settled below the sampling point. The cumulative undersize distribution by mass of the powder is obtained directly by weighing the residue after removal of the suspending medium from each extracted sample.

5.2 Apparatus

5.2.1 Sedimentation vessel

The sedimentation vessel is of glass of about 5 cm internal diameter and having a graduated scale from 0 cm to 20 cm marked on the side of the vessel (Figure 1). The graduated scale may be subdivided at 5 mm or 10 mm intervals. The zero graduation should be not less than 25 mm from the inside base of the vessel so that the capacity, when filled to the 20 cm mark, is about 500 ml. It is important that the walls of the cylinder are vertical. The scale should also be vertical and have an accuracy of ± 1 mm.



Key

1 Graduation mark
2 10 ml capacity
3 Discharge tube
4 Pipette bulb
5 Two-way tap
6 Dome (vented)
7 Pipette capillary stem
8 Sedimentation vessel

Figure 1 — Fixed position pipette (Andreasen)

5.2.2 Pipette

The pipette is fitted with a two-way tap and a side discharge tube. The capacity of the pipette to the graduation mark is 10 ml. A bell-shaped dome (with vent hole, not shown in Figure 1) is fused to the pipette with a ground-glass joint to fit the neck of the sedimentation vessel. The pipette bulb should be shaped as in Figure 1. The inlet to the pipette stem should be level with the zero mark on the sedimentation vessel and the stem should be parallel to the walls of the sedimentation vessel when in position. The stem from the pipette bulb to the sampling inlet is constructed of capillary glass tube with a bore of not less than 1 mm nor more than 1,3 mm. The tube above the bulb should be a 3,5 mm bore.

NOTE A variation of the fixed position Andreasen pipette exists as the Leschonski modification. In this variation, the pipette is extended to the bottom of the vessel and the sample is typically withdrawn through four apertures around the circumference of the pipette at a fixed depth of about 30 mm above the bottom of the vessel. Additionally, a subsidiary bulb with a volume matching that of the capillary is intended to remove the systematic positive error (i.e. over-estimation of the percentage undersize) resulting from liquid retained in the capillary tube after each withdrawal. In this way, the sample residue is removed from the capillary before taking the next sample. In practice, differences between results obtained using these modifications and results using the Andreasen pipette method may not be significant for most materials.

5.3 Ancillary apparatus

The ancillary apparatus includes:

— constant temperature enclosure. For prolonged analyses (greater than 1 h), a constant temperature bath is required into which the sedimentation vessel can be immersed. It is essential that no vibration be imparted to the sedimentation vessel during the analysis. See 6.3;

— dispersing vessel of appropriate dimensions;

— flexible spatula;

— ultrasonic bath or probe, bottle shaker or high speed mechanical stirrer;

— balance, having an accuracy of 0,1 mg, or better;

— drying oven capable of being maintained within the temperature range suitable for the evaporation of the suspending liquid, e.g. 378,15 K to 383,15 K for water;

— wide-mouthed weighing bottles or beakers suitable for evaporation, of capacity not less than 20 ml or aluminium foil containers. Aluminium foil containers, as purchased, may contain a layer of grease and should be oven dried before use. These are preferable to glass containers since they typically weigh about 1 g compared to 20 g to 30 g for the glass containers. Since a difference in mass is required, a greater accuracy can result from the use of aluminium foil containers. With certain sample systems, filtration may be preferred;

— desiccator in which the sample containers can be cooled after drying. Some dispersing agents are hygroscopic and the sample may absorb moisture if allowed to cool in ambient conditions;

— timer with a range of at least 3 600 s and a resolution of 1 s.

6 Preparation

6.1 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. Make up the test portion to give a concentration of about 0,2 % by volume. Record the use of ultrasonics or mechanical stirring to aid dispersion.

6.2 Calibration of sedimentation vessel

Determine the volume, (V), of the sedimentation vessel, to an accuracy of 0,3 %, by filling with distilled water up to the 20 ml mark with the pipette in position. Remove typically eight 10 ml samples and determine the final height h_n after each withdrawal.

6.3 Calibration of pipette

Clean and dry the pipette. Partly fill the sedimentation vessel with distilled water. Set the tap in the sampling position and suck water into the bulb up to the graduation mark. Turn the tap to the discharge position and allow the water to drain into a tared weighing container. Apply minimum pressure to blow any water remaining in the bulb and in the discharge tube into the weighing container. Weigh the container to the nearest 0,001 g and calculate the internal volume V_p of the pipette.

The values for the volume of the sedimentation vessel V, and the pipette volume V_p, are used to calculate the sample concentrations in the sedimentation vessel and the pipette (see Table A.1). The calculated concentration using V and V_p should agree (within statistical uncertainty) with the initial concentration for samples extracted at time zero in Table A.1.

6.4 Temperature

Maintain the temperature in accordance with ISO 13317-1.

6.5 Withdrawal time calculation

Calculate the withdrawal time for the first fraction using Stokes law in the following form:

$$t_1 = \frac{18\eta h}{(\rho_s - \rho_l) g x_1^2} \quad (1)$$

where

x_1 is the Stokes diameter for the first fraction, which must not exceed the upper critical Stokes diameter, see ISO 13317-1.

The initial height h is normally 20 cm, but other heights in the 10 cm to 20 cm range may be used. Extract at times calculated from selected diameters or at times in a ratio of two to one. The first sample extraction time should be not less than 60 s after the test begins.

If the size distribution extends above the coarse end of the size for the liquid, then repeat the analysis using a higher viscosity liquid until there is sufficient overlap between the two determined distributions.

7 Procedure

7.1 Initial sample preparation

Fill the sedimentation vessel to about the 20 cm fiduciary mark (about 500 ml) with an initial homogeneous concentration of about 0,2 % by volume. Extract 2 × 10 ml from the agitated suspension for drying and weighing separately as a check for the 100 % value. It is preferable to use the pipette for this extraction so that the volumes throughout the analyses are measured with the same pipette. This sample should be dried at the same time and under the same conditions as the measurement sample, since the mass loss of the dispersing agent may depend on drying conditions. It is also advisable to dry a sample (ca. 20 ml) of the dispersing solution in order to determine whether the dried mass adsorbs moisture from air over time.

7.2 Sample extraction procedure

The time to fill the pipette should be about 20 s; commence extraction 10 s before the withdrawal time and terminate about 10 s after. Discharge the fraction into a tared weighing container. When the pipette bulb has drained, draw 5 ml to 7 ml of suspending liquid without dispersant into the bulb through the outlet tap.

Use vacuum suction to draw liquid into the pipette bulb. Do not use oral suction.

Remove the container whilst maintaining a slight vacuum in order to draw air into the pipette bulb; then apply pressure and wash the liquid into the tared weighing container. This removes any particles adhering to the surface. Maintain the stem of the pipette full of suspension.

7.3 Measurement procedure

Transfer the sedimentation vessel to a constant temperature enclosure or bath and allow to stand until temperature equilibrium has been attained, and record this temperature. When temperature equilibrium has been attained, mix the contents thoroughly by placing a finger over the vent hole in the dome and inverting continuously for 1 min.

Withdraw two samples as described in 7.2. If it is found that the masses of these two dried samples do not agree to within 2 %, reject the analysis. These measurements are used to determine the initial concentration of the

suspension. The average of these two masses constitutes the 100 % mass. Repeat the mixing then set the sedimentation vessel in a vertical position whilst simultaneously starting the timer. Withdraw fractions at time intervals according to 6.5.

The depth of immersion of the pipette decreases as each fraction is withdrawn. Determine the exact decrease in depth by experiment and allow for this decrease in the subsequent calculations. For example, if the decrease is 0,40 cm for each fraction withdrawn, and that at time zero the depth of immersion of the pipette was 19,2 cm, then the depth after the first timed fraction has been withdrawn will be 18,8 cm and the mean depth used for calculation of the initial diameter will be 19,0 cm. The mean depth for the second fraction will be 18,6 cm, and so on.

With some viscous liquids, a 20 s extraction time may prove insufficient and will need to be increased. If air bubbles are drawn in through the stopcock, then grease should be applied to it.

Repeat the sampling procedure at times in a two to one progression, e.g. 2, 4, 8, 16, 32, 64 and perhaps 128 min, depending on the fineness of the powder. The analysis may be terminated when the extracted suspension is relatively clear.

8 Assay of fractions

Accurately assay each fraction. If this is made by drying and weighing, then adopt the following procedure.

Evaporate each fraction in its weighing container to dryness (as indicated by constant weight) in an oven maintained at a temperature suitable to the particular suspending liquid, and subsequently cool each container in a desiccator.

Weigh the container and the dried contents to the nearest 0,1 mg and determine the mass W_n of solid material in each of the fractions (n = 1, 2, 3...). Make allowance for the mass of the dispersing agent. This is best made by drying a 20 ml sample of suspending liquid plus dispersant. With powders or dispersing agents that are hygroscopic, it is necessary to weigh without delay after removal from the desiccator.

The removal of the suspending liquid may be expedited by first centrifuging the collected fractions in centrifuge tubes and decanting the supernatant liquid from the firmly compacted solid material. Then proceed as described above.

Fractions may be assayed by other appropriate methods, e.g. chemical or colorimetric.

9 Tests in duplicate and validation

9.1 Tests in duplicate

Two test portions shall be analysed initially to determine the particle size distribution. The results are acceptable if the proportions by mass for the same Stokes diameter do not differ by more than 2 %. If the results do not agree within 2 %, then the analysis shall be repeated.

9.2 Validation

The checking at regular intervals of both operator procedure and instrument performance is essential to validate the test results. The frequency of checking is a matter for each laboratory to determine.

It is recommended that validation be done using certified reference materials. Suitable materials would be the Certified Reference Materials from the Bureau Community of Reference (BCR) and the US National Institute of Science and Technology (NIST), as referred to in ISO 13317-1:2000.

A record of all validation activities shall be maintained.