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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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Contents

Page

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

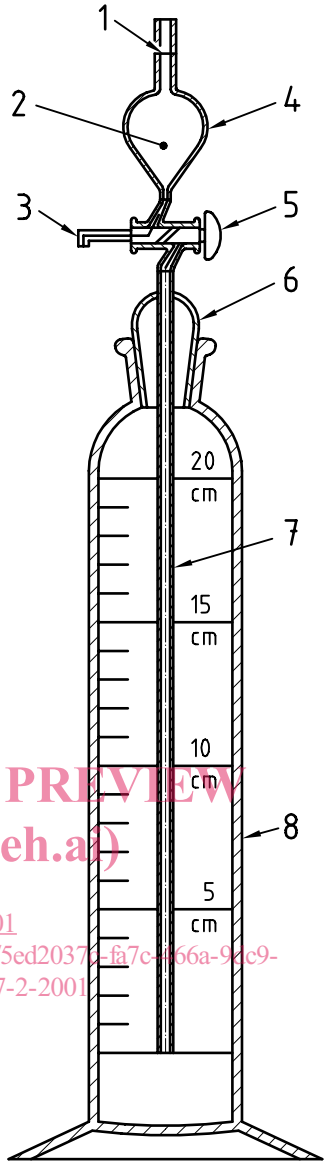
5.1 Principle

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.