INTERNATIONAL STANDARD

ISO 13317-4

First edition
2014-11-15

Determination of particle size distribution by gravitational liquid sedimentation methods —

Part 4: Balance method

Détermination de la distribution granulométrique par les méthodes de sédimentation par gravité dans un liquide —
Partie 4: Méthode de la balance

Reference number
ISO 13317-4:2014(E)

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Published in Switzerland

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

The committee responsible for this document is ISO/TC 24, *Particle characterization including sieving*, Subcommittee SC 4, *Particle characterization*.

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*

— *Part 4: Balance method*

Introduction

This document is a part of the ISO 13317 series. It describes a method to determine particle size distribution by use of the mass of particles deposited at a balance. This method is based on a direct mass measurement and gives immediately the mass-based distribution of particle diameter. This method does not use any fitting parameters. The results obtained are Stokes diameters.

Determination of particle size distribution by gravitational liquid sedimentation methods —

Part 4:
Balance method

1 Scope

This part of ISO 13317 specifies the method for the determination of particle size distribution by the mass of particles settling under gravity in liquid. This method is based on a direct mass measurement and gives the mass distribution of equivalent spherical particle diameter. Typically, the gravitational liquid sedimentation method applies to samples in the 1 µm to 100 µm size range and where the sedimentation condition for particle Reynolds number less than 0,25 is satisfied.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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*

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*

ISO 14488, *Particulate materials — Sampling and sample splitting for the determination of particulate properties*

3 Terms and definitions

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

3.1
apparent particle density
particle mass divided by the volume it would occupy including all internal pores

4 Symbols

For the purposes of this part of ISO 13317, the following symbols apply.

Quantity	Symbol	Unit	Derivative Unit
Mass of dispersion medium	m_l	kg	—
Maximum amount of sample same as the first line	m_s	kg	—
Apparent particle density	ρ_s	$kg \cdot m^{-3}$	
Liquid density	ρ_l	$kg \cdot m^{-3}$	
Cumulative mass for particle diameter greater than x_i	M_i	kg	
Total mass of particles	M_{max}	kg	
Sedimentation time for particle having a diameter x_i and time, respectively	t_i, t	s	—
Particle diameter	x_i	m	—
Liquid viscosity	η	$Pa \cdot s$	
Sedimentation distance	h	m	
Gravity acceleration	g	$m \cdot s^{-2}$	
Cumulative distribution by mass for particle diameter x_i	$Q_{3,i}$	dimensionless	—
Sedimentation mass at time t_i and t_{end}, respectively	G_{ti}, G_{end}	kg	—
Particle diameter corresponding to time t required to move distance h	x	m	—
Maximum particle diameter	x_{max}	m	—
Sedimentation velocity	$v(x)$	$m \cdot s^{-1}$	
Response function	$g(t,x)$	dimensionless	—
Distribution density by mass	$q_3(x)$	m^{-1}	
Distribution density by mass at time t_i	$q_{3,i}(x)$	m^{-1}	
Parameter defined by Formula (A.6)	$\gamma_i^{(k)}$	dimensionless	—

5 Principle of Method

This method is based on particle settling in a gravitational field and uniformly dispersed particles at start (homogeneous technique). The relationship between settling velocity v, that means the time t required to settle the distance h, is defined by the following formula according to Stokes law.

$$v = \frac{h}{t} = \frac{(\rho_s - \rho_l) g x^2}{18\eta} \tag{1}$$

From Formula (1), the Stokes diameter x is directly obtained.

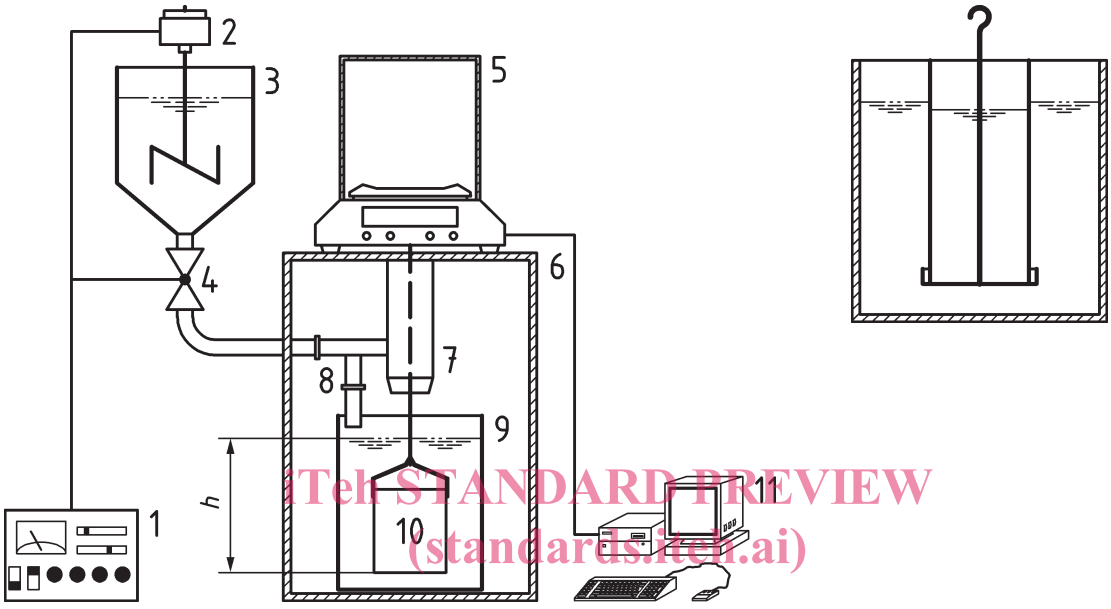
$$x = \sqrt{\frac{18\eta h}{(\rho_s - \rho_l) g t}} \tag{2}$$

The above formulae can be applied for Reynolds numbers of sedimenting particles less than 0,25. The determination of the particle size by gravitational sedimentation is a cumulative method (see ISO 13317-1). In this case, the method determines the rate at which solid particles settle from the suspension in a known volume of cylindrical vessel to a given distance. The mass of particles settled at time t is summed up from the mass of all particles of a diameter greater than x and in part of particles of diameters less than x. This method does not use any fitting parameters to obtain particle size distribution.

6 Measurement apparatus

a) Measurement apparatus to obtain the mass of the sediment

The apparatus measures continuously the increase of the mass of the particles sedimented out from the suspension. The apparatus shown in Figure 1 a) typically consists of a sedimentation container and mass measuring system (see Reference [2]). Figure 1 b) shows other type of sedimentation tray (see Reference [3]). For the mass measurement apparatus (electronic balance), detection precision shall be at least 1 % of the total mass of particles in the detection tray.



Key

1	controller	7	main inlet pipe
2	stirrer	8	bypass
3	dispersion bath	9	sedimentation container
4	valve	10	detection tray
5	precision electronic balance	11	personal computer
6	glove box		

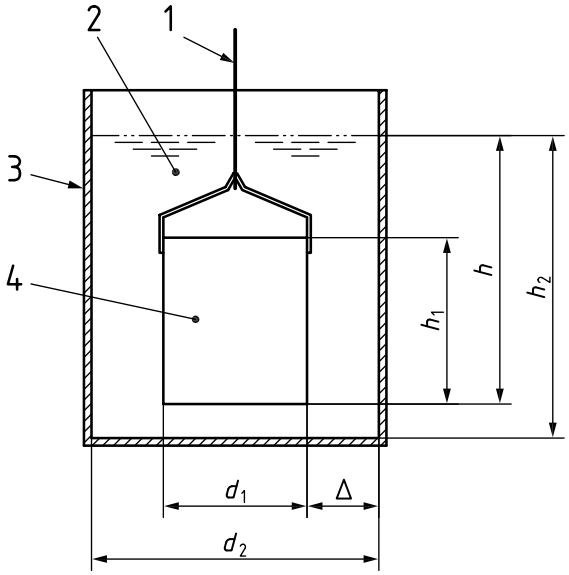
Figure 1 — Measurement apparatus — Sedimentation balance for particles in liquid

b) Sedimentation bath

A typical sedimentation bath is shown in Figure 2. The detection tray has a cylindrical side wall and the clearance between the side wall of the tray and sedimentation bath shall be large enough to avoid interaction between them. Dimensions for the tray are shown in Figure 2. The following ratios should apply:

— $0{,}88 < h / d_2 < 1{,}15,\ 1{,}15 < h_2 / d_2 < 1{,}48;$

— $0{,}43 < d_1 / d_2 < 0{,}71,\ 0{,}61 < h_1 / d_2 < 0{,}90.$



Key

1 support wire
2 suspension
3 sedimentation bath
4 detection tray
h sedimentation distance

Figure 2 — Detection container

c) Dispersion bath

In the bath, the particles have to be dispersed before measurement and the dispersion state has to be checked (see ISO 14887).

d) Measuring system

Figure 1 shows a schematic diagram of the measuring system. By use of a time-controlled valve (key 4), a precision electronic balance (key 5), and a personal computer (key 11), the cumulative mass of the sediment on the tray is automatically recorded.

7 Measuring method

7.1 Measurement of density

The apparent particle density for the setting shall be measured (refer to ISO 13317-1:2001, 5.4).

7.2 Preparation method of suspension

A representative sample according to ISO 14488 shall be dispersed according to ISO 14887 in a dispersion medium.

7.2.1 Dispersion medium

When the test particles are not well dispersed by the dispersion medium, it is necessary to use a suitable dispersing agent. In this case, the dispersion medium should satisfy the following requirements.

a) Viscosity of the dispersion medium has to be in a suitable range regarding the sedimentation time.

b) Flocculation and agglomeration shall be avoided also during sedimentation process.

c) In the dispersion medium, the solid phase shall be insoluble, chemically and hydro-dynamically stable, and not change its volume.

d) Avoid using volatile liquid as sedimentation medium.

7.2.2 Suspension

A suitable amount of test particles, dispersion medium, and if necessary, a dispersion agent shall be well mixed in the dispersion bath.

The volume concentration of test particles in the dispersion medium should be less than 0,1 %. At higher volume concentrations, the hindrance function accounting for the hydrodynamic hindered settling effect has to be taken into account. Particle settling velocity reads:

$$v = \frac{h}{t} = \frac{(\rho_s - \rho_l) g x^2}{18\eta} f(\varepsilon) \tag{3}$$

where $f(\varepsilon)$ is the sample specific hindrance function (see ISO 13317-1) and ε is liquid volume fraction defined by

$$\varepsilon = \frac{(1-w)\rho_s}{w\rho_l + (1-w)\rho_s} = 1 - \phi \tag{4}$$

The values of ϕ and w are particle volume fraction and mass fraction, respectively.

7.3 Measurement

The following procedures shall be performed to take a measurement.

a) Have the dispersion medium prepared. The temperature of the dispersion medium should be constant during the preparation and measurement (see Annex A). Initial particle mass of W gram is given into a small beaker and 10 cm^3 of dispersion medium is directly added to the test particles and well mixed to prepare a pre-suspension. After adding the appropriate amount of dispersing medium, the supersonic vibration is applied to the pre-suspension. Finally the necessary amount of dispersion medium has to be added, and the suspension has to be well mixed. After stirring of the suspension, it is supplied to the sedimentation bath and the measurement is started. During the preparation and the supply of the suspension to the dispersion bath, air bubbles shall be avoided.

b) The measurement is stopped when all particles are settled down and the mass of sediment shows no further increase. In the case that fines are still suspended in the dispersion, the suspension is sampled with a siphon. After drying the suspension, the mass of the fines is determined.