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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

ICS 19.120

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Foreword

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ISO 13317-4 was prepared by Technical Committee 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*

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Introduction

This document is a part of ISO 13317 “Determination of particle size distributions by gravitational liquid sedimentation methods”. 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.

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Determination of particle size distribution by gravitational liquid sedimentation methods —

Part 4:
Balance method

1 Scope

This standard specifies the method for 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.

2 Normative references

The following referenced documents are indispensable for the application 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*

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 of 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 excluding all pores, and surface fissures (ISO 13317-1)

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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	kg/m/s	Pa 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 Equation (A.6)	$\gamma_i^{(k)}$	dimensionless	—

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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 equation according to Stokes law.

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

From equation (1) the Stokes equivalent spherical diameter x is directly obtained.

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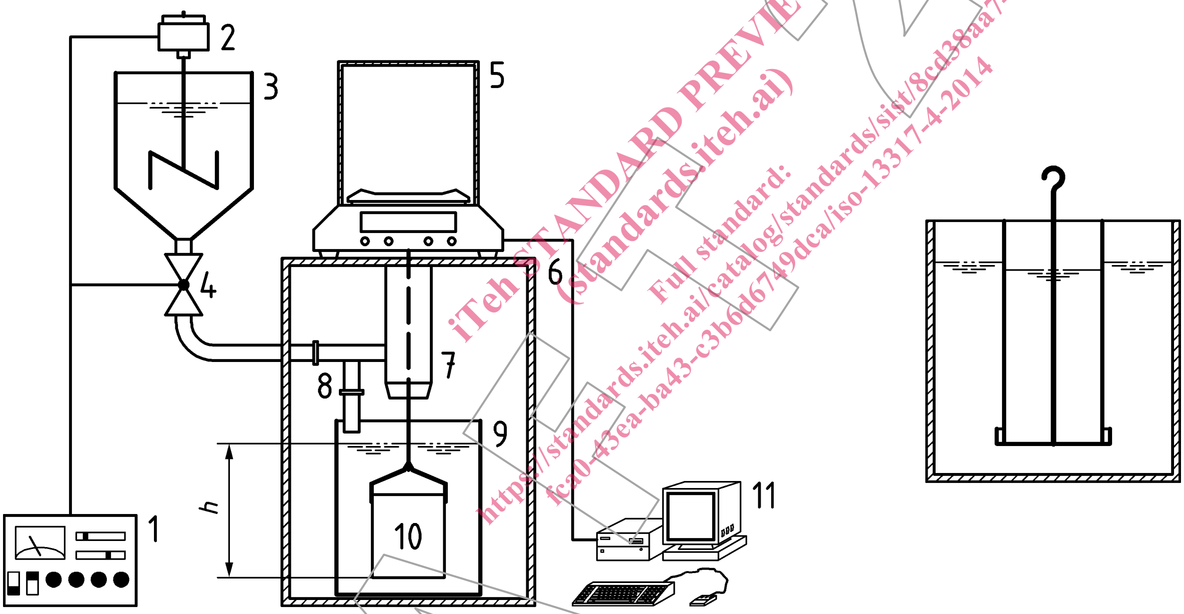
$$x = \sqrt{\frac{18\eta\, h}{(\rho_\mathrm{s} - \rho_\mathrm{l})g\, t}} \tag{2}$$

The above equations 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 (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 Measuring apparatus

a) Measuring 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 (ref. 2). Figure 1-(b) shows other type of sedimentation tray (ref. 3). For the mass measuring apparatus (electronic balance) detection precision shall be at least 1 % of total mass of particles in the detection tray.



a) b)

Key

1 Controller
2 Stirrer
3 Dispersion bath
4 Valve
5 Precision electronic balance
6 Glove box
7 Main inlet pipe
8 Bypass
9 Sedimentation container
10 Detection tray
11 Personal computer

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:

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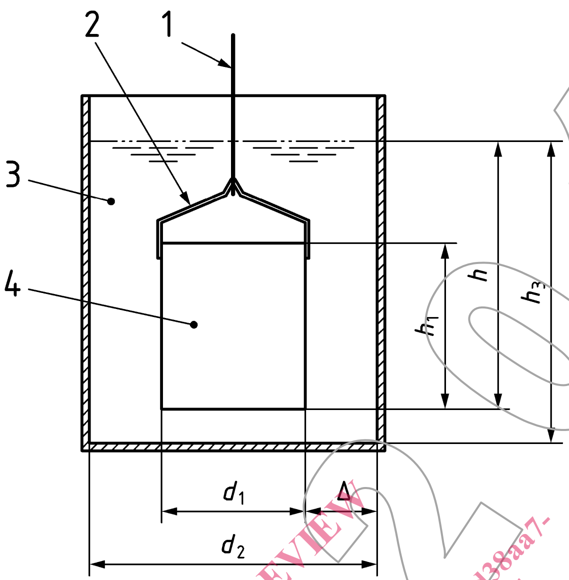
$h/d_2 = 1.14$, $h_3/d_2 = 1.48$,

$0.55 < d_1/d_2 < 0.71$, $0.78 < h_1/d_2 < 0.90$



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 4, a precision electronic balance 5 and a personal computer 11, the cumulative mass of the sediment on the tray is automatically recorded.

7 Measuring method

7.1 Measurement of density

The effective particle density shall be measured (refer to 5.4 of ISO 13317-1:2001).

7.2 Preparation method of suspension

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

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