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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

Reference number
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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 · 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.

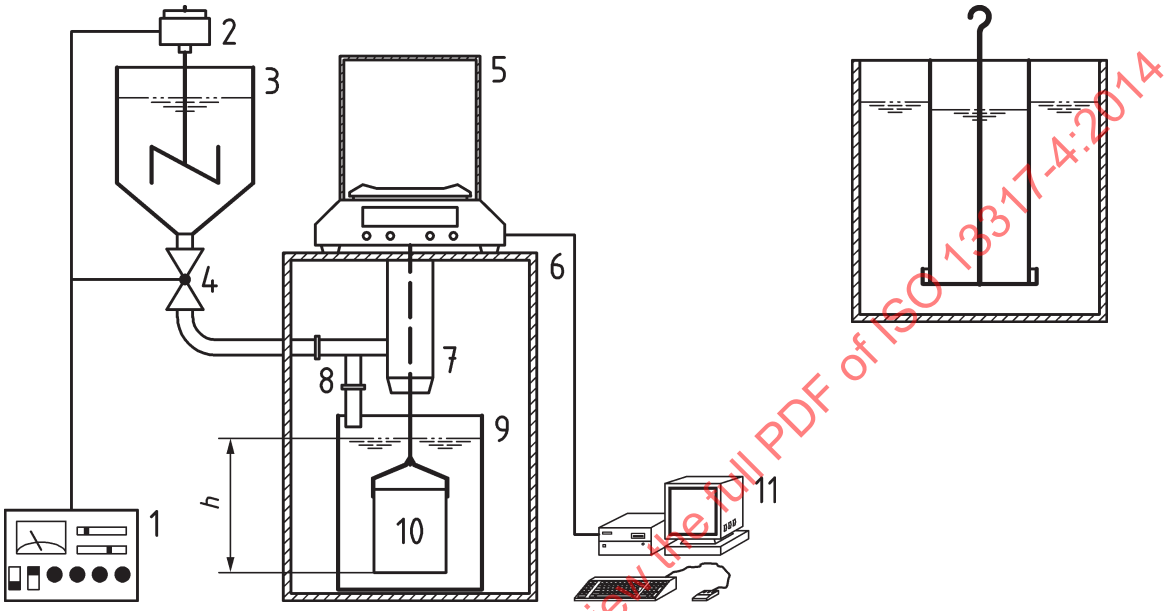
$$x = \sqrt{\frac{18\eta h}{(\rho_s - \rho_l) g t}} \tag{2}$$

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.



a) b)

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		

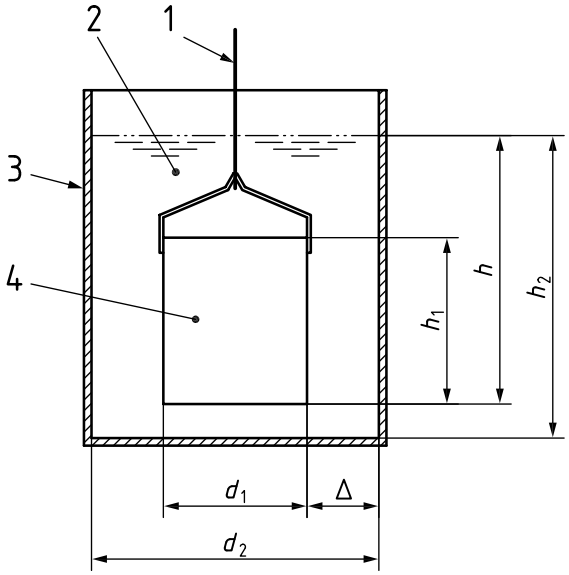
Figure 1 — Measurement apparatus — Sedimentation balance for particles in liquid

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

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

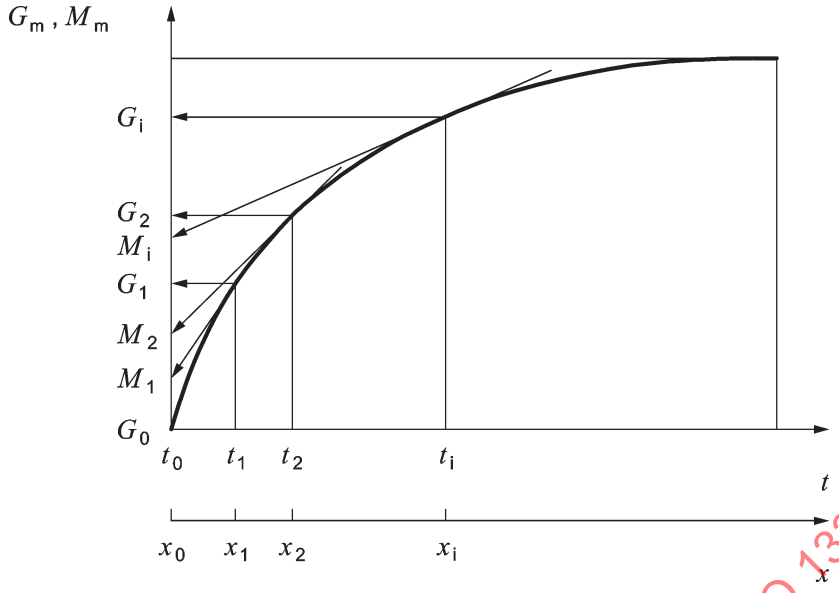
The values of ϕ and w are particle volume fraction and mass fraction, respectively.

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.



Key

t time [s]

x particle diameter [µm]

Figure 3 — Typical sedimentation curve

7.4 Data analysis

Figure 3 depicts a typical record from the balance indicating the mass increase of the sediment with time. This is the sedimentation curve $G(t)$. In Figure 3, G_i is the mass at the sedimentation time t_i, and M_i is the intercept of the tangential line at the point (t_i, G_i) of the sedimentation curve. When the mass of sediment reaches a constant value G_m (see Figure 3), maximum time, corresponding to the minimum Stokes diameter, is obtained assuming that all particles have the same apparent density.

7.4.1 Differential method

a) Sedimentation times t_1, t_2... t_n corresponding to particle diameters x_1, x_2... x_n are calculated using Formula (1).

$$t_i = \frac{18\eta h}{g(\rho_s - \rho_l)x_i^2} \tag{5}$$

where

t_i is the sedimentation time (s);

h is the sedimentation distance from water surface to the bottom of the detection tray (m);

η is the viscosity of dispersion medium (Pa·s);

g is the gravity acceleration (m/s^2);

ρ_s, ρ_l are the densities of particle and dispersion medium, respectively (kg/m^3);

x_i is the particle diameter corresponding to t_i (Stokes diameter) (m).

In case of volume concentrations higher at 0,1 %, Formulae (3) and (4) apply.

$$t_i = \frac{18\eta h f(\varepsilon)}{g(\rho_s - \rho_l) x_i^2} \tag{6}$$

b) From the experimental sedimentation curve, the relation between the sedimentation time and mass of sediment is obtained (see Figure 3). For each particle diameters $x_1, x_2, \ldots x_n$ corresponding to sedimentation times $t_1, t_2, \ldots t_n$, a tangential line is drawn and by extrapolating the tangential lines to the ordinate, the values of intercepts $M_1, M_2, \ldots M_n$ are obtained. These values are proportional to the cumulative distribution by mass for the particle classes of diameters $x_1, x_2, \ldots x_n$.

c) The cumulative distribution by mass for the particle diameter x_i is calculated as follows:

$$Q_{3,i} = 1 - \frac{M_i}{M_{max}} \tag{7}$$

where

$Q_{3,i}$ is the cumulative distribution by mass;

M_i is the cumulative mass for the particle diameter greater than x_i;

M_{max} is the total mass of particles.

For the case that small particles are left in the sedimentation bath, the mass of suspended small particles in the cylindrical volume of the detection tray shall be determined by the method in 7.3 (b). The mass of sediment is the sum of the final data of the sedimentation curve and mass of the particles left in the sedimentation bath.

7.4.2 Data reduction by matrix method

When the masses of particles at time $t = t$ and $t = t_{end}$ are represented by G_t and G_m respectively, the following formula may be applied.[1]

$$\frac{G_t}{G_m} = \int_0^{x_{max}} g(t,x) q_3(x) dx \tag{8}$$

The value of $g(t,x)$ is defined as follows:

$$g(t,x) = \frac{v(x)t}{h} : 0 < x \le x_e \tag{9}$$

$$g(t,x) = 1 \quad : x_e < x \le x_{max} \tag{10}$$

where h is the sedimentation distance and x_e is a particle diameter corresponding to the particle which requires time t to pass the sedimentation distance h. x_{max} is the maximum particle diameter and $v(x)$ is the sedimentation velocity for a particle of diameter x.

By using Formula (8) for each particle size range, a matrix is obtained. Particle size distribution $q_3(x)$ is determined by solving the matrix through iterative procedure.

8 Accuracy

The theoretical maximum sedimentation mass G_m is calculated by Formula 11.

$$G_m = W\frac{V_1}{V}\frac{\rho_s - \rho_l}{\rho_s} \tag{11}$$

where W is the initial particle mass in volume V and V_1 is the volume of the detection tray from liquid surface to the tray bottom. Due to the uncertainty of the apparent particle density, the uncertainty of the theoretical sedimentation mass is estimated by Formula 12.

$$\Delta G_m = \sqrt{\left(\frac{\partial G_m}{\partial \rho_s}\Delta\rho_s\right)^2} = \sqrt{\left(W\frac{V_1}{V}\frac{\rho_l}{{\rho_s}^2}\Delta\rho_s\right)^2} \tag{12}$$

Due to the uncertainty of the apparent particle density, the uncertainty of the Stokes diameter is calculated from Formula 13.

$$\Delta x = \sqrt{\left(\frac{\partial x}{\partial \rho_s}\Delta\rho_s\right)^2} = \sqrt{\left(\frac{18\eta h}{4gt}\right)\frac{1}{(\rho_s - \rho_l)^3}{\Delta\rho_s}^2} \tag{13}$$

9 Size range of measurement

Maximum and minimum particle diameters [x_1 to x_2 (µm)] should be in the range determined by the following formulae.

$$x_1 = \sqrt{\frac{18\eta h}{(\rho_s - \rho_l)gt_1}} \quad x_2 = \sqrt{\frac{18\eta h}{(\rho_s - \rho_l)gt_2}} \tag{14}$$

where t_1 and t_2 correspond to maximum and minimum measuring time, respectively.

On the other hand, the maximum size has to be determined by Formula (15).

$$x_1 \leq 0{,}25\frac{\eta}{\rho_l \nu} \tag{15}$$

10 Data representation

The measurement results shall be documented according to ISO 13317-1 and include the following information:

— a reference to this part of ISO 13317, i.e. ISO 13317-4;

— date of the test;

— unique report identification;

— operator identification;

— instrument type used;

— sample identification;

— sample apparent density;

— sample mass and concentration;

— suspending liquid;

— temperature;

— liquid density;

— liquid viscosity;

— dispersing agent and concentration;

— method of dispersion;

— suspension volume;

— minimum and maximum time.

Data can be illustrated by a figure whose abscissa depicts equivalent particle diameter and ordinate displays the corresponding cumulative distribution.

Annex A
(informative)

Data reduction by matrix method

A.1 Experimental conditions

a) Temperature control is necessary to avoid convective flow due to local liquid density difference and to know the exact material properties according to Formula (1). The temperature control should ensure a measurement temperature of ± 1 K. Change of temperature with time should be less than 0,05 K/min.

b) Any vibrations as well as air circulation in the place of the measurement apparatus have to be avoided.

A.2 Data reduction by matrix method

Data reduction by use of differential method on sedimentation curves sometimes causes errors due to the differential processing of curve. On the other hand, this error can be reduced by data reduction by means of the matrix method.

When the mass of particles at time $t = t$ and $t = t_{end}$ is represented by G_{ti} and G_m respectively, and sedimentation distance by h, Formula (A.1) is satisfied.

$$\frac{G_{ti}}{G_m} = \int_{x_e}^{x_{max}} q_3(x)dx + \int_0^{x_e} \frac{v(x)t_i}{h} q_3(x)dx \tag{A.1}$$

where x_e is a particle diameter corresponding to the particle which requires time t to move sedimentation distance h. x_{max} is a maximum particle diameter and $v(x)$ is a sedimentation velocity for a particle of diameter x.

Particle size distribution at time t_i is denoted by $q_{3,i}(x)$, Formula (A.2) is satisfied.

$$G(t_i) = G_m \int_0^{x_{max}} g(t_i, x) q_{3,i}(x)dx \quad (i = 1, \ldots, n) \tag{A.2}$$

$$g(t,x) = \frac{v(x)t}{h} \quad (0 < x \leq x_e) \tag{A.3}$$

$$g(t,x)=1 \quad (x_e < x \le x_{max}) \tag{A.4}$$

Discretising Formula (A.2), Formula (A.5) is obtained.

$$q_{3,i}^{(k+1)}(x)=\{1-g(t_i,x)\}q_{3,i-1}^{(k)}(x)+\gamma_i^{(k)}g(t_i,x)q_{3,i-1}^{(k)}(x) \tag{A.5}$$

where i indicates time t_i and upper suffix (k) means number of iterations. $\gamma_i^{(k)}$ is defined as follows and tends to unity when numerical results converge.

$$\gamma_i^{(k)}=\frac{G(t_i)}{G_m\sum_{j=1}^{m}g(t_i,x)q_{3,i-1}^{(k)}(x_j)\Delta x_j} \tag{A.6}$$

where m is division number of particle size, and Δx_j is the width of the particle size interval j. Converged solution can be obtained with iterative procedure assuming initial particle size distribution $q_{3,i}^{(0)}(x)$. Converged solution is obtained when Formula (A.7) is satisfied.

$$\sum_{j=1}^{m}\left|q_{3,i}^{(k+1)}(x_j)-q_{3,i}^{(k)}(x_j)\right|<\varepsilon \tag{A.7}$$

where ε is a small value to determine the criteria of convergence.

A.3 Example of measurement

The example data of sedimentation mass are shown in Figure A.1 and Table A.1. The detection tray with the following dimensions (see Figure 2) was used in the experiment: d_1 = 39 mm, d_2 = 70 mm, h = 80 mm, h_1 = 62 mm, h_2 = 104 mm. The accuracy of the electronic balance was ±0,01 mg. The data reduction was carried out based on the matrix method. The measurement results of the other two kinds of test particles (JIS No. 2 and 8) are also shown in Figure A.2.

— Reference to the International Standard	ISO 13317-4
— Date of the test	10/24 2011
— Unique report identification	Report number: 123
— Operator identification	Takayuki Tuyama
— Instrument type used	Liquid sedimentation mass balance method
— Sample identification and Lot Number	Barium Titanate Glass (MBP3-30), No.B0330
— Sample apparent density	4 200 kg · m^{-3}
— Sample mass and concentration	2 500 g, 0,62 wt %
— Suspending liquid	Distilled water
— Temperature	293,15 K
— Liquid density	999 kg · m^{-3}
— Liquid viscosity	1,005 mPa · s
— Dispersing agent and concentration	0,05 wt %, Sodium hexa-meta-phosphate, $(NaPO_3)_6$