INTERNATIONAL STANDARD

ISO 13318-2

Second edition
2007-09-15

Determination of particle size distribution by centrifugal liquid sedimentation methods —

Part 2: **Photocentrifuge method**

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

Partie 2: Méthode photocentrifuge

Reference number
ISO 13318-2:2007(E)

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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 13318-2 was prepared by Technical Committee ISO/TC 24, *Sieves, sieving and other sizing methods*, Subcommittee SC 4, *Sizing by methods other than sieving*.

This second edition cancels and replaces ISO 13318-2:2001, of which it constitutes a minor revision, due to the extension of Clause 4 and 5.2, and the addition of Figure 3 and the Bibliography.

ISO 13318 consists of the following parts, under the general title *Determination of particle size distribution by centrifugal liquid sedimentation methods*:

— *Part 1: General principles and guidelines*

— *Part 2: Photocentrifuge method*

— *Part 3: Centrifugal X-ray method*

Introduction

The sample suspension in a photocentrifuge may be contained in a cuvette or a disc. Sample concentration is determined by changes in a light signal monitored at a known radius. The cuvette photocentrifuge can only be run in the homogeneous mode whereas the disc photocentrifuge may be run in either the homogeneous or the line-start mode. Some systems permit the coarse end of the distribution to be measured in a gravitational mode and the fine end in the centrifugal mode. The use of light to determine particle size distribution requires a calibration factor to be applied as the particle size approaches the wavelength of the light, due to the inapplicability of the laws of geometric optics.

Determination of particle size distribution by centrifugal liquid sedimentation methods —

Part 2: Photocentrifuge method

WARNING — This part of ISO 13318 may involve hazardous materials, operations and equipment. This part of ISO 13318 does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this part of ISO 13318 to establish appropriate safety and health practices and determine the applicability of the regulatory limitations prior to its use.

1 Scope

This part of ISO 13318 covers methods for determining the particle size distribution of particulate materials by means of centrifugal sedimentation in a liquid. Solids concentrations are determined by the transmission of a light beam. The resulting signal enables conversion to a particle size distribution.

The method of determining the particle size distribution described in this part of ISO 13318 is applicable to powders that can be dispersed in liquids, powders that are present in slurry form and some emulsions. Typical particle size range for analysis is from about 0,1 µm to 5 µm. The method is applicable to powders in which all particles have the same density and comparable shapes and do not undergo chemical or physical change in the suspension liquid. It is usually necessary that the particles have a density higher than that of the liquid.

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 13318-1, *Determination of particle size distribution by centrifugal liquid sedimentation methods — Part 1: General principles and guidelines*

ISO 14887, *Sample preparation — Dispersing procedures for powders in liquids*

3 Terms, definitions and symbols

For the purposes of this document, the terms, definitions and symbols given in ISO 13318-1, and the following symbols, apply.

D optical density

E_i extinction coefficient for a particle of diameter x_i

F(surface) frequency undersize by surface

G constant dependent upon the geometry of the system, the dimensions of the light beam and on the shape of the particles

l transmission of the emergent light beam, at the time t, after the start of sedimentation

l_0 transmission of the emergent light beam when no particles are present

M distance from rotation axis to measurement zone (mm)

n_i number of particles of diameter x_i in the beam

R distance from rotation axis to centrifuge wall, inner disc radius (mm)

S distance from rotation axis to liquid/air interface of sample (mm)

x_0 diameter of the smallest particle in the light beam (µm)

x_{St} diameter of the largest particle in the light beam, i.e. the Stokes diameter (µm)

4 Principle

A stable, finely collimated beam of light passes through a spinning disc or cuvette and sedimenting sample and is detected at a known radius. Light rays, typically from either a white light source (e.g. incandescent bulb) or a monochromatic coherent source (e.g. laser), pass through the suspension and are detected by a photodiode or photomultiplier. The disc photocentrifuge can be operated in the line-start or homogeneous mode whereas the cuvette photocentrifuge can be operated only in the homogeneous mode. The signal of the light beam is monitored over the analysis time. The mass percentage of sample present in the beam is determined by calculating the ratio of the light transmission signal, by use of a clear dispersing liquid, to the light transmission signal with the sample present.

In the line-start mode the disc initially contains clear fill liquid to give maximum light transmission. Then the sample is injected as a thin layer on top of the spinning fill liquid and begins to settle outward radially. When the largest particles present reach the light beam the light transmission decreases, returning to the original transmission value when the smallest particle present passes through the beam. A buffer layer is usually injected over the fill liquid to prevent suspension breaking through the interface in a phenomenon known as “streaming”.

In an alternative configuration, the determination of the particle size distribution by centrifugal liquid sedimentation method can also be accomplished using a photocentrifuge containing a line light source and a line sensor detector system aligned with the sample cell. In this configuration, light intensity/extinction alterations during centrifugation are measured simultaneously over the whole sedimentation zone as a function of both time and of position. From these data, the particle size distribution may be calculated either from the time course of the extinction at a freely selectable position within the sample [numerical integration, ISO 13318-1:2001, Equation (11) in 4.3.3.3] or from the extinction profile along the sample at a freely selectable time (analytical integration, for details see Reference [1]).

5 Apparatus

5.1 Disc photocentrifuge, with a chamber consisting of a hollow disc with an entry port coaxial with the axis of rotation (see Figure 1). Typically this is mounted vertically, or at a small angle to the vertical, on to the shaft of an electric motor with a digitally variable speed typically between 500 r·min^{-1} and 15 000 r·min^{-1}. A white light source and detector assembly measures transmittance through the suspension as a function of time. The instrument can be used in either a line-start or homogeneous mode. Extinction coefficient corrections need to be applied for the breakdown in the laws of geometric optics for both line-start and homogeneous modes. Additionally, a correction is required for radial dilution effects when the homogeneous mode is used. Software is provided with commercial equipment to convert the data directly into size distributions in the form of tables or graphs of cumulative mass percentage versus particle size.

5.2 Cuvette photocentrifuge, in which the disc is replaced with a rectangular cell containing a homogeneous suspension (see Figure 2). Corrections need to be made for both radial dilution and light scattering effects as described in ISO 13318-1. Cuvette photocentrifuges can typically be run in both the gravitational and centrifugal modes. Additionally, some systems may offer a gradient mode permitting the centrifuge to accelerate throughout the analysis in order to reduce the measurement time.

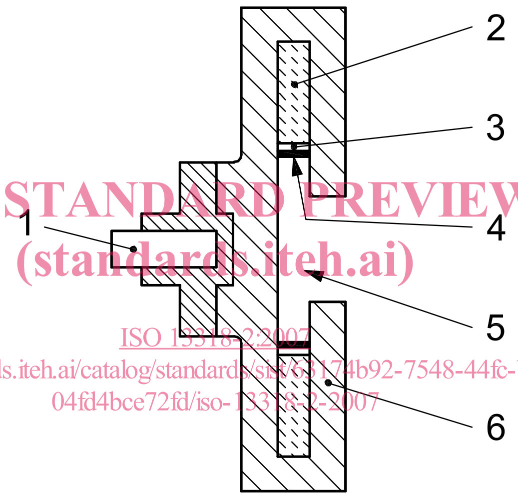
When determining particle size distribution using an apparatus containing a line light source and a line sensor detection system, transmittance is measured along the entire sedimentation zone simultaneously (see Figure 3).

5.3 Ancillary apparatus, consisting of:

— dispersing vessel, e.g. glass beaker or bottle, of appropriate dimensions;

— flexible spatula;

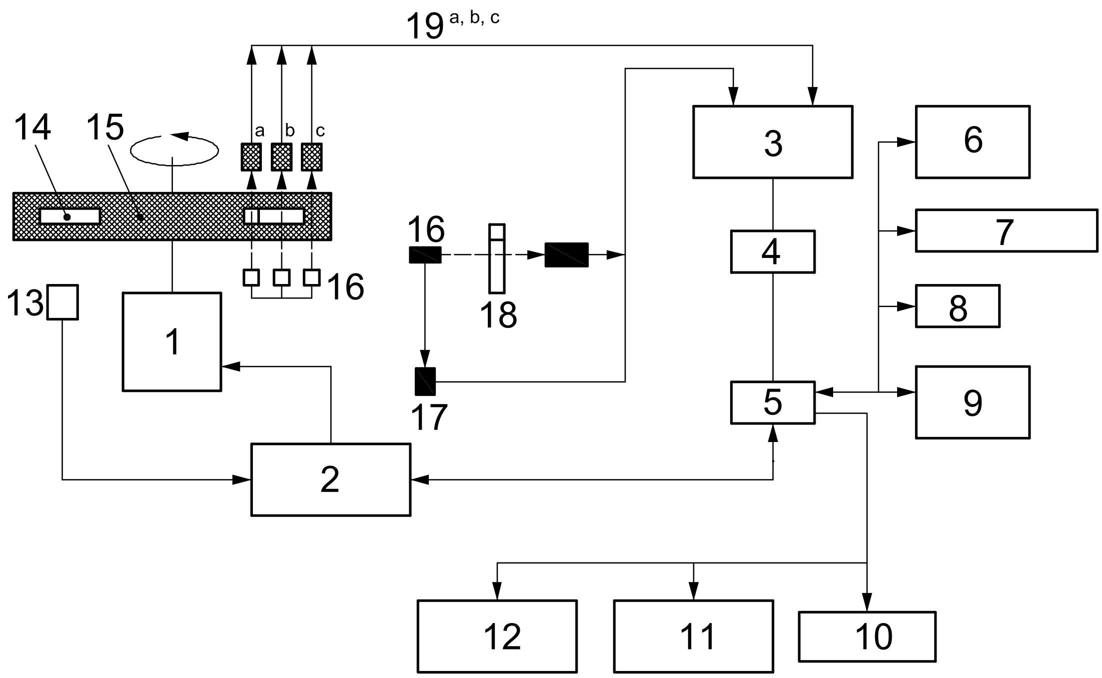
— ultrasonic bath or probe, a bottle shaker or high speed mechanical stirrer capable of rotating at 500 r·min^{-1} to 1 000 r·min^{-1}.



Key

1 motor shaft
2 spin fluid
3 buffer layer
4 suspension
5 entry port
6 polymethylmethacrylate disc

Figure 1 — Side view of the disc of a disc photocentrifuge

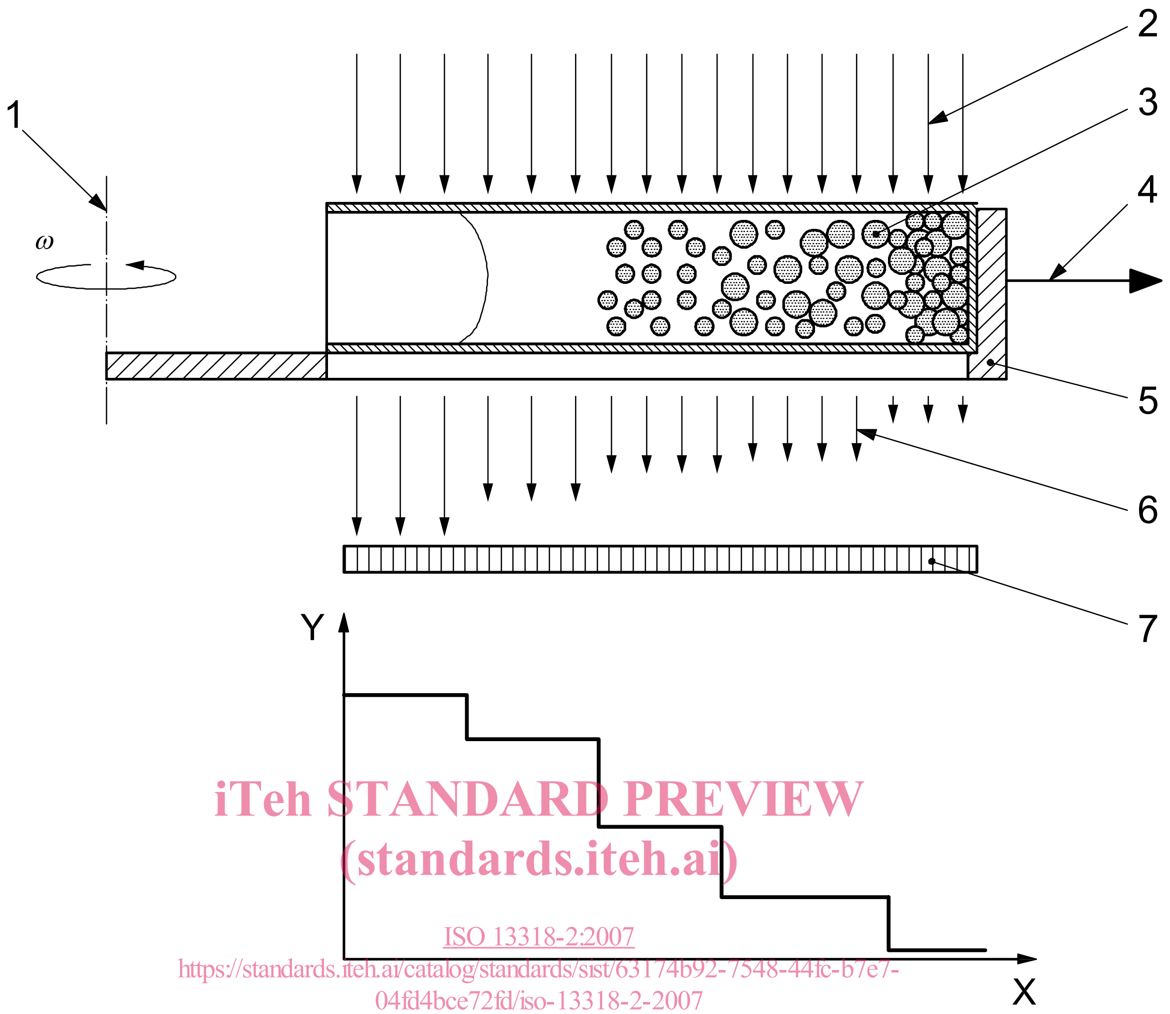


Key

1 motor
2 motor control
3 signal process
4 analogue to digital converter (ADC)
5 central processing unit (CPU)
6 date time
7 analysis parameters
8 revolutions per minute (RPM)
9 keyboard
10 printer
11 analog interface
12 computer interface
13 photosensor revolutions per minute (RPM)
14 centrifugal cell
15 rotating disc
16 light-emitting diode (LED)
17 photocell (reference)
18 photocell (sample)
19 photocells

[a] Synchro-signal (reference).
[b] Analog signal.
[c] Synchro-signal (sample).

Figure 2 — Schematic diagram of a typical cuvette photocentrifuge



Key

X position for one selected time

Y intensity

1 axis of rotation

2 incident parallel light

3 sample cell

4 centrifugal force

5 rotor

6 transmitted light

7 line detector array

Figure 3 — Schematic diagram of a cuvette photocentrifuge with line light source and line detector array