INTERNATIONAL STANDARD

ISO 13318-2

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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
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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.

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 part of ISO 13318 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*.

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*

Annexes A to C of this part of ISO 13318 are for information only.

Introduction

The sample suspension in a photocentrifuge may be contained in a cuvet or a disc. Sample concentration is determined by changes in a light signal monitored at a known radius. The cuvet 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 normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 13318. 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 13318 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 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

3.1 Terms and definitions

For the purposes of this part of ISO 13318 the terms and definitions of ISO 13318-1 apply.

3.2 Symbols

For the purposes of this this part of ISO 13318 the symbols of ISO 13318-1 and the following apply:

D optical density

E_i extinction coefficient for a particle of diameter x_i

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 cuvet 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 cuvet 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”.

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.

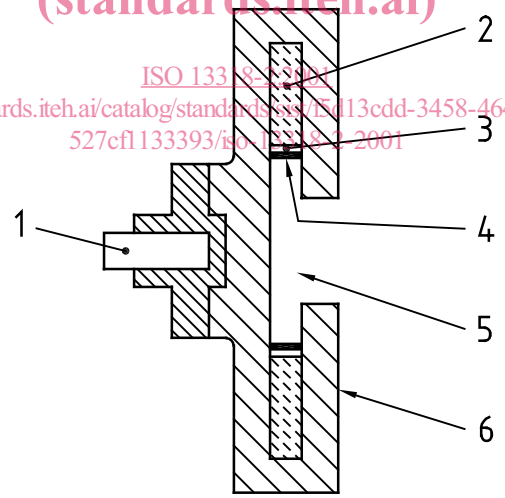
5.2 Cuvet photocentrifuge, in which the disc is replaced with a rectangular cell containing a homogeneous suspension (see Figure 2). Corrections need to be made for both radial dilution and light scattering effects as described in ISO 13318-1. Cuvet photocentrifuges can typically be run in both the gravitational 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.

5.3 Ancillary apparatus, consisting of:

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

— flexible spatula;

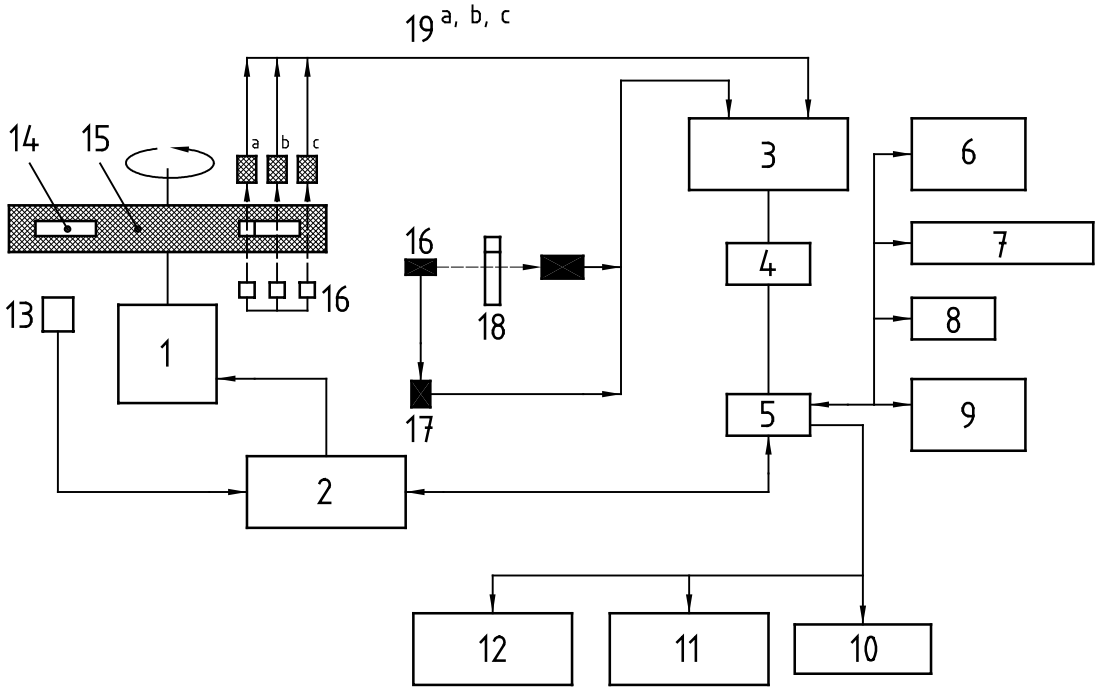
— ultrasonic bath or probe, a bottle shaker or high speed mechanical stirrer capable of rotating at 500 r·min^{-1} to 1 000 r·min^{-1}.



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 ADC
5 CPU
6 Date time
7 Analysis parameters
8 RPM
9 Key board
10 Printer
11 Analog interface
12 Computer interface
13 Photo sensor RPM
14 Centrifugal cell
15 Rotating disc
16 LED
17 Photocell (reference)
18 Photocell (sample)
19 Photocells

a Synchro-signal (reference)
b Analogue signal
c Synchro-signal (sample)

Figure 2 — Schematic diagram of a typical cuvet photocentrifuge

6 Sampling

For sampling see ISO 13318-1.

7 Preparation

7.1 Sample preparation

An analysis sample shall be prepared as described in ISO 13318-1. The volume and concentration required depends upon the volume of the centrifuge disc (or cuvet), the sensitivity of the optical-electronic system and whether the line-start (disc only) or homogeneous method is to be used. In general, lower concentrations are required than for other sedimentation methods. A concentration typically less than 0,25 % m/v and providing an attenuation preferably in the 20 % to 30 % range compared to the spin fluid without sample is required.

7.2 Temperature

The temperature of the spin fluid (line-start method) or suspension (homogeneous method) shall be determined and recorded before and after analysis in accordance with ISO 13318-1. The liquid viscosity and liquid density shall be recorded for the spin fluid or suspension at the temperature of the analysis. The temperature shall be maintained in accordance with ISO 13318-1.

7.3 Dispersion

For dispersion see ISO 13318-1 and ISO 14887.

8 Procedure

8.1 Line-start methods

8.1.1 General

The line-start method is one in which a layer of sample suspension is deposited on a spinning fill liquid i.e. all particles are considered to have started to sediment from the same initial radius. However the sample suspension has a tendency to penetrate the surface of the spin liquid as globules of suspension which then “stream” rapidly through the spin liquid. The streaming effect is due to an uneven outward diffusion of the sample suspension. On injection of the sample suspension the particles can become concentrated by surface tension effects at the interface. This leads to a region of high density above one of lower density and causes a bulk transfer of concentrated suspension into the spin fluid. Streaming is particularly prone to happen if the sample suspension is injected directly on to the spin fluid (two-layer method); consequently the two-layer method is in general replaced by the three-layer method (see 8.1.2) due to its applicability to a wider range of sample types. The following methods all attempt to cushion the hydrodynamic shock experienced by the particles as they enter the spin fluid and thus obtain a smooth transition and laminar flow.

Hydrodynamically stable sedimentation can generally be achieved by using the three-layer method, the buffered line or the external gradient method.

8.1.2 Three-layer method

Partially fill the centrifuge disc with a known volume of clear liquid (the spin fluid, typically approximately 15 ml) and allow the disc to attain the required running speed. Set up the light source at the required sampling radius (M). A steady baseline represents maximum transmission (I_0). Introduce into the disc a small volume of buffer liquid, typically 0,5 ml or 1 ml, that is of lower density than the spin fluid and determine the vortex radius (S). This can be done from a knowledge of the dimensions of the disc and the total volume of liquid introduced. Introduce the required volume of suspension (typically 0,25 ml of a 0,25 % m/v concentration) into the disc and activate the timer.