INTERNATIONAL STANDARD

ISO 13318-3

First edition
2004-07-15

Determination of particle size distribution by centrifugal liquid sedimentation methods —

Part 3:
Centrifugal X-ray method

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

Partie 3: Méthode centrifuge aux rayons X

Reference number
ISO 13318-3:2004(E)

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Published in Switzerland

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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 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 13318-3 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*

Introduction

The X-ray centrifuge monitors particle concentration changes at a fixed or variable radius. In some configurations, the instrument can also be used in a gravitational mode (see ISO 13317-1) and those data blended with other data determined in the centrifugal mode, thus extending the typical upper size limit above 5 µm.

Determination of particle size distribution by centrifugal liquid sedimentation methods —

Part 3: Centrifugal X-ray 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 standard 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 describes a method for determining the particle size distribution of homogeneous particulate material using centrifugal sedimentation in a liquid. Solids concentrations are determined by the attenuation of an X-ray beam. The resulting signal enables conversion to a particle size distribution.

The method of determining the particle size distribution described in this standard is applicable to powders which can be dispersed in liquids or powders which are present in slurry form. The typical particle size range for analysis is from 0,1 µm to 5 µm. The method is applicable to powders in which all particles have the same effective density, chemical composition and comparable shapes. Materials possessing elements with an atomic number greater than about 12 can be expected to produce adequate X-ray opacity. Particles should not undergo chemical or physical change in the suspension liquid. It is necessary that the particles have a higher density 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*

3 Terms and definitions

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

4 Symbols

For the purpose of this document, the symbols of ISO 13318-1 and the following apply.

B	function of the atomic number of the sample elements in the beam
C	concentration of sample in the beam
I_0	transmission of the emergent X-ray beam through the suspending fluid with no particles present
I	transmission of the emergent X-ray beam through suspension at radius M and time t

D X-ray density [$\log_{10}(I_0/I)$]

x_{St} diameter of the largest particle in the X-ray beam, i.e. the Stokes diameter, expressed in micrometres

M distance, expressed in centimetres, from rotation axis to measurement zone

S distance, expressed in centimetres, from rotation axis to liquid-air interface of sample

5 Sampling

For information regarding sampling, see ISO 13318-1.

6 Principle

A stable, finely collimated beam of X-rays passes through a suspension containing the sample particles and is detected at a known radius. The centrifuge disc containing the sample is of known dimensions and a known amount of suspension is used so that the surface radius for the suspension can be calculated. The measured settling radius can be reduced during the analysis for the purpose of obtaining a more rapid analysis than would be possible if the radius were fixed. The mass percentage of sample present at a given time, and at a known measurement radius, is determined by calculating the ratio of

— the X-ray transmission through a clear dispersing liquid, and

▪ the signal with the sample present.

The Stokes diameter (x_{St}) at measurement radius, M, and time, t, is given by the following equation:

$$x_{St} = \sqrt{\frac{18\eta \ln \frac{M}{S}}{(\rho_s - \rho_1)\omega^2 t}} \quad (1)$$

The transmission, I, of the emergent X-ray beam having passed through the suspension is proportional to the mass of powder in the beam.

$$I = I_0 \exp\left[-BC(r,t)\right] \quad (2)$$

where $C(r,t)$ is the concentration of the sample in the beam at radius, r, and time, t.

The X-ray density, D, at measurement radius, r, and time, t, is given by the following equation:

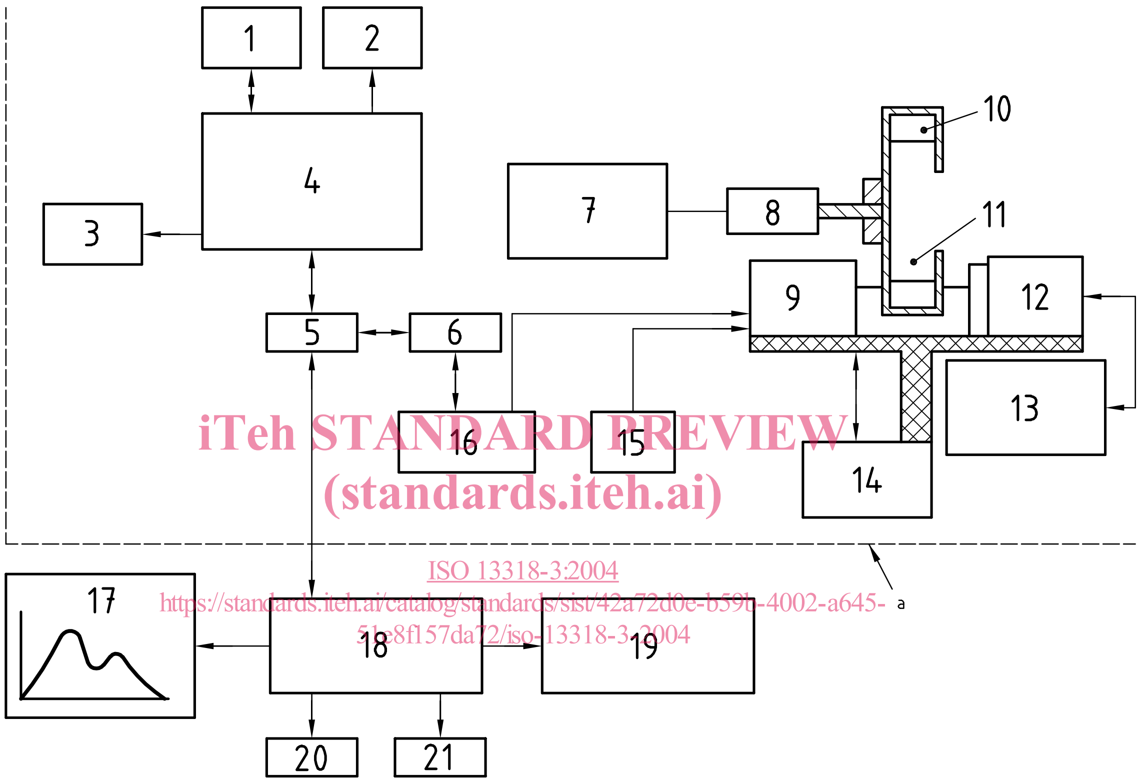
$$D = -BC(M,t)\log_{10} e = \log_{10}(I_0/I) \quad (3)$$

The X-ray density of the emergent beam, after correction for radial dilution effects, is proportional to the mass of sample in the beam.

7 Apparatus

7.1 Basic apparatus

The instrument (Figure 1) consists typically of a shallow bowl-shaped X-ray-transparent disc of known internal radius and depth. The bowl is mounted vertically on the shaft of an electric motor with a variable speed, typically between 500 r·min^{-1} and 15 000 r·min^{-1}. An X-ray source and detector assembly, which may also scan radially, measures transmission through the suspension as a function of time and radial position. Software provides for the conversion of data directly into frequency distributions in the form of tables or graphs of cumulative mass percentage versus particle size.



Key

1 arm display
2 signal display
3 rotational motion (r·min^{-1})
4 instrument keyboard and display
5 central processing unit
6 analog digital converter
7 motor control and power supply
8 motor
9 detector
10 suspension
11 cell
12 X-ray source
13 power supply and beam control
14 scanning system
15 power supply
16 signal cond.
17 colour monitor
18 computer data system program
19 data analysis, archiving comparison
20 plotter
21 printer

[a] Parameter and data transfer.

Figure 1 — Line diagram of an X-ray scanning disc centrifuge