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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
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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:

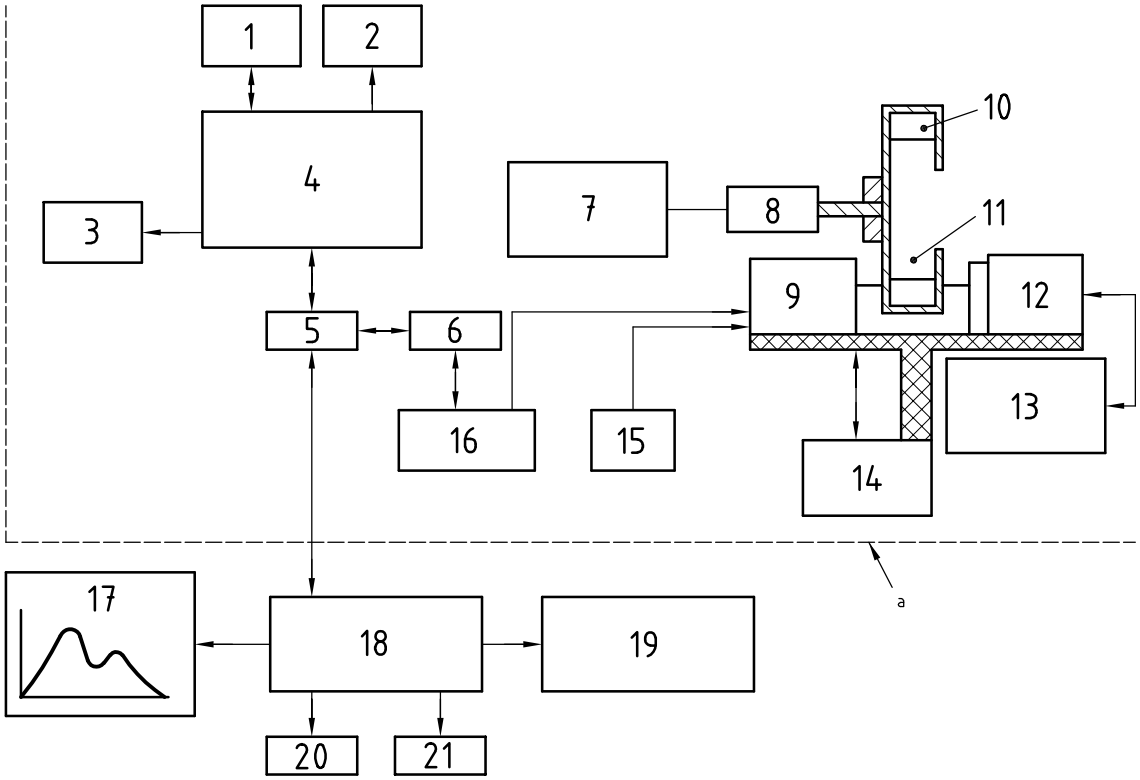
$$D = -BC(M,t)\log_{10} e = \log_{10}(I_0/I) \quad (3)$$

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 $r \cdot min^{-1}$ and 15 000 $r \cdot min^{-1}$. 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	8	motor	15	power supply
2	signal display	9	detector	16	signal cond.
3	rotational motion ($r \cdot min^{-1}$)	10	suspension	17	colour monitor
4	instrument keyboard and display	11	cell	18	computer data system program
5	central processing unit	12	X-ray source	19	data analysis, archiving comparison
6	analog digital converter	13	power supply and beam control	20	plotter
7	motor control and power supply	14	scanning system	21	printer

a Parameter and data transfer.

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

7.2 Ancillary apparatus

The ancillary apparatus consists of

— dispersing vessel, of appropriate dimensions,

— flexible spatula, and

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

8 Preparation

8.1 Sample preparation

Prepare an analytical sample as described in ISO 13318-1.

Prepare an initial sample for analysis having a homogeneous concentration of about 0,2 % by volume. If this is not sufficient to give an adequate signal, it may be necessary to increase the concentration.

Carry out duplicate tests at higher concentrations in order to determine whether delayed settling is occurring. If the second test gives a significantly higher percentage of small particles, then discard these data.

8.2 Temperature

Measure and record the temperature of the suspension before and after analysis, in accordance with ISO 13318-1. Record the liquid viscosity and liquid density for the spin fluid or suspension at the temperature of the analysis. Maintain the temperature in accordance with ISO 13318-1.

8.3 Dispersion

For information regarding the dispersion, see ISO 13318-1.

9 Measurement procedure

Switch the instrument on 20 min before use in order to obtain stable operating signal. Determine the attenuation of the emergent X-ray beam with only the suspension liquid in the disc. To this end, inject sufficient clear suspension liquid into the disc to ensure that the X-ray beam path is below the liquid surface in order to provide the maximum X-ray transmission reading. The suspension liquid is then removed. For more precise results, a base-line scan of the entire measuring portion of the disc may be determined for subsequent correction of the corresponding suspension data.

Set the run conditions prior to analysis by keying into the computer the operating variables such as effective particle density, liquid density and liquid viscosity. Then switch the centrifuge on to the desired speed and feed the suspension into the centrifuge disc without delay and activate the timer.

10 Tests in duplicate and validation

10.1 Tests in duplicate

For information regarding duplicate tests, see ISO 13318-1.

10.2 Validation

For information regarding validation, see ISO 13318-1.

11 Calculation of results

11.1 Calculation of particle size

Calculate the Stokes diameters in accordance with ISO 13318-1.

11.2 Calculation of cumulative mass percentage

In the centrifugal X-ray method, the attenuation of the X-ray beam is directly proportional to the mass of particles in the path of the beam through the suspension and the result is presented automatically.

12 Test report

The report shall conform with the requirements in accordance with ISO 13318-1. Results shall typically be presented as a plot of Stokes diameter versus cumulative distribution by mass reported to the nearest 0,1 %. On graphic presentations of the plot, the diameters are placed on the abscissa and the cumulative mass percentage on the ordinate.

The report shall include

— reference to this part of ISO 13318,

— name of the testing establishment,

— date of the test,

— operator identification,

— instrument type used,

— mode of operation (e.g. fixed radius or scanning),

— test sample identification,

— sample density (and mass, if available),

— suspending liquid, its temperature, density, viscosity and, if applicable, its volume,

— dispersing agent and concentration of agent used,

— method of dispersion of the suspension, including dispersion time,

— centrifuge speed,

— any other operations not specified in this part of ISO 13318 and which might have an influence on the results.

The following instrument characteristic, typically determined by the manufacturer and pre-programmed into the equipment software, may be optionally reported if available:

— Measurement radius, M (not fixed in scanning mode)

Annex A
(informative)

Worked example

A.1 General

Experimental data are presented in Table A.1. Supplementary data include the following.

The measurement radius, M, at the start of the analysis is 4,88 cm and the surface radius, S, is 4,41 cm. After 1 min, the source and detector scan towards the surface at constant speed and M decreases as t increases such that after 7 min scanning the X-ray beam is 0,2 cm below the surface.

The opacity to X-ray is determined by the count level of a scintillation counter and converted into a percentage to give the measured concentration. The example data presented here represent smoothed data taken every 15 s. In an actual analysis, the data may be taken more frequently and data smoothing may be performed automatically in some instrument configurations.

Using Equation (1), the Stokes diameter, x_1, in micrometres, at 15 s is given by

$$x_1 = \sqrt{\frac{18 \times 8{,}90 \times 10^{-4} \ln\left(\frac{4{,}88}{4{,}41}\right)}{(2\,500 - 1\,000)\,15(50\pi)^2}} = 1{,}71 \qquad \text{(A.1)}$$

At 1 min, the Stokes diameter is 0,855 µm. Scanning commences after 1 min and causes the measurement radius to decrease with increasing time. Subsequent diameters, expressed in micrometres, are, therefore, determined using the Stokes equation in the following form:

$$x_i = 20{,}80 \sqrt{\frac{\ln\left(\frac{M_\mathrm{i}}{4{,}41}\right)}{t_\mathrm{i}}} \qquad \text{(A.2)}$$

A.2 Example

ISO reference	ISO 13318-3
Testing establishment	MAL
Date	2000-11-15
Report	Quartz 123
Operator	DM
Instrument	Model X234
Operation mode	Scanning
Sample identification	Quartz A157
Material	Quartz

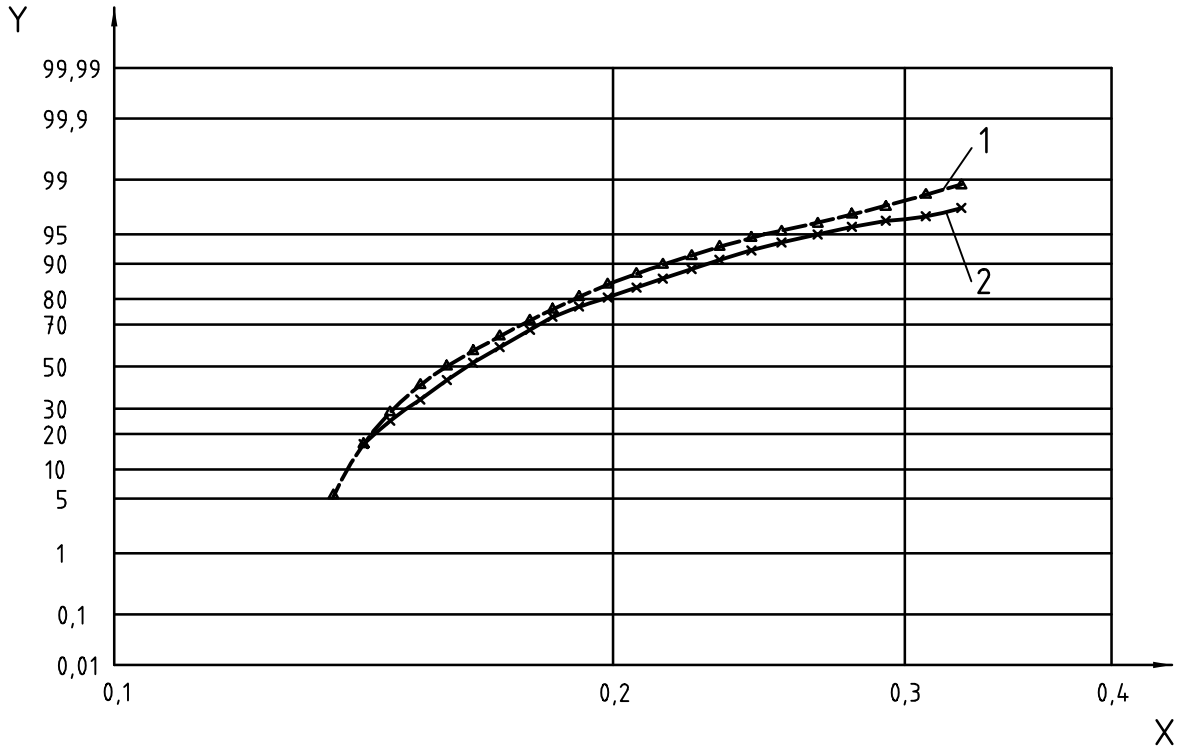
Sample density	2 500 kg·m^{-3}
Mass of powder	0,112 g
Spin liquid	Distilled water
Temperature	298,15 K
Liquid density	1 000 kg·m^{-3}
Liquid viscosity	0,890 4 mPa·s
Volume of suspension:	20 ml
Dispersant	Sodium polymetaphosphate, 0,1 % (volume fraction) in distilled water (1 g/l)
Preparation	1 min ultrasonic probe in water-cooled jacketed container
Centrifuge speed	1 500 r·min^{-1}
Powder pre-treatment	Oven dried, 1 h at 383,15 K

NOTE The signal count with water in the disc gives I_0 = 56 000 and the minimum count with sample, I = 36 744.

Table A.1 — Conversion of measured X-ray sedimentation data into cumulative mass percentage

Time t_i s	Radius M_i cm	Size x_i µm	X-ray density D_i	Concentration $C(M_i, t_i)$	Mass under Q_i
Maximum			0,183		
165	4,81	0,48	0,179	97,6	98,8
180	4,80	0,45	0,177	96,8	97,9
195	4,79	0,43	0,177	96,4	97,6
210	4,78	0,41	0,176	95,9	97,3
225	4,77	0,39	0,173	94,6	96,3
240	4,76	0,37	0,171	93,6	95,5
255	4,75	0,35	0,169	92,2	94,5
270	4,74	0,34	0,166	90,4	93,1
285	4,73	0,33	0,162	88,3	90,9
300	4,72	0,31	0,157	85,9	89,0
315	4,71	0,30	0,153	83,5	86,9
330	4,70	0,29	0,148	80,6	84,3
345	4,69	0,28	0,141	76,9	80,9
360	4,68	0,27	0,133	72,4	76,6
375	4,67	0,26	0,123	66,9	71,2
390	4,66	0,25	0,110	60,2	64,4
405	4,65	0,24	0,095	51,7	55,5
420	4,64	0,23	0,078	42,8	46,5
435	4,63	0,22	0,063	34,3	37,8
450	4,62	0,21	0,046	25,0	27,2
465	4,61	0,20	0,029	15,7	17,1
480	4,60	0,19	0,010	5,7	6,1

NOTE The data in Table A.1 giving the cumulative mass percentage versus Stokes diameter are graphed in Figure A.1.



Key

1 concentration
2 mass, %
X particle size, µm
Y percent

Figure A.1 — Plot of measured concentration and derived mass percentage undersize, for a centrifuge operating in a scanning mode

ICS 19.120

Price based on 9 pages