BRITISH STANDARD

BS ISO 13317-3:2001

Determination of particle size distribution by gravitational liquid sedimentation methods —

Part 3: X-ray gravitational technique

ICS 19.120

National foreword

This British Standard reproduces verbatim ISO 13317-3:2001 and implements it as the UK national standard. Together with BS ISO 13317-1:2001 and BS ISO 13317-2:2001 it supersedes BS 3406-2:1984, which is withdrawn.

The UK participation in its preparation was entrusted by Technical Committee LBI/37, Sieves, screens and particle sizing, to Subcommittee LBI/37/4, Sizing by methods other than sieving, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this subcommittee can be obtained on request to its secretary.

Cross-references

The British Standards which implement international publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled “International Standards Correspondence Index”, or by using the “Find” facility of the BSI Standards Electronic Catalogue.

This British Standard, having been prepared under the direction of the Sector Committee for Materials and Chemicals, was published under the authority of the Standards Committee and comes into effect on 15 July 2001

Summary of pages

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INTERNATIONAL STANDARD

ISO 13317-3

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Determination of particle size distribution by gravitational liquid sedimentation methods —

Part 3: X-ray gravitational technique

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

Partie 3: Méthode aux rayons X par gravité

Reference number
ISO 13317-3:2001(E)

Contents

Annex

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.

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 13317 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 13317-3 was prepared by Technical Committee ISO/TC 24, *Sieves, sieving and other sizing methods*, Subcommittee SC 4, *Sizing by methods other than sieving*.

ISO 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*

Annex A of this part of ISO 13317 is for information only.

Determination of particle size distribution by gravitational liquid sedimentation methods —

Part 3:
X-ray gravitational technique

SAFETY PRECAUTIONS — This part of ISO 13317 does not purport to address all of the safety considerations associated with its use. It is the responsibility of the user of this part of ISO 13317 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 13317 describes a method for the determination of the particle size distribution of a powder dispersed in a liquid using gravity sedimentation. The measurement of the concentration of solids settling in a liquid suspension is achieved by monitoring the incremental signal absorption from a beam of X-rays.

The method of determining the particle size distribution described in this part of ISO 13317 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 about 0,5 µm to about 100 µm. The method is used for materials containing particles of the same chemical composition which produce adequate X-ray opacity.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 13317. 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 13317 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 787-10, *General methods of test for pigments and extenders — Part 10: Determination of density — Pyknometer method*.

ISO 8213, *Chemical products for industrial use — Sampling techniques — Solid chemical products in the form of particles varying from powders to coarse lumps*.

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*.

3 Terms and definitions

For the purposes of this part of ISO 13317, the terms and definitions given in ISO 13317-1 apply.

4 Symbols

For the purpose of this part of ISO 13317, the symbols given in ISO 13317-1 and the following apply.

x_{St}	Stokes equivalent spherical diameter (m) (practical unit: micrometres, μm)
η	Suspending liquid viscosity (Pa·s), (practical unit: mPa·s)
h	Sedimentation height (m)
ρ_{s}	Sample density [effective particle density] (kg·m^{-3})
ρ_{l}	Liquid density (kg·m^{-3})
g	Acceleration due to gravity (9,807 m·s^{-2})
t	Sedimentation time, (seconds, s)
B	Function of atomic number of sample elements in beam
C	Concentration of sample in beam
I_0	Attenuation of emergent X-ray beam passing through the suspending fluid
I	Attenuation of emergent X-ray beam through suspension at settling height h, at time t
D	X-ray density [$\lg_{10} (I_0/I)$]

5 Sampling

The sampling procedure according to ISO 13317-1 shall be followed.

6 Representation of results

The results shall be represented according to ISO 9276-1.

7 Principle

The concentration of dispersed sample is measured by attenuation of an X-ray beam. A stable, narrow, collimated beam of X-rays passes through a suspension of the sample: it is detected at a known height from the top of the sample cell. The sample cell is completely filled with sample suspension for the duration of the analysis. The settling height, h, at which the particle concentration, C, is determined, may be reduced during the analysis for the purpose of obtaining a more rapid analysis than would be possible if all measurements were made at the same value of, h. The cumulative mass percent of sample present at a given sedimentation height is continuously determined. The X-ray signal attenuation at the known height is compared to the attenuation with suspending liquid and also to the attenuation with the homogeneously dispersed sample present in the liquid. The attenuation of the emergent X-ray beam is proportional to the mass of powder in the beam, and is expressed by the following formula:

$$I = I_0 \exp(-BC) \tag{1}$$

or

$$C = (-1/B) \ln(I/I_0) \tag{2}$$

The X-ray density, D, is expressed as follows:

$$D = -BC\,\mathrm{lg}\,\mathrm{e} \tag{3}$$

also

$$D = \mathrm{lg}\left(I_0/I\right) \tag{4}$$

thus

$$D = -BC\,\mathrm{lg}\,\mathrm{e} = \mathrm{lg}\left(I_0/I\right) \tag{5}$$

The Stokes diameter x_{St}, corresponding to the X-ray density at settling height h and time t, is given by:

$$x_{\mathrm{st}} = \left[\frac{18\eta h}{\left(\rho_{\mathrm{s}} - \rho_{\mathrm{l}}\right) g t}\right]^{1/2} \tag{6}$$

The X-ray density D is proportional to the concentration C, and thus mass, of sample in the beam. A plot of the X-ray density D, taken as a function of time t and h, versus

$$\sqrt{18\eta h/\left(\rho_{\mathrm{s}} - \rho_{\mathrm{l}}\right) g t}$$

provides the cumulative mass distribution versus equivalent spherical diameter.

8 Apparatus

8.1 Main apparatus

The main apparatus (see Figure 1) typically consists of a temperature-controlled analysis compartment and mixing chamber; a plumbing system for circulation of suspending liquid or sample suspension; an X-ray source/detector system and a control module for apparatus control, data acquisition and reduction. The sedimentation cell within the temperature-regulated analysis compartment may be repositioned throughout the analysis relative to the signal source and detector to reduce analysis time. Alternative designs, such as one in which the X-ray source and detector move while the sedimentation cell remains stationary, may also be used.

The use of a magnetic stirrer should be avoided if magnetically susceptible particles are to be tested. The dispersion may then be maintained by means of a mechanical stirrer.

8.2 Ancillary apparatus

Ultrasonic bath, probe or high-speed mechanical stirrer capable of 500 revolutions to 1 000 revolutions per minute.

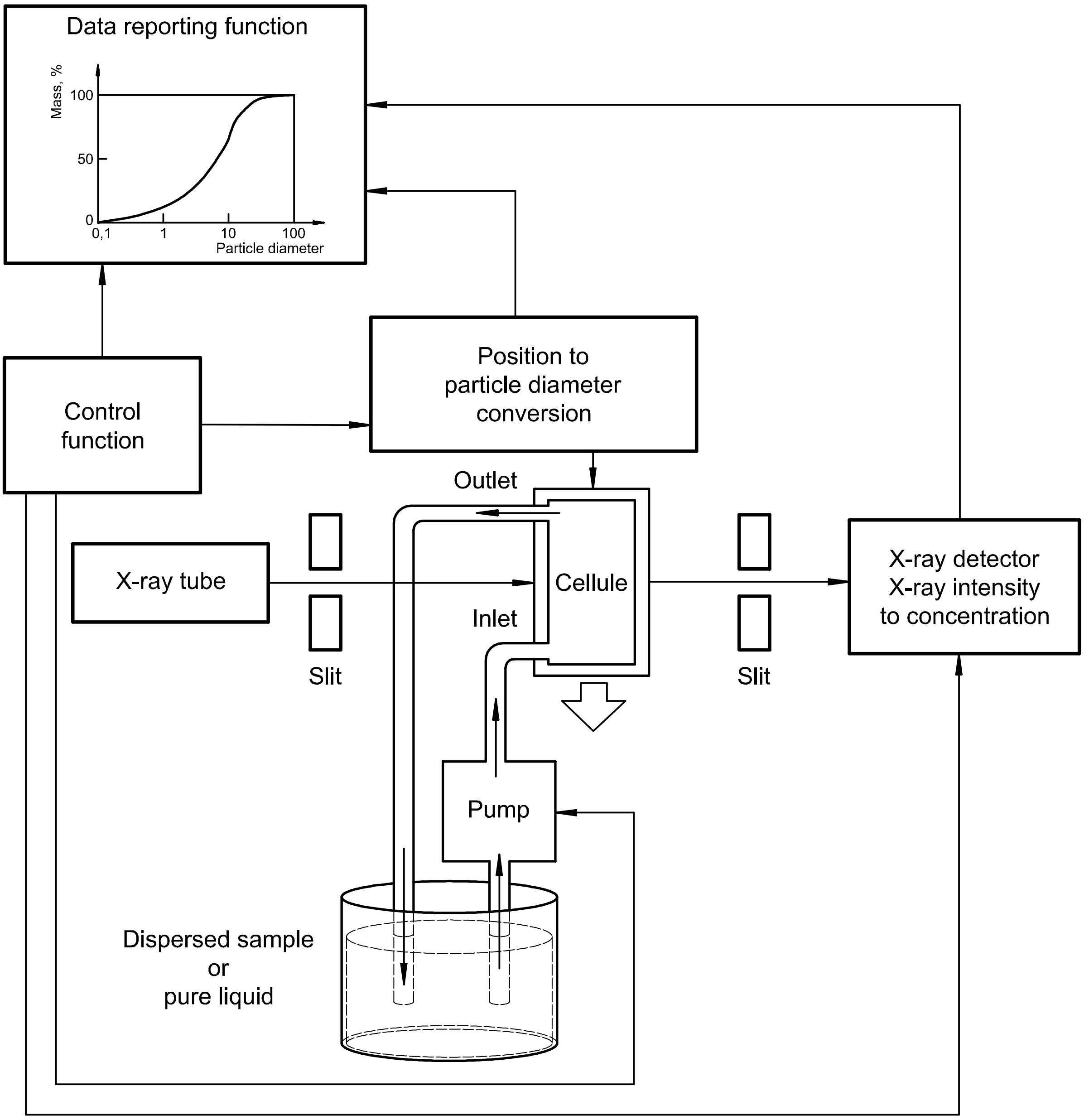

Figure 1 — Schematic of typical apparatus using the X-ray gravitational technique

9 Sample preparation

A representative sample for analysis shall be taken according to ISO 8213. It shall be dispersed according to ISO 14887 in a suspending medium of adequate viscosity and X-ray transparency. The use of dispersing agents and ultrasonics or mechanical stirring to aid dispersion will be recorded. Typically 50 ml of dispersed sample is required for the analysis. The sample concentration will be prepared in accordance with the manufacturers' instructions. Higher sample concentrations may be required with powders having a low X-ray absorptivity coefficient. Knowledge of the exact sample concentration is not required, but the influence of concentration should be checked. The minimum concentration of suspension compatible with the measuring method is preferred (see ISO 13317-1). The required sample concentration will typically produce a reduction in the beam signal of 10 % to 30 % relative to the signal observed with the suspending liquid. The sample may be dispersed in either an aqueous or an organic medium. Any liquid compatible with sample cell materials and having low absorptivity for X-rays may be used. Typical liquids are water, glycols, kerosene, mineral oils, alcohols and mineral spirits. It is recommended that the viscosity of the suspending medium should have a value such that the largest particle to be measured has a Reynolds number of $\leqslant 0{,}25$ (see ISO 13317-1). Any temperature change of the suspension should be minimized during a measurement (see 10.3).

The viscosity and density of the suspending liquid at the temperature of the analysis will be reported. The effective particle density of the sample is also reported. The suspending liquid, including dispersants at the analysis concentration, may be used as the displacement fluid to experimentally determine the effective particle density.

10 Procedure

10.1 General

See ISO 13317-1 for guidance on restrictions of upper size limit and lower size limit, and on test conditions. The validity of the measurement will be dependent on the Stokes equation applying to creeping flow for the suspension under study. The lower size limitation will in part be a function of the ability of the system to minimize thermal convection and mechanical effects. Additionally, for the fine size fraction, Brownian motion effects may contribute to the vertical spread of particles of the same size originating from the same initial settling height.

10.2 Base-line determination

Determine the attenuation of the X-ray signal with only suspending liquid in the sample cell (0 % solids). Where possible, a base-line scan of the entire measuring portion of the cell is advised for subsequent correction of the corresponding sample suspension data. The base-line scan of the entire measuring portion of the cell is recommended as this will provide optimum correction for variability in the cell window thickness, or non-parallel alignment of the cell windows.

10.3 Temperature equilibration

Place the dispersed sample suspension in the mixing chamber, and circulate the suspension through the sample cell for typically 60 s to 90 s. Confirmation is obtained that the analysis compartment is operating within the specified temperature band, preferably within ± 1 K of the temperature set point.

10.4 Bubble elimination

Check for the presence of air bubbles in the sample cell either manually or automatically. If bubbles are detected, remove them by manual or automatic operation. If air bubbles are detected, make a further check prior to commencing the analysis. Repeat the bubble detection and elimination steps until confirmation is obtained that no bubbles are detected.

10.5 Concentration setting

Whilst the suspension continues to circulate through the sample cell, determine the 100 % initial sample concentration setting, according to the manufacturers' instructions. Depending on the apparatus, this will typically require either a manual setting of the 100 % point on the sample-recording graph paper or the automatic detection and storage of the signal for subsequent computation. A scan of the entire measuring portion of the cell for the 100 % concentration setting may be performed; this will provide optimum correction for variability in cell window thickness, or non-parallel alignment of the windows, when applied in combination with a similar base-line scan.

10.6 Measurement

Initiate the analysis by cessation of pumping of the dispersed sample through the sample cell. Elapsed sedimentation times and the corresponding sample concentrations, at known sedimentation heights, are automatically recorded. The cumulative signal obtained as the analysis progresses permits termination of the analysis prior to achieving a base-line corresponding to a zero concentration of detected sample. Registration of the complete cumulative size distribution is preferred for the better detection of anomalies. However, for routine procedures, termination of the measurement prior to reaching the base-line is permitted. The requirements for an abbreviated analysis will depend on the sample diameters of interest to the operator. When the measurement is completed, a repeat analysis may be performed, following adequate redispersion of the sample sediment in the sample cell.

10.7 Cleaning of instruments

Thoroughly rinse the sample vessel, measurement cell and associated tubing with the suspending liquid following the final repeat analysis. Run a new base-line scan to determine if any sample remains from the previous analysis.

11 Tests in duplicate, validation and verification

11.1 General

The X-ray gravitational technique is based on first principles and does not require calibration by the operator. Correct instrument operation may however be confirmed by a validation procedure.

11.2 Tests in duplicate

Carry out tests in duplicate on representative analysis samples taken from the same laboratory sample. The results for duplicate analyses should normally differ by less than 2 % for the proportions of mass at the same Stokes diameter. This figure would be expected to hold for the Certified Reference Materials referred to in 11.3. Note that a wider tolerance may be required for other sample types with more narrow distributions.

11.3 Validation

The total measurement procedure should be considered, including sampling, sample dispersion, sample transport through the measuring zone, and the measurement itself. It is essential that the total operational procedure be adequately described in detail. Primary validation can be made with acceptable reference materials certified by a standard gravity sedimentation technique. Suitable materials would include, for example, Certified Reference Materials from the Bureau Centre of Reference, Brussels, Nos 66, 67, 69 and 70 (all quartz), or U.S. National Institute of Science and Technology (NIST), Gaithersburg MD, No. SRM 1978 (zirconium oxide). The response of a sedimentation instrument is considered to meet this part of ISO 13317 if the mean values of x_{10}, $x_{50}$and x_{90}, coming from at least three independent measurements, are within the range of the certified values i.e. the mean values together with their standard deviation. Mixtures by mass of two or more certified reference materials, having the same properties, can be applied to test the accuracy of the reported fractional quantities, the size resolution and the sensitivity for fines or coarse material. Representative sampling is of particular concern in the case of a test of mixed reference materials where fractional quantities can be very small.

The precise position of the sample cell relative to the X-ray beam is critical for an accurate measurement. Where possible, the correct positioning at the start of the analysis is confirmed by monitoring the height from the initial analysis position to that point at which intersection of the finely collimated X-ray beam occurs with the top inner edge of the metal cell (hence referred to as an “X-ray beam-split test”). Where applicable, an X-ray beam-split test may be performed as part of each validation test measurement, to corroborate precise positioning of the movable parts of the measurement system. This test verifies or establishes the travel of the moving parts from their initial position, for a given number of repositioning steps.

11.4 Verification

The checking at regular intervals of both operator procedure and instrument performance is essential to verify the test results. The frequency of checking is a matter for each laboratory to determine. Instrument drift is best monitored using control charting.

Whilst the X-ray gravitational technique does not require calibration, it is recommended that an X-ray beam-split test, when applicable, be performed regularly to corroborate precise positioning of the movable parts of the measurement system. Where practical, it is recommended that the beam-split test be incorporated as part of each test measurement. This test verifies or establishes the travel of the moving parts from their initial position, for a given number of repositioning steps.

It is recommended that both the operator procedure and instrument performance be verified by the regular use of Certified Reference Materials. Product samples of typical composition and particle size distribution for a specified class of products may be used, provided that their particle size distribution has been proven to be stable over time. The results should comply with previously determined data with the same precision and bias as those for the Certified Reference Materials; otherwise a re-run of Certified Reference Materials is indicated to verify instrument performance.

12 Calculation of results

12.1 Calculation of particle size

Use equation (6) to calculate the equivalent Stokes diameter for the height and time at which each measurement of X-ray density is recorded. Present the result as percentage oversize or undersize.

12.2 Calculation of cumulative distribution by mass

Use equation (4) to compute X-ray density, which represents the cumulative distribution by mass below the equivalent Stokes diameter corresponding to the time and height of the measurement.

13 Test report

Results are typically tabulated as the cumulative percentage by mass (reported to the nearest 0,1 %) at a near-logarithmic series of diameters or plotted as cumulative mass percent versus diameter (on an exponential logarithmic scale). In the case of a plot, the diameters will be placed on the abscissa and the cumulative mass on the ordinate. The representation of results shall conform to ISO 9276-1.

The test report shall indicate:

- reference to this part of ISO 13317, i.e. ISO 13317-3;
- name of the testing establishment;
- date of the test;
- unique report identification;
- reference to any other appropriate International Standard;

- operator identification;
- instrument type used;
- test sample identification;
- sample density;
- suspending liquid;
- density of suspending liquid at the analysis temperature;
- viscosity of suspending liquid at the analysis temperature;
- temperature of the suspension during analysis;
- dispersing agent and concentration of agent used;
- method of dispersion, including dispersing time;
- any other operations not specified in this part of ISO 13317.

Annex A
(informative)

Example of typical report

Testing establishment: MAL Brussels
Date: 1998-11-21
Report identification: MAL 123
International Standard reference : ISO 13317-3
Operator: RM
Instrument type: Model XXXX
Sample identification: Quartz (BCR No. 70)
Sample density: 2 642 kg·m^{-3}
Suspending liquid: De-ionized water.
Liquid density: 994 kg·m^{-3}
Liquid viscosity: 0,727 mPa·s
Analysis temperature: 307,85 K
Dispersing agent: 0,1 % weight per volume, tetrasodium pyrophosphate decahydrate
Dispersion method: Ultrasonic probe, 1 min

Table A.1 — Particle size distribution of quartz (BCR No. 70)

Diameter µm	**Cumulative mass distribution** %
50,0	99,6
40,0	99,1
30,0	99,2
25,0	99,2
20,0	99,0
15,0	98,4
10,0	95,5
8,0	92,7
6,0	86,5
5,0	80,4
4,0	70,3
3,0	53,8
2,0	29,0
1,5	14,9
1,0	3,9
0,8	1,2
Mass median diameter: 2,8 µm Mass modal diameter; 2,7 µm	

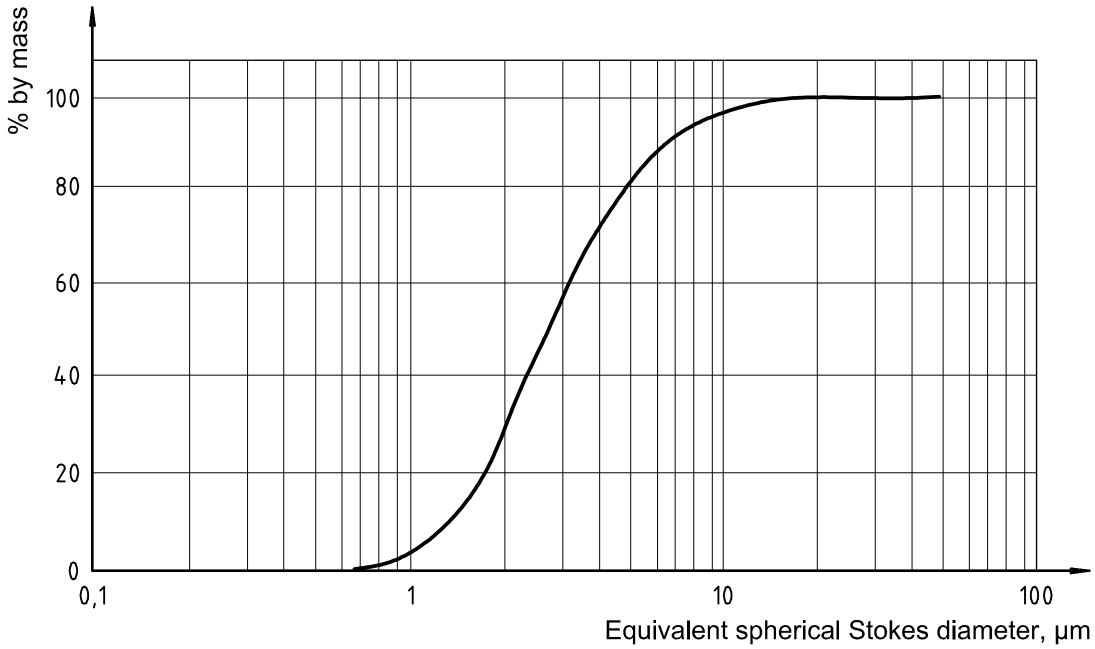

Figure A.1 — Particle size distribution of quartz (BCR No. 70)

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