INTERNATIONAL STANDARD

ISO 13322-2

Second edition 2021-12

Particle size analysis — Image analysis methods —

Part 2: **Dynamic image analysis methods**

Analyse granulométrique — Méthodes par analyse d'images — Partie 2: Méthodes par analyse d'images dynamiques

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 24, *Particle characterization including sieving*, Subcommittee SC 4, *Particle characterization*.

This second edition cancels and replaces the first edition (ISO 13322-2:2006), which has been technically revised.

The main changes compared to the previous edition are as follows:

- the text has been aligned with changes introduced in ISO 13322-1:2014;
- clauses on instrumentation (principle) and operational procedures have been significantly expanded;
- a new clause on accuracy and instrument qualification using particulate reference materials has been added.

A list of all parts in the ISO 13322 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The ISO 13322 series is applicable to the analysis of images for the purpose of determining particle size distributions. The purpose of this document is to provide guidance for measuring and describing particle size distribution, using image analysis methods where particles are in motion. This entails using techniques for dispersing particles in liquid or gas, taking in-focus, still images of them while the particles are moving and subsequently analysing the images. This methodology is called dynamic image analysis.

There are several image capture methods. Some typical methods are described in this document.

ISO 13322-1 on static image analysis methods assumes that an adequate image has already been captured and concentrates upon the analysis of these images.

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Particle size analysis — Image analysis methods —

Part 2:

Dynamic image analysis methods

1 Scope

This document describes a method to transfer the images from particles having relative motion to binary images within practical systems, in which the particles in the images are individually separated. Images of moving particles are created by an optical image capture device. Effects of particle movement on the images are either minimized by the instrumentation or corrected by software procedures. This method is applicable to the particle images that are clearly distinguishable from static background. Further processing of the binary image, which is then considered as static, is described in ISO 13322-1. A dynamic image analysis system is capable of measuring a higher number of particles compared to static image analysis systems. This document provides guidance on instrument qualification for particle size distribution measurements by using particulate reference materials. This document addresses the relative movement of the particles with respect to each other, the effect of particle movement on the image (motion blur), the movement and position along the optical axis (depth of field), and the orientation of the particles with respect to the camera.

2 Normative references / standards item ai)

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 9276-1, Representation of results of particle size analysis — Part 1: Graphical representation

ISO 9276-2, Representation of results of particle size analysis — Part 2: Calculation of average particle sizes/diameters and moments from particle size distributions

ISO 9276-6, Representation of results of particle size analysis — Part 6: Descriptive and quantitative representation of particle shape and morphology

ISO 13322-1, Particle size analysis — Image analysis methods — Part 1: Static image analysis methods

ISO 14488, Particulate materials — Sampling and sample splitting for the determination of particulate properties

ISO/IEC 17025, General requirements for the competence of testing and calibration laboratories

3 Terms, definitions and symbols

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 13322-1 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

3.1.1

acceptable depth of field

<dynamic image analysis> depth with respect to the focal depth where the sharpness of the edges of the particle images is accepted for segmentation

Note 1 to entry: The acceptable depth of field is decided by the software based on the sharpness of the images and is also dependent on the particle size.

3.1.2

accuracy

closeness of agreement between a test result or measurement result and the *true value* (3.1.20)

Note 1 to entry: In practice, the accepted reference value is substituted for the true value.

Note 2 to entry: The term "accuracy", when applied to a set of test or measurement results, involves a combination of random components and a common systematic error or bias component.

Note 3 to entry: Accuracy refers to a combination of trueness (3.1.19) and precision (3.1.12).

[SOURCE: ISO 3534-2:2006, 3.3.1]

3.1.3

certified reference material CRM

reference material (3.1.13) characterised by a metrologically valid procedure for one or more specified properties, accompanied by an RM certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability

Note 1 to entry: The concept of value includes a nominal property or a qualitative attribute such as identity or sequence. Uncertainties for such attributes may be expressed as probabilities or levels of confidence.

Note 2 to entry: Metrologically valid procedures for the production and certification of RMs are given in, among others, ISO 17034 and ISO Guide 35.

Note 3 to entry: ISO Guide 31 gives guidance on the contents of RM certificates.

Note 4 to entry: ISO/IEC Guide 99:2007 has an analogous definition. $\frac{4b63-b8b3-14b3}{1036cbc8/iso-13322-2-2021}$

[SOURCE: ISO Guide 35:2017, 3.2]

3.1.4

flow cell

measurement cell inside which the gas- or liquid-particle mixture flows

3.1.5

frame coverage

<dynamic image analysis> fraction of the image area that is obscured by the projection area of all segmented particles counted in the image

Note 1 to entry: Frame coverage can be expressed as a part or percentage of the image area.

3.1.6

intermediate precision

<dynamic image analysis> accuracy (3.1.2) and precision under intermediate precision conditions (3.1.7)

[SOURCE: ISO 3534-2:2006, 3.3.15, modified — "and precision" and the field of application "dynamic image analysis" have been added.]

3.1.7

intermediate precision conditions

<dynamic image analysis> conditions where test results or measurement results are obtained on different dynamic image analysis instruments and with different operators using the same prescribed method

Note 1 to entry: There are four elements to the operating condition: time, calibration, operator and equipment.

3.1.8

image capture device

matrix camera or line scan camera for converting an optical image to digital image data

3.1.9

measurement zone

volume in which particles are measured by an image analyser, formed by the measurement frame including a third dimension from the *acceptable depth of field* (3.1.1)

Note 1 to entry: The measurement zone is defined by the software (see 3.1.1).

3.1.10

orifice tube

tube with an aperture through which a stream of fluid with dispersed particles flows

3.1.11

illumination

continuous illumination for an *image capture device* (3.1.8) with an electronic exposure time controller, or illumination of short duration for a synchronized image capture device

3.1.12

precision

closeness of agreement between independent test/measurement results obtained under stipulated conditions

Note 1 to entry: Precision depends only on the distribution of random errors and does not relate to the *true value* (3.1.20) or the specified value.

Note 2 to entry: The measure of precision is usually expressed in terms of imprecision and computed as a standard deviation of the test results or measurement results. Less precision is reflected by a larger standard deviation.

Note 3 to entry: Quantitative measures of precision depend critically on the stipulated conditions. *Repeatability conditions* (3.1.15) and reproducibility conditions are particular sets of extreme stipulated conditions.

[SOURCE: ISO 3534-2:2006, 3.3.4]

3.1.13

reference material

RM

material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process

Note 1 to entry: RM is a generic term.

Note 2 to entry: Properties can be quantitative or qualitative, e.g. identity of substances or species.

Note 3 to entry: Uses may include the calibration of a measurement system, assessment of a measurement procedure, assigning values to other materials, and quality control.

Note 4 to entry: ISO/IEC Guide 99:2007 has an analogous definition, but restricts the term "measurement" to apply to quantitative values. However, ISO/IEC Guide 99:2007, 5.13, Note 3 (VIM), specifically includes qualitative properties, called "nominal properties".

[SOURCE: ISO Guide 35:2017, 3.1]

3.1.14

repeatability

precision (3.1.12) under repeatability conditions (3.1.15)

Note 1 to entry: Repeatability can be expressed quantitatively in terms of the dispersion characteristics of the results.

[SOURCE: ISO 3534-2:2006, 3.3.5]

3.1.15

repeatability conditions

observation conditions where independent test/measurement results are obtained with the same method on identical test/measurement items in the same test or measuring facility by the same operator using the same equipment within short intervals of time

Note 1 to entry: Repeatability conditions include:

- the same measurement procedure or test procedure;
- the same operator;
- the same measuring or test equipment used under the same conditions;
- the same location;
- repetition over a short period of time.

[SOURCE: ISO 3534-2:2006, 3.3.6]

3.1.16

sampling volume

volume in which the particles are within the field of view of the image analyser including a third dimension from the *sampling volume depth* (3.1.17)

3.1.17

sampling volume depth

length which describes the extent of the particle field in front of the camera | 463 f036cbc8/iso-13322-2-2021

3.1.18

sheath flow

particle-free fluid flow surrounding particle-laden fluid for directing particles into a specific measurement zone (3.1.9)

3.1.19

trueness

closeness of agreement between the expectation of a test result or a measurement result and a *true value* (3.1.20)

Note 1 to entry: The measure of trueness is usually expressed in terms of bias.

Note 2 to entry: Trueness is sometimes referred to as "accuracy of the mean". This usage is not recommended.

Note 3 to entry: In practice, the accepted reference value is substituted for the true value.

[SOURCE: ISO 3534-2:2006, 3.3.3]

3.1.20

true value

value which characterizes a quantity or quantitative characteristic perfectly defined in the conditions which exist when that quantity or quantitative characteristic is considered

Note 1 to entry: The true value of a quantity or quantitative characteristic is a theoretical concept and, in general, cannot be known exactly.

Note 2 to entry: For an explanation of the term "quantity", refer to ISO 3534-2:2006, Note 1 of 3.2.1.

[SOURCE: ISO 3534-2:2006, 3.2.5]

3.2 Symbols

In this document the symbol *x* is used to denote the particle sizes. However, it is recognized that the symbols *d* and *D* are also widely used to designate these values (see ISO 9276-2).

a moving distance of a particle during time t

 A_i projected area of particle i after segmentation

 $A_{\rm real}$ projected area of the static spherical particle whose shape has been approximated by an

ellipsoid

 $A_{\rm meas}$ projected area of the measured particle whose shape has been approximated by an ellipsoid

b measured size of binary particle image, including effects from motion blur

k coverage factor

*q** distribution density

 q_0^* distribution density by number

q₃* distribution density by volume

 Q_r cumulative undersize distribution of quantity r

 $Q_r(x)$ cumulative distribution at selected particle sizes x

quantity type; number (r=0), area (r=2) or volume (r=3)

 $\sigma_{\rm s}$ $\,$ standard deviation of the test samples

 σ standard deviation

t effective exposure time

T threshold level

u_m measurement uncertainty

 u_{CRM} uncertainty of an assigned value of a certified reference material

 u_{RM} uncertainty of a characterized value of a reference material

 U_{lim} total value of the uncertainty used as the final acceptance/rejection limits for qualification tests

v particle velocity

x particle size

 $x_{10,r}$ particle size corresponding to 10 % of the cumulative undersize distribution

 $x_{50,r}$ particle size corresponding to 50 % of the cumulative undersize distribution

 $x_{90.r}$ particle size corresponding to 90 % of the cumulative undersize distribution

 $x_{\rm A}$ area equivalent diameter

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 $x_{A,\text{real}}$ area equivalent diameter of a static particle

 $x_{A,\text{meas}}$ area equivalent diameter of the measured particle

 $x_{\rm F}$ Feret diameter of projected area perpendicular to the direction of motion

 $x_{A,i}$ projected area equivalent diameter of particle i

 $x_{\mathrm{Fmax},i}$ maximum Feret diameter of particle i

 $x_{\text{Fmin},i}$ minimum Feret diameter of particle i

 ε ratio of the measured particle size *b* (under motion) to the static particle size *x*

4 Principle

4.1 Key components of a dynamic image analyser

Each system designated as a dynamic image analyser consists of the following essential key components. Additionally, some optional components can be used to either enhance the quality of the measurements or to deal with particular set-up characteristics:

- a) essential:
 - illumination,Teh State
 - particle feed,
 (https://standards.iteh.ai)
 - optical system,
 - image capture device,
 - image analysis,

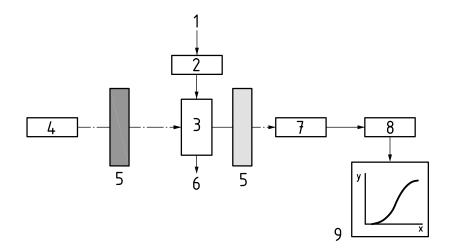
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http://conversion to meaningful particle size parameters, and 4b63-b8b3-14b3f036cbc8/iso-13322-2-2021

statistical representation of descriptors;

- b) optional:
 - particle dispersers, and
 - particle positioning.

A general diagram for dynamic image analysis is shown in Figure 1 and Figure 2. The illumination can be set-up in a transmitted light arrangement (see Figure 1), in a reflection arrangement (see Figure 2) or in a combination of both. In a reflection arrangement, a reflecting device, the vessel wall or even the particles can reflect the light back through the measurement zone as transflected light. The type of lighting has a great influence on the appearance of the particle images.

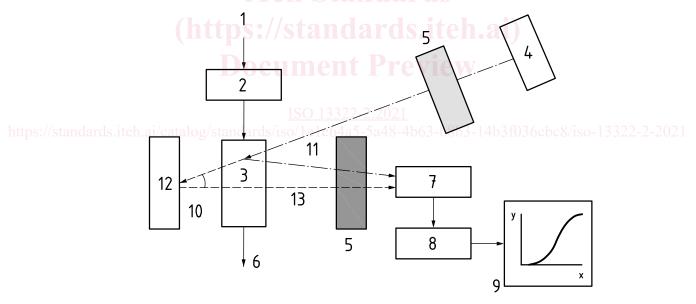


Key

- 1 dispersed particles
- 2 device for control of particle feed (optional)
- 3 measurement zone
- 4 illumination
- 5 optical system

- 6 particle flow
- 7 image capture device
- 8 image analyser
- 9 representation of results

 $Figure \ 1-Flow\ diagram\ for\ typical\ dynamic\ image\ analysis\ method\ (transmission\ set-up)$



Key

- 1 dispersed particles
- 2 device for control of particle feed (optional)
- 3 measurement zone
- 4 illumination
- 5 optical system
- 6 particle flow
- 7 image capture device

- 8 image analyser
- 9 representation of results
- 10 angle of illumination (can be set-up to zero)
- 11 reflected light from particles
- 12 reflecting objects (mirror, wall or particles, optional)
- 13 transflected light

Figure 2 — Flow diagram for typical dynamic image analysis method (reflection set-up)

4.2 Illumination

4.2.1 Time performance

4.2.1.1 General

An optimum exposure time is a crucial component of proper imaging. In principle, there are two different methods to achieve a time performance balanced between minimised motion blur and sufficient contrast. In both cases, the instrument manufacturer shall care for providing as much intensity as required for sufficient contrast between background and particles.

4.2.1.2 Pulsed illumination

At first, limiting the exposure time via short light pulses has been a method for several decades. Various electrical illumination sources in combination with condenser and collector lenses such as electric discharge flashlight bulbs, light emitting diodes (LED) and laser diodes have different properties like slew rate when switching on and off, light intensity, stability and durability.

4.2.1.3 Continuous illumination

The second method uses a permanent light source while the capturing device itself electronically handles the exposure time (shuttered detection). Typically, cold cathode fluorescent lamp (CCFL) tubes, permanent LED grids or lamps in combination with condenser and collector lenses are used. Another solution is the usage of an adapted wide light screen.

4.2.2 Direction of illumination

4.2.2.1 General

At least two different set-ups are widely common: illumination from the back of the particles (transmission set-up, see Figure 1) or direct illumination from the front with an angle between the direction of illumination and that of observation (reflection set-up, see Figure 2). Both methods shall care to have sufficient contrast between background and foreground (particles) and hence for detectable particle edges.

4.2.2.2 Back illumination

Back illumination requires a set-up with a light source and image capture device at opposite sides of the particles. Back illumination provides a projection area like a shadow of the particle perpendicular to the direction of observation (shadow or bright field method). Parallel light minimises reflected light on the sides of the particles which can otherwise reduce the contrast of the image edges. The method should cope with the challenge of (partially) transparent particles creating even more complex shadow structures. It delivers the projection area of the particle and information about its shadow's shape whereas colour and 3D information of the particles on the single instance are lost.

4.2.2.3 Illumination from the front and other directions

Front illumination is widely used in classic photography, for example, flashlights or ring illumination mounted near to the camera lens. As in photography the capturing device as well as the subsequent image processing should deal with the classical drawbacks of this set-up like reflections, deflections and refractions. As for back illumination, the image quality of the edges becomes important for the quality of the results. For the reflected light used to obtain the information about the particles, some information of the visible particle surface and the edges is obtained.