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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

ICS: 19.120

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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).

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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.

The committee responsible for this document is 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:

— consideration of changes to the last revision of ISO 13322-1:2014

— significantly expanded sections on instrumentation (principle) and operational procedures

— new section on accuracy and instrument qualification using particulate reference materials

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

Introduction

The purpose of this second part of ISO 13322 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 second part of ISO 13322.

Identification of patent holders, if any.

Particle size analysis — Image analysis methods —

Part 2:

Dynamic image analysis methods

1 Scope

ISO 13322 is applicable to the analysis of images for the purpose of determining particle size distributions.

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

ISO 13322-2 describes the transfer of images from particles having relative motion to binary images within practical systems, in which the particles are highly diluted. 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. The application of this method requires the particle images to be clearly distinguishable from a 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 higher number of particles compared to static image analysis systems. This International Standard provides guidance on instruments qualification and particle size distribution measurement by using particulate reference materials. This part 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

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 13322-1:2014, *Particle size analysis — Image analysis method — Part 1: Static image analysis methods*

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 14488:2007, *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 13322-1:2014 and the following apply.

3.1.1
acceptable depth of field
<dynamic image analysis> depth with respect to focal depth where the sharpness of the edges of the particle images is accepted for segmentation

3.1.2
accuracy
closeness of agreement between a test result or measurement result and the true value

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 and precision.

[Source, ISO 3534‐2:2006, clause 3.3.1]

3.1.3
certified reference material
CRM

reference material (RM) 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 (5.14).

[Source, ISO Guide 35:2017, 3.2]

3.1.4
flow-cell
measurement cell inside which the fluid-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 in the image

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

3.1.6
image capture device
matrix camera or line scan camera for converting an optical image to digital image data

3.1.7
measurement zone
volume in which particles are measured by an image analyser. The measurement zone is formed by the measurement frame including a third dimension from the acceptable depth of field.

Note 1 to entry: the measurement zone is defined by the software

3.1.8
orifice tube
tube with an aperture through which a stream of fluid with dispersed particles flows

3.1.9
particle illumination
continuous illumination for image capture device with an electronic exposure time controller, or illumination of short duration for synchronized image capture device

3.1.10
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 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 and reproducibility conditions are particular sets of extreme stipulated conditions.

[Source, ISO 3534 - 2:2006, clause 3.3.4]

3.1.11
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[3] has an analogous definition (5.13), 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]

3.1.12
repeatability
precision under repeatability conditions

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

[Source ISO 3534 - 2:2006, clause 3.3.5]

3.1.13
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, clause 3.3.6]

3.1.14
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

3.1.15
sampling volume depth
length which describes the extent of the particle field in front of the camera

3.1.16
sheath flow
clean fluid flow surrounding particle-laden fluid for directing particles into a specific measurement zone

3.1.17
trueness
closeness of agreement between the expectation of a test result or a measurement result and a true value

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, clause 3.3.3]

3.1.18
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 1 to entry: For an explanation of the term "quantity", refer to [ISO 3534 - 2:2006.]

3.2 Symbols

a moving distance of a particle during time t

A_i projected area of particle i

b measured diameter of binary image

CF coverage factor

$D_{10,r}$ particle diameter corresponding to 10 % of the cumulative undersize distribution

$D_{50,r}$ particle diameter corresponding to 50 % of the cumulative undersize distribution

$D_{90,r}$ particle diameter corresponding to 90 % of the cumulative undersize distribution

Q_r cumulative undersize distribution of quantity r

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

s standard deviation of the test samples

σ standard deviation

t exposure time

u_m measurement uncertainty

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

u_{RM} uncertainty of a characterized values 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 or D diameter of particle

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

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

$x_{90,r}$ particle diameter corresponding to 90 % of the cumulative undersize distribution

x_{Ai} projected area equivalent diameter of particle *i*

$x_{i\max}$ maximum Feret diameter of particle *i*

$x_{i\min}$ minimum Feret diameter of particle *i*

ε ratio of the measured particle diameter to the static particle diameter

4 Principle

4.1 Key components of a dynamic image analyser

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

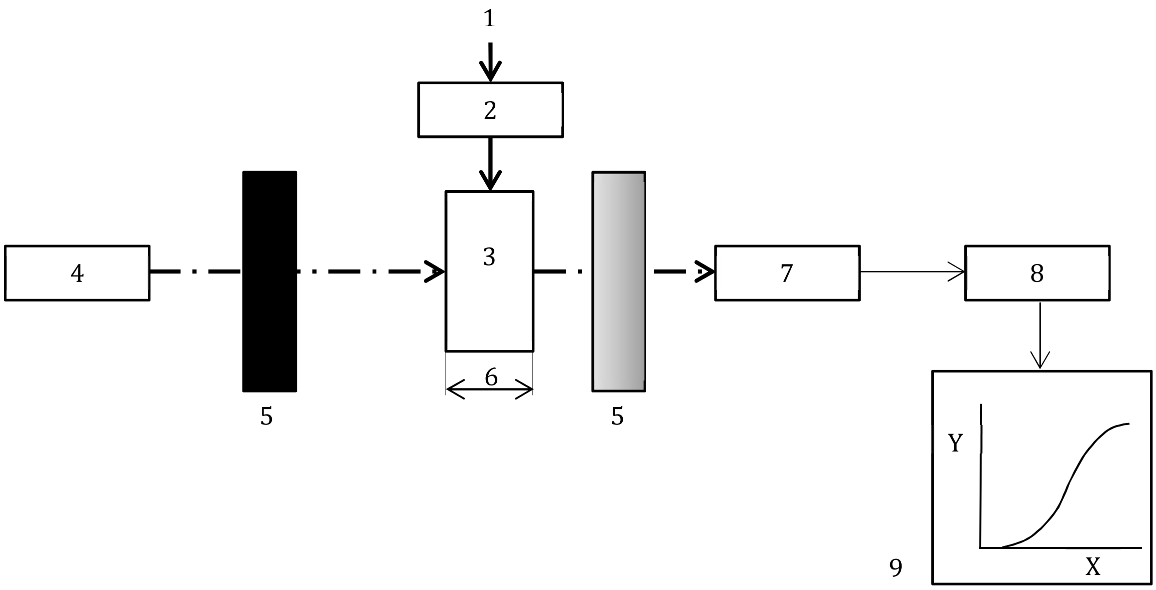
a) Essential

- Illumination
- Particle motion
- Optical system
- Image capture device
- Image analysis
- Conversion to meaningful particle size parameters
- Statistical representation of descriptors

b) Optional

- Particle dispersers
- Particle positioning

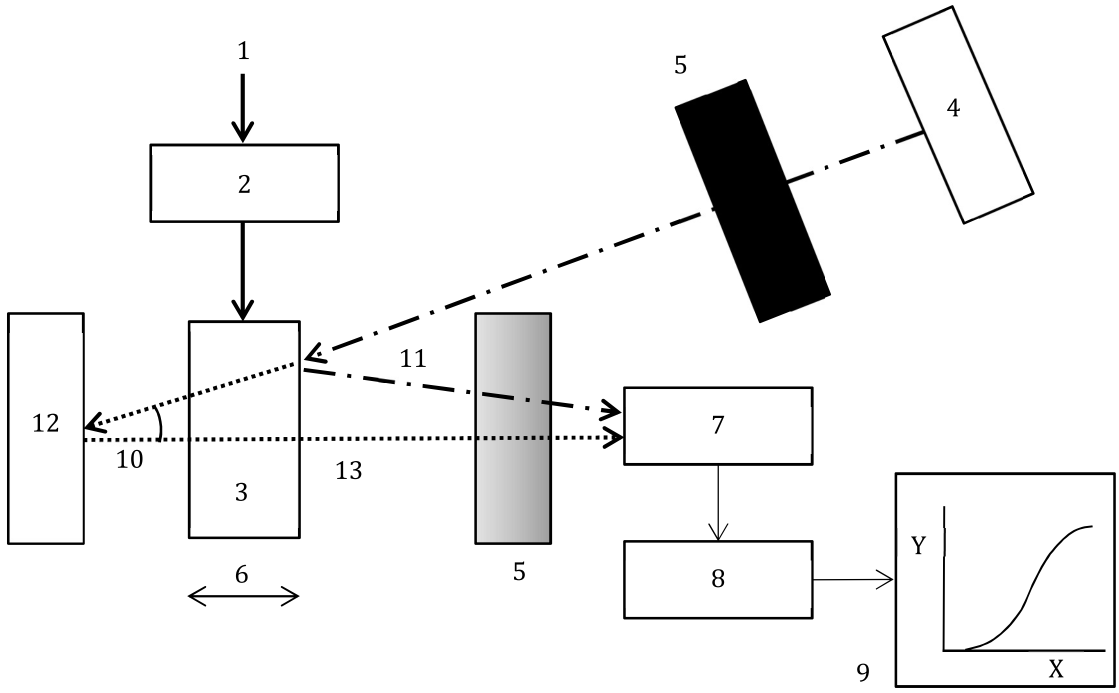
A general diagram for dynamic image analysis is shown in Figure 1 & Figure 2. The illumination can be set-up in a transmitted light arrangement (Figure 1), in a reflection arrangement (Figure 2) or in a combination of both. In a reflection arrangement a reflecting device, the vessel wall or even the particles may 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 motion (optional)
3 measurement zone
4 light source
5 optical system
6 acceptable depth of field
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 motion (optional)
3 measurement zone
4 light source
5 optical system
6 acceptable depth of field
7 image capture device
8 image analyser
9 representation of results
10 angle of illumination (may be set-up to zero)
11 reflected light from particles
12 reflecting objects (mirror, wall or particles)
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 has to care for providing as much intensity as required for a sufficient contrast between background and particles.

4.2.1.2 Pulsed illumination

At first, limiting the exposure time via the time performance of the light source has been a well-established method for several decades. Various electrical illumination sources such as: electric discharge flash light 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 whilst the capturing device itself electronically handles the exposure time (shuttered detection). Typically, CCFL-tubes, permanent LED grids or lamps in combination with condenser and collector lenses are used. Another solution is the usage of 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 a small angle between the direction of illumination and that of observation (reflection set-up, see Figure 2). Both methods shall care for a sufficient contrast between background and foreground and hence for detectable particle edges.

4.2.2.2 Back illumination

Back illumination requires a set-up with 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). The method has to 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, e.g. flash lights or ring illumination mounted near to the camera lens. As in photography the capturing device as well as the subsequent image processing has to 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 is used to obtain the information about the particles, some information of the visible particle surface and the edges is obtained.

4.2.3 Spectrum of illumination

4.2.3.1 Polychromatic

Polychromatic illumination allows for colour information of the particles whereas additional errors like chromatic aberration may have to be taken into account. In addition, the position of the particle edges and possible blurs may depend on the used spectrum. Typical light sources providing polychromatic light are the classic flash light, day light, incandescent lamps and some multiply coloured LEDs.

4.2.3.2 Monochromatic

As a consequence of using single-colour LEDs or lasers for illumination monochromatic light is also used. Obviously, no colour information is obtained. Using laser light for illumination the capturing device and the image evaluation respectively may have to deal with speckles and interference effects from the coherent light source.

Together with the numerical aperture of the imaging lens the wavelength of the illumination limits the theoretical maximum optical resolution of a lens.