INTERNATIONAL STANDARD

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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

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Foreword

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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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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.

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

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.

[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

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_{real}	projected area of the static spherical particle whose shape has been approximated by an ellipsoid
A_{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
$q_3{}^*$	distribution density by volume
Q_r	cumulative undersize distribution of quantity r
$Q_r(x)$	cumulative distribution at selected particle sizes x
r	quantity type; number ($r=0$), area ($r=2$) or volume ($r=3$)
σ_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_A	area equivalent diameter

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

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

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

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

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

$x_{\mathrm{Fmin},i}$ 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:

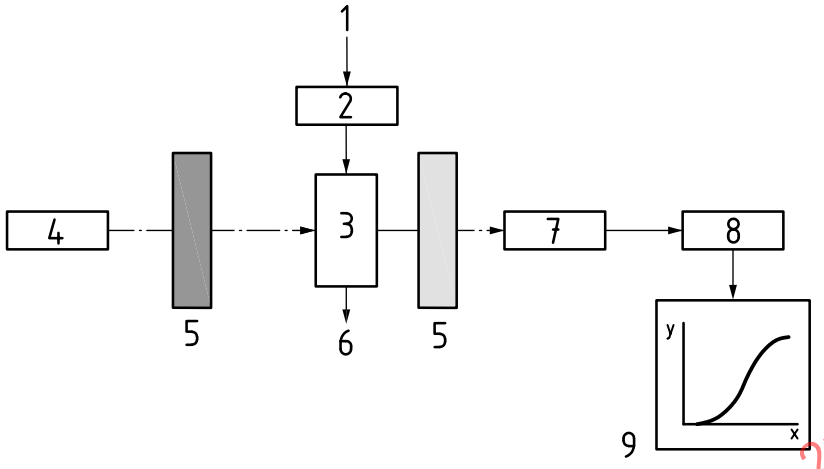
a) essential:

- illumination,
- particle feed,
- optical system,
- image capture device,
- image analysis,
- conversion to meaningful particle size parameters, and
- statistical representation of descriptors;

b) optional:

- particle dispersers, and
- particle positioning.

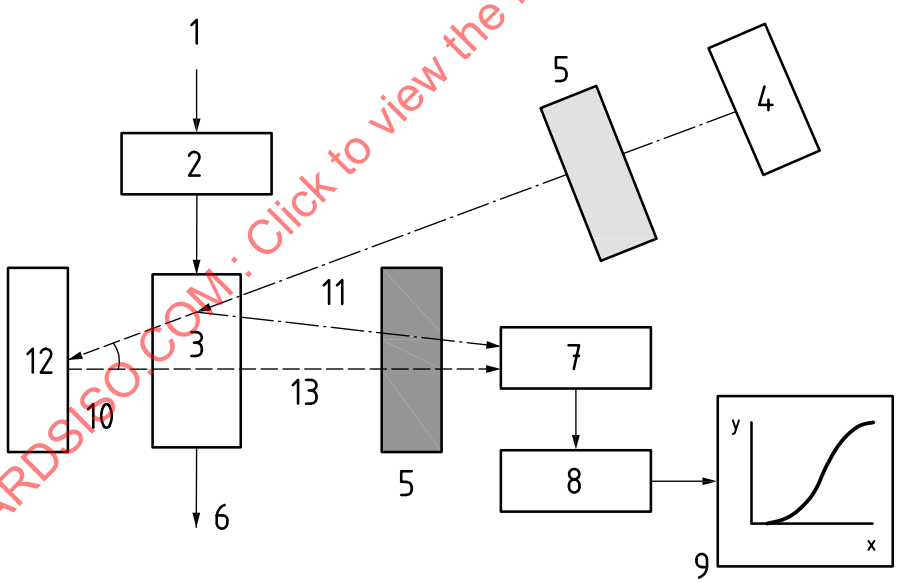
A general diagram for dynamic image analysis is shown in Figure 1 and Figure 2. The illumination can be set-up in a transmitted light arrangement (see Figure 1), in a reflection arrangement (see Figure 2) or in a combination of both. In a reflection arrangement, a reflecting device, the vessel wall or 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.

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 should be taken into account. In addition, the position of the particle edges and possible blurs can depend on the used spectrum. Typical light sources providing polychromatic light are the classic flashlight, daylight, 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 of the capturing device, the image evaluation respectively should 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 (see Reference [4]).

4.2.4 Stability of the light source

All systems should illuminate the particles' images captured at different times with at least comparable intensity to avoid later contrast or segmentation fluctuations or even imaging artefacts. Illumination stability should be given in the long run as well as from image to image (pulse-to-pulse). Instability should be handled by using adapted segmentation algorithms.

4.2.5 Special types of illumination

4.2.5.1 Dark and bright field illumination

Some special types of illumination can enhance the contrast of fine structures, for example, dark field illumination where the unscattered beam from the illumination is excluded from the image. The main limitations of dark field microscopy are the low light levels in the final image and the interpretation of the image structures.

Bright field microscopy can use critical or Köhler illumination to increase the optical resolution, but then bright field microscopy typically has low contrast with transparent particles.

4.2.5.2 Polarized light

Polarized light and filters can be used to enhance the contrast by using optical phase contrast microscopy techniques.

4.3 Particle motion

Moving particles can be introduced into the measurement zone by three means:

a) particle motion in a moving fluid (e.g. particles in suspension, in an aerosol, in a duct, in an air jet, in a sheath flow, in turbulent flow or in a push-pull flow regime);

b) particle motion in a still fluid, i.e. in an injection or free-falling system, where particles are intentionally moved by an external force (e.g. gravity, electrostatic charge);

c) particle motion with a moving substrate, where particles are on the moving substrate (e.g. conveyor belt), provided that the frame coverage of the particles is low and the particles are separated.

4.4 Particle positioning

Images are taken when particles reach the measurement zone. The depth of the measurement zone in front of the image capture device is determined either by the sampling volume depth of the particles (i.e. the width of a flow cell) or by the acceptable depth of field. The acceptable depth of field is a combination of the depth of field of the optical system together with a software decision to accept or reject blurred particle images. The acceptable depth of field and thus the measurement zone effectively depends on the particle size (see D.3). There are two possible arrangements for a dynamic particle image analyser.

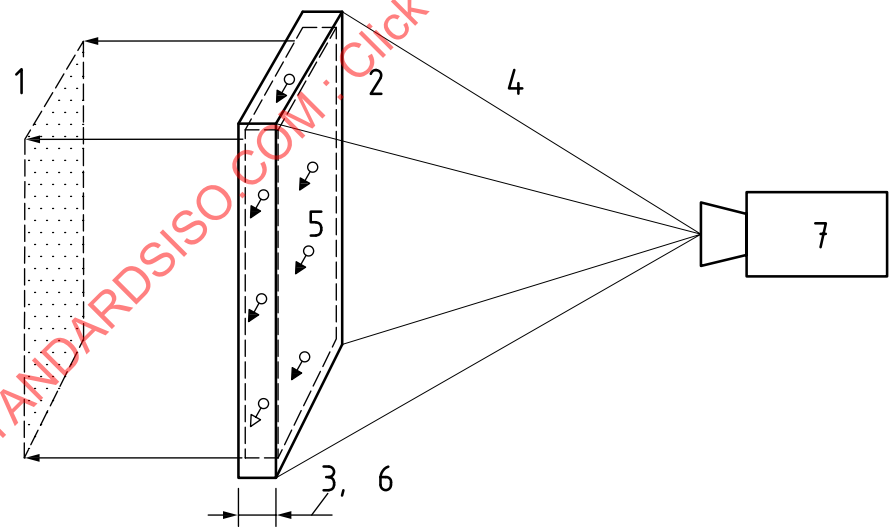
a) The particle movement can be controlled in order for all particles to be within the acceptable depth of field of the smallest particles measured. By using this arrangement, all particle images in the measurement frame shall be accepted for segmentation. Figure 3 shows an example of this type of arrangement.

b) The particles can be allowed to move freely into or out of the acceptable depth of field. Since all recorded images of particles detected outside the acceptable depth of field shall be rejected, corrections to the proportions of the particle numbers shall be applied to the result. Figure 4 shows an example of this type of arrangement.

NOTE The particles can also be allowed to move freely into or out of the acceptable depth of field if the focus of the image capture equipment can be controlled fast enough to acquire the exact image of the particles moving in the fluid for example by capturing the image of the moving particles only when they pass through the measurement zone of the image capture equipment. In this case, a correction of the particle count is not necessary.

It is also required that the particles move freely relative to each other. It is also necessary that all particles traverse the measurement zone at the same velocity if the effects of velocity bias are to be avoided. If the different velocities are known, corrections may be applied as if the particles travel at the same speed.

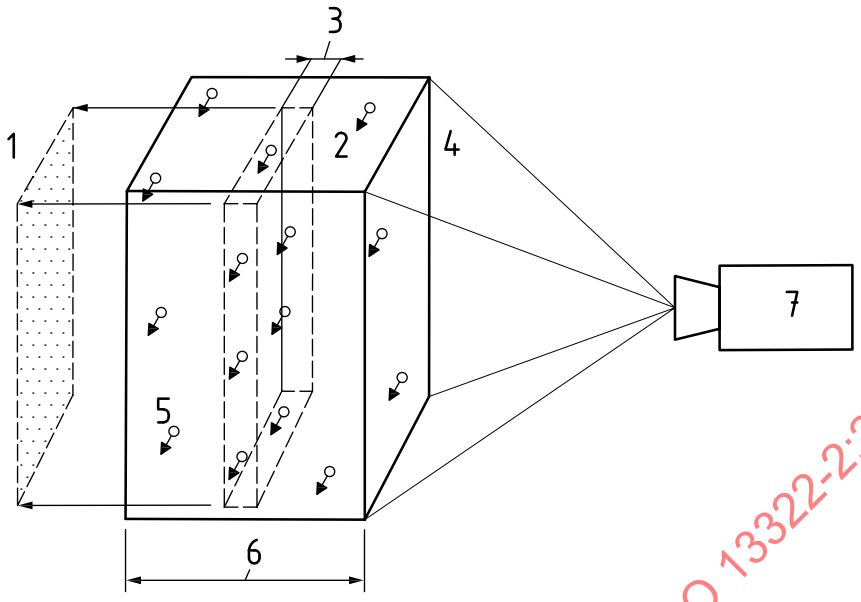
The orientation of the particles with respect to the measurement zone affects the interpretation of particle size and shape.



Key

1	measurement frame (2D dimension of Key 2)	5	dispersed particle
2	measurement zone (3D volume)	6	sampling volume depth
3	acceptable depth of field	7	image capture device
4	field of view		

Figure 3 — Example of a measurement zone where the particle movement is only within the depth of the measurement zone



Key

1	measurement frame (2D dimensions of Key 2)	5	dispersed particle
2	measurement zone (3D volume)	6	sampling volume depth
3	acceptable depth of field (size dependent)	7	image capture device
4	field of view		

Figure 4 — Example of a measurement zone where particles are moving within and outside of the measurement zone as well as movements pointing into and out of the measurement zone

4.5 Optical system

4.5.1 General

Precise focus is possible at only one distance between the object and the lens of the instrument. Only within a plane at that distance (object plane), a point object produces a point image at the image capture plane (image plane) which is usually determined by the sensor surface of the image capture device (see Annex A).

4.5.2 Lens design

General imaging lenses exhibit varying magnification for objects at different distances from the lens. Using these so-called entocentric lenses, the size dependence on the particle's position (the distance from the lens to the particle) should be taken into account.

Telecentric lenses provide an orthographic projection, providing the same magnification at all distances. An object can still be out of focus, but the resulting blurry image can still have the same size as the correctly focused image.

Telecentric lenses can be combined with telecentric back illumination parallel to the optical axis. This allows for a smaller aperture stop of these lenses which enlarges the depth-of-field and the contrast of transparent objects but can limit the optical resolution because of decreasing the numerical aperture.

4.5.3 Optical magnification

Optical magnification is the ratio between the apparent size of an object in its image and its true size.

4.5.4 Optical resolution

The measure of how closely lines can be resolved in an image is called spatial resolution. The term optical resolution is sometimes used to distinguish the resolution of the optical systems from the sensor resolution (spatial density) of the image capture device (see also 4.6.5).

4.5.5 Lens errors

An optical system may show distortion where the magnification in the image plane changes with the distance to the optical axis. This shall be determined and corrected in a suitable manner (see A.3).

The image plane is determined by the flat sensor of the image capture device. Yet, the object plane upon which a lens focuses may not be a literal flat plane and will show a field of curvature.

4.6 Image capture device

4.6.1 Matrix camera

A digital area image sensor contains a two-dimensional array of pixel sensors allowing the conversion of incoming photons into electron charges or current. It is recommended that the matrix sensor uses a square grid of pixel elements in order to simplify the particle size and shape calculations.

4.6.2 Line scan camera

A line scan camera has a single or up to four lines of pixel sensors, which are read out at a certain frequency. The recorded lines are stacked and create the picture of the scanned object (particle) – camera or object move (e.g. copy or fax machine). The velocity of moving objects (particles) or the moving camera needs to be known to calculate the image. The line scan camera creates a continuous picture without multiple upper and lower picture edges of the particle stream. It allows a high number of pixels in one row and reduces edge effects on two sides of the recorded image (top and bottom).

4.6.3 Exposure time

The exposure time is the integration time of the image capture device either defined by the light pulse (flash) or the electronics of the image capture device. The exposure time shall be adapted to the particle motion to reduce the motion blur of the image.

4.6.4 Frame rate/line rate

The frame rate is the frequency at which an imaging device records consecutive images (frames). The frame rate has statistical relevance on the result.

a) The flowing particle sample should be recorded in a constant frame rate so that the recorded images are representative of the complete sample. A high frame rate is required to capture a sufficient number of particles in a shorter time.

b) When using a high frame rate, overlapping images may be recorded counting particles multiple times. If the particles are rotating, they may be captured from various sides.

c) A very small number of particles are allowed per frame to avoid particle image overlap or particles touching each other.

d) As a line scan camera is a one-dimensional camera, the line rate defines the resolution in the second dimension, the moving direction.

4.6.5 Sensor resolution

The sensor resolution is specified using the pixel size and pixel number in both directions of the two-dimensional image array. The sensor resolution should be matched to optical resolution. There is no

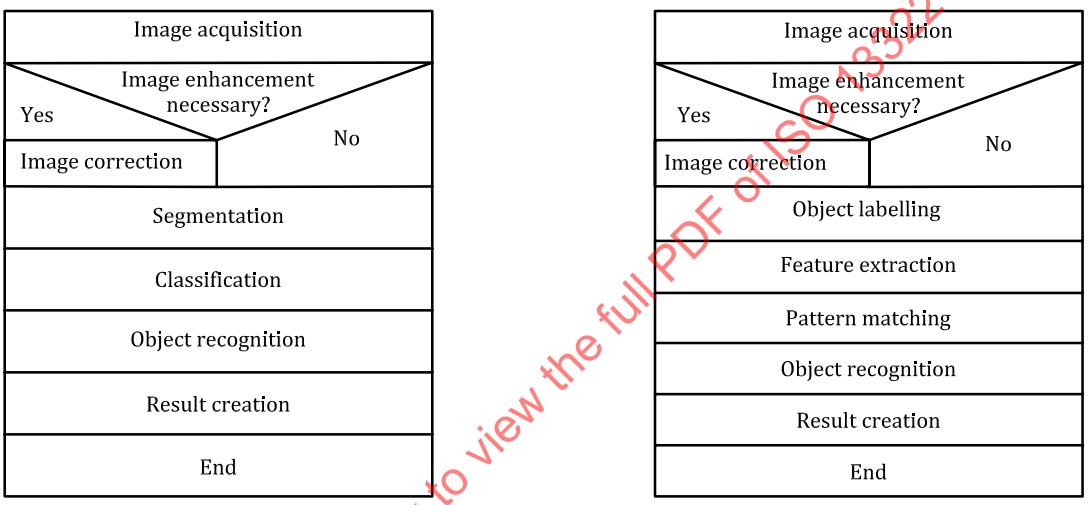
gain in image information when the sensor resolution exceeds the optical resolution limited by the numerical aperture of the imaging device significantly.

It can be beneficial to use 2 times to 3 times higher digital resolution than the optical resolution to evaluate the image sharpness for segmentation and to describe the particle shape. A digital resolution increased by oversampling the optical image does not improve the measurement of the particle size from a projection surface.

4.7 Image analysis methods

4.7.1 Image analysis process

Possible sequences of image analysis methods used for particle size analysis are shown by flow diagrams in Figure 5.



a) Flow-diagram for classical segmentation analysis

b) Flow-diagram for pattern matching analysis

Figure 5 — Image analysis process

4.7.2 Robustness of the image analysis method

Many image analysis methods depend on one or several parameters. If these parameters are set by the user, the robustness of the settings shall be checked by using particulate material to ensure that a small variation of the parameters leads only to a small change of the results (see Annex F).

4.7.3 Image correction

The digital images shall be corrected prior to segmentation, if required.

Image geometry correction is the process of digitally manipulating image data so that the image's projection matches a specific projection shape. Image geometry correction shall be used to correct local changes in the imaging scale (distortion).

Image noise is a random variation of brightness information in images and is usually an aspect of electronic or unavoidable shot noise of the photon detection. Excessive image noise influences the segmentation method and should be corrected using digital filters prior to segmentation.

Image correction may also contain image enhancement like shading correction, resizing, filtering, morphological filters on grayscale images and image type conversion.

4.7.4 Segmentation methods

4.7.4.1 General

Image segmentation is the process of partitioning a digital image into multiple segments. In dynamic image analysis, the pixels of an image are divided into background pixels and into sets of pixels belonging to the particle images. Several algorithms and techniques have been developed for image segmentation. This document considers only methods using full pixel numbers to be compatible with ISO 13322-1. The filling of holes in the images of the particles can be considered as an additional step of the segmentation method.

The algorithms for size and shape calculation shall be adapted to the segmentation method in order to obtain identical results from the same particle images.

4.7.4.2 Threshold

In this method, all pixels whose (grey-value) intensity is above a certain threshold value are considered part of a foreground object. Pixels whose (grey-value) intensity is below a certain threshold value are considered part of the background. An inverse binarization process is possible. Such an image is usually displayed as a binary image using black and white pixels to distinguish the particles from the background.

4.7.4.3 Edge detection

Edge detection algorithms can also be used to determine contours around objects tracing with a greyscale level or highest derivative. One problem with these methods is that they do not necessarily yield closed curves.

4.7.4.4 Pattern matching

If the particles have similar characteristics and boundary features, for example, oil droplets or air bubbles in a multiphase system, typical features can be extracted and be correlated during pattern matching to identify similar objects in a series of images. The image series is processed to identify particles that match the characteristics contained in the generated pattern (pattern matching). The same principle of pattern-based searching can be applied to all kinds of shapes, colours or other systematic structures. Automatic object recognition is most effective when objects appear in high contrast to the background.

4.7.5 Particle classification

After the particles have been filed as binary image objects they can be further classified with respect to their shape and size. Setting up filter conditions with respect to shape descriptors and diameters, allows for the separation of different classes of particles, for example, gas bubbles from irregularly shaped particles. The particle size and shape result can then be calculated for each class independently.

4.8 Conversion to meaningful particle descriptors

See ISO 13322-1 and ISO 9276-6.

The measured particle size distribution (by number and also by volume) is not an exact representation of the total number of measured particles because several corrections can be employed. See, for example, 4.4, 5.2.3.2 and 5.2.3.4.

4.9 Statistical representation of descriptors

See ISO 9276-2.

4.10 Particle dispersion technique

Dispersion controls the separation, positioning and optical concentration, and shall minimize segregation and velocity bias for a homogenous and representative distribution of the particles in the measurement volume.

4.11 Systematic corrections dealing with set-up characteristics

Some parameters of the systems shall be determined in addition to correct the images or the particle size results by software:

a) particle velocities to calculate a two-dimensional image from a line camera system;

b) particle positioning for a constant working distance of particles in non-telecentric (entocentric) imaging to correct for differences in magnification;

c) assessment of blurred particles to exclude them from the measurement result; this can depend on particle size (see 4.4).

5 Operational procedures

5.1 General

A pre-requisite for accurate particle size measurement using this method requires a full understanding of the settings and calibration applied within the image capture device as well as a consideration of the purpose for conducting the measurement.

5.2 Instrument set-up and calibration

5.2.1 Preliminaries

The dynamic imaging instrument shall be set up and operated in accordance with the manufacturer's recommendations.

5.2.2 Site of installation

The instrument and its equipment shall be installed under sufficiently stable and clean conditions (see 5.2.1). External influences, for example, direct sunlight, heating, vibrations, shall be avoided in order to obtain high-quality results.

5.2.3 Magnification and sensor resolution

5.2.3.1 Optical calibration

The equipment shall be calibrated to convert pixels into SI length units (e.g. nanometres, micrometres and millimetres) for the final results. The calibration procedure shall include verification of the uniformity of the field of view. Corrections shall be applied in case of image distortion (see 4.7.3). An essential requirement of the calibration procedure is that all measurements shall be traceable to the standard metre. This can be achieved by calibration of the image scale with a certified standard stage micrometre. It is also possible to calibrate the magnification factor using circular objects like chrome particles on glass. As the measurement and calibration is done by area, more pixels are involved compared to a linear scale. This leads to a higher resolution of the local magnification measurement but is dependent on the segmentation procedure. When a microscopic scale cannot be placed into the measurement zone, for example, in process environments, spherical, monodisperse particles, the diameter of which is traceable to the metre, may also be used. These procedures are a required calibration of the optical components. This does not qualify the entire system which includes segmentation, particle positioning and velocity effects.

The magnification shall be set up such as to provide a minimum number of pixels for the smallest particle consistent with the accuracy demanded and set to achieve a sharp focus. The largest particles shall be able to pass through the measurement volume.

Both the number of pixels forming the image of the particle and the relative position of the centring of the image with respect to the fixed pixel pattern can have a material influence upon the final particle size assessed from each particle image (see ISO 13322-1). Table 1 is provided as a recommendation for the minimum number of pixels for a single particle image. These numbers are only valid, if the optical resolution is better or about 1 pixel. Different size and shape parameters have different demands on the number of pixels used.

Table 1 — Recommendation for the minimum number of pixels

Minimum pixel number in one direction	Effect on the evaluation of an image of a single particle
1	Prone to noise
3	Acceptable size results
9	Acceptable size and shape results

5.2.3.2 Field of view

The image of the largest particles shall fit onto the sensor surface which defines the measurement frame. All particles cut by the edge of the measurement frame shall be neglected. This creates the situation where the probability for a particle to be included in the result varies inversely with the size of the particle and the population of the particle in its size class shall be corrected by this probability (see ISO 13322-1). The smallest recommended probability for correction shall be 0,5. Therefore, the magnification shall be selected so that the maximum diameter of the largest particle does not exceed one-third of the shorter side of a rectangular image frame of the measuring area.

Such a correction is not required if the method ensures that each particle is observed at least once (see 5.3.2.2).

5.2.3.3 Resolution

The theoretical limit for the resolution of objects by size using image analysis is 1 pixel, and counts shall be stored particle by particle, with the maximum resolution of 1 pixel. However, it is necessary to define size classes for the final reporting of the result, which is a function of the total number of particles, the dynamic range and the number of pixels included in the smallest considered objects. It is recommended that pixel size be converted to actual size prior to any reporting of size for quantitative analysis.

5.2.3.4 Focusing and acceptable depth-of-field

The measurement volume shall be selected to achieve a sharp focus consistent with the accuracy demanded for the smallest particles measured. The position of the moving particles shall be controlled so that they pass only within the measurement zone of the image capture equipment. Systems where the sampling volume depth exceeds the measurement zone shall include a regime to control and monitor the correct particle representation which should be calibrated with a certified reference material with a wider distribution or with multi-modal-particle standards (see ISO/TS 14411-1) or by scanning through the sample volume depth with reference objects of different certified sizes.

5.2.4 Illumination

In order to achieve accurate particle size measurements, it is preferred that the illumination be uniform over the total field of view and of a type designed to create images of high contrast. The instrument shall employ an acceptance criterion for the illumination set-up.

5.2.4.1 Contrast

Distinguishing a particle image from its background a sufficient contrast is crucial for both detection and size measurement. The brightness of the image shall allow for a safe distinction between foreground pixels belonging to particle objects and background pixels, i.e. the contrast ratio or dynamic range shall be maximised.

5.2.5 Segmentation

5.2.5.1 Brightness and threshold calibration

In an image, the particle edge shall be defined by a suitable threshold level. The technique for doing this depends on the sophistication of the image analysis equipment.

It is strongly recommended that the threshold level be adjusted by comparing the processed binary images with the original grey images, in order to ensure that they are a reliable representation of the original grey images or the images obtained in the focal plane.

Threshold calibration shall be performed by reference materials like reticules or particles ideally having similar optical properties to the particles to be measured.

If a calibration of the segmentation process is required, particles shall be selected with similar optical properties as the real particles to be measured. Their size should include the dynamic range of the entire system. It is recommended to calibrate with multimodal mixes of certified particles, i.e. with values near the mid-point and minimum particle sizes to be measured with the system.

Performance qualification shall only be demonstrated using real particles as reference material.

CAUTION — Failure to set an appropriate threshold level can lead to significant bias in the determination of the particles size. This bias depends on particle size. All particles are affected but the relative value of influence to the particles size increases with decreasing particle size under a given resolution.

5.2.5.2 Automatic set-up

The preferred method is to "auto-threshold" the image. Such an auto-threshold procedure shall be validated against a certified reference material (particles or reticule) preferably having optical properties similar to that of the particles under test. Ensure that the threshold applied is independent of particle size.

5.2.5.3 Manual set-up

A threshold method can be established manually, if necessary.

EXAMPLE If a half-amplitude method is applicable, a small region of the background located a few pixels away from the boundary of a typical particle is selected to establish the background value. The amplitude of the signal from pixels just fully responding to the particles' presence is selected to establish the foreground value. The average of these two values is used to set the threshold level.

NOTE Manual threshold levels can be subjectively checked by direct comparison of the threshold image with that of the original image. This subjective method does not constitute validation but readily detects incorrect settings.

5.2.6 Contamination

Contamination of the optical system can have an impact on image quality and should be avoided as much as possible. The instrument shall provide a method to quantify the amount of contamination. The

contamination is acceptable unless the particle size distribution changes significantly or the change in contrast level is below a critical limit.

NOTE Systems with a wider depth of field are more sensitive to contamination in the optical path than systems with a smaller depth of field. This is important in case the protection screens are relatively close to the measurement volume or the acceptable depth of field. Systems using coherent light for illumination are more sensitive for contamination than systems using incoherent light.

5.3 Dispersing systems

5.3.1 Preliminary considerations

A dispersing system should be selected according to the properties of the sample. The dispersing system can affect the location, velocity and orientation of the particles in the measurement volume, depending on their size and shape. This can also depend on dispersing parameters like particle concentration, circulation speed and air pressure.

In the case of wet systems, a test sample is dispersed in an appropriate transparent liquid medium, and transported to an optical (sample) cell, in which the measuring zone is formed. A recirculation system is often used, consisting of an optical cell, a sample bath (with either agitation or sonication, or both), a pump and tubing. In this case, the particles pass through the measurement zone multiple times. The measurement may be repeated using the same sample.

In dry powder systems, particles are converted into aerosols by a dry powder feeder and a disperser, before being introduced into the measuring zone. The aerosolised particle stream is either blown or sucked through the measurement zone. It is preferred that the concentration of aerosolised powder remains steady during the averaging of the scattered light. Dry dispersion is restricted to single shot analysis where the particles pass through the measurement zone only once. This requires controlling the particle velocities in these dry systems.

Systems using free falling particles, conveyor belts or other methods of sample feeding may also be employed (see Annex H).

5.3.2 Particle velocity

5.3.2.1 General

The maximum velocity of the particles shall be controlled in order to minimize the effects on the images.

5.3.2.2 Still image resolution and motion blur

The resolution of an image captured by a dynamic image analysis system depends not only on the optical system (lens magnification and camera resolution) but also on the lighting system and the velocity of the particles.

When a spherical particle of diameter x (pixel) moves at a velocity v (pixel/s), the centre of the projected area of the particle moves a distance a (pixel) during a time t (s), where t is either the strobe light emission time or camera shutter opening time (see Figure C.1), i.e. $a = v \times t$

Without appropriate grey level handling, a shall not exceed either 0,5 pixel or $x \times (\varepsilon^2 - 1)$ pixel, where ε is an acceptable ratio of the particle diameter measured at the particle image enlarged by motion to the diameter measured at a static particle (see Annex C). Grey level handling between pixel level and background level reduces the difference between both diameters.

The total system resolution should be determined based on the particle size distribution and the desired confidence limits (see also ISO 13322-1).

5.3.2.3 Missed particles and multiple detection

5.3.2.3.1 Matrix camera systems

The frame rate of the image capture device should be adapted to the requirements of the measurement. Either the particles are measured one time, are measured multiple times or are missed in between two frames. In general, it cannot be ensured that each particle is measured only once. If the particles of the sample are still distributed homogeneously in the image frames, a bias with respect to the frame rate is not observed in a measured particle size distribution.

5.3.2.3.2 Single shot measurement

If the frame rate is sufficiently high to observe all particles at least one time, and the particles are tracked and processed by software in such a way that even if each particle is observed multiple times, these multiple instances are assembled into a single representation of each particle. Such particle counting is also possible for virtual line matrix and line scan camera systems, as all the particles passing the camera are measured only once.

5.3.3 Frame coverage

The number of particles images overlapping each other should be minimised. It is a prime requirement of the method that measurements shall be made on isolated particles. Overlapping particle images measured as one particle without a proper separation can introduce error in particle area and related parameters.

Errors introduced by overlapping particle images have a larger effect on parameters like aspect ratio, equivalent area diameter and length than on other descriptors like the smallest maximum chord or Wadell-roundness.

It is often not possible to reliably detect overlapping particles by image analysis alone, but the influence of touching particles on the result can be investigated experimentally by increasing or decreasing the number of particles per image.

5.3.4 Medium

5.3.4.1 Wet

Any suitable, optically transparent liquid may be used. The medium shall not introduce additional obscuration either by absorption or scattering from fine particles below the instrument's resolution.

The number of bubbles shall be minimized. The influence of the bubbles on the result can be corrected in software if the bubbles can be discriminated from the sample particles either by shape or optical properties.

5.3.4.2 Dry

For dry dispersion, compressed gas can be used as a medium. The dry medium shall be clean with respect to particle contamination.

5.3.5 Homogeneous dispersion and segregation

The transport conditions for the particles through the measurement zone should also be considered. Adequate flow should be applied to ensure that particles of all sizes pass the measurement zone either at a similar velocity in order to avoid velocity bias in the result or the velocity should be detected for a correct representation of the particles. Particles with a low aspect ratio tend to show preferred orientations at the flow conditions existing in the measurement cell. Even at turbulent conditions, their orientation may not be fully random, so care should be taken if random orientation is required.

5.4 Operational qualification

The correct operation of the instrument shall be verified by a qualification procedure.

Movement of particles during the capture of particle images, especially for smaller particles, the presence of out-of-focus particles and other optical effects can introduce serious error in the segmentation procedure for determining particle sizes. It is therefore required that the whole system be qualified with particles of a reference material under motion.

Operational qualification shall be demonstrated by using a microscopic scale as a certified reference material (see E.3) or by using a (ideally certified) spherical reference material as described in E.3 following the tests described in 7.2.

Performance qualification shall be demonstrated using spherical or non-spherical reference material as described in E.3 and E.4 following the tests described in 7.4.

5.5 Image enhancement algorithms

Modern image analysers usually have algorithms to enhance the quality of the image prior to analysis. It is acceptable to use enhancement algorithms provided that the measured results agree with the system qualification requirements.

5.6 Measurements

5.6.1 Particle size and shape

It is recommended that the primary measurands used to determine the particle sizes are:

a) the projected area of each particle in pixels (A_i),

b) the projected area of each particle is converted to the area equivalent circular diameter, $x_{A,i}$, $x_{A,i} = \sqrt{\frac{4A_i}{\pi}}$,

c) a long linear dimension of each particle in pixels (e.g. the maximum Feret diameter, $x_{Fmax,i}$),

d) a short linear dimension of each particle in pixels (e.g. the minimum Feret diameter, $x_{Fmin,i}$), and

e) the aspect ratio is defined as, $x_{Fmin,i}/x_{Fmax,i}$.

The Feret diameters are used as an example of linear direct size parameters with large discrimination. Other diameters can also be used as particle sizes under the definition. The measurement of the perimeter of a particle is heavily dependent on the image analysis system used, on the digital resolution and on the achievable sharpness of the images. Thus, it is not recommended to use a parameter based on the perimeter for qualification procedures.

5.6.2 Pixel to length conversion

The digital images shall be composed by a rectangular grid of pixel elements. A square grid of pixel elements is recommended (see 4.6.1). Other patterns like hexagonal and octagonal pixel grids are not supported by this document.

All particles measured should be sized and stored with a resolution of one pixel.

The equipment shall be calibrated to convert pixels into SI length units according to ISO 13322-1 and the result of the particle sizes shall be reported in SI units.

5.6.3 Size class limits

When the particle size distribution is analysed, the particle sizes are counted in size classes. The required size class limits depend on sensor resolution, measuring range (magnification) and the desired precision with respect to the width of the sample's size distribution.

Basically, size results are in pixel area or length resolution. Hence, size classes narrower than a single pixel do not provide a meaningful analysis for particles with low pixel counts.

The number of particles per class should be as high as the desired maximum statistical error requires. (see ISO 13322-1). Unless the number of measured particles cannot be increased, it is sometimes required to reduce the size class resolution, i.e. to combine size classes, in order to decrease the statistical error to an acceptable level.

The evaluation software shall ensure that all potentially measured particles sizes be covered by the selected set of class limits. Otherwise, the resulting loss of information introduces preventable errors to the reported size distributions.

6 Sample preparation

6.1 Sample splitting and reduction

If only a small amount of material is needed to prepare a test sample, this should be sub-divided from the whole sample in a manner that ensures that the test sample is representative of the whole as specified in ISO 14488.

6.2 Touching particles

The number of particles in the dispersion medium should be controlled so that overlapping images of particles are minimised (see ISO 13322-1).

Separation methods are common and useful to separate touching particles. Only for convex particle shapes a separation or rejection of touching and overlapping particles may be allowed within a qualification procedure.

6.3 Number of particles to be counted

Considerable care shall be exercised in order to ensure that the analysis is representative of the bulk sample and contains a minimum number of particles to meet a specific standard error of the required quantity of interest as described in ISO 14488. This can be demonstrated by splitting the bulk sample into at least three test samples. Each test sample should contain sufficient particle numbers for a full measurement. Statistical analysis of the data reveals the repeatability of the method, including sampling and dispersion.

The sub splitting into test samples can also be done by virtually splitting the image data of the full bulk sample measurement into sub measurements. With the availability of the full image data, the confidence interval of any statistical quantity of interest can also be estimated by using a bootstrap method[5].

Characteristic size values below 5 % and above 95 % of the cumulative undersize distribution are likely to be vulnerable to additional uncertainty and systematic error, as a result of sampling problems.

7 Accuracy and instrument qualification

7.1 General

Before commencing any of the tests specified in Clause 7, the operator should consider the protocol of measurement, the operating manual of the instrument and be guided by the recommendations of Clause 5. In general, the tests may be applied to both liquid and dry dispersions.

The functionality of the unit shall have passed the supplying manufacturer's operational qualification (OQ) test or equivalent, with the date and result of the test recorded. The analysis mode, if selectable, shall be suitable for this class of measurement.

Instrument qualification shall be demonstrated by using particulate reference materials which fulfil the requirements described in E.2.

The result presentation software shall be set to produce an output of the cumulative undersize distribution in accordance with ISO 9276-1. The cumulative undersize distribution Q_r shall be obtained as a distribution either by number $(r=0)$, area $(r=2)$ or volume $(r=3)$. The appearance of the distribution can be significantly altered by using different quantities (see Annex B).

If the particle size distribution of the material is monomodal, the following particle size parameters, shall be used for the comparison: $x_{10,r}$, $x_{50,r}$, $x_{90,r}$. For a multimodal distribution, values of the cumulative distribution, $Q_r(x)$ at selected particle sizes x shall be used.

For the comparison of the measurement result with the reference values of the material the measurement uncertainties, u_m, shall be known for each value. Several approximations exist to estimate measurement uncertainties.

a) The within-laboratory reproducibility standard deviation (intermediate precision) as determined from, for example, quality control charts (see ISO 7870-2) may be used as a (rough) estimation of u_m.

b) A reproducibility standard deviation from other sources (e.g. an interlaboratory comparison or provided by the manufacturer of the instrument) may be used.

c) The standard deviation of the measurements as obtained in 7.3.2 may be used as a very rough estimation. This estimation is typically underestimating the real uncertainty, especially if only a single instrument is involved in the determination of u_m.

7.2 Trueness

7.2.1 General

An instrument demonstrates trueness if the results obtained using CRMs, which fulfil the requirements described in E.3, are within acceptable limits. For the qualification test on trueness, spherical particulate CRMs should be used. This ensures that the instrument is correctly functioning as an analytical platform without a significant systematic bias.

It shall be ensured that the values of the particle size parameters and the associated uncertainties of the material have been determined for the same quantity type used for the undersize distribution measured at the instrument.

At the moment, assigned values for a commercially available certified reference materials can only exist for a particle size distribution by volume, but tests based on number or area weighted distributions are also allowed.

7.2.2 Qualification test

The test protocol of the CRM shall be followed during the measurement. At least three separate test samples shall be measured, more than three separate test samples are preferred. Each test shall be conducted at an adequate sample concentration and number of measurement frames to allow a sufficient number of single particles to be analysed (see ISO 14488).

7.2.3 Qualification acceptance

The qualification test shall be accepted as fulfilling the requirements of this document if the resulting measured particle size distribution achieves the following criteria.

a) The number of accepted particles that are measured exceeds the number required for the desired accuracy within the specified confidence limits (see ISO 14488).

b) The number of touching particles is below specified limits; using spherical CRMs, their influence on the result may be reduced with shape descriptors as filters or by software-based separation (see 6.2).

c) The average cumulative distribution value from the three tests or five tests, at the required percentile or quantile shall be calculated and compared to the corresponding certified value of the reference material.

The total value of the uncertainty associated with each parameter which is used as the final acceptance/rejection limits, shall be calculated using the following formula (see E.5 for an example):

$$U_{\text{lim}} = \pm\, k \cdot \sqrt{{u_m}^2 + {u_{\text{CRM}}}^2}$$

The value for the coverage factor k is usually a number between two and three and shall be chosen on the basis of the desired level of confidence. The uncertainties, u_{CRM}, of the assigned values of the reference material are provided on the certificate of the reference material.

In the event of a failure of this test, then all aspects of the measurement method adopted by the operator and of that of the instrument should be examined extensively.

7.3 Repeatability

7.3.1 General

For the wet dispersion method, this test is carried out using one instrument, a single operator and the same dispersed sample within a short time interval that avoids segregation and enables stable conditions.

For the dry dispersion method, different aliquots of the same sample shall be used. This type of repeatability (method repeatability) includes additional variability due to sampling and dispersion.

Any material being either spherical or non-spherical may be employed for this investigation. The material shall follow the requirements described in E.2 with the exception that assigned (certified) values are not required.

7.3.2 Repeatability test

Perform at least six consecutive measurements with the same dispersed test sample (repeatability) or with different aliquots of the same sample (method repeatability) at an adequate sample concentration and measurement period to allow a sufficient number of particles to be analysed (see ISO 14488). The measurements should be conducted within a short period to ensure sample stability.

The standard deviation obtained is then compared with a target set by the user. The limits for this test should be chosen high enough in order not to disqualify an instrument based on an insufficient number of replicates[2].

EXAMPLE 1 The 95 % confidence interval for a standard deviation σ of six results is $0{,}6\ \sigma_s < \sigma < 2{,}5\ \sigma_s$, where σ_s is the standard deviation of the test samples.

EXAMPLE 2 The standard deviation of the results of a method repeatability test using different sample aliquots of the same sample can be used as an estimation for the measurement uncertainties, u_m, required in 7.2.3 and 7.4.2. In this case, it is recommended to perform 20 measurements to 30 measurements to obtain a statistically significant result for the uncertainties.

7.4 Intermediate precision

7.4.1 General

The performance of a dynamic image analysis system should also be qualified using more realistic materials, like non-spherical particles. Irregular particles are prone to flow orientation and the conversion of the projected area to a size or to a size distribution is more dependent on the method employed when compared with spherical particles. This material is, therefore, more sensitive to changes in particle motion and orientation. Such a RM is very often provided by the manufacturer of the instrument and has been characterized on several dynamic image analysis instruments of the same kind and set-up using the same prescribed method. It is allowed to create an in-house reference material for this type of test.

This material may then be used to qualify the performance of instruments of the same kind following the same procedure as described in 7.2.2.

7.4.2 Qualification acceptance

The qualification test shall be accepted if the resulting measured particle size distribution achieves the following criteria.

a) The number of accepted particles that are measured exceeds the number required for the desired accuracy within the specified confidence limits (see ISO 14488).

b) The number of touching particles is below specified limits; if using spherical RMs, their influence on the result may be reduced by using shape descriptors as filters.

c) The average cumulative distribution value from the three tests or five tests, at the required percentile or quantile shall be calculated and compared to the corresponding certified or characterized value of the reference material.

The total value of the uncertainty associated with each parameter shall be calculated by using the following formula:

$$U_{\text{lim}} = \pm\, k \cdot \sqrt{{u_m}^2 + {u_{\text{RM}}}^2}$$

The value for the coverage factor k is usually a number between two and three and shall be chosen on the basis of the desired level of confidence. The uncertainty, u_{RM}, of the characterized value of the reference material shall be provided by the reference material producer. If only a single instrument has been used to characterize the RM, the RM can be valid for this instrument only and the uncertainties, u_{RM}, are dependent on the number of measurements to characterize the assigned value. In this case, k shall be set to three. Measurement results within the determined limits indicate that an instrument is in statistical control.

NOTE If the assigned value is based on more than nine results, the uncertainty of the assigned value is small compared to the measurement uncertainty.

8 Test report

8.1 General

The results shall be reported in accordance with ISO/IEC 17025, ISO 9276-1, ISO 9276-2, and ISO 9276-6. Moreover, the information listed in this clause should be available in the form of a written protocol or reported so that the measurements can be readily repeated by different operators in different laboratories.

For each result, the following information given in 8.2 to 8.5 should be available either in the test report itself or in laboratory records.

8.2 Sample

a) complete sample identification, such as name, chemical type, batch number and/or location, date and time of sampling,

b) sampling procedure, i.e. sampling method and sample splitting procedure,

c) sample pre-treatment (optional), e.g. pre-sieving, type and conditions,

d) amount of sample, and

e) date of analysis.

8.3 Dispersion

For dry dispersion:

a) specific details of dispersing device, e.g. diameter of the delivery tube, primary pressure,

b) type of dosing/feeding device,

c) dosing rate,

d) dispersion pressure, and

e) outlet width/sampling volume depth.

For wet dispersion:

f) dispersion liquid: identification, volume and, if necessary, temperature,

g) dispersant(s): type and concentration,

h) sonication: type of unit, frequency (energy), duration and pause before starting measurement,

i) pump speed, and

j) sampling volume depth of the flow-cell.

8.4 Image analysis instrument

a) Instrument type and serial number,

b) software version,

c) segmentation method and threshold applied,

d) volume of dispersion unit,

e) optical arrangement applied (measuring range, optical module, magnification)

f) date and time of the last alignment,

g) date of last qualification test,

h) date and time of measurement,

i) frame coverage,

j) trigger thresholds for start/stop conditions (if applied), and

k) threshold for acquisition of valid data (if applied).

8.5 Analyst identification

a) Name and address of laboratory, and

b) operator's name or initials.

Annex A
(informative)

Theoretical background

A.1 Object and image planes

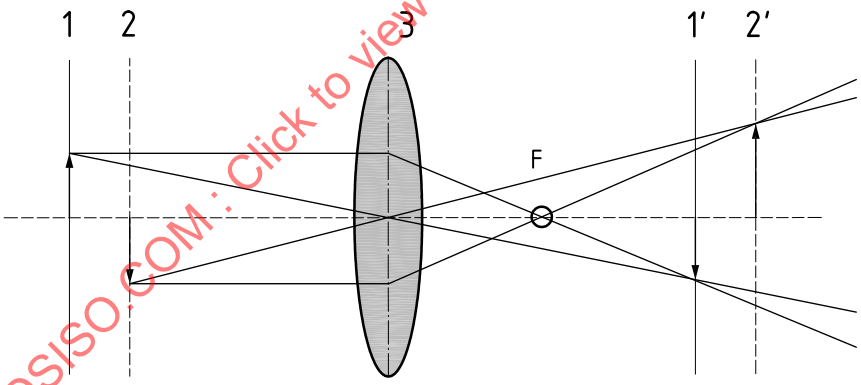
The optical image of an object is created by means of an optical system that combines light emanating from the object point to an image point. An image is the superposition of all the individual image points within the image plane which represent all object points of a given object plane. The geometrical image construction at a simple lens considers two special rays[3].

a) Any ray that enters parallel to the axis on one side of the lens proceeds towards the focal point F on the other side.

b) Any ray that passes through the centre of the lens does not change its direction.

Only objects located at a single object plane are in focus on the same image plane. Other objects are out of focus in that plane and are focused on a different plane. If a flat image sensor is located at a single plane in image space, only objects at the corresponding object plane will be in focus on that sensor.

At a single lens the magnification depends on object position and thus leads to a perspective image. This also applies to entocentric (non-telecentric) imaging lenses (see Figure A.1).



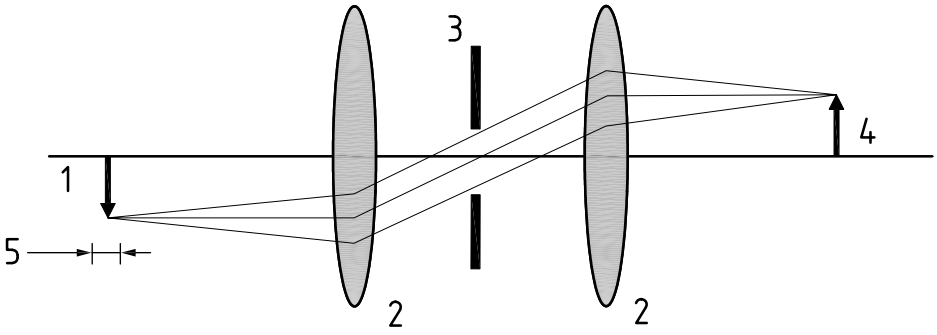
Key

1	object plane of image plane 1'	1'	image plane of object plane 1
2	object plane of image plane 2'	2'	image plane of object plane 2
F	focal point	3	lens

Figure A.1 — Construction of an image at a single lens

A.2 Object and image space telecentric lenses

Telecentric lenses are special optical lenses to capture objects without perspective distortion. At a telecentric lens, all of the collected principal rays in the object space are parallel to the optical axis. Therefore, the front lens has to be at least as large as the object to be imaged.



Key

1	object	4	image
2	lens	5	acceptable depth of field
3	aperture stop		

Figure A.2 — Construction of an image within a bilateral telecentric lens

Two types of telecentric lenses are appropriate for dynamic image analysis systems for particle size measurement. Within the acceptable depth of field, the object (particle) remains at constant magnification.

a) In an object-sided telecentric lens, a small change in the distance from the object to the lens does not change the magnification of the resulting image. However, a small change of the (generally fixed) camera position can change the magnification.

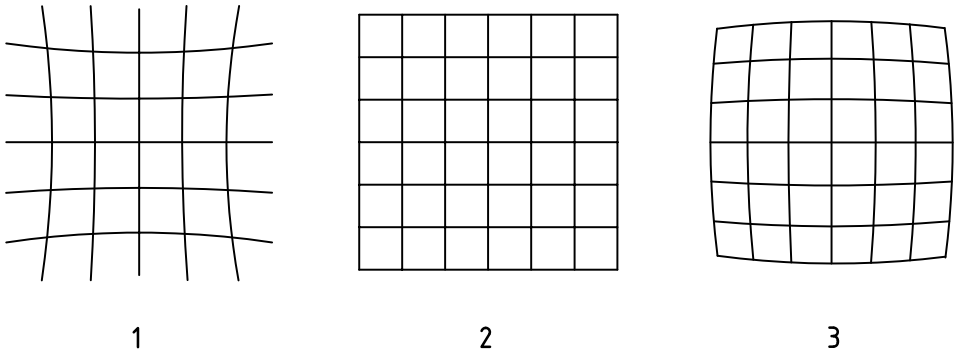
b) In a bilateral telecentric lens (see Figure A.2), the magnification is independent of the object and of camera position.

The relationship between object and image plane also applies to telecentric lenses. With a fixed image sensor, a telecentric lens focuses at only one working distance to the object.

A.3 Lens aberrations and distortions

Image aberrations of lenses occur when the different light beams emanating from the object point are not all focused in one image point. The most common aberrations are because of the spherical surface of lenses and because of chromatic shifts of the focal length. Aberrations introduce additional image blur.

The distortion is a geometric imaging error of optical systems which leads to a local change in the imaging scale. The scale change is based on a change in the magnification with an increasing distance of the image point from the optical axis. Distortion can be quantified during system calibration and corrected by software.



Key

1 pincushion distortion
2 no distortion
3 barrel distortion

Figure A.3 — Images showing pincushion and barrel distortion

A.4 Optical resolution

A.4.1 Optical resolution

Optical resolution describes the ability of an imaging system to resolve detail in the object that is being imaged. The pixel resolution of the image sensor should be adapted to the optical resolution of the system. A pixel resolution higher than the optical resolution does not provide additional information about the object being imaged, but it can be beneficial to use two times to three times higher digital resolution than the optical resolution to evaluate the image sharpness for segmentation and to describe the particle shape. The digital resolution should not be used to describe the measurement range of the instrument in case it is higher than the optical resolution.

A.4.2 Diffraction limit

Geometrical optics, or ray optics as shown in Figure A.1, describes light propagation in terms of rays. This is a simplification of light propagation. Geometrical optics does not account for optical effects such as diffraction and interference[4]. There is a principal limit to the resolution of any optical system. For microscopic instruments, the diffraction-limited spatial resolution is proportional to the light wavelength, and to the numerical aperture of either the objective or the object illumination source, whichever is smaller.

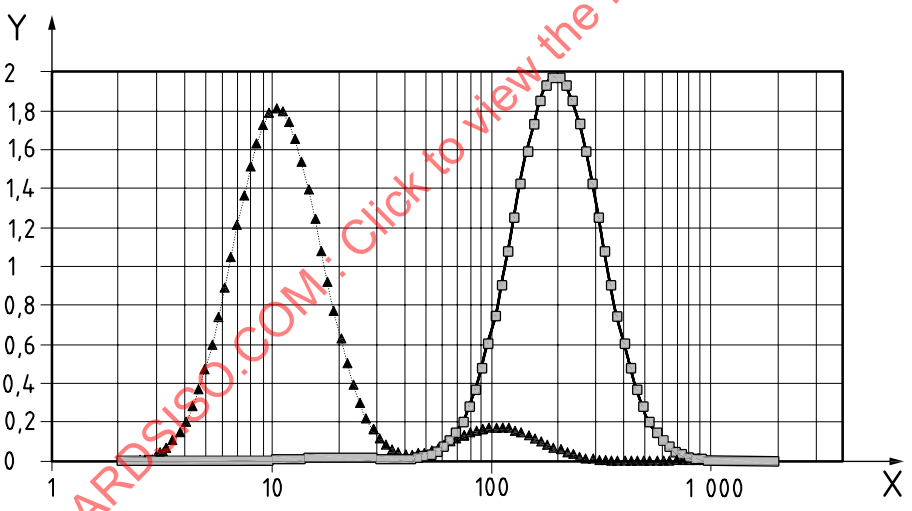
Annex B
(informative)

Comparison between particle size distributions by number and by volume

Various types of quantities can be used for the representation of a particle size distribution. If individual particles of similar size are counted, the quantity type is the number. If the mass of all particles of similar size is weighed, the quantity type is the mass or the volume. In the graphical representation of a particle size distribution, the equivalent diameter is plotted on the abscissa and the measurement quantity is plotted on the ordinate.

When interpreting or comparing particle size distributions, it should be respected that the appearance of the distribution can be significantly altered by applying different equivalent diameters or different quantities.

For the following example, a theoretical bimodal mass distribution was composed of two logarithmic normal distributions centred about 20 µm and 200 µm. The mass of the 20 µm component was chosen to contribute only 1 % to the full mass of the distribution. This size distribution was calculated into a size distribution by number. Since the mass of a spherical particle having a diameter of 200 µm is 1 000 times higher than the mass of a 20 µm particle, the appearance of the distributions differs considerably.



Key

X particle size (µm)

Y distribution density q^*

distribution density by number $q_0{}^*$

distribution density by volume $q_3{}^*$

Figure B.1 — Comparison of distribution by number versus by volume

Annex C
(informative)

Recommended particle velocity and exposure time

Special precautions are required when measuring small particles in motion by dynamic image analysis.

When a spherical particle of diameter x [pixel] moves at a velocity v [pixel/s] and the exposure time is t [s], the centre of the area of the particle moves the distance a [pixel] during this period, i.e.

$$a = v \times t$$

The observed diameter of the particle b [pixel] in the direction of motion is between $(x + a)$ and $(x - a)$, depending on the threshold level used (see Figure C.1).

Consequently, when the image of a moving spherical particle is captured as a grey image and then converted into a binary image with a given threshold level, the shape appears to be a prolonged ellipsoid rather than circular. The maximum dimension of the binary particle image is then:

$$b = x + a$$

In order to make the results of dynamic particle measurement consistent with those obtained by static particle measurement, it is recommended that the difference between x and b be less than 0,5 pixel, i.e.

$$a = v \times t < 0{,}5$$

However, if the measurement is performed only with large particles (e.g. x is larger than 10 pixel, with a given error in the measured area equivalent diameter), the difference between x and b (which is equal to a) can be calculated as follows:

$$x_{A,\mathrm{real}} = \sqrt{\frac{4 \times A_{\mathrm{real}}}{\pi}} = x$$

$$A_{\mathrm{real}} = \frac{\pi}{4} \times x^2$$

$$x_{A,\mathrm{meas}} = \sqrt{\frac{4 \times A_{\mathrm{meas}}}{\pi}}$$

$$A_{\mathrm{meas}} = \frac{\pi}{4} \times x \times b$$

where

$x_{A,\mathrm{real}}$ is the area equivalent diameter of a static particle;

$x_{A,\mathrm{meas}}$ is the area equivalent diameter of the measured particle;

A_{real} is the projected area of the static spherical particle whose shape has been approximated by an ellipsoid;

A_{meas} is the projected area of the measured particle whose shape has been approximated by an ellipsoid.

The ratio of the measured particle diameter to the static particle diameter ε is given by:

$$\varepsilon = \frac{x_{A,\text{meas}}}{x_{A,\text{real}}} = \sqrt{\frac{b}{x}} = \sqrt{\frac{x+a}{x}} = \sqrt{1+\frac{a}{x}}$$

This formula can also be expressed as follows:

$$a = x\left(\varepsilon^2 - 1\right)$$

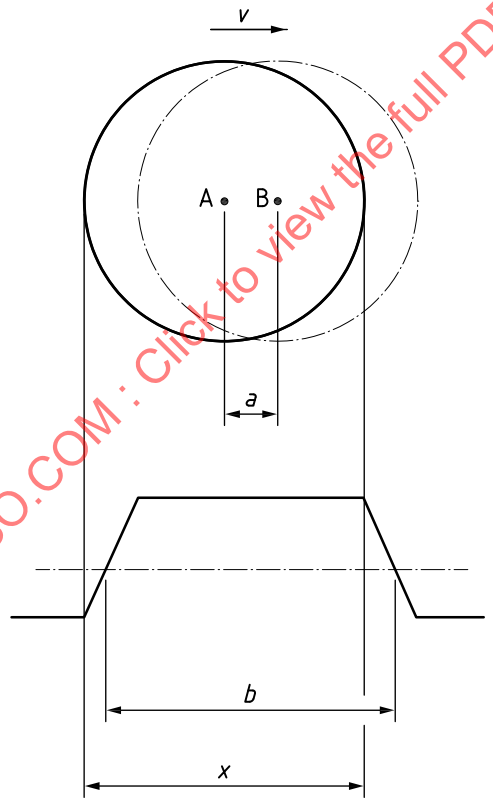
For instance, when ε is less than 1,1 (which corresponds to a relative error of 10 % in particle diameter),

$$\frac{a}{x} < 0,21$$

Therefore, a can be as large as 2 pixels when the minimum particle size measured is 10 pixels, i.e.

$$a = v \times t < 2,1$$

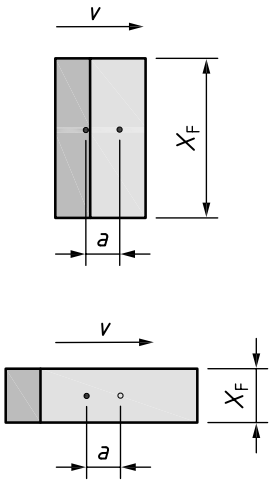
Figure C.1 illustrates particle image and threshold level, and Figure C.2 illustrates an extension for particles of arbitrary shape.



Key

a	travelled distance during the exposure time [pixel]	A	particle position at start of image capturing
b	measured diameter of binary-imaged particle, including effects from motion blur [pixel]	B	particle position at end of image capturing
v	direction of motion and velocity [pixel/s]	_·_·_·_	threshold line
x	diameter of static particle [pixel]	———	grey level

Figure C.1 — Particle image and threshold level



Key

	projected area of the static particle	x_F	Feret diameter of projected area perpendicular to the direction of motion
	maximum error caused by the particle movement	A	particle position at start of image capturing
a	travelled distance during the exposure time [pixel]	B	particle position at end of image capturing
v	direction of motion and velocity [pixel/s]		

Figure C.2 — Extension of Figure C.1 for particles of arbitrary shape

In Figure C.2, the following formula can be used:

$$A_{\text{err}} = a \times x_F$$

where x_F depends on the particle orientation relative to the moving direction.

$$\varepsilon = \frac{x_{A,\text{meas}}}{x_{A,\text{real}}} = \sqrt{\frac{A_{\text{real}} + A_{\text{err}}}{A_{\text{real}}}} = \sqrt{1 + \frac{4 \times a \times x_F}{\pi \times x_{A,\text{real}}^2}}$$

Annex D
(informative)

Particle diameter dependence on threshold selection

D.1 General

The measured particle size depends on the selection of the threshold value which is used to discriminate between background and foreground pixels of the recorded images. This effect can be demonstrated using simulations and real images of particles.

D.2 Influence of the threshold selection caused by the pixel structure of digital images

In the first simulation, the optical image forming was not considered which can introduce additional image blur. Only perfect circular shapes of the same diameter are placed at random on the pixel grid of a digital image sensor. Within this simulation pixels lying completely within the circular shapes are set to black; pixels lying outside the shape are set to white. At the edges of the circles, the pixels are only partially covered by the circle and grey values are assigned proportionally to the area inside and outside of the particle for each pixel. Thus, even without any optical blur, grey edge pixels are created and a threshold method is needed to distinguish between background and shape. If an 8-bit representation of the grey values is implemented using a value of 0 for black and a value of 255 (100 %) for white, the theoretically correct threshold value equals 127,5 (50 %).

The simulation starts with eight times higher pixel resolution in each direction in order to calculate the partial coverage of pixels. The target nominal diameter of the circles is 30 pixels.

Simulation procedure:

a) 100 circles having a diameter of 240 pixels have been drawn, where the centre of each circle has been randomly placed on the grid with sub pixel resolution.

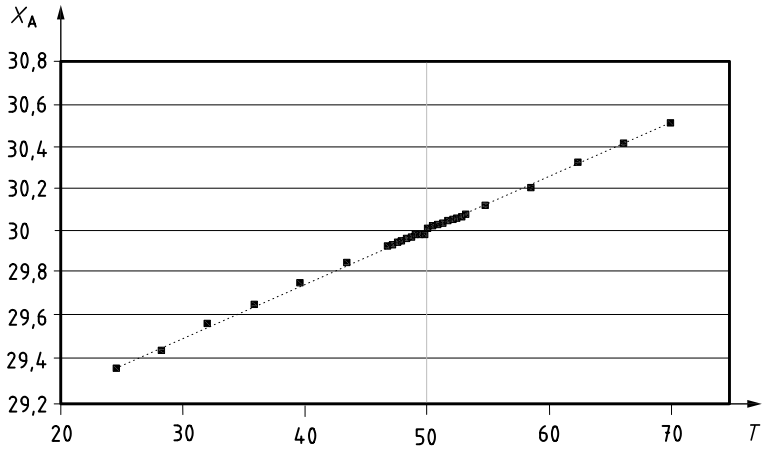
b) If the centre of a pixel was inside the circle, it was considered to be part of the circle and its grey level value was set to 0. All other pixels were set to 255.

c) The images of the circles have been down-sampled by a factor of 8 using a bilinear interpolation method. By using this method, grey levels between 0 and 255 are assigned to pixels at the edge of the circular shape, approximating the partial coverage by the exact circle. This method results in 100 images of circles having a diameter of 30 pixels.

d) A threshold was applied and the images were converted to a binary representation.

e) The area of the circles was measured by counting pixels and averaged over all 100 images. The area equivalent diameter was calculated from the average area.

The results of the simulation are shown in Figure D.1. The trend shows a linear relationship around the theoretically correct threshold level of 50 % where the simulation yields the correct result of 30 pixels. Overall, the expected average error from discretisation is very small for area calculations by counting pixels.



Key

X_A area equivalent diameter [pixel]

T threshold level [%]

Figure D.1 — Detected particle diameter versus threshold selection for a simulated circular shape having a diameter of 30 pixels placed randomly on a digital pixel grid

D.3 Influence of the threshold selection for particles within and outside the depth of field

The influence of the threshold on the particle size determined is significantly greater at real images of particles. This is because the real images of particles are very often not perfectly sharp on the image sensor. Image blur is introduced by the limited optical resolution and by particles being out of the optimum focus position.

In this example, images of two glass spheres of different sizes which have been fixed on a glass slide have been taken at three different focal positions resulting in three images each showing a different level of sharpness.

a) The focal positions have been selected to demonstrate an optimum sharpness, an acceptable sharpness and a very blurred picture where the edges of the particles are no longer recognizable.

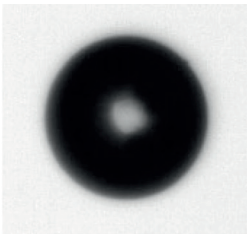
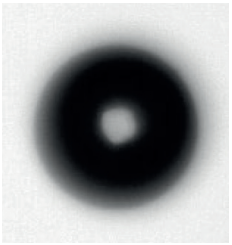
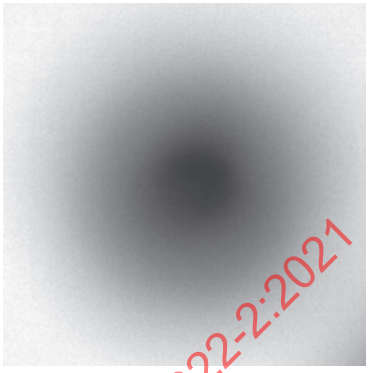
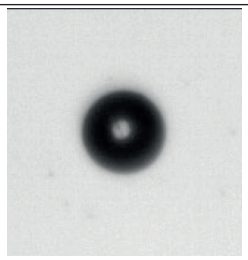
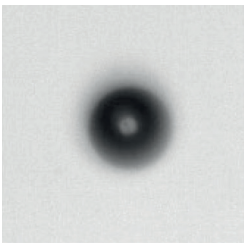
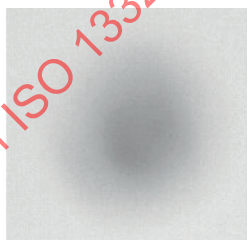
b) Different threshold levels have been applied to each image for segmentation.

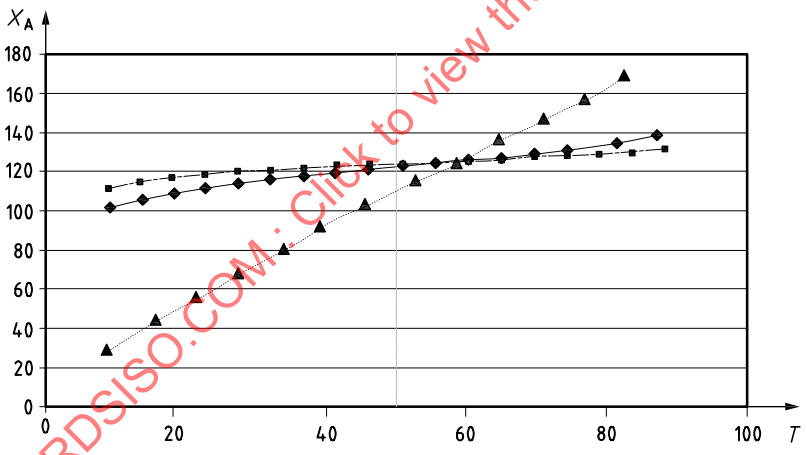
c) The threshold is given as a percentage of the background value.

d) The particle images show a centre hole because of the transparent particle material. This hole has been filled in the binary image prior to area measurement by counting pixels.

e) The spherical equivalent diameter is calculated from the measured area in pixel.

The results are shown in Figure D.2 and D.3.

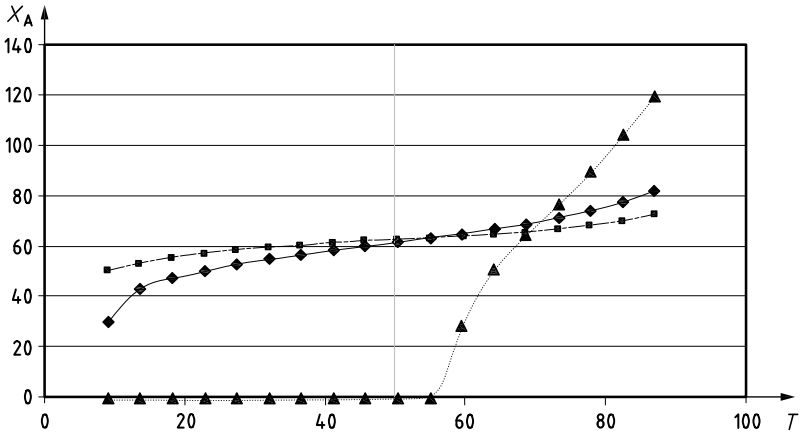
Optimum sharpness at object distance: 64,04 mm	Acceptable sharpness at object distance: 64,18 mm	Blurred image at object distance: 64,48 mm
		
		



Key

X_A	area equivalent diameter [pixel]	◆	acceptable sharpness
T	threshold level [%]	▲	blurred image
■	optimum sharpness		

Figure D.2 — Detected particle diameter versus threshold selection for the coarser particle at different focus positions (object distances)



Key

X_A	area equivalent diameter [pixel]	◆	acceptable sharpness
T	threshold level [%]	▲	blurred image
—■—	optimum sharpness		

Figure D.3 — Detected particle diameter versus threshold selection for the smaller particle at different focus positions (object distances)

The influence of the threshold selection on the measured particle size increases with the blurriness of the particle images.

— The relative error of the measured particle size is greater for the smaller than for the larger particles.

— Even if the edge of a particle cannot be observed in its blurred image, a threshold around the 50 % level can still provide an acceptable result.

— With increasing distance to the focal position, the smaller particles disappear first in the binary image. The depth of the measurement zone which is defined by the acceptable depth of field is smaller for the smaller particles than for larger particles.

The effective measurement volume depends on particle size if the particle positions are not controlled to be contained completely within the acceptable depth of field of the smallest particles measured. In this case, corrections need to be applied to the measured particle size distribution.

Annex E
(normative)

Requirements for reference material

E.1 Calibration standards

Certified reference material shall be used for calibration.

The conversions from pixel of the instrument to micrometers shall be calibrated by a stage micrometer or a calibration slide (see 5.2.3.1).

NOTE According to the definition of certified reference material, a certified calibration standard like a stage micrometer is a certified reference material.

A reference stage graticule having arrays of known objects of different sizes shall be used to calibrate the threshold level of the instrument. Alternatively, spherical monodisperse particles shall be used to test the threshold calibration of the instrument.

CAUTION — The use of a graticule or a particulate reference material having different optical properties to that of the material under test especially when the particles are suspended in a liquid can lead to substantial bias in the reported particle size when this reference is used to establish a threshold level.

Systems with a wider depth of measurement zone than the acceptable depth of field shall include a calibration for the correct particle representation with multi-modal-particle standards (see ISO/TS 14411-1) or by scanning through the sample volume depth with reference objects of different certified sizes (see 5.2.3.4).

E.2 General requirements for particulate reference materials suitable for image analysis

Reference material particles for use in particle size measurements by dynamic image analysis may be spherical or non-spherical, certified or non-certified depending upon the application. In addition to being produced in compliance with the general requirements for reference materials (see ISO 17034) they shall comply with the following properties.

a) Aspect ratio, shape, size distribution width: the certified reference material shall be suitable for the image analysis technique and consist of a known distribution having a range of particles with an $x_{90,r} / x_{10,r}$ ratio of 1,5 to 10. Additional uncertainties introduced by possible flow orientation and segregation problems, with particles having a low aspect ratio <0,5 or a very wide size distribution >20:1, shall be avoided during the selection of reference materials suitable for dynamic image analysis.

b) Optical properties, refractive index: the optical homogeneity of the material shall be as uniform as possible. The optical appearance of a real particle depends on the refractive index of the particle, the refractive index of the surrounding medium, the surface structure, and the type of illumination.

c) Apparent density (wet application): the apparent density of the material shall exceed the density of the dispersing liquid for the particles not to float in wet applications. Furthermore, the apparent density should not be too high for avoiding sedimentation effects. Therefore, a value within the range from 1 000 kg/m^3 to 2 500 kg/m^3 seems to be optimal for aqueous applications. Particles of higher densities can be used if a liquid with higher density or viscosity is used.

d) Stability (chemical, mechanical, long term): for wet application, the particles shall have a high chemical stability and be non-soluble in dispersant media. The particles should not be disrupted by ultrasound pressure in dispersant media. For dry application, the mechanical strength shall be as high as possible since the material should be able to withstand a typical dry dispersion procedure without breakage. The material should provide a shelf life of at least two years after production without measurably changing its physical properties.

e) Dispersability, swelling behaviour (wet application): the material shall be easily dispersible in the chosen liquid. No particle agglomerates or flocculation should be detectable after dispersion. It is allowed to support the particle dispersion using dispersing agents or ultrasound. The swelling of the material suspended in pure dispersant media should be as low as possible and shall be specified in the sample preparation procedure.

f) Dispersability (dry application): the particles should not agglomerate under normal environmental conditions. Their electrostatic behaviour should not cause any significant deposition on a feeding mechanism.

g) Amount, sampling, sub-sampling: sampling particles for the reference material shall be carried out from a significant stocked amount to guarantee a prolonged period of application. The sample division shall be carried out by use of a suitable method (see ISO 14488). The material should not cause major problems during sub-sampling, if required. It is essential that a robust procedure is available that fully describes the sub-sampling and sample preparation.

h) Documentation, protocol: the chosen reference material should possess sufficient and robust, written sampling / dispersion / measurement protocols, suitable for dynamic image analysis.

i) Required property values: if the particle size distribution of the material is monomodal, the following particle size parameters are required: $x_{10,r}$, $x_{50,r}$, $x_{90,r}$. For a multimodal distribution, values of the cumulative distribution, $Q_r(x)$, at selected particle sizes x are required. The standard uncertainty u_{CRM} or the expanded uncertainty $k^* u_{\mathrm{CRM}}$ of each particle size parameter shall be quoted. Additional property values may be added for better relation to product properties.

E.3 Selection of (certified) reference materials

Certified reference materials, for example, particles with assigned particle size parameters that have been characterized by a metrologically valid procedure for image analysis, shall be used for the determination of accuracy and trueness of any dynamic image analysis unit.

Reference materials may be used for repeatability tests (see 7.3) which do not require assigned particle size parameters and also for performance qualification (PQ) under intermediate precision conditions (see 7.4), provided that values for the particle size parameters have been quoted with corresponding values for the expanded uncertainties. These values coming from image analysis in one or more instrument types can be certified according to ISO Guide 35 or can be 'in-house' characterized by a metrologically valid procedure.

E.4 Selection and characterization of spherical and non-spherical reference materials

Particle size determination by dynamic image analysis, in common with other techniques, describes the size of three-dimensional objects with a single value of length, the 'area equivalent diameter' (see ISO 13322-1). It is for this reason that the equivalent spherical diameter reported for non-spherical particles depends upon the technique employed. The sphere is the only shape whose properties can be fully defined by a single dimension of length, without ambiguity.

The dynamic image analysis results of non-spherical materials can differ from those coming from other particle sizing techniques. Therefore, care shall be taken that the certified or characterized values of non-spherical reference materials also apply for image analysis, i.e. were obtained by dynamic image analysis units, known to be in conformance with 7.2.

In case this material is applied to other dynamic image analysis units of different designs using different dispersing methods creating a change in particle orientation, these differences can then lead to a systematic deviation (bias) between the measured result and the assigned values of the material. Such a bias should be carefully examined when selecting non-spherical reference materials.

E.5 Calculation of the acceptance limits (informative example)

In this example a fictional certified reference material has been used to demonstrate the calculations required for the instrument qualification as it is described in 7.2. The certificate of this material provides values for certified diameters versus mass fractions as well as certified mass fractions versus diameters[5]. Thus, the instrument's software was set-up to provide mass-based diameters.

The $x_{90,r}$ / $x_{10,r}$ ratio is about 2,38. The distribution density is monomodal and sufficiently narrow and therefore the qualification check should be based on the values of the diameters $x_{10,3}$, $x_{50,3}$, $x_{90,3}$.

In this example the certified particle diameter value at the mass fraction of 50 % is 78,4 µm. The corresponding uncertainty of this value is 1,5 µm. This value is the standard uncertainty u_{CRM} and not an expanded uncertainty.

When the uncertainty provided in the certificate is an expanded uncertainty, it shall be divided by the coverage factor to obtain the standard uncertainty required for the calculation. A 95 % level of confidence usually implies a coverage factor of $k = 2$.

For comparison of the values, the measurement uncertainties, u_m, are required for this type of measurement. In this example, a reproducibility standard deviation from an interlaboratory comparison is not available. Therefore, the qualification check requires a minimum of five samples to be measured in order to estimate the uncertainty of measurement for this particular instrument. The recommended procedure is to use a microriffler to divide the 43 g sample into sub-samples until a suitable sub-sample mass is obtained. Refer to ISO 14488 for guidance on the minimum recommended sample size.

The laboratory measurements gave an average of 76,2 µm ± 0,4 µm (as a single standard deviation of six measurements). The standard deviation is divided by the square root of the number of measurements, as the average of the results is compared with the certified value. The measurement uncertainty u_m for the average $x_{50,3}$ value is therefore estimated as $0,4\ \mu\mathrm{m} / \sqrt{6} = 0,16\ \mu\mathrm{m} < 0,2\ \mu\mathrm{m}$. This estimation is typically underestimating the real uncertainty, especially if only a single instrument and method are involved. The acceptance limits U_{lim} are then calculated according to 7.2.3:

$$U_{\mathrm{lim}} = \pm\, k \cdot \sqrt{0,2^2 + 1,5^2}\ \mu\mathrm{m} = \pm\ 2 \times 1,51\ \mu\mathrm{m} \approx \pm\ 3,0\ \mu\mathrm{m}$$

The laboratory measurements gave an average of 76,2 µm. This result is within the range of 78,4 µm ± 3,0 µm, so the measured mean value is therefore not significantly different from the certified value.

The same procedure and calculations shall be repeated for the $x_{10,3}$ and $x_{90,3}$ values to pass the qualification test.

Annex F
(informative)

Robustness and ruggedness of the image analysis method

F.1 Robustness

A robust analytical method is one which exhibits insensitivity to changes of the operational parameters. This should be ascertained during the method development and determines the allowable (acceptable) limits for all critical parameters that affect the final result.

F.2 Ruggedness

A rugged analytical method is one which exhibits insensitivity against inadvertent changes of known operational variables. These variables often make themselves known when transferring methods between sites, even if results on a single site were acceptable. Experiments are normally conducted, using well-defined procedures following robustness testing and provide information on a procedure's inter-laboratory transferability.

F.3 Investigation of parameters

F.3.1 General

An ideal time to study the robustness of a method is during its development, but sometimes investigations can occur as part of a troubleshooting exercise.

Each parameter can be classified into defined types. Each of these parameters can also be classed as being either a noise factor, a control factor or an experimental factor.

F.3.2 Noise factors

Noise factors are unintentional variations which if identified as potentially critical can require ruggedness testing to assess their impact. These can be controlled in a broad sense. Small variations can show up as small unavoidable differences in results.

Typically, noise factors occur either from the environment, the material or the instrument electronics and mechanics respectively:

— environment: humidity, temperature, environmental light;

— material: dispersant source, dispersant grade, shape, inhomogeneities;

— instrument: electronic noise, mechanical tolerances.

F.3.3 Control factors

Control factors form a crucial part of the method (such as the sample preparation procedure). They should be well defined (by describing them in detail) by a method.

This kind of factors occurs during all steps and many components of a particle sizing method:

— analyst: pre-dispersion method, sampling method, shaking, weighing method;

— instrument: instrument model, analysis range, cleanliness, extraction, gas supply;

— measurement: alignment of camera and illumination;

— material: sample source (batch), verification materials, gas quality;

— method: optical properties, sample transfer to an instrument, sample preparation.

F.3.4 Experimental factors

Experimental factors are those that should be varied as part of method development to find the most appropriate ones for the procedure in question.

— instrument: analysis settings;

— measurement: time, sample concentration, sonification, pump speed, stirring speed, flow rates, pressure.

F.4 Examples

F.4.1 General

A complete listing of all parameters mentioned in G.3 goes beyond the scope of this annex. Instead, some examples are given.

F.4.2 Noise factor: Material statistics

A few large particles measured within a small sample may have a huge impact on the particle size distribution by mass or volume.

F.4.3 Control factor: Camera electronics correction

All digital cameras to some degree exhibit blemishes which affects the correct reproduction of the optical image. Most apparent are black and white pixels of the sensor (dead pixels or hot pixels). These pixel defects are rarely a major issue unless they extend to too many pixels. They can be removed by taking a flat fielding reference or by post processing using interpolation methods to reduce their effects.

F.4.4 Control factor: Overexposure (illumination)

An overexposure of the images leads to a significantly underestimated particle size or causes other problems.

F.4.5 Control factor: Contamination

Contamination can create artefacts that are interpreted as particles finally falsifying the result. A maximum contamination should be specified.

F.4.6 Control factor: Sampling technique

Improper sampling technique, leading to a non-representative sample in the measurement zone. This type of error is especially significant when using an inadequate sample splitting technique in the case of a large batch of free-flowing material having a wide size distribution.

F.4.7 Experimental factor: Particle concentration

A particle concentration exceeding the limits can cause too many particle overlaps. As a consequence, and without further corrections, the resulting particle size distribution as well as its characteristic value can be shifted to the coarse.