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Particle size analysis — Image analysis methods —

Part 1: Static image analysis methods

Analyse granulométrique — Méthodes par analyse d'images —

Partie 1: Méthodes par analyse d'images statiques

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 24, *Particle characterization including sieving*, Subcommittee SC 4, *Particle characterization*.

This second edition cancels and replaces the first edition (ISO 13322-1:2004), which has been technically revised.

ISO 13322 consists of the following parts, under the general title *Particle size analysis — Image analysis methods*:

— *Part 1: Static image analysis methods*

— *Part 2: Dynamic image analysis methods*

Introduction

The purpose of this part of ISO 13322 is to give guidance when using images for particle size analysis.

Image analysis is a technique that has gained popularity in different applications. The aim of this part of ISO 13322 is to give a standardized description of the technique used and its validation. This part of ISO 13322 does not describe specific instruments and is restricted to those parts of the acquisition of images that are relevant to the accuracy of the particle size analysis.

This part of ISO 13322 includes methods of calibration verification and recommends using a certified standard as a reference scale. However it is sensible to make some measurements on particles under study, or other reference objects, of known size so that the likely systematic uncertainties introduced by the equipment can be assessed.

Errors introduced at all stages of the analysis from sub-division of the sample to generation of the final result add to the total uncertainty of measurements and it is important to obtain estimates for the uncertainty arising from each stage.

Essential operations are identified to ensure that measurements made conform to this part of ISO 13322 and are traceable.

Particle size analysis — Image analysis methods —

Part 1: Static image analysis methods

1 Scope

This part of ISO 13322 is applicable to the analysis of images for the purpose of determining particle size distributions where the velocity of the particles against the axis of the optical system of the imaging device is zero. The particles are appropriately dispersed and fixed in the object plane of the instrument. The field of view may sample the object plane dynamically either by moving the sample support or the camera provided this can be accomplished without any motion effects on the image. Captured images can be analysed subsequently.

This part of ISO 13322 concentrates upon the analysis of digital images created from either light or electron detection systems. It does not address the method of creating the image although the detection settings chosen together with its calibration are important to particle sizing accuracy. This part of ISO 13322 considers only image evaluation methods using complete pixel counts.

Both the type of distribution, (by number or by volume) together with the width of the particle size distribution has a very material influence upon the number of particles to be measured to secure the desired accuracy within the specified confidence limits. An example is shown in Annex A.

Automation of the analysis is possible in order to measure sufficient particle numbers for a required degree of precision.

This part of ISO 13322 does not address the sample preparation. However, the sub sampling, dispersion and presentation of particles to be measured are a vital part of the operational chain of actions necessary to ensure accuracy and precision of any final result.

NOTE Further details about sampling and sample preparation can be found in ISO 14887 and ISO 14488.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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: The calculations of average particle sizes/diameters and moments from particle size distributions*

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

3 Terms and definitions and list of symbols

3.1 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1.1
area equivalent diameter
diameter of a circle having the same area as the projected image of the particle

Note 1 to entry: It is also known as the Heywood diameter or as the equivalent circular diameter.

3.1.2
binary image
digitized image consisting of an array of pixels, each of which has a value of 0 or 1, whose values are normally represented by dark and bright regions on the display screen or by the use of two distinct colours

3.1.3
contrast (of an image)
<particle size analysis> difference between the intensity of the particle image with respect to the background near to the particle

3.1.4
edge detection
methods used to detect transition between objects and background

Note 1 to entry: See *segmentation method* (3.1.13).

3.1.5
Feret diameter
distance between two parallel tangents on opposite sides of the image of a particle

3.1.6
field of view
field which is viewed by the viewing device

Note 1 to entry: The full image frame of a digital imaging device corresponds to its field of view.

SEE: Figure 1.

3.1.7
grey image
image in which multiple grey level values are permitted for each pixel

3.1.8
image analysis
processing and data reduction operation which yields a numerical or logical result from an image

3.1.9
measurement field
field which is composed by the set of all measurement frames

SEE: Figure 1.

3.1.10
measurement frame
selected area from the field of view in which particles are sized and counted for image analysis

SEE: Figure 1.

3.1.11
pixel
picture element
individual sample in a digital image that has been formed by uniform sampling in both the horizontal and vertical directions

3.1.12
raster pattern
scanning order of measurement frames in the total measurement field

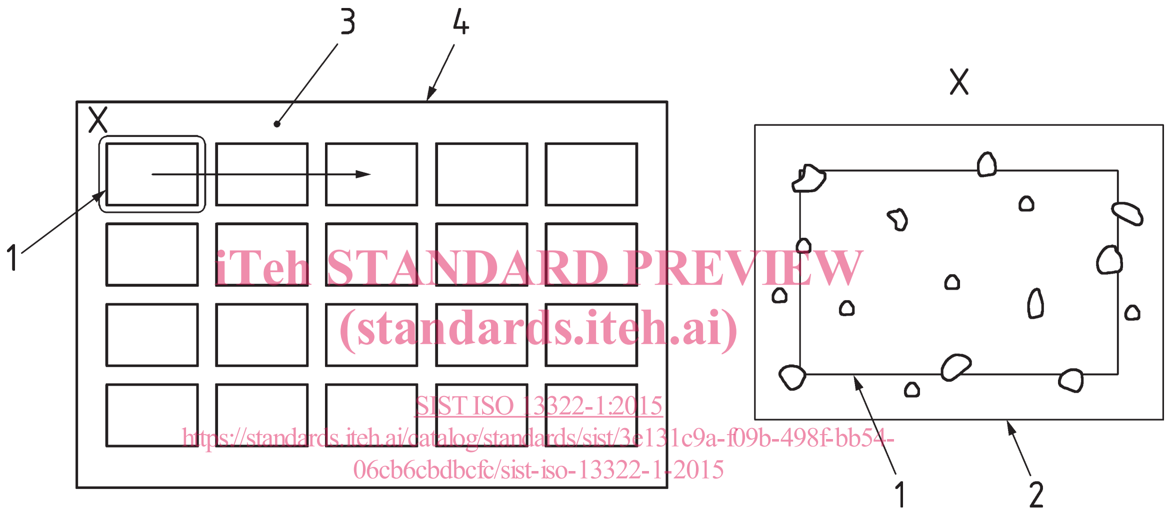
SEE: Figure 1.

3.1.13
segmentation method
strategy employed to separate the objects of interest from their surroundings

Note 1 to entry: Method of dividing the particle image from the background.

Note 2 to entry: See *edge detection* (3.1.4).

3.1.14
threshold
grey level value which is set to discriminate objects of interest from background



Key

1 measurement frame
2 field of view
3 raster pattern of measurement frames
4 measurement field
X enlarged view of a field of view

Figure 1 — Relationship between the terms “field of view”, “measurement frame”, “raster pattern” and “measurement field”

3.2 Symbols

A_i	projected area of particle i
α_1	horizontal calibration factor
α_2	vertical calibration factor
d	minimum feature length
d_c	diameter of a circle
N	number of particles to be measured
n_c	measured number of pixels within a circle
n_j	numbers of particles in size interval Δx_j
P_i	probability that particle i exists in the measuring frame (also called Miles-Lantuéjoul factor)
φ_i	shape descriptor
σ	standard deviation
V_i	volume of particle i
$x_{A,i}$	area equivalent diameter of particle i
x_{F1}	horizontal Feret diameter of object
x_{F2}	vertical Feret diameter of object
x_i	dimension of particle i
$x_{Fmax,i}$	longest dimension of particle i, also called maximum Feret diameter
$x_{Fmin,i}$	shortest dimension of particle i, also called minimum Feret diameter
x_1	horizontal dimension of object
$x_{1,m}$	horizontal dimension of object in SI unit
$x_{1,p}$	horizontal dimension of object in pixel
x_2	vertical dimension of object
$x_{2,m}$	vertical dimension of object in SI unit
$x_{2,p}$	vertical dimension of object in pixel
$x_{10,3}$	particle size corresponding to 10 % of the cumulative undersize distribution by volume
$x_{90,3}$	particle size corresponding to 90 % of the cumulative undersize distribution by volume
Z_1	horizontal side length of the rectangular measurement frame
Z_2	vertical side length of the rectangular measurement frame

4 Preparation for image capture

4.1 Introduction

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.

The final settings and calibration of the image capture device need to be established via an iterative approach. The size range of the particles within an unknown test sample has an influence upon the settings required within the image capturing device. These remain unknown until the first image has been taken, the result observed and the necessary adjustments to the image capture device to achieve the desired accuracy of particle size measurement required. A fully trained operator shall conduct the assessment.

The imaging instrument should be set up and operated in accordance with the manufacturer's recommendations considering the conditions prevailing.

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 magnification should be 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 number of pixels for the smallest dimension of a particle is relevant for cases where linear dimensions or combinations thereof are measured.

Distortion in the image might arise from a number of causes, but its presence and effect on the image may be determined by selecting known sized particles or other reference objects of similar optical properties at a number of points and orientations in the field of view. It is important to note that the measurements made provide only two-dimensional, X and Y, information.

4.2 Procedures

The operator should decide why the result of the image analysis is required. Is a size distribution by the number of particles in each size class required or is the volume of particles in each size class the requirement? What accuracy and precision is required for the final result? These decisions will have a significant influence upon the choice of settings and the method employed in conducting the measurement.

For each material to be analysed and for each instrument employed the person conducting the analysis shall ensure that the following procedures are followed.

a) Ensure an adequate calibration for both the X and Y-axis of the measurement frame exists for the imaging instrument being employed, preferably by using a certified graticule or equivalent reference of equal standing.

b) Ensure that the optical magnification employed is suitable and such as the image of the smallest particle to be measured covers a sufficient number of pixels to support the required accuracy of measurement.

c) Ensure that the illumination method and setting of any focus is correctly established to give a good contrast and uniformity of illumination of any image gathered.

d) Ensure that the number of particles within the measurement frame is such as to minimize the number of touching particles.

e) Ensure that a sufficient number of images of separate aliquot samples are gathered to provide a suitable total number of particles with respect to the type of distribution, number or volume based, and the width of the particle size distribution (see ISO 14488) and that they contain an adequate statistical number of the largest particle of the target material (see Annex A).

f) Some implementations of the image analysis technique employ a large area X, Y servo or manually controlled sample slide assembly. Such large slides enable many measurement frames of the particles deposited to be examined. Should the method of fully separate measurement frames be employed then any frame overlap shall be avoided. If the method of overlapping measurement frames, or other methods of analysing particles that interact with the measurement frame edge is used, then procedures shall be employed to ensure that each particle is only included into the total count for the appropriate size class, once. For more information, see 8.3.

g) If appropriate, ensure that the image quality consisting of illumination, focus and magnification has not changed at the end of the measurement. The requirement for this step depends on the variability of the instrument employed.

h) For the case when image analysis is to be used for certified reference material measurement at the end of the image gathering procedure, the calibration outlined in a) should be repeated and any measured deviation recorded.

i) All the conditions, set up or established, for the target material shall be fully documented.

5 Sample preparation demands for method description

5.1 Sample splitting and reduction

As 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.

5.2 Touching particles

In order to assess the degree of touching particles, a suitable optical resolution setting of the imaging system should be chosen. The optical resolution should also meet the criteria set out in 4.2 b).

The number of particles touching each other should be minimized. It is a prime requirement of the method that measurements shall be made on isolated particles. Touching particles measured as one particle without a proper separation will introduce error.

It is often not possible to reliably detect touching 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. If the number of particles cannot be changed, the influence on the results can be investigated using a reference material with similar size and shape.

5.3 Particle distribution

There should be an adequate distribution of particles in the field of view. It may be necessary to examine several fields of view if a large total particle count is required. The whole area of the measurement field should be examined to ascertain whether there is noticeable segregation of particles (by size). The requirements set out in 4.2 f) should be followed.

5.4 Number of particles to be counted

The number of particles to be analysed depends upon whether the final result is a particle size distribution by the number or by the volume of particles. Considerable care has to be exercised in order to ensure that the analysis is representative of the bulk sample 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 will reveal the repeatability of the method including sampling and dispersion.

NOTE See Annex A for more information.