INTERNATIONAL STANDARD

ISO
14488

First edition
2007-12-15

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

Matériaux particulaires — Échantillonnage et division des échantillons pour la caractérisation des propriétés particulaires

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

iTeh Standards
(https://standards.iteh.ai)
Document Preview

ISO 14488:2007
https://standards.iteh.ai/catalog/standards/iso/fc7323fd-4ec7-44fc-9aca-7575b1a77a35/iso-14488-2007

Contents

Page

Foreword

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

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 14488 was prepared by Technical Committee ISO/TC 24, *Sieves, sieving and other sizing methods*, Subcommittee SC 4, *Sizing by methods other than sieving*.

iTeh Standards
(https://standards.iteh.ai)
Document Preview

ISO 14488:2007
https://standards.iteh.ai/catalog/standards/iso/fc7323fd-4ec7-44fc-9aca-7575b1a77a35/iso-14488-2007

Introduction

The characterization of particle properties like size, form and specific surface area requires very careful sampling and sample splitting practices to be followed. The distributions of the values of such properties are related to the number of particles, which cannot be increased as in sampling for chemical analysis. Deviations from statistical values occur due to the presence of particles of different sizes and shapes for each component in a powder obtained from a sampled mass of powder.

iTeh Standards
(https://standards.iteh.ai)
Document Preview

ISO 14488:2007
https://standards.iteh.ai/catalog/standards/iso/fc7323fd-4ec7-44fc-9aca-7575b1a77a35/iso-14488-2007

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

1 Scope

This International Standard specifies methods for obtaining a test sample from a defined bulk of particulate material (powder, paste, suspension or dust) that can be considered to be representative of that bulk with a defined confidence level. It is particularly relevant to the measurement of particle size, size distribution and surface area.

2 Normative references

The following referenced documents are indispensable for the application 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 3165, *Sampling of chemical products for industrial use — Safety in sampling*

ISO 6206, *Chemical products for industrial use — Sampling — Vocabulary*

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 14887, *Sample preparation — Dispersing procedures for powders in liquids*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 6206 and the following apply.

3.1
bias
systematic difference between true (or accepted) value and measured value

3.2
"critical" size class
specific size class, whose sampling error, in its fractional mass, has a significant influence upon the product properties

3.3
error
difference between a measured value and the true value, which may have a random or a systematic nature

3.4
gross sample
primary sample, composed of several sample increments

3.5
grab sample
sample that has not been taken under well-defined conditions

3.6
primary sample
sample (single or composed) taken from a defined bulk product

3.7
representative sample
sample that has the same properties as a defined batch of material and represents the bulk material, within a defined confidence limit

3.8
sample
part of a defined bulk product taken for the purpose of characterization

3.9
sample increment
single sample, taken from any of a defined set of locations in a bulk product or at any of a defined set of times from a production/transportation line, to be mixed with other increments to form a gross sample

3.10
sampling sequence
sequence of sampling, sample division and combination steps that result in a test sample for a defined bulk product

3.11
spot sample
sample, taken at a defined location or production time, from a batch of material

3.12
test sample
sample that is entirely used for a property characterization

4   Abbreviations and symbols

For the purposes of this document, the following abbreviations and symbols apply.

CV	coefficient of variation, i.e. standard deviation relative to the corresponding mean value, expressed as fraction or percentage
F_{ϕ_1,ϕ_2}	standard F-distribution value with ϕ_1 and ϕ_2 degrees of freedom
FE	fundamental error
MMD	mass median diameter
n	total number of particles in sample or sample increment
n_0	number of particles in a defined size class
n_{min}	number of particles in a sample or sample increment required to obtain a defined maximum deviation ε_{max} with a defined level of confidence
n_{MMD}	required number of particles in a sample to meet the stated error of the MMD
n_r	total number of particles in the log-normal particle size distribution required to reach a maximum coefficient of variation of 3,16 % in x_{90}
N	number of measured samples
N_r	number of samples required to obtain a defined maximum deviation ε_{max}, with a defined level of confidence, between the estimated and the true mean value of a property of interest

$Q_0(x_i)$	cumulative number-based particle size distribution
$Q_3(x_i)$	cumulative volume- or mass-based particle size distribution
r	dimensionality (type of quantity) of a distribution: $r = 0$: number; $r = 1$: length; $r = 2$: area; $r = 3$: volume or mass
SD	standard deviation
s_y	estimate of standard deviation of y, coming from measurements
t	Student's t-factor for statistical significance, which depends on the confidence level taken and the number of degrees of freedom ($N - 1$) (to be taken from statistical tables)
Var	variance
x	particle size
x_5	5 percentile size of the particles
x_{95}	95 percentile size of the particles
x_i	particle size corresponding to percentile i
y	value of any property of interest of the particulate material, e.g. specific size, shape, surface area
$\overline{y}$	mean value of y
z_c	critical z-value related to a defined confidence level according to the standard normal distribution (to be taken from statistical tables)
ε_{max}	defined maximum level of deviation at defined confidence level (half-width of the stated confidence interval)
γ	granulometric factor, related to the width of the particle size distribution, expressed by the ratio x_{95}/x_5 of the undersize particle size distribution; $\gamma \approx 0{,}25$ for wide particle size distribution with $x_{95}/x_5 > 4$; $\gamma \approx 0{,}5$ for $2 < x_{95}/x_5 < 4$; $\gamma \approx 0{,}75$ for $1 < x_{95}/x_5 < 2$; and $\gamma \approx 1$ for $x_{95}/x_5 \approx 1$
ρ	density of particles in kg/m^3
σ	standard deviation; square root of variance (theoretical value)
σ_g	geometric standard deviation of the log-normal particle size distribution
σ_P	fundamental error (standard deviation) of mass fraction of particles smaller than or equal to x_i, i.e. $Q_3(x_i)$

5 Principles of sampling and sample splitting

5.1 General

Particulate materials consist of discrete particles, each having its own properties such as size, shape, surface area, density and/or composition. Sometimes, the material is well mixed and the properties show only random variations with respect to location in the bulk and/or time of production. More often, however, segregation occurs due to the free-flowing behaviour of the material and/or fluctuations in the production process. This can result in a systematic deviation between the mean properties at different locations and at different times. For representative sampling, each of the particles in a bulk product must have the same probability of being sampled, in their proportions. For well-mixed materials, a single sample of adequate quantity may suffice. For most materials, some degree of segregation is to be expected. Then, several sample increments must be taken from different locations or at different production times. These are either analysed as such or combined into one primary sample. In most cases, there is no recipe for representative sampling. The quality of the

sampling procedure can only be assessed by measurement. Often, the primary sample collected in this way is too large for characterization of the required property. Then, sample splitting is necessary until the required quantity for the test is obtained.

The total error of sampling and sample splitting consists of two parts: the fundamental error and the segregation error.

The fundamental error is related to the discrete nature of particles given their different properties. It is a statistical error, related to the random variations of the properties with respect to location. It represents the lower limit of the total sampling error. This error depends on the amount (number, mass) of sample taken.

The segregation error is related to the degree of segregation, or the degree of "de-mixing" of particles according to their size, shape and density. It cannot be predicted, but only assessed by measurement of samples taken at different locations in the bulk and at different times of production. These measurements provide an estimation of the segregation error (see 5.3).

Given the complex behaviour of particulate materials, it is required that the complete procedure for sampling and sample splitting be described in a sampling plan (see Clause 6).

5.2   Fundamental error

5.2.1   Number distributions $Q_0(x)$

For number-based size distributions, the fundamental error [expressed as the variance Var or squared standard deviation σ of a fraction $Q_0(x_i)$ at any point x_i] can be calculated using the mathematics of binomial distributions [1], [6]:

$$\text{Var}[Q_0(x_i)] = \sigma_{Q0,i}{}^2 = Q_0(x_i)[1 - Q_0(x_i)]/n \tag{1}$$

If the number of particles in a size class or above a certain size is very small in comparison to the total number of particles taken into account for a measurement, then Poisson statistics may be used for estimating the variance or the standard deviation:

$$\text{Var}(n_0) = \sigma(n_0)^2 = n_0 \tag{2}$$

For example, for the number of particles larger than x_{90}, it can be seen that

$$n_0 = [1 - Q_0(x_{90})]n \tag{3}$$

and, since $Q_0(x_{90}) = 0{,}9$ or almost 1, Equations (1) and (3) give approximately the same answers.

If the fundamental error is the only error, the minimum number of particles $n_{\min}$ required to obtain a defined maximum deviation $\varepsilon_{\max}$ with a defined confidence can be derived from:

$$\varepsilon_{\max} = z_c \sigma_{Q0,i} = z_c \sqrt{Q_0(x_i)[1 - Q_0(x_i)]/n_{\min}} \tag{4}$$

or

$$n_{\min} = \{Q_0(x_i)[1 - Q_0(x_i)]\}z_c{}^2/\varepsilon_{\max}{}^2 \tag{5}$$

where z_c is the critical z-value related to the defined confidence level according to the standard normal distribution and may be obtained from statistical tables.

The standard deviation of x_i can be calculated from $\sigma_{Q0,i}$ through multiplication by the reciprocal value of the slope of the cumulative size distribution at point x_i:

$$\sigma_{xi} = \sigma_{Q0,i}\, \text{d}x_i/\text{d}Q_0(x_i) \tag{6}$$

The coefficient of variation of x_i can be calculated from this standard deviation by multiplication by 100 and division by x_i:

$$\text{CV}_{xi} = 100\,\sigma_{xi}/x_i \tag{7}$$

5.2.2 Volume- or mass-based distributions $Q_3(x)$

For volume- or mass-based size distributions, the general calculation of the fundamental error is not simple. One way is to use a spreadsheet programme (e.g. Excel) to convert the measured volume-based size distribution of a typical sample into the corresponding number distribution. The conversion principles and equations given in ISO 9276-2 shall be used.

This estimation of the minimum amount of sample in view of a stated minimum fundamental error shall always be the first step in a sampling procedure.

As an example of the results from such calculations, Figure 1 and Figure 2 are presented for a 1 % coefficient of variation in x for various characteristic sizes as log-normal size distributions. The calculations were conducted for distributions around a median size of 30 µm with a material density of 1 000 kg/m³. A detailed calculation is given in Annex B.

Figure 1 shows that, for a constant percentile, the sample mass required to reach a coefficient of variation of 1 % increases with particle size distribution width. It increases from about 10 mg to 1 kg as the distribution width ratio $(x_{90,3}/x_{10,3})$ increases from 2 to 100. The sample mass required is also increased if the fundamental error is to be maintained for percentiles ever closer to the highest limit of the distribution $(x_{90,3}, x_{95,3}, x_{98,3}$ and $x_{99,3})$. For a distribution by mass, it should be noted that a 10 times higher mean particle size requires a 1 000 times higher sample mass and *vice versa*. Note also that acceptance of a 10 times larger coefficient of variation in the fundamental error requires 100 times less sample mass.

Figure 2 illustrates that, for larger distribution width ratios, larger numbers of particles must be counted to ensure a result whose error remains below the target of a 1 % coefficient of variation. For a very narrow size distribution, counting a few hundred particles is all that is required. However, for very broad distributions, a number in excess of 10 000 000 will need to be assessed and counted.

5