INTERNATIONAL STANDARD

ISO 14488

First edition
2007-12-15

AMENDMENT 1
2019-11

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

AMENDMENT 1

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

AMENDEMENT 1

Reference number
ISO 14488:2007/Amd.1:2019(E)

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Published in Switzerland

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

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Annex B

Add the following additional clause.

B.7 Simple approach to the calculation of the fundamental sampling error (FSE) and minimum mass required for a specified standard error

This clause provides a method to approximately determine the magnitude (mg/g/kg) of the minimum mass required to meet a specific standard error. The approach approximates the precise calculations described in the document. With a knowledge or assumption of the actual mass utilized in the particle size analysis experiment, the best standard error achievable based solely on the heterogeneity of the material can also be estimated.

The fundamental sampling error (FSE) is one of 8 errors (implies culpability) or variables (implies statistical variation) originally described by Pierre Gy[12] – see Table B.3. It represents the smallest possible variation in sample to sample reproducibility based solely on the heterogeneity of the material or distribution involved.

Table B.3 — Old (Gy) and new notations (Pitard/Esbensen)

Errors	Old term	New term
Heterogeneity fluctuation error	CE	HFE
Quality fluctuation error	QE	QFE
Fundamental sampling error	FE	FSE
Grouping and segregation error	GE	GSE
Increment weighting error	WE	IWE
Increment delimitation error	DE	IDE
Increment extraction error	EE	IEE
Increment preparation errors	PE	IPE

The FSE is identical to the standard error familiar to mathematicians and statisticians[13][14][15]. The standard error (SE) of a parameter is the standard deviation (σ, theoretical value) or an estimate of the standard deviation (s, absolute value coming from measurements) of a sampled distribution. If the parameter or the statistic is the mean, it is called the standard error of the mean (SEM). It represents how close the result gets to the ‘true’ mean with repeated samplings or an increased proportion of the actual distribution.

In particle size distribution considerations, the FSE is inversely proportional to the square root of the number of particles present in the distribution or part of the distribution. In the following derivations, no assumption is made of the form of the original particle size distribution; only, that the samples withdrawn are normally distributed (as is the standard case).

$$\text{SEM } \alpha\ 1/\sqrt{n} \text{ or } n\ \alpha\ 1/\sigma^2$$

For 1 % SEM it can be shown that:

$$n = 1/(0{,}01)^2 = 10\,000$$

Thus, 10 000 particles in total will be needed to specify the mean to 1 % SE. See also Reference [18]. The implication is that to specify any other point of the distribution to 1 % SE, at least 10 000 particles will be needed in the portion of the distribution above that point. The worst-case situation is considered first; specifying the x_{99} point in the distribution to a standard error of 1 %. This requires 10 000 particles in the x_{99}+ part of the distribution. This x_{99}+ part represents only 1/100 of the total sample mass of the entire distribution. Thus, only the mass (= volume × density) of 10 000 particles needs to be calculated at some known or assumed x_{99} point in the distribution and multiplied by the appropriate value to compute the total mass of the distribution. Using the value of the x_{99} point is more convenient than attempting to use a more correct midpoint between the x_{99} and (unknown) x_{100} point. It will slightly underestimate the minimum mass required but calculations show this to be minor.

Making two assumptions, the minimum mass of the portion M_Q can be calculated: the particles are spherical and all particles of the portion have the minimum diameter x_Q.

$$M_Q = n_{\min} \times (\pi/6) \times x_Q{}^3 \times \rho$$

where

$n_{\min}$ is the minimum number of particles for the specified precision;

$(\pi/6)$ is the shape factor for a sphere;

x_Q is the largest point in the distribution that is to be specified;

ρ is the density of the particles in the sample.

NOTE 1 $(\pi/6) \times [x_Q]^3$ is the volume of a single particle of size x_Q and multiplied by the density ρ gives the mass.

This gives the minimum required total mass M_{tot}:

$$M_{\text{tot}} = \frac{M_Q}{1 - Q_3(x_Q)} = \frac{n_{\min} \times (\pi/6) \times x_Q{}^3 \times \rho}{1 - Q_3(x_Q)} = \frac{(\pi/6) \times x_Q{}^3 \times \rho}{(SE)^2 \times \left(1 - Q_3(x_Q)\right)}$$

where $Q_3(x_Q)$ is the volume-based cumulative distribution for the diameter x_Q.

NOTE 2 All parameters are given in SI units.

M_{tot}, is thus M_Q multiplied by a multiplier, which takes the calculated mass fraction, M_Q to the total mass of sample (e.g. for the x_{99} point then this is 1 % of the total sample so the multiplier is 100; for the x_{95} point this multiplier would be 20. Stricter statistical considerations[12] show 98 and 18 respectively for the multiplier for the x_{99} and x_{95} but this does not detract from the broad conclusions presented here).