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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

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

AMENDMENT 1

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).

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

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}:

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

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).

One simple example can illustrate the calculation, based on an assumed top end parameter x_{99} of 1 000 µm (0,001 m) of particles with a density of 2 500 kg/m³. The required standard error SE is 1 % (0,01) and according to its definition $Q_3(x_{99})$ = 99 %:

$$M_{\text{tot}} = \frac{(\pi/6)\times(0{,}001\ \text{m})^3\times 2\ 500\ \text{kg}/\text{m}^3}{(0{,}01)^2\times(1-0{,}99)} = \frac{(\pi/6)\times 2{,}5\times 10^{-6}\ \text{kg}}{10^{-6}} \approx 1{,}3\ \text{kg}$$

The minimum total mass M_{tot} required for an SE of 1 % for an x_{99} of 1 000 µm is 1,3 kg (see also Table B.4).

It is easy to spreadsheet such calculations. The example above can be expanded to cover sizes lower and higher in order of magnitude steps. See Table B.4.

Table B.4 — Mass of test sample required to reach 1 % standard error for the upper 1 % of a volume particle size distribution with particle density 2,5 g/cm³

x µm	Mass in top size fraction g	Total mass g (= Last column × 100)	Note
1	1,309E-08	1,31E-06	
10	1,309E-05	1,31E-03	~1 mg
100	1,309E-02	1,31E+00	~1 g
1 000	1,309E+01	1,31E+03	~1 kg
10 000	1,309E+04	1,31E+06	~1 000 kg
	This is the mass of 10 000 particles Assuming spheres	This is where 1 % of the particles are in the top size band (or 1 % in last size band — × 99)	

In summary, it can be stated that at least a test sample or sample increment of 1 g is required to reach a fundamental error (standard deviation) of 1 % for a $x_{99,3}$ (the 99th percentile of a volume distribution). Thus, the required sample size mass in mg, g, kg or metric tons is based solely on the top end point in the distribution as above. Table B.4 is in concordance to data displayed in Reference [16].

In a more practical case, the calculated sample masses would be reduced by a factor of 250 to meet the 5 % x_{90} requirement in ISO 13320:2009 [17] (25-fold from 1 % SE to 5 % SE and a factor of 10 from x_{99} to x_{90}).

It is possible to reverse the calculation and to calculate the best sample-to-sample precision based solely on the heterogeneity of the material. Therefore, the following parameters need to be known:

— the point in the distribution to specify;

— the density of the material;

— the amount of sample used in the experiment.

$x_{99,}$ can be taken as an example with sample sizes of 0,1 g/1 g/10 g and with various top end sizes from 1 µm to 2 000 µm, using a low density of 1,5 g/cm³ typical of a pharmaceutical. See Table B.5.

Table B.5 — Standard error calculations for the x_{99} point of the distribution with varying sample masses (0,1 g/1 g/10 g) and density 1,5 g/cm^3

x µm	x/10 000 (conversion to cm)	x^3 cm^3	ρ g/cm^3	**Mass used** g	**Mass of one particle** g	**No. particles**	**Calculated SE** %
1	0,000 1	1E-12	1,5	0,1	7,853 98E-13	1 273 239 545	0,003
1	0,000 1	1E-12	1,5	1	7,853 98E-13	12 732 395 447	0,001
1	0,000 1	1E-12	1,5	10	7,853 98E-13	1,273 24E+11	0,000
20	0,002	8E-09	1,5	0,1	6,283 19E-09	159 155	0,25
20	0,002	8E-09	1,5	1	6,283 19E-09	1 591 549	0,08
20	0,002	8E-09	1,5	10	6,283 19E-09	15 915 494	0,03
50	0,005	1,25E-07	1,5	0,1	9,817 48E-08	10 186	0,99
50	0,005	1,25E-07	1,5	1	9,817 48E-08	101 859	0,31
50	0,005	1,25E-07	1,5	10	9,817 48E-08	1 018 592	0,10
100	0,01	0,000 001	1,5	0,1	7,853 98E-07	1 273	2,80
100	0,01	0,000 001	1,5	1	7,853 98E-07	12 732	0,89
100	0,01	0,000 001	1,5	10	7,853 98E-07	127 324	0,28
200	0,02	0,000 008	1,5	0,1	6,283 19E-06	159	7,93
200	0,02	0,000 008	1,5	1	6,283 19E-06	1 592	2,51
200	0,02	0,000 008	1,5	10	6,283 19E-06	15 915	0,79
500	0,05	0,000 125	1,5	0,1	9,817 48E-05	10	31,3
500	0,05	0,000 125	1,5	1	9,817 48E-05	102	9,9
500	0,05	0,000 125	1,5	10	9,817 48E-05	1 019	3,1
1 000	0,1	0,001	1,5	0,1	0,000 785 398	1	88,6
1 000	0,1	0,001	1,5	1	0,000 785 398	13	28,0
1 000	0,1	0,001	1,5	10	0,000 785 398	127	8,9
1 500	0,15	0,003 375	1,5	0,1	0,002 650 719	0	162,8
1 500	0,15	0,003 375	1,5	1	0,002 650 719	4	51,5
1 500	0,15	0,003 375	1,5	10	0,002 650 719	38	16,3
2 000	0,2	0,008	1,5	0,1	0,006 283 185	0	250,7
2 000	0,2	0,008	1,5	1	0,006 283 185	2	79,3
2 000	0,2	0,008	1,5	10	0,006 283 185	16	25,1

At the larger sizes (>100 µm or so), the minimum variation is substantial if sample masses are kept below ≈10 g. This has implications for particle size distributions obtained by such techniques as electron microscopy.

At 20 mg total sample mass, the variation on the x_{99} (the number of calculated particles is in a mass of 0,000 2 g for the x_{99}) is given in Table B.6.

Table B.6 — Standard error calculations for the x_{99} point in the distribution with sample mass 20 mg and density 1,5 g/cm^3

x µm	ρ g/cm^3	**Mass used** g	**Mass of one particle** g	**No. particles**	**Calculated SE** %
1	1,5	0,02	7,853 98E-13	254 647 909	0,01
20	1,5	0,02	6,283 19E-09	31 831	0,56
50	1,5	0,02	9,817 48E-08	2 037	2,22

Table B.6 *(continued)*

x µm	*ρ* g/cm³	**Mass used** g	**Mass of one particle** g	**No. particles**	**Calculated SE** %
100	1,5	0,02	7,853 98E-07	255	6,27
200	1,5	0,02	6,283 19E-06	32	17,72
500	1,5	0,02	9,817 48E-05	2	70,06
1 000	1,5	0,02	0,000 785 398	0	198,17
1 500	1,5	0,02	0,002 650 719	0	364,05
2 000	1,5	0,02	0,006 283 185	0	560,50

The above calculations provide the most suitable sample-to-sample variation based solely on the heterogeneity of the material. All other errors or variation will add to this minimum.

Bibliography

Add the following references.

[12] Pitard F.F. *Pierre Gy's Sampling Theory and Sampling Practice*. Heterogeneity and Sampling CRC Press, Boca Raton, Vol. 1, 1989

[13] Brookes C.J., Betteley I.G., Loxston A.M. *Mathematics and Statistics for Chemists*. John Wiley and Sons, Hoboken, 1966, pp. 266

[14] Lucas H. *Statistical Methods*. Butterworths, 1970, pp. 77

[15] Annels A.E. *"Mineral Deposit Evaluation: a Practical Approach" Chapman and Hall, London, 1991 referenced in MOON, C J. et al., "Introduction to Mineral Exploration*. Blackwell, Oxford, Second Edition, 2006, pp. 206

[16] Richards R.H. *Ore Dressing*. Hill Publishing, New York, Vol. II, 1908, pp. 850

[17] ISO 13320:2009, *Particle size analysis — Laser diffraction methods*

[18] K Sommer Sampling of powders and bulk materials Springer-Verlag New York, Incorporated, New York, NY, U.S.A (1986) Table 9.2.2 page 191