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Fertilizers — Sampling — Minimum mass of increment to be taken to be representative of the total sampling unit

Engrais — Échantillonnage — Masse minimale du prélèvement en cas de traitement de la totalité de l'unité d'échantillonnage à prélever

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ISO/TR 7553 was prepared by Technical Committee ISO/TC 134, *Fertilizers and soil conditioners.*

The reasons which led to the decision to publish this document in the form of a technical report type 3 are explained in the Introduction.

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0 Introduction

Within the framework of its work on sampling, Technical Committee 134, *Fertilizers and soil conditioners*, has, through its sub-committee 2, carried out studies on the minimum mass which an increment may have in order to remain representative of the fertilizer sampled.

This work complements other standards for fertilizers, currently under preparation, particularly those which describe sampling plans for lots of various sizes, and those related to sample reduction.

This study, which is different from those usually published as International Standards, is intended to act as a complement to them, as a technical report (type 3) for the sampling of fertilizers.

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1 Scope and field of application

When sampling is carried out on a batch of fertilizer, an increment is taken from N units. Each of these increments may be taken either by means of partial sampling devices, if their validity (absence of any particular bias) is assured, or by treating the complete item with a suitable reduction device (absence of bias).

This Technical Report specifies, for the latter case, the extent of mass reduction beyond which the error in the representativity of the increment can no longer be ignored. It may also be used to establish the minimum mass of any intermediate increment during the treatment of the sample, up to analysis. Where partial sampling devices are used, this limiting mass can be much more important.

2 Basic theory

Taking into account the granular nature of solid fertilizers, the sampled mass cannot be reduced indefinitely without losing all representativity.

However, it is possible to relate the mass of the sample to a minimum error of representativity, as a function of the variability in composition of the product sampled and of its particle size distribution.

Emphasis should, however, be placed on the nature of the minimum limit value of this error to which can, in practice, be added large errors due to the variability of the increments, often associated with defective sampling devices (spears in particular).

It should also be noted that this error occurs at each reduction operation on the sample after its constitution. Particular attention is drawn to the fact that, in preparing the test portion from the laboratory sample, representativity can easily be lost if the masses sampled at a given point are insufficient with respect to particle size distribution and variability in composition or if they do not follow the rule of equiprobability of choice.

This concept is, in fact, empirically admissible because, in the case of determinations where grinding is allowed, such an operation is generally carried out before the test portion is taken.

Statistical studies now make it possible to calculate accurately a relation between these magnitudes. In cases where grinding is not possible (for example determination of particle size distribution), they also enable a relationship to be obtained between the mass of the test portion and the error of representativity.

Two types of fertilizer may be encountered:

a) Fertilizer in which all the particles are essentially of the same nature

Once the variability in analysis from one particle to another is known, it is possible to calculate statistically, on the basis of a normal distribution, the number of particles, and thus the minimum mass, required so as not to exceed a given uncertainty.

b) Fertilizer in which the particles are of a different nature (bulk blends)

Once the percentage of the least abundant constituent to be analysed is known, it is possible to calculate statistically, on the basis of a binomial distribution, the number of particles, and thus the minimum mass, required so as not to exceed a given uncertainty.

3 Fertilizers composed of particles of the same nature

The analyses vary continuously from particle to particle following a normal or an approximately normal distribution with a coefficient of variation generally between 10 % and 40 % (40 % in extreme cases with fertilizers obtained via an almost dry mixing stage).

Calculation on the basis of the theory of normal distributions indicates that, if the standard deviation for variations in particle analysis is called σ' and the desired precision at 95 % bilateral probability $2\,\sigma_\tau$, the mass of this increment is given, approximately, by the formula

$$C \times \frac{\pi}{6}\,\phi_m^3 \left(\frac{\sigma'}{\sigma_\tau}\right)^2 \varrho \times 10^{-3}$$

where

ϕ_m is the mean diameter of the particles as defined in the annex;

ϱ is the density of the particles;

$2\,\sigma_\tau$ corresponds to the overall error of judgement desired and is expressed in the same way as σ', for example as an absolute value;

C is a coefficient which renders the error connected with the limitation of the increment mass negligible compared with σ_τ. It will be chosen as equal to at least 5, as random errors are added to the square. Hence, approximating $\pi/6$ to 0,5,

$$m \geqslant \frac{C}{2} \phi_m^3 \varrho \left(\frac{\sigma'}{\sigma_\tau}\right)^2 \times 10^{-3}$$

The value of C will depend on the number of operations or divisions carried out on the initial increment(s), up to taking the test portion. The relationship is given in table 1.

Table 1 — Value of C in relation to number of dividing operations

Number of dividing operations	0	1	2	3	4
C	5	10	15	20	25

Table 2 gives limit values of m for typical values of ϱ, C, $\frac{\sigma'}{\sigma_\tau}$ and ϕ_m.

Table 2 — Examples of limit values of m
ϱ = 2 g/ml, C = 5

σ'/σ_τ \ ϕ_m	1 mm	2,5 mm	4 mm	10 mm
5	0,12 g	2 g	8 g	125 g
10	0,5 g	8 g	32 g	500 g
20	2 g	32 g	130 g	2 000 g
40	8 g	125 g	510 g	8 000 g

4 Fertilizers composed of particles of different nature

The second group of fertilizers includes dry mixtures (bulk blends or others) without any notable agglomeration of the constituents.

In these products, the variation in composition of the constituents is low compared with the heterogeneity of the constituents and it is ultimately the latter factor which dictates the mass of the increment. Since the density of each of the constituents is approximately the same, to within ± 20 %, calculation on the basis of the binomial law leads to the equation

$$m \geqslant \frac{C}{2} \times \frac{y\,(1-y)\,\phi_m^3}{\sigma_\tau^2} \varrho \times 10^{-3}$$

if the size distributions of the constituents are similar.

σ_τ is expressed as a function of unity and not as a percentage, in the same way as y;

y corresponds to the amount of the least abundant constituent, expressed as a fraction of unity.

Table 3 gives the values of this minimum mass for ϕ_m = 1, 2 and 4 mm and y = 0,05, 0,15 and 0,50 assuming that ϱ = 2 g/ml and C = 5.

Table 3 — Values of minimum mass for similar size distributions

ϕ_m	1 mm			2 mm			4 mm		
y \ σ_τ/y	0,01	0,02	0,05	0,01	0,02	0,05	0,01	0,02	0,05
0,05	950 g	240 g	40 g	7 600 g	1 900 g	300 g	60 800 g	15 200 g	2 450 g
0,15	285 g	70 g	12 g	2 260 g	570 g	90 g	18 100 g	4 530 g	720 g
0,50	50 g	12 g	2 g	400 g	100 g	16 g	3 200 g	800 g	130 g

If the size distributions vary greatly (segregation during transport causes problems, so that the manufacture of this type of fertilizer should be avoided) the equation becomes

$$m \geqslant \frac{C}{2\sigma_\tau^2} \varrho\, y\, (1 - y)\, [y\, \phi_2^3 + (1 - y)\, \phi_1^3] \times 10^{-3}$$

The index 1 is attached to the component corresponding to the amount y.

Table 4 gives the values of this minimum mass for $\phi_1 = 1$ mm and $\phi_2 = 2$ mm; $\phi_1 = 2$ mm and $\phi_2 = 1$ mm and $y = 0{,}05$, 0,15 and 0,50, assuming that $\varrho = 2$ g/ml and $C = 5$.

Table 4 — Values of minimum mass for size distributions which vary greatly

	σ_τ / y	$\phi_1 = 1$ mm $\phi_2 = 2$ mm		$\phi_2 = 1$ mm $\phi_1 = 2$ mm	
		0,01	0,05	0,01	0,05
y	0,05	1 285 g	52 g	7 250 g	290 g
	0,15	580 g	24 g	2 000 g	80 g
	0,50	225 g	9 g	225 g	9 g

The case in which the densities of the two constituents are very different is even more hazardous with regard to precision of sampling, segregation giving rise to even greater errors.

In this case, the equation is again modified and becomes

$$m \geqslant \frac{C}{2\sigma_\tau^2}\, y\, (1 - y)\, [y\, \varrho_2\, \phi_2^3 + (1 - y)\, \varrho_1\, \phi_1^3] \times 10^{-3}$$

For $\varrho_1 = 1{,}5$ g/ml, $\varrho_2 = 2{,}5$ g/ml and $C = 5$

the following sample table (table 5) is obtained:

Table 5 — Values of minimum mass for two constituents with very different densities

	σ_τ / y	$\phi_1 = 1$ mm $\phi_2 = 2$ mm		$\phi_2 = 1$ mm $\phi_1 = 2$ mm	
		0,01	0,05	0,01	0,05
y	0,05	1 150 g	46 g	5 450 g	218 g

It can be seen that a mean ϱ can be taken without great error if the minor constituent is not that with the larger mean diameter.

5 General comment on the reduction of samples

It can be seen that, according to the type of product for sampling, the precautions which have to be taken can vary greatly.

These conditions of the mass/particle size ratio must also be respected at all stages in the preparation of reduced or final samples up to and including the stage at which the test portion is taken for analysis.

It follows that any reduction in the sample quantity should, in general, be accompanied by crushing. If the fertilizer is to be kept in its original state, then a large quantity of sample must be retained, or a loss in representativity must be accepted.

For example, for a mixed granular fertilizer with $y = 0{,}15$, if the starting value of ϕ_m is 4 mm, the initial increment is approximately 4 500 g for a 2 % precision. If this is reduced to 200 g while retaining the same representativity, it must be crushed until a value of ϕ_m of about 1,4 mm is obtained, and for a test portion for analysis of 2 g, it must be ground so that $\phi_m \approx 0{,}3$ mm.

If the sample is reduced to 500 g without crushing, an additional error of representativity is introduced which will more than quadruple the standard deviation of the overall uncertainty, which will go from 2 % to 8 % relative.

6 Selection of the mass of the smallest increment in the case of grouping of the increments (k by k before analysis)

It this case, the minimum masses defined above shall be respected throughout all the dividing operations up until testing, but at the start it is sufficient that each grouping of k increments attain this minimum mass.

It would then be possible to select a mass for the initial increment k times smaller.

It is important to be aware of the need for particular care with regard to the validity of the sampling apparatus, which must provide an absolute guarantee of the equiprobability of the particles comprising the increments.

Annex

Calculation of ϕ_m

If the particle size distribution of the fertilizer is relatively broad, the mean diameter ϕ_m may be calculated from a size distribution analysis.

By definition, ϕ_m corresponds to the mean diameter of a hypothetical size distribution having a single particle size classification which, with an increment of mass

$$M = \eta \frac{\pi}{6} \phi_m^3 \varrho$$

would enable the same precision

$$\sigma_p^2 = \frac{(\sigma')^2}{\eta}$$

to be obtained as that obtained with the actual distribution.

The increment of mass of the actual distribution corresponds to

$$M = \sum \left(\eta_i \phi_i^3 \frac{\pi}{6} \varrho\right) = \frac{\pi}{6} \varrho \sum \left(\eta_i \phi_i^3\right)$$

and the precision is derived as follows:

$$\sigma_p^2 = \sigma^2(x_m) = \sigma^2\left(\sum \frac{m_i}{M} x_i\right) = \sum \left(\frac{m_i}{M}\right)^2 \sigma^2(x_i) = \sum \left[\frac{(\sigma')^2}{\eta_i} \frac{m_i^2}{M^2}\right] = \frac{(\sigma')^2}{M^2} \sum \left(\frac{m_i^2}{\eta_i}\right)$$

Thus,

$$M = \eta \frac{\pi}{6} \phi_m^3 \varrho = \frac{\pi}{6} \varrho \sum \left(\eta_i \phi_i^3\right) \qquad \text{hence} \quad \phi_m^3 = \frac{1}{\eta} \sum \left(\eta_i \phi_i^3\right) \qquad \ldots (1)$$

and

$$\sigma_p^2 = \frac{(\sigma')^2}{\eta} = \frac{(\sigma')^2}{M^2} \sum \left(\frac{m_i^2}{\eta_i}\right) \qquad \text{hence} \quad \frac{1}{\eta} = \frac{1}{M^2} \sum \left(\frac{m_i^2}{\eta_i}\right) \qquad \ldots (2)$$

Also

$$m_i = \eta_i \frac{\pi}{6} \phi_i^3 \varrho \qquad \text{hence} \quad \eta_i = \frac{6\, m_i}{\pi\, \phi_i^3 \varrho} \qquad \ldots (3)$$

and

$$\sum m_i = M \qquad \ldots (4)$$

Substituting for $1/\eta$ from equation (2), equation (1) becomes

$$\phi_m^3 = \frac{1}{M^2} \sum \left(\frac{m_i^2}{\eta_i}\right) \sum (\eta_i \phi_i^3)$$

Substituting for η_i from equation (3), this becomes

$$\phi_m^3 = \frac{1}{M^2} \sum \left(\frac{\pi \phi_i^3 \varrho m_i}{6}\right) \sum \left(\frac{6 m_i}{\pi \varrho}\right) = \frac{1}{M^2} \sum (\phi_i^3 m_i) \sum m_i$$

Substituting from equation (4), gives

$$\phi_m^3 = \frac{1}{M} \sum (\phi_i^3 m_i)$$

Therefore

$$\phi_m = \sqrt[3]{\frac{1}{M} \sum (\phi_i^3 m_i)}$$

where

- η is the number of particles in the increment;
- η_i is the number of particles in each size classification;
- ϱ is the density of the particles;
- ϕ_i is the mean diameter of each size classification;
- m_i is the mass of each class;
- x_i is the content of each class;
- x_m is the mean content of the increment;
- σ' is the standard deviation of the content on the scale of the particle;
- $\sigma(x_i)$ is the standard deviation of the content on the scale of the size classification;
- σ_p is the standard deviation of the content on the scale of the increment.

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