# INTERNATIONAL STANDARD

ISO 8634

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# Solid fertilizers — Sampling plan for the evaluation of a large delivery

iTeh SMatières fertilisantes solides Plan d'échantillonnage pour l'évaluation d'une grosse livraison (standards.iteh.ai)

<u>ISO 8634:1991</u> https://standards.iteh.ai/catalog/standards/sist/8ebc519c-fbc6-454b-9890-7e0d9f37a5f9/iso-8634-1991



Reference number ISO 8634:1991(E)

## 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.

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.

International Standard ISO 8634 was prepared by Technical Committee ) ISO/TC 134, Fertilizers and soil conditioners.

Annex A forms an integral part of this International <u>Standardo Annex B</u> is for information only. https://standards.iteh.ai/catalog/standards/sist/8ebc519c-fbc6-454b-9890-7e0d9f37a5f9/iso-8634-1991

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## Introduction

This International Standard on the sampling and evaluation of a large delivery of solid fertilizer has been prepared on the basis of a statistical study which forms the subject of ISO/TR 5307.

Each country has its own regulations applicable to the fertilizer trade and an official department is responsible for checking the application of these regulations. If they are violated, sanctions may be taken against those responsible for placing the fertilizer in that country. In the case of an imported delivery, it is the representative of the manufacturing company in the country, or the importer, who is considered by the relevant authorities to be responsible for the contents declared on labels or other documentation accompanying the fertilizer.

iTeh S This International Standard concerns the case of an importer who resells, under his own reponsibility, a large amount of fertilizer received from abroad. After unloading, this delivery is resold in smaller lots to Straders (dealers or agricultural cooperatives) who will themselves directly supply farmers. In the case in question, it is the importer whose name is associated with the fertilizer; therefore it is he who will be considered by the retailers and users to be responsible for the declared contents.

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# Solid fertilizers — Sampling plan for the evaluation of a large delivery

#### 1 Scope

This International Standard specifies a method for sampling a delivery of more than 250 t of fertilizer and, after analysis of the sample or samples, presents rules for assessing whether the delivery can be accepted by a buyer, allowing for given reselling risks under given local legal conditions (or if he wishes to guarantee to the final buyer a given mean assay with a given risk). **Teh STANDAR**  mum mass of increment to be taken to be representative of the total sampling unit.

ISO 7742:1988, Solid fertilizers — Reduction of samples.

ISO 8358:1991, Solid fertilizers – Preparation of samples for chemical and physical analysis.

assay with a given risk). **iTeh STANDARD PREVIEW** The weighing of the bags or lots, the date limit for **s.itshDefinitions** sampling, etc., which are of necessity involved in the

definitive evaluation of the delivery, are not dealt

with in this International Standard.

https://standards.iteh.ai/catalog/standards/si This sampling plan is applicable to a large delivery of fertilizer supplied to a third party for resale under his own responsibility, in small lots, each of which would be subject to legislation.

By "large amount" is understood, for example, a full boat-load (5 000 t, 10 000 t or more) corresponding to a relatively long period of manufacture, but the plan may be used for any delivery of 250 t or more.

#### 2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 5306:1983, Fertilizers — Presentation of sampling reports.

ISO/TR 7553:1987, Fertilizers — Sampling — Mini-

ISO 8634:1991 For the purposes of this International Standard, the following definitions apply. These definitions are givery so-863 taken from ISO 8157/Add.1. ISO 8157 contains addiunder tional definitions relating to the sampling of fertiwhich lizers.

**3.1 lot; sample portion:** The total quantity of material, assumed to have the same characteristics, to be sampled using a particular sampling plan.

**3.2 delivery:** A quantity of material transferred at one time.

**3.3 sampling unit**: A defined quantity of material having a boundary which may be physical, for example a container, or hypothetical, for example a particular time or time interval in the case of a flow of material.

**3.4 increment:** A representative quantity of material taken from a sampling unit.

**3.5 aggregate sample:** A combination of all increments from the lot.

NOTE 1 In this International Standard, all the increments are grouped in equal numbers to form aggregate samples. 3.6 reduced sample: A representative part of the aggregate sample obtained by a process of reduction in such a manner that the mass approximates to that of the final (laboratory) samples.

3.7 final sample: A representative part of the reduced sample or, where no intermediate reduction is required, of the aggregate sample.

#### Symbols 4

The following symbols appear in this International Standard and have the meanings assigned to them below.

- Ν Number of sampling units to be selected during the sampling of the delivery. (Increments.)
- N' Number of analyses to be carried out on the N increments during the inspection of the delivery.
- Number of sampling units contained in the  $N_{\mathsf{R}}$ smallest lot presented for resale.
- k Number of increments to be combined into each aggregate sample for analysis
- **A**KI Calculation intermediate used to facilitate the Number of sampling units which will be n calculation of k and N. mandatorily selected during the official sam dards.i pling of a lot of  $N_{\rm R}$  sampling units.
- Estimate of  $\sigma/\sqrt{k}$  with the aid of N' analyses, <u>ISO 8654:1</u> General plan of application S where  $\sigma$  is the standard side viation in the tweet of the standards of the standard side via the standard si the sampling units in the delivery.
- Analytical result obtained on the sample of  $\chi_i$ rank i.
- Estimate of the mean value of the delivery  $\overline{\chi}_i$ with the aid of the N' analyses.
- L Official inspection limit value which depends on the declared value (D). It may be equal to D or less than D by a prescribed tolerance which may depend on the size of the lot sold.
- Probability that the mean value of *n* sampling  $r_{a}$ units is lower than the official limit value (L), just acceptable by the importer.
- Probability that the mean value of *n* sampling  $r_{\rm r}$ units is lower than the official limit value (L), just unacceptable by the importer.
- Probability of rejection of a delivery of just α acceptable quality (seller's or producer's risk).
- ß Probability of acceptance of a delivery of just unacceptable quality (importer's or consumer's risk).
- Value of the standardized normal variable  $u_{1-r_{a}}$ such that  $Pr [u > u_{1-r_a}]$  equals  $r_a$ .

- $u_{1-r_{e}}$  Value of the standardized normal variable such that  $Pr [u > u_{1-r_{r}}]$  equals  $r_{r}$ .
- Value of the standardized normal variable  $u_{1-\alpha}$ such that  $Pr [u > u_{1-\alpha}]$  equals  $\alpha$ .
- Value of the standardized normal variable  $u_{1-\beta}$ such that  $Pr [u > u_{1-\beta}]$  equals  $\beta$ .
- K Calculation coefficient which is dependent on *n*, the risk levels  $\alpha$  and  $\beta$  and the probability levels  $r_a$  and  $r_r$ .
- a Constant factor dependent on N' which represents the uncertainty associated with the estimate of the standard deviation.
- Non-centrality parameter. δ
- Value of the non-central Student ratio corre $t_0$ sponding to the level of probability for a noncentrality parameter equal to  $\sqrt{N} u_{1-r} / \sqrt{n}$ .
- Limit value of the estimate calculated from  $B_0$ *t*<sub>0</sub>.
- A, BCalculation intermediates used during the estimation of the lot after analysis.
- 7e0d9f37a5f9/The863pplication of this International Standard in
  - volves a certain number of successive operations, which are described in clause 6 to clause 8.

#### 5.1 Determinations prior to sampling

Determination of the number of sampling units N to be sampled and determination of the number of analyses N' to be carried out (see clause 6)

### 5.2 Sampling

- a) Designation and separation of the sampling units resulting in increments:
  - fertilizer in bags (see 7.1.2);
  - fertilizer in bulk (see 7.2.2).
- b) Increments from the sampling units:
  - fertilizer in bags (see 7.1.3);
  - fertilizer in bulk (see 7.2.3).
- c) Preparation of N' aggregate samples by grouping N increments k by k (see 7.1.4).

- d) Preparation by mixing and corresponding reduction of the N' aggregate samples into N' reduced samples for laboratory testing (see 7.1.4).
- e) Drawing up the sampling report (see clause 8).

### 5.3 Analyses

#### 5.4 Conclusions

# 6 Determination of the number of increments N and number of analyses N'

Assign numerical values to the following parameters:

 $n, r_a, r_r, \alpha$  and  $\beta$ 

Refer to table A.1 to find the corresponding values of the reduced normal variables:

 $u_{1-r_a}, u_{1-r_r}, u_{1-\alpha}$  and  $u_{1-\beta}$ 

Calculate the values of N and N' as a function of these values, using either the complete procedure given in 6.1 or the simplified procedure given in 6.2. The simplified calculation may be used without excessive error as long as N' is greater than 5 damages in 6.2.

NOTE 2The derivations of formulae used below are to<br/>be found in ISO/TR 5307 together with typical values 8664:1991NN and N'.https://standards.iteh.ai/catalog/standards/sist/8eba

## 6.1 Calculation of N and N' by the complete procedure

To determine the minimum value of N', calculate

$$\left(\frac{1-a^2}{a^2}\right)_0 = \left(\frac{u_{1-r_a}-u_{1-r_r}}{u_{1-\alpha}u_{1-r_r}+u_{1-\beta}u_{1-r_a}}\right)^2$$

Using table A.2, determine the value of N' such that the ratio

$$\frac{1-a^2}{a^2}$$

corresponding to this value of N' is just less than

$$\left(\frac{1-a^2}{a^2}\right)_0$$

calculated above.

Take  $N'_0$  to be this particular value of N'; this is the smallest possible value for N'.

Leaving  $(1 - a^2)/a^2$  undetermined, calculate the expression

$$F = n(u_{1-\alpha} + u_{1-\beta})^{2} \{ (u_{1-r_{a}} - u_{1-r_{r}})^{2} - [(1-a^{2})/a^{2}](u_{1-\alpha}u_{1-r_{r}} + u_{1-\beta}u_{1-r_{a}})^{2} \}^{-1}$$

With the aid of table A.2, determine the values of

$$\frac{1-a^2}{a^2}$$

for  $N' = N_0$ ,  $N_0 + 1$ ,  $N_0 + 2$ , etc. and calculate for each of these values of N' the corresponding values of F and hence of k, where k is the whole part of

$$\frac{F}{N'}$$
 + 1

and thus N, where N = kN'.

Stop when a value N' is obtained for which the corresponding value of F is less than this value N'. Let this value be  $N'_m$ .

Transfer all the results to table 1.

a function of	Table 1				
te procedure given in 6.2.RD d without ex-	PŘEV	$1-a^2$	F	k	Ν
d without ex-	$e_{N_0+1}^{N_0+1}$				
d below are to bical va <b>lues86</b> \$4:1991	N' <sub>0</sub> + 2				
ai/catalog/standards/sist/ 7e0d9f37a5f9/iso-8634	-1991	:6-454b-989	0-		
complete	N' <sub>0</sub> +				

With the aid of table 1, all possible pairs of N and N' can be obtained.

For practical application, these pairs of N and N' may be given in a simplified table (table 2).

Table 2

N	N'					

# 6.2 Calculation of N and N' by the simplified procedure

Use the formula

$$N \ge n \left( \frac{u_{1-\alpha} + u_{1-\beta}}{u_{1-r_{a}} - u_{1-r_{r}}} \right)^{2} \left( 1 + \frac{K^{2}}{2} \right)$$

where

$$K^{2} = \frac{k}{n} \left( \frac{u_{1-r_{a}} u_{1-\beta} + u_{1-r_{r}} u_{1-\alpha}}{u_{1-\alpha} + u_{1-\beta}} \right)^{2}$$

and prepare table 3, noting that N' = N/k and therefore that the value of N multiples of k immediately above the calculated figure must be chosen.

Table 3

k	N′	N
1	$Z_{1}/1$	<i>Z</i> <sub>1</sub>
2	7 <sub>2</sub> /2	$Z_2$
3	Z₃/3	$Z_3$
4	$Z_{4}/4$	$Z_4$
5	Z <sub>3</sub> /3 Z <sub>4</sub> /4 Z <sub>5</sub> /5	$Z_5$
1		

#### Sampling 7

#### 7.1 Products in bags

This is the general case for products packaged in small units of mass between 10 kg and 100 kg Abagdards.iteh.ai) usually weighs 50 kg.

#### 7.1.1 Sampling unit

#### ISO 8634:1991

## https://standards.iteh.ai/catalog/standards/sist/8ebc519c-fbc6-454b-9890samples

In this case, the sampling unit is a bag.

#### 7.1.2 Designation of bags from which increments are taken

The designation of the bags is made at random, for example by numbering the bags 1, 2, 3, etc. and using a table of random numbers.

#### 7.1.3 Increments

Take one increment per bag chosen, i.e. a total of N increments.

Because of the more or less granular nature of solid fertilizers, the masses sampled or handled cannot be reduced indefinitely without losina all representativity.

Each increment or divided fraction shall comply with two essential rules. It shall

- a) contain a sufficient number of grains;
- b) not favour the sampling of any type of grain or any particular locality (equiprobability of selection).

Condition b) is very exacting as regards the choice of sampling tool, which should be known to have no bias with the type of fertilizer sampled. In case of doubt, preliminary tests on a closely related synthetic mixture are essential.

The use of a tool which deals with the complete sampling unit is generally preferable (riffle, cone sample divider, automatic rotary sample divider, etc.) as long as it does not give rise to elutriation phenomena (separating out of the fine particles, centrifuging of the coarse particles, etc.).

If the sampling and division operations are performed on the complete sample unit from which the increment is taken, or on the sample for division. mathematical rules can be used to link the precision obtained with the granulometry and reduced size obtained.

It should be noted that all these comments concern all stages of handling of the product sampled, from each increment to the test portion for analysis. The requirements for the minimum mass at each stage of the procedure are set out fully in ISO/TR 7553.

Place these increments individually in clean. iTeh STANDAthem 1, 2, 3, etc.

> Group the increments k by k so as to obtain N' aggregate samples (k = N/N').

> Prepare N' clean containers which can be hermetically sealed.

> Place the increments numbered 1 to k in the first container and the increments numbered from (k + 1) to 2k, etc. in the second container.

> Mix, as thoroughly as possible, each of the aggregate samples thus produced using a device (V-mixer or double-cone mixer) or, if this is not possible, by hand.

> Reduce each of the aggregate samples to a sufficient mass to provide the required number of final samples of the required mass. Place these reduced aggregate samples in clean, watertight and sealable containers and number them 1, 2, 3, etc.

> Subsequent treatment of these samples will depend on the purpose of the sampling and the analyses to be performed. For information on sample reduction and division, see ISO 7742 and on sample preparation, see ISO 8358.

#### 7.2 Products in bulk, during loading or unloading

#### 7.2.1 Sampling unit

If the loading or unloading operation is carried out (grabbing grabbing equipment crane usina automatic-shovel loader), the sampling unit is composed of the quantity of material corresponding to one grab.

If the operation is carried out, at least in part, using continuous apparatus (conveyor belt, pneumatic device, etc.), each sampling unit is made up of a mass of approximately 50 kg taken during the operation.

#### 7.2.2 Designation and separation of the sampling units from which increments are taken

The sampling units from which increments are taken shall be designated at random throughout the operation.

Knowing the mass of the lot and the mass of the sampling unit, start by calculating the total number of sampling units contained in the lot-ANDARI

Number the sampling units in chronological order of their actual formation (grabbing equipment) or virtual formation (continuous apparatus). In the latter case, the time intervals are numbered, taking into account the operating rate of the apparatus. tandards/sis

Designate at random N sampling units, Torlexampleo-8634h) all observations which could, in the opinion of the using a table of random numbers.

With grabbing equipment, discharge separately the N sampling units from which increments are to be taken.

With continuous apparatus, separate the N sampling units of approximately 50 kg on the transporting equipment, and collect them separately.

#### 7.2.3 Increments

Proceed exactly as for bags, given that a bag is replaced here by a sampling unit (see 7.1.3).

#### 7.2.4 Preparation of aggregate samples and reduced aggregate samples

Proceed exactly as for bags (see 7.1.4).

### 7.3 Products in bulk, in storage or in transport

The case of products in storage corresponds to that of products in bulk, in store and in piles; the case of products in transport concerns products in dump trucks, hopper wagons, barges, ships, etc.

In view of the extreme difficulty and sometimes impossibility (in the case of large piles, for example) of rigorously sampling products stored under such conditions, sampling shall always be carried out during loading or unloading, in accordance with 7.2.

#### 8 Sampling report

The sampling report shall include, in addition to the information specified by the various legal requlations:

- a) the date and location of sampling;
- b) the date of arrival of the delivery of fertilizer, if sampling has been carried out on the client's premises;
- c) the characteristics of the fertilizer sampled according to the information given on the bags or on the sale documents if it is in bulk, with the statement of the guaranteed elements;
- d) the nominal size of the delivery (number of bags, mass, etc.); W
- KE e) the designation of the sampling unit taken;
- en.ai f) the number of increments taken (N):
- sampler, be of importance (e.g. packaging);

g) the number of aggregate samples prepared (N'):

a declaration stating that sampling has been i) carried out in accordance with this sampling procedure.

In addition the sampling report shall mention all operating details not covered in this International Standard, or which are optional, together with any events which may have had an effect on the results.

A more detailed form of sampling report is given in ISO 5306.

#### Analyses of the reduced aggregate 9 samples

A separate analysis should be made on each of the N' reduced aggregate samples. Carry out the analyses in accordance with the methods from the relevant standards, taking the usual precautions.

The test report shall, in accordance with these standards, include in particular the values  $\chi_i$  of the analysis results of each of the laboratory samples (thus the corresponding reduced aggregate samples).