SLOVENSKI STANDARD
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Gnojila, sredstva za apnjenje in inhibitorji - Vzorčenje in priprava vzorcev - 1. del: Splošne zahteve za vzorčenje

Fertilizers, liming materials and inhibitors - Sampling and sample preparation - Part 1: General sampling provisions

Düngemittel, Kalkdünger und Inhibitoren - Probenahme und Probenvorbereitung - Teil 1: Allgemeine Festlegungen zur Probenahme

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Fertilizers, liming materials and inhibitors - Sampling and sample preparation - Part 1: General sampling provisions

Düngemittel, Kalkdünger und Inhibitoren - Probenahme und Probenvorbereitung - Teil 1: Allgemeine Festlegungen zur Probenahme

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 260.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Contents

Page

European foreword

This document (prEN 1482-1:2023) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

This document will supersede EN 1482-1:2007.

In comparison with the previous edition, the following technical modifications have been made:

— Title, Introduction, Scope, Normative References, Terms and definitions have been updated;

— Requirements on sampling for detonation testing, oil retention and other tests added to 4.3.2;

— The mass of the final sample in 4.3.4 has been specified;

— 5.11 has been updated.

EN 1482 “Fertilizers, liming materials and inhibitors — Sampling and sample preparation” consists of four parts:

— Part 1: General sampling provisions;

— Part 2: General sample preparation provisions;

— Part 3: Sampling of static heaps;

— Part 4: Sampling for microbiological presence in fertilizers.

This document has been prepared under a standardization request given to CEN by the European Commission and the European Free Trade Association.

Introduction

This document (prEN 1482-1) covers the following aspects of sampling, derived from the International Standards and documents indicated but presented in a simplified and condensed form. The titles of the International Standards are given in the Bibliography.

— Sampling plans and quantitative data: ISO 8634, ISO/TR 5307, ISO/TR 7553 and Regulation (EU) 2019/1009;

— Sampling methods: ISO 3963;

— Reduction: ISO 7410, ISO 7742, ISO 8358;

— Sampling reports: ISO 5306.

prEN 1482-2 covers the general methods for the reduction and preparation of samples of inorganic fertilizers, liming material and inhibitors for analysis. prEN 1482-3 covers the sampling of specified fertilizers when stored in a static heap. prEN 1482-4 covers the sampling of specified fertilizers to be tested for the presence of regulated microbes. It is under investigation whether this standard covers organic and organo-mineral fertilizers.

Figure 1 gives a schematic diagram of the sampling and sample preparation process for solids.

The fundamental principle of representative sampling is that every particle has an equal chance of being selected or rejected. In some circumstances this principle cannot easily be complied with, particularly in the case of bulk heaps of solid fertilizers not specified in prEN 1482-3, or large storage tanks of liquid fertilizers as the majority of the material cannot be reached by any sampling device. The fertilizer or inhibitor in these cases should be sampled during transfer, during the building up of the heap, during the filling of the storage tank, during dispatch or where it is being moved solely for sampling purposes.

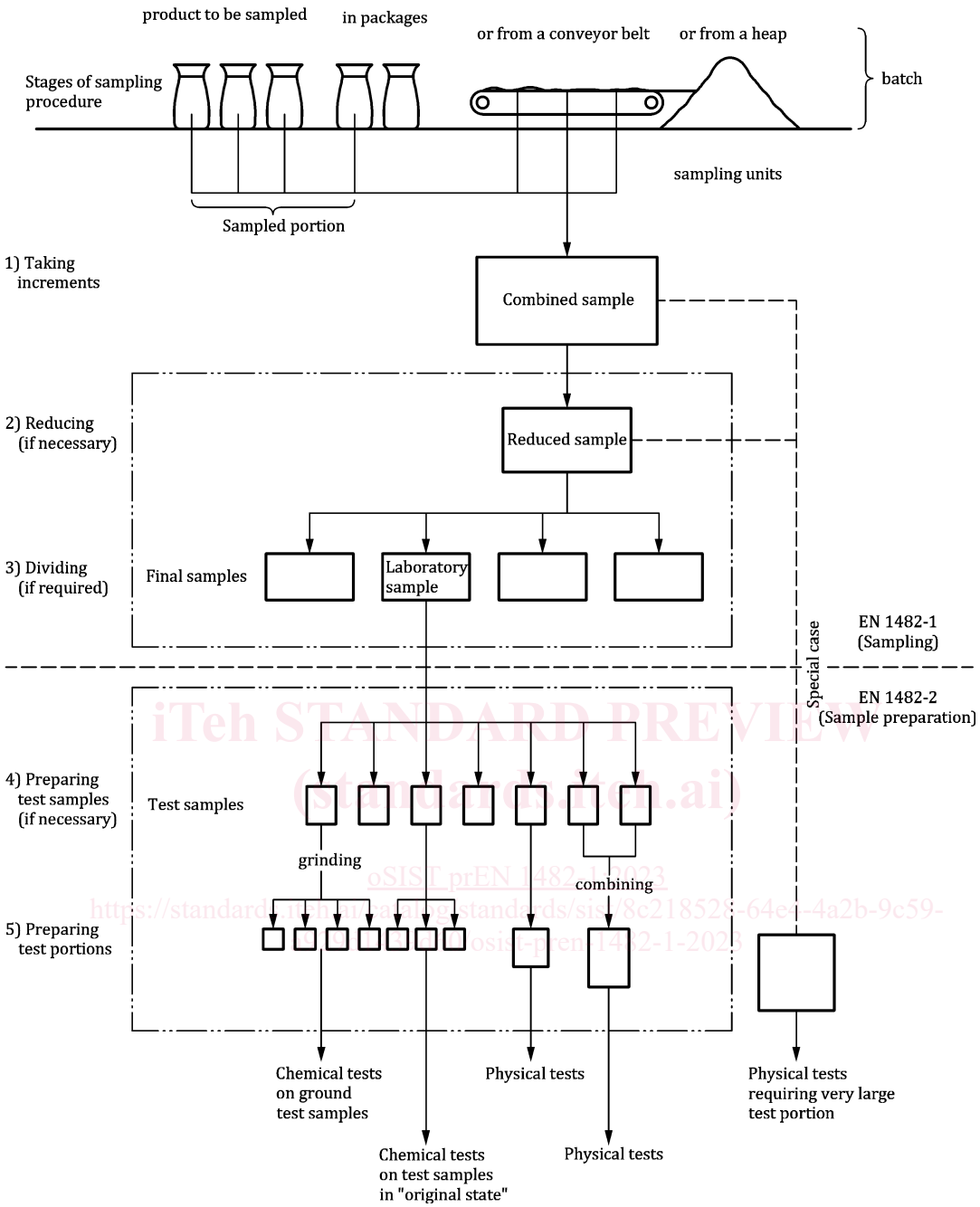

Figure 1 — Schematic diagram of sampling process for solids

1 Scope

This document specifies sampling plans and methods of representative sampling of inorganic fertilizers, liming materials and inhibitors for physical and chemical analysis, from packages and containers up to and including 1 000 kg, in liquid and solid form. This document covers sampling of products in bulk only while in motion.

NOTE 1 The sampling of bulk heaps of specified types of fertilizers is covered in prEN 1482-3. Sampling for detection of microbial presence is covered by prEN 1482-4.

NOTE 2 The term product is used throughout the body of this document and is understood to include inorganic fertilizers, liming materials and inhibitors unless otherwise indicated.

It is applicable to the sampling of batches of fertilizer, liming material and inhibitors supplied or ready for supply to third parties, as such, or in smaller batches, each of which would be subject to local, national or regional legislation.

This document does not cover complete, statistical sampling plans.

This document is also applicable to the blends of products where inorganic fertilizers, liming materials, or inhibitors are the main part of the blend in quantity. If fertilizers, liming materials, or inhibitors are not the main part of the blend, the European Standard for the main part of the blend applies. In case a blend of fertilizing products is composed of parts in equal quantity, the user decides which standard to apply. Special care is needed to ensure that the blend is/stays homogeneous and well mixed when sampled.

NOTE 3 It is the responsibility of manufacturers, importers and sellers, however, to ensure they supply a product that complies with its label declaration at the moment of delivery and fulfils the expectations of the end user at the moment of application.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

EN 1235, *Solid fertilizers - Test sieving (ISO 8397:1988 modified)*

ISO 2602, *Statistical interpretation of test results — Estimation of the mean — Confidence interval*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— IEC Electropedia: available at https://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

3.1
batch
total quantity of material present assumed to have the same characteristics

Note 1 to entry: A batch is produced by the same process at the same time, under the same conditions and labelled in the same manner, and assumed to have the same characteristics to be sampled using a particular sampling plan.

3.2
sampled portion
in relation to sampling for chemical and physical testing, the quantity of material from the same batch from which one representative combined sample is taken

Note 1 to entry: In some instances, the batch and the sampled portion will be the same.

3.3
sampling unit
defined quantity of material having a boundary, which can be physical or hypothetical

Note 1 to entry: An example of a physical boundary is a container. An example of a hypothetical boundary is a time interval for a flow of material.

3.4
incremental sample
quantity of material taken from a sampling point

3.5
combined sample
combination of all incremental samples taken from one sampled portion

3.6
reduction
process of producing a representative smaller mass of material from a larger mass, with the remainder being discarded

3.7
reduced sample
representative part of the combined sample obtained by a process of reduction in such a manner that the mass is at least the mass of the required final samples

3.8
division
process of producing a number of representative smaller portions, approximately equal in mass to each other, from a larger mass

3.9
final sample
in relation to chemical and physical testing, a representative part of the combined sample taken from the sampled portion obtained, where necessary, by a process of reduction

3.10
delivery
quantity of material transferred at one time

3.11
laboratory sample
in relation to chemical and physical testing, a final sample intended for laboratory testing

3.12
ammonium nitrate fertilizer of high nitrogen content
straight or compound solid inorganic macronutrient fertilizer, which is ammonium nitrate-based and contains 28 % or more by mass of nitrogen (N) as a result of ammonium nitrate

4 Sampling plans and quantitative data

4.1 General

Correct sampling is a difficult operation which requires great care. The need to obtain a fully representative sample for both the chemical and physical testing of fertilizers cannot be stressed too much. Sampling plans have been produced to cover a range of quantities of fertilizer and these form the basis of International Standards (see Bibliography).

The sampling plans given in this document are not based on strict statistical principles but samples obtained by following the procedures described in this clause shall be considered to be representative of the original batch or sampled portion.

This clause specifies sampling plans for the evaluation of deliveries of fertilizers as well as statutory control plans which have to be followed in certain circumstances.

For statutory control and the simple commercial evaluation of a small quantity of fertilizer, one final sample is sufficient but this may subsequently be divided into a number of identical samples.

For the commercial evaluation of a large delivery which is supplied for resale in smaller batches a number of samples representing parts of the delivery are required in order to assess the variability of the batch.

For example a delivery of 5 000 t should be treated as at least five deliveries of 1 000 t each and five separate samples should be collected and prepared. The determination in this document is based on a simple relationship between the amount to be sampled and the minimum number of increments to be taken.

The methods of sampling to be used are described in Clause 5.

4.2 Sampling plans

4.2.1 Determination of the number of sampling units which form the sampled portion

4.2.1.1 General

The number of sampling units from which increments are to be taken depends on the size of the batch.

4.2.1.2 Product in packages or containers

In the case of product in packages or containers, the sampling unit is a package and the minimum number of individual packages from which incremental samples are to be taken should be in accordance with Table 1. In this context a package is normally taken to hold no more than 50 kg – larger containers such as Intermediate Bulk Containers (IBC's) should be treated according to the procedure in 5.9 or 5.10. For packages weighing less than 1 kg each, it might be necessary to increase the number taken to ensure a sufficiently large combined sample.

Table 1 — Minimum number of individual packages from which incremental samples are to be taken

Batch size	Minimum number of sampling units
4 or fewer packages	All packages
More than 4 and up to 10 packages	4
More than 10 and up to 400 packages	The nearest whole number above the square root of the number of packages present.
More than 400 packages	20

4.2.1.3 Product in bulk

In the case of product in bulk, the minimum number of sampling units from which incremental samples should be taken depends on the total mass present. The minimum number of sampling units to be sampled should be in accordance with Table 2.

Table 2 — Minimum number of sampling units from which incremental samples are to be taken

Batch size	Minimum number of sampling units
25 t or less	10
More than 25 t and up to 400 t	The nearest whole number above the square root of 4 times the number of tonnes present.
More than 400 t	40

4.2.2 Identification of the sampling units to be sampled

4.2.2.1 Solid and liquid fertilizer in packages or containers

Identify the packages in the batch or sampled portion consecutively and, by using a source of random numbers, select the packages from which incremental samples are to be taken and mark them.

4.2.2.2 Solid and liquid fertilizer in bulk during movement

Where the movement relates to loading or unloading using grabbing equipment such as a crane or automatic shovel loader, the sampling unit is the quantity of material corresponding to one grab. If the movement is a continuous operation such as on a conveyor belt or through a pipe, each sampling unit is made up of a mass of no more than 5 t.

Calculate the number of sampling units present from the total mass and by using a table of random numbers select the sampling units from which increments are to be taken during the movement. Number the sampling units in chronological order of their formation.

Estimate the time taken for the material to pass the sampling place. Divide this time into equal time intervals such that the number of intervals is at least twice the minimum number of sampling units to be sampled in accordance with Table 2 and each sampling unit is not more than 5 t. The time intervals are the sampling units. From these sampling units randomly select the number from which increments are to be taken. Within each of the selected sampling units randomly select a time at which the increment is to be taken.

As there will be some variation in the speed of the belt or the flow in the pipe and the quantity at any one point, it is recommended that the number of sampling units selected is at least 10 % more than the minimum in Table 2.

Automatic mechanical samplers normally work at fixed time intervals. In this case the increments are collected over the whole timescale and cannot be regarded as having been taken randomly. For formal sampling purposes the mechanical sampler must be operated at the selected random times.

4.2.3 Collection of increments

4.2.3.1 General

All incremental samples shall be of approximately the same mass/volume.

4.2.3.2 Solid fertilizer in packages or containers up to and including 50 kg

Take one increment from each of the selected packages (sampling units 4.2.2.1), by the use of a divider (5.6 or 5.7) or by the manual method described in 5.8.

4.2.3.3 Product in intermediate bulk containers

Collect the relevant number of increments by using the method described in 5.9 and/or 5.10.

4.2.3.4 Solid fertilizer in bulk

Collect the relevant number of increments by using one of the methods described in 5.2 to 5.5.

4.2.3.5 Liquid fertilizers

Follow the appropriate procedure described in 5.11.

4.3 Quantitative data

4.3.1 Mass of increments

Increments should normally be of at least 250 g each. For blended fertilizers and for liming materials coarser than 80 % passing 0,315 mm the minimum mass of each increment should be 500 g. For packages weighing 4 kg or less, the entire contents are taken as the increment.

4.3.2 Mass of single combined/reduced samples

Combine and mix all the collected increments. When necessary, reduce the combined sample as described in Clause 6, so that the final mass for chemical testing is at least 2 kg and for physical testing at least 4 times the maximum amount required for the physical test method.

When sampling ammonium nitrate fertilizers of high nitrogen content for testing of detonation resistance, the quantity shall be 75 kg.

When sampling ammonium nitrate fertilizers of high nitrogen content for oil retention and other tests, the quantity shall be 4 kg.

4.3.3 Mass of multiple combined samples

Combine and mix all the collected increments for one sample before reduction to final samples. Each sample shall have at least a final mass equal to 4 times the maximum amount required for testing. Repeat this procedure for each sample.

4.3.4 Mass of final sample

The mass of each final sample for chemical analysis shall be at least 500 g. For physical testing the mass is dependent on the test(s) to be carried out.

Final samples of ammonium nitrate fertilizers of high nitrogen content for testing shall be as follows:

(a) For chemical analysis to check against declared content 500 g;

(b) For porosity (oil retention) testing minimum 1 kg;

(c) For resistance to detonation testing shall be 25 kg.

Final samples of ammonium nitrate fertilizers of high nitrogen content shall be kept at a temperature between 0 °C and 25 °C.

5 Incremental sampling methods

5.1 General

Packages of up to and including 50 kg in mass may be sampled by a process of reduction (see 5.6), starting with the total contents of the package, or by spear sampling from the selected packages but the latter only

when the product is uniform or a single chemical (such as urea, ammonium nitrate or ammonium sulfate) and the sampling is only for chemical analysis. Intermediate bulk containers are best sampled by the method described in 5.9. All packages and IBC's may be sampled by emptying the contents as in the method described in 5.8.

Mechanical sampling devices, if installed in a transfer system, can be used to collect increments, provided they have been tested for the absence of bias (see Annex A) and the timing of the incremental samples can be controlled manually.

The sampling apparatus shall be clean, dry and inert (i.e. fabricated of materials which will not affect the characteristics of the fertilizers to be sampled).

All sampling operations should be carried out in such a way as to minimize changes to sample properties, e.g. moisture content.

5.2 Solid fertilizer in bulk being moved by conveyor belt – Stopping the belt method

5.2.1 General

The sample is taken from a conveyor by stopping the belt.

Taking a representative sample from a consignment of fertilizer by sampling from a conveyor by stopping the belt is time-consuming and interrupts the loading or unloading process considerably. The method should, therefore, only be used if no other more convenient method is available.

NOTE This sampling technique is also used as a reference method to assess the accuracy of other techniques or apparatus.

WARNING — This sampling method involves contact with machinery which is normally in motion. It is essential that precautions be taken so that there is no possibility of the conveyor starting up while the increments are being taken. An override start/stop button should be provided at the point of sampling.

The sampler shall be able to reach the whole cross-section of the belt without undue physical strain. The position for sampling should be made as safe and convenient as possible, for example by using a suitable platform.

5.2.2 Principle

Stopping of the belt conveying the fertilizer. Insertion of two parallel rigid sheets into and at right angles to the stream of fertilizer and to the axis of the conveyor belt. Removal of the material between the sheets as an increment.

5.2.3 Apparatus

Two parallel rigid sheets, shaped to the characteristics of the trough of the belt, sufficiently long to project beyond the sides of the belt by about 500 mm and sufficiently wide for the upper edge to be at least 50 mm above the top of the fertilizer on the belt. It is recommended that a metal frame be made to carry the rigid sheets. This frame can then be placed across the belt in a single operation. Failing this, two marks should be made on the supporting structure on each side of the belt so that the sheets can be inserted in the same places each time.

5.2.4 Procedure

Stop the belt at the times selected as described in 4.2.2.2. Once the belt has stopped, insert the two parallel rigid sheets at a sufficient distance apart to give an increment of at least 1 kg as follows:

a) if the conveyor belt is horizontal, insert the sheets vertically downwards into the stream of fertilizer;

b) if the conveyor belt is inclined, insert the sheets quickly, at right angles to the stream, so as to avoid any backflow.

Push any fertilizer obstructing the insertion of the sheets as follows:

c) in the case of the downstream sheet, into the sample;

d) in the case of the upstream sheet, out of the sample.

As quickly as possible, completely remove the material between the two parallel rigid sheets into a suitable closed container.

Remove the sheets and make sure that nothing has been left on the belt which could cause damage further down. Restart the belt.

Repeat the process for each increment.

5.3 Solid fertilizer in bulk – Mechanical sampling whilst in motion

5.3.1 General

Mechanical sampling devices installed in a fertilizer handling system are a convenient means of collecting samples providing the timing of the taking of the incremental samples can be controlled manually to allow randomness in sampling times. A number of different types are available and this document does not recommend any particular type over another. All might be suitable provided they have been shown to be capable of operating without bias. Before any samples are taken by the device for control purposes, it should be checked for bias using the procedure described in Annex A.

The Annex A bias check test is applicable to any form of mechanical sampling device installed at some point in a bulk handling system, providing that either the fertilizer passes along a conveyor belt, before or after the device, or it is subsequently packed in bags in order that a reference collection can be made.

The mechanical sampling device may be used for the collection of samples for chemical analysis as well as for physical testing.

5.3.2 Procedure

Obtain increments by operating the mechanical sampling device at the times selected as described in 4.2.2.2.

5.4 Solid fertilizer in bulk – Manual sampling from falling stream

WARNING — Manual sampling from bulk fertilizer in motion should only be undertaken when the operations can be performed safely.

5.4.1 Principle

Representative increments are taken by means of randomly timed cuts of the stream.

5.4.2 Apparatus

To sample a free-falling stream as shown in Figure 2, a stainless steel sampling cup shall be used as shown in Figure 3. The length of the cup should be at least three times the depth of the falling stream to be sampled and the edges of the opening shall be thin to ensure a clean cut. The minimum capacity should be 500 g, the maximum capacity should be 5 kg. The width of the active opening of the cup shall be at least three times the maximum diameter of the particles of the product to be sampled.