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Fertilizers and liming materials — Sampling and sample preparation —

Part 2: Sample preparation

Engrais et amendements minéraux basiques — Échantillonnage et préparation de l'échantillon —

Partie 2: Préparation des échantillons

Reference number
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Contents

Page

Foreword ..... **iv**
Introduction ..... **v**
1 Scope **1**
2 Normative references **1**
3 Terms and definitions **1**
4 Principle **2**
5 Apparatus **2**
6 Procedure **3**
6.1 General 3
6.2 Preparation of test samples in their original condition 3
6.3 Further preparation of test samples which are to remain in their original condition 3
6.3.1 General 3
6.3.2 Preparation of test portions for physical testing 3
6.3.3 Preparation of test portions for moisture analysis 3
6.3.4 Preparation of test portions for other chemical analyses 4
6.4 Further preparation of test samples for chemical analysis 4
6.4.1 General 4
6.4.2 Use of sample grinder 4
6.4.3 Use of mortar and pestle 4
6.5 Products which are difficult to grind mechanically 4
6.6 Organic matter 5
6.7 Fertilizers comprising several different materials 5
6.8 Fluid fertilizers 5
6.9 Foreign matter 5
6.10 Storage 5
6.11 Taking test portions 5
6.11.1 Solid fertilizers 5
6.11.2 Fluid fertilizers 5
7 Labelling **5**
8 Sample preparation report **6**
Bibliography **7**

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

ISO 14820-2:2016 was prepared by CEN/TC 260, *Fertilizers and liming materials* (as EN 1482-2:2007) and was adopted without modification other than those stipulated below by ISO/TC 134, *Fertilizers and soil conditioners*.

— The EN references (EN 1482-1 and EN 1482-2) have been changed to ISO references (ISO 14820-1 and ISO 14820-2).

— The definition in 3.2 have been modified slightly to align it with that in ISO 8157:2015. ISO 8157 has been added to the Bibliography.

— Clause 5 has been renumbered.

— In 5.4 (formerly 5.5) and 6.4.2, notes have been changed to full text.

ISO 14820 consists of the following parts, under the general title *Fertilizers and liming materials — Sampling and sample preparation*:

— *Part 1: Sampling*

— *Part 2: Sample preparation*

Introduction

This part of ISO 14820 covers the following aspects of sample preparation, derived from the International Standards and documents indicated but presented in a simplified and condensed form. The titles of these International Standards are given in the Bibliography.

— Reduction and preparation of samples for analysis: ISO 7410, ISO 7742, ISO 8358 and EEC 77/535 (superseded by Regulation (EC) No 2003/2003);

— Sampling reports: ISO 5306 and EEC 77/535 (superseded by Regulation (EC) No 2003/2003).

ISO 14820-1 covers the sampling of fertilizers and liming materials.

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

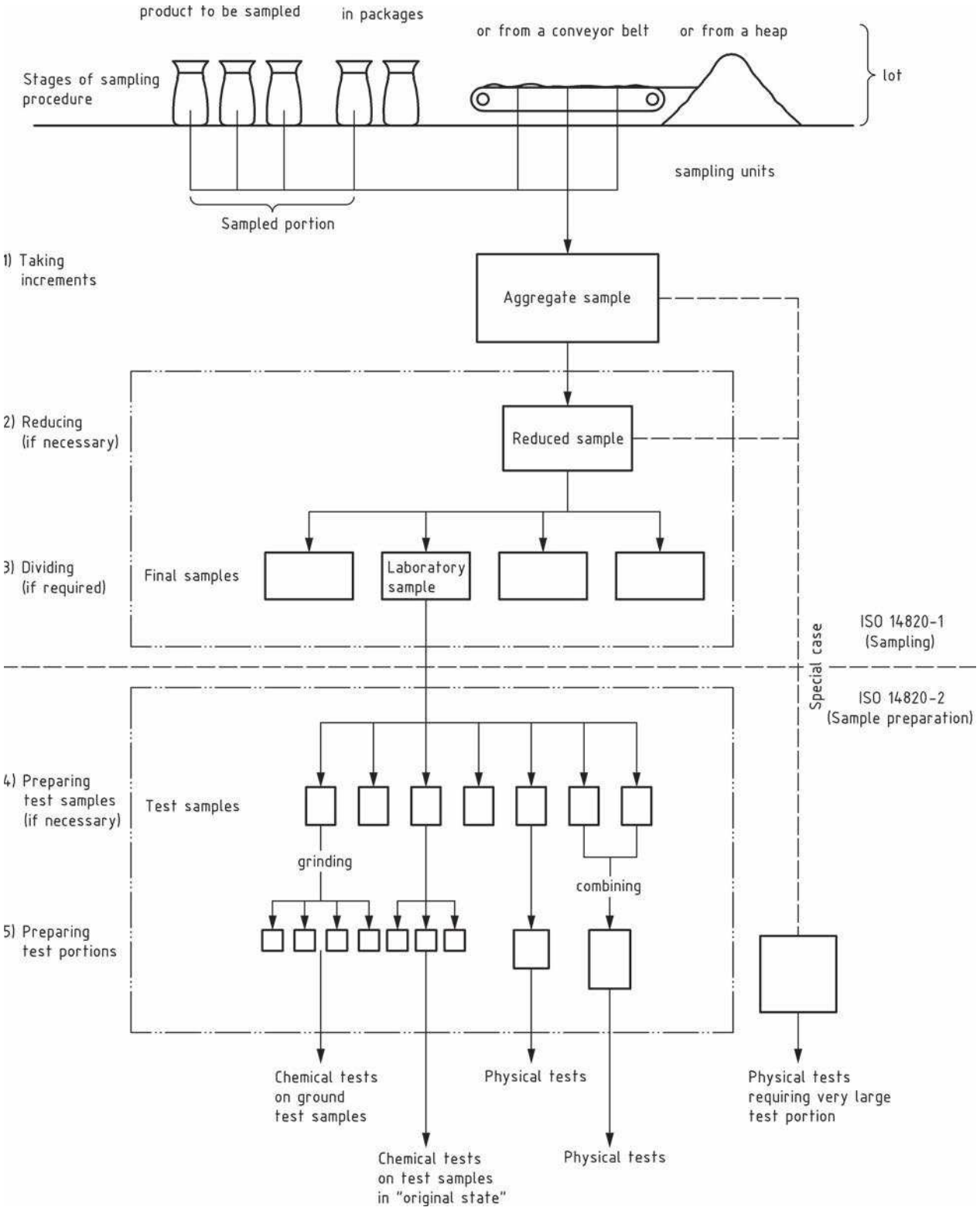

Figure 1 — Schematic diagram of sampling process for solids

Fertilizers and liming materials — Sampling and sample preparation —

Part 2: Sample preparation

1 Scope

This part of ISO 14820 specifies methods for the reduction and preparation of samples of fertilizers and liming materials and sets out the requirements for sample preparation reports. It also specifies methods for the preparation of test samples and test portions from laboratory samples of fertilizer for subsequent chemical or physical analysis. It does not cover the preparation of samples for certain physical tests which require test portions of more than 2 kg. It is applicable to all fertilizers.

NOTE The term "fertilizer" is used throughout the body of this part of ISO 14820 and is taken to include liming materials unless otherwise indicated.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 14820-1:2016, *Fertilizers and liming materials — Sampling and sample preparation — Part 1: Sampling*

3 Terms and definitions

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

3.1
division
process of producing a number of representative smaller portions, approximately equal in mass to each other, from a larger mass

3.2
final sample
representative part of the reduced sample or, where no intermediate reduction is required, of the aggregate sample

Note 1 to entry: Often, more than one sample is prepared, at the same time, from the reduced sample (or from the aggregate sample). One or more of these final samples will be used as a laboratory sample or as laboratory samples, while others may be stored for reference purposes.

[SOURCE: ISO 8157:2015, 2.6.6]

3.3
laboratory sample
final sample intended for laboratory inspection or testing

3.4
reduction
process of producing a representative smaller mass of fertilizer from a larger mass, with the remainder being discarded

3.5
test portion
quantity of material taken from the test sample (or if both are the same, from the laboratory sample) and on which the test or observation is actually carried out

3.6
test sample
sample prepared from the laboratory sample and from which test portions will be taken

4 Principle

Reduction and division of the laboratory sample, as necessary, to produce test samples. Preparation of test portions from the test samples by division, with or without previous grinding, or by combination, as appropriate.

5 Apparatus

Apparatus used in the preparation and storage of samples shall be clean and dry and made from materials which will not affect the characteristics of the fertilizer.

5.1 Rotary sample divider, conforming to the requirements specified in ISO 14820-1:2016, 5.6.3, or **riffle divider**, conforming to the requirements specified in ISO 14820-1:2016, 5.7.2.

5.2 Sample grinder, capable of taking the whole sample at one pass and, preferably, totally enclosed. It shall have a screen, or other mechanism without a screen, which allows the ground material to pass through the machine into a collecting vessel and away from the cutters or grinding discs, to avoid over-grinding. In the case of a grinder with screens, the fineness of grind can be adjusted by the fitting of different mesh screens. Grinding shall continue until as much as possible of the fertilizer has passed through the machine.

NOTE If the grinder is of the open type, the moisture content of the fertilizer can change significantly during grinding.

Any machine used for grinding samples as required by this part of ISO 14820 shall be checked for satisfactory performance. Particular points to be checked are:

a) the fineness of grinding achieved;

b) the temperature rise of the material being ground (see 6.3);

c) non-contamination of the sample.

5.3 Mortar and pestle, of suitable material and size.

5.4 Test sieves, conforming to ISO 3310-1, of nominal aperture sizes 1,0 mm, 0,5 mm and 0,18 mm.

In cases where national regulations or the nature of the material require sieves of different aperture sizes, these may be used but the fact should be noted in the sample preparation report.

5.5 Sample containers, made of plastics material and/or glass, or any other material of adequate resistance and fitted with air-tight closures.

6 Procedure

6.1 General

All operations connected with this procedure shall be carried out as quickly as possible to minimize the absorption or loss of water.

6.2 Preparation of test samples in their original condition

Thoroughly mix the whole of the laboratory sample and follow one of the procedures described in ISO 14820-1:2016, Clause 6 to reduce (if necessary) and divide the total mass to obtain the appropriate number of representative test samples, each of about 0,5 kg in mass.

Reject, by random selection, any test samples in excess of those required and place the remaining *N* test samples in some of the air-tight containers (5.5).

NOTE The maximum number of test samples which can be produced by this method depends on the mass of the original laboratory sample. The minimum number of 0,5 kg test samples which is required depends on the nature of the analyses to be carried out and the number of replicates required. In some instances, when only chemical analyses are to be carried out and only a small laboratory sample is available, the whole of this sample is used as the test sample.

6.3 Further preparation of test samples which are to remain in their original condition

6.3.1 General

Test samples in this category include all those for physical testing, those for certain chemical analyses and those which, by their nature, should not be ground.

6.3.2 Preparation of test portions for physical testing

If the mass of the test portion required is greater than 0,5 kg, select at random two or more of the *N* test samples (6.2). Mix these together and, if the mass required is not an exact multiple of 0,5 kg, reduce it to the required size by following one of the procedures described in ISO 14820-1:2016, Clause 6.

If the mass of test portion required is less than 0,5 kg, select at random one of the *N* test samples (6.2) and continue the reduction and division following one of the procedures described in ISO 14820-1:2016, Clause 6 until test portions of the required mass for the test are obtained. During the division process, replicate test portions are obtained and these are suitable for replicate tests without further treatment. Discard any unwanted material.

The representivity of the sample might be lost during this further subdivision.

6.3.3 Preparation of test portions for moisture analysis

Do not grind test portions for moisture analysis if grinding is likely to alter the moisture content of the fertilizer.

NOTE Some types of grinding mill can alter the moisture content during processing. For example: single-pass hammer mills with interchangeable screens do not alter the moisture content; static sample mills of the coffee grinder type tend to reduce the sample moisture content unless the processing time is kept to an absolute minimum.

Cyclone type mills shall not be used as the rapid airflow in the cyclone causes a reduction in moisture content.

If necessary, the size of the larger particles can be reduced by crushing.

Select, at random, one of the *N* test samples (6.2) and carry out reduction and division, following one of the procedures described in ISO 14820-1:2016, Clause 6 until test portions of the required size are obtained.

6.3.4 Preparation of test portions for other chemical analyses

Some chemical analyses have to be performed on test portions taken from samples which have not been ground. Reference should be made to this in the relevant analytical method. Some fertilizers, for example ammonium nitrate, might decompose during grinding and it is essential that these are not ground before starting analysis.

Select, at random, one of the *N* test samples (6.2) and carry out reduction and division following one of the procedures described in ISO 14820-1:2016, Clause 6 until test portions of the required size are obtained.

6.4 Further preparation of test samples for chemical analysis

6.4.1 General

NOTE See 6.3.3 and 6.3.4 for those special cases where the test sample should remain in its original condition.

Take care during the following grinding operations that the temperature of the fertilizer does not rise above 45 °C, to avoid loss of ammonia, etc.

Select, at random, one of the *N* test samples (6.2) and either follow the procedure described in 6.4.2 or, if a suitable sample grinder is not available, that described in 6.4.3, or, if special treatment is necessary, that described in 6.5, 6.6 or 6.7 as appropriate. For fluid fertilizers, follow the procedure described in 6.8.

6.4.2 Use of sample grinder

Grind the test sample in the grinder (5.2) until all the sample has passed through, or for the specified time, depending on the type of grinder. To check that the grinding has been adequate, thoroughly mix a small representative portion of the ground sample and sieve it through the test sieve of aperture size 0,5 mm (5.4) and discard it. If the whole of this portion does not pass through the sieve, return the sample to the grinder and repeat the grinding until satisfactory grinding is achieved.

For mixtures containing one or more very hard components, it might be difficult to grind and homogenize all the components. The procedure described in 6.4.2 should not be used if the over grinding of some of the softer components is to be avoided. In these, and only these, cases two or more parts of the same sample should be prepared.

6.4.3 Use of mortar and pestle

Sieve the total laboratory sample on the test sieve of aperture size 0,5 mm (5.4). Grind the residue on the sieve, using the mortar and pestle (5.3), until all the material passes, without residue, through the sieve.

Grinding beyond the fineness required shall in all cases be avoided where this affects the solubility in various reagents. Carefully homogenize all the samples.

NOTE In this case, the moisture content of the fertilizer can change significantly during the grinding.

6.5 Products which are difficult to grind mechanically

Products with abnormal moisture content or products such as superphosphate can become doughy if ground mechanically. In these cases, crush the fertilizer in the mortar (5.3 so that all the material passes through the test sieve of aperture size 1,0 mm (5.4).

6.6 Organic matter

Some organic materials can be of such a nature that the procedures described above cannot be used (examples of these are: fresh guano, leather, wool and animal residues). In these cases the analyst should use the best practicable means to reduce the material size and to obtain a representative sample and record the method used in the test report.

6.7 Fertilizers comprising several different materials

These fertilizers include materials with marked differences in texture or mechanical properties (hardness, density, etc.). They can be difficult to grind completely, e.g. mixtures of organic and inorganic materials, or they might segregate during handling, e.g. potassium magnesium sulfate. Special procedures are necessary in these cases.

Follow the procedure described in 6.4.2 using the test sieve of aperture size 0,18 mm (5.4).

6.8 Fluid fertilizers

No further preparation of the test samples obtained as described in 6.2 is required.

6.9 Foreign matter

If the test sample contains foreign matter which cannot be ground, remove and weigh this, and allow for it in the results of the analysis. This material should be retained and, if possible, its nature recorded in the test report.

6.10 Storage

Place all the prepared sample from 6.3, 6.4, 6.5, 6.6, 6.7 or 6.8 in one of the clean containers (5.5) and seal it hermetically until required for analysis.

6.11 Taking test portions

6.11.1 Solid fertilizers

Before taking each test portion for analysis, thoroughly mix the whole test sample, e.g. by rolling it on a piece of hard paper or by mixing small portions using a spatula. Form the material into a flattened cone and take the required test portion at random in small increments with the spatula.

6.11.2 Fluid fertilizers

Before taking each test portion for analysis, thoroughly homogenize the whole test sample by shaking, ensuring that the test sample is inverted several times, or by stirring, ensuring there is no remaining deposition. Pour the required test portion into the appropriate vessel.

Continuous mechanical agitation of the sample might be necessary where solids are likely to settle out rapidly. The test portion should be taken during the agitation when possible. If this is not possible, the test portion should be taken as soon as mixing has stopped.

Attention is drawn to the possibility of ammonia loss during mixing. If this occurs, it shall be included in the sample preparation report (see ISO 7103).

7 Labelling

Label all those containers (5.5) into which final portions of the laboratory sample have been placed, in accordance with 6.10.

8 Sample preparation report

All information on the preparation of the sample shall be recorded, for example as a separate report or as a part of the test report. The sample preparation report shall include the following particulars:

a) reference to the method used for preparation, i.e. the number of this part of ISO 14820, i.e. ISO 14820-2 and the appropriate clause(s) used;

b) all information necessary for the complete identification of the sample;

c) any unusual features noted during this preparation;

d) any operation not specified in this part of ISO 14820, or in the standards to which reference is made, or any operation regarded as optional.

Bibliography

[1] ISO 5306, *Fertilizers — Presentation of sampling reports*

[2] ISO 7103, *Liquefied anhydrous ammonia for industrial use — Sampling — Taking a laboratory sample*

[3] ISO 7410, *Fertilizers and soil conditioners — Final samples — Practical arrangements*

[4] ISO/TR 7553, *Fertilizers — Sampling — Minimum mass of increment to be taken to be representative of the total sampling unit*

[5] ISO 7742, *Solid fertilizers — Reduction of samples*

[6] ISO 8157:2015, *Fertilizers and soil conditioners — Vocabulary*

[7] ISO 8358, *Solid fertilizers — Preparation of samples for chemical and physical analysis*

[8] *Regulation (EC) No 2003/2003 of the European Parliament and of the Council of 13 October 2003 relating to fertilisers*, Official Journal L 304, 21/11/2003, P. 0001-0194

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