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Solid fertilizers — Preparation of samples for chemical and physical analysis

Matières fertilisantes solides — Préparation des échantillons pour analyse chimique et physique

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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 8358 was prepared by Technical Committee ISO/TC 134, *Fertilizers and soil conditioners*, Sub-Committee SC 2, *Sampling*.

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Introduction

This International Standard covers the operations necessary to pass from the laboratory sample to the taking of the test portion for a particular analysis or test. Usually, several chemical and/or physical tests will be performed on one laboratory sample and this International Standard describes the preparation of test samples from which the test portions for the individual tests are taken. A schematic diagram showing the sampling process is given in figure 1.

The preparation of one or more laboratory samples from the aggregate sample is described in ISO 7742. Other standards exist, either published or in the course of preparation, which describe sampling techniques and sampling plans to be used in obtaining the aggregate sample from the lot.

It is essential that all the operations described in this International Standard are carried out carefully and exactly so that the representativity of the original sample is preserved throughout.

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Solid fertilizers — Preparation of samples for chemical and physical analysis

1 Scope

This International Standard specifies methods for the preparation of test samples and test portions from laboratory samples of solid 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.

This International Standard is applicable to all solid fertilizers.

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 3310-1 : 1982, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth.*

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

ISO 7410 : 1983, *Fertilizers and soil conditioners — Final samples — Practical arrangements.*

ISO/TR 7553 : 1987, *Fertilizers — Sampling — Minimum mass of increment to be taken to be representative of the total sampling unit.*

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

ISO 8157 : 1984, *Fertilizers and soil conditioners — Vocabulary.*

3 Definitions

For the purposes of this International Standard, the definitions given in ISO 8157 and the following definitions apply.

3.1 reduction: The process of producing a representative smaller mass of fertilizer from a larger mass, with the remainder being discarded.

3.2 division: The process of producing a number of representative smaller portions, approximately equal in mass to each other, from a larger mass with little or no remainder.

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

5.1 Rotary sample divider or riffle divider, satisfying the requirements of ISO 7742.

5.2 Sample grinder

Any machine used for grinding samples as required by this International Standard 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.

The grinder shall be capable of taking the whole sample at one pass and should preferably be totally enclosed. It shall have a screen, or other mechanism without a screen, which will allow 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 may change significantly during grinding.

5.3 Mortar and pestle, of suitable material and size.

5.4 Test sieves, complying with the requirements of ISO 3310-1, of nominal aperture sizes 1,0 mm, 0,5 mm and 0,18 mm.

NOTE — 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 airtight closures (see ISO 7410).

6 Procedure (see figure 1)

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

6.1 Preparation of test samples in their original condition

Mix the whole of the laboratory sample and follow the procedure described in ISO 7742, 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 airtight containers (5.5).

NOTE — The maximum number of test samples which can be produced by this method will depend on the mass of the original laboratory sample. The minimum number of 0,5 kg test samples which is required will depend 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, only a small laboratory sample will be available and the whole of this sample will be used as the test sample.

6.2 Further preparation for test samples which must remain in their original condition

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

6.2.1 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 (6.1). 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 the procedure described in ISO 7742.

If the mass of test portion required is less than 0,5 kg, select at random one of the N test samples (6.1) and continue the reduction and division following the procedure described in ISO 7742, until test portions of the required mass for the test are obtained. During the division process, replicate test portions will be obtained and these are suitable for replicate tests without further treatment. Discard any unwanted material.

NOTE — Representativity of the sample may be lost during this further sub-division and reference should be made to ISO/TR 7553.

6.2.2 Preparation of test portions for moisture analysis

Test portions for moisture analysis shall not be ground because grinding is likely to alter the moisture content of the fertilizer. If necessary, the size of the larger particles may be reduced by crushing.

Select, at random, one of the N test samples (6.1) and carry out reduction and division, following the procedure described in ISO 7742, until test portions of the required size are obtained.

6.2.3 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 decompose during grinding and it is essential that these are not ground before starting analysis. The following fertilizers may be subject to such considerations: calcium nitrate, calcium magnesium nitrate, sodium nitrate, calcium cyanamide, ammonium sulfate, ammonium nitrate with more than 30 % of N, urea, basic slag, natural phosphate partially solubilized, dicalcium phosphate, sintered alumina and calcium phosphate and soft natural phosphate and slow-release fertilizers.

Select, at random, one of the N test samples (6.1) and carry out reduction and division following the procedure described in ISO 7742 until test portions of the required size are obtained.

6.3 Further preparation for test samples for chemical analysis

See 6.2.2 and 6.2.3 for those special cases where the test sample must remain in its original condition.

WARNING — Care should be taken during the following grinding operation that the temperature of the fertilizer does not rise above 45 °C, to avoid loss of ammonia, etc.

6.3.1 General

Select, at random, one of the N test samples (6.1) and follow preferably the procedure described in 6.3.2 or, if a suitable sample grinder is not available, that specified in 6.3.3, or, if special treatment is necessary, that specified in 6.3.4, 6.3.5 or 6.3.6 as appropriate.

6.3.2 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 and sieve a small representative portion of the ground sample 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.

NOTE — For mixtures containing one or more very hard components, it may be difficult to grind and homogenize all the components. The procedure given in this subclause should not be used if the overgrinding of some of the softer components is to be avoided. In these, and only these, cases it will be necessary to prepare two or more parts of the same sample.

6.3.3 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 to the fineness required shall in all cases be avoided where this will affect the solubility in various reagents. Carefully homogenize all the sample.

NOTE — In this case the moisture content of the fertilizer may change significantly during grinding.

6.3.4 Products which are difficult to grind mechanically

Products with abnormal moisture content or products such as superphosphate may 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).

6.3.5 Organic matter

Some organic materials may 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 should record the method used in the test report.

NOTE — Many of these materials may be ground after intense cooling, for example in liquid nitrogen.

6.3.6 Fertilizers comprising several different materials

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

A grinding machine capable of grinding the whole of the sample in one pass to the required fineness is strongly recommended. Follow the procedure specified in 6.3.2 using the test sieve of aperture size 0,18 mm (5.4).

6.3.7 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.

6.3.8 Storage

Place all the prepared sample from 6.3.2, 6.3.3, 6.3.4, 6.3.5 or 6.3.6 in one of the clean containers (5.5) and seal it hermetically until required for analysis.

6.3.9 Taking test portions

Before taking each test portion for analysis, mix the whole test sample well, 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.

7 Labelling

Label all those containers (5.5) into which final portions of the laboratory sample have been placed, following the procedures described in ISO 7410.

8 Sample preparation report

A copy of the sample preparation report should remain with each final portion at all times. Reference should be made to the sample preparation report in any sampling report produced (see ISO 5306).

The sample preparation report should include the following particulars:

a) reference to the method used for preparation, i.e. the number of this International Standard and the appropriate clause(s) used;

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

c) any unusual features noted during this procedure;

d) any operation not specified in this International Standard, or in the International Standards to which reference is made, or any operation regarded as optional.

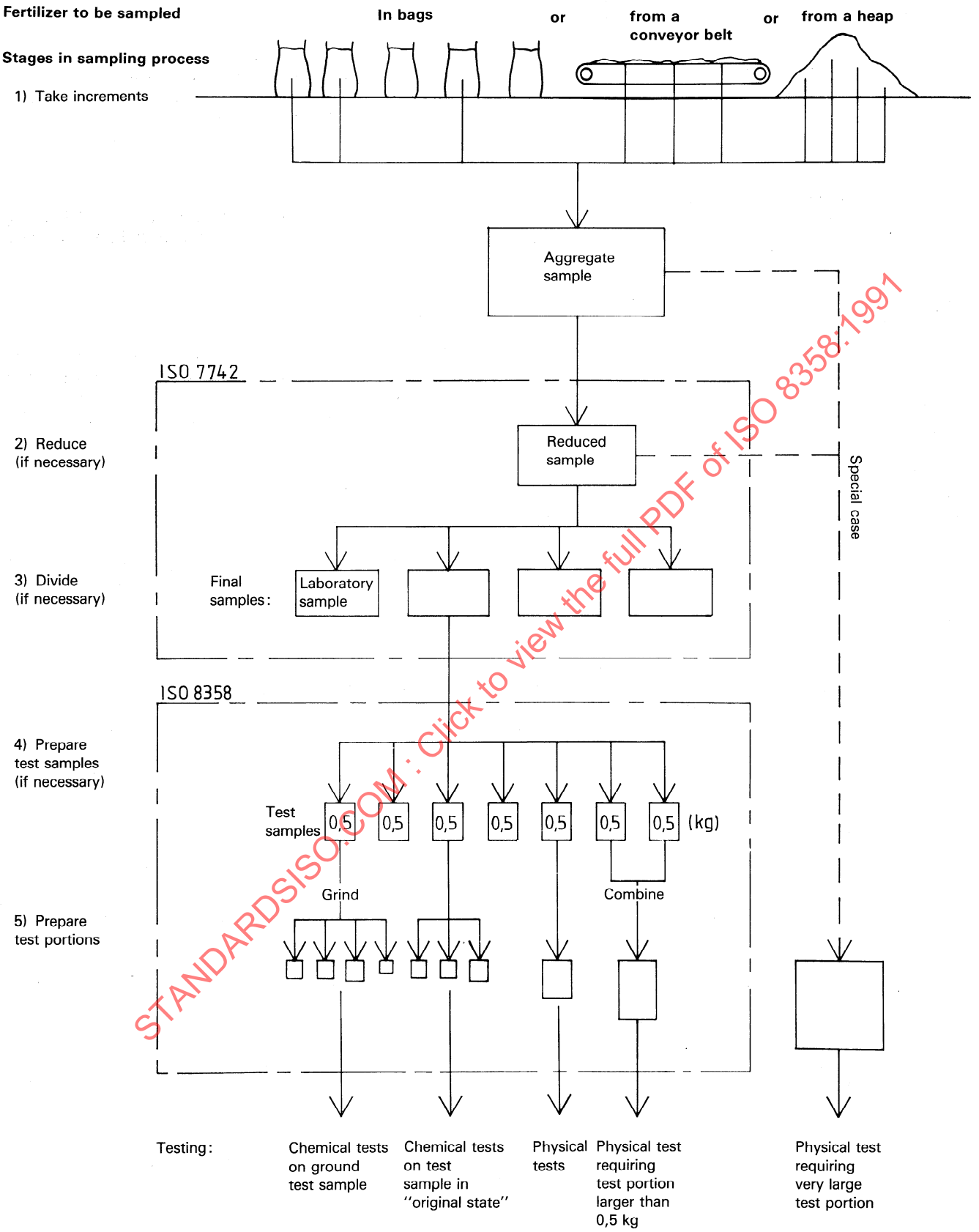

Figure 1 — Schematic diagram of sampling process

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