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Solid fertilizers — Reduction of samples

Matières fertilisantes solides — Réduction des échantillons

Reference number
ISO 7742 : 1988 (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 approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 7742 was prepared by Technical Committee ISO/TC 134, *Fertilizers and soil conditioners.*

Annex A forms an integral part of this International Standard. Annex B is for information only.

Solid fertilizers — Reduction of samples

1 Scope

This International Standard specifies a method suitable for the reduction of a sample of a solid fertilizer to a smaller quantity such as may be used for analysis or for further reduction after suitable comminution.

By choosing suitable equipment, the method is applicable to the reduction of a sample of any mass above a minimum defined by the size and number of particles. The method can be applied to the division of samples into a number of equally representative sub-samples.

Other reduction devices and methods are described in annex A but they are not as accurate as the recommended procedure.

This International Standard does not include information on the methods of obtaining the original sample.

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 listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

ISO 8397 : 1988, *Solid fertilizers and soil conditioners — Test sieving.*

3 Principle

Passage of the material through a rotating sample divider; collection of the fractions, followed by rejection or recombination of some of the fractions.

4 Apparatus

4.1 Basic requirements

Rotating sample dividers are of several basic types. They may operate by collecting increments from a falling stream (cutter type) or by extracting a helical ribbon from a falling cylindrical curtain, such as is created by allowing the fertilizer to fall on to the apex of a cone distributor. In the case of the cutter type, each increment shall consist of a complete cross-section of the stream.

All sample dividers shall conform to the following basic requirements:

a) The effective opening of the cutter or slot shall be at least three times, but preferably five times, the maximum particle size of the fertilizer to be divided. In practice, this means a minimum dimension of at least 15 mm.

b) The divider shall be constructed and operated in such a manner that every particle has an equal opportunity of being included in the sub-sample. Provided that all parts of the stream are sampled in due proportion, an unbiased sample should be obtained.

c) At least 50 to 60 increments shall be taken from the gross sample at each stage of division.

4.2 Test for bias

The sample divider is considered acceptable for a certain type of fertilizer only after it has been installed and has been shown to comply with the test requirements for bias and precision. The tests should be based on the particle size distribution of the fertilizer (see ISO 8397) as this is likely to be the property most sensitive to bias. Thus, it is likely that errors in chemical analysis may themselves arise from errors in the particle size distribution of the fertilizer. At least three sieves should be used in the particle size analysis, giving at least four different fractions, none of which should contain less than 5 % or more than 40 % of the total.

The particle size distribution of two analysis samples collected from each of ten gross samples from the same fertilizer is obtained by sieving through at least three sieves. The mean difference between the percentages retained on the smallest of the sieves used is calculated and used to estimate the errors of sample division. To provide an unbiased estimate, the two analysis samples shall be as independent as possible. To do this, two separate samples shall be obtained at the one stage of division, if necessary by sampling the discarded material.

The estimation of the errors of sample division is itself liable to error. The most satisfactory procedure is therefore to test the results to ensure that the errors are not greater than permitted. For example, if the mean difference between ten duplicate preparations is $|\bar{d}|$ and S_d^2 is the estimated variance of the set of ten differences, $|\bar{d}|$ must be smaller than 0,72 S_d. If two successive sets of ten duplicate preparations satisfy this condition, it may be assumed that the division is satisfactory.

4.3 General description

The sample divider shall be fed from a hopper fitted with one of a series of interchangeable orifices so that the time taken for the sample to flow out is about 1 min.

The hopper may be on the vertical axis of the receiver, feeding via the distributing cone, or off-centre when no such cone is needed.

The standard divider operates at a rotational frequency of 60 min^{-1} but this rotational frequency can be increased up to 360 min^{-1}, the variance of the sample division being reduced as a larger number of increments is taken. However, care is needed to ensure that there is no bias because of larger particles bouncing on the rapidly moving edges of the sample receiver.

Examples of rotating sample dividers are shown in figure B.1

5 Procedure

Follow the procedure specified in 5.1, or 5.2 or 5.3, depending on the mass of the bulk sample.

5.1 Bulk sample is small enough for the apparatus to handle the whole quantity in one pass

5.1.1 Set the rotating sample divider in motion and allow time for it to reach its steady rotational frequency (a period of 15 to 20 s is normally sufficient).

Fill the feed hopper with the sample and open the retaining device at the base of the hopper.

Top up the hopper from the remainder of the sample, making sure that at no time can material run directly from the sample container through the hopper outlet.

Continue until the whole of the sample has been passed through the divider.

5.1.2 Depending on the size of the reduced sample required, take and combine an appropriate number of the fractions produced by the divider and discard the remainder.

5.1.3 Repeat steps 5.1.1 and 5.1.2 on the combined fractions if further reduction is needed.

NOTE — See clause 7 and the note to 5.2.4 before carrying out any further reduction.

5.2 Sample is too large for the apparatus to handle in one pass

5.2.1 Set the rotating sample divider in motion and allow time for it to reach its steady rotational frequency (a period of 15 to 20 s is normally sufficient).

Fill the feed hopper with the sample and open the retaining device at the base of the hopper.

Top up the hopper from the remainder of the sample, making sure that at no time can material run directly from the sample through the hopper outlet.

Continue until the collecting devices are about 80 % full.

5.2.2 Depending on the size of the reduced sample required, take and combine an appropriate number of the fractions produced by the divider and place them in a covered container. Discard the remainder.

5.2.3 Repeat steps 5.2.1 and 5.2.2, adding the selected fractions to the container and discarding the remainder, as often as is necessary to reduce the whole of the bulk sample.

5.2.4 If further reduction of the material collected as in 5.2.1 to 5.2.3 is required, repeat step 5.1 or 5.2 as appropriate.

NOTE — Provided that a rotating sample divider is used throughout for the reduction, it is not necessary to mix the material passed through, before further reduction as in 5.1.3 or 5.2.4.

5.3 Sample is too large for the apparatus to handle in one pass and masses of the fractions produced differ from each other by more than 3 %

5.3.1 Divide the original sample into n equal parts by weighing ($n = M/m$, where M is the total mass of the original sample and m is the capacity of the divider).

5.3.2 Pass the first of the n parts through the divider as in 5.1.

5.3.3 Take a number of the fractions depending on the mass of reduced sample required and the variation between the fractions. Place this (or these) fraction(s) in a covered container and discard the remainder.

5.3.4 Repeat steps 5.3.2 and 5.3.3 on the remainder of the n parts, adding the selected fractions to the container. The masses of the portions collected from the n operations of 5.3.3 should be as nearly as possible equal to each other.

5.3.5 If further reduction of the material collected in 5.3.4 is needed, repeat the procedure described in 5.1, 5.2 or 5.3 as appropriate.

NOTE — Provided that a rotating sample divider is used throughout for the reduction, it is not necessary to mix the material passed through, before reduction as described in 5.1.3, 5.2.4 or 5.3.5.

6 Precautions

6.1 Ensure that all equipment is clean and dry before use.

6.2 Carry out all the operations described in clause 5 as rapidly as possible to avoid loss or gain of moisture.

6.3 Store samples in airtight containers except during the actual process of reduction.

7 Further reduction

The procedures described in clause 5 can be applied to all samples, provided that the final sample mass is greater than the minimum recommended.

Information on the minimum mass of the final sample for various purposes may be found in ISO/TR 7553.

Reduction below this minimum mass is not recommended without comminution. Thus sample reduction below this mass may not be permitted if certain physical tests (for example particle size analysis, bulk density, etc.) are to be carried out. A further reduction should only be carried out after due consideration of the nature of the material and the tests to be performed.

8 Sample division

If it is required to produce a number of equally representative final samples from one sample, the same procedures (clause 5 or annex A) may be followed except that in 5.1.2 or 5.2.2 the fractions are not combined and may be kept separately.

Rotating sample dividers normally produce six, eight or ten fractions. Various combinations and subsequent division can provide virtually any number of sub-samples with a minimum of residue.

Annex A
(normative)

Alternative methods

A.1 General

If a rotating sample divider as described in clause 4 is not available, or cannot be used for lack of power supply, it is still possible to effect reduction of samples. The two procedures described in A.2 and A.3 are known to be less precise and may introduce bias. The extent of this bias will depend on the nature of the fertilizer and the tests which are subsequently to be carried out. For example, the standard deviations for the results of particle size analysis of replicate samples obtained by the three methods of reduction described are in the following approximate ratios:

rotating divider: riffle divider: coning and quartering = 1 : 1,5 : 3,5.

A.2 Riffle divider

A riffle divider is a two-way divider without moving parts. It consists of a hopper having two vertical sides and two sloping sides which run the full length of the riffle divider. The hopper feeds a series of at least 12 rectangular slots, each having a width of at least twice the maximum particle size plus 5 mm. Each slot constitutes an opening to a chute; alternate chutes deliver in opposite directions to two receivers. Riffle dividers are commercially available in many sizes ranging from bench-size to large floor-mounted models.

With the receivers in position, the fertilizer sample is tipped gently into the hopper with a shovel the width of which equals the length of the riffle divider, so that a thin curtain of material falls vertically and equally into all the slots. If a vertical curtain is not maintained then more material will tend to fall into the receiver furthest from the operator, giving unequal and biased samples.

For samples whose volume is less than the capacity of the riffle divider, the contents of one receiver are discarded and those of the other are poured back through the riffle divider into two fresh receivers. Depending on the degree of reduction, this process is repeated, the contents of alternate receivers being discarded. Where a large number of sub-samples is required, the contents of both receivers are reduced separately until the required number of sub-samples is obtained. For greater precision, each sub-sample is further divided and the reduced sub-samples at opposite ends of the 'tree' are recombined.

Samples whose volume is greater than the capacity of the riffle divider should be divided into portions of equal size, each being within the capacity of the riffle divider. Each portion is riffled separately, the contents of one container being retained and those of the others being discarded. The retained material is well mixed and again divided into equal portions, each within the capacity of the riffle divider. The riffling procedure is repeated until the sample size is within the capacity of the riffle divider.

An example of a riffle divider is given in figure B.2.

NOTES

1 Mixing, if necessary, may be effected by passing all the material through the riffle divider three times, recombining it between each pass.

2 Riffling should be carried out as quickly as possible to avoid loss or gain of moisture.

3 Careful handling throughout is necessary to avoid attrition.

4 Feeding the hopper from alternate receivers from alternate directions helps to eliminate biases due to imprecise engineering and handling technique.

A.3 Coning and quartering

This is the simplest of all methods of sample reduction and requires no special apparatus.

A.3.1 Form the fertilizer into a conical heap on a clean, dry, smooth surface.

A.3.2 Flatten the top of the cone and divide the fertilizer into four along two diameters at right angles to each other.

A.3.3 Remove and discard two diagonally opposite quarters, leaving a clean surface in these spaces.

A.3.4 Mix the remaining quarters and repeat steps A.3.1 to A.3.3 until the required mass of sub-sample is obtained.

Annex B
(informative)

Examples of apparatus

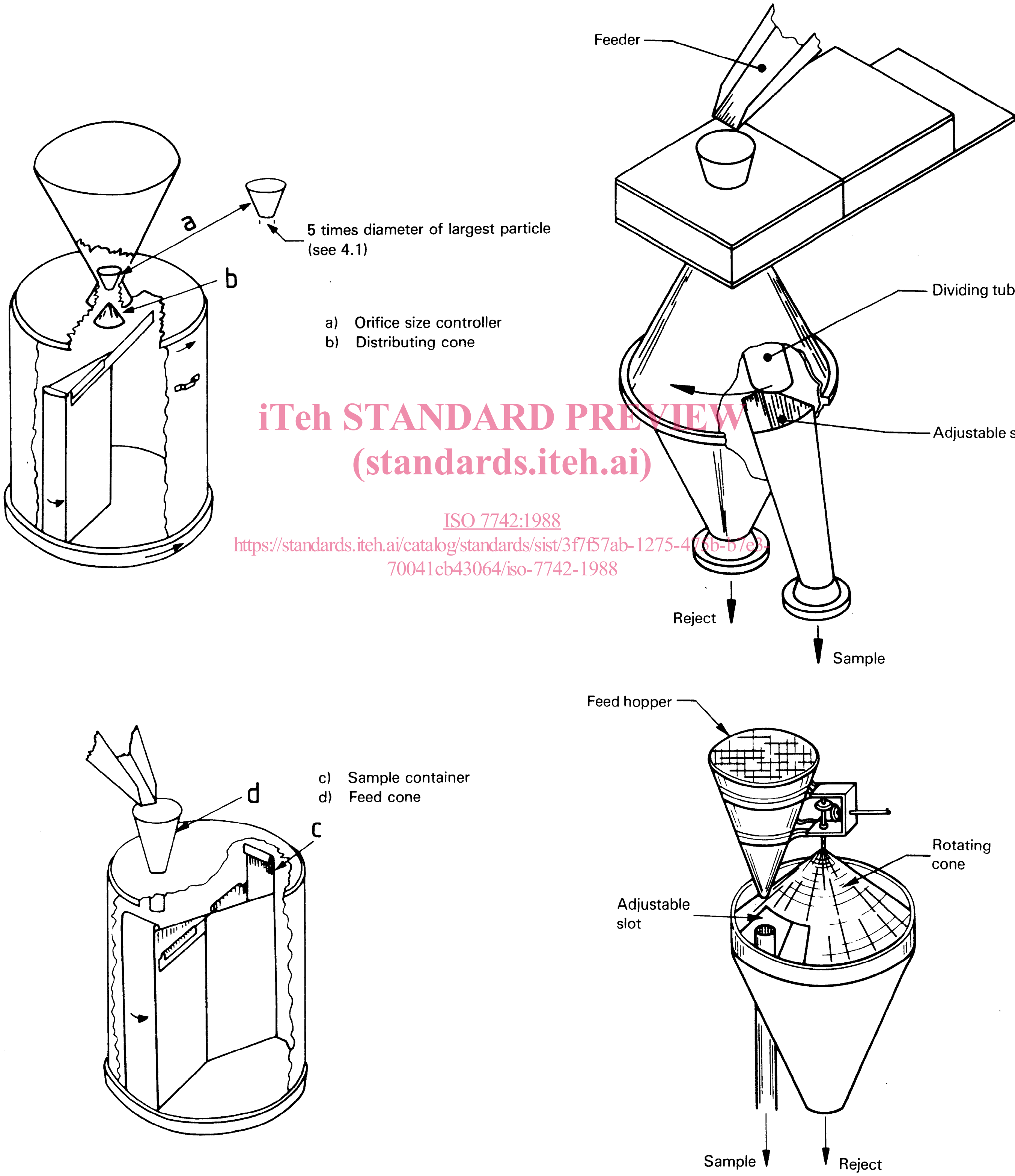

Figure B.1 — Rotating sample dividers

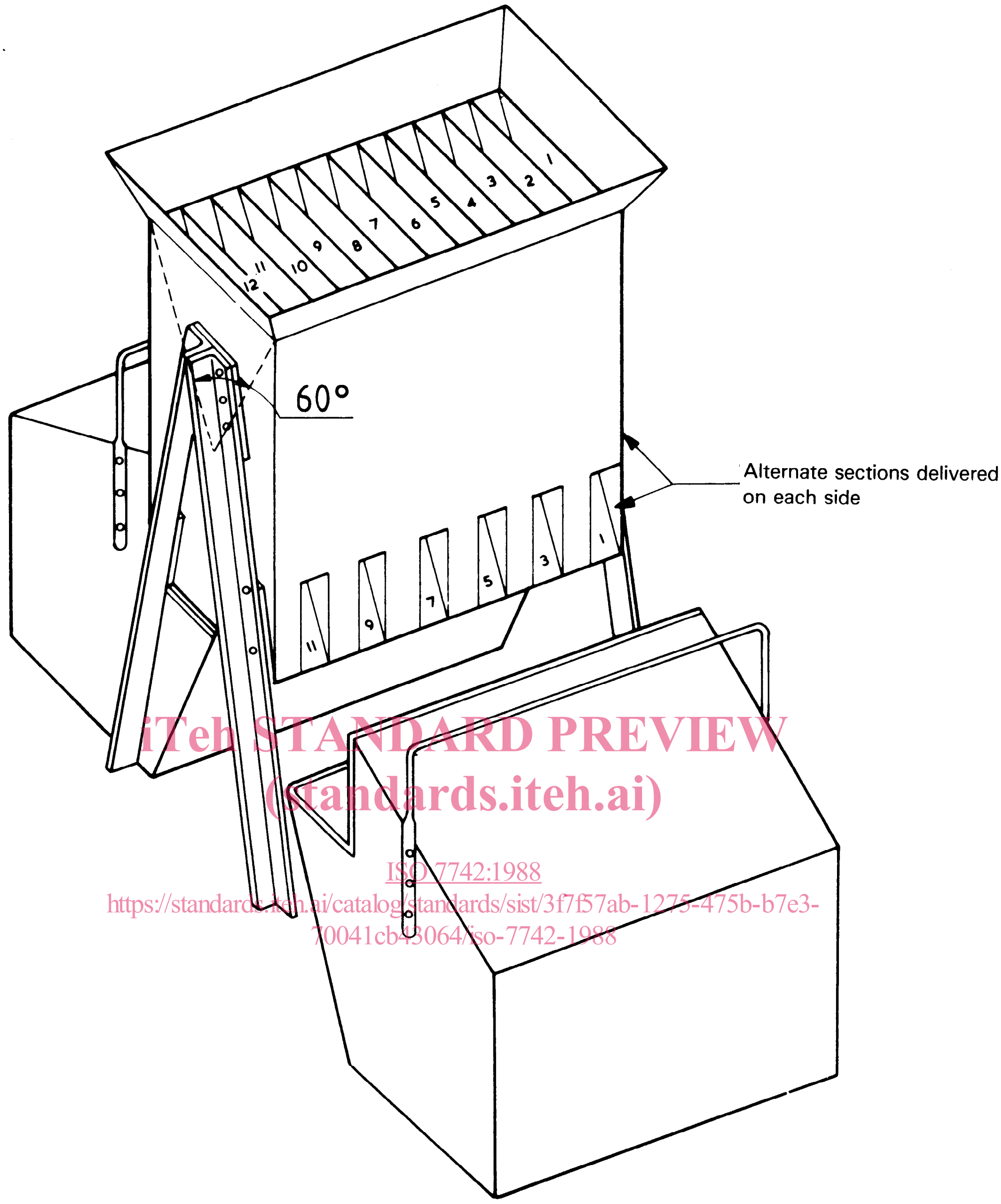

Figure B.2 — Riffle sample divider

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