BRITISH STANDARD

BS EN
1482-1:2007

Fertilizers and liming materials — Sampling and sample preparation —

Part 1: Sampling

The European Standard EN 1482-1:2007 has the status of a British Standard

ICS 65.080

BSI
British Standards

National foreword

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A list of organizations represented on CII/37 can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Compliance with a British Standard cannot confer immunity from legal obligations.

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Düngemittel und Calcium-/Magnesium-Bodenverbesserungsmittel - Probenahme und Probenvorbereitung - Teil 1: Probenahme

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Foreword

This document (EN 1482-1:2007) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by July 2007, and conflicting national standards shall be withdrawn at the latest by July 2007.

Together with Part 2, this document supersedes EN 1482:1996.

This European Standard has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association to provide a means of conforming to Essential Requirements of the Regulation (EC) No 2003/2003 of the European Parliament and of the Council of 13 October 2003 relating to fertilizers.

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

— Part 1: Sampling

— Part 2: Sample preparation

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Introduction

This European Standard (EN 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 EEC 77/535 (superseded by Regulation (EC) No 2003/2003).

— Sampling methods: ISO 3963, and EEC 77/535 (superseded by Regulation (EC) No 2003/2003).

— Reduction: 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).

EN 1482-2 covers the reduction and preparation of samples for analysis.

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. This principle cannot easily be complied with in the case of bulk heaps of solid fertilizers or large storage tanks of fluid fertilizers as the majority of the material cannot be reached by any sampling device. The fertilizer 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.

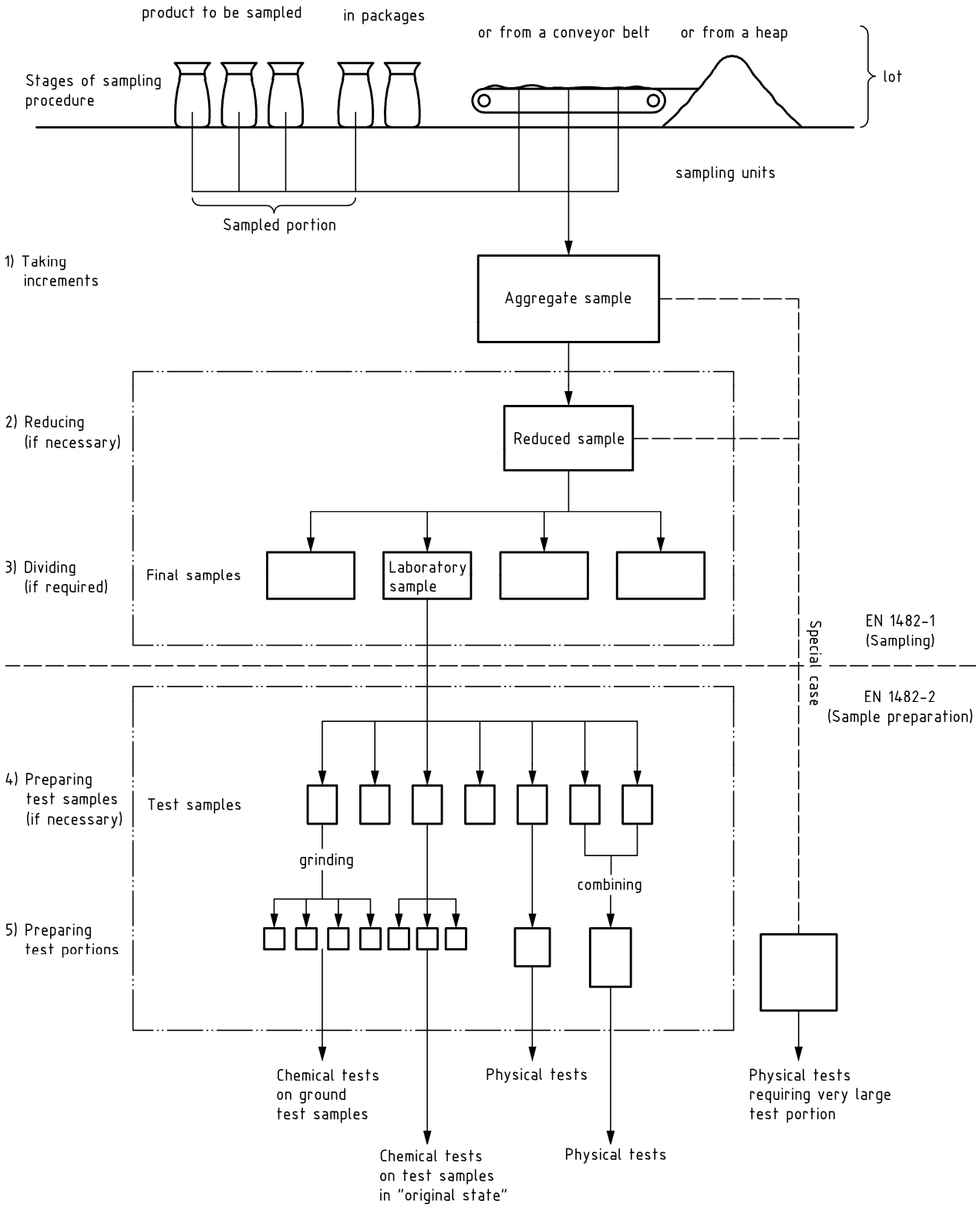


Figure 1 — Schematic diagram of sampling process for solids

1 Scope

This European Standard specifies sampling plans and methods of representative sampling of fertilizers and liming materials to obtain samples for physical and chemical analysis, from packages and containers up to and including 1 000 kg, from fluid products and from fertilizers in bulk provided the product is in motion.

It is applicable to the sampling of lots of fertilizer or liming material supplied or ready for supply to third parties, as such, or in smaller lots, each of which would be subject to local, national or regional legislation. Where legislation so requires, samples are taken in accordance with this European Standard.

NOTE The term fertilizer is used throughout the body of this European Standard and should be taken to include liming materials unless otherwise indicated.

This European Standard does not cover complete, statistical sampling plans.

2 Normative references

The following referenced documents are indispensable for the application 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.

3.1
aggregate sample
combination of all increments from the lot or sampled portion

NOTE The increments may be grouped together in equal numbers in order to form several aggregate samples which can be reduced and analysed separately.

3.2
delivery
quantity of material transferred at one time

3.3
division
process of producing a number of representative smaller portions, approximately equal in mass to each other, from a larger mass

3.4
final sample
representative part of the reduced sample or, where no intermediate reduction is required, of the aggregate sample

NOTE 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 is used as a laboratory sample or as laboratory samples, while others may be stored for reference purposes.

3.5
increment
quantity of material taken from a sampling unit

NOTE An increment may be constituted from a number of sub samples.

3.6
laboratory sample
one final sample intended for laboratory inspection or testing

3.7
lot
total quantity of material, assumed to have the same characteristics, to be sampled using a particular sampling plan

3.8
reduced sample
representative part of the aggregate sample obtained by a process of reduction in such a manner that the mass is at least the mass of the required final samples

3.9
reduction
process of producing a representative smaller mass of fertilizer from a larger mass, with the remainder being discarded

3.10
sampling unit
defined quantity of material having a boundary, which can be physical or hypothetical

NOTE An example of a physical boundary is a container. An example of a hypothetical boundary is a time interval for a flow of material.

3.11
sampled portion
quantity of a material consisting of all the sampling units from which increments are to be taken and having characteristics presumed to be uniform

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 European Standard 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 lot 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 lots a number of samples representing parts of the delivery are required in order to assess the variability of the lot.

NOTE 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 European Standard 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 lot.

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 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 aggregate sample.

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

Lot 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 number of sampling units from which incremental samples should be taken depends on the total mass present. The number of sampling units to be sampled should be in accordance with Table 2.

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

Lot 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 fluid fertilizer in packages or containers

Identify the packages in the lot 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 fluid 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 point.

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.

NOTE 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 legislative purposes the mechanical sampler shall 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 Fluid 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 aggregate/reduced samples

Combine and mix all the collected increments. When necessary, reduce the aggregate 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.

4.3.3 Mass of multiple aggregate 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.

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:

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

b) 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 air-tight 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 European Standard 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.

NOTE 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

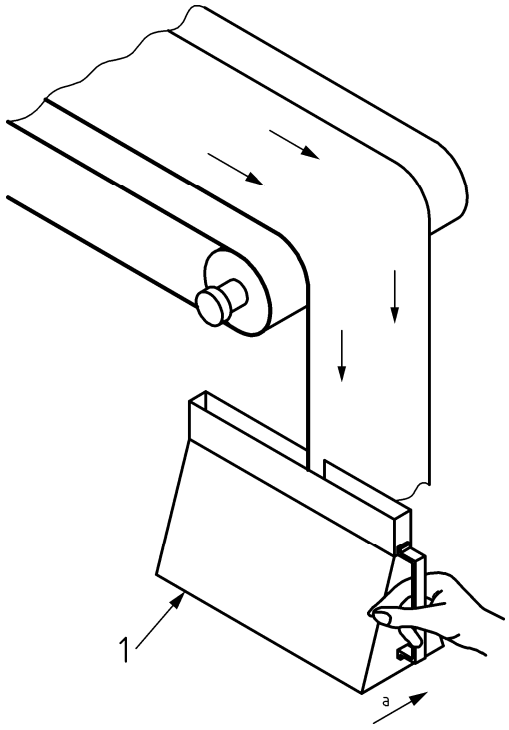
To sample a free-falling stream as shown in Figure 2, a stainless steel sampling cup shall be used as shown in Figure 3. The length of the cup should be at least three times the depth of the falling stream to be sampled and the edges of the opening shall be thin to ensure a clean cut. The minimum capacity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

5.4.3 Procedure

Sample the fertilizer during the free fall by arranging the sampling cup in such a way that it passes horizontally through the falling stream. Ensure that the sampling cup extends completely through the stream (see Figure 2). Ensure that the sampling cup when not in use is protected from the stream.

Pass the cup through the stream at random times within each sampling unit as designated in accordance with 4.2.2.2, throughout the transfer operation. Make sure that passes are made at a uniform speed such that the cup is approximately half filled each time.

Empty the contents of the cup from each pass into a suitable air-tight container.



Key

1 sampling cup
a direction of sampling cup movement

Figure 2 — Method of sampling a free-falling stream

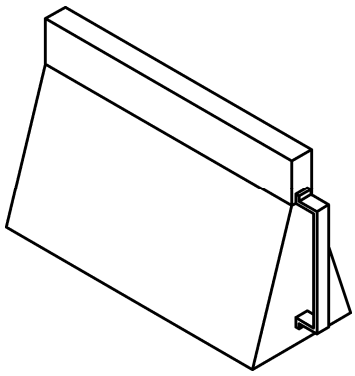


Figure 3 — Example of stream sampling cup

5.5 Solid fertilizer in bulk - Manual sampling method by moving the bulk

5.5.1 General

Where the fertilizer to be sampled is in a bulk static heap and is not to be moved at a time or by a method convenient for any of the other methods of sampling described above, then the heap will need to be moved by the sampling official.

This can be achieved by using a mechanical shovel to move the fertilizer which is then passed either through an overhead hopper, with a controllable bottom outlet and of sufficient volume to take at least one shovelful from the mechanical shovel, or along a conveyor belt.

5.5.2 Procedure

The individual shovel contents are taken as the sampling units. Select the units to be sampled using random numbers. Obtain increments from each selected sampling unit by either of the following means:

a) passing through an overhead hopper. Calculate the time it will take for the selected sampling unit to pass through the hopper. Load the fertilizer into the hopper and obtain increments using the method for sampling IBC's in 5.9. All increments should be of approximately the same mass and stored in an air-tight container until required to form the aggregate sample;

b) loading it onto a conveyor belt and taking increments at times selected as described in 4.2.2.2 using the methods described in 5.2, 5.3 or 5.4.

5.6 Solid fertilizers in packages - Reduction method using a rotating mechanical sample divider

5.6.1 General

This subclause specifies a method suitable for the reduction of a mass of a solid fertilizer to a smaller quantity which forms the incremental sample from the package.

NOTE The method may also be used to prepare reduced samples, final samples or laboratory samples.

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.

5.6.2 Principle

Passage of the material through a rotating mechanical sample divider. Collection of the fractions, followed by rejection or recombination of some of the fractions to give the desired quantity for the incremental sample.

5.6.3 Apparatus

5.6.3.1 General

Rotating mechanical sample dividers are of several basic types. They can operate by collecting sub-samples 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 onto the apex of a cone distributor. In the case of the cutter type, each sub-sample consists of a complete cross-section of the stream.

The sample divider is fed from a hopper fitted with one of a series of interchangeable orifices so that the criteria below can be met.

A standard divider operates at a rotational frequency of about 60 rounds min^{-1} but this rotational frequency can be increased up to about 360 rounds min^{-1}, the variance of the sample division being reduced as a larger number of sub-samples are 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 or because particles are shattered.

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

Examples of rotating sample dividers are shown in Annex B.

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) During reduction, there shall be at least 50 rotations of the cup(s) so that at least 50 increments are taken from the gross sample at each stage of division.

5.6.3.2 Test for bias

A suitable test for bias is given in Annex C.

5.6.4 Procedure

5.6.4.1 General

Follow the procedure specified in 5.6.4.2, or 5.6.4.3 depending on the mass of the bulk sample.

5.6.4.2 Sample small enough for the apparatus to handle the whole quantity in one pass

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

Fill the feed hopper from the contents of the package and open the retaining device at the base of the hopper.

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

Continue until the whole of the contents of the package has been passed through the divider.

5.6.4.2.2 Depending on the size of the incremental sample required, take and combine an appropriate number of the sub-samples produced by the divider. Place in an air-tight container and discard the remainder.

5.6.4.2.3 Repeat the operations described in 5.6.4.2.1 and 5.6.4.2.2 on the combined fractions if further reduction is needed.

5.6.4.3 Sample too large for the apparatus to handle in one pass

5.6.4.3.1 Follow the procedure described in 5.6.4.2.1.

Continue to top up the hopper from the remainder of the contents of the package, making sure that at no time can material run directly from the sample container through the hopper outlet, until the collecting devices are about 80 % (volume fraction) full.

5.6.4.3.2 Depending on the size of the incremental sample required, take and combine an appropriate number of the sub-samples produced by the divider and place them in an air-tight container. Discard the remainder.

5.6.4.3.3 Repeat the operations described in 5.6.4.3.1 and 5.6.4.3.2, adding the selected fractions to the container and discarding the remainder, as often as is necessary to completely empty the package.

5.6.4.3.4 If the masses of the sub-samples produced differ from each other by more than 3 % (mass fraction) follow the procedure described in 5.6.4.3.5.

5.6.4.3.5 Divide the original package contents into *n* equal parts by weighing (*n* = *M*/*m*, where *M* is the total net mass of the original package and *m* is the capacity of the divider).

Pass the first of the *n* parts through the divider in accordance with 5.6.4.2.

Take a number of the sub-samples depending on the mass of the incremental sample required and the variation between the sub-samples. Place this (or these) sub-sample(s) in an air-tight container and discard the remainder.

Repeat these operations on the remainder of the *n* parts, adding the selected sub-samples to the container. The masses of the portions collected from the *n* operations should be as nearly as possible equal to each other.

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 in accordance with 5.6.4.2.3.

5.6.5 Precautions

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

5.6.5.2 Carry out all the operations described in 5.6.4 as rapidly as possible to avoid loss or gain of moisture.

5.6.5.3 Store samples in air-tight containers except during the actual process of reduction.

5.7 Solid fertilizers in packages - Reduction method using a riffle divider

5.7.1 General

If a suitable rotating sample divider is not available, or cannot be used for lack of power supply, it is still possible to obtain incremental samples by other reduction methods. The procedure described in 5.7.2 is known to be less precise and might 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 two methods of reduction described and by coning and quartering (see 6.2.2.3) are in the following approximate ratios:

$$s_r : s_f : s_c = 1{,}0 : 1{,}5 : 3{,}5$$

where

s_r is the standard deviation for a rotating divider;

s_f is the standard deviation for a riffle divider.

s_c is the standard deviation for coning and quartering

5.7.2 Apparatus

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.

An example of a riffle divider is given in Figure 4.

5.7.3 Procedure

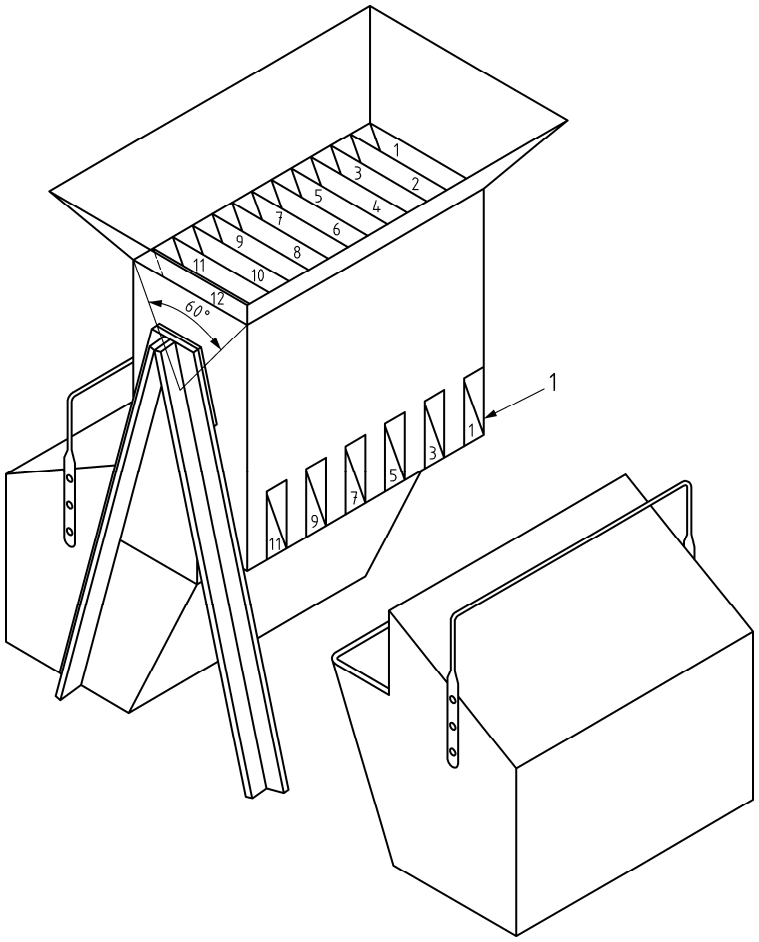
With the receivers in position, tip the fertilizer from the package gently into the hopper right across the width of the riffle divider, so that a thin curtain of material falls vertically and equally into all the slots. If a vertical curtain is not maintained then more material will tend to fall into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

For packages whose volume is less than the capacity of the riffle divider, discard the contents of one receiver and if the quantity is greater than the mass required for the incremental sample pour back the contents of the other through the riffle divider into two fresh receivers.

Depending on the degree of reduction, repeat this process, the contents of alternate receivers being discarded.

Packages 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. Riffle each portion separately, the contents of one container being retained and those of the others being discarded. Mix the retained material well and divide it again into equal portions, each within the capacity of the riffle divider. Repeat the riffling procedure until the sample size is within the capacity of the riffle divider.

Place all incremental samples in an air-tight container until required to form the aggregate sample.



Key

1 alternate sections delivered on this side

Figure 4 — Riffle sample divider

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

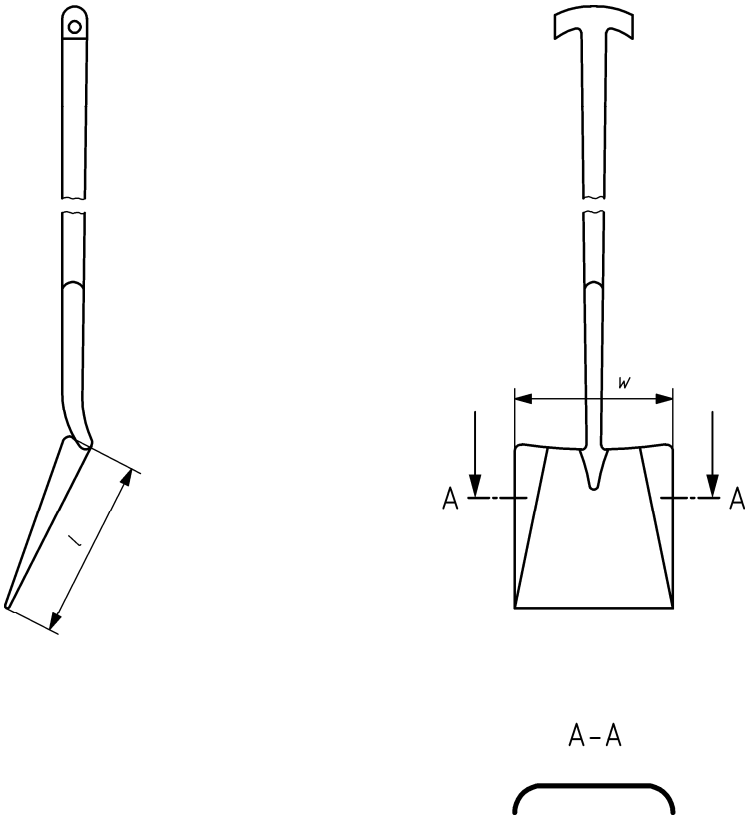
NOTE 2 Riffling should be carried out as quickly as possible to avoid loss or gain of moisture.

NOTE 3 Feeding the hopper from alternate receivers from alternate directions helps to eliminate biases due to imprecise engineering and handling technique.

NOTE 4 Equal masses differing by less than 5 % (mass fraction) should be obtained.

5.8 Sampling of solid fertilizers in packages - Manual method

Empty the contents of each package separately onto a clean dry surface, mixing thoroughly with a shovel (see Figure 5) and remove one shovelful as the incremental sample from that package. Return the remainder to the package. Store samples in airtight containers.



Key

l length of the shovel blade
w width of the shovel blade

Figure 5 — Example of a shovel

5.9 Sampling from intermediate bulk containers (IBC's) by controlled flow

5.9.1 General

This method applies in circumstances where the material is free-flowing and the IBC is not to be reused. Examples would be in a factory where the material can be re-circulated and re-packed or on a farm where the material is about to be used.

5.9.2 Principle

Containers from which incremental samples are to be obtained are selected in accordance with 4.2.2.1 and Table 1 in 4.2.1.2. Each container is then treated as a quantity of loose material and individual sub-samples are taken from each as if they were incremental samples, the number of sub-samples being decided by reference to Table 2 in 4.2.1.3.

5.9.3 Safety

The following personal protective clothing is recommended:

— waterproof boots with toe protection, overalls, gloves, hard hat, goggles, dust mask.

Critical safety points:

a) fixing the straps around lifting device to secure funnel;

b) positioning of all personnel while the lifting device with IBC/funnel is being positioned over the receptacle;

c) ensuring that no person works directly under the equipment;

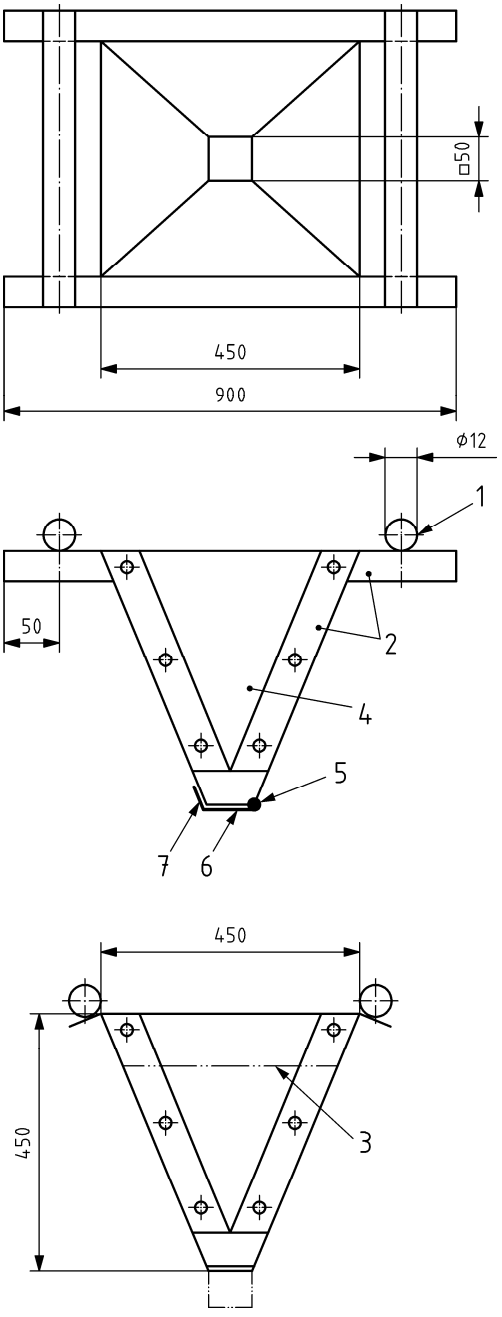
d) ensuring that step ladders if used are attached to a suitable solid object.

5.9.4 Apparatus

5.9.4.1 Flow control funnel

The funnel should be constructed as shown in Figure 6. Dimensions are not critical though it is recommended that the outflow be 50 mm square. This gives a reasonable flow rate of approximately 3 $t \cdot h^{-1}$.

Dimensions in millimetres



Key

1 eye bolts through angle iron. Suspension "U" shackles 10 mm with belting sewn through
2 slotted angle iron reinforcement
3 level of grill and of head of material height line, 100 mm from top of funnel
4 100 mm plywood or appropriate material
5 hinge
6 hinged gate to prevent flow
7 hook and eye to keep gate closed when required

Figure 6 — Flow control funnel (top elevation, side elevation, end elevation)

Draw a marker line round the inside of the funnel approximately 100 mm from the top: The level of material in the funnel should not be allowed to drop below this mark. This maintains the head of material and thereby the consistency of the flow-rate.

A metal grill having bars approximately 20 mm apart fits inside the funnel at marker line level to catch large lumps of material.

Suspension of the hopper shall be by straps of suitable material, i.e. wire, chain or fabric belting. This shall be securely fixed to the 'U' shackles, which in turn are secured to the eye bolts. The suspension material, its fixing method, the 'U' shackles, the eye bolts and the frame shall be capable of supporting at least 1 t.

5.9.4.2 Paddle to control outflow from IBC into funnel

A paddle approximately 400 mm by 200 mm with a 1,2 m handle is needed to cover the hole created in the base of the IBC so that the level of material in the funnel is kept at the desired height.

5.9.4.3 Sample taker

A small open tray approximately 150 mm × 100 mm with 25 mm sides with a 1,2 m handle.

5.9.4.4 A fork lift truck or other similar lifting device capable of lifting an IBC approximately 2,5 m above the level of the receptacle into which the fertilizer is to flow.

5.9.4.5 A suitable set of step ladders (1,5 m working height should be sufficient).

5.9.4.6 A suitable cutting blade on a 1,2 m handle to cut open the base of the IBC.

5.9.4.7 A clean dry plastics container with air-tight lid capable of holding 20 kg to 25 kg of material.

5.9.4.8 Random number tables or generator.

5.9.4.9 Stopwatch

5.9.4.10 Calculator.

5.9.5 Obtaining increments

5.9.5.1 General

To ensure a representative sample, each IBC is treated as a separate loose amount and at least 10 increments should be taken from each IBC. The increments are taken at random times selected as described in 4.2.2.2.

5.9.5.2 Procedure

Determine the number of IBC's to be selected from the lot and from the sampled portion by reference to Table 1 in 4.2.1.2 and select them randomly from the sampled portion.

Lift each selected IBC in turn off the ground and fit the flow control funnel (5.9.4.1) beneath it. This is achieved by passing the suspension straps around the arm of the lifting device, down each side of the IBC and attaching to the funnel suspension points. Leave a gap of approximately 350 mm between the base of the IBC and the top of the funnel to allow access for the cutting blade (5.9.4.6) and the control paddle (5.9.4.2). The gap can be adjusted by further loops of the supporting strap round the lifting arm. Raise the IBC and funnel together and position over the receptacle. Place the plastics container (5.9.4.7) in such a position that the sub-samples taken can be emptied into it as soon as possible without spilling any contents. It is recommended that the same person takes all sub-samples from all the IBC's to be sampled to ensure a consistent mass of sub-sample.

A minimum of two people is required to take the sub-samples. The first person needs to have access to the base of the IBC at the gap left between it and the top of the funnel. This may be by use of the steps or other safe working position. At no time should this person be directly under the IBC. All implements should have handles which allow safe access. The second person should take up a position such that he/she can safely take sub-samples right across the outflow from the base of the funnel using the sample taker (5.9.4.3).

Using the cutting blade (5.9.4.6) make an X cut in the base of the IBC 150 mm to 200 mm long directly above the funnel. Ensure that the cut goes through both the outer material and the inner polyethylene.

Control the flow of product into the funnel by covering the hole with the paddle (5.9.4.2) and stop the flow when the material reaches the marked lines inside the funnel to ensure a consistent head of material and flow-rate.

It is important that all material passes through the funnel - none should be lost over the side.

Open the hinged gate at the base of the funnel and start the stop watch (5.9.4.9). Take sub-samples across the whole flow at the times selected as described in 4.2.2.2.

If for any reason the head of material falls significantly below the lines, shut the gate and stop the stop watch until the problem has been resolved.

Break up any large lumps caught on the grill using the paddle or other suitable equipment so that all the material flows through the funnel. If necessary, stop the flow and the stop watch to allow the breaking up to take place.

Place all the sub-samples in the container (5.9.4.7) forming the increment.

Repeat the process for each IBC from which an increment is to be taken, ensuring that the same number of sub-samples is taken from each.

Mix all the increments (sub-samples) from all the IBC's to form the aggregate sample.

Proceed with reduction and division to final samples (see Clauses 6 and 7).

5.9.6 Precautions

Do not separate the top of the inner polyethylene bag from the suspension handles of the IBC. Doing so will allow the inner bag to slip down and obstruct the hole in the IBC during the final emptying stage. If the inner polyethylene bag is not secured to the handles, use string to ensure it is so secured.

If the product is dusty fix a curtain of fabric or paper to the base of the IBC around as much of the hole as it is practicable bearing in mind the need for paddle access. The curtain should extend downwards to the surface of the material in the hopper thus preventing loss of dust.

5.10 Sampling from intermediate bulk containers IBC's - Manual method

5.10.1 Principle

This method is used when the IBC is to be refilled. The method is extremely physically demanding and should only be used as a last resort. It involves treating the IBC as a package as in 5.8.

5.10.2 Procedure

Proceed as described in 5.8 or, if a suitable large mechanical sample divider or riffle divider is available, 5.6 or 5.7. Because of the bulk of the IBC's at least four shovelfuls should be taken from each IBC.

5.11 Sampling of fluid fertilizers

5.11.1 General

For safety reasons manual sampling (5.11.2.2) is not recommended for fluid products containing free ammonia.

Solutions, slurries and suspensions may be sampled manually provided the product is homogenized (see Annex E for methods of mixing and associated precautions).

There is a risk that portions of multiphase fluids, sampled through narrow tubes or apertures, might not be truly representative. Consequently, it is important to ensure that the internal dimensions of the sampling devices are sufficiently large, i.e. in the region of 50 mm, to avoid this problem.

5.11.2 Apparatus

5.11.2.1 General

The sampling apparatus shall be clean, dry and made from materials which will not affect the characteristics of the fertilizer to be sampled.

NOTE The special properties of fluid fertilizers, including vapour pressure and stratification should be taken into account when choosing sampling apparatus.

5.11.2.2 Manual sampling devices

5.11.2.2.1 A tube that can be introduced vertically into a tank or container and capable of closure at one or both ends. Typical devices are illustrated in Annex D, Figures D.1 and D.2.

5.11.2.2.2 A weighted bottle or other vessel, capable of being lowered into the product, sealed with a device to enable it to be opened at any specific depth. A variant of this provides for gradual filling of the sample bottle as it is lowered from the surface of the fluid to the base of its container. Typical devices are illustrated in Annex D, Figures D.3, D.4 and D.5.

5.11.2.3 Continuous sampling devices

5.11.2.3.1 A sample valve on the storage vessel (illustrated in Annex D, Figure D.6).

5.11.2.3.2 A sample valve on a loading line out of the storage vessel (illustrated in Annex D, Figure D.7).

5.11.2.3.3 A sample valve on an external line through which product in storage is circulated (illustrated in Annex D, Figure D.8).

5.11.3 Procedure

5.11.3.1 Incremental samples from solutions, slurries or suspensions in storage vessels of capacity not less than 1 000 l (1 m^3)

5.11.3.1.1 Sampling at source

If the fertilizer is being withdrawn from a tank and there is a tap of adequate dimensions in the outlet pipe, draw incremental samples from the tap (after first withdrawing twice the volume in the pipe to remove any residues in the pipe) into a clean dry vessel at random time intervals.

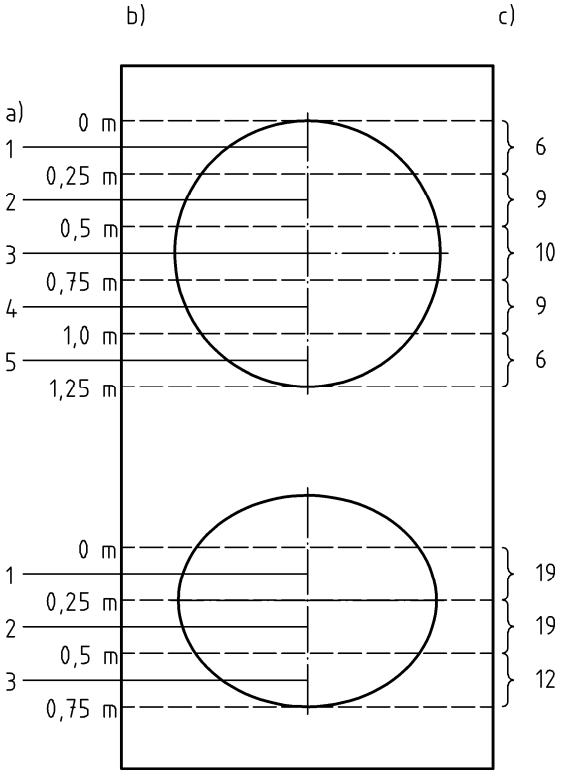
5.11.3.1.2 Homogeneous fluids

Draw about 1 l of fluid from a convenient outlet in the tank (after first withdrawing twice the volume in the pipe to remove any residues in the outlet) into a clean dry vessel.

5.11.3.1.3 Heterogeneous fluids

If it is possible for it to be homogenized, mix the fluid, e.g. as described in Annex E, then take a sample in accordance with 5.11.3.1.2 or from a convenient outlet in a recirculation pipe.

If it is not possible to make the liquid homogeneous or if it is considered that the procedures in 5.11.3.1.1 and 5.11.3.1.2 are not appropriate, sample the contents by lowering an open tube of suitable dimensions (long enough to reach the bottom) (see 5.11.2.2.1) vertically into the tank. Then close one or both ends of the tube and transfer its contents into a clean dry vessel. If sampling by tube is impracticable, take incremental samples from various levels of the tank with a sampling bottle so as to obtain an aggregate sample representative of the lot (see Figure 7). Repeat the operation until a quantity of at least 10 l has been withdrawn. If the tank has a non-uniform cross section then take several portions, as necessary, of each incremental sample at various depths and combine them in a ratio reflecting the variations in cross-sectional area. The total amount taken shall not be less than 10 l.



Key

a) sample point
b) distance from surface
c) proportional volume

Figure 7 — Typical sampling scheme for a full cylindrical tank and a partially filled elliptical tank

5.11.3.2 Sampled portion consisting of two or more containers

Take increments from each, proceeding in the manner described in 5.11.3.1.1, 5.11.3.1.2 or 5.11.3.1.3 as appropriate.

5.11.3.3 Solutions or suspensions in storage vessels of capacity less than 1 000 l

5.11.3.3.1 The number of vessels from which the increments are to be taken should be selected in accordance with 4.2.1.2 and treated as described in 4.2.2.2.

5.11.3.3.2 If the selected vessels each contain not more than 1 l, the entire contents are treated as the increment and should be transferred into a clean dry vessel.

5.11.3.3.3 If the vessels each contain more than 1 l and not more than 1 000 l shake or agitate the selected containers well to ensure uniformity. Then take an approximately equal proportion of fluid immediately from each of the selected containers and transfer into a clean dry vessel.

6 Reduction of aggregate sample

6.1 General

Where the amount of the aggregate sample is larger than the minimum mass required for all the final samples the quantity may be reduced.

Reduction below this minimum mass is not recommended without comminution. Thus sample reduction below this mass might not be possible 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.

6.2 Solid fertilizers

6.2.1 General

Aggregate samples of solid fertilizers may be reduced using the methods described in 5.6 or 5.7 or by coning and quartering.

6.2.2 Procedure

6.2.2.1 Mechanical sampling device

Carry out the operations described in 5.6 but using the whole aggregate sample instead of the package.

If the reduced quantity is still too great then further reduction of the material collected in 5.6.4.2.2, 5.6.4.3.3 or 5.6.4.3.5 is needed, repeat the procedure described in 5.6.4.2 or 5.6.4.3 as appropriate.

6.2.2.2 Riffle divider

Carry out the operations described in 5.7.

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.

6.2.2.3 Coning and quartering

6.2.2.3.1 General

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

6.2.2.3.2 Procedure

Carry out the following steps:

a) form the fertilizer into a conical heap on a clean, dry, smooth surface;

b) flatten the top of the cone and divide the fertilizer into four along two diameters at right angles to each other;

c) remove and discard two diagonally opposite quarters, leaving a clean surface in these spaces;

d) mix the remaining quarters and repeat the procedures described in a) to c) until the required mass of reduced sample is obtained.

6.3 Fluid fertilizers

6.3.1 Apparatus

Reduction of fluids can be carried out using a mechanical sampling device or by placing the aggregate sample into a clean dry and inert vessel which can be well agitated.

6.3.2 Procedure

6.3.2.1 A mechanical sampling device

Carry out the operations described in 5.11.

6.3.2.2 A vessel

Place the aggregate sample into the vessel and agitate to ensure that its contents are homogeneous. Final samples may be poured directly from the vessel.

7 Division into final samples

If it is required to produce a number of equally representative final samples from one aggregate/reduced sample, the sample shall be carefully mixed to obtain a homogenized sample. This shall then be divided into the number of final samples required using one of the methods described in 5.6 and 5.7 without discarding. The final samples shall be approximately the same quantity.

If a rotating sample divider is used to divide the aggregate into final samples it normally produces six, eight or 10 fractions. Various combinations and subsequent division can provide virtually any number of sub-samples. There should be no residue.

8 Practical arrangements for final (laboratory) samples

NOTE The treatment of final samples might be subject to legal requirements in certain countries.

8.1 Containers

Appropriate containers, which shall be clean, dry and impervious to moisture, shall be made of glass and/or plastics materials, or any other inert material of adequate resistance, capable of maintaining the sample in its original condition. The containers shall be fitted with airtight closures.

8.2 Sealing of containers

Secure and seal each container in such a manner that it can be opened only by breaking the seal. Otherwise, the container shall be placed in a sealed, robust package in such a manner that no part of the sample can be removed without breaking the seal of the package. The label (see 8.3) shall be incorporated in the sealing arrangement such that it cannot be removed without breaking the seal.

This procedure applies to samples which are to be kept for official purposes or when required by third parties.

8.3 Labelling of final samples

Each final sample shall be labelled in an identical manner. The label attached to each final sample shall be adequately secured and sealed to prevent its loss. It shall carry at least the following information:

a) final sample number or other distinguishing mark;

b) name of the material and its nominal composition;

c) date and place of sampling.

NOTE Further details of the information required concerning the sample are given in Clause 9.

8.4 Dispatch of the final sample

If it is necessary to submit the final sample to a third party, it can either be delivered by hand or sent by any other appropriate method, provided that the containers are suitably protected against accidental damage and all appropriate precautions are taken for the storage of the sample.

8.5 Storage of final samples

Store all final samples under conditions that do not permit deterioration.

9 Sampling report

9.1 General

In many cases, sampling is carried out in accordance with legal requirements, and, in these cases, a statutory sampling report has to be completed. For all other cases, or for cases where it is considered that insufficient information is provided by the statutory sampling report, the information specified in this clause should be given.

A sampling report should be completed for each sample taken. If the sample is divided into a number of equivalent portions, a copy of the sampling report shall accompany each portion.

9.2 Essential information

The following information shall always be given in the sampling report:

a) the name of the sampler and the department or organization to which he or she belongs;

b) the name, description or designation associated with the fertilizer, and whether it is in bulk or in packages;

c) any declared information on the composition or the fineness of grind of the fertilizer, and, if available, a copy of any labels attached to the original packages containing the product;

d) any lot or consignment numbers for the complete identification of the lot, and, if possible, the date of manufacture or delivery;

e) the quantity of fertilizer sampled (i.e. the sampled portion in terms of mass and/or the number of packages) and its relation to the total amount present;

f) if the fertilizer is in packages, the nature of the packages and the method of sealing;

g) the sampling plan adopted and the number of increments taken (if the sampling was carried out in accordance with a particular standard, its reference shall be given);

h) any relevant observations made during the sampling procedure, including assessment of the condition of the fertilizer and its storage environment;

i) the date, time and postal address of the place of sampling, including, where appropriate, the name of any vessel or the registration number of any vehicle from which the sample was taken;

j) the identification mark or reference number given to the sample by the sampler;

k) the method of sealing the sample containers, with a description of any seals;

l) the names and addresses of the parties to the relevant transaction, for example manufacturer, importer or vendor and purchaser or holder of the sampled fertilizer;

m) the destinations of the final samples and information for the analyst;

n) any relevant safety information;

o) the signature of the sampler and the name and signature of any independent witness or person from whom any of the information given in the report was obtained.

9.3 Additional information

The sampler can, if he or she so wishes, or shall, if instructed by the client, annex to the report any other information not required by 9.2. If the sampler has been so instructed by the client, the latter should supply a detailed list of the items of additional information required.

Annex A
(normative)

Test for bias in mechanical samplers

A.1 Principle

Collection of two series of increments, one using the mechanical device and one using the reference method, from a quantity of fertilizer passing through a bulk handling system and comparison of the means and variances of the particle size distribution of the two samples.

A.2 Apparatus

A.2.1 Rotary sample divider, conforming to the requirements given in 5.6.3.

A.2.2 Sample containers

A.2.3 Set of sieves, conforming to ISO 3310-1, of nominal aperture sizes 4,00 mm; 3,35 mm;

2,80 mm; 2,36 mm; 2,00 mm; 1,40 mm; 1,00 mm and 0,50 mm.

A.2.4 Sieve shaking machine

A.3 Test conditions

Carry out the method of test specified in this annex using a granular fertilizer.

NOTE 1 The use of a blended product might introduce additional sources of variation. If only blended fertilizer is available for this test, collect increments of at least 250 g (see A.4.1.2).

NOTE 2 It might be necessary to check the variability of the product before carrying out this method of test in order to decide whether to take more increments than are specified, or to be able to conduct the test over a shorter period of time.

Carry out the test while at least 20 t and preferably not more than 100 t of fertilizer passes through the mechanical sampling device.

A.4 Procedure

A.4.1 Collection of increments

A.4.1.1 Carry out the operations described in A.4.1.2 and A.4.1.3 concurrently.

A.4.1.2 Using the mechanical sampling device under test, collect at least 50 increments and label them from A1 to A50 etc.

A.4.1.3 Carry out one of the following reference collections:

a) if the fertilizer is conveyed by a conveyor belt feeding to, or extracting from, the mechanical sampling device, collect the same number of increments as collected in A.4.1.2 by the reference method described in 5.2. If possible, synchronize the taking of the increments by the device under test with the taking of the increments by the reference method so that corresponding increments are taken from the same part of the fertilizer bed (see A.5.2 below). For example, if the reference sample is taken after the mechanical device, it is possible to see the gap in the fertilizer bed on the conveyor belt;

b) collect the same number of full containers as the number of increments specified in A.4.1.2 from the same tonnage of fertilizer, at intervals corresponding as closely as possible to the time intervals at which the increments are taken in accordance with A.4.1.2. Reduce each container, in accordance with 5.6 or 5.7, to about 1 kg.

Label the reference increments or containers R1 to R50 etc.

A.4.2 Preparation of samples

Treat each sample from each series in exactly the same way.

Combine the increments into equal groups to give at least 10 individual samples taken by the same method (A.4.1.2 and A.4.1.3). The groups shall be formed of consecutive increments, for example A1 to A5, A6 to A10, ... A46 to A50 etc.

Thoroughly mix each of the individual samples so formed and in accordance with 5.6 or 5.7, reduce each sample to 250 g for particle size analysis.

A.4.3 Analysis of samples

Carry out a particle size analysis on each of the reduced samples, obtained in accordance with A.4.2 by the method described in EN 1235, using four or five sieves from the set (A.2.3), chosen to suit the fertilizer used in the test. Use the same sieves for the analysis of each reduced sample. Record the percentage of fertilizer retained on each sieve.

A.5 Expression of results

A.5.1 General case where the increments taken by the two methods do not correspond exactly to each other

For each series of *n* reduced samples, calculate the mean and the standard deviation for the percentage of fertilizer retained successively on each of the sieves and the percentage passing through the sieve of the smallest aperture used.

For each successive test sieve, calculate the value *t* by means of the following equation:

$$t = \frac{\sqrt{n} \times (\bar{x}_A - \bar{x}_R)}{\sqrt{s_A^2 + s_R^2}} \qquad \text{(A.1)}$$

where

$\bar{x}_A$ and s_A are respectively the mean and standard deviation for the reduced samples obtained using the device (A.4.1.2);

$\bar{x}_R$ and s_R are respectively the mean and standard deviation for the reduced samples obtained using the reference method (A.4.1.3).

Refer to statistical tables such as those in ISO 2602 for the significance of the values of *t* obtained with 2(n – 1) degrees of freedom.

A.5.2 Special case where the increments taken by the two methods correspond exactly to each other

For each pair of reduced samples, calculate the difference between the percentages of fertilizer retained on each sieve in turn and the percentages passing through the sieve of smallest aperture size used.

Calculate the mean ($\bar{d}$) and the standard deviation (s_d) of these differences for each test sieve in turn and calculate the value of *t* by means of the following equation:

$$t = \frac{\sqrt{n} \times \bar{d}}{s_d} \qquad \text{(A.2)}$$

Refer to statistical tables such as those in ISO 3301 for the significance of the values of t obtained with n – 1 degrees of freedom.

A.6 Interpretation of the results

The interpretation of the individual results depends on the precision that is both acceptable and practically feasible. It is to be expected that the most significant bias is shown by the results for the percentages of fertilizer retained on the sieve of largest aperture size and passing through the sieve of the smallest aperture size used for the test, and the least significant bias by the results for the intermediate sieves.

A mechanical sampling device should be rejected if any of the values of *t* for the percentages of fertilizer retained on the sieve of largest aperture size and the percentages of fertilizer passing through the sieve of smallest aperture size are significant at the 95 % confidence level.

The device can be accepted as being without bias if all the values of *t* are not significant at the 95 % confidence level.

In other cases, e.g. where one of the values of *t* is significant at the 95 % confidence level, indicating the possibility of bias, the tests should be repeated on a larger number of samples.

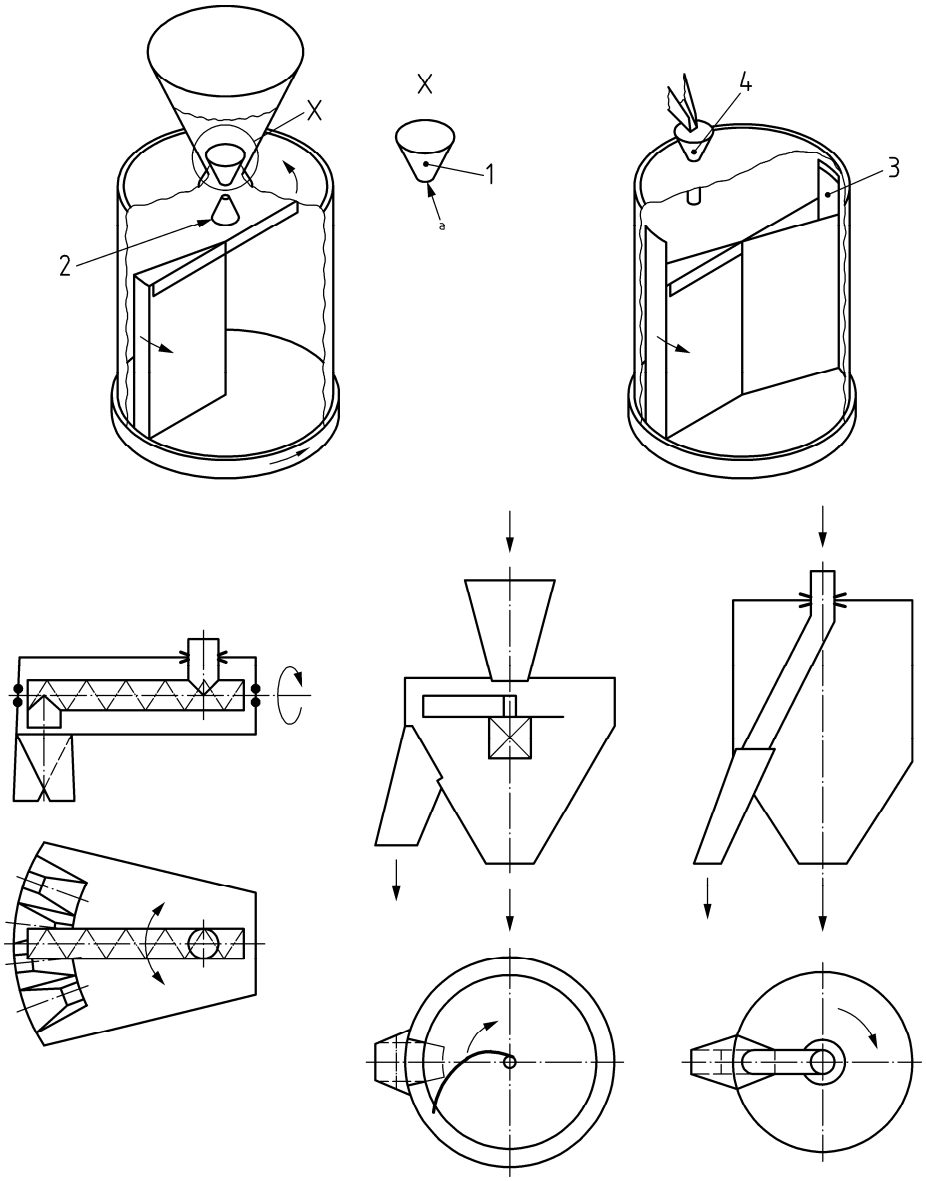
Additional information on the performance of the device can be obtained by comparing the variances s_A^2 and s_R^2 obtained in A.5.1 using the test described in Table G of ISO 2854:1976 (the F-test).

In the special case (A.5.2), s_A^2 and s_R^2 may be calculated in the normal way from the individual results.

If a significantly larger variance is obtained for the mechanical device, this would imply that the device is not reliable.

Annex B
(informative)

Examples of rotating sample dividers



Key

1 orifice size controller
2 distributing cone
3 sampling segment
4 feed cone
a 5 times diameter of largest particle

Figure B.1 — Example 1

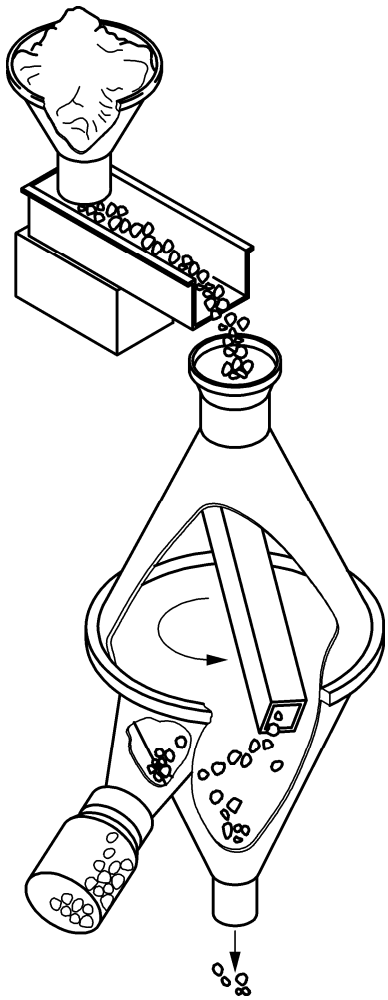


Figure B.2 — Example 2

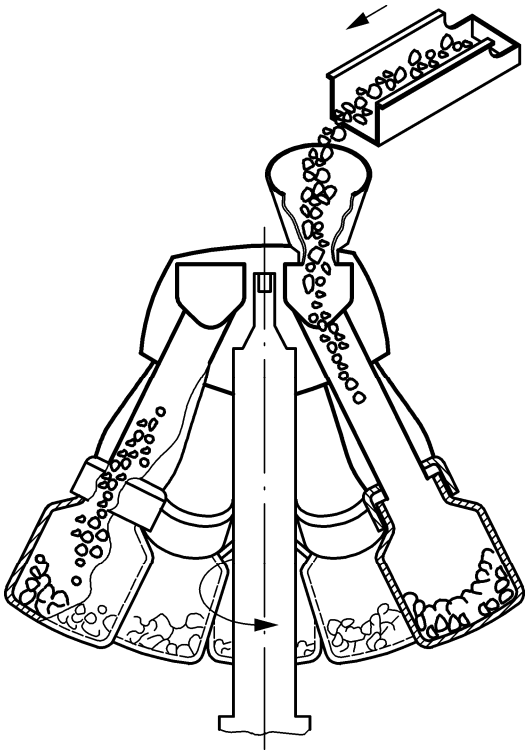


Figure B.3 — Example 3

Annex C
(normative)

Test for bias in a rotary divider

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 EN 1235) as this is likely to be the property most sensitive to bias. Thus, it is likely that errors in chemical analysis might 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.

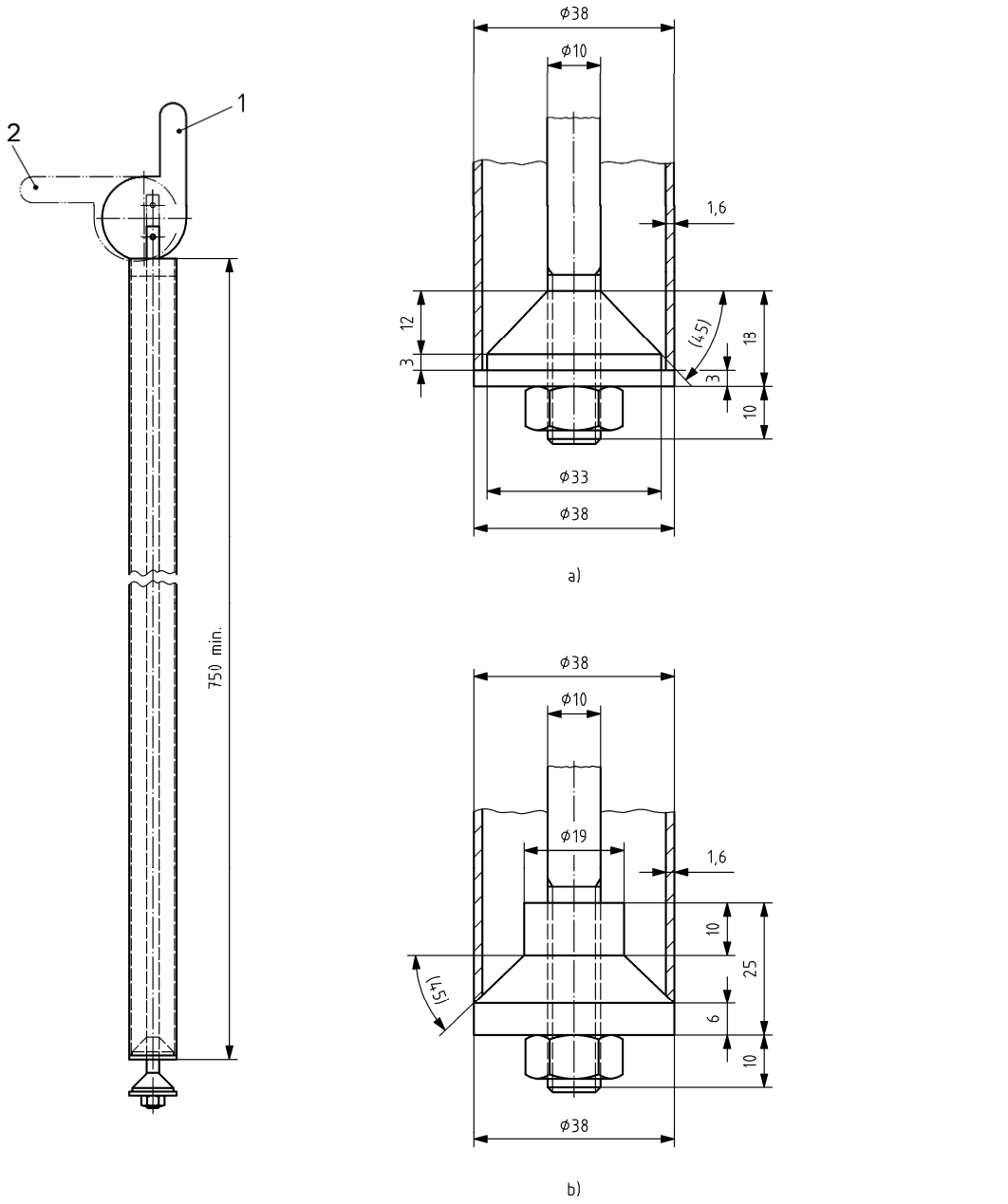
The particle size distribution of two analysis samples collected from each of 10 gross samples from the same fertilizer is obtained by sieving through at least three sieves. The mean difference between the percentages retained on the smallest of the sieves used is calculated and used to estimate the 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 10 duplicate preparations is ldl and s_{d}^2 is the estimated variance of the set of 10 differences, ldl has to be smaller than 0,72 s_{d}. If two successive sets of 10 duplicate preparations satisfy this condition, it may be assumed that the division is satisfactory.

Annex D
(informative)

Examples of apparatus for sampling fluid fertilizers

Dimensions in millimetres

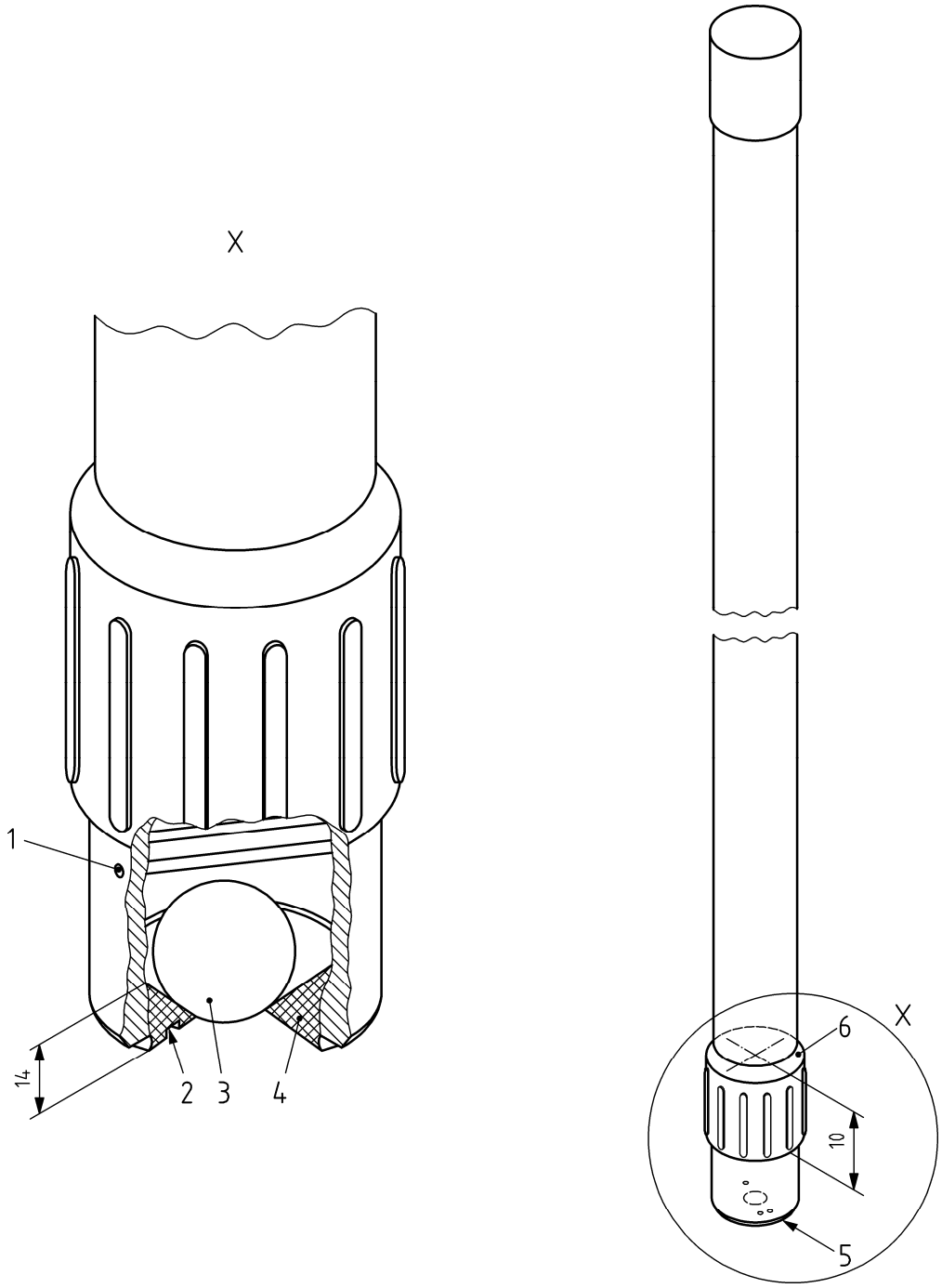


Key

1 hand lever (open position)
2 closed position

Figure D.1 — Typical bottom-valve sampling tube – a) Type A and b) Type B

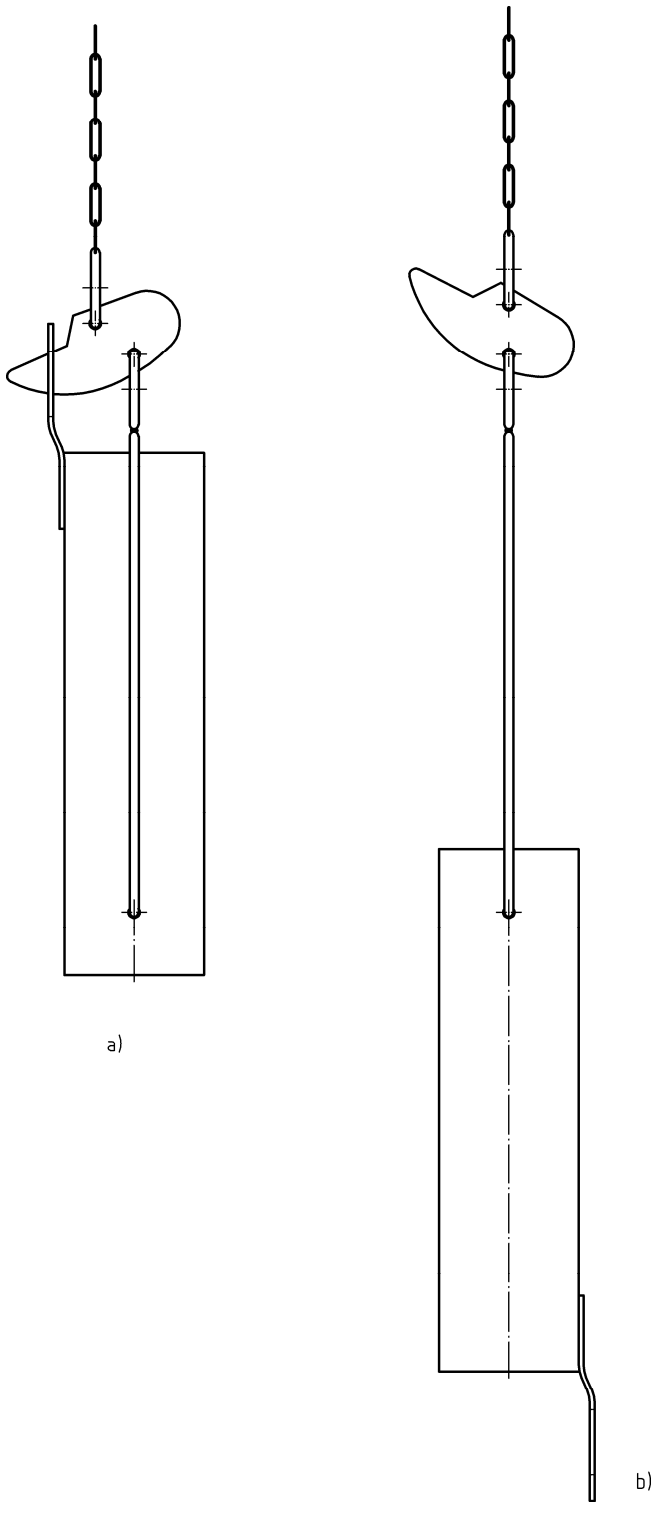
Dimensions in millimetres



Key

1 retaining pin, tight press-fit (two required)
2 groove for lug wrench (spanner)
3 25 mm stainless steel ball
4 threaded plug-form seat
5 19 mm diameter
6 PVC coupling

Figure D.2 — Texas tube

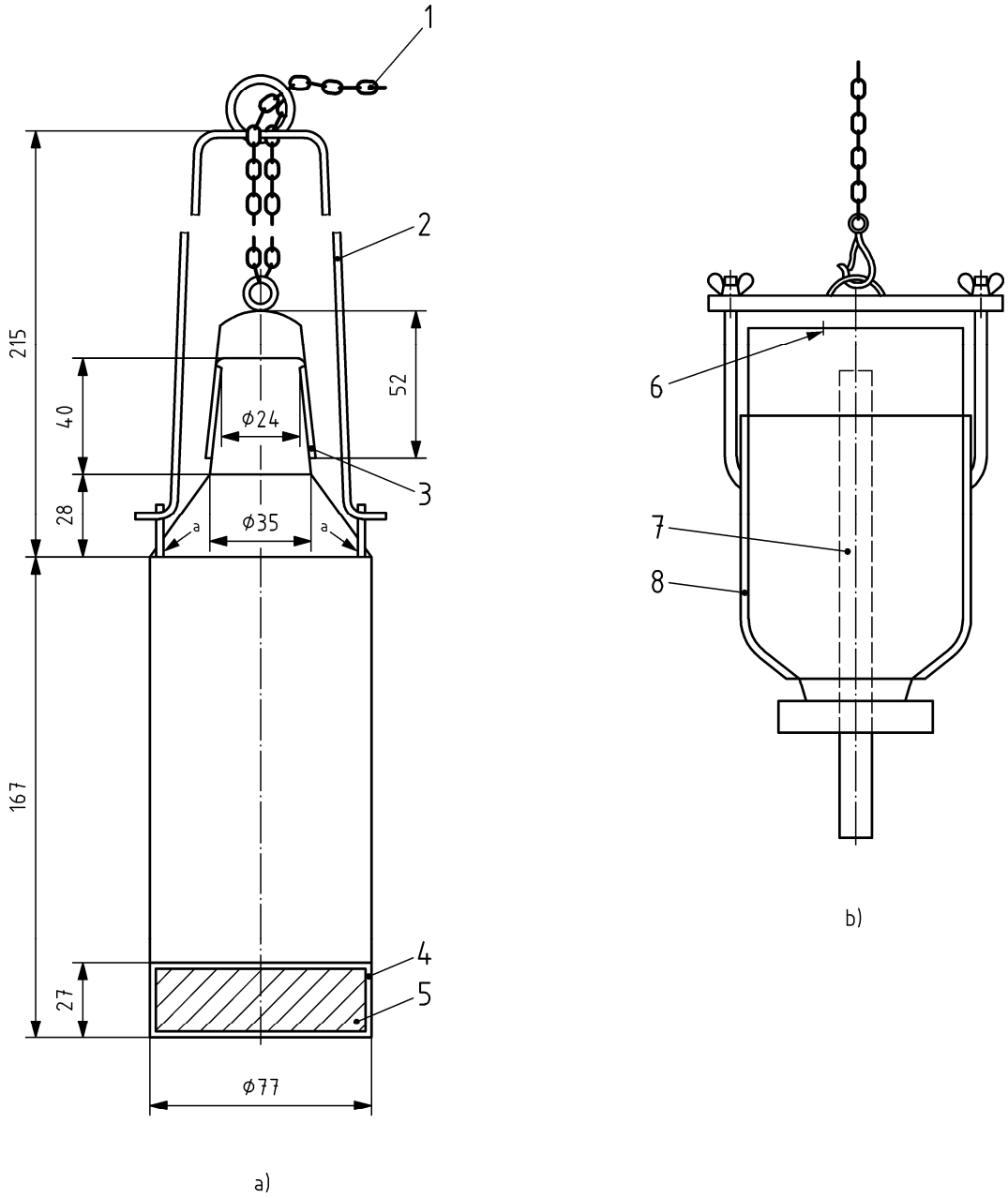


Key

a) lowering position
b) filling position

Figure D.3 — Tipping dipper

Dimensions in millimetres

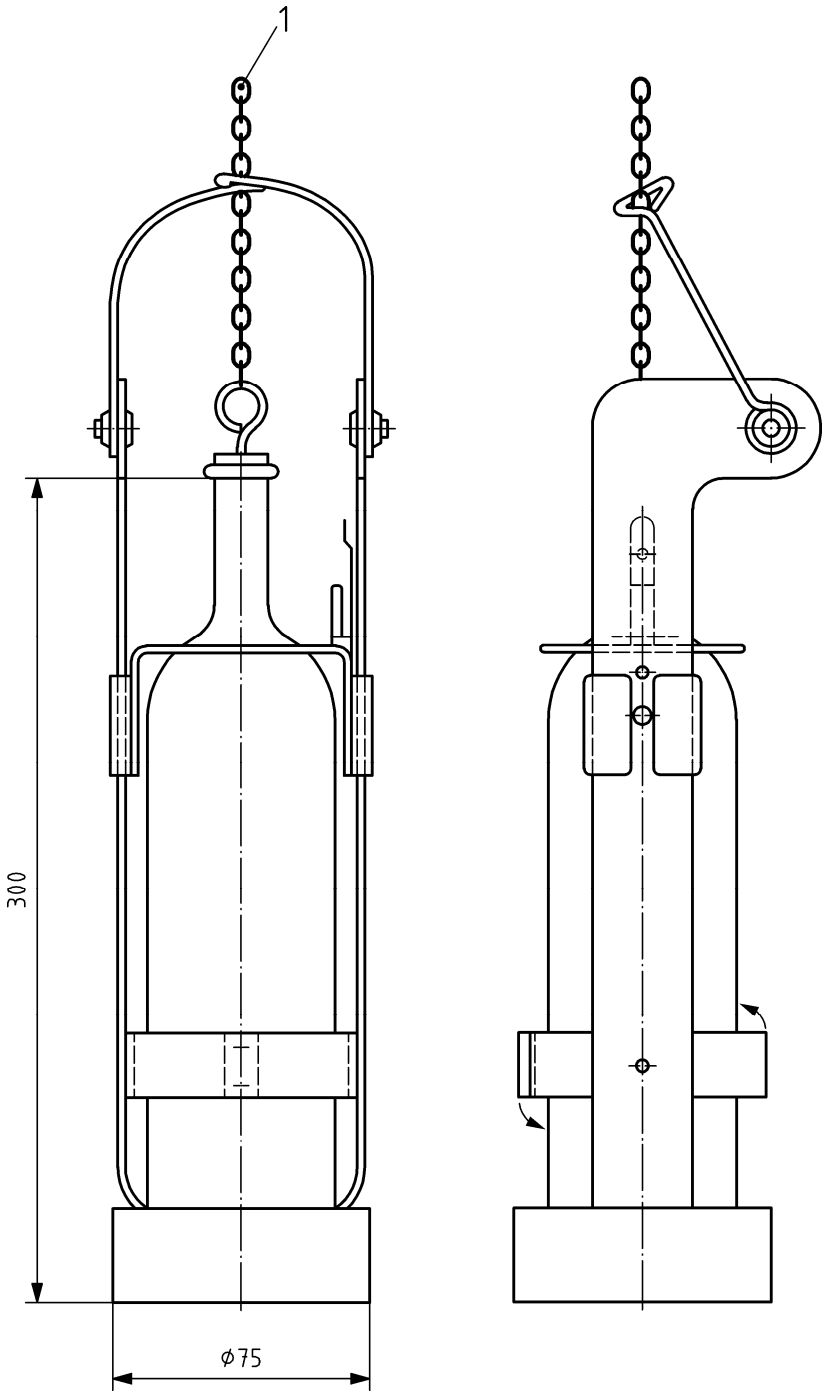


Key

1 graduated chain
2 wire handle
3 conical cup of tight fit
4 liquid tight false bottom
5 lead weight approximately 1 kg in sealed compartment
6 air hole 3 mm diameter
7 fluid intake tube 7 mm diameter
8 stainless steel jacket

Figure D.4 — Typical weighted sampling cans

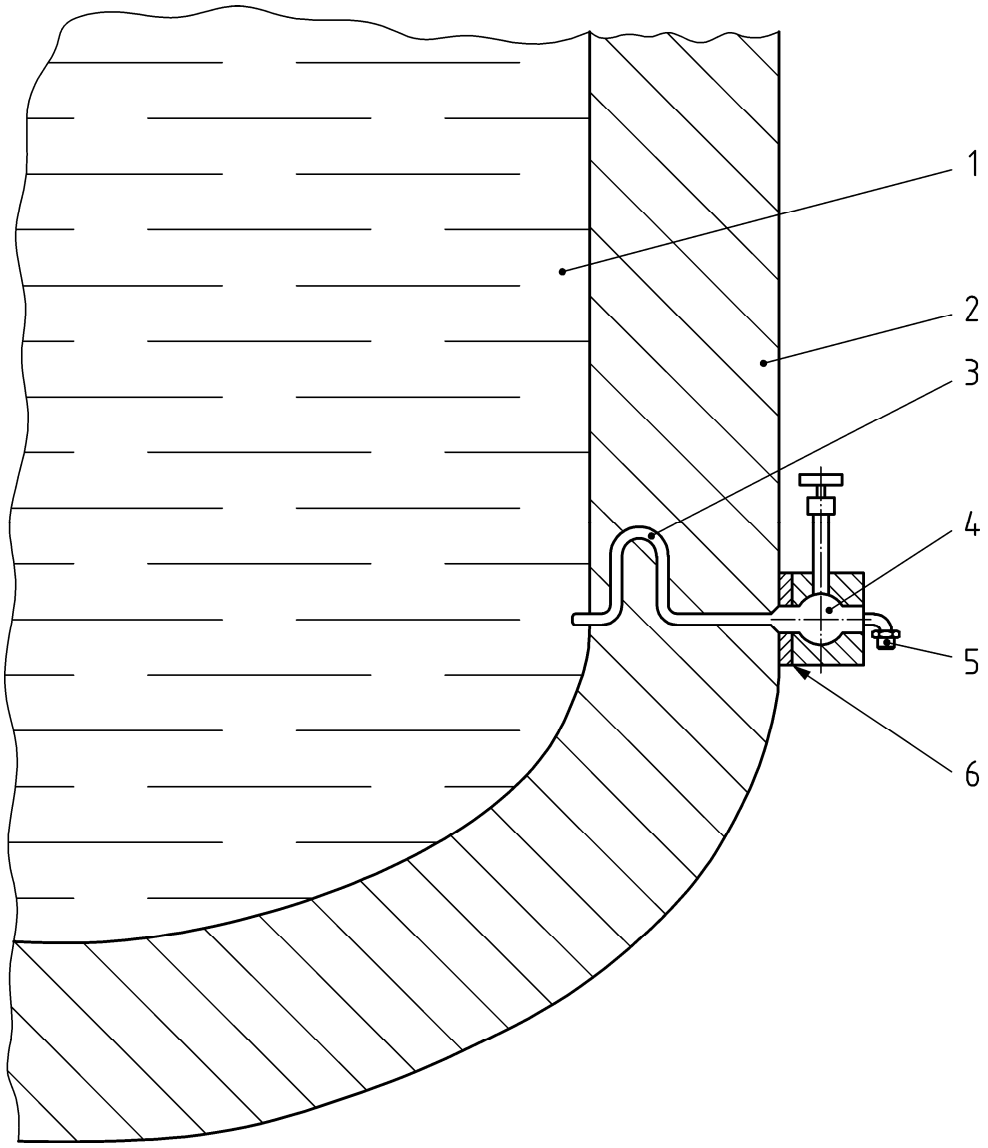
Dimensions in millimetres



Key

1 graduated chain

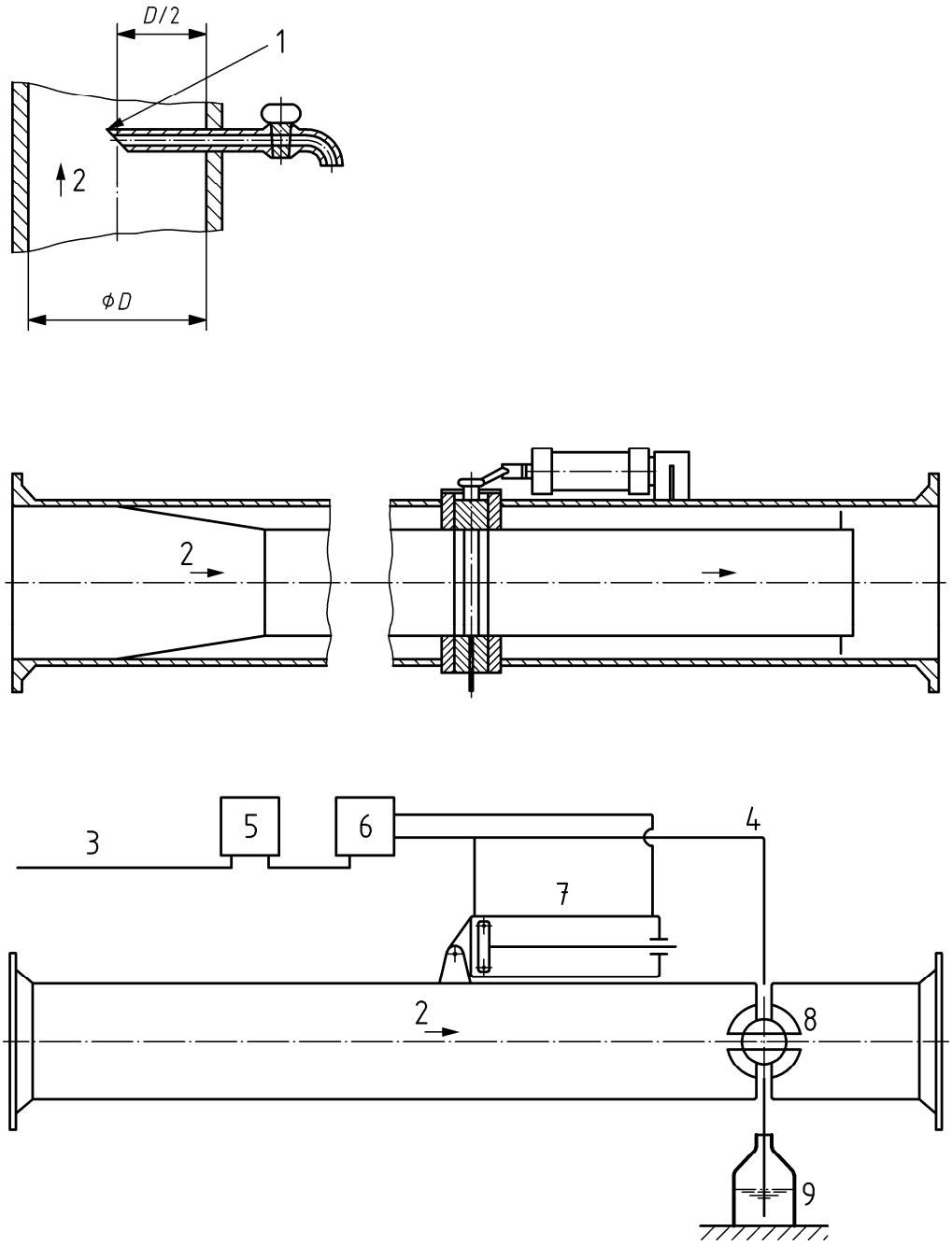
Figure D.5 — Typical sampling cage



Key

1 liquid
2 lagging
3 goose-neck liquid seal
4 extended spindle valve
5 union for connecting sampling apparatus
6 heat break in outer wall of vessel

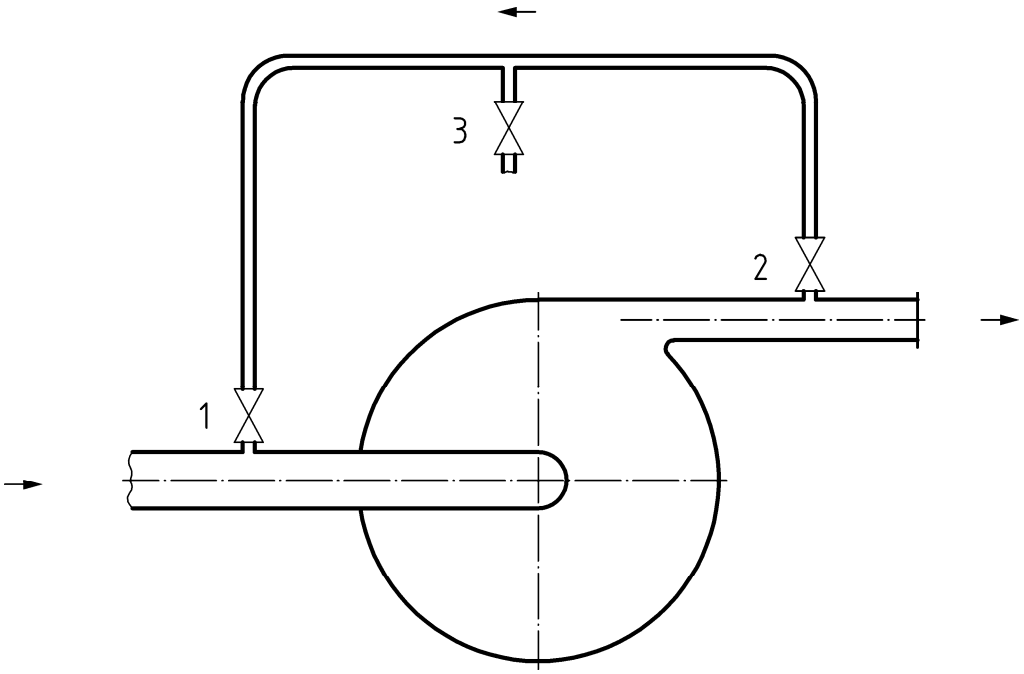
Figure D.6 — Typical sampling point installed in storage tank



Key

1 end of pipe bevelled at 45° to face the flow of liquid
2 flow
3 signal from flow meter
4 compressed air
5 integrator
6 impulse unit
7 compressed air
8 parallel-plug valve (shown horizontal for clarification)
9 sampling flask

Figure D.7 — Pipeline probes



Key

1 valve into bypass sampling pipe
2 valve out of bypass sampling line
3 sample taking valve

Figure D.8 — Typical bypass sampling apparatus

Annex E
(normative)

Methods of mixing for fluid fertilizers

E.1 General

Recommendations for mixing methods and precautions are given in E.2 to E.5 and necessary precautions associated with mixing are given in E.6 to E.8.

The effectiveness of any mixing method should be determined by testing random spot samples. If the variation between samples is found to be satisfactory the method and time of mixing should be recorded for future reference.

NOTE Any method of mixing might generate static charges in a liquid (see E.8).

E.2 Small containers

E.2.1 Hand shaking

Containers up to 10 l capacity may be shaken by hand, the container being inverted several times during the process.

E.2.2 Rocking

Heavier containers up to 20 l capacity may be rocked on the edge of the container but some ullage is essential for effective mixing by this method.

E.3 Drums and casks (up to about 1,8 m deep)

E.3.1 Rocking in a see-saw fashion

This method is effective only if there is ullage in the drum or cask and the drum or cask is laid on a suitable support and rocked rapidly. A strong sack stuffed with a resilient substance such as cork makes a suitable support. A support with sharp, hard edges should be avoided.

E.3.2 Rolling to and fro

This method is effective only if there is ullage in the drum or cask and if there are 2 to 4 complete revolutions between each reversal of direction. At least 20 complete cycles are necessary.

E.3.3 Mechanically driven drum shaker or roller

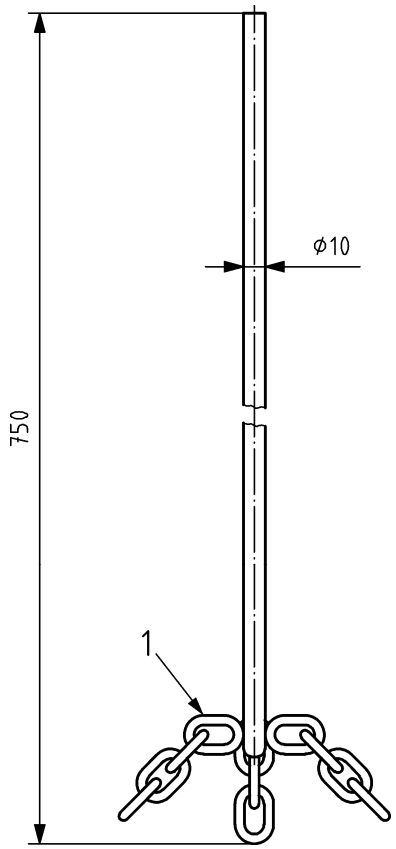
Mechanical action may be substituted for either of the actions described in E.3.1 and E.3.2 but the same limitations apply. If an electric motor is used a flexible drive from the motor is advantageous.

E.3.4 Mechanical mixing

Two types of mixer are shown in Figures E.1 and E.2 which are driven by hand drill, air or electric motor. If the latter is used a flexible drive is advantageous.

The dimensions of the fixed links (see Figure E.1) are chosen so that the mixer can be inserted into the container. When using these types of mixer care shall be taken to avoid damage to the inner surface of the container.

Dimensions in millimetres



Key

1 three links welded symmetrically around the rod

Figure E.1 — Typical mechanical mixer with fixed links

Dimensions in millimetres

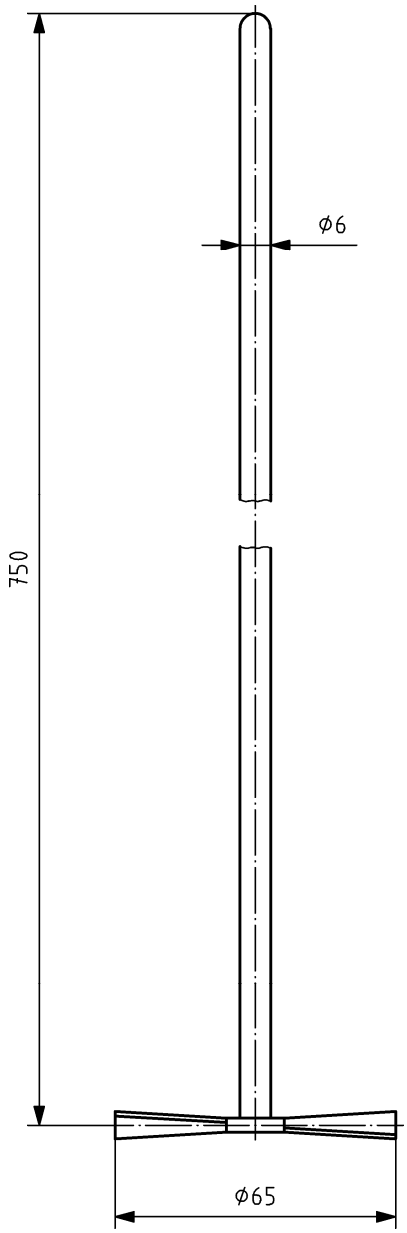


Figure E.2 — Typical mechanical mixer

E.3.5 Hand mixing

Two types (a and b) of hand mixer are shown in Figures E.3 and E.4. That shown in Figure E.4 is limited in use to open-topped containers. Both mixers are also suitable for small containers. They should be constructed such that no lodgement of material can occur between the blade or the disc and the shaft.

The hand mixer a) shown in Figure E.3 is suitable for mixing most liquids and should be used in such a way that the liquid is thrown from the bottom to the top of the container. It is particularly suitable for liquids containing firmly deposited solids, which can be dislodged using it and then dispersed by manual or mechanical mixer.

The hand mixer b) or plunger shown in Figure E.4 should be of sufficient area to produce adequate disturbance of the liquid and of sufficiently low mass to ensure that the operator is able to move it rapidly

through the liquid. It is suitable for multi-phase liquids and emulsions but not for those liquids containing solids capable of settling. The number of plunges (i. e. pushing the plunger quickly to the bottom of the container and then pulling it out of the liquid) required to mix the particular liquid should be determined.

Dimensions in millimetres

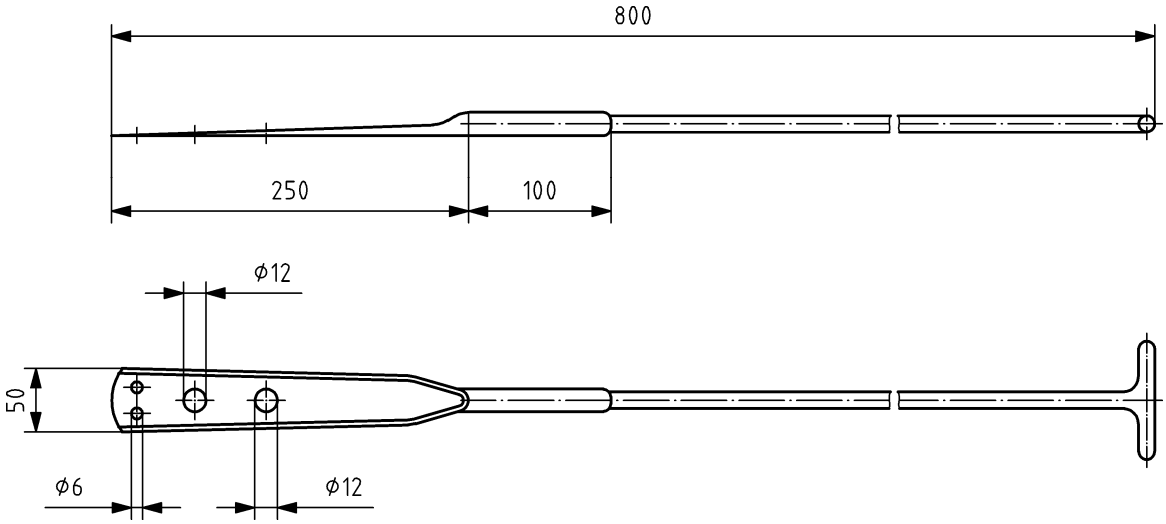


Figure E.3 — Typical hand mixer a)

Dimensions in millimetres

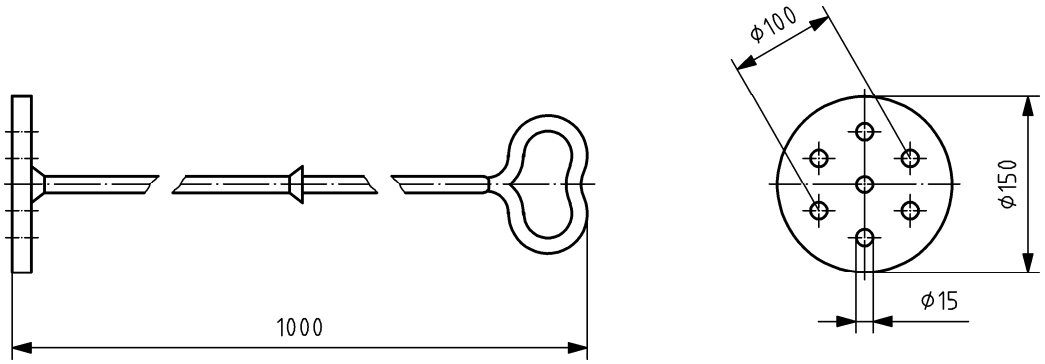


Figure E.4 — Typical hand mixer b)

E.3.6 Compressed gas

The gas used has to be inert and is generally air or nitrogen. It should only be used if it has been ascertained that loss of components, particularly volatile ones, is impossible, that the gas contains no deleterious impurities, e.g. entrained water or oil, and that the gas will not react with any of the components in the liquid.

E.4 Shallow tanks

Use the procedures described in E.3.4, E.3.5 and E.3.6.

E.5 Deep tanks

E.5.1 General

The apparatus for mixing the contents of tanks more than about 1,8 m deep usually forms part of the permanent equipment attached to the tank.

E.5.2 Pumped circulation

An effective and economical means of mixing the contents of large tanks is by means of pumped circulation through submerged jets.

E.5.3 Compressed gas

Follow the recommendations of E.3.6.

E.6 Precautions for sampling multi-phase fluids, including slurries

E.6.1 Materials in this category are fluids comprising separate liquid or solid phases, e.g. slurries, the whole of which is to be sampled. The separate phase might be suspended or might settle quickly. This category does not include fluids containing a solid contaminant, which are sampled according to the nature of the fluid.

E.6.2 The sampling methods described in 5.11 should be used but the following points should be noted:

a) the material should first be examined for the presence of a surface skin; if found, the thickness and nature of the skin should be noted and the skin should then be carefully removed;

b) a preliminary examination for the presence of adventitious matter on the bottom of a container might not be possible due to the presence of sediment;

c) due to the necessity of mixing before use these materials are usually supplied in containers not exceeding 20 l nominal capacity;

d) if the material is in bulk tanks these are normally fitted with built-in mechanical mixing devices otherwise the material may be rapidly mixed by other means, e.g. ball mills.

E.6.3 If sampling from a storage vessel that has been allowed to stand for any length of time it should be tested with the hand mixer (see E.3.5) or other suitable probe to determine the depth and hardness of any material deposited at the bottom. If found, this should be broken away with the hand mixer and dispersed by hand or mechanical mixing. Alternatively, for a smaller container the supernatant liquid may be decanted into a separate container and the settled solids broken up and stirred to a smooth paste. The decanted liquid should then be added back to the paste slowly whilst stirring. If the settled material is so hard that it cannot be re-dispersed, record the depth of settling and report the consignment as suspect.

E.6.4 Whilst taking a representative sample it is essential that the material be continuously agitated during the whole of the period during which the sample or samples are to be collected. Mechanical agitation should be used if the solid settles rapidly. If this is not possible the sample should be taken as rapidly as possible once mixing has ceased.

The sample collection has to be rapid, therefore sampling cans should not have narrow necks.

In order to prevent loss of solid matter it might be necessary to use a closable sampling tube rather than an open sampling tube. Care should be taken to ensure that a tight seal is obtained at the end of this operation.

E.7 Precautions for sampling fluids with significant vapour pressure

E.7.1 Introduction

For many gases that are stored or handled as liquids the composition of the gaseous phase in contact with the liquid differs from that of the liquid phase. Representative sampling of the liquid phase might, however, be difficult if it is not possible to mix the liquid and when only a single fixed sampling or discharge point is available.

NOTE Attention is drawn to ISO 7103 [7].

E.7.2 General precautions

Filters should not be used as impurities might be present as suspensions. Any part of the sampling device that comes into contact with the liquid or gas shall be capable of operation without grease or other material which might contaminate the sample.

The sampling methods described in this annex inevitably involve considerable spillage of these liquids and their vapours. Areas where samples are being taken should be adequately ventilated, preferably in the open, and operators taking samples should be made aware of the hazards involved.

E.7.3 Gases liquefied by pressure at ambient temperatures

E.7.3.1 Small storage vessels

Small containers are shaken to mix the liquid then inverted and sampled using a sampling bomb. Alternatively, if the pressure is low, the liquid may be driven into a Dewar vessel or passed through a vaporizer and collected as a gas in a suitable container.

E.7.3.2 Large storage vessels

Large containers should be fitted with at least one sampling point consisting of a pipe which should extend into the container for a short distance with its inner end below the lowest expected liquid surface level. The outer end of the pipe should terminate in a valve and union and should be of sufficient length for the sampling apparatus to be attached to the valve union.

The size of sample taken will depend on the requirements. A sampling bomb of appropriate size should be chosen. Alternatively, if the pressure is low the liquid may be driven into a Dewar vessel or passed through a vaporizer and collected as a gas in a suitable container.

E.8 Precautions against static electricity

WARNING — A discharge of static electricity might also give an electric shock to personnel.

E.8.1 Generation of static electricity

Liquids can be considered as containing equal numbers of positive and negative electric charges distributed evenly throughout their mass. If these charges are separated and remain separated a static electricity hazard arises. Separation of these charges only occurs if work is done on the liquid, e.g. if it is pumped, stirred, or if it evaporates or condenses. Maintenance of this state of charge separation only occurs when either the liquid or its container is a relatively poor conductor of electricity. When the liquid is composed of two phases, particularly if one of these is a conductor, charge separation can occur within the liquid and might be maintained if the major phase is not a good conductor of electricity. Evolution of dissolved gases might also produce charge separation.

E.8.2 Discharge of static electricity

No substance is a perfect insulator and all static electricity charges will eventually decay or leak away harmlessly. However, if another body capable of sharing the charge, or alternatively an earthed conductor, approaches a charged liquid a rapid redistribution of the charge will occur and some of the energy of the charge might appear as a spark bridging the gap.

As sampling often involves the introduction of sampling apparatus into the liquid it is clear that the act of sampling might cause sparking and/or an electric shock to the operator.

If charge separation occurs, the liquid and the equipment containing it frequently become oppositely charged. Such equipment is normally made from a conducting material, e.g. steel. It should be routine practice to ensure that all equipment is electrically bonded and well earthed. This prevents sparks arising outside the system and makes it safe to approach or touch the equipment. The charge persists in the liquid until it leaks away, usually by conduction through the liquid, to the container.

Earthing the container does not accelerate this process. If a sampling point is near an isolating joint on a cathodically protected pipeline care should be taken to avoid short-circuiting the isolating joint.

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