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Water quality — Sampling —

Part 13:
Guidance on sampling of sludges

Qualité de l'eau — Échantillonnage —

Partie 13: Lignes directrices pour l'échantillonnage de boues

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 5667-13 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 6, *Sampling (general methods)*.

This second edition cancels and replaces the first edition (ISO 5667-13:1997), which has been technically revised.

ISO 5667 consists of the following parts, under the general title *Water quality — Sampling*:

— *Part 1: Guidance on the design of sampling programmes and sampling techniques*

— *Part 3: Preservation and handling of water samples*

— *Part 4: Guidance on sampling from lakes, natural and man-made*

— *Part 5: Guidance on sampling of drinking water from treatment works and piped distribution systems*

— *Part 6: Guidance on sampling of rivers and streams*

— *Part 7: Guidance on sampling of water and steam in boiler plants*

— *Part 8: Guidance on the sampling of wet deposition*

— *Part 9: Guidance on sampling from marine waters*

— *Part 10: Guidance on sampling of waste waters*

— *Part 11: Guidance of sampling of groundwaters*

— *Part 12: Guidance on sampling of bottom sediments*

— *Part 13: Guidance on sampling of sludges*

— *Part 14: Guidance on quality assurance of environmental water sampling and handling*

— *Part 15: Guidance on the preservation and handling of sludge and sediment samples*

— *Part 16: Guidance on biotesting of samples*

— *Part 17: Guidance on sampling of bulk suspended solids*

— *Part 19: Guidance on sampling of marine sediments*

— *Part 20: Guidance on the use of sampling data for decision making — Compliance with thresholds and classification systems*

— *Part 21: Guidance on sampling of drinking water distributed by tankers or means other than distribution pipes*

— *Part 22: Guidance on the design and installation of groundwater monitoring points*

— *Part 23: Guidance on passive sampling*

Introduction

This part of ISO 5667 should be read in conjunction with ISO 5667-1 and ISO 5667-15. The general terminology used is in accordance with the various parts of ISO 6107.

Sampling and the determination of the physical and chemical properties of sludges and related solids are normally carried out for a specific purpose. The sampling methods given are suitable for general use but do not exclude modification in the light of any special factor known to the analyst receiving the samples or any operational reason dictating the need for sampling. Personnel taking samples should be fully aware of safety requirements before sampling occurs.

The importance of using a valid sampling technique cannot be overemphasized if the subsequent analysis is to be worthwhile. It is important that the personnel taking and analysing the sample be fully aware of its nature and the purpose for which the analysis is required before embarking on any work programme. Full cooperation with the laboratory analysing the samples ensures that the most effective application of the sampling occasion can be made. For example, the use of method-specific sample preservation techniques assists in the accurate determination of results.

This part of ISO 5667 is applicable to sampling motivated by different objectives, some of which are to:

a) provide data for the operation of activated sludge plants;

b) provide data for the operation of sludge treatment facilities;

c) determine the concentration of pollutants in wastewater sludges for disposal to landfill;

d) test whether prescribed substance limits are contravened when sludge is used in agriculture;

e) provide information on process control in potable and wastewater treatment, including:

    1) addition or withdrawal of solids,

    2) addition or withdrawal of liquid;

f) provide information for legally enforceable aspects of the disposal of sewage and waterworks sludges;

g) facilitate special investigations into the performance of new equipment and processes;

h) optimize costs, e.g. for the transport of sludges for treatment or disposal.

When designing a sludge sampling programme, it is essential that the objectives of the study be kept in mind, so that the information gained corresponds to that required. In addition, the data should not be distorted by the use of inappropriate techniques, e.g. inadequate sample storage temperatures or the sampling of unrepresentative parts of a sludge-treatment plant.

Water quality — Sampling —

Part 13:
Guidance on sampling of sludges

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This part of ISO 5667 gives guidance on the sampling of sludges from wastewater treatment works, water treatment works and industrial processes. It is applicable to all types of sludge arising from these works and also to sludges of similar characteristics, e.g. septic tank sludges. Guidance is also given on the design of sampling programmes and techniques for the collection of samples.

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.

ISO 5667-1, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes and sampling techniques*

ISO 5667-10:1992, *Water quality — Sampling — Part 10: Guidance on sampling of waste waters*

ISO 5667-12, *Water quality — Sampling — Part 12: Guidance on sampling of bottom sediments*

ISO 5667-14, *Water quality — Sampling — Part 14: Guidance on quality assurance of environmental water sampling and handling*

ISO 5667-15:2009, *Water quality — Sampling — Part 15: Guidance on the preservation and handling of sludge and sediment samples*

ISO 6107 (all parts), *Water quality — Vocabulary*

ISO/TR 8363, *Measurement of liquid flow in open channels — General guidelines for selection of method*

ISO 18283, *Hard coal and coke — Manual sampling*

CEN/TR 13097, *Characterization of sludges — Good practice for sludge utilisation in agriculture*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 6107 and the following apply.

3.1
batch
unit of production produced in a single plant using uniform production parameters — or a number of such units, when stored together — and that can be identified for the purposes of recall and re-treatment or disposal should tests show that to be necessary

3.2
composite sample
two or more samples or sub-samples, mixed together in appropriate known proportions (either discretely or continuously), from which the average value of a desired characteristic can be obtained

NOTE 1 The proportions are usually based on time or flow measurements.

NOTE 2 Adapted from ISO 6107-2:2006, 29.

3.3
critical control point
point, step or procedure at which control can be applied and is essential to prevent or eliminate a hazard or reduce it to an acceptable level

3.4
draw-off head
height of sludge above the extraction point providing hydraulic pressure available for withdrawal of sludge when removal is dependent upon gravity flow

3.5
flow-related sampling
samples taken at varying time intervals governed by material flow

NOTE "Flow-related sampling" usually applies to liquid sludges; for further guidance, see ISO 5667-10.

3.6
grab sample
discrete sample taken randomly (with regard to time and/or location) from a body of sludge

NOTE Adapted from ISO 6107-2:2006, 128.

3.7
heap
pile of dewatered sludge of approximately equal dimensions

3.8
liquid sludge
sludge flowing under the effect of gravity or pressure below a certain threshold

[CEN/TR 15463:2007[7]]

3.9
long pile
pile of dewatered sludge with length greater than width

3.10
open channel
pipe or conduit where the liquid surface is at atmospheric pressure

3.11
proportional sampling
technique for obtaining a sample from flowing sludge in which the frequency of collection (in the case of discrete sampling), or the sample flow rate (in the case of continuous sampling), is directly proportional to the flow rate of the sampled sludge

[ISO 6107-2:2006, 91]

3.12
quality control point
point, step or procedure at which control can be applied and is important or even critical for acceptable quality, but not necessarily for safety

3.13
sampling performance
precision of sampling assessed by quality control methods, e.g. repeated sampling, field blanks, field controls, intersampler comparisons, and sampling at reference stations

3.14
sludge
mixture of water and solids separated from various types of water as a result of natural or artificial processes

NOTE Adapted from ISO 6107-1:2004, 67.

3.15
sludge cake
sludge generated from dewatering devices

EXAMPLE Filter press, centrifuge.

[EN 1085:2007[5], 9490]

3.16
static belt
stationary conveyor where material is conveyed on a belt

3.17
stockpile
storage of treated sludge until it is utilized or disposed of

4 Developing a sampling plan

4.1 Sampling objectives

Definition of the objectives of the sampling programme is an essential step towards defining the type and quality of information that is to be obtained through sampling.

The type of sampling that is undertaken depends upon whether the objective of the sampling programme is monitoring for process control or for effluent quality. Typically, a sampling programme targets the critical control points and quality control points in conjunction with in-line process instrumentation. Consult CEN/TR 13097 for details of hazard analysis critical control point, an approach to identifying critical control points and quality control points.

A sampling programme might include:

— influent monitoring;

— in-process monitoring;

— effluent monitoring;

— equipment inspection and testing.

4.2 Variability considerations

Variability, in time and space, is probably the most significant aspect to be considered in the design of sampling plans. Variability determines the number of sites, number of replicates, and the frequency of sample collection. High variability of environment or industrial discharge combined with poor sampling design or too few samples can result in data that are too variable to reveal an impact, disturbance or trend. Local heterogeneity, sampling variance and analytical variance can be estimated and vetted against data quality requirements (i.e. by the method of Reference [8]).

Examples of variation in wastewater due to process variability include:

- daily and weekly variation: particular processes, e.g. scheduled cleaning, can always occur on the same day of the week, leading to a consistent pattern of variation in the quality of the discharge;
- seasonal variation: in communities with large seasonal load changes, e.g. a holiday resort or where there is a food processing operation (fish, fruit, or vegetables), the characteristics of the sewage sludge can vary over the course of a year;
- event variation: the influent (and effluent) from sewage treatment plants varies after a rainfall event due to the infiltration and inflow into the sewage system diluting the concentration, but increasing the volume of wastewater.

How process variability considerations are taken into account in the design of a monitoring plan depends upon the objective of monitoring, e.g. to determine the maximum concentrations of a pollutant, the variability of discharge or the average concentration.

5 Sampling equipment and containers

5.1 General

The sampling of sludge from fixed points can require the installation of permanent equipment, even if this is only an additional pipe and valve to the processing plant. It is important to verify that any such equipment is regularly cleaned and that it is free from corrosion. In addition, it is necessary to assess the potential for interference on any test results that the equipment can have. In general, the laboratory performing the sludge examination should be consulted before installation of any fixed-point equipment or at the implementation of a new sampling scheme.

5.2 Sampling equipment

In general, sludge sampling equipment is most practical if it is as simple in design and construction as possible. The physical properties of sludges depend on their type and solids content. Guidance on selection of sampling equipment for different situations is given in Annex A. Some specific examples of equipment for liquid sludges under particular circumstances are given in Annexes B and C. Sampling equipment should be robust and free of any contaminating influence; equipment should be kept clean and corrosion free.

Composite samples of liquids taken as time or flow proportional samples are often collected by an automatic sampling unit programmed to collect individual samples of liquid at selected intervals. Generally, the sampling unit automatically purges the sample connection and tubing before collecting a sample.

5.3 Containers and sample preservation

Sample containers should be chosen with care. Obtain specific guidance on containers and sample preservation from ISO 5667-15 and in all cases consult the analyst.

Samples for total moisture determination should be collected and stored in containers that are both leak-tight, to prevent leakage or ingress, and airtight, to reduce moisture loss by evaporation. The sample containers should be shielded from any direct source of heat, including the sun, at all times and returned to the laboratory for refrigerated storage and/or rapid analysis to alleviate the risk of gas build-up in the containers.

Except for samples to be analysed for trace organic materials, double polyethylene bags can be used for sludge cake samples. Polyethylene, polypropylene, polycarbonate, and glass containers are satisfactory from the point of view of chemical stability when sludge sampling. However, caution should be exercised since containers can become pressurized due to gas production in wastewater sludges and explosive situations can occur. Care should be taken, particularly when glass containers are used, to prevent build-up of gas pressure and to minimize the dispersion of fragments if an explosion occurs. Further guidance on overcoming the problem is given in Clause 8. Some manufacturers offer self-regulating pressure equalization closures for glass containers. For additional safety guidance, consult ISO 5667-15.

Glass containers should be used when organic constituents, e.g. pesticides, are to be determined, whereas polyethylene containers are preferable for sampling parameters of general interest, e.g. pH and dry matter. It is possible that polyethylene containers are not suitable for collecting samples to be analysed for some trace metals, e.g. mercury. These containers should only be used if preliminary tests indicate acceptable levels of interference.

The introduction of aged material from the dead space in sample lines can also contribute to contamination of samples due to corrosion (see 6.3.4) and can prove to be a serious potential source of error if not eliminated.

Sample containers should be made of a material appropriate to the preservation of the natural properties of both the sample and the expected range of contaminants. For guidance on the type of sample container to be used and recommendations for the preparation of containers, consult ISO 5667-15.

6 Sampling procedure

6.1 Sampling regime

6.1.1 General

The most appropriate way of sampling in any situation depends on several factors:

a) access to the sampling point by personnel;

b) the practicality of installing and maintaining automatic equipment, if appropriate;

c) the practicalities of interrupting safely a stream of moving liquid sludge or cake when manually sampling;

d) the nature of the chamber or tank design with respect to stratification of liquid sludges.

For a fixed plant, site safety, practicality of sample collection and representativeness of collected samples should be taken into account when selecting sampling site locations.

Where sludge is passing in an accessible stream, either continuous or intermittent sampling should be considered. The greater the number of samples taken, the greater the degree of confidence in the representativeness of the sludge sample. Consult ISO 5667-1 and ISO 5667-14 for further information. There can be a requirement to consider the representative nature of solid sludges. Consult ISO 18283 for guidance on the statistical assessment of bulk loads of solid materials. When a non-representative sample has been collected, the analytical data need to be interpreted with caution.

Nevertheless, it is often desirable to take daily or shift samples for control purposes, since definitions of batches and periods vary from plant to plant. Continuous sampling is more likely to be practicable where a fixed conveyor discharge can be sampled automatically. Intermittent sampling is more suited to manual sampling from a wagon or tanker.

6.1.2 Sample type

The basic types of sample which can be required are:

a) a composite sample which can be generated from either continuous or grab samples from stockpiles, sampling of liquid or cake sludges;

b) a grab sample, which is taken at random from a liquid or conveyor flow of cake or from a single sample point in a stockpile. A programmed series of grab samples analysed individually, which can be liquid or cake samples, is a refinement of this technique.

6.1.3 Time-basis sampling

Time-basis samples can be a programmed series of grab samples that are to be analysed individually or combined into a composite sample.

Calculate the maximum sampling interval, t, in minutes, between taking samples when using time-based sampling, using Equation (1):

$$t = \frac{60 \times m}{qn} \quad (1)$$

where

60 is the number of minutes per hour;

m is the mass, in tonnes, of the batch;

q is the maximum mass flow rate, in tonnes per hour;

n is the number of samples.

6.1.4 Composite sampling

6.1.4.1 General

A composite sample is made from a number of discrete samples that have been collected from a body of material and combined into a single sample. This single, composite, sample is representative of the average conditions in that sampled body of material.

The composite sample should be homogenized before analysis and can be reduced to provide multiple sub-samples (see 6.4).

6.1.4.2 Number of sub-samples

To calculate the minimum sample number for taking composite samples, Equation (2) should be used:

$$n = \left(\frac{1{,}96 \times s}{E} \right)^2 \quad (2)$$

where

1,96 is the z-value (number of standard deviations from the mean) for 95 % confidence;

s is the standard deviation estimated from test sampling;

E is the maximum permitted error, expressed in the same units as s;

n is the number of samples.

The standard deviation can be estimated by repeated collection and analysis of a large number ($\geqslant$30) of grab samples, e.g. at plant set-up, and checked at intervals.

See Annex D for an example calculation.

6.1.4.3 Continuous sampling

In continuous sampling at regular intervals, the samples are taken uniformly throughout the whole supply of sludge, but are then grouped together into composite samples.

6.1.4.4 Intermittent or consignment sampling

For this type of sampling, the sludge is regarded as a series of batches and only a proportion is selected for sampling. The selected batches are spread uniformly throughout the whole supply of sludge, and the samples are taken uniformly from each batch selected for sampling. For example, one might sample by randomly picking tankers irrespective of the source of the sludge or the mass transported.

With this type of sampling scheme, it is necessary to allow for the fact that the time-interval average is influenced by the variation between batches, which cannot be predicted. More samples are required over the time interval to achieve a given confidence than if continuous sampling had been carried out, since the error of sampling a batch is now only a portion of the total error.

6.1.4.5 Flow-related sampling

This is accomplished by extracting a mass of sludge proportional to the flow rate at the sampling point at the end of each time interval. This can either be added to a composite sample or to a partial composite sample. This method is applicable when sampling primary sludge at the time of draw-off, i.e. as the draw-off head falls, the discharge rate drops and the flow proportionality changes. If there is a requirement for mass transfer information, it is prudent to measure the associated flow rate or batch size of the sludge. For example, daily metals-loading information can be required for sludge pumped to agricultural land. For further guidance, consult ISO/TR 8363.

6.2 Replicate sampling

In a situation where automatic sampling is to be installed, e.g. on a conveyor belt, it is preferable to establish that the point at which the samples are being taken is representative of the output from that particular part of the plant. Under these circumstances, replicate sampling should be used to assess the variability of the output stream at the proposed sampling point. This technique can be applied to liquid as well as cake sludges.

For example, when replicate sampling is in progress, two samples should be taken by placing samples alternately in two containers labelled A and B. After a number of samples have been collected in replicate, the results should be examined and the number of samples or the number of batches sampled should be changed in accordance with the guidance given in ISO 5667-1 and ISO 5667-14. After carrying out this test, it might be found that fewer samples can be taken in the future than were at first estimated to achieve the required confidence defined by the need for sampling. Consult ISO 18283 for details of the calculation of the number of samples, if the material can be likened to a mineral.

If occasional confirmation of the sampling performance is required, replicate sampling is ideal. This should be achieved by taking a run of 10 paired replicate samples (i.e. 20 samples) after every 40 ordinary samples. It is not possible to assess whether there has been a change in sampling behaviour until two sets of 10 paired replicate results have been obtained and compared. If, at any time, there is reason to believe that sampling conditions have changed, a further set of 10 paired replicate samples should be collected and statistically tested before any decision to alter the regime is made.

It is important to ensure that samples taken for confirmation of the sampling performance are not taken with more than ordinary care. One way of ensuring this is always to sample in replicate pairs, but to amalgamate the two sub-samples together and prepare the combined sample when replicate results are not required.

6.3 Methodology

6.3.1 General

There is no definitive guidance that can be given on the need to sample sludges as cakes or liquids. For example, it can be necessary to sample sludge in both forms on any particular plant in order that the process can be optimized and the quality of the final output monitored for disposal purposes.

6.3.2 Sample size

6.3.2.1 Liquid sludges

It should be noted that thin liquid sludges (of low solids content) require the preparation of relatively large volumes of the sampled material to provide sufficient dry matter to allow for a truly representative analysis of constituents, e.g. metals. The analyst should always be consulted as to the quantities of sludge required, and the sample reduced accordingly in the field before returning to the laboratory. Large volumes of sample accrued by the combination of representative samples need to be homogenized before sub-sampling. The mixing process should preferably be tested to ensure efficacy of mixing. The homogenization can be achieved in a container such as a plastic dustbin using a suitable paddle to prevent settlement.

6.3.2.2 Sludge cake

To obtain a representative sample of sludge cake, the mass accumulated is always too large for laboratory manipulation at the bench. Sample size reduction is, therefore, best carried out in the field in accordance with the procedures specified in 6.4.

NOTE Little guidance can be given as to the size of samples. This is because this criterion is dependent on the variability of the sampled material and the type of analysis to be carried out.

6.3.3 Sampling from tanks and road tankers

The performance of tanks used for sedimentation or consolidation of wastewater or sewage sludges, digesters and other vessels, cannot always be gauged from samples taken from the inlet and outlet pipelines. The segregation of solids likely to occur can be detected by sampling different sections and depths of a tank. Access to different strata is often provided by a design feature, e.g. stepped draw-off pipework. Examples of equipment that could be used when this is not the case are given in Annex A.

Usually a composite sample of the sludge is required. The sludge in the tank should, where possible, have been thoroughly mixed before sampling. Alternatively, grab samples can be taken either using a long-handled ladle to sample the discharge or by diverting the flow at random intervals into a separate container to allow separate mixing and subsequent sampling. This technique assists in removing some of the problems of stratification that can occur when some sludges are left standing in tanks or tankers (e.g. with easily settleable sludges).

6.3.4 Sampling from pipes

See 6.1.4.5 for guidance on flow-related sampling. If pumping is taking place, correct sampling can be achieved with samples being taken at appropriate intervals at the pump outlet or a similar convenient place. However, factors such as the nature of the sludge, the flow rate, the diameter of the pipes and the roughness of the pipe can produce turbulent flow that results in mixing of the sludge. Minimizing the influence of this potential problem can be achieved by allowing the flow to equilibrate before collecting a portion from which to take a sub-sample after mixing. Any side arms or valves utilized in the sampling arrangement should be flushed with at least three times the standing volume to ensure that any stagnant material is removed from the pipework. When taking the sample in this manner, visual checks should be made to ensure that the flow rate and consistency remain constant. Blockages in pipes due to fibrous materials can effectively filter the sludge, thereby giving spurious results. This can go undetected at the time of sampling, and requires repetition of the exercise to assess the reliability of results.

A special case is the sampling of conditioned sludge from a high-pressure line prior to plate filter pressing. In this case, if sludge were to be sampled in a conventional manner allowing rapid decompression, its filtration properties would probably deteriorate markedly due to shear in the sampling valve. To sample a conditioned sludge with the minimum of shear, the simple apparatus shown in Annex B can be used. This type of sampling is usually required if testing for specific resistance to filtration has been requested to assess the potential efficacy of chemical dosing on press performance.

6.3.5 Sampling from open channels

A weighted bucket or a pump should be used, depending on the solids content of the sludge. When using a pump to sample from an open channel, a solids content of up to 5 % mass fraction can be sampled, provided the velocity in the suction pipe is sufficient to keep all the particulates in suspension. This velocity has to be established on a site-specific basis using a transparent section of the pump uptake tube to visually assess the performance of the suction. Samples should be taken across the width and depth of the channel to ensure that a representative composite sample is obtained after mixing individual samples. It should be borne in mind that the physical characteristics of the sludge can change on passing through a pump, due to shear of the particulate matter. The practice of sampling from open channels is probably only likely to occur when dealing with wastewater activated-sludge plants, and therefore a weighted bucket is often more appropriate.

6.3.6 Sampling of sludge cake from heaps and stockpiles

In general, sampling of sludge cake from heaps and stockpiles is prohibited by safety requirements, and is not technically required. However, if it is necessary to sample from heaps and stockpiles, the following guidance applies.

When sampling heaps of air-dried sludge lifted from drying beds or stockpiles of sludge cake, it is important to obtain portions of sludge from throughout the mass and not just from the surface layer. The sludge taken off drying beds should be free of the bed media, since inclusion of grit or sand distorts measurements of dry matter content. The inclusion of any grit or sand is only applicable if it is representative of the whole mass of sludge being processed. A mechanical excavator can be the most practical tool, but particular care is necessary to ensure representative sampling.

If, after assessment of safety requirements, no concerns are apparent and equipment is available, core sampling can be considered as a means of obtaining samples. Samples should be taken throughout the depth of the heap or stockpile. A composite sample can then be prepared from a statistically significant number, n_{sp}, of such cores. Statistically, n_{sp} is given by the equation:

$$n_{\mathrm{sp}} = \frac{\sqrt{V}}{2}$$

where V is the nominal volume, in cubic metres, of the stockpile.

The value of n_{sp}, rounded to the nearest whole number, should lie between 4 and 30. Consult ISO 5667-12 for further guidance on core sampling.

Major variations in data throughout stockpiles can be found, particularly old ones, in which the top layers desiccate to form crusts which allow anaerobic activity to increase below and aerobic activity to proliferate in the upper near-surface layers. The migration of nutrient species due to leaching in these situations can also cause difficulty when attempting to take representative samples or use analytical results. The surface layers can therefore give rise to misinterpretation when coring to the centre or full depth of the heap, due to surface area-to-volume ratio inconsistencies dependent on the stockpile shape. In certain circumstances, accessing the cross-sections of a heap with a mechanical excavator should be considered if it can be safely undertaken to enable representative sampling.

6.3.7 Sampling from wagons

The only method considered satisfactory for sampling from wagons is to take samples in such a way that they are representative of all parts of the sludge in the wagon. Normally, most of the sludge in a wagon is inaccessible and methods usually involve sampling after unloading. The method to be adopted depends upon the method of handling wagon-borne sludge and upon the types of wagons concerned. Sampling from wagons should not be routine practice, but where circumstances dictate (e.g. on delivery to a landfill) the following guidance applies.

It is evident that samples of sludge taken from the tops of wagons cannot be representative of its moisture content if the sludge has been exposed for some time to rain or snow, or to the drying effects of exposure to the open air during transport. Consequently, sampling from the tops of wagons for moisture or ash content is not satisfactory. In addition, appropriate safety precautions for regular access make this practice an unlikely routine measure.

If sampling is for the purpose of determining the dry solid or ash content, then a homogenized intermittent sample should be taken at the point of discharge from the wagon after unloading, if it is judged safe to do so, i.e. not at the tipping face of a working landfill.

6.3.8 Sampling from conveyor belts

6.3.8.1 General

Fragments of pressed or otherwise consolidated sludge tend to segregate by size and density when agitated and fines are likely to drop to the bottom. In order to obtain a representative sample of material on a conveyor belt, a complete cross-sectional portion should be removed, including fines. If the solid material is of approximately uniform size, lumps can be removed, with caution, from a moving belt at random.

6.3.8.2 Sampling from a static belt

The samples taken from a stationary conveyor under these conditions should originate from the full width and depth of the stream. A complete section should then be taken from a sufficient length to give the required mass. It is convenient to define a suitable position for regular sampling by marking the framework adjacent to the belt.

6.3.8.3 Sampling from a falling stream

This sampling is best done by a device which temporarily collects the whole flow at a transfer station or conveyor discharge point, e.g. diversion of the flow to a sampling bin or barrow.

Where it is not possible to stop the conveyor, the sludge should be sampled while the conveyor is in motion. When practicable, the sample can be taken as the sludge passes from the loading booms into wagons or hoppers. When this is not possible, the sludge can be sampled as it passes from one conveyor to another, if there is sufficient fall for the insertion of a sampling scoop. A suitable point is often found where the sludge falls from a belt on to the loading boom or ramp and a platform can be erected to make access for manual sampling easy and safe (see Clause 8).

Such techniques are useful for taking representative samples when sludge cake is being loaded on to wagons. If it is not possible to use such a collection technique at a transfer station or discharge point, an alternative procedure is to stop the conveyor periodically and treat the material on it as a long pile (see 6.3.9).

6.3.8.4 Manual sampling from a moving belt

A scoop or shovel should be used for sampling from a moving belt. It is essential that the stream be sampled in such a way that no bias is introduced. The scoop can be inserted, for alternate samples, from the left, and from the right, and passed entirely through the stream to ensure that the sludge cake from the full width is included in the sample. If the size of the stream is too great to be sampled as a whole, successive samples can be taken from adjacent parts of the stream.

Whether the belt is stopped or not, the sample loading should be controlled as far as possible so that the samples are not of excessive mass (see Clause 8).

6.3.8.5 Mechanical sampling from a moving belt

Machinery has been developed for the sampling of minerals from conveyor belts and falling streams which can be converted for use with sludge cake. However, the adaptation of such equipment is not common. If a particular situation arises where the use of such equipment appears practical, a statistical analysis of the performance should be carried out before implementation on a full-time basis is contemplated.

6.3.9 Sampling from long piles

With this technique, the conveyor flow is deflected into an area which has been marked out, or specifically designed as a long bin with removable dividers. If convenient, the dividers can be put in place prior to the next stage. The material should be poured into the pile area or bin in such a way that it is distributed uniformly.

If preplacement is not an option, pairs of braced dividers should be inserted into the pile at fixed intervals along its length. The bracing is necessary to prevent the dividers being forced together during the removal of the sample. These dividers should make good contact with the base. The sample comprises all the material between the pairs of dividers, including the fines at the bottom.

6.3.10 Sampling after dewatering by centrifugation

Installations that incinerate sludge locally use this type of technology. Generally the sampling point is put after the high-pressure pump (similar to a concrete pump) used to convey the paste-like sludge from the centrifugation device to the oven.

NOTE It is possible that the internal pipework needs more frequent inspection than when used for concrete.

6.4 Sample homogenization and sub-sampling for sludge cakes (quartering)

6.4.1 General

It is usually necessary to reduce the mass of any bulk solid sample. This results in a laboratory sample which in turn is reduced to obtain a test portion of appropriate mass. The sample reduction therefore needs to be carried out in such a way as to obtain at each stage a representative part of the sample.

The sample should be thoroughly mixed by heaping it on to a clean, flat and hard surface to form a cone. This is then turned over, e.g. with a shovel, to form a new cone, the operation being carried out three times. Each conical heap should be formed by depositing each shovelful of material on the apex of the cone, so that the portions which slide down the sides are distributed as evenly as possible, and the centre of the cone is not displaced.

The heap should then be divided into quarters, which should be uniform in thickness and diameter, giving due regard to the irregular shape. Diametrically opposed quarters are retained and should be recombined. The process is repeated until the final two quarters produce the required sample mass.

Sludges that have a gelatinous appearance, and behave more like a jelly than a mineral solid-like gravel, are unlikely to be suitably homogenized by this technique. Mixing such as that employed for the hand or mechanical preparation of cement mortar can be more appropriate. Division into sub-samples can still be achieved by the combination of diametrically opposed quarters.

6.4.2 Sample reduction to provide multiple sub-samples

When two or more laboratory samples are required from a bulk sample, the sample mass should be reduced by quartering. All of the excess bulk sample rejected should be recombined at the individual division stages, mixed thoroughly and reduced again to provide a second laboratory sample. This should be repeated as necessary to provide the required number of laboratory samples.

In the laboratory, when handling dry material, quartering is often performed on a plastic sheet which can then be used for mixing the material and forming a new mound ready for the next mass reduction. The mixing is performed by the repeated turning in of opposite quarters, by lifting the edges of the sheet and folding it into the middle or by the use of plastics implements.

This procedure should also be adopted if division into laboratory duplicates is required. The procedure thus ensures the maximum homogeneity of the two duplicates from the same bulk sample, e.g. after homogenization of a sub-sample prepared for the analysis of metals.

Alternatively, the sample can be poured into a conical mound which has been divided into four using flat dividers larger than the pile. Diametrically opposite quadrants should be removed (including fines) and combined. This process is repeated until the sample is sufficiently reduced in volume to provide the analyst with an appropriate sample size. When samples have been dried and homogenized, devices such as riffle boxes (see Figure 1) can be used for sub-division if sufficient material exists. Where riffle boxes are used, the material should be distributed evenly across the width to ensure the sample is divided representatively. If wet samples are treated in this manner, they do not divide properly and can cause clogging.

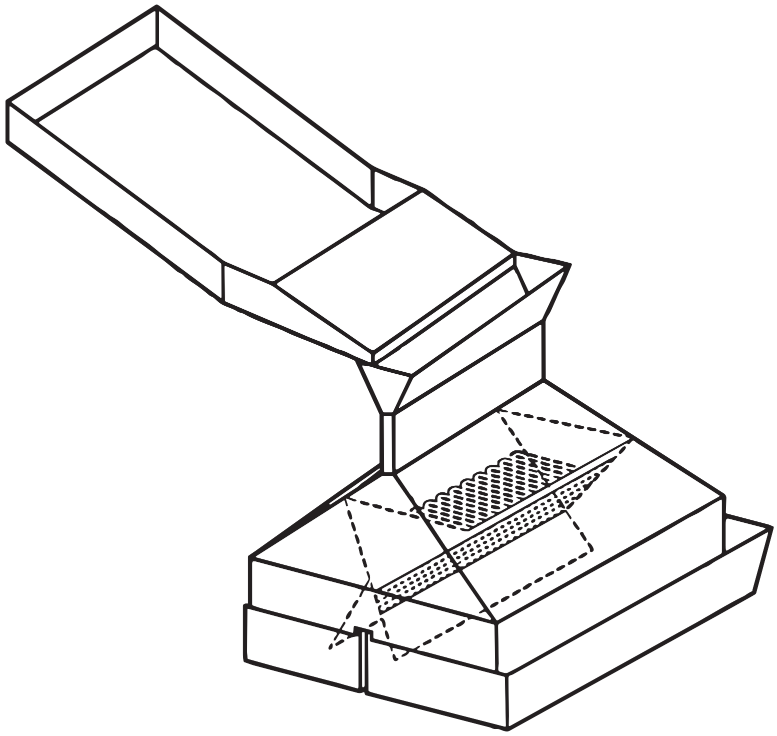


Figure 1 — Typical riffle box used for subdivision of samples in the laboratory

7 Sample storage

7.1 General

The methods of sampling can be time dependent in terms of the analytical technique to be used (e.g. pH change over time). In addition, if there is an immediate operational requirement for information, a loss of statistical confidence can be acceptable. Judgements have to be made on a situation-specific basis. For example, when temperature is the parameter to be monitored, it is possible that sample homogeneity is not regarded as critical. For further guidance, see ISO 5667-15.

For some types of liquid sludge, particularly raw sewage sludge, gross atypical solids, e.g. rags, can be removed by passing the sample through a stainless steel or plastics screen of nominal size of openings not less than 5 mm.

NOTE Stainless steel contains chromium and nickel. Neither are expected to be a significant problem in terms of release to the sample, but awareness of the presence of these metals is prudent when extremes of pH are encountered. With plastics screens, the plasticizer used in manufacture can interfere with biocide analysis.

Atypical solids can be needed for further examination and should be retained. Some samples can change significantly because of biological activity. It is therefore important that such samples be analysed as soon as possible after collection.

Some form of heat treatment, such as autoclaving, can be appropriate prior to storage, e.g. if volatile components are of no interest and the sample is being retained for composite metals determination. Specific guidance should always be sought from the receiving analyst.

7.2 Storage

Samples should be stored at the laboratory until all data are obtained and checked, in case spurious results require investigation. The storage duration and conditions of sludge samples within the laboratory are specific to the analyte(s) to be analysed.

Samples should be stored no longer than the maximum storage duration times. Samples should be stored in containers appropriate to the analyte(s) to be analysed, with preservatives added if necessary. For further guidance, consult ISO 5667-15:2009, Table 1.

Specific guidance should always be sought from the receiving analyst.

8 Safety

It can be necessary to take samples from a sewer system as well as a sewage treatment works or similar site, but in either case certain risks are likely to be present. When selecting sampling locations and when sampling potentially hazardous sludge, safety and health aspects should be observed. In general, the safety advice given in other parts of ISO 5667 is relevant on many occasions when sludge is sampled (e.g. refer to ISO 5667-10:1992, Clause 6).

However, these guidelines cannot be substituted for the provisions of local or national health and safety regulations, which should always be carefully studied and put into effect before sampling occurs.

9 Labelling and reporting

Sample containers should be clearly and unambiguously marked, so that subsequent analytical results can be properly interpreted. All details relevant to the sample should be recorded on a label attached to the sample container, in addition to the results of any on-site tests carried out by the sampler (e.g. pH). Alternatively, a codified system, e.g. using barcodes, should be used. When many sample containers are needed for a single occasion, the containers should be identified by code numbers and all relevant details entered on a sample record form. Labels or forms should always be completed at the time of sample collection.

The detailed form of sample report depends on the objectives of sampling. Details which should be considered for inclusion are:

a) the name of the plant;

b) the sampling site (this description should be complete enough to allow another person to find the exact location without further guidance);

c) the date and time of sample collection;

d) the name of the sample collector;

e) the weather conditions at the time of sampling;

f) the appearance of the sample;

g) information on any sample preservation technique used;

h) information on any specific sample storage requirements (e.g. whether refrigeration is necessary).

Annex A
(informative)

Support on the selection of equipment

Suggested applications for generic types of sampling equipment are listed in Table A.1.

Table A.1 — Suggested applications for generic types of sampling equipment

Sampling tool	Main application	Application[b]			Size of openings	Sample length or volume	Maximum sampling depth	Level of disturbance[b]	Fit for stratified sampling[b]	Can be sterilized	Fit for analysis[b,e]
		L[a]	SC/P[c]	P[d]	mm		m				
Dutch or Edelman Auger		–	++	–	40 to 200	10 cm to 15 cm	5 to 10	0	++	yes	tm +++ vol –
Bucket auger		–	++	–	40 to 200	10 cm to 15 cm	3	0	+	yes	tm +++ vol –
Gouge operated with body weight	Soft and cohesive materials	–	+++	–	10 to 60	30 cm to 100 cm	0,5 to 10	+	+++	yes	tm +++ vol –
Handhammer operated gouge	Cohesive materials	–	+++	–	25	30 cm to 100 cm	0,5 to 10	+	+++	yes	tm +++ vol –
Peat sampling device	Sediments/sludges	–	+++	–	25	50 cm	<10	++	+++	yes	tm +++ vol +
Flap gouge auger (small peat sampling device)	Powders/sediments/sludges/bio-reactors	–	+++	–	12	25 cm	<3	++	+++	yes	tm +++ vol –
Sampling device for volatiles (no headspace)	Materials to be analysed for volatiles	–	++	–	15 to 40	5 cm to 20 cm	<10	+++	+++	yes	tm +++ vol +++
Piston sampling devices	Liquid-saturated uncohesive materials	–	+	–	30 to 50	75 cm to 200 cm	<10	+++	+++	difficult	tm +++ vol +
Piston sampling devices with lockable cutting head	Liquid-saturated uncohesive harder materials	–	+	–	60 to 70	50 cm to 150 cm	<10	+++	+++	difficult	tm +++ vol +
Remote operated piston sampling devices	Liquid-saturated uncohesive materials at larger depths	–	0	–	50 to 70	500 cm	>5	+++	+++	no	tm +++ vol +
Spoon	Fine dry or moist bulk material	–	+	+[f]	30		0	0	–	yes	tm +++ vol –

Table A.1 (*continued*)

Sampling tool	Main application	Application[b]			Size of openings	Sample length or volume	Maximum sampling depth	Level of disturbance[b]	Fit for stratified sampling[b]	Can be sterilized	Fit for analysis[b,e]
		L[a]	SC/P[c]	P[d]	mm		m				
Spade (flat)	Dry or moist bulk material; soil	–	++	++	200	20 cm	<2	+	+	yes	tm +++ vol +
Spade with sides	Waste with debris	–	++	++	150 to 300	20 cm	<1	+	–	yes	tm +++ vol +
Scoop/trowel		–	+	+[f]		15 cm	<0,5	0	–	yes	tm ++ vol –
Weighted bottle/weighted cage with sample bottle	Liquids and free-flowing slurries	+++[g]	–	–		500 ml	15	–	–	yes	tm ++ vol +
Simple weighted can	Liquids	+++	–	–		500 ml	15	–	–	yes	tm ++ vol +
Valve sampling cylinder (sinker sampling device)	Liquids	+++	–	–				++	++	yes	tm ++ vol +
Bottom sampling device	Liquids	+++	–	–		500 ml	15	–	–	yes	tm ++ vol +
Column sampling device	Liquids	+++	–	–				+++	+++	yes	tm ++ vol +
Drum and tank sampling device	Liquids	+++	–	–			1	+++	+++	yes	tm ++ vol –
Tube sampling device	Liquids	+++	–	–	20 to 40		1,5	+++	+++	yes	tm +++ vol +++
Pond sampling device	Liquids	+	–	–		150 ml to 600 ml	2,5 to 4,5	–	–	yes	tm ++ vol –
Vacuum pumps	Liquids and free-flowing particulates	+++	–	+++				–	–	yes	tm ++ vol –
Peristaltic pump	Liquids, sludges and slurries in containers	+	–	–				–	–	yes	tm ++ vol –

Table A.1 (*continued*)

Sampling tool	Main application	Application[b]			Size of openings	Sample length or volume	Maximum sampling depth	Level of disturbance[b]	Fit for stratified sampling[b]	Can be sterilized	Fit for analysis[b,e]
		L[a]	SC/P[c]	P[d]	mm		m				
Valve	Liquids under pressure	+++	–	–				–	–	difficult	tm ++ vol –
Tap	Liquids; particulates free-flowing under gravity	+++	–	+++				–	–	difficult	tm ++ vol –
Thief (grain sampling device)	Particulates	–	–	+[h]	12 to 25	60 cm to 100 cm		–	–	yes	tm ++ vol –
Trier	Soft to firm solids	–	+	+[h]	12 to 25	60 cm to 100 cm		++	++	yes	tm ++ vol –
Split tube		–	+	++	35 to 125	30 cm to 90 cm	<1	+++	+++	yes	tm ++ vol +
Thin-walled (Shelby) tube sampling device		–	++	–	50 to 100	25 cm to 90 cm	<1	+++	+++	yes	tm ++ vol +
Pile sampling device		–	+	++	0 to 140	<140 cm		++	++	yes	tm ++ vol –

a Liquids.

b –/0/+/++/+++ a scale ranging from “not appropriate” to “most appropriate”.

c Sludge cake or paste.

d Particulate material.

e The symbols used in this column are:
tm suitable for trace metals;
vol suitable for volatiles.

f Not suitable for representative samples.

g Not suitable for viscous liquids.

h Can be difficult to retain the sample with very dry granular material.

Annex B
(informative)

Vacuum sampling devices

B.1 Thick sludge from open vessels

For thick, e.g. primary, sludges, the vacuum sampler illustrated in Figure B.1 has been used successfully in circumstances such as storage tanks that have not been fitted with pipework for stratified sampling. Sample uptake pipes can be set to sample at predetermined depths from the top of the tank. Construction consists of a pipe of 25 mm bore, electrically earthed to the tank, in up to a maximum of five 2 m sections joined by screw connections which do not reduce the bore. This is connected via a flexible pipe and valve to a 10 l bottle or Buchner flask which is surrounded by a guard to prevent injury should it collapse; it can be evacuated either by hand or by an electrically operated vacuum pump fitted with an intrinsically spark-free motor.

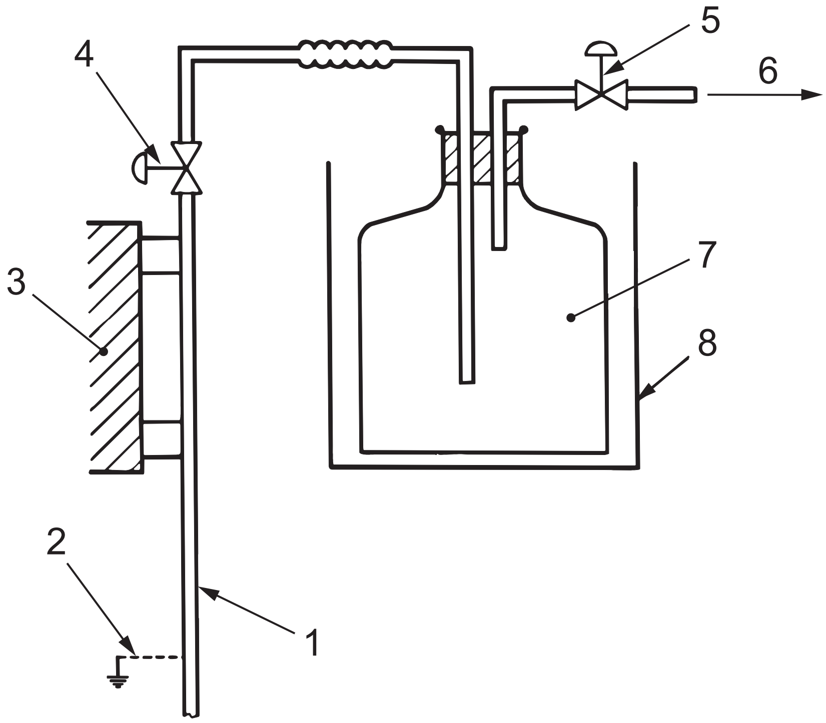
It is necessary to obtain a good vacuum in the bottle before opening the valve to the sampling line. Before taking a sample, some sludge should be withdrawn into another clean 10 l bottle to flush out the pipe. The volume of the purge sample should be three times the standing volume of the sampling arm. This method is particularly suitable for sampling from digesters, either through a port on the roof or through a sludge seal in floating head types. It is important to remove encrusted sludge from the sampling point before inserting the aluminium pipe. In order to ensure that the sample of sludge is representative, the siting of the uptake tube needs careful consideration.

NOTE In situations in which a very viscous layer develops in a stratified sludge, such equipment can draw up less viscous material and overlying rainwater, thus generating an unrepresentative sample.

The equipment has been demonstrated to be unsuitable for sampling sludges with dry solids contents greater than a mass fraction of 6 % to 8 %.

B.2 Thin sludge

For sampling thin sludges (i.e. those of low solids content), e.g. in blanket clarifiers or final settlement tanks, a suitable commercial sampler using small-bore (not less than 6 mm diameter) plastics tube can be used. Care should be taken to clean all pipework after use to avoid accumulation of bacterial slimes or algal growths on the internal pipe surfaces.



Key

1	25 mm bore (intake)	5	outlet valve
2	electrical earth to tank	6	to vacuum
3	support	7	10 l sample bottle
4	intake valve	8	protective covering

Figure B.1 — Suggested apparatus for the sampling of thick liquid sludge under vacuum

Annex C
(informative)

Apparatus for sampling from pipes under pressure

The suggested valve arrangement (see Figure C.1) should be connected to a system of gauges capable of measuring and equilibrating pressures in the sampling vessel with the pressurized pipe to which it is connected. The device acts as a pressure lock to allow a controlled decompression into the sample vessel. This operation is an aid to the safe handling of the sample and considerably reduces the effects of shear on the sludge. The following steps outline the operating procedure.

a) Connect the apparatus vertically to the high-pressure line at point A with all valves closed.

b) Open valve D and admit compressed air until the pressure in the apparatus is equal to the operating pressure of the filter press or pipe in question.

c) Close valve D and open valve B.

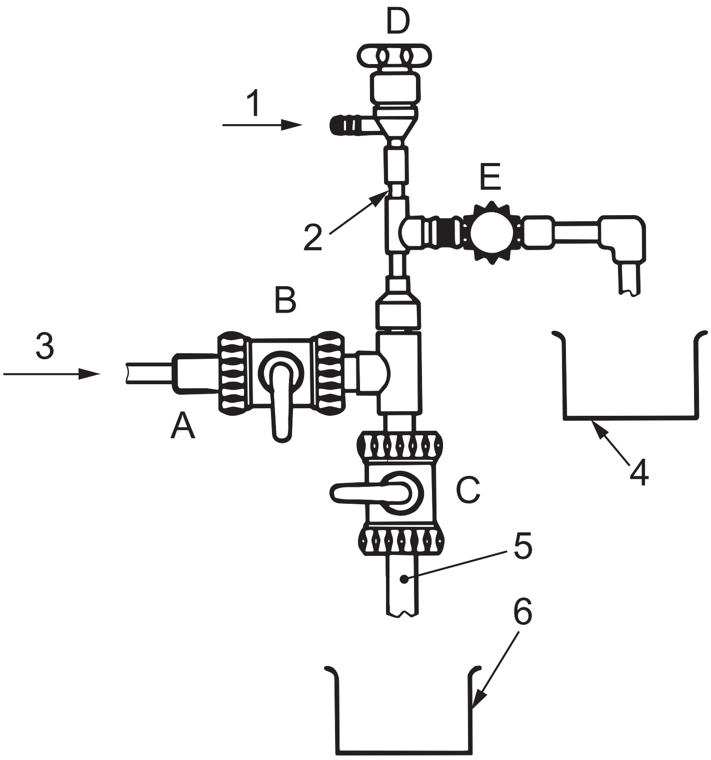
d) Slightly open valve E to allow air to escape and sludge to be sampled through the open valve B.

e) When sludge appears at the outlet of valve E, the sampling compartment is full of sludge. Close valve E.

f) Close valve B and open valve E to reduce the pressure to ambient atmospheric conditions.

g) Open valve C and withdraw the sludge sample.

In order to compensate for the dead volume of sludge in the pressure sampling line A, the above procedure should be repeated to provide a purge equivalent to three times the volume of the pipe being sampled. This ensures that new sludge is drawn off as the sample.



Key

1	compressed air intake	4	container
2	pipe, 15 mm outside diameter	5	PVC pipe, 30 mm outside diameter
3	high-pressure sludge line	6	sample-collecting container
A, B, C, D, E	see text		

Figure C.1 — Suggested valve arrangement for sampling liquid sludge under pressure

Annex D
(informative)

Minimum number of samples in a composite sample — Calculation example

D.1 General

The minimum number of sub-samples required for taking composite samples is specified in 6.1.4.2. D.2 and D.3 describe an example to illustrate the process.

D.2 Test sampling

A test sampling was carried out to determine typical variability between sludge samples and to determine how many samples should be collected for a composite sample. The analytical results from 30 samples collected are presented in Table D.1.

Table D.1 — Mass fraction of copper in 30 test samples

Copper mass fraction mg/kg dry mass		
235	275	218
216	270	242
284	274	289
291	290	323
293	289	297
238	229	264
224	276	295
256	201	240
322	346	336
321	335	319

The average copper mass fraction in the sludge, estimated from the 30 samples, is 276,3 mg/kg dry mass.

It is decided that the maximum permitted error in the estimated sludge copper mass fraction should be ±30 mg/kg dry mass.

D.3 Calculation

To calculate the minimum sample number for taking composite samples, Equation (2) is used:

$$n = \left(\frac{1{,}96 \times s}{E} \right)^2 \qquad (2)$$

where

1,96 is the z-value (number of standard deviations from the mean) for 95 % confidence;

s is the standard deviation estimated from test sampling;

E is the maximum permitted error, expressed in the same units as s;

n is the number of samples.

Based on the test sampling data, the standard deviation of the copper mass fraction, $s = 39{,}7$ mg/kg dry mass.

It is decided that the maximum permitted error in the copper mass fraction, $E = \pm 30$ mg/kg dry mass.

Substituting these values into the equation:

$$n = \left(\frac{1{,}96 \times 39{,}7}{30} \right)^2 = 2{,}60^2 = 6{,}73 \approx 7$$

Since it is not possible to take part of a sample, the result is rounded up to the nearest integer.

So, in this example, seven samples should be collected and mixed together to form a composite sample.

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