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Water quality — Sampling —

Part 13:
Guidance on sampling of sludges from sewage and water treatment works

Qualité de l'eau — Échantillonnage —

Partie 13: Guide pour l'échantillonnage de boues provenant d'installations de traitement de l'eau et des eaux usées

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Foreword

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

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

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

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

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

— *Part 2: Guidance on sampling techniques*

— *Part 3: Guidance on the preservation and handling of samples*

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

— *Part 5: Guidance on sampling of drinking water and water used for food and beverage processing*

— *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 on sampling of groundwaters*

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

— *Part 13: Guidance on sampling of sludges from sewage and water treatment works*

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

— *Part 16: Guidance on biotesting of samples*

Annexes A, B and C of this part of ISO 5667 are for information only.

Introduction

This part of ISO 5667 should be read in conjunction with ISO 5667-1, ISO 5567-2 and ISO 5667-3. 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.

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 that will be 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 will assist in the accurate determination of results.

Water quality — Sampling —

Part 13:

Guidance on sampling of sludges from sewage and water treatment works

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, for example septic tank sludges. Guidance is also given on the design of sampling programmes and techniques for the collection of samples.

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

— provide data for the operation of activated sludge plants;

— provide data for the operation of sludge treatment facilities;

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

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

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

  a) addition or withdrawal of solids;
  b) addition or withdrawal of liquid;

— provide information for legally enforceable aspects of the disposal of sewage and waterworks' sludges;

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

— optimize costs; for example for the transport of sludges for treatment and/or disposal.

NOTE 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, such as inadequate storage temperatures or the sampling of unrepresentative parts of a treatment plant.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 5667. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 5667 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 5667-2:1991, *Water quality — Sampling — Part 2: Guidance on sampling techniques.*

ISO 5667-3:1994, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples.*

ISO 5667-12:1995, *Water quality — Sampling — Part 12: Guidance on sampling of bottom sediments.*

ISO 5667-14: —[1], *Water quality — Sampling — Part 14: Guidance on quality assurance of environmental water sampling and handling.*

ISO 8363: —[2], *Measurement of liquid flow in open channels — General guidelines for selection of method.*

ISO 10381-6:1993, *Soil quality — Sampling — Part 6: Guidance on the collection, handling and storage of soil for the assessment of aerobic microbial processes in the laboratory.*

3 Definitions

For the purposes of this part of ISO 5667, the following definitions apply:

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

[Based on ISO 6107-2]

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

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

[Based on ISO 6107-2]

[1] To be published.
[2] To be published. (Revision of ISO 8363:1986)

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

NOTE This usually applies to liquid sludges; further guidance can be drawn from ISO 5667-10.

3.4 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 flowrate (in the case of continuous sampling), is directly proportional to the flow rate of the sampled sludge

4 Sampling equipment

4.1 Materials

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 will be necessary to assess the potential for interference on any test results that the equipment may have. For example, the use of aluminium extension pipes to a sampling valve would be inappropriate if the samples were being taken for the analysis of an aluminium flocculation assister. 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.

Tools should be chosen to avoid contamination by substances of interest. They should be kept clean and corrosion free. Plastics utensils and polytetrafluoroethylene pallet knives may be used if they prove to be robust and the absence of any contaminating influence can be demonstrated. High alloy steels should be avoided if trace metals are to be determined. The use of stainless steel tools is routinely adopted but the possibility of contamination needs to be recognized and tested for if analyses for elements such as chromium are to be performed on the sludge sample. Old, rusty tools or those with chipped or flaking surface coatings and painted surfaces should not be used, as they may contribute to random contamination of samples.

Polyethylene, polypropylene, polycarbonate and glass containers are satisfactory from the point of view of chemical stability when sludge sampling (see also 6.1). However, caution should be exercised since containers can become pressurized due to gas production in wastewater sludges and explosive situations may occur. Guidance on overcoming this problem is given in clause 7.

Glass containers should be used when organic constituents, such as pesticides, are to be determined whereas polyethylene containers are preferable for sampling parameters of general interest such as pH and dry matter. Polyethylene containers may not be suitable for collecting samples to be subjected to some trace metal analysis (for example 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 5.3.3, and can prove to be a serious potential source of error if not eliminated.

Refer to standard analytical procedures for detailed guidance on the type of sample container to be used. For guidance on the cleaning of sample containers, see ISO 5667-3.

4.2 Equipment

In general, sludge sampling equipment is usually most practical if it is as simple in design and construction as possible. The characteristics of a sludge can vary according to type and solids content, and therefore the manner of handling in a sampling device is dependent upon the physical properties; no general recommendations can therefore be given but some specific examples of equipment for liquid sludges under particular circumstances are given in annexes A and B.

5 Sampling procedure

5.1 Sampling regime

The most appropriate way of sampling in any situation will depend 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; and

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

On a fixed plant, when planning a sampling exercise, it is recommended that a review of the practicalities of the site location is undertaken prior to establishing the safest and most practical position for manual sampling. The representative nature of the resultant sample will also play a key role in the final choice of position.

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. For further information see ISO 5667-1 and ISO 5667-14. There may be a requirement to consider the representative nature of solid sludges. For this purpose further guidance on the statistical assessment of bulk loads of solid materials can be found in ISO 1988.

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

5.1.1 Sample type

The basic types of sample which may be required are:

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

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

To calculate the maximum sampling interval, t, in minutes, between taking samples when using time-based sampling, equation (1) should be used;

$$t = \frac{60\,Q}{Gn} \qquad \text{. . . (1)}$$

where:

Q is the mass of the batch (in tonnes);

G is the maximum flowrate (in tonnes/hour);

n is the number of samples.

5.1.2 Composite sampling

5.1.2.1 Continuous sampling

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

5.1.2.2 Intermittent or consignment sampling

For this type of sampling, samples are not normally taken at uniform intervals throughout the whole supply of sludge before being composited. Instead, 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, 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 will be influenced by the variation between batches, which cannot be predicted. More samples will be 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.

5.1.2.3 Flow-related sampling

This is accomplished by extracting at the end of each time interval a mass of sludge proportional to the flowrate at the sampling point. 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 will drop and the flow proportionality will change. If there is a requirement for mass transfer information, it is prudent to measure the associated flowrate and/or batch size of the sludge. For example, daily metals-loading information may be required for sludge pumped to agricultural land. For further guidance see ISO 8363.

5.2 Replicate sampling

In a situation where automatic sampling is to be installed, for example 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 duplicate 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 duplicate, the results should be examined and the number of samples or the number of batches sampled should be changed to reflect the guidance given in ISO 5667-1 and ISO 5667-14. After carrying out this exercise, it may 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. ISO 1988 gives details on the calculation of the number of samples if the material can be likened to a mineral.

If an occasional confirmation of the sampling performance is required, replicate sampling is ideal. It is recommended that this is achieved by taking a run of 10 samples in duplicate (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 duplicate results have been obtained and compared. If at any time there is reason to believe that sampling conditions have changed, it is recommended that a further set of 10 duplicate samples be collected and statistically tested before any decision to alter the regime is made.

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

5.3 Methodology

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

5.3.1 Sample size

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.

a) Liquid sludges

It should be noted that thin liquid sludges (of low solids content) will 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 such as 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 will need to be homogenized before subsampling. The mixing process should preferably be tested to ensure efficacy of mixing. The homogenization can be achieved in a container such as a plastics dustbin using a suitable paddle to prevent settlement.

b) Sludge cake

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

5.3.2 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 such as stepped draw-off pipework. Inspection of the tank concerned will usually reveal the presence of these facilities if they have been built in. 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 and the sludge in the tank should, where possible, have been thoroughly mixed before sampling. This practice minimizes the need for sampling stratified material, since the whole sludge production is treated as a composite. When this cannot be achieved, interpretation of analytical data will need to be carried out with caution.

A grab sample can be taken from a road tanker by sampling the discharge using a long-handled ladle. A valuable procedure for obtaining a composite sample from a tanker discharge is to divert the flow at random intervals into a separate container such as a barrow to allow separate mixing and subsequent sampling. This technique assists in removing some of the problems of stratification that may occur when some sludges are left standing in tanks or tankers, for example with easily settleable sludges.

5.3.3 Sampling from pipes

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. (see 5.1.1). However, factors such as the nature of the sludge, the flowrate, the diameter of the pipes and the roughness of the pipe can affect the tendency of the dynamic system to allow streaming of the flow. 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 subsample after mixing. Any sidearms 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 flowrate and consistency remain constant. Blockages in pipes due to fibrous materials will often influence the nature of the sludge by a filtering action, thereby giving spurious results. This may go undetected at the time of sampling, necessitating repeating the exercice to assess the reliability of results.

After sampling, the samples may be bulked to provide a composite sample or analysed separately to determine a profile, for example the withdrawal of sludge from a blanket clarifier or a primary tank. Sampling the discharge from road tankers can be achieved by using a long-handled ladle.

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 to reduce this problem. 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.

5.3.4 Sampling from open channels

A weighted bucket or a pump should be used, depending on the solids content of the sludge. A solids content of up to 5 % can be sampled from an open channel provided, when using a pump, the velocity in the suction pipe is sufficient to keep all the particulates in suspension. This velocity will have 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 may 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.

5.3.5 Sampling of sludge cake from heaps and stockpiles

In general, this is often not required and safety requirements usually prohibit routine sampling in this manner. 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 will distort 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 may be the most practical tool, but care is particularly necessary to ensure representative sampling.

If after assessment of safety requirements and the availability of equipment, core sampling can be considered as a means of obtaining samples. Samples should be taken through the depth of the heap/stockpile, and a composite sample prepared from, nominally, n_{sp} such cores,

$$n_{sp} = \frac{\sqrt{V}}{2} \quad \text{[given to the nearest whole number]}$$

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

It is recommended that the value of n_{sp} lie between 4 and 30. Further guidance on core sampling can be obtained from ISO 5667-12.

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 and/or use analytical results. The surface layers may 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.

5.3.6 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 is not recommended as routine practice, but where circumstances dictate it is necessary, for example on delivery to a landfill, then the guidance given in 5.3.6.1 will apply.

5.3.6.1 Sampling from the tops of wagons

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.

5.3.7 Sampling from belt conveyors

5.3.7.1 General

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

5.3.7.2 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.

5.3.7.3 Sampling from a belt terminus

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

Where it is not possible to stop the conveyor, the sludge should to be sampled whilst the conveyor is in motion. When practicable, the sample may be taken as the sludge passes from the loading booms into wagons/hoppers; when this is not possible, the sludge may 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 may be erected to make access for manual sampling easy and safe, see clause 7.

Such techniques are useful for taking representative samples when sludge cake is being loaded onto 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 5.3.8).

5.3.7.4 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 is 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 may 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 will not be of excessive mass, see clause 7.

5.3.7.5 Mechanical sampling from a moving belt

Machinery has been developed for the sampling of minerals from conveyor belts and falling streams which may be converted for use with sludge cake. However, the adaption 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.

5.3.8 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 may 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 Storage, preservation and handling

6.1 General

The methods of sampling may be time-dependent in terms of the analytical technique to be used (for example pH change over time). In addition, if there is an immediate operational requirement for information, a loss of confidence may be acceptable. Judgements will have to be made on a situation-specific basis. For example, when temperature is the parameter to be monitored, the sample homogeneity may not be regarded as critical. For further guidance see ISO 5667-3 and ISO 10381-6.

NOTE 1 Further specific guidance is being developed on the preservation and handling of sludge samples; it is intended that this work eventually be published as ISO 5667-16.

For some types of liquid sludge, particularly raw sewage sludge, gross atypical solids, such as rags, may be removed by passing the sample through a stainless steel or plastics screen of aperture size not less than 5 mm.

NOTE 2 It should be remembered that stainless steel contains chromium and nickel. Neither would be expected to be a significant problem in terms of release to the sample, but awareness of the presence of these metals would be prudent when extremes of pH are encountered. With plastics screens, the plasticizer used in manufacture may interfere with biocide analysis.

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

6.2 Containers and sample preservation

Sample containers should be chosen with care. Specific preservation guidance can be obtained from ISO 5667-3 and in all cases the analyst should be consulted (also see note 1 to 6.1.)

Samples for total moisture determination should be collected and stored in containers that are both leaktight, 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 buildup in the containers.

It is recommended that all glass containers used for sludge sampling where gassing may occur be wrapped with waterproof adhesive tape or other equivalent measures taken, such as wrapping in plastics mesh. These measures will then minimize the dispersion of fragments of the container if an explosion occurs. Some manufacturers may be able to offer self-regulating pressure equalization closures for glass containers. Except for samples to be analysed for trace organic materials, double polyethylene bags can be used for sludge cake samples. The sample should be placed in the inner bag and sealed, and this sample bag and the label placed inside the outer polyethylene bag and that sealed. Cloth bags are unsuitable, as they are neither waterproof nor dustproof.

6.3 Storage

See also note 1 to 6.1.

For classical analyses, full sample containers should be stored in a cool place, preferably at a temperature lower than that prevailing when the sample was taken, giving due regard to local climatic conditions. Samples should be identified, packed, stored and transported wherever possible at 4 °C ± 2 °C to avoid the possibility of loss of volatiles and to minimize biologically induced change. Storage in the dark should be maintained to avoid stimulation of biological activity.

NOTE — If microbiological examination of the samples is required, the preservation temperature may be critical to specific microorganism populations. In this case guidance should be sought from the examining laboratory with respect to the precise requirements.

When trace organic determinations are required, the analysis is usually performed on samples ‘as-received’ (not dried before use). Only glass containers should be used, with appropriate precautions applied with respect to gas production and pressure buildup. If trace organics are not suspected to be significantly volatilized into the gas phase, a regime of regularly opening the container to relieve pressure during storage can be adopted. If significant gas phase volatility is suspected, analysis should be undertaken as soon as possible after sampling. Fermentable samples (nearly all biologically derived sludges) should, where possible, not be stored in glass containers without rendering the biological activity inert, for example by refrigeration, because of the risk of explosion due to gas generation.

Storage at the laboratory is recommended until all data are obtained, in case spurious results require investigation. In some cases preservatives may need to be added. If the sample is air-dried, then the sieved sample should be stored in a closed polyethylene or glass container.

Some form of heat treatment, such as autoclaving, prior to storage may be appropriate, for example 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.

6.4 Sample size reduction for sludge cakes (quartering)

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 portion of mass appropriate to the test in hand. 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, for example 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, may be more appropriate. Division into subsamples can still be achieved by the combination of diametrically opposed quarters.

6.4.1 Sample reduction to provide multiple subsamples

When two or more laboratory samples are required from a bulk sample, the sample mass should be reduced by quartering. All 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 and/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. For example, after homogenization of a subsample 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) may 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 will not divide properly and may cause clogging.

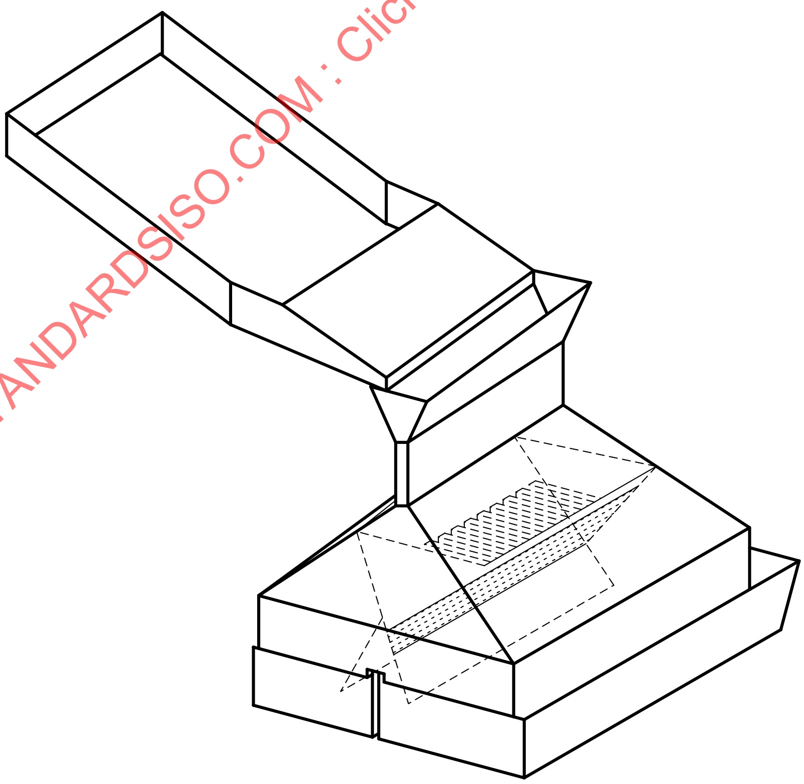

Figure 1 — Typical riffle box

7 Safety

It may 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. In general, the safety advice given in other parts of ISO 5667 is relevant on many occasions when sludge is sampled, for example see ISO 5667-10. Examples of the type of risk that may be present with respect to sludge sampling in particular are given below.

a) Sludge-handling plants are often associated with wet flooring which can be covered in minor sludge spillage, for example press houses. Slipping on floors while sampling can be avoided if chosen sampling points are protected by raised slatted floor panels.

b) Bacterial and parasitic infection can occur through accidental ingestion or infection via unprotected broken skin and mucus membranes. These risks can be minimized by the adoption of strict personal hygiene codes and the wearing of gloves and goggles.

c) Dangerous atmospheres exist in confined spaces such as the wet wells of pumping stations, for example oxygen deficiency, toxic gases and vapours, flammable and explosive gases and vapours. Most sampling environments display signs warning of potential dangers, but atmospheric constituents should always be monitored if the conditions have not previously been assessed. It is essential that personnel engaged in sampling make themselves familiar with the safety equipment and procedures available and the operation of gas/vapour testing equipment that is used to assess the quality of the atmosphere. Particular care should be taken at anaerobic digestion plants due to potential explosion risks. Entry into confined spaces under these conditions is often subject to national regulations.

d) Flooding is a risk probably only associated with sewers and holding tanks. The overriding rule should always be that such areas are never entered during sampling unless exceptional circumstances dictate the need. Where a flooding risk exists, safety personnel should always be present.

e) Sampling from sludge cake stockpiles is not without some risk of personal injury, and care should be taken to avoid the risk of becoming trapped by inducing slippage or sinking.

f) Regard should be given to the requirements for safe working practice when working with moving machinery, for example conveyor belts and press plates.

g) It is suggested that the maximum mass of a sample to be lifted by an individual be 25 kg.

h) Where sludge is sampled using pressure or vacuum systems, care should be taken to prevent exposure of personnel to sludge aerosols formed during the sampling process. Otherwise infectious agents can enter the body via the lungs.

The provisions of national health and safety regulations should always be carefully studied and put into effect before sampling occurs.

8 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 including the results of any on-site tests carried out by the sampler (for example pH). Alternatively, a codified system, for example using bar codes, should be used. When many sample containers are needed for a single occasion, it is recommended to identify the containers by code numbers and to record all relevant details on a sample record form. Labels or forms should always be completed at the time of sample collection.

The detailed form of sample report will depend 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 (for example, keep refrigerated).