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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;

$$t = \frac{60\,Q}{Gn} \quad \ldots (1)$$

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.