INTERNATIONAL STANDARD

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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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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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):

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

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:

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

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.