International Standard

ISO 5667-10

Water quality — Sampling —

Part 10: Guidance and requirements on sampling of waste water

Third edition
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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

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

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This third edition cancels and replaces the second edition (ISO 5667-10:2020), which has been technically revised.

The main changes as follows:

— EN 16479[10] has been integrated in this document;

— microplastic sampling has been excluded from the scope.

A list of all parts in the ISO 5667 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Water quality — Sampling —

Part 10: Guidance and requirements on sampling of waste water

1 Scope

This document specifies the sampling of urban and industrial waste water, i.e. the design of sampling programmes and techniques for the collection of samples. This document covers waste water in all its forms, i.e. industrial waste water, radioactive waste water, cooling water, raw and treated urban waste water.

This document deals with various sampling techniques used and the rules to be applied to ensure the samples are representative.

This document does not cover sampling of accidental spillages, although the methods described in certain cases can also be applicable to spillages.

This document is not applicable to sampling of microplastics in waste waters. Appropriate guidance is given in ISO 5667-27[1].

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2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

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

ISO 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples*

ISO 5667-7:1993, *Water quality — Sampling — Part 7: Guidance on sampling of water and steam in boiler plants*

ISO 5667-14:2014, *Water quality — Sampling — Part 14: Guidance on quality assurance and quality control of environmental water sampling and handling*

ISO 5667-16, *Water quality — Sampling — Part 16: Guidance on biotesting of samples*

ISO 19458, *Water quality — Sampling for microbiological analysis*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

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3.1
composite sample
two or more samples or sub-samples, mixed together in appropriately known proportions (either discretely or continuously), from which the average value of a desired characteristic can be obtained

Note 1 to entry: The number of samples or sub samples are usually based on time, flow measurements, area or depth profile sampling.

EXAMPLE Composite sample can be made in different ways:

— constant volume variable time sampling (CVVT): flow proportional sampling based on collecting equal volumes of sample at frequencies proportional to flow.

— constant time variable volume sampling (CTVV): flow proportional sampling based on collecting samples at fixed time intervals but where the volume of sample is varied in proportion to the flow.

— constant time constant volume sampling (CTCV): equal volumes of sample or sub-sample collected at equal increments of time.

[SOURCE: ISO 6107:2021, 3.126[2], modified — the EXAMPLE has been added.]

3.2
sampling point
precise position within a *sampling site* (3.3) from which samples are taken

[SOURCE: ISO 5667-12:2017, 3.8[3]]

3.3
sampling site
general area or location from which samples are taken

[SOURCE: ISO 5667-6:2014, 3.10[4]]

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3.4
spot sample
discrete sample taken randomly (with regard to either time or location, or both) from a body of water, usually taken manually, but can be taken by automatic sampling equipment or by event-triggered automatic samplers

3.5
qualified spot sample
special form of a *composite sample* (3.1), consisting of at least five *spot samples* (3.4), taken and mixed within a maximum period of two hours and at an interval of not less than two minutes

Note 1 to entry: In this definition, mixing signifies to carry out using either constant volume or variable volume, in relation to the monitoring objective. The proportion in which spot sample need to be mixed should be clearly mentioned.

3.6
radioactive liquid effluent
water or waste water that contains radioactive substances, resulting from a process and that can be either recycled, treated or discharged to the environment, or all

Note 1 to entry: The activity concentration of the radioactive liquid effluent is usually measured before being discharged in the environment to verify that it is lower than the authorized levels in order to comply with national regulations.

3.7
supernatant
solid or liquid phase present on the surface of an effluent

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3.8
planned discharge
discharge subject to prior agreement further to a consultation between several parties based on knowing certain predefined parameters and referring to limit values (regulatory or otherwise)

Note 1 to entry: These parameters can be, for example, physical, chemical and radiological measurements, the estimated discharge volume, the discharge period or the maximum discharge flow rate.

3.9
permanent discharge
direct discharge into a channel or collector or water body, which is not subject to a specific prior agreement, but respects limit values

3.10
storage container
hollow object, very variable in size and shape, used to hold liquids

Note 1 to entry: Covers the usual names such as tank, lagoons and basins. The content of this storage container is intended for direct and indirect liquid discharge to the environment or to a specific treatment.

3.11
event-triggered sampling
sampling which is triggered when a pre-determined criterion has been met (e.g. rainfall, change in electrical conductivity, pH or the introduction of a polluting substance), when samples need to be taken manually or by automatic equipment

3.12
bias
estimate of a systematic measurement error

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Note 1 to entry: The systematic measurement error is a component of measurement error that in replicate measurements remains constant or varies in a predictable manner.

[SOURCE: ISO/IEC Guide 99:2007, 2.18[5], modified — Note 1 to entry has been added.]

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3.13
measurement repeatability
repeatability
measurement precision under a set of repeatability conditions of measurement

Note 1 to entry: A repeatability condition of measurement is a condition of measurement, out of a set of conditions that includes the same measurement procedure, same operators, same measuring system, same operating conditions and same location, and replicate measurements on the same or similar objects over a short period of time

Note 2 to entry: A condition of measurement is a repeatability condition only with respect to a specified set of repeatability conditions.

Note 3 to entry: In chemistry, the term “intra-serial precision condition of measurement” is sometimes used to designate this concept.

[SOURCE: ISO/IEC Guide 99:2007, 2.21[5], modified — Notes 1, 2 and 3 to entry have been added.]

3.14
representative sample
sample with the same quality and characteristics for the material of interest as that of its source at the time of sampling

[SOURCE: ISO 2889:2023, 3.63[6]]

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4 General aspects

4.1 Design of sampling programme

Sampling is usually the first step in carrying out an investigation and largely determines the quality of the whole investigation. It is therefore recommended that a detailed sampling strategy be drawn up, often based upon a preliminary investigation in which an assessment has been carried out to identify the important aspects. Both the purpose and the ambient situation determine the way in which the sampling is to be carried out. General aspects for sampling programme design given in ISO 5667-1 shall be followed.

4.2 Sampling point selection — Representativeness

The selected sampling point should be appropriate for the collection of samples that are representative of the waste of discharge to be examined. In some waste waters, this representativeness can be difficult to obtain because of the spatial and temporal heterogeneity of the water body. It is necessary to carry out the sampling in the sections where the flow is well mixed and homogeneous.

The term “representativeness” encompasses two notions depending on the type of environment to be sampled:

— representativeness in a flow (canal, sewer, manhole, pressurized pipes, etc.);

— representativeness in a storage container (tank, lagoons, basins, etc.).

These two options should be treated in different ways, but the goal remains to obtain a representative sample of the water body.

Sampling points can be clearly identified by regulatory text. If they are not, a preliminary investigation is recommended. This is generally the case for the selection of sewer sampling locations. By first studying technical drawings of the sewer system, potential sampling points should be identified. Subsequently, a site inspection should be conducted to ensure that these sampling are representative. Each sampling point should be documented. It is important to gather, for example, the following information: sampling point identification, sampling site (photos, geographical coordinates), type of flow (open, closed), access conditions and sampling technique.

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The following facilities should be available for the sampling sites involving a fixed automatic sampling device:

— access for suitable carrier to the immediate vicinity of the sampling site;

— flat working surfaces at appropriate height above the sampling point for the set-up of sampling devices;

— adequate lighting and power connection;

— water supply to clean the equipment after sampling;

— adequate safety precautions (e.g. grids, railings, fall arresting devices); and

— flow meters in the case of a flow-dependent sampling.

If the hydraulic conditions do not ensure the representativeness of the sample (e.g. absence of flow, reduced activity, abnormal load rise), this unusual situation should be noted on the sampling report and the customer and the analytical laboratory should be informed.

4.3 Frequency and time of sampling

4.3.1 Number of samples

The decision on the required number of samples taken during each period should be decided based on statistical techniques (see ISO 2602[7], ISO 3534 (all parts)[8] and ISO 5667-1). However, the number of samples to be taken can often be decided by a regulatory body or pollution control authorities.

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4.3.2 Sampling time for effluent stream

The objective of a sampling programme often dictates when and how a sample is collected and is often determined by legislation or directives. Generally, when sampling sewages and effluents, it is normal to make allowances for the following sources of variation in quality:

a) diurnal variations (i.e. within-day variability);

b) variations between days of the week;

c) variations between weeks and months;

d) variations between seasons;

e) variations due to storm water episodes;

f) trends; and

g) incident (general accident).

If there is little or no diurnal variation, or day-to-day variations, then the time of day or day of the week for sampling is relatively unimportant.

If the identification of the nature and magnitude of peak load are important, sampling should be restricted to those periods of the day, week or month when peak loads are known to occur.

When assessing industrial effluent discharges that are either seasonal or batch-based, sampling times should be aligned with the process being monitored. As the discharge will not be continuous in either case, the sampling programme shall be take this into account.

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Sampling for the detection of trends needs careful planning. For example, when detecting trends on a month-to-month basis, it can be appropriate to always sample on the same day of the week, in order that any diurnal and daily variations are eliminated from the overall variability of data, thus allowing trends to be more efficiently detected.

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When the number of samples has been decided upon according to 4.3.1, the sampling times should be determined. The samples should normally be taken at fixed intervals during the whole sampling period. The sampling period may be one year, a number of months or weeks, or even shorter periods of time.

If the sampling period covers one year, the days of sampling may be determined using a formula. An example of this is:

— Formula (1) should be applied when the number of samples (n) is equivalent or larger than 25 and Formula (2) should be applied when number of samples is less than about 25.

— Formula (1) indicates the day number during which sampling should take place.

$$A+\frac{365}{n}, A+\frac{365\times 2}{n}, A+\frac{365\times 3}{n}, \ldots, A+\frac{365\times n}{n} \quad (1)$$

where

n is the number of samples;

A is a random number in the interval between $-\frac{365}{n}$ and 0.

Formula (2) indicates the week number during which the sampling should take place. The day of each week should be determined so that samples are taken on every weekday.

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$$B+\frac{52}{n}, B+\frac{52\times 2}{n}, B+\frac{52\times 3}{n}, \ldots, B+\frac{52\times n}{n} \quad (2)$$

where

n is the number of samples;

B is a random number in the interval between $-\frac{52}{n}$ and 0.

Similar formulae can be used for other periods, for example, one month, three months, six months, etc. The period chosen should cover any seasonal variations.

After determining the intervals and the day or week number, it should be ensured that the sampling does not lead to any risk of systematic error, for example, by always taking samples on one day, or by systematically omitting weekdays.

5 Sampling at specific locations

5.1 General

The concentration profiles of substances (hydrophilic and hydrophobic substances) to be measured in waste waters are often heterogeneous because they depend on the hydraulic conditions and transport conditions of the solid phase in the water body. The observations of concentration profile of these substances in body of water may be as following:

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— a vertical gradient of concentration, due to the flow velocities or the shear stresses near the bottom are low;

— a very dense layer at the interface between the deposit of the bottom and the water circulating in the structure;

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— an increase of the concentration near the walls;

— an increase of the concentration near the surface of the flow due to the presence of floating matter.

To take a sample representative of the average concentration of the measured section, the sampling point should be placed about halfway up the water column and at a sufficient distance from the walls and from deposits at the bottom to avoid measurement bias.

5.2 Sampling from sewers, channels and manholes

The sampling point should be chosen where the effluent has a high turbulent flow to ensure good mixing conditions. Difficult access, lack of security or power supply unavailable can preclude the selection of alternative sampling points.

Sewers, channels and manholes are generally designed to cope with waste water or storm water discharge conditions. In some cases, channels have to cope with combined effluent and storm water discharges. In the absence of storm water discharge, this type of channel is oversized, resulting in laminar flow.

In the absence of turbulent flow conditions at a permanent sampling point, means for restricting the flow should be implemented, for example using a baffle or weir. The restriction should be made in such a way that sedimentation upstream does not occur (e.g. foresee enlargement of the downstream effluent channel compared to the upstream channel, to avoid any pressure increase of the effluent downstream).

The sampling intake point should always be located downstream from the restriction and, as a general rule, it should be located at least three times the pipe diameter, or width of the channel, downstream of the restriction. The inlet of the sampling probe should preferably face the direction of flow but may face downstream if too many blockages result.

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The exact location of the sampling point should be evaluated with respect to variations in water level, types and concentrations of the determinands, etc. The sampling point should be at a minimum distance from the bottom and walls to avoid sample contamination by deposits or the biofilms that develop. Generally, a sampling point between one third and one half of the effluent water depth below the surface of the water may be recommended.

Whenever practicable, permanent sampling locations should be established, with care being taken to ensure reproducible sampling conditions.

Before proceeding with the sampling of industrial discharges and if the information is accessible, the conditions inside the plant (e.g. processes and production rates) should be noted and recorded along with any potential hazards.

5.3 Sampling from waste water treatment plant

When choosing sampling points for waste water treatment plant, it is again important to refer to the objective of the data collection programme, of which the sampling is a part.

Typical objectives are:

— control of the performance of the entire treatment plant: samples should be collected at the main inlet and main outlet points;

— control of the operation of individual processing units, or groups of units: samples should be collected at the inlet and outlet of the units in question.

When sampling at the waste water treatment plant, the importance and the relevance of any bypass flow should be evaluated. Sampling of such bypass flows can be needed for the sampling to be representative of the overall effluent.

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When sampling at the inlets of plants, there can be a need to sample raw sewage in the mixture with recirculated processing liquid (e.g. in the assessment of primary sedimentation tank loadings and efficiency).

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In other cases, it can be necessary to exclude the effect of these liquids (e.g. when collecting data designed to assess either domestic or industrial loadings to a plant, or both, and to assist in industrial effluent control).

Representative sampling is often facilitated setting the sampling point downstream of a measuring flume or weir (see also 5.2).

When sampling effluents from processes employing more than one individual treatment unit (e.g. several sedimentation storage container), care should be exercised in ensuring that the sample is representative of the overall effluent stream rather than any one specific treatment unit (unless that unit forms the basis of a specific study).

Frequent reviews of a plant's sampling points need to be made to ensure that any relevant changes in the operation of unit processes are considered when sampling. For example, the percolating filter operation can be changed from a “single-pass” operation to a “recirculation” or “alternating-double filtration” operation. Treatment plant operation may involve changes in the manner in which feed or return liquors are introduced to the plant (e.g. return of sewage from storm tanks, changes in the position at which processing liquors are returned to the treatment plant).

Whenever sampling waste waters, great care should be exercised to overcome or minimize the substantial heterogeneity caused by suspended solids that are often present. Similarly, thermal stratification of separate industrial effluent streams may be found when sampling effluents or discharges from industrial processes, and measures have to be taken to promote the mixing of such streams before sampling.

5.4 Sampling from industrial sites

The sampling of industrial effluent can involve taking representative samples either before or after the transfer of effluent to discharge channels or collectors. In some cases, samples may be taken both before and after transfer.

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The sampling method depends on the type of discharge or transfer taking place. The following should therefore be distinguished:

— planned discharges, which require a preceding inspection before the discharge can take place: The effluent in temporary storage containers, should meet the set requirements prior to being transferred to general collectors or buffer pools for further discharge into the environment (refer to 7.4).

— permanent discharges, which concern effluents that are continuously discharged: They are monitored based on sequential or continuous sampling of the discharged effluent. These can be, for example, discharges from an effluent collector at the outlet of an industrial site prior to discharge into the environment.

As such, the kind of sampling and the creation of representative samples shall be adapted to suit the type of discharge and its potential heterogeneity (e.g. the presence of suspended matter or any density or thermal stratification).

For this, and to guarantee the representative nature, the samples shall be produced:

— ensuring sampling only takes place after obtaining suitable effluent homogeneity;

— ensuring there is no change to the nature of the effluent or cross-contamination; and

— taking account of the volumes of the storage container, the flow rates and the flow conditions in the lines, to enable quantification of the discharges from the facility in question (see Figure A.1 for an example of sampling from storage containers).

Sampling may be performed based on several configurations, depending on the facilities:

— in a storage container prior to its discharge (see Figures A.1 and A.3);

— in a continuous or discontinuous flow in a line or a discharge channel (see Figure A.1).

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5.5 Sampling from cooling systems

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The selection of sampling points in industrial cooling processes using water as a coolant depends on the cooling water system to be tested.

In continuous-flow cooling systems, fresh water (ground water, bank filtrate, surface water) or saline water are used in one or repeated flow. The sampling points are located both in front and behind the aggregates, which shall be cooled.

During cooling via the primary or secondary circuits, the two cooling circuits are coupled together. In this process, the closed secondary circuit is cooled again by the primary circuit. The primary circuit can consist of a continuous cooling system, an open or closed recooling plant or a refrigerating plant (e.g. brine cooling). The sampling site should be on the return side and on a well through-flow site of the circulatory system.

In many cases, the cooling water is precooled in an open system by evaporation cooling, whereby water losses due to evaporation, spraying and desludging (desalination) will be replaced by fresh water. In the closed recooling process, the cooling water flows through pipes, which are cooled from outside by air or water. The methods of open and closed recooling can be combined. The sampling site is located on the return side of the circulatory system.

Representative sampling locations shall be defined throughout the system for periodic microbiological and hygiene checks. Microbiological sampling guidance is given in ISO 19458. Samples are preferably taken from the circulating water between the running pump and the spraying or trickling stage. A sampling facility allowing disinfection (preferably by flaming) and draining shall be provided at this location. Before sampling, sampling lines, connectors and taps should be sterilized (if possible) by autoclaving, by flaming or if not possible, by cleaning with a disinfectant solution (refer to ISO 19458). Bottles and sampling equipment should be clean and sterile, sterile both inside and out.

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Allow the water to drain for at least 30 seconds before sampling. Sampling shall be performed in such a way that the results are not distorted by biocide dosage. The sampling location shall be upstream of the biocide dosage point.

If sampling is not possible at this location, a sample can be taken from the sprayed water or by bailing from the circulating-water basin[9].

Special cooling processes are usually used when the medium to be cooled is very hot (e.g. waste heaters, heat pumps) or when very low flow temperatures are required (e.g. brine cooling). For special cooling processes in the high temperature range (i.e. temperature greater than 100 °C), the arrangements specified in ISO 5667-7:1993, 6.4 and 6.5 shall be used.

6 Main types of waste waters sampling

6.1 Spot sampling

In the case of spot sampling (refer to Annex B), it is possible to implement:

a) direct sampling in waste waters:

   1) directly by using laboratory bottles;

   2) by using a ballasted sample collector equipped with laboratory bottles;

   3) by using an automatic sampler.

b) indirect sampling in waste waters:

   1) by using a sampling rod equipped with a collection container;

   2) by using a bucket or other equipment.

Sample Document

NOTE Spot samples are usually taken manually but can be taken by automatic sampling equipment or by event-triggered automatic samplers.

Full document from standards.iteh.ai

6.2 Composite sampling

Composite sampling consists of collecting several discrete samples and combining them into a single (composite) sample. It can be done automatically or manually (see 3.1, Figure A.2 and Annex B).

There are several ways of performing composite sampling:

— constant volume variable time sampling;

— constant time variable volume sampling;

— constant time constant volume sampling.

For manual composite sampling, this approach is equivalent to sampling with fixed time, fixed flow and fixed volume.

7 Waste water sampling

7.1 General aspects

7.1.1 General

The purpose of the sampling should clearly be defined so that the sampler can implement the appropriate sampling strategy.

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