
Water quality — Sampling —

Part 17:

**Guidance on sampling of bulk suspended
solids**

Qualité de l'eau — Échantillonnage —

*Partie 17: Lignes directrices pour l'échantillonnage des matières solides
en suspension*

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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 5667-17 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 5667-17:2000), 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: Guidance on the 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 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 15: Guidance on 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 18: Guidance on sampling of groundwater at contaminated sites*
- *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*

The following parts are under preparation:

- *Part 21: Guidance on sampling of drinking water distributed by tankers or means other than distribution pipes*
- *Part 22: Guidance on design and installation of groundwater sample points*
- *Part 23: Determination of significant pollutants in surface waters using passive sampling*

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Introduction

This part of ISO 5667 reflects the important role of suspended solids in flowing water, especially of the silt plus clay (< 63 µm) component and associated carbon, as a transport medium for nutrients (especially phosphorus), trace metals, and certain classes of organic compounds (see Clause A.1).

Although analysis of suspended solids has been carried out for many years, there are no standard methods for field sampling of suspended solids for water quality purposes (i.e. for physical, chemical, biological and/or toxicological characterisation). While standard methods exist for sampling of water for sedimentological purposes (see ISO 5667-1 [1], ISO 5667-4 [2] and ISO 5667-6 [3]), these are often not appropriate for the chemical analysis of suspended solids due to contamination from the sampler itself and to a lack of sufficient sample volume for reliable chemical analysis. Often, indirect methods of assessing the chemical contribution of the solid fraction (e.g. method of differences, see Clause A.3) provide erroneous results (see Clause A.2) due to problems caused during the filtration process and through the manipulation of analytical results to determine the concentrations of chemical analytes in the particulate phase (see Clauses A.2 and A.3). Because of the lack of standards for sampling of suspended solids for water quality purposes and the improbability of achieving complete standardisation because of differences in the objectives of water quality programmes and the lack of standard apparatus, this part of ISO 5667 provides guidance to the various sampling procedures, their biases, and alternatives. This part of ISO 5667 excludes sampling protocols that apply to conventional water sampling. Field and laboratory filtration procedures that are conventionally used to measure the quantity of suspended solids are also excluded. Any reference to these methods is solely for the purpose of demonstrating their profound limitations for suspended solids quality purposes.

The objectives of a water quality programme will dictate the size of sample required and therefore the type of apparatus to be used. Generally, however, the analysis of physical, chemical, biological, and toxicological properties can require samples of mass measurable in grams to hundreds of grams to be collected, depending on the analysis to be undertaken. Examples of programme objectives that require bulk collection of suspended solids include:

- ambient monitoring for water quality assessment, control or regulation;
- in-river monitoring of effluents for regulatory or control purposes, especially for chemical and toxicological properties;
- research into water quality, including physico-chemical processes that affect the pathways, fate, and effects of suspended solids, and their associated nutrient and contaminant chemistry;
- recovery of suspended solids for purposes of physical analysis, including particle size, organic content including particulate organic carbon, suspended solids geochemistry, inorganic and organic chemistry of suspended solids, and toxicity of suspended solids;
- collecting of suspended solids samples for the purpose of long-term storage (Reference [35]).

Water quality — Sampling —

Part 17:

Guidance on sampling of bulk suspended solids

1 Scope

This part of ISO 5667 is applicable to the sampling of suspended solids for the purpose of monitoring and investigating freshwater quality, and more particularly to flowing freshwater systems such as rivers and streams. Certain elements of this part of ISO 5667 can be applied to freshwater lakes, reservoirs, and impoundments; however, field sampling programmes can differ and are not necessarily covered here.

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

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

ISO 5667-15, *Water quality — Sampling — Part 15: Guidance on preservation and handling of sludge and sediment samples*¹⁾

3 Terms and definitions

For the purposes of this part of ISO 5667, the following terms and definitions apply.

3.1

suspended solids

(bulk sampling) solids with a diameter greater than 0,45 µm that are suspended in water

3.2

bulk suspended solids

solids that can be removed from water by filtration, settling or centrifuging under specified conditions

NOTE Adapted from ISO 6107-2:2006^[4], 139, “suspended solids”.

3.3

isokinetic sampling

technique in which the sample from a water stream passes into the orifice of a sampling probe with a velocity equal to that of the stream in the immediate vicinity of the probe

[ISO 6107-2:2006^[4], 56]

1) To be published. (Revision of ISO 5667-15:1999)

4 Strategies and goals of sampling suspended solids

4.1 Sampling programme and sampling plan

Among the most important steps in monitoring and risk assessment programmes is the design of a suitable sampling plan which should be drawn up in line with the individual goals of the assessment and with the specific demands on the quality of the data.

Sampling strategy includes: identification of the area under investigation, choice of procedure and type of analysis, and choice of location and number of sampling sites. These are then integrated into a sampling programme that takes account of time-related requirements such as seasonality and input patterns.

Sampling should take into account the required accuracy of results, the types of local substrates, the topographic and hydrographical conditions in the area under investigation, information on local sources of pollution, as well as (where available) insights gained from earlier assessments. The number of the sampling points, their location, the number of samples to be taken at each site, and the sample identification system should be determined in advance. Any appropriate adjustments can then be made in the field, in which case the reasons for such changes should be explained logically on the sampling record. Where an investigation of trends is planned, it is important to take the required statistical confidence of the data into account if conclusions on measurable variations during a defined period are to be reached; this requires a statistical evaluation. From a statistical point of view, potential errors during sampling and/or measurement especially affect the variance of the data. For further details on how to devise sampling programmes, see ISO 5667-1 [1].

4.2 The dependency of the content of suspended solids on discharge

The suspended solids content of flowing water is determined in the first instance by the flow velocity, and thus by the discharge of the water under consideration. The higher the speed of flow, the greater too is its eroding power and the period during which sediment particles remain in suspension. This is the reason for the dynamic nature of the transport of suspended particulate matter. In sections where there is a reduced speed of flow (e.g. in dammed areas or in docks) suspended solids deposit as sediments, to be transported further if channel flow begins to increase (Reference [36]).

An accurate interpretation of suspended solids analysis presupposes, therefore, knowledge of the discharge in question and taking the origin (sampling point) into account. For example, as the discharge increases, the suspended solids content often increases exponentially, so that rising floods transport significant parts of the suspended solids, whereby the highest concentrations of suspended solids may occur before the flood has reached its flood peak. The higher contaminant concentrations may cause a significant increase in potential toxicity of the suspended solids (Reference [37]). The supply of sediment is greatly reduced prior to the peak of flow such that lower concentrations of suspended solid may occur after the flood has reached its peak. Often these hydrological phenomena are integrated into the time-integrated sample that is collected.

The composition of the suspended solids can be a reflection of increased erosion and the increased entry of particles from run-off caused by heavy rainfall. Particularly waters with high plankton concentration usually show noticeable increases in the mineral content (shown as a proportion of ignition residues) as drainage increases.

Where waters have been dammed up or regulated and there is only little discharge, both an increased primary production in the reservoirs and an increase of mineral particle sedimentation has been observed — the latter because the particle density is greater than that of the plankton. As drainage increases, the opposite holds, as the lighter plankton are rapidly washed away while the sedimented mineral particles are resuspended (Reference [38]).

4.3 Sampling frequency, duration, and timing

The frequency, duration, and timing of sampling are particularly dependent on the purpose of the investigation.

Depending on the issue under investigation, a single analysis may be all that is required, while for estimating loads, or for making long-term predictions, particularly when measurements show values distributed over a wide range, an adequately based conclusion may require monthly or weekly analyses. Statistical analysis (see ISO 5667-1 [1]) can be useful in assessing whether variations are random (i.e. showing normal distribution) or systematic (trends, cyclic variations).

The length of the period set for collecting the suspended solids depends mainly on the quantity of suspended matter in the water and the mass of sediment required for analytical purposes. Depending on the sampling process, the time needed to obtain the sediment can range from a few hours to several weeks.

The amount of suspended solids is primarily a function of the runoff (discharge) of the water course, and is thus mostly independent of the time of day. Particular hydraulic events such as high and low tide should also be included in the routine so that sampling is truly representative (Reference [39]).

Many contaminants (e.g. those associated with road runoff) can be carried in the early stages of a fresh event. In some cases, it may be useful to target this period to ensure that loadings of contaminants of potential concern are not underestimated.

4.4 Sampling points

Sampling points should be selected so that the results of measurements are representative of an extended section of the river. Site appointment should take account of the existing network of water-monitoring points so that corresponding and complementary results can be obtained for both compartments.

Where causes of pollution are to be identified, sampling points should be sited appropriately in relation to the emission sources under investigation. Often practical considerations, such as access to the water, the accessibility of the sampling point, a suitable site for the portable centrifuge, or the protection of the sampling equipment from vandals, should be taken into account.

Tributary loadings may be needed to enable identification of where source control might be necessary. To facilitate calculations of tributary loadings, it is advantageous to collect suspended sediment samples as far downstream as possible, but above any locations where confluence might be felt.

There should be preliminary investigations at different potential measurement sites to determine for which area, and for which characteristics, a sampling site is representative before making a decision on the site appointment of permanent monitoring points (Reference [39]).

The sampling site should be described by its co-ordinates (easting and northing) and the exact position of each measuring point. In addition, the site should be documented with 1:5 000 and 1:25 000 scale maps and photographs, and the access route described so that new sampling personnel, for example, are able to locate the sampling point. If possible, the sampling point should be marked (e.g. by buoys).

Suitable sampling points are often in the vicinity of bridges or gauging stations, as they are easy to locate. Usually waters are accessible at such points even when water levels are higher than normal. The corresponding discharge can be determined from water gauges.

5 Sampling equipment

5.1 General

There are a number of different sampling techniques with differing apparatus for the bulk collection of suspended solids. Many of these samplers are specific to site conditions and can require deployment from boats, bridges or by wading.

Guidance on the volumes of material that are required for various types of physical, chemical, biological and toxicological analysis is given in ISO 5667-15.

5.2 Passive samplers

This class of samplers includes the conventional suspended solids samplers such as depth integrating and point samplers. Passive samplers are placed in the water column where they fill under ambient conditions using isokinetic sampling methods. These samplers are generally used in conjunction with standard sampling protocols for the collection of the most representative mineral solids sample in a given riverine cross-section, such as the equal discharge increment and equal width increment methods (References [7], [8], [9]).

The majority of standard samplers described in Reference [9] were developed for quantity and not quality determinations of suspended solids. Their use is not recommended for solids quality sampling, due to small sample volumes, contamination of the sample by the materials used in the construction of these samplers, and other technical and methodological factors (Reference [14]).

5.3 Bag sampler

The large-bag passive sampler (6,5 l) described in Reference [10] was developed specifically for suspended solids quality due to its large capacity and construction from chemically inert materials. Multiple bag samples were generally composited to produce a sample of sufficient volume to obtain enough suspended solids for subsequent chemical analysis. The bag sampler is also used in conjunction with bulk samplers described in 5.4.

5.4 Bulk samplers

Bulk samplers are used for dewatering large (bulk) quantities of suspended solids. Field bulk samplers include tangential flow filtration and centrifugation. These both require a large volume of water/solids mixture to be taken, or pumped, from the water column to the bulk sampler. This part of ISO 5667 refers only to those methods that can be deployed in the field. Therefore, bench centrifuges and other laboratory methods of dewatering such as sedimentation, are not dealt with here.

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6 Methods for sampling suspended solids

6.1 General

As there are as yet no standardised instructions for sampling suspended solids, it is important to observe a standard procedure so that long-term observations are comparable.

The following criteria (Reference [25]) are significant when deciding on a sampling procedure:

- a) the horizontal distribution of suspended solids;
- b) the vertical distribution of suspended solids;
- c) the spatial and temporal distribution of suspended solids at constant rates of discharge (basic discharge) or during fast variations of discharge (flood discharge);
- d) the varying composition of suspended solids, depending on the sampling strategy or procedure;
- e) sample quantity, to minimise the error resulting from irregular distribution of suspended solids in the water and to meet analytical requirements.

Suspended solids are sampled by a variety of sampling methods that use different equipment:

- a) centrifuging methods (e.g. continuous-flow centrifuges);
- b) sedimentation methods (e.g. sedimentation tanks and boxes, floating collectors);
- c) filtration methods (normal, pressure, and vacuum filtration).

Some of these methods involve the extraction of larger volumes of water/solids mixture from a river. This part of ISO 5667 is only concerned with *in situ* procedures, which is why laboratory centrifuging and other laboratory-based separation methods are not dealt with here.

6.2 Centrifuging methods

6.2.1 General

Sampling devices that rely on centrifuging procedures are referred to as clarifiers or, more usually, centrifuges. These devices operate with a constant flow; the water is pumped through the centrifugal force field where the solids are separated from the aqueous medium (see Clause B.2). While there are a number of different types of continuous-flow centrifuges, they all function according to the same principle. All require:

- a) a drive (an electric motor or petrol engine) to rotate the centrifuge bowl at high speed;
- b) a pump to deliver the suspended solid/water mixture to the centrifuge bowl;
- c) a centrifuge bowl (separator, clarifying cylinder) which retains the dewatered suspended solid.

In centrifuges, the raw water is pumped from the top or the bottom into the centre of the bowl. Centrifugal force pushes the solids, which are denser than the water, out to the side of the bowl where cohesive and adsorptive forces hold them. The clarified water flows out of the bowl. These systems are effective for collecting suspended solids if the concentration of organic matter is not excessive (see ISO 5667-15). The smallest particle size which can be separated out depends on the geometry of the bowl, the centrifugal force (speed of rotation), and the physical characteristics of the suspended solids (size distribution, chemical composition and density) (see ISO 6107-2 [4]).

The recovery efficiency also depends on the above three characteristics as well as on the suspended solids concentration and the amount of organic matter in the sample. Retention of more than 90 % of the suspended solids in the water/solids mixture is described in Reference [12]. Also, the percentage of retained solids that were less than 0,45 µm in diameter was often more than 50 % of the solids sample.

Details on deployment, strategies for continuous-flow centrifuges and pumps can be found in References [13], [14], [18], and [19]. Operation of the centrifuge should always be in accordance with manufacturer's specifications. Safety is particularly important and is covered in Clause 11. Although dedicated to whole water samples, refer to ISO 5667-3 for container preparation (cleaning) prior to sample collection.

All centrifuge components that come into contact with the water/solids mixture should be made of stainless steel or lined with polytetrafluoroethylene (PTFE) to avoid sample contamination. This is especially critical if the clarified water discharged from the clarifier (permeate) is to be used for further chemical analysis and/or metals are to be analysed in the attained suspended solids. It is preferable that PTFE be used where inorganic analyses (e.g. metals) are to be performed while sterile stainless steel is favoured for organic analysis.

Recovering suspended solids from the bowl or tubular chamber is generally not covered in manufacturers' instructions. Depending on the type of analysis to be performed, recovered solids should be removed either by using PTFE or stainless steel spatulas (bowl-type centrifuges) or by removing the PTFE liner (tubular chambers).

The use of a centrifuge allows separate hydrological events (e.g. flood water sampling, or spatial distribution of suspended elements) to be logged as they occur. The results of analyses permit target compliance to be monitored and show the current level of pollutant load in the suspended solids. Sampling with centrifuges involves individual samples, which can be directly correlated with the discharge at the time of sampling, so that when an adequate number of samples has been taken for a year (every 2 weeks, for example) assessments of the load or other hydrological evaluations (e.g. sources of pollution) can be made.

Continuous-flow centrifuges can be used in a number of ways:

- *in situ* extraction (direct extraction from the body of water);
- stationary deployment (installation of a continuous-flow centrifuge in a water-monitoring station);
- mobile deployment (e.g. installation of a continuous-flow centrifuge in a boat or on a trailer);
- laboratory extraction (input from a reservoir/container).

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6.2.2 Advantages of centrifuging processes

The advantages of centrifuging processes are:

- a) rapid resolution of sampling during unusual events (flood, pollutant waves);
- b) sampling method can be varied depending on the suspended solids load;
- c) they allow the collection of large amounts of suspended solids within a few hours;
- d) they achieve a good separation of solids and water, i.e. separation rates of between 91 % and 98 % (Reference [40]);
- e) loads of substances which mainly bind with suspended solids can be estimated;
- f) hydrological assessments of the suspended solids load can be made by direct relating to discharge levels;
- g) the sample remains unchanged after extraction (it is immediately refrigerated or frozen);
- h) samples can be taken from a number of different sampling points within a few days, depending on their location;
- i) mobile installation on a boat permits horizontal and depth profile measurements to be made.