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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

Reference number
ISO 5667-17:2008(E)

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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*

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]

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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.

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).

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.

6.2.3 Disadvantages of centrifuging processes

The disadvantages of centrifuging processes are:

a) high cost of acquisition;

b) expense of servicing when continuously operated;

c) replacement parts are expensive;

d) mobile deployment is personnel intensive;

e) extraction of sediments with a continuous-flow centrifuge is only advantageous for local sampling points (see also 6.2.4);

f) they do not supply a comprehensive picture, but only snapshots;

g) riverbanks are inaccessible in some places where the topography is rugged;

h) water samples cannot be taken at sub-zero temperatures.

6.2.4 Operational considerations for continuous flow centrifuges

The operation and servicing of the centrifuges should be as specified in the operating instructions. Particular attention should be paid to safety (see Reference [7]).

In addition, attention should be paid to the following points.

a) The time taken for sampling varies according to the suspended solids content of the water being analysed, and depending on the suspended solids content of the water, the collection of larger sample quantities can often take several hours. As the sampling time increases, it may smooth out temporal variations in the composition of suspended solids and their associated chemistry.

b) Ensure that non-contaminating materials are used (e.g. non-PVC hose, stainless steel pump).

c) Select the dimensioning of the hose diameter and the power of the pump so that no solid particles are deposited on the hose.

d) Adjust the flow so that a maximum separation of suspended solids is achieved (also when sampling flood waters). The flow velocity of the water should be adjusted so that the retention time of the suspended particles in the bowl is between 20 s and 25 s.

e) Measure the volume of water throughput, as this value is needed for calculating the suspended solids content (dry sediment/total water throughput volume).

f) Take the sample from the cylinder and treat (using refrigeration when appropriate) immediately after sampling. The separation of the sediment from the bowl or the tubular chamber is not usually specified in manufacturers' instructions. The sediment should be removed using a PTFE or stainless steel spatula (rotor centrifuges), or by removing the PTFE lining (tubular chamber centrifuges). In either case, this should be done with particular care to avoid contaminating the sample.

g) When taking the sediments from the separator, take a representative wet subsample for determining particle sizes.

h) Clean all equipment, including the bowl, each time it is used.

The efficiency of continuous-flow centrifuges depends on the relative density of the solids, internal turbulence of the apparatus, the centrifugal force produced by the apparatus, etc. The efficiency of small, portable centrifuges is usually inadequate for silts and clays. It is just these particles with their large specific surface area and consequent high adsorption capacity that are important factors for the assessment of water quality.

Laboratory centrifuges with low throughput rates should be operated for several days to obtain samples of comparable size. The water to be centrifuged (which can be as much as several cubic metres, depending on the suspended solids content) should be brought to the laboratory. Even when it can be ensured that all sampled sediments reach the laboratory, this is only advantageous for local sampling points. The restricted capacity of the water containers means that the sediment mass that can be obtained is also limited, and as a result, it may not be possible to carry out all the chemical and physical measurements. On the other hand, sedimentation centrifuges that are installed in laboratories may be used for sampling when the external temperature is below 0 °C.

Continuous-flow centrifuges which have been modified for field use are usually heavy, and are difficult to move in the field. Some devices also require a large amount of electricity, the supply of which should be assured. Because of the high rotational frequency, the equipment is potentially very hazardous. Therefore, sampling personnel should be appropriately trained.

6.3 Settling methods

6.3.1 General

The process whereby gravity settles out suspended solids in zones where the flow velocity has been reduced into an appropriate collection system as it does in harbour basins or groyne fields is known as settling. Suspended or floating particles with a density approaching, or less than, 10^3 kg/m^3 are thus not registered by these methods. The material collected in this way is known as fresh sediment derived from suspended solids.

There are stationary settling processes which take place in a measuring station, and systems which are portable and can be deployed independently of any installation.

Because the equipment is easy to operate, it is a relatively simple matter to collect adequate quantities of sample material of fresh sediment for the subsequent analysis of a number of characteristics. The results of the analysis of the mixed sample obtained over several weeks represent the average load of a longer period. When sedimentation tanks are deployed in monitoring stations, the load for a complete year can be monitored without any gaps.

Primarily the data that are collected are used for checking targets and characterising the relevant section of water according to its classification system. In addition to statements on current pollution levels, trend assessments can be performed, even more so when sampling has continued without any interruptions over a period of years.

6.3.2 Stationary settling methods (sedimentation tanks)

6.3.2.1 General

Sedimentation basins are usually installed in water-monitoring stations. The tanks are usually made of polymethylmethacrylate (PMMA) so that the settling processes, and in particular, both the removal of the water above the sediment and the collection of the sample, can be observed. The fact that this material, particularly where the sample has organic components, can interact with the sediments and influence the results of the analysis, should be taken into account here. This is why the walls of the basin should not be wiped down after the excess water above the sediment has been drained off; otherwise the contact layer might be introduced into the sample for analysis.

Part of the flow of water entering the water-monitoring station is led through the sedimentation tank via the water intake valve which can be adjusted so that the incoming flow is reduced, resulting in a current in the tank of about 0,01 m/s and allowing part of the suspended solids load to settle out in the 1 m long flow section (see Clause B.3).

Usually damp material is collected during a sampling period of a month or so. The finest, low-density suspended solids and free-floating plankton organisms pass the tank. The efficiency of settling tanks is between 20 % and 40 %, depending on the amount of suspended material (Reference [41]). The tanks are covered with opaque plastic film in stations that are naturally lit because of the increased growth of plant organisms in such stations.

6.3.2.2 Advantages of sedimentation tanks

The advantages of sedimentation tanks are:

a) they require little servicing or maintenance, and are not at all personnel intensive in operation. Usually personnel are only deployed once a month to extract the sample material and clean the basin and its intake lines;

b) low acquisition and running costs;

c) the volume of the sample is usually sufficient for an extensive range of tests;

d) they can ensure an uninterrupted, continuous monitoring of suspended solids loads over a complete year;

e) a similar kind of settling out of the suspended solids is achieved in sedimentation tanks as there is in harbour basins and groyne fields.

6.3.2.3 Disadvantages of sedimentation tanks

The disadvantages of sedimentation tanks are:

a) only a small proportion of the suspended solids is collected (20 % to 40 %, depending on the quantities of suspended solids and particle size distribution);

b) the analysis of loads requires additional daily tests of the suspended solids concentrations and the discharge;

c) possible ageing of the sediments and changes to the content during the sampling period at room temperature;

d) not all small particles are collected.

6.3.3 Mobile settling methods (sedimentation boxes)

6.3.3.1 General

Sedimentation boxes (see Clause B.4) are placed in the water to collect sediment; they may be attached to a buoy or anchored to the bank. In principle, they can be deployed anywhere in the water; the flow velocity should not, however, be above 1 m/s. The sampling period varies between 1 week and 4 weeks depending on the amount of suspended solids. In modified construction (connection of in- and outflow hoses or pipe), sediment boxes may also be used in monitoring stations (Reference [34]).

6.3.3.2 Advantages of sedimentation boxes

The advantages of sedimentation boxes are:

a) minimal demands on personnel; the equipment is easy to operate;

b) inexpensive.

6.3.3.3 Disadvantages of sedimentation boxes

The disadvantages of sedimentation boxes are:

a) not all suspended solids are collected;

b) the sediments may age during the sampling period;

c) poor results where current velocities exceed 1 m/s;

d) small number of deployments, as the equipment wears out relatively fast;

e) possible losses caused by vandalism and floods;

f) effort of protecting the equipment from being damaged by boats or vessels;

g) limited applicability, as they cannot be used when there is drifting ice, for example.

There are other mobile settling methods to collect suspended solids. For example, the plate sediment traps described in Clause B.6, and the flask sediment traps in Clause B.7.

6.3.4 Floating collectors

6.3.4.1 General

Floating collectors (see Clause B.5) are designed for use directly in the water. The collectors are hung from a supporting buoy and placed in the water. The water flows through the inlet nozzle, which can be used to adjust the inflow, into the funnel-shaped interior, which functions as a settling basin. Through redirecting the horizontal flow into a circular flow, the retention time during which the suspended solids can settle out is increased. The suspended solids drop into the collecting flasks which function as sample receptacles. Depending on the amount of suspended solids, sampling periods may vary from 2 weeks to 4 weeks (Reference [42]).

6.3.4.2 Advantages of floating collectors

The advantages of floating collectors are:

a) minimal demands on personnel; the equipment is easy to operate;

b) relatively inexpensive;

c) the equipment can be deployed at different depths.

6.3.4.3 Disadvantages of floating collectors

The disadvantages of floating collectors are:

a) exchange of the sample receptacles often involves the use of a boat;

b) the sediment collector should be protected from river traffic;

c) the equipment cannot be deployed when there is drifting ice;

d) only a small proportion of the sediment is collected.

6.4 Filtration methods

6.4.1 General

Another method for collecting sediment samples is the filtration process. Filtration always requires a pressure difference between the supply and discharge sides of the filter system; this means that in practice either overpressure or vacuum filter containers are deployed. For water chemistry purposes, filters with 0,45 µm pores are commonly used.

The water sample, which is either one random sample, or a mixed sample made up of a number of random samples, should be filtered as rapidly as possible to minimise any change in the balance of components being analysed which are either dissolved or bound to particles (possible precipitation, etc.).

Attention should be given to ensure that:

a) the water sample is homogenised immediately before it is poured into the funnel of the filtration apparatus;

b) the membrane filters being used (cellulose acetate, for example) are cleaned (pre-rinsed) and weighed;

c) the filtration vessel is of a material that will not affect the results of the analysis (to avoid contamination, use glass or PTFE, for example);

d) the filtration process does not take too long — the volume of the water sample to be filtered should be one that permits the filtration process to be completed within a few hours, before any post-precipitation can set in;

e) when employing a pressure filtration method the gas used to produce the pressure has no effect on the results of the analysis.

If the filtrate is also to be analysed, attention should be given to ensuring that:

a) the blank values of the batch of the membrane filters being used are regularly determined, as they are subtracted from the overall results of the analysis;

b) the membrane filters being used do not have too large a diameter (up to about 100 mg for a filter diameter of 49 mm), as otherwise the blank value, relative to the analytical value, would be high.

The sediment sample is carefully rinsed with de-ionised water, dried, and weighed. Until they are analysed, the residues in the filters are stored in airtight conditions (e.g. in sealable polyethylene bags).

As taking water samples precedes the filtration process, it can be applied in a number of areas. Depth profiles can be made of the water body by taking the samples from somewhat varying depths (using the appropriate scoops). Samples can be taken from land, from boats, or from helicopters, allowing the procedure to be applied for longitudinal and cross-section. Water samples can also be taken from monitoring stations.

The results of analyses allow compliance with target values to be checked, they show the current state of the load, and can be used when drawing up load balances. As the water samples are usually taken at random, the sediment findings can be assigned to a very short time period. When tests are made frequently enough, the good temporal definition allows the pronounced variations in suspended solids contents (extreme values due to floods, etc.), which are observed during a year of investigation, to be detected.

6.4.2 Advantages of filtration processes

The advantages of filtration processes are:

a) good temporal definition (snapshots);

b) the effort required for taking the sample is relatively low;

c) good sediment/water separation;

d) the suspended solids collected comprise a specific size range: > 0,45 µm;

e) fairly inexpensive;

f) advantageous when calculating loads, because the dissolved portion, which may not be negligible in determining the load, is immediately accessible;

g) sample taking can be variable (e.g. for establishing depth and horizontal profiles).

6.4.3 Disadvantages of filtration processes

The disadvantages of filtration processes are:

a) the filters rapidly become clogged;

b) only a very small mass of sediment is collected, which can lead to relatively large analytical errors;

c) filtration can be very slow (vacuum filtration may take several days), and so the original equilibrium may shift from dissolved to particulate-bound;

d) as the system is relatively susceptible to contamination (filtering, drying), contamination originating elsewhere, although very slight, can have a strong influence on the results;

e) only limited suitability for organic contents;

f) because of the small mass of sediment, other analyses such as particle size analysis or analysis of the fine-particle fraction are not possible.

6.5 Tangential-flow filtration

6.5.1 General

This type of particle/fluid separation, also known as ultra-filtration, is generally used for the separation of the < 3 µm fraction including colloids. However, this size is dependent upon the nominal pore size of the filters used, flow rate and other factors. A sample is initially collected using other samplers, such as those from the passive category or by pumping into a storage container. The system employs a stack of membrane filters, separated by gaskets that channel the flow across the surface of the membranes. The suspended solid/water mixture is pumped (generally with a peristaltic pump) across the filters with the retained suspended solid (that of a size larger than the nominal pore size of the filters) swept tangentially across the filter stack and out into the original sampler container where it is recycled through the system again. Filtrate and suspended solid which is small enough to pass through the pores of the filters is removed from the system. This recycling process is generally continued until the original sample volume is reduced to less than 1 l (References [13], [14]).

Deployment strategies for tangential flow filtration can be found in References [13] and [14]. Operation of the unit should be according to manufacturers' specifications. There is no published literature on quality assurance of tangential-flow units.

CAUTION — Re-use of filters and attached tubing requires cleaning, with reagent or pesticide-grade solvents. Handling and disposal of such solvents requires great care and should be in accordance with national regulations. Care should be taken to collect samples in appropriately cleaned sample

containers (see ISO 5667-3) and with proper inert utensils when working with the bulk suspended solids.

6.5.2 Operational considerations for tangential flow filtration

The following factors should be considered.

a) The final suspended solid sample can contain particle sizes into the colloidal size range, depending on the filter pore size employed. This size fraction, because of its large surface area, has the highest concentration of adsorbed chemical substances. However, suspended solid separation is relatively slow (slower than continuous-flow centrifugation).

b) Filter clogging can result in a downward shift in nominal pore size, resulting in better retention of very small particles, but at reduced flow rates.

c) Frequent filter replacement is often required, depending on the manufacturer and nature of use, can involve significant cost and generally has a greater clean-up time required than for other methods.

d) This technique has not been widely used in routine field situations. Thus, there is relatively little known of the operational problems associated with tangential flow apparatus from different manufacturers.

e) This system is much smaller than continuous-flow systems and is less expensive to purchase, but can be more expensive to operate because of cost of replacement filters (Reference [14]).

6.6 Pumping requirements

Most bulk samplers require that the suspended solids/water mixture be pumped from the water column into the bulk sampler or into a storage container. There are numerous types of pump ranging from submersible to peristaltic pumps, which have been used for water quality sampling. The composition (plastics such as PTFE, metal, etc.) of pump parts that are in contact with the water, and the composition of the hosing that carries the water to the sampling apparatus can be important, depending on the type of chemical analyses required in the sampling programme. Submersible pumps should be magnetically driven so that there is no chance of leakage of lubricating and cooling oil from the submersible electric motor into the impeller housing.

Pumped samples are appropriate for all aquatic environments, providing that isokinetic sampling (3.3) is not a requirement of the sampling protocol. Very few pumps can sample isokinetically. However, in practice, this is not generally a problem, especially as:

a) in many rivers the majority of suspended solids are silt plus clay particles; and

b) the particle-size range of interest in the chemistry of suspended solids is usually the < 63 μm fraction (Reference [20]).

Pumps are difficult to use for depth-integrated sampling. Therefore, if a river transports a significant proportion of sand particles, pump samplers are likely to undersample this population (sand is generally transported near the bed). For environmental chemistry this might not be critical, because chemical enrichment of solids is mainly in the silt plus clay < 63 μm fraction.

There are a number of practical advantages to using pumps as follows.

a) Pumps are the only practical means of moving large volumes of water to the bulk sampler and can be easily deployed in any aquatic environment.

b) Because of the length of time involved, pumps average out any short-term temporal variations in solids chemistry that are commonly observed in the water column.

c) Pumps are relatively inexpensive and are easily dismantled for cleaning.

d) Fine suspended solid in water is usually transported as flocculated solids (Reference [11]), however pumps break up these flocs. While not important for solids chemistry, this is important if the sampling programme is interested in measuring the natural particle sizes that exist in the water column.

e) Particle sizes sampled can be biased due to the non-isokinetic nature of most pump samplers.

f) The chemical composition of the hose material (e.g. rubber) and pump construction can affect the suspended solid chemistry. Hosing can be obtained in stainless steel, PTFE and other non-contaminating materials.

For research purposes, the deployment of sampling pumps depends upon the questions to be resolved by the data collected by the pumping programme. However, for routine monitoring purposes the following protocol is recommended, especially when there are constraints of time and budget.

— For small rivers (< 10 m in width), one pump should be located in the middle (or in the zone of maximum velocity) of the channel, at approximately 0,5 m from the surface of the water or at mid-depth, whichever is less. The success of this simplistic technique is based on the fact that, in many instances (but not always), the fine-grained < 63 µm fraction (the chemically active fraction) is generally evenly distributed in the vertical section, although this might not be true in the cross-section (References [15], [17]), depending on the nature of inputs of solids upstream.

— For larger rivers, several pumps should be placed at equal intervals across the river and at depths as above. The pumped water should be integrated into a single sample (either in one container, or combined into a single hose feeding into the bulk sampler). For very large rivers, pumps might have to be deployed sequentially in time across the section.

— Any portable electrical generator should be placed downwind of all sampling apparatus, to avoid contamination of equipment and sample by engine exhausts.

— It is essential that hoses be cleaned after use, to avoid sample contamination. A recommended operating procedure is to pump water under field conditions through the hosing for 5 min to 10 min prior to taking the sample.

— Long hose lengths should be avoided, especially under hot field conditions. Temperature rise in the hose, especially if there is a long transit time from water column to bulk sampler, can affect chemical partitioning between suspended solid and water.

7 On site measurements

To assess how representative the sampling is, and to determine the suspended solids load, it is necessary to have information on the discharge, as well as from other sediment analyses taking place at the same time.

Generally, the suspended solids content should be calculated from the amount of sediment collected and the volume of water that has passed through the collecting equipment.

Other possibilities to assess the suspended sediment content are to determine the content of substances that can be filtered out, or the turbidity measurement method, which is both simple and fast.

Depending on the physical, chemical, biological, or toxicological characteristics of the suspended solids that are being investigated, other variables may need to be measured on site to aid interpretation of the results of the sample programme, e.g. water temperature, pH, dissolved oxygen.

8 Post collection sample handling and analysis

8.1 General

Depending on the type of analysis to be performed, collected bulk suspended solid samples should be placed within inert sampling containers. These will generally take the form of acid-washed polycarbonate or glass bottles. Container preparation (cleaning) prior to sample collection should follow the instructions provided in ISO 5667-3 (although for whole water samples, similar procedures can be followed for the containers used for bulk suspended solids).

8.2 Identification of samples

Sample containers should be labelled in a clear manner with indelible ink.

Note the date and time of sampling, the name of the person who took the sample, location, sampling instrument number (if more than one has been deployed) and any additional information required, such as weather conditions or unusual occurrences. The uses of pre-printed labels, forms, etc. can facilitate the practical attainment of these requirements.

8.3 Sampling record

A sampling record should be prepared for each sample taken to include the sampling data. The laboratory and the client will thus receive information on the type of sample, the sampling site, activities taking place, and measurements being made at the time of sampling, as well as information which is important for the analysis and later assessment purposes.

All observations should be recorded immediately on site. Also, marginal conditions, or any unusual occurrences, such as injections or unusual features of the riverbanks which are important for interpreting the results and water quality findings, should be recorded.

Further details of the sampling site, the date, the sample number, the flow, and the time taken for sampling should be noted.

To ensure correct identification of the sample, its label should match the details on the corresponding sampling record. The sampler is responsible for the sample, and for the details on the sampling record; the sampler should confirm this with his signature (Reference [39]).

8.4 Preservation

While some chemical amendments can be applied to whole water samples depending on the nature of the analysis to be performed (see ISO 5667-3), generally no preservatives should be employed with bulk suspended solid samples due to the non-fluid nature of the sample. For the characteristics that are usually investigated, sample pre-treatment and conservation in accordance with ISO 5667-15 is recommended.

Note that the storage time given in ISO 5667-3 is incorrect as the high level of biological activity results in considerable development of mould within a few days, even when cooled to < 2 °C. Therefore, it should be ensured that there is an unbroken cooling chain, and that the sample is promptly processed in the laboratory.

Refrigerated samples should not be retained for longer than 2 days.

Samples should be preserved immediately after sampling, as the first few hours after taking the sample are always the most critical for any changes that may take place.

Generally preservation is attained through keeping the samples in the dark and refrigerated (or on ice during transportation) at between 2 °C and 5 °C (ISO 5667-15). Lack of refrigeration can result in rapid chemical and biological changes due to chemical reactions and metabolism of organisms. Samples can be frozen if analysis requires such a step (e.g. particle size analysis). Where certain organic components are to be determined deep-freezing or the addition of chemicals is recommended.

It should be noted that freezing (slow or flash freezing) can change the physical (e.g. particle structure), chemical, and biological characteristics of the suspended solid through cell breakage (ISO 5667-15, Reference [20]) and is not recommended if toxicological analysis of the sample is to be conducted.

8.5 Transport of samples

Containers holding samples should be protected and sealed in such a way that samples do not deteriorate and do not lose any of their constituents during transport. Packaging material should protect the containers from possible external contaminant and breakage and should not be a source of contamination itself.

The sample containers should be made of a suitable material, which neither contaminates the sample, absorbs any of the components to be analysed, nor reacts with any of them. They should also be easy to clean, and disposable of in an environmentally friendly way.

Depending on the analysis parameters, containers of PTFE, glass or polyethylene should be used. When analysing organic contamination, use brown glass bottles with ground glass stoppers.

Full sample containers should be stored where they cannot be broken, away from heat and light, and taken to the laboratory, preferably in coolboxes or refrigerators, as soon as possible. The laboratory can then carry out any further treatment of the samples or split them into subsamples.

9 Quality assurance of field samples

9.1 General

9.1.1 Introduction

General water quality assurance guidelines should be consulted within ISO 5667-14; however, samples taken with bulk sampling apparatus should also conform to the guidance in 9.1.2 to 9.1.4.

9.1.2 Precision

One field sample should be split and analysed in duplicate for every sampling day. These samples should not be analysed consecutively in the laboratory in order to limit any bias that can be introduced by their position in the analytical run. Precision is based on the relative percent differences in analytical values between the samples. Its magnitude will depend on the precision that can be realistically achieved for a given analysis. Acceptable limits for precision are typically in the range 10 % to 50 %, depending on the analysis and the way the results are to be used.

9.1.3 Accuracy

Accuracy is derived by the analysis of a standard reference material in the laboratory and cannot be determined as part of the field programme.

9.1.4 Field method blanks

Field blanks, commonly used in conventional water quality sampling, are not feasible. It is neither practical nor realistic to carry enough "blank" water to run through a centrifuge to check for field contamination or potential contamination of metals from stainless steel (or other metallic) apparatus; hence good field practice that emphasises cleanliness of apparatus, connecting hosing, etc. is essential.

9.2 Quality assurance specific to centrifuges

This guidance is independent of the quality assurance required for laboratory analysis of samples. No standardised quality assurance programme exists for centrifuges. Evaluations of internal contamination of the sample from contact with metal or non-metallic parts, oils and greases, etc. have been carried out

(References [14], [18]). Quality assurance programmes that have been used by various practitioners include pumping suspensions of laboratory clays, for which the chemical composition is known, into the centrifuge (this includes all hosing, pumps and any other apparatus used as part of the field centrifuge set-up), followed by analysis of the collected suspended solid for comparison with the original suspended solid. This is not, however, a procedure that can be easily carried out and is usually reserved for ensuring, on an occasional basis, that there is no sample contamination caused by the apparatus itself.

Field cleaning of apparatus is essential to ensure that there is no carry-over of oils and greases or other materials that might have been sampled at other sites. Rigorous cleaning of the centrifuge bowl with acetone should be carried out at each site. Approximately 15 min of pumping a field sample through connecting hoses (but not through the centrifuge) prior to discharge into the cleaned centrifuge prevents cross-contamination from hosing used at previous sites. However, consideration should be given to using separate pump and hose apparatus when the field programme includes both very contaminated and very clean conditions. If one set of apparatus has to be used, then the cleanest site should be sampled first, followed by successively more contaminated sites.

All apparatus should be cleaned when returned to the laboratory by pumping suitable cleaning solutions through all apparatus. Solvent cleaning of the centrifuge bowl is also essential.

9.3 Suspended solids characterisation

Generally bulk suspended solids samples are reserved for chemical analysis where elements are bound strongly to the mineralogy of the particles. Some fractional loss of elements can occur due to the extremely high energy imparted onto the particles by bulk sampling methods. For example, the loosely bound exchangeable metals can be desorbed. This fraction of the elemental composition is, however, generally minimal relative to the total and so these methods are ideal for bulk chemical characterisation of suspended solids.

Determination of particle physical characteristics such as floc size, shape and porosity (Reference [21]) should not be attempted with bulk sampling methods due to particle breakage and re-amalgamation upon deposition.

NOTE Given the high energy imparted onto suspended solids by the various bulk sampling methods, they are generally not ideal for biological analysis due to the uncertainty of microbial removal and cell disruption.

10 Interpretation of data

10.1 General

The chemical nature of suspended solids is generally used for two purposes: a) to determine the presence and amount of the chemical parameters at the time of sampling; and b) to calculate the chemical loadings that are being transported over some designated period of time.

Interpretation of suspended solids data is never exact because of the impossibility of knowing the actual distribution of total suspended solids and associated chemical values through time and space (across the sampled section).

10.2 Variability in time

Variation in chemical nature within the fluvial environment is highly dependent on discharge and on upstream sources of natural or anthropogenic pollution. Because sampling programmes are usually unable to continuously monitor for temporal changes, the variability associated with time is usually not well known; therefore, analyses of chemical values from single samples should be regarded with caution.

10.3 Variability in space

Sampling in the vertical section should, if applicable, take advantage of the fact that research in many rivers has demonstrated that silt and clay-sized particles tend to be fairly evenly distributed in the vertical section (References [16], [23]). However, use of near-surface sampling should not necessarily assume that the sample is typical of the whole profile. This becomes especially important for analysis of metals on suspended solids when the sand-size fraction is being included. Sand-size material is depth dependent and requires depth-integration sampling.

Sampling in the cross-section adds considerably to the expense of the field programme. Typically, water sampling programmes collect a sample near the centre of the channel, or at the point of maximum flow in the cross-section. Rivers are not necessarily well mixed in the cross-section, especially where tributaries or point sources enter the main stream, upstream of the sampling location. Only detailed sampling can determine if one position in the cross-section is typical of the section. Additionally, sampling from near-riverbank locations should always be avoided because of “edge effects” which arise from localised interactions between the river and its banks. This can lead to high levels of contamination in the river edges when there are polluting substances on banks upstream and could lead to overestimation of chemical concentrations and/or loads of the cross-section as a whole.

10.4 Implications for data interpretation

It is essential that the determination of the presence and concentration of solids-associated chemical parameters at a point in time, especially when this is determined from one sample (the normal procedure) should take into account the variability that is associated with single samples. This variability can be quite large (up to 100 %). It is imperative that the use of such data for regulatory or other purposes should take this variability into account.

For determining solids-associated chemical loadings, the situation is quite different. The error in loadings values is mainly associated with the product of discharge and total suspended solids (TSS) concentrations (which typically vary over several orders of magnitude) and only marginally with the solids-associated chemical parameters (which usually vary by much less than an order of magnitude). Therefore, the variability in the chemical concentration is a minor factor (Reference [22]) even though the physical concentration of suspended matter can change greatly, even within a single storm event. The cumulative effect of these variabilities is that loadings values are typically within an order of magnitude of the actual (but usually unknown) value (Reference [22]). Greater accuracy is dependent upon a high-frequency sampling protocol for discharge and TSS, but not for suspended solid chemistry. Generally, it is preferable to ensure that the range of solids-associated chemical parameters is known, especially if seasonal effects or runoff events affect solids chemistry. Examples include sources of suspended solid that are seasonally dependent such as during spring runoff, or solids runoff from storm events in urban environments.

10.5 Field methods for reducing uncertainty

Because of the cost of operating field programmes, automatic samplers which composite small samples in real time can reduce uncertainty in total suspended solids concentrations. Composited samples might not, however, be suitable for certain types of chemical evaluation due to restricted sample volume and potential changes in pH, temperature, and microbial degradation of organic contaminants during the period of storage in the sampler.

There are no other means of reducing uncertainty, apart from more intensive sampling in time and space.

11 Safety precautions

All field programmes involving sampling in lakes and rivers require extreme care. The use of a manual for standard field operating procedures that prescribes all safety elements is recommended. Consideration should be given to carrying out a “hazard assessment” to identify all potential hazards and to develop an appropriate response strategy, as is required in some countries.

When sampling from flowing waters, there is usually no equipment available that meets safety-at-work and accident prevention requirements. Usually, therefore, one has to take samples from the water’s banks, from boats or bridges.

Be aware of the risk of accidents when sampling and observe the current accident prevention rules.

Observe operating instructions and all safety-at-work rules.

If there is any risk of drowning because of high water levels and/or fast currents, always wear a life jacket and safety gear.

Centrifuges and electric pumps are run with portable generators and cables — often in wet weather. When using electrical equipment under such conditions, particular care is required to ensure that there is no risk of electric shock. The electrical equipment should be fitted with a residual current protective device and be earthed with an earthing spike. Always operate the centrifuge, the generator, and the pump as described in manufacturers’ operating instructions. Always check the equipment for damage before using it. Replace defective or damaged parts immediately.

Some passive samplers are very heavy, and should only be lifted with mechanical hoists.

Depending on their origin, suspended solids samples can contain pathogens. Infection with bacteria or parasites may result from inadvertent ingestion with food, through hand-mouth contact (smoking), or through unprotected skin or mucous membranes. These hazards can be reduced to a minimum by complete compliance with the rules for personal hygiene, wearing gloves and protective clothing (such as disposable overalls, safety goggles), and not smoking. Sampling personnel should also be offered the opportunity of having themselves immunised, e.g. against hepatitis.

Handling of solids samples can involve use of organic solvents such as acetone for cleaning sampling equipment, sampling bottles, etc. Solvents and their vapours are toxic and it is essential they are handled and disposed of with extreme care and in accordance with any national regulations. See ISO 5667-3 for precautions to be taken in the preparation and use of appropriate containers.

Annex A
(informative)

Information on suspended solids and their sampling

A.1 Relationship of certain analytes to suspended solids

There is abundant literature which demonstrates the strong affiliation of many chemical constituents with the particulate phase during transport in aquatic systems. These include phosphorus, metals, chlorinated organic compounds [PCB, dioxins, organochlorine pesticides (e.g. DDT and its metabolites, pentachlorophenol)], polycyclic aromatic hydrocarbons (PAHs), etc. In soil science, the preferential association of nutrients, metals and other agrochemicals leads to the concept of enrichment ratio, which describes the increase in the concentration of chemicals associated with transported suspended solids in comparison with natural concentrations in uneroded soils.

The association of phosphorus and metals with suspended solids is variable and depends on many site-specific factors such as type of suspended solid, pH, and redox potential. Suspended solids have been demonstrated to be the primary mode of transport for these chemical parameters (Reference [24]). Studies in North America and Europe show that as much as 90 % of total phosphorus transport in rivers can be in association with suspended solids. The association of phosphorus and metals with suspended solids can vary widely and is dependent on a variety of physicochemical factors, including: grain size, surface area, relative density, surface charge, adsorption, precipitation, co-precipitation, organometallic bonding, cation exchange, incorporation of crystalline minerals, interstitial water, and the presence of carbonates, clay minerals, hydrous iron and manganese, oxides, sulfides and silicates (Reference [25]).

The association of hydrophobic xenobiotic organic chemicals with suspended solids depends firstly on the octanol/water partition coefficient, K_{ow}, which is well known for all organic chemicals. Further, it is believed that there is a particular relationship between these chemicals and the organic carbon fraction of the suspended solids (Reference [26]). The partitioning relationship with organic carbon is characterised as K_{ow}.

A.2 Relationship of suspended solids concentration to analyte concentration

The amount (concentration) of suspended solids in the water column plays an important role in the amount of the chemical which is associated with the suspended solid phase. For organic compounds, the relationship with the organic carbon fraction is believed to be the most significant.

The significance of the linkage between analytes and suspended solids is as follows.

a) A significant but variable proportion of the total load is carried by the suspended solids phase.

b) Sampling for these chemicals using whole water samples can underestimate the concentration of these chemicals in the water column because of the inability to fully extract that portion of the chemistry which is associated with the particulate phase.

c) For organic chemicals that are associated with particulate material, analysis giving a “not detectable” result, which is common in analysis of water samples, can misrepresent the actual presence/absence of the chemical and lead to serious errors in interpreting the laboratory results.

d) Measurement of toxicity of the water column can be seriously compromised, insofar as the measurement technique is usually insensitive to the particulate phase in a water sample (References [15], [23]).

A.3 Problems inherent in conventional analyses of suspended solids analyte concentration

Conventionally, the concentration of an analyte that is associated with the particulate phase is determined by the method of differences (M of D), i.e. the determination of the total analyte in a whole water sample, minus the determination of the analyte in the filtered fraction. Irrespective of problems of filtration (see below), it is known that total analysis of raw water can underrepresent the particulate fraction, especially for organic analytes and metals, due to incomplete extraction from the particulate phase. The M of D can be very inconsistent and highly dependent on the TSS concentration. At very low TSS concentrations, negative values can occur because of the minimal difference in the compositions of dissolved and total water samples and because of higher analytical error at lower concentrations. At high TSS concentrations, contaminant concentrations analysed directly from the suspended solid can often be 50 % greater than that calculated by M of D (Reference [23]). As such, if a contaminant is transported primarily in the particulate phase, a more accurate estimate of contaminant concentrations can be derived from a direct analysis of the suspended solid.

Notwithstanding the rationale for direct analysis of the solids fraction, filtration is still widely used to infer solids chemistry through the M of D procedure noted above. For this purpose, filtration has significant methodological problems. Apart from well-documented difficulties of quality control, especially for filtration techniques used in the field, filtration is highly susceptible to inherent and often uncontrollable methodological problems (References [27], [28]). Detailed studies of filtration in water quality measurement have demonstrated (References [29], [30], [31], [32]) that analytical values of filtered samples can be an artefact of the filtration methodology employed. ISO 5667-3 provides a discussion on the contamination and/or adsorption effects of certain filters. Hence, the analytical accuracy of filtered samples cannot be assured and, in some cases, cannot be determined. Clearly this is most undesirable for important analytes that are associated with the solids phase.

Annex B
(informative)

Description of sampling devices

B.1 General

The following is a description of a selection of devices used for taking suspended solids samples. It is not possible here to present a comprehensive description and explanation of all the systems that are available and in use.

B.2 Continuous-flow centrifuge types

B.2.1 General

Three types of centrifugal samplers are used for the bulk collection of suspended solids for chemical investigations. These samplers follow the same principles related to centrifugal separation of solids from water and mainly differ in their bowl (the collection unit) configuration. However, there are significant differences in collection efficiency and field performance associated with bowl configuration. The three types of bowls are multi-chamber, multi-disc, and single-chamber tubular bowls (Reference [13]).

B.2.2 Multi-chamber bowl

This is a four-chamber bowl system where water is pumped via a submersible pump into the top of the bowl unit (a rate of 6 l/min is generally used for highly inorganic riverine sampling) where it is evenly distributed by a vane insert. The bowl spins at approximately 10 000 r/min and allows gross size separation with the largest or densest particles collected in the inner bowl(s) and the smaller or lighter particles collected in the outer bowl(s). The clarified water (effluent) is then discharged from the top of the bowl (Reference [14]).

B.2.3 Multi-disc bowl

The centrifuge bowl, operating at a speed of approximately 9 000 r/min, is configured with approximately 50 discs stacked on a hollow centre distributor (spindle) with spacing < 1 mm between the discs. Water is pumped into the top of the distributor (a rate of 4 l/min is generally used for highly inorganic riverine sampling) where the water is accelerated; the water then passes out the bottom of the distributor and is forced upwards between the discs. Particle settling occurs between the discs, thereby allowing high clarifying efficiency, as the individual particles have less distance to settle out of suspension than for chambered bowls. The solids migrate outwards on the discs under centrifugal force and collect on the wall of the centrifuge bowl. The majority of suspended solid is thus pushed out to the sides of the bowl by centrifugal force. The clarified water is then discharged through the outlets on the side of the device and into the bowl hood where it is ejected from the machine (References [12], [14], [18]).

B.2.4 Single tubular chamber bowl

See Figure B.1.

This bowl is essentially a long tube (sizes can vary) which is spun around its long axis at approximately 16 000 r/min for a 105 mm × 711 mm bowl (the rate will vary for different bowl sizes). Water is fed in at the base of the bowl generally at a rate of 2 l/min. Experience has demonstrated that a flow rate of 2 l/min gives a removal efficiency of greater than 95 % for predominantly inorganic solids. At higher flow rates, it has been found that the removal efficiency can decrease significantly. It is therefore recommended that an appropriate flow rate be determined by undertaking preliminary removal tests. The denser solids are collected on the outer