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Water quality — Sampling —

Part 17: Guidance on sampling of suspended sediments

Qualité de l'eau — Échantillonnage —

Partie 17: Lignes directrices pour l'échantillonnage des sédiments 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 3.

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 part of ISO 5667 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 5667-17 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 6, *Sampling (general methods)*.

ISO 5667 consists of the following parts, under the general title *Water quality — Sampling*:

— *Part 1: Guidance on the design of sampling programmes*

— *Part 2: Guidance on sampling techniques*

— *Part 3: Guidance on the preservation and handling of samples*

— *Part 4: Guidance on sampling from lakes, natural and man-made*

— *Part 5: Guidance on sampling of drinking water and water used for food and beverage processing*

— *Part 6: Guidance on sampling of rivers and streams*

— *Part 7: Guidance on sampling of water and steam in boiler plants*

— *Part 8: Guidance on the sampling of wet deposition*

— *Part 9: Guidance on sampling from marine waters*

— *Part 10: Guidance on sampling of waste waters*

— *Part 11: Guidance on sampling of groundwaters*

— *Part 12: Guidance on sampling of bottom sediments*

— *Part 13: Guidance on sampling of sludges from sewage and water-treatment works*

— *Part 14: Guidance on quality assurance of environmental water sampling and handling*

— *Part 15: Guidance on preservation and handling of sludge and sediment samples*

— *Part 16: Guidance on biotesting of samples*

— *Part 17: Guidance on sampling of suspended sediments*

— *Part 18: Guidance on sampling of groundwater at contaminated sites*

Annex A of this part of ISO 5667 is for information only.

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Introduction

This part of ISO 5667 reflects the important role of suspended solids in flowing water, especially of the silt + clay ($< 63\ \mu m$) component and associated carbon, as a transport medium for nutrients (especially phosphorus), trace metals, and certain classes of organic compounds (see 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. While standard methods exist for sampling of suspended mineral sediment for sedimentological purposes these are often not appropriate for the chemical analysis of suspended solids. Because of the lack of standards for sampling of suspended solids for water quality purposes and the improbability of achieving complete standardization 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 sediment 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 may require gram-size samples. Examples of programme objectives that require bulk collection of suspended solids include:

a) ambient monitoring for water quality assessment, control or regulation;

b) in-river monitoring of effluents for regulatory or control purposes, especially for chemical and toxicological properties;

c) 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;

d) recovery of suspended solids for purposes of physical analysis, including particle size, organic content including particulate organic carbon, sediment geochemistry, inorganic and organic chemistry of suspended solids, and toxicity of suspended solids.

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Water quality — Sampling —

Part 17: Guidance on sampling of suspended sediments

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 may be applied to freshwater lakes, reservoirs and impoundments, however field sampling programmes may differ and are not necessarily covered here.

2 Terms and definitions

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

2.1
suspended solids
solids removed by filtration or centrifuging under specified conditions

[ISO 6107-2]

2.2
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]

3 Sampling equipment

3.1 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 may require deployment from boats, bridges or by wading.

3.2 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 [9], [10], [11].

The majority of standard samplers described by the National Handbook of Recommended Methods for Water-Data Acquisition [11] 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 [16].

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3.3 Bag sampler

The large-bag passive sampler (6,5 l) described in [12] 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 3.4.

3.4 Bulk samplers

3.4.1 General

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 solids-water 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.

3.4.2 Pumping requirements

3.4.2.1 General

Most bulk samplers require that the sediment-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, metal, polytetrafluoroethylene (PTFE), 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 (2.2) 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 + clay particles; and

b) the particle-size range of interest in the chemistry of suspended solids is usually the $< 63\ \mu m$ fraction [24].

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

3.4.2.2 Operational considerations for pumping to bulk samplers

There are a number of practical advantages to using pumps.

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, they 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 sediment in water is usually transported as flocculated solids [13], 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.

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e) Particle sizes sampled may 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 sediment chemistry. Hosing can be obtained in stainless steel, PTFE and other non-contaminating materials.

3.4.3 Clarifiers/centrifuges

3.4.3.1 General

Centrifugal samplers can be referred to both as clarifiers and as centrifuges These types of centrifuge operate in continuous-flow mode whereby the water medium is pumped continuously through the centrifugal force field where the solids are separated from the aqueous medium. While there are a number of different types of continuous-flow centrifuge (see clause A.4), they all function under the same principles. All require:

a) power (electrical or gas motor) in order to rotate the centrifuge bowl at a high speed;

b) a pump to deliver the sediment/water mixture to the centrifuge bowl;

c) a centrifuge bowl which retains the dewatered sediment.

All centrifuge components that come in contact with the water/solids should be of stainless steel construction or PTFE-coated to avoid sample contamination. This is especially critical if the water discharge from the clarifier (effluent) is to be used for water quality analysis. In centrifuges, the raw water is pumped into the centre of the bowl from the top or bottom. The denser sediment (relative to water) is pushed out to the side of the bowl by centrifugal force and the clear water (effluent) passes out of the bowl. These systems are effective in dewatering suspended solids provided that the organic matter concentration is not excessive [14]. The smallest size of sediment retained by the centrifuges is dependent on bowl geometry, bowl rotation speed, and the physical characteristics of the suspended particles (size distribution, composition and density) [15].

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 solids-water mixture is described by [14]. Also, the percentage of retained solids that was less than 0,45 μm in diameter was often more than 50 % of the solids sample.

3.4.3.2 Operational considerations for continuous-flow centrifuges

a) Bulk samples provide sufficient quantities for sediment quality analysis. The alternative, the analysis of trace elements and organic micropollutants on filtering media involves use of microsamples which are very easily contaminated in the field and laboratory. Although the ability to carry out quantitative analysis of organic micropollutants on microsamples has made great progress, the amount of sample required for certain types of analysis is generally larger than can be obtained by filtration.

b) Bulk samples are less prone to field and handling contamination than are microsamples, except where microsamples are collected in prepackaged and sterile cartridges or capsules.

c) The ability to carry out replicate analyses or several different types of analyses on the same sample are limited or impossible with conventional methods (e.g. particulate-P, particle size and organic carbon, or an organic micropollutant analyte and toxicity). Multiple analysis is possible only when there is a bulk sample from which to draw subsamples for the different analyses. [Reference to ISO/TC 190 International Standards on physical methods may give some useful guidance.]

d) Sampling for bulk collection typically takes from 30 min to several hours, depending on the concentration of suspended solids in the sampled medium. This can smooth out temporal variations in suspended solids concentrations and their associated chemistry.

e) The collection efficiency of continuous-flow centrifuges depends on the specific gravity of the solids, internal turbulence of the apparatus, gravitational forces produced by the apparatus, etc. Small “portable” tubular clarifiers tend to be very poor at recovering silts and clays, which are the most important constituent for water quality purposes.

f) There is no source of commercially-produced field-useable continuous-flow centrifuges. However, commercial centrifuges have been successfully adapted for field use. Continuous-flow centrifuges are heavy, are slow to de-

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ploy in the field, and some units have large power requirements. These units use high rotational speeds and can be dangerous if used incorrectly.

3.4.4 Tangential-flow filtration

3.4.4.1 General

This type of particle/fluid separation, also known as ultra-filtration, is generally used for the separation of the $< 3\ \mu m$ fraction including colloids. However, this size is dependent upon the nominal pore size of the filters used, flowrate 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 sediment/water mixture is pumped (generally with a peristaltic pump) across the filters with the retained sediment (that 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 sediment 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 one litre [15], [16].

3.4.4.2 Operational considerations for tangential flow filtration

The following factors should be considered.

a) The final sediment 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, sediment 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 flowrates.

c) Frequent filter replacement is often required, depending on the manufacturer and nature of use, and may 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 may be more expensive to operate because of cost of replacement filters [16].

4 Sampling procedure

4.1 General

No definitive guidance can be given on the methods to be used for sampling of suspended solids for chemical determinations because of the lack of standard protocols in this field. It is therefore important to maintain consistency of technique in order to retain the value of long-term monitoring. Unlike the non-cohesive sediment load (sand-sized material), there is no unifying theory on cohesive solids transport that can be used as the basis for developing a standard methodology.

The following factors [17] are essential to consider in deciding on the sampling regime:

a) horizontal variations in suspended solids and associated contaminants;

b) vertical variations in suspended solids and associated contaminants;

c) variations in suspended solids and associated contaminants in space and time during periods of constant discharge (base flow) and during periods when discharge changes quickly (storm flow).

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d) variations in suspended solids and associated contaminants induced by differences in sampling strategy or methodology; and

e) the volume of sample required to minimize the error-producing effects of inhomogeneities in the aquatic system, and to meet analytical requirements.

All of these factors can be accommodated by using bulk-collection techniques (see 3.4) provided that the river and solids chemical regimes are not dominated by sand-sized solids transport, for the reasons given in annex A.

4.2 Pumps

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 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 may not be true in the cross-section [18], [19]), 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 may 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 15 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 sediment and water.

4.3 Centrifuges

4.3.1 General

Details on deployment strategies for continuous-flow centrifuges and pumps can be found in [15], [16], [20] and [21]. Operation of the centrifuge should always be in accordance with manufacturer's specifications. Safety is particularly important and is covered in clause 6. Recovering sediment from the bowl or tubular chamber is not covered in manufacturer's instructions. Sediment should be removed either by PTFE or stainless steel spatulas (bowl-type centrifuges) or by removing a PTFE liner (tubular chambers). In all cases, great care is essential not to contaminate the sample.

4.3.2 Quality assurance specific to centrifuges

This guidance is independent of the quality assurance required for laboratory analysis of samples. No standardized 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 [16], [20]. 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 sediment for comparison with the original sediment. 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.

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Field cleaning of apparatus is essential to ensure that there is no carry-over of oils and greases or other materials that may 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.

4.4 Tangential-flow filtration units

Deployment strategies for tangential flow filtration can be found in [15] and [16]. Operation of the unit should be according to manufacturer's specifications. There is no published literature on quality assurance of tangential-flow units. However, re-use of filters and attached tubing requires cleaning, probably with reagent or pesticide-grade solvents. Handling and disposal of such solvents requires great care and should be in accordance with national regulations.

4.5 Quality assurance of field samples

Samples taken with bulk sampling apparatus should conform to the following guidance:

— **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 may be introduced by their position in the analytical run. Precision is based on the relative percent differences in analytical values between the samples with an acceptable limit of no more than 10 %.

— **Accuracy:** Accuracy is derived by the analysis of a standard reference material (SRM) in the laboratory and cannot be determined as part of the field programme.

— **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 emphasizes cleanliness of apparatus, connecting hosing, etc. is essential.

5 Interpretation of data

5.1 General

The chemical nature of suspended solids is generally used for two purposes — one is to determine presence and amount of the chemical parameters at the time of sampling; the second is to calculate 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).

5.2 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.

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5.3 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 [18], [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-integrating 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-river bank locations should always be avoided because of “edge effects” which arise from localized 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 over-estimation of chemical concentrations and/or loads of the cross-section as a whole.

5.4 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 (Q) and solids concentrations (TSS) [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 [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 [22]. Greater accuracy is dependent upon a high-frequency sampling protocol for discharge and TSS, but not for sediment 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 sediment that are seasonally dependent such as during spring runoff, or solids runoff from storm events in urban environments.

5.5 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 may 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.

6 Safety precautions

All field programmes involving sampling in lakes and rivers require extreme care. In particular, the danger of drowning is always present. The use of a standard field operating procedures manual 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.

Field apparatus involves specific types of safety considerations. Some passive samplers can be very heavy and should not be lifted without mechanical winches. Wading samplers place the operator at risk of drowning; personnel should always wear personal flotation devices. Centrifugal clarifiers and electrical pumps involve the use of electrical cables and portable electrical generators, often under wet conditions. Use of electrical equipment outdoors requires

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special care to avoid electrocution. It is imperative that high speed centrifuges are operated at all times according to the manufacturer's instructions or they may fail and cause extensive injury or death to personnel.

Handling of solids samples may 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.

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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, etc.)], 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 sediment 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 sediment, pH, redox potential, etc. Suspended solids have been demonstrated to be the primary mode of transport for these chemical parameters [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. Phosphorus and metal association with suspended solids can vary widely and is dependent on a variety of physicochemical factors, including: grain size, surface area, specific gravity, surface charge, adsorption, precipitation, coprecipitation, organometallic bonding, cation exchange, incorporation of crystalline minerals, interstitial water, and the presence of carbonates, clay minerals, hydrous Fe and Mn, oxides, sulfides and silicates [25].

The association of hydrophobic xenobiotic organic chemicals with suspended solids depends firstly on the octanol-water partitioning 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 [26]. The partitioning relationship with organic carbon is characterized as K_{oc}.

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 the following:

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 may under-estimate 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 result of ND (not detectable), which is common in analysis of water samples, may 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 may be seriously compromised, insofar as the measurement technique is usually insensitive to the particulate phase in a water sample [17], [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) — that is, the determination of the total analyte in a whole water sample, less 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 may under-represent 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

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suspended solids (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 SS concentrations, contaminant concentrations analysed directly from the sediment can often be 50 % greater than that calculated by M of D [23]. As such, if a contaminant is transported primarily in the particulate phase, a more accurate estimate of contaminant concentrations may be derived from a direct analysis of the sediment.

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 problems of quality control, especially for filtration techniques used in the field, filtration is highly susceptible to inherent and often uncontrollable methodological problems [27], [28]. In detailed studies of filtration in water quality measurement, it has been determined [29], [30], [31], [32] that analytical values of filtered samples may be an artifact of the filtration methodology employed. 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.

A.4 Centrifuge types

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 (1) multi-chamber, (2) multi-disc, and (3) single-chamber tubular bowls [15].

a) **Multi-chamber:** 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 out the top of the bowl [16].

b) **Multi-disc:** 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 less than a millimetre spacing 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 sediment 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 [14], [16], [20].

c) **Single tubular chamber:** 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 10,5 × 71,1 cm 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 flowrate of 2 l/min gives a removal efficiency of greater than 95 % for predominantly inorganic solids. At higher flowrates it has been found that the removal efficiency can decrease significantly. It is therefore recommended that an appropriate flowrate be determined by undertaking preliminary removal tests. The denser solids are collected on the outer surface of the stainless steel bowl and the clarified water forced out at the top of the bowl. This centrifuge bowl can be easily coated with PTFE and or be used with a PTFE lining to minimize contamination and facilitate easy cleaning. Smaller units having narrow-diameter bowls suffer from a lack of sufficient gravitational forces to efficiently recover silt-clay sizes [15].

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