

# The Potential for Error in Sampling

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*Editor's note: The measurement of water quality parameters in environmental laboratories follows standard quality control protocols using methodologies approved by the U.S. Environmental Protection Agency. However, little attention has been given to quality assurance and quality control in activities outside the laboratory. This article describes some of those issues for sampling suspended sediment loads in streams and rivers.*

Recommended standard methods of measuring water quality parameters emphasize the importance of accurate laboratory techniques but overlook the importance of appropriate sampling design and protocols for sample collection. Quality assurance also should be applied to handling and storage of samples and computational procedures. All sources of error in a measurement program should be evaluated, and quality assurance efforts should be prioritized based on the magnitude and tractability of each error.

More emphasis needs to be placed on adequately representing the temporal and spatial variability of sediment in transport. The concentration and discharge of fluvial suspended sediments are very difficult to determine accurately due to their natural variability in time and space.

The difficulties begin with defining what is meant by *suspended*. According to one definition, "the particles lifted up by eddies in the main flow and moving long distances downstream before settling to the bed constitute the suspended load. The particles, usually much larger, that are rolled, dragged, skipping, or saltating constitute the bed load." The meaning of "long distances" is subject to interpretation. The American Geologic Institute's *Dictionary of Geological Terms* defines *suspended sediment* as "sediment which remains in suspension in water for a considerable period of time without contact with the bottom." Here, the vagueness is simply shifted from the spatial to the tem-

poral domain. As a practical matter, the distinction between modes of transport is defined by the mode of sampling. Hence, suspended load is defined as the material collected by standard hand-held or cable-and-reel samplers that obtain samples from a column of water.

## Limitations of Samplers

Suspended-sediment samplers cannot entirely exclude particles that might have been skipping along the bed. The degree of "contamination" by such particles is difficult to control because of changing flow and channel conditions, as well as human error committed by the individual operating the sampler. Furthermore, natural streams continually exchange material between bed load and suspended load.

Suspended-sediment samplers also cannot exclude organic material. As a result, samples may contain organic debris up to the size of the orifice of the sampling container. This material is normally included as part of the suspended-sediment load. To characterize such data properly, the size of the sampler orifice should be reported, as well as any organic material manually removed from samples before weighing. If the organic fraction is to be accurately quantified or entirely excluded, it should be removed by oxidation with hydrogen peroxide before the final weight determination.

The concentration of suspended silt and clay generally is evenly distributed throughout a stream cross-section, unless the section is near a tributary and mixing is incomplete. The distribution of sand can be quite uneven, especially in large rivers. A single-point sample or a single depth-integrated vertical sample may not be representative of the cross-sectional mean. In some streams, a correction coefficient may be applied to concentration at a single vertical. A single coefficient cannot be expected to produce consistently accurate results. In describing approved methods for fluvial

sediment data collection, T.K. Edwards and G.D. Glysson (1998, *Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 3, Chapter C2*) present guidelines for determining the number of verticals needed to achieve a given relative standard error, based on the percentage of sand in the sample and an index of variability computed from stream velocity and depth.

For multiple-vertical sampling, samples are best obtained at equally spaced verticals across the stream using an equal transit rate. The transit rate of the sampler must be kept constant up and down through each vertical and identical at all verticals. This permits verticals to be composited and ensures that the total sample volume is weighted by discharge; sediment discharge then can be obtained as the product of water discharge and the discharge-weighted concentration.

Depth-integrating samplers are designed so that water enters the sample bottle at the same velocity as the surrounding stream velocity. If the transit rate is too rapid, or if the sampler is permitted to swing downstream or from side to side, sampling will not be isokinetic and concentrations will not be discharge-weighted. In addition, bottles filled to less than 4 or 5 cm from the top result in too high a concentration. Another potential source of error in sand-bed streams is sample enrichment by striking the sampler nozzle into a dune.

Pumping samplers, which are useful for automating sample collection, only collect samples at a fixed point in the stream and are, therefore, most effective in streams carrying predominantly fine sediments. When sediments are not well mixed in the cross-section, pumped samples may be a poor representation of the mean concentration. In some cases, the fixed-point concentrations can be corrected using regression relations with simultaneous depth-integrated samples from mul-

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multiple verticals. The introduced error can be estimated from the regression statistics.

The efficiency of a pumping sampler is affected by the orientation of the intake nozzle. The nozzle normally should not be pointed upstream, because it can become clogged with sediment or debris. Pointing the intake downstream, however, can lead to serious undersampling of medium and coarse sands when flow velocities are high, particularly if the pump is located high above the intake nozzle.

Another potential problem with pumping samplers is contamination of samples with sediment that remains in the intake tubing from previous samples or that collects in the nozzle between samples and is not completely purged before the next sample is collected. Samples should be capped tightly and stored upright to prevent spillage. The water level in the bottle should be marked when the sample is removed from the field to ensure that no spillage or evaporation has occurred. If samples must be stored for more than a few days, they should be acidified and stored in the dark to prevent algae growth.

#### **Fallible Filters**

In the laboratory, significant losses of fine sediment can occur through the use of a standard filter size. At one location in northwestern California, concentrations up to 30 mg/L or higher in the zero to 1- $\mu$ m size range have been found. But 1- $\mu$ m filters often are used in concentration

analyses. From catchments in New South Wales, one researcher has reported significant proportions (commonly 12% or more) of the mass of suspended sediment passed through a 0.45- $\mu$ m filter.

Lab errors also can arise from incomplete washing, sieving, or drying of samples, spillage, weighing errors, and recording errors. These types of errors usually can be limited through proper training of lab personnel, regular calibration of balances and oven thermometers, and checking results on a portion of all analyses using reference samples.

Detailed quality-assurance guidelines are given by J.C. Knott, C.J. Sholar, and W.J. Matthes (1992, *USGS Open-File Report, 92-33*) and W.J. Matthes, C.J. Sholar, and J.R. George (1991, *USGS Open-File Report, 91-467*).

In 1996, the U.S. Geological Survey initiated a sediment laboratory quality-assurance project to identify and eliminate systematic bias and to quantify the precision of sediment analyses done by and for the federal agency. The project has an Internet link on the Branch of Quality Systems Web site at <http://bqs.usgs.gov>. The project prepares and distributes standard reference samples for measuring physical sediment properties. Results from five studies in the first 3 years of the project vary widely among the 12 participating labs, with median percent errors in suspended sediment concentration varying from zero to 18%.

However, five of the labs had median errors of 2% or less.

#### **Greatest Source of Error**

Estimating suspended sediment loads combines all the above errors with errors in water discharge and errors in interpolating or extrapolating sediment concentrations over time. Errors in water discharge can occur in many ways and easily can exceed 10%, but the greatest source of error in estimating suspended-sediment loads in streams is infrequent sampling.

Suspended-sediment loads often are estimated on the basis of a small number of very accurately determined concentration measurements, but the estimates are poor because the processes associated with sediment transport operate at frequencies that are too high for ordinary water sampling programs to characterize. Load estimates from infrequently collected sample data are highly error-prone, often based on poor relationships between concentration and water discharge, or applied beyond the range of the sample data. Recent advances in statistical methods have been developed for load estimation. Load estimates also can be improved by augmenting concentration data with continuous turbidity data.

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