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U.S. Geological Survey Quality-Assurance Project for Sediment Analysis

Introduction

Sediment is derived primarily from natural weathering of rock and is an assemblage of individual mineral grains that are then deposited by some physical agent, such as water, wind, ice, or gravity (Fetter, 1988). The U.S. Geological Survey (USGS) samples sediments and collects data on the amount of sediment in selected waterways. The most pressing sediment-related problems are associated with environmental questions, such as the transport and fate of attached pollutants, effects of sediment on aquatic biota and their habitats, and effects on sediment transport from land-use changes. Current (2000) sediment issues require that sediment studies address multiple objectives in water-resources management (Koltun and others, 1997).

To support sediment research, the USGS operates laboratories for the analysis of the physical characteristics of sediment. Sediment laboratories producing data for the USGS have two principal functions: (1) the determination of suspended-sediment concentration in samples and (2) the determination of sand/fine separations. The reliability of these determinations and the usefulness of the data are dependent on the accuracy and reliability of the laboratory analyses (Guy, 1969).

Quality Assurance—A Top Priority

In 1992, the USGS, Office of Surface Water, reviewed each of its 19 sediment laboratories to observe the quality of laboratory operations and determined the need for more rigorous quality assurance of laboratory processes and results. The review revealed that there were occasional departures from documented procedures, a lack of state-of-the-art equipment, few quantitative laboratory quality-assurance plans, and a need to document quality-control practices. To ensure that the physical sediment data produced or used by the USGS are of a known quality and are sufficient to provide long-term comparability and consistency, the USGS began the Sediment Laboratory Quality-Assurance (SLQA) project in August 1996 (U.S. Geological Survey Office of Surface Water Technical Memorandum No. 98.05, 1998). This project was developed to (1) evaluate the precision and bias of suspended-sediment concentration and sand/fine separations, and (2) evaluate sediment analyses on an ongoing basis in order to monitor intralaboratory (analyses comparison within each laboratory) and interlaboratory (analyses comparison between laboratories) precision and accuracy (U.S. Geological Survey Office of Surface Water Technical Memorandum No. 98.05, 1998).

Description of the Quality-Assurance Project

Reference Material Preparation

Two materials were used to represent naturally occurring sediments in streams and rivers. To represent the fine material portion of the sediment, AC Spark Plug[1] dust (a natural fine-size material collected in Arizona) was purchased from General Motors[1] AC Spark Plug Division (http://www.bardyne.com/glossary.htm). This AC Spark Plug dust contains 40 percent clay-size material and has a specific gravity of about 2.7. The AC Spark Plug dust was tested for dissolution in water, dried at 105 degrees Celsius (°C) for a minimum of 24 hours, and sieved to less than 62 micrometers (µm). It was determined that about 1 percent of the spark plug dust was less than 0.45 µm. When the AC Spark Plug dust was added to deionized water, the size fraction that was less than 0.45 µm was considered dissolved. The sand used in the project was collected in South Dakota. It was also tested for dissolution in water, dried at 105°C for a minimum of 24 hours, and then sieved to between 63 and 125 µm. The recovery of the sand-size material ranged from 99.6 to 100.3 percent, indicating that the sand-size material did not dissolve. The recovery of sand-size material that measured greater than 100 percent was attributed to a slight error in measurement (Gordon and others, 1999).

Standard reference sediment samples were prepared by adding both fine-size and sand-size material to deionized water. Samples were created in three broad sample classes to simulate the wide range of sediment concentration and sand- to fine-size material ratios found in the environment. The mass of the fine-size material added to each sample was determined gravimetrically in milligrams. The sand was then added to the fine-size material in an amount equal to between 5 and 32 percent of the fine-size material mass (table 1) (Gordon and others, 1999). Figures 1 and 2 show the samples as they are prepared for use in the SLQA project.

[1]The use of brand, trade, or firm names in this report is for identification purposes only and does not constitute endorsement by the U.S. Geological Survey.

Figure 1. Deionized water is added to the dry sediment sample. A meniscus line is then drawn, showing the top of the water level.

Table 1. Sample fine-size material mass and percent sand-size material in each Sediment Laboratory Quality-Assurance study between August 1996 and July 1999.

Study number	Sample class	Range of fine-size material mass in each size class (milligrams)	Percentage of sand-size material added to the mass of fine-size material
96-1	1	50 to 100	10, 15, 20
	2	101 to 300	10, 15, 20
	3	2,200 to 3,200	10, 15, 20
96-2	1	50 to 100	10, 15, 20
	2	101 to 300	10, 15, 20
	3	2,200 to 3,200	10, 15, 20
97-1	1	50 to 100	10, 18, 28
	2	101 to 300	09, 15, 23
	3	2,200 to 3,200	12, 17, 24
97-2	1	50 to 100	11, 18, 28
	2	101 to 300	09, 15, 23
	3	2,200 to 3,200	17, 24, 12
98-1	1	50 to 100	11, 18, 28
	2	101 to 300	09, 15, 23
	3	2,200 to 3,200	17, 24, 12
98-2	1	50 to 100	15, 21, 32
	2	101 to 300	08, 18, 27
	3	2,200 to 3,200	05, 13, 25
99-1	1	50 to 100	15, 21, 27
	2	101 to 300	15, 23, 27
	3	2,200 to 3,200	05, 13, 14

Figure 2. Samples are placed in pint-size and quart-size bottles.

Study Approach

The USGS, Branch of Quality Systems (BQS), currently distributes 18 standard reference sediment samples annually in two separate sets to each participating laboratory. A total of nine samples—three from each sample class—are sent to each of the laboratories (table 1). The laboratories report the sediment concentration, the mass of sediment, and the percentage of sand-size and fine-size material (sand/fine separations) (Gordon and others, 1999).

Upon completion of the analyses by the participating laboratories, the analytical results along with the methods of analysis are returned to the BQS. Analytical results from all sediment quality-control samples are compiled and statistically summarized by BQS personnel on an intra- and inter-laboratory basis and entered into a national data base. Laboratories participating in the study and their customers can access their results, summary tables, graphs, control charts, and reports on the SLQA Web page at http://sedserv.cr.usgs.gov/. An example of a control chart is presented in figure 3. These control charts are prepared after each study by using the median percent difference as the central tendency line and ± 3 F-pseudosigma as the control limits. The closer the percent difference is to zero, the more accurate the individual determination is considered to be. The smaller the F-pseudosigma, the more precise the overall results are considered to be.

Laboratories that produce data that fall outside the control limits are evaluated in a follow-up study to resolve the source of the measurement discrepancies.

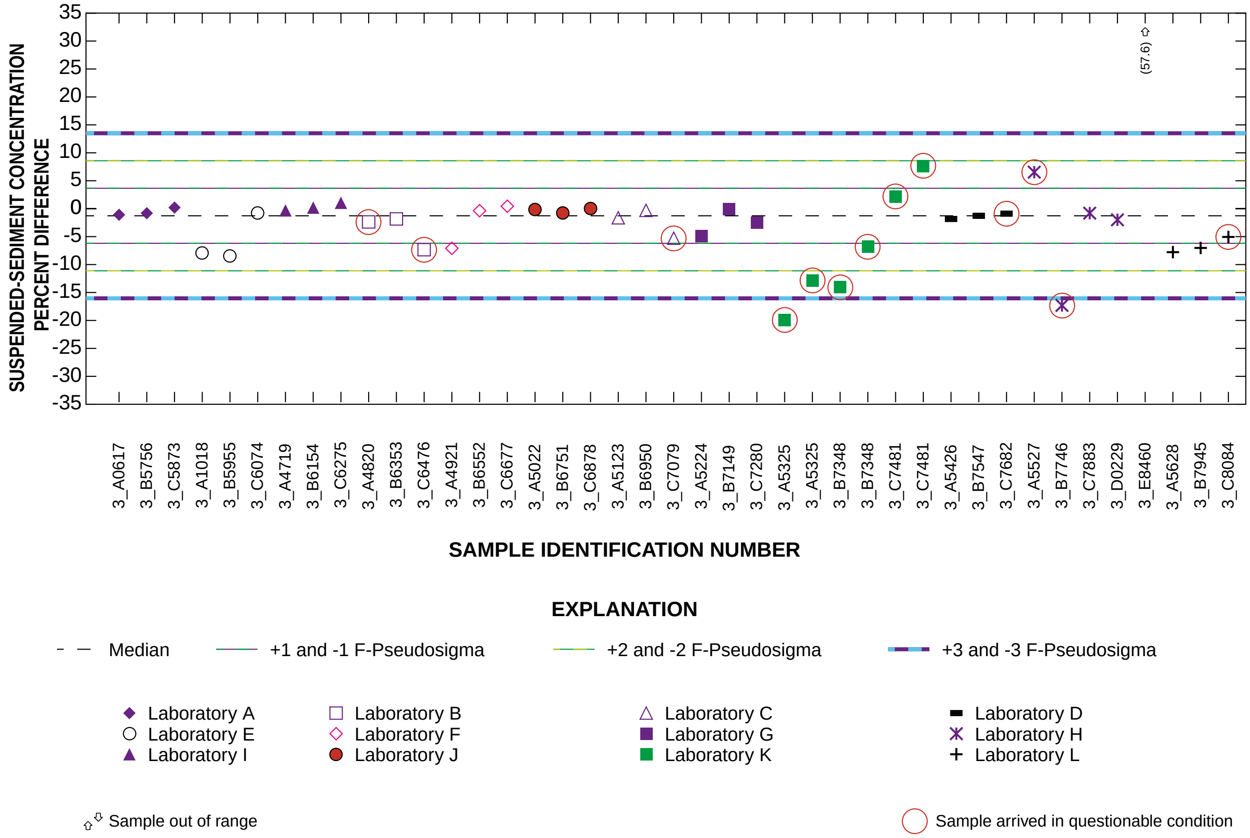


Figure 3. Control chart depicting sediment-concentration percent difference for samples containing 2,200 to 3,200 milligrams of fine-size material from study number 98-1.

Results of the First Seven Studies

The percent differences for each laboratory were tabulated for the seven studies completed between August 1996 and July 1999. The percent differences were calculated using the known and reported values for sediment mass, suspended-sediment concentration, and sand/fine separations. The median differences between the reported and known values can be used as an indication of laboratory bias. If the median difference from all of the pooled data reported by the laboratories were zero, the median difference line for each control would also be zero. The fact that the median difference is consistently offset from zero indicates that the data for each of the physical sediment properties are biased (fig. 4).

Fine-size material mass is consistently negatively biased, whereas sand-size material mass is generally positively biased. The negative bias for fine-size material mass may be due in part to dissolution of a small amount of the fine-size material. The positive bias observed for sand-size material mass may be due in part to a small amount of fine-size material adhering to the sand-size grains (Gordon and others, 1999). In addition, laboratories may be experiencing difficulty separating the fine-size material from the sand-size material due to the high percentage of clay-size material (about 40 percent) in the AC Spark Plug dust.

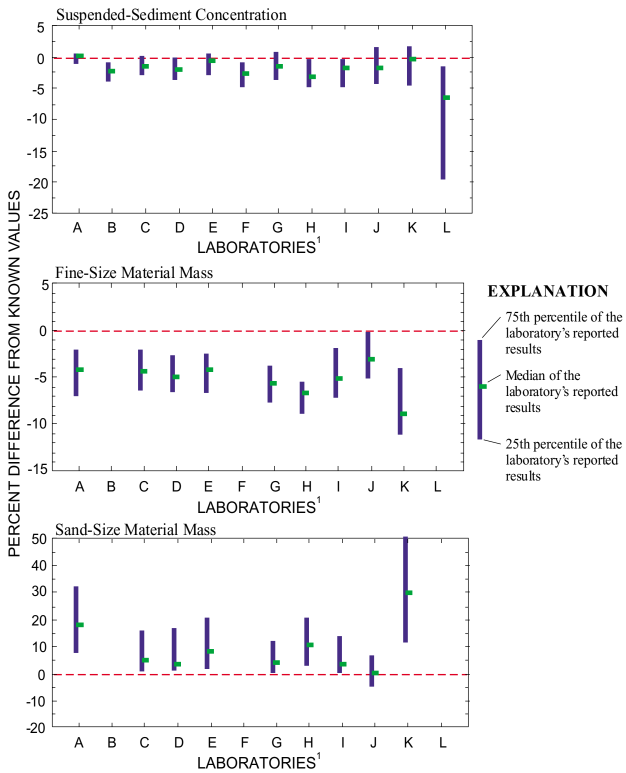
Project Expansion

Follow-Up Investigations

Beginning in 1999, follow-up studies were performed when a laboratory returned a specified number of analyses that plotted outside statistically determined data-quality boundaries or for which substantial deficiencies in a laboratory's methods were detected. The laboratory was then asked to process additional quality-control samples to determine if the deficiency had been corrected. The intent of the follow-up evaluation is to identify and eliminate the source of any discrepancies, increase the competence of the laboratory, and improve the data the laboratory produces (U.S. Geological Survey, Office of Surface Water Technical Memorandum No. 98.05, 1998). The laboratory under review, the USGS Office of Surface Water, and the BQS discuss the results of follow-up evaluations before the data are added to the national data base. Recommendations and any subsequent quality-assurance testing are based on the outcome of the follow-up evaluation (Gordon and others, 1999).

Double-Blind Study

A new double-blind study began in 1999. This study provides the capability for customers that submit environmental suspended-sediment samples to participating laboratories also to submit quality-control samples that are disguised as environmental samples. In the double-blind study, samples are processed and shipped from the collection site to provide infor-



[1]Two laboratories are contract laboratories and 10 laboratories are U.S. Geological Survey laboratories.

Figure 4. Summary charts of qualitative findings for suspended-sediment concentration, fine-size material mass, and sand-size material mass shown by individual laboratory.

mation on bias and variance attributable to routine shipping, processing, and handling steps. The double-blind study also augments information on the bias, precision, and accuracy of suspended-sediment data provided by the single-blind studies (U.S. Geological Survey, Office of Surface Water Technical Memorandum No. 98.05, 1998).

Summary

The U.S. Geological Survey (USGS) is the leading agency in assessing the Nation's water quality. To be able to study sediment effectively, the USGS operates sediment laboratories that determine the suspended-sediment concentration and sand/fine separations of fluvial samples. In 1996 the USGS began the Sediment Laboratory Quality-Assurance (SLQA) project to provide information on the bias and variability of the data received from its sediment laboratories.

For each participating laboratory, percent differences for suspended-sediment concentration, mass of fine- and sand-size material, and net sediment mass were tabulated for the seven studies completed between August 1996 and July 1999. The median differences between the reported and known values can be used as an indication of laboratory bias. If a laboratory showed no bias, then the median percent difference for that laboratory would be zero.

References

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Koltun, G.F., Landers, M.N., Nolan, K.M., and Parker, R.S., 1997, Sediment transport and geomorphology issues in the Water Resources Division: Proceedings of the U.S. Geological Survey Sediment Workshop, Reston, Virginia, February 4-7, 1997, p. 1.

U.S. Geological Survey, 1998, A national quality assurance project for sediment laboratories operated or used by the Water Resource Division: U.S. Geological Survey, Office of Surface Water Technical Memorandum No. 98.05, issued March 2, 1998, at URL http://water.usgs.gov/admin/memo/SW/sw98.05.txt

—--John Gordon and Carla Newland

Information on technical reports and data related to the Sediment Laboratory Quality Assurance project can be obtained from:

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The Sediment Laboratory Quality-Assurance project is one component of a national program to quality assure the sediment data produced by laboratories operated or used by the U.S. Geological Survey. The other components are: training in laboratory operational procedures, onsite laboratory evaluations, documentation of laboratory quality-assurance plans, and quality-control procedures. Questions regarding the other components of the national program to quality assure sediment data can be addressed to:

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