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Quality-Assurance Plan for the Analysis of Fluvial Sediment by the Northeastern Region, Kentucky District Sediment Laboratory

By Clyde J. Sholar *and* Elizabeth A. Shreve

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CONVERSION FACTORS

Multiply	By	To Obtain
centimeter (cm)	0.3937	inch
gram (g)	0.03527	ounce, avdp
gram per cubic centimeter (g/cm^3)	0.03613	pound per cubic inch
liter (L)	33.818	ounce, fluid
liter (L)	2.113	pint
liter (L)	1.057	quart
liter per minute (L/min)	0.2642	gallon per minute
milliliter (mL)	0.03381	ounce, fluid
millimeter (mm)	0.03937	inch

Degrees Celsius (°C) can be converted to degrees Fahrenheit (°F) by use of the following equation:

$$°F = (9/5)(°C) + 32$$

ABBREVIATIONS

Abbreviated water-quality units used in this report:

µS/cm - microsiemens per centimeter at 25 degrees Celsius
mg/L - milligrams per liter
ppm - parts per million

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Abstract

This report describes laboratory procedures used by the U.S. Geological Survey, Northeastern Region, Kentucky District sediment laboratory for the processing and analysis of fluvial-sediment samples for concentration of sand and finer material. The report details the processing of a sediment sample through the laboratory from receiving the sediment sample, through the analytical process, to compiling results of the requested analysis. Also described are procedures for preserving sample integrity, calibrating and maintaining of laboratory and field instruments and equipment, analyzing samples, internal quality assurance and quality control, and validity of the sediment-analysis results. The report includes a list of references cited and a glossary of sediment and quality-assurance terms.

INTRODUCTION

This report describes a quality-assurance (QA) plan for the Northeastern Region, Kentucky District sediment laboratory to assure that standard procedures are being followed. This plan is based on quality-assurance and quality-control (QA/QC) guidelines described in Knott and others (1992) and Matthes and others (1991). Analytical methods described by Guy (1969) are followed and the methods documented.

This QA plan is described in the context of the sediment laboratory operated by the U.S. Geological Survey (USGS) in Louisville, Kentucky. The laboratory is operated with a staff of one full-time hydrologic technician (certified laboratory chief) and two part-time personnel under the direction of the Chief, Hydrologic Surveillance Section, who reviews all outgoing analytical results.

QUALITY ASSURANCE OF SAMPLE MANAGEMENT

The reliability of analyses provided by the sediment laboratory is affected by sample documentation received with the sample, as much as the quality of the sample provided. Erroneous sample documentation, incorrect analytical procedures, or loss of sample integrity may result from samples received by the laboratory that have illegible or incomplete sample-label data, inadequate analysis instructions, or were damaged during shipment.

Sediment Sample Shipment-Log Form

Before samples are sent to the laboratory, the originating office will complete the sediment sample shipment-log form (fig. 1). The completed form provides basic information required by laboratory personnel to correctly identify each sample and will be included with the corresponding shipping case of samples. Bottles are placed in shipping cases using the placement guides noted on the shipment-log form. Sample information is completed on the forms with bottle information corresponding to the sample number in the shipping case. If the shipping case contains samples from more than one stream location, a separate form must be completed for each location and included with the case.

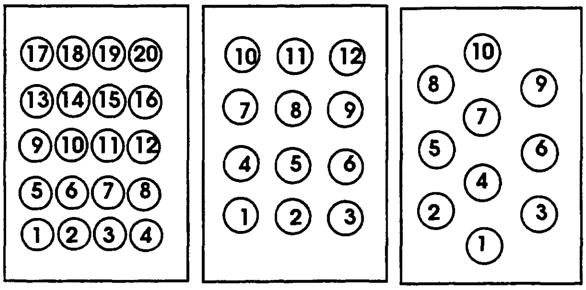
SEDIMENT SAMPLE SHIPMENT-LOG FORM

*********** PLEASE USE A SEPARATE LOG SHEET FOR EACH STATION IN A CASE ***********

STATION # ______________ *STATION NAME* ______________
LATITUDE ______________ *LONGITUDE* ______________
CURRENT DATE ______________ *OFFICE* ______________
CONTACT PERSON FROM YOUR OFFICE ______________ *ACCOUNT # FOR BILLING* ______________
PHONE # ______________ *CONDITION OF SAMPLES* ______________
POSSIBLE CONTAMINANTS: Y ___ *N* ___ *If yes, please explain:* ______________

LAB USE ONLY	*BOTTLE NUMBER*	*DATE*	*TIME*	*ANALYSIS REQUIRED*	*GAGE HEIGHT*	*REMARKS*
	1					
	2					
	3					
	4					
	5					
	6					
	7					
	8					
	9					
	10					

LAB USE ONLY	*BOTTLE NUMBER*	*DATE*	*TIME*	*ANALYSIS REQUIRED*	*GAGE HEIGHT*	*REMARKS*
	11					
	12					
	13					
	14					
	15					
	16					
	17					
	18					
	19					
	20					



BOTTLE PLACEMENT GUIDES

SHIPPING INSTRUCTIONS

DATE AND MILITARY TIME SAMPLE WAS COLLECTED MATCH DATA ON THE BOTTLE (NOT NECESSARILY THE "BEGIN TIME" OF A X-SECT SAMPLES TO BE ENTERED INTO QW FILE)

MARK ANALYSIS REQUIRED:
1 = CONCENTRATION ONLY
2 = COMPOSITE CONCENTRATION
3 = SAND/FINE BREAK *(includes total concentration)*
4 = COMPOSITE SAND/FINE BREAK *(includes total concentration)*

REMARKS: INCLUDE TYPE OF SAMPLE, MAY USE MORE THAN ONE SYMBOL COMBINATION.
O = OBSERVER
T = TECHNICIAN SAMPLE FROM FIXED LOCATION
X-EWI = CROSS-SECTION EQUAL WIDTH INCREMENTS
X-EDI = CROSS-SECTION EQUAL DISCHARGE INCREMENTS
P = FROM PUMPING SAMPLER
REP = REPLICATE SAMPLE
AC = AUTO CENTROID

LAB USE ONLY

DATE SAMPLES RECEIVED___/___/___
DATE SAMPLES LOGGED IN ___/___/___
RACK NUMBER ______________
DATE ANALYSIS COMPLETED ___/___/___
REMARKS:

PREPARED BY KENTUCKY DISTRICT SEDIMENT LABORATORY 7/22/98

Figure 1. Example of Sediment Sample Shipment-Log Form.

Suggestions for preparation and shipping of samples are as follows:

- Use permanent markers for completion of the shipment-log forms
- Include the account number (for billing purposes) in the designated space on the shipment-log form
- **Indicate any known or possible contaminants that might be present in the samples**
- Following the placement guides on the shipment-log form, arrange samples in the shipping case in chronological order matching the bottle number information on the shipment-log form with the corresponding bottle number in the shipping case. Samples will be processed in the order in which they are placed in the case.
- Have field personnel mark the water line of at least two bottles per case with a grease pen to assist laboratory personnel in detecting if leakage or evaporation occurred at any point from the time the sample was collected until sample analysis.
- Stream location names and downstream order numbers should be accurate and complete. Samples collected from more than one stream location may be shipped in the same case, but there must be a separate form for each sample location included in the case.
- The required analysis must be noted for each sample.
- Record on the form if the bottles from a cross-section or multi-bottle samples are to be composited for analysis or analyzed as discrete samples. If two or more cross sections are collected, indicate the type of analysis requested for each cross-section. Mark the field-sample labels for a cross section as "1 of 12, 2 of 12, . . . 12 of 12."
- Record the sample shipping date. Laboratory personnel will record the date the samples were received and the condition of the samples upon arrival. The sediment sample shipment-log forms will be returned to the customer along with applicable notes on the condition of the samples such as algae, underfilled or overfilled containers, foreign material present, leakage, or breakage.
- Include the name and telephone number of the person responsible for the samples on the shipment-log form to allow questions by laboratory personnel to be resolved in a timely manner.
- Check to see that the number of samples shipped agrees with the total on the shipment-log form.

A sample in a damaged container is discarded unless it is determined the sample can be reconstituted. It may be possible to transfer the sample to a new container or to determine the amount of water/sediment loss. Pertinent comments regarding sample condition are recorded on the shipment-log form, and customers are promptly notified of sample deficiencies. Customers are notified immediately if any samples were damaged during shipment.

Shipment-log forms and sample cases are tagged with corresponding temporary case numbers upon arrival at the laboratory. The forms are then sorted based on the analysis requirements of the samples. After the forms have been sorted by analysis type, water year, stream location, and chronological order, the cases of samples are sorted into analysis racks using the forms as guides. The cases of samples are renumbered for laboratory tracking purposes and stored in a cool, dark room until start of analysis.

Sediment-Sample-Bottle Label

The sediment-sample-bottle label (fig. 2) is completed at the time of sample collection. A permanent marker should be used to write on the label. Required label information supplied by the person collecting the sample is the water body (Stream), location, date, and time (use military time). Gage height (G.H.), measured water discharge (Qm.), water temperature (W.T.), specific conductance (Cond.), sample number (i.e., 1 of 12), and the initials of the collector (Party) may also be provided. The "Additional Data" section on the label may be used to record the collection station and method (box, point, EWI, EDI, grab, or other).

STREAM ____________________
LOCATION ____________________
DATE ____________ TIME ________
G.H.____ Qm.____ W.T.____ Cond.____
SAMPLE NO.________ PARTY ________
ADDITIONAL DATA:____________________

EXPLANATION

G.H. – Gage height
Qm. – Measured water discharge
W.T. – Water temperature
Cond. – Specific conductance

Figure 2. Example of sediment-sample-bottle label.

Samples from an automatic sampler require that the following information is completed on the labels: the station location, the date the sample set begins, and sequence number 1 on the first bottle. If the bottles are kept together as a set, only the first and last bottle require both a sequence number and date. All other bottles require only a sequence number. A completed sediment sample shipment-log form, with bottle numbers and corresponding sequence numbers, must accompany these samples.

Sample Shipment and Storage

Under normal conditions, few problems are encountered when shipping samples during the warm-weather months. Extra care must be taken when shipping samples during cold weather because freezing may cause minerals to form a precipitate, glass bottles to fracture, or plastic bottles to split. Any of these conditions will compromise sample validity. **Also, handling of fractured glass bottles by the carrier and laboratory personnel is a potential**

hazard. Avoid shipping samples when the air temperature is below freezing (0°C).

The U.S. Army Corps of Engineers Waterways Experiment Station Hydraulics Laboratory sediment-sample cases are the recommended shipping cases for all glass bottles; they are hard plastic cases with foam liners on all sides. **These cases must be used if a shipment is mailed using the U.S. Postal Service.** 3M Polyethylene Film Tape (or the equivalent) should be used on each bottle within the shipping case to secure plastic bottle lids to prevent loosening and leakage.

The Northeastern Region, Kentucky District sediment laboratory provides a written information sheet that summarizes laboratory policy for sample shipment and storage. Each customer is provided with instructions for shipment and storage of samples. Sample storage instructions are displayed in the laboratory.

Proper storage of samples minimize the potential for sample contamination or loss. Improper storage may result in algal growth, dirt and grime accumulation on the exterior of the sample bottles, evaporation of water, spillage or breakage of samples, and loss of labels. The customer is advised of any problem.

When samples are received in the sediment laboratory, the weight and specific conductance are measured and recorded on the sample bottles before they are stored. If it is not possible to measure the weight of the samples when they are received, it is obtained within 2 weeks of arrival to prevent error in analysis as a result of evaporation. Bottles are wiped with a cloth to remove excess dirt from the exteriors before gross weight is determined. Loose or cracked container caps are replaced; samples are stored in a cool, dark location to prevent algal growth.

QUALITY ASSURANCE OF LABORATORY EQUIPMENT AND COMPUTER SOFTWARE

A variety of specialized equipment is required for weighing, processing, and analyzing sediment samples. Most equipment such as balances, ovens, specific-conductance meters, vacuum pumps, thermometers, and calculators can be purchased from commercial sources. Decanting and filtering devices must be fabricated to best fit the needs of the laboratory.

Reliable analytical results are achieved by the use of calibrated equipment. The laboratory maintains the calibration of its equipment by systematic checking against reference standards and routine-maintenance schedules. Documentation is essential for support of the QA programs of the laboratory; therefore, all equipment used in the laboratory has a logbook documenting calibration records.

Balances

Two types of balances are used in the sediment laboratory. The macro balance is used to weigh items ranging from 100 g to 3,000 g, with a precision of +/- 0.5 g. The analytical balance is used to weigh items less than 100 g with a precision of +/- 0.0005 g. Both balances are mounted on anti-vibration tables to maximize stability during use. The analytical balance has a protective hood over the weighing pan to minimize air-current disturbances. Both balances are fitted with RS232 connectors for electronic data transfer directly into a computer.

Balance calibration is checked prior to, and at the end of each weighing session. The macro balance is checked with at least two Class S weights, traceable to the National Bureau of Standards (Friedman and Erdmann, 1982), that bracket the range of expected weights of the samples. The macro balance is recalibrated if there is an error of more than +/- 0.5 g. The analytical balance is also checked with at least two Class S traceable weights that bracket the expected weight to be measured. The analytical balance is recalibrated if the measured weight deviates more than 0.001 g from the standard weight. The balances are serviced by a technical representative if calibration does not bring them within required tolerances.

Balances are serviced and calibrated at least annually by a service representative using National Bureau of Standards traceable weights. All balance checks, calibrations, and professional servicing for each balance are recorded in dedicated instrument logbooks.

During computer entry, the analyst compares the weight displayed on the balance with the weight displayed on the computer terminal screen to confirm all entries. The empty balance is checked for a zero reading between each weighing with the balance being readjusted to zero after every ten readings.

Ovens

A convection-type drying oven is used to dry the sediment contained in crucibles and evaporating dishes. The oven is required to maintain a temperature in the range of 80 to 103°C, +/- 2°C.

Oven temperatures are checked at least twice during each drying operation. The temperature of the oven thermometer is recorded in the logbook. A calibration thermometer is used to check the oven thermometer; the calibration thermometer temperature is checked and recorded in the logbook. If the oven thermometer differs by more than 2°C from the calibration thermometer, the oven thermometer is replaced.

Conductivity Meter

The specific-conductance meter is used to measure the specific conductance of sediment samples. These specific conductance values assist in QA for samples collected at field sites, and assure the purity of deionized water used in laboratory processes. The units of conductance are measured in microsiemens per centimeter at 25 degrees Celsius (µS/cm). Specific conductance of a water-sediment mixture is a useful indicator of the conductivity of a stream at the time a sample was collected.

Prior to measuring sample conductivity, meter linearity is checked with at least three standard solutions in the range of expected sample values. The meter is recalibrated if more than a 2 percent difference exists between the meter readings and the standard solutions. The laboratory chief checks the meter on a quarterly basis with standards to verify meter operation. All calibrations are recorded in the specific-conductance meter logbook.

When the meter readings differ +/- 2 percent or more from the standards, the probe should be cleaned or replaced following the manufacturer's suggested procedures. Sediment laboratory personnel participate in the nationwide USGS National Field Quality Assurance testing by the USGS laboratory in Ocala, Florida.

Decanting Equipment

The decanting equipment consists of a J-shaped decanting nozzle attached to rubber tubing that is connected to a vacuum system. The vacuum system should be adjusted prior to each use to decant at a rate not to exceed 1 to 1-1/2 L/min to avoid disturbing the sediment.

Filtering Equipment

The filtration-method analysis of sediment samples requires the use of filtering equipment. The equipment consists of crucible holders (with filter adapters and glass funnel stems), rubber tubing, shut-off valves, storage tank for collecting surplus water, and a vacuum device. Crucible holders are mounted on a filtration rack.

Computer Software

Computer software programs are used to record, calculate, and store data from various instruments. Data transcription errors are minimized by the use of these automated programs, because electronic data transfer eliminates keypunch and inaccurate transcription errors.

The computer software is maintained by the Northeastern Region, Kentucky District computer section. The computer programs are tested by processing a sample for which the analytical results are known. The results from the predetermined entry are compared with the computer-entry results. Any difference in the results of the computer output indicates a problem in the computer hardware or software. All analyses are discontinued until the problem is resolved.

QUALITY ASSURANCE OF LABORATORY APPARATUS AND STANDARD SOLUTIONS

A variety of apparatus and standard solutions are commonly used in the laboratory when analyzing samples. Various laboratory apparatus include desiccators, sample sieves, and crucibles. Standard solutions include deionized water and specific-conductance standards. Written cleaning procedures for sample containers, evaporating dishes, and crucibles are posted in the laboratory near the cleaning area. All laboratory personnel are trained in cleaning techniques for the various types of apparatus.

Desiccators

Desiccator cabinets are used to store crucibles and evaporating dishes after they are removed from the drying oven to prevent the samples from reabsorbing moisture from the air during cooling. Hygrometers mounted in the desiccator doors indicate the humidity level of each desiccator cabinet. Desiccant containing a color indicator that monitors moisture is placed in the lower portion of each desiccator cabinet. When the hygrometer reading exceeds the acceptable range or the desiccant color indicator has changed, the desiccant is removed from the desiccator and oven-dried overnight at 110°C. The desiccant is replaced if it does not return to its original color after drying. Because humidity levels vary slightly in each desiccator, sample containers are assigned to a specific desiccator and are maintained in that desiccator throughout the processing sequence.

Desiccators are washed periodically with a laboratory detergent. The desiccant is dried and new desiccant or indicator is added as needed. Dates of cleaning and desiccant changes are recorded in the desiccator logbook.

Sample Bottles

Sediment samples may be collected in many types of sample containers. The 1-pint glass milk bottles and 1-quart glass bottles are the most common types of bottles used in standard USGS depth-integrating and point samplers. Automatic samplers use plastic or glass containers of varying sizes and shapes.

Empty sample bottles are weighed on the macro balance at the Northeastern Region, Kentucky District sediment laboratory to obtain the tare weight of the sample bottles to the nearest 1 g. This tare weight is then etched on the glass bottle or marked on plastic bottles with a permanent waterproof marker. Tare weights followed by an asterisk indicate the bottle weight includes the weight of the field label.

When samples have been analyzed, the empty glass sample bottles are first rinsed with an 8-percent solution of hydrochloric acid (HCL) to remove any calcium build-up or clouding of the bottle. Rinsing is done under a chemical-fume hood in the water-quality laboratory. Protective gloves, clothing, and goggles are worn when using acid to pre-clean sample bottles.

Glass bottles are returned to the sediment laboratory after the acid rinse and are washed with tap water, a low-residue detergent, and an electric bottle brush. The low residue detergent removes oil, grease, and other hard-to-remove material. A thorough rinse in tap water removes detergent and any remaining residue. The bottles are placed upside down on a bottle rack and air-dried overnight. The dried bottles are inspected for cleanliness, re-labelled, capped, and packed in shipping cases. If necessary, the washing procedure is repeated on bottles that were not thoroughly cleaned after the first washing. If chipped or cracked glass bottles are found during inspection, the bottles are discarded.

Plastic bottles are usually more difficult to clean. Their shapes do not conform to the shape of the bottle brush, and the bottle brush could scratch the plastic allowing sediment to become trapped in etch marks during subsequent use. Residue inside the bottles is difficult to see because most plastic containers are opaque. If a residue build-up is evident, the bottle is rinsed with an 8-percent HCL solution, washed with laboratory detergent, and the interior brushed manually. The bottle is thoroughly rinsed in tap water to remove detergent residue, and the drying procedure for glass sample bottles is followed. Plastic bottles are examined for cracks and interior scratches; defective bottles are discarded.

Five percent of the cleaned, dry containers are weighed to confirm tare weights. If there is a difference of more than +/- 1 g on any bottle from the set, another 5 percent are checked. If any bottle from this set is determined to have a difference of more than +/- 1 g, all containers from that group are retared. The corrected weights are etched on the bottles, and the bottles are capped and stored in shipping cases. Checking of tare weights is documented in a designated logbook.

Evaporating Dishes

Evaporating dishes are made of Pyrex glass and are used to weigh sediment in samples requiring the evaporation analytical process. The dishes are washed manually using a nylon scrub pad or a brush, followed by a thorough rinse in tap water. Dishes not in use are stored in closed drawers, while dishes for analysis are oven dried at 103°C +/- 2°C and cooled in assigned desiccators prior to use. Dishes are weighed to the nearest 0.0001 g with the analytical balance to obtain their tare weights prior to analysis.

Sieves

Sieves are used in determining the particle-size distribution of sample material that is larger than 0.062 mm. The sieves are inspected before each use for particle-clogged areas of the screen or tearing in the wire cloth. If needed, a magnifying glass is used for inspection. Sieves must retain a taut wire mesh without tears or clogging in order to maintain the integrity of the screen size (Tyler Industrial Products, 1976). Sieves are cleaned with an ultrasonic cleaner after each use; sieves that cannot be cleaned are replaced.

Crucibles

Pyrex brand Gooch crucibles with fritted discs are used in the laboratory. The fritted disc in the base of the crucible is fitted with a Whatman #934-AH glass fiber filter that allows for 1.5 μm retention of suspended solids. The crucible is used for samples estimated to have sand concentrations less than about 10,000 parts per million (ppm) and clay concentrations less than about 200 ppm. If the quantity of fine sediment is too great, the filter will become clogged, and some liquid will be retained in the crucible.

Crucibles are prepared for use in analyses by washing them in 1 gallon warm water and 1 ounce household bleach with a nylon scrub pad to remove any deposit build-up. Crucibles are thoroughly rinsed with tap water, placed under vacuum, and seated with glass-fiber filters.

Prior to seating the filters, deionized water is filtered through the crucibles to remove any loose filter fibers remaining in the fritted disc. If filtering time exceeds 30 seconds for a crucible, ultrasonic cleaning is required. This requires crucibles to be submerged in water mixed with an ultrasonic cleaning solution according to manufacturer's recommendations and cleaned for no longer than 10 minutes in a Sonicor Ultrasonic Cleaner. The time of cleaning varies with the type of material that is clogging the fritted-glass filter. The crucibles are thoroughly rinsed, and deionized water is again filtered through the crucibles. If filtering time remains at or exceeds 30 seconds, an 8-percent solution of HCL is used to clean the fritted disc. Crucibles are discarded when they no longer filter properly.

Filters are seated by pouring deionized water into each crucible, placing a glass-fiber filter centered in the crucible suspended on the water, and applying vacuum. Deionized water is again filtered through the crucible, which aids in seating of the filter and helps remove loose glass fibers. While the filter is under vacuum, it is checked for air escaping around its perimeter. If air is detected, the vacuum is turned off, the filter is adjusted and rewetted with deionized water, and the vacuum re-applied. If the filter does not seat properly it is replaced.

The crucibles with properly seated filters are placed in a Pyrex tray and oven dried 2 to 4 hours at 103°C +/- 2°C (Matthes and others, 1991). They are immediately placed in assigned desiccator cabinets to cool at least 3 hours before weighing. Each crucible with filter is weighed to the nearest 0.0001 g before use. Crucibles are assigned a specific desiccator cabinet to minimize humidity changes during the analyses procedures. Tongs or gloves are used by laboratory personnel when handling crucibles to avoid contaminating crucibles with moisture, dirt, or oil. A filter logbook of the lot numbers, dates received, and dates used is maintained to document filter usage in the event a certain production lot is found to be defective.

Deionized Water

Deionized water is used in the laboratory to prevent the addition of dissolved solids when the sediment sample is rinsed into a crucible, evaporating dish, or during wet-sieving. Deionized water is produced by passing tap water through a mixed cation-anion exchange resin. The deionized water is prepared by the laboratory with a Barnstead Nanopure II purification system. The specific conductance of deionized water at 25°C must not exceed 10 µS/cm (Knott and others, 1992). Specific conductance of the deionized water is measured for each analytical sequence and the readings are recorded in a deionized water logbook.

Storage bottles for deionized water require periodic cleaning with deionized water and liquid bleach to remove organic build-up. A cleaning solution of about 250 mL of liquid bleach and 20 L of detergent water is added to the storage bottle. The storage bottle is agitated and allowed to soak for 5 minutes; this procedure is repeated at least three times. A brush is used to remove material that cannot be removed by agitating, then the bottle is rinsed with deionized water until there is no evidence of residue. Cleaning of the storage bottles is annotated in the deionized water logbook.

Specific-Conductance Standard Solutions

Specific-conductance standard solutions are obtained from the USGS in laboratory Ocala, Fla. All standards are labeled with an expiration date and are discarded after that date. Specific-conductance standard solutions are stored in tightly capped containers to avoid contamination and evaporation. After calibration procedures, solutions are discarded after each use.

QUALITY ASSURANCE OF ANALYTICAL PROCEDURES

The Northeastern Region, Kentucky District sediment laboratory follows the methods described in Guy (1969) when analyzing fluvial-sediment samples for concentrations or sand-fine separations.

The standard scientific unit used for expressing sediment concentration in the laboratory is milligrams per liter (mg/L). The sediment concentration is calculated automatically by a computer program as follows:

$$\text{mg/L} = \text{Concentration (ppm)} = \text{Concentration}\left[\frac{\text{Weight of sediment x } 10^6}{\text{Weight of water-sediment mixture}}\right]. \qquad (1)$$

USGS policy requires publishing sediment concentration values in mg/L; therefore, a conversion from ppm to mg/L is required. The conversion factors for concentrations ranging from 0 to 539,000 ppm are listed in table 1. Correction factors are applied automatically by the computer program for sample concentrations of 8,000 ppm or greater.

Table 1. Factors for converting suspended-sediment concentration from parts per million (ppm) to milligrams per liter (mg/L)

[The values of C are based on the assumption that the density of water is 1.000 grams per cubic centimeter plus or minus 0.005 grams per cubic centimeter, the range of temperature is 0-29 degrees Celsius, the specific gravity of sediment is 2.65, and the dissolved-solids concentration is less that 10,000 ppm]

Concentration range (ppm)	Conversion factor (C)	Concentration range (ppm)	Conversion factor (C)
0 - 7,900	1.00	327,000 - 336,000	1.26
8,000 - 23,700	1.01	337,000 - 346,000	1.27
23,800 - 39,200	1.02	347,000 - 356,000	1.28
39,300 - 54,300	1.03	357,000 - 366,000	1.29
54,400 - 69,200	1.04	367,000 - 375,000	1.30
69,300 - 83,700	1.05	376,000 - 385,000	1.31
83,800 - 98,000	1.06	386,000 - 394,000	1.32
98,100 - 112,000	1.07	395,000 - 403,000	1.33
113,000 - 126,000	1.08	404,000 - 412,000	1.34
127,000 - 139,000	1.09	413,000 - 421,000	1.35
140,000 - 153,000	1.10	422,000 - 429,000	1.36
154,000 - 166,000	1.11	430,000 - 438,000	1.37
167,000 - 178,000	1.12	439,000 - 446,000	1.38
179,000 - 191,000	1.13	447,000 - 455,000	1.39
192,000 - 203,000	1.14	456,000 - 463,000	1.40
204,000 - 215,000	1.15	464,000 - 471,000	1.41
216,000 - 227,000	1.16	472,000 - 479,000	1.42
228,000 - 239,000	1.17	480,000 - 487,000	1.43
240,000 - 251,000	1.18	488,000 - 495,000	1.44
252,000 - 262,000	1.19	496,000 - 502,000	1.45
263,000 - 273,000	1.20	503,000 - 510,000	1.46
274,000 - 284,000	1.21	511,000 - 517,000	1.47
285,000 - 295,000	1.22	518,000 - 525,000	1.48
296,000 - 306,000	1.23	526,000 - 532,000	1.49
307,000 - 316,000	1.24	533,000 - 539,000	1.50
317,000 - 326,000	1.25		

Concentrations

The procedures followed by the Northeastern Region, Kentucky District sediment laboratory for concentration determination are listed below. These procedures focus on QC and include specifics not contained in Guy (1969).

Two methods used for the analysis of suspended-sediment concentrations are the filtration method and the evaporation method. Both methods require the sediment-free water to be decanted from the sample without disturbance of the settled sediment.

The filtration method requires use of a crucible, a filter, and the vacuum system. The sample is poured into a crucible, and vacuum pressure is applied. After the water-sediment mixture is forced through the filter, the crucible is oven dried, cooled, and weighed. The advantage of this method is that the dissolved solids present in the sample water will pass through the filter; therefore, mathematical adjustments for dissolved solids are not needed. A disadvantage to this method is that large quantities of sediment in the sample can clog the filter, which slows filtration.

The evaporation method is used when the concentration of the water-sediment mixture exceeds about 10,000 mg/L of sediment that is mostly sand and about 200 mg/L of sediment that is mostly clay (Guy, 1969). After washing the water-sediment mixture into the evaporation dish, the sample is dried and weighed. The primary disadvantage of the evaporation method is that a correction for dissolved solids may be required. A dissolved-solids correction needs to be applied when the dissolved-solids concentration is greater than 200 mg/L and the sediment concentration is less than 200 mg/L (Guy, 1969).

Login of Samples for Concentration Analysis

Upon receipt at the laboratory, samples and sediment sample shipment-log forms are examined for sample condition and requested analysis type. The samples are checked for damage during shipment when the laboratory receives the samples and damage is noted on the form and customers are notified. The forms are used by the laboratory to verify that all samples have been received.

The data entry procedures described are used with a suspended-sediment concentration computer program loaded on the UNIX operating system. Documentation for this program is maintained in the laboratory.

Use of the computer during data entry and processing eliminates errors that are common during manual processing of samples. Transcription errors are eliminated by electronic transfer of balance readings. Tests are built into the computer software to check the entry of weights, acceptance of the weights in the computer, and the validity of the computations.

The samples are logged-in chronologically. Login of a concentration sample consists of entering sample field label information; measurement of specific conductance; entering the bottle tare weight, and the gross weight of the bottle and water-sediment mixture; and the selection of the method to be used for the analysis. The computer program stores the data in data files, which are compared with information on the sediment sample shipment log forms. Mislabeled or missing samples are noted on the log forms. If sample information cannot be resolved in the laboratory, the customer is contacted.

The final entry in each new data file is a QC, precalculated system-check sample that validates the operation of the computer and software. Thirty-one check-sample entries are available for this validation—one for each day of the month. The results from the check samples cover the normal range of sediment concentrations analyzed by the laboratory. Data are entered for the check sample from the system-check list for the current date. When the final results for a data file are made available, the check-sample results from the data file are compared for agreement with the precalculated results of the check sample. Any error would indicate a problem with the computer hardware or with the sediment-concentration software program.

Five percent of the samples analyzed are QC sample blanks or field replicates. During the login process, sample blanks are entered into the data file along with the field samples. The laboratory chief is responsible for providing sample blanks, while field replicates are provided by the customer. Results of the precalculated system-check sample, sample blanks, and field replicates provide quantitative information about computer-program performance, analytical efficiency, and sample-collection technique.

After the login part of the analysis is complete, a worksheet is printed by use of the computer program. The worksheet is used by laboratory personnel during the remainder of the analysis for comparison with sample information and data. The worksheet is used to rebuild the data file if the computer malfunctions, and the data file is lost.

A listing of the station locations in a data file and the number of samples analyzed for each location are available as part of the computer-generated results. The laboratory retains a copy of this listing and records the inclusive dates of the analysis in a log of the samples analyzed.

Data Entry of Concentration Analyses

A computer program calculates sediment concentration following three distinct stages of data entry. Stage 1 inputs sample information from the sediment sample shipment-log form and sediment-sample-bottle label. Procedures during stage 1 include the following:

- Bottles are checked with corresponding bottle data on the sediment sample shipment-log form.
- Bottles are assigned a unique record number that will be kept throughout the analysis procedure.
- Information from the sediment-sample-bottle label is checked against the information on the sediment sample shipment-log form, and the bottle tare weight is entered into the computer. If no tare weight is available, the sample is marked and a tare weight is determined after the bottle is washed and dried. The data file is then edited to reflect the correct tare weight for calculation purposes.
- Gross weights obtained from uncapped samples and specific conductance are entered into the computer.
- Sample net weight is automatically calculated by the computer program when stage 1 is complete.
- Final bottle entry for a data file is a precalculated check sample for the current date.
- Stage 1 data files are printed and reviewed by the laboratory technician before proceeding.
- Sample bottles are moved to a decanting table to allow sediment to settle. Samples remain on the decanting table overnight before proceeding with the decanting process. Decanting is delayed if the samples appear cloudy after a 24-hour period, and an extended settling period may be considered.

During stage 2 of data entry, the tare weights of crucibles and (or) evaporation dishes are entered into the computer. Procedures during stage 2 include the following:

- Crucibles and evaporating dishes are numbered with unique identification numbers for use in the laboratory. Numbered crucibles are selected for use to correspond with assigned record numbers of samples.
- Crucibles are used in the filtration method.
- Evaporating dishes may be used with samples having net weights greater than 0.1 g.
- The dish number is recorded in the data file.
- Crucible or dish weight transfer is made by placing the container on the analytical balance. When the balance is stabilized, the letter "g" is displayed on the balance screen. By pressing the return key on the computer keyboard, the weight is electronically transferred into the data file.

- Crucible or dish weights and container identification numbers must be entered for all samples before proceeding to sample processing.
- A worksheet of the sample entry is printed at completion of stage 2 to prevent loss of data in the event of a computer failure. The software updates the sample information to the data file after each sample entry.

During stage 3 of data entry, the containers plus dried sediment are weighed. Only the number of samples that can be weighed in less than 1-minute exposure time to the ambient temperature are removed from the desiccator. The desiccator is closed between each removal process. Samples are weighed to the nearest 0.0001 g on the analytical balance and the gross weight is electronically transferred in the same method as the tare weight. Crucibles and dishes are transferred from the desiccator to the balance by using tongs, plastic gloves, or rubber fingertip pads.

Quality-Control Measures for Data Entry of Concentrations

- Sediment sample shipment-log forms are compared with data entered in the computer to confirm sample documentation.
- The analytical balance is connected to the computer, and the data are transferred electronically. Each digital display is compared with the weight entry on the screen to assure validity of the weight. The 0.0-g empty weight display is checked between each weighing.
- Weight displayed on the balance is compared with weight shown on the computer screen.
- Tongs or rubber gloves are used to handle any glassware.
- Desiccator door is tightly closed between weighings.
- Each crucible or dish is weighed within 1 minute of removal from the desiccator.
- Data worksheets of the concentration notes are reviewed, edited if needed, and initialed by the reviewer. A final print of notes is presented to the laboratory chief for final review.

Analysis of Samples for Concentration

The analysis of samples for concentration require supplies and equipment, the determination of sampling method (filtration or evaporation), calculation of dissolved-solids corrections, and quality-control measures. Equipment and methods are described in Guy (1969), with the exception of quality-control measures. The following lists highlight the key components of each category for the analysis of samples for concentration.

Supplies and Equipment

- Pyrex brand Gooch crucibles with fritted discs and bottom diameters of 2.4 to 4.0 cm capacity for 30 mL of solution
- Whatman #934-AH glass-fiber crucible filters
- Crucible holders
- Pyrex evaporating dishes (100- and 200-mL capacity)
- Deionized water
- Vacuum system
- Analytical and macro balances
- Drying oven
- Decanting equipment
- Desiccator cabinets
- Computer and concentration software program

Filtration Method

- Sample data on the worksheet are compared with data on the field-sample label and with the bottle tare value. Errors are recorded on the worksheet and corrected in the edit option of the concentration software program.

- Most of the sediment-free water in the sample bottle is decanted using care not to disturb or remove sediment. A J-shaped tube and a vacuum system are used to suction the supernatant water from the top, away from the sediment. Care is taken not to tilt the bottle, which could disturb the sediment. The decanting rate should not exceed 1 to 1-1/2 L/min.
- The crucible is placed in the crucible holder, the filter wetted with deionized water, and suction applied to seat the filter.
- The crucible identification number is checked against the container identification number on the sample to assure agreement.
- All sediment is washed from the sample container with deionized water into the appropriate crucible. Care is taken not to spill any of the sample during the transfer.
- Crucibles are placed in a convection oven and dried at 103°C +/- 2°C for a minimum of 3 hours or overnight.
- Crucibles are immediately placed into a desiccator to cool for at least 3 hours or overnight.

Evaporation Method

The evaporation method is used if it is evident the sample contains a large amount of sediment that would be difficult to filter in a crucible. The water-sediment mixture is poured into the evaporating dish, and the sample is dried and weighed. If required, a dissolved-solids sample is collected and analyzed prior to the evaporation analysis.

- Evaporating dishes are aligned sequentially on the countertop.
- Sample data on the worksheet are compared with data on the field-sample label and with the bottle tare value. Errors are recorded on the worksheet and corrected using the edit option of the concentration program.
- Dish identification number is checked against the container identification number on the sample to assure agreement.
- All sediment is washed from the bottle into the dish using deionized water. A dish of sufficient volume is used to avoid spillage when transferring the dish to the convection oven.
- Evaporating dishes are placed in the oven. They are dried at 90-95°C until all visible water is removed to avoid any loss of sediment from boiling. When all visible water is removed, samples are then dried for an additional 3 hours at 103°C +/- 2°C or overnight. Containers are then cooled in a desiccator for a minimum of 3 hours, or overnight.

Dissolved-Solids Correction Calculation

- The tare weight of a 100 mL evaporating dish is measured and recorded.
- Before decanting, 25 mL of sediment-free native water is removed from the sample, by use of a pipet, and placed in the evaporating dish.
- The evaporating dish is dried at 85 to 95°C until visible moisture has evaporated. The oven temperature is raised to 103°C +/- 2°C for 1 hour to complete the drying process.
- The dish is placed in a desiccator cabinet to cool. (Note: Evaporation dishes used to calculate dissolved-solids corrections are never placed in desiccators containing environmental samples.)
- The gross weight of the dish and solid residue are calculated and recorded.
- The net weight of the residue is calculated and recorded.
- The dissolved-solids correction (C) is computed as follows:

$$C = \frac{\text{net weight of residue x sample volume (after decanting)}}{\text{25 mL (pipet volume)}} . \qquad (2)$$

The net weight is measured to the nearest 0.0001 g, and the volume of sample (after decanting) is estimated to the nearest 1 mL. The dissolved-solids correction is then subtracted from the sediment net weight before calculating the concentration of the sample.

Quality-Control Measures

- Time to dry and desiccate crucibles is replicated when drying and desiccating samples.
- Crucibles and evaporating dishes are assigned to a specific desiccator throughout the analyses to compensate for variations in humidity.
- Label information, bottle tare weight, and crucible identification are compared with corresponding data on the worksheet for accuracy.
- Precalculated system-check, blank, and field replicate samples are immediately reviewed to verify analytical results.

Sand/Fine Separations

Sand/fine separations are used to determine sample concentration and the amount of material that is less than or greater than sand size. The term fine fraction refers to particles that pass through a .062 mm mesh sieve, and sand fraction refers to particles large enough to be retained on a sieve.

Login of Samples for Sand/Fine Analysis

A logbook is maintained for each customer to record receipt of samples. Identification numbers are assigned to each sample in the laboratory, and the identification number, station name and number, date, and number of bottles are listed in the logbook. The identification number is a unique number comprised of the prefix "sf," the water year of sample analysis with a three or more digit sequential number. Two letters are added to the unique number to identify the customer, and a rack number is appended to identify the group of samples. An example of the first Kentucky suspended-sediment sample analyzed in the 1998 water year for the percentage of sediment finer than 0.062 mm would be sf-98001-ky.01.

Data Entry for Sand/Fine Analyses

A particle-size computer program is used to store and compute the data and maintain a record of analytical status of samples. Documentation for the particle-size program is maintained in the laboratory.

Quality-Control Measures for Data Entry of Sand/Fine Analyses

- The sediment sample shipment-log form is compared with sample data entered in the computer to confirm sample documentation.
- The analytical balance is connected to the computer; the sample gross weights and the evaporating dish and crucible tare and gross weights are transferred electronically. The 0.0 g empty weight is checked between each weighing.
- Weight displayed on the balance is compared with weight shown on the computer screen.
- Tongs or rubber gloves are used when handling glassware.
- Desiccator door is tightly closed between evaporating dish or crucible weighings.
- Each crucible or dish is weighed within 1 minute of removal from the desiccator.

Analysis of Samples for Sand/Fine Separation

The analysis of samples for sand/fine separation requires supplies and equipment, wet-sieve processing calculation of dissolved-solids correction, and quality-control measures. The particle-size software is used to store information and compute results for concentration and the percentage of sediment finer than 0.062 mm. User documentation for this program is kept on file in the laboratory.

Supplies and Equipment

- A 0.062 mm mesh sieve
- Pyrex brand Gooch crucibles with fritted discs and bottom diameters of 2.4 to 4.0 cm capacity for 30 mL of solution
- Whatman #934-AH glass-fiber crucible filters
- Crucible holders
- Pyrex evaporating dishes (100- and 200-mL capacity)
- Deionized water
- Vacuum system
- Analytical and macro balances
- Drying oven
- Decanting equipment
- Desiccator cabinets
- Computer and a particle-size computer program

Wet-Sieve Processing

Wet-sieve processing is used to separate sand and coarser material from finer material.

- Thoroughly wet the surface of the sieve and place over Pyrex evaporating dish.
- The sample is washed onto the sieve with deionized water. The screen is rinsed with a gentle stream of water (gravity flow) to wash the particles through and into the assigned dish.The sieve is then rinsed with deionized water on the sides and underneath to remove any fine particles that may have adhered to the sieve.
- Five percent of the sand/fine splits analyzed in the laboratory receive an additional final rinse procedure. A clean crucible is assigned for one final rinse of the sample. Using a clean evaporating dish, the sample is rinsed thoroughly with deionized water, and this water is poured into the clean crucible. The purpose for this step is to monitor the thoroughness of the sand/fine separation.
- The sample fractions are dried according to the methods listed in the "Analysis of Samples for Concentration" section.
- The gross weights of sample fractions are obtained electronically by use of the analytical balance and the particle-size computer program.

Dissolved-Solids Correction Calculation

If a sample is dried in evaporation dishes, a dissolved-solids correction may be needed. This correction should be subtracted from only the fine fraction since the sand fraction is rinsed entirely with deionized water.

Quality-Control Measures

- Sieve screens are checked with a magnifying glass before and after each use and cleaned, repaired, or replaced, as needed.
- Crucibles or evaporating dishes, with unique identification numbers, are assigned to each sample to obtain sand weights. The identification number of the crucible or dish is written on the worksheet. Crucibles are assigned to each sample for use in obtaining fine weights; more than one crucible may be necessary. The crucible identification numbers are written on the worksheet.

QUALITY ASSURANCE OF LABORATORY PROCEDURES

Internal QA procedures provide a guide for monitoring the quality of the data. These procedures include the analysis of QA samples, review of logbooks and computer procedures, training, equipment checks, data reviews, and documentation. If expected results are not obtained, immediate action is taken by the laboratory chief to identify and resolve the problem.

Quality-Assurance Samples

Normally, 5 to 10 percent of the samples analyzed by the laboratory will be QA samples inserted by the analyst or the laboratory chief in addition to the QA samples from the customers. These samples consist of field-collected replicate samples, and blank samples. Blank samples are prepared from deionized water and will be distributed throughout the set of concentration-analysis samples. These samples will be clearly marked as QC samples. The laboratory is provided with a copy of the QC samples for a log record from the computer. Analyses from the QC samples are reviewed by the laboratory chief and the supervisor; corrective actions are taken as required. The notes of these corrective actions are retained in the QC logbook.

The Northeastern Region, Kentucky District sediment laboratory participates in the Sediment Laboratory Quality Assurance (SLQA) program of the Office of Water Quality, Branch of Quality Systems, and the QA/QC exercises developed by the National Stream Quality Accounting Network (NASQAN).

Logbooks

A logbook is maintained to document receipt of all samples by the laboratory. A summary sheet is prepared and attached to a hardcopy of sample results for each set of samples analyzed. One hardcopy of sample results is kept on file in the laboratory.

Computer Procedures

Documentation for the concentration and sand/fine separation programs used by the Northeastern Region, Kentucky District sediment laboratory is available on site. Only the system administrator, district sediment specialist, laboratory chief, and laboratory staff have access rights to the sediment-laboratory data files.

Data are archived on a hard-disc drive every 2 days. To avoid loss of data during a computer failure, a worksheet copy is made after each stage is completed in the concentration and sand/fine programs and a final formatted copy upon the completion of stage 3 of the program. At the end of the third water year, which begins on October 1 and continues to September 30 of the next calendar year, the data files for the first water year are archived on magnetic tape. The magnetic back-up tapes are stored off-site for added security. Hard-copy data files are maintained on site for a period of 5 years.

Training

The training program for new staff members improves productivity and proficiency in sediment-laboratory techniques and the use of the computer programs. A typical training program is as follows:

1. During the first week, each employee is given a tour of the laboratory and shown the various analytical processes done in the laboratory. Procedures are demonstrated for the following: use of the laboratory equipment, use of the computer concentration program, and login of samples.
2. Each laboratory employee is provided with a copy of the "Northeastern Region Kentucky District Sediment Laboratory Student/Employee Guidebook."
3. Each employee reads the QA plan to help provide a general understanding of the laboratory operations.
4. Each employee determines concentrations for 2 to 4 weeks under the supervision of the laboratory chief.
5. The work of the employee is reviewed carefully.
6. The next 4 to 8 weeks are used to increase proficiency and productivity of the employee. The work is reviewed weekly by the laboratory chief, and recommendations for further training are discussed with the district sediment specialist.

Equipment Checks

A logbook is maintained for each balance, meter, and oven. A logbook is also maintained to record information relative to maintenance of apparatus and checking of deionized water and standard reagents.

Data Reviews

The analyst computes, initials, and dates all results. Analysis results are reviewed to assure they are complete and reasonable. The person responsible for QA activities provides a final review of the QC samples. The Chief, Hydrologic Surveillance Section reviews the final results prior to these results being sent to the customer. Any corrective actions are included in a QA/QC logbook and in appropriate files.

Documentation

Basic references, a procedures manual, logbooks, and laboratory documents and correspondence are required documents needed to support the QA/QC program of the sediment laboratory.

1. The basic references maintained in the laboratory are Fishman and Friedman (1985), Friedman and Erdmann (1982), Guy (1969), Knott and others (1992), and Matthes and others (1991).
2. A QA/QC manual specific to the Northeastern Region, Kentucky District sediment laboratory is required to describe the specific methods, procedures, instruments, and equipment that are used by the laboratory. The manual is updated periodically to document changes in equipment, apparatus, or facilities. Changes or modifications to analysis methods would only be made with approval by the USGS Office of Surface Water, Reston, Virginia.
3. Logbooks are required to provide documentation of maintenance and calibration of equipment, analysis of QA/QC samples, and necessary corrective actions would be logged, if needed.
4. The laboratory QA Plan, shipping and login records, copies of worksheets and analysis results, and related correspondence are the basic documents on file in the laboratory.

DATA MANAGEMENT

Data are stored in computer files and on paper analysis forms. Each type of documentation is vulnerable to potential data loss, and back-up measures are used. Copies of all results provided to the customer are maintained in the laboratory files for a period of 5 years.

Data loss during analysis is decreased by printing working copies of each data entry stage for the samples. The working copies contain enough sample information to re-enter the files into the computer system, if necessary. The computer files are archived every other night. Only the district sediment specialist, laboratory personnel, and the system administrator have access rights to edit analysis results in the computer files.

SAFETY

New laboratory employees receive a safety orientation by the District Safety Coordinator. They each receive a copy of the Kentucky District "Emergency Procedures" handbook for reference, as needed. Laboratory safety and hazard-communication training is provided to all laboratory employees.

Safety concerns specific to the Northeastern Region Kentucky District Sediment Laboratory are documented in the laboratory safety handbook. The laboratory chief instructs laboratory personnel in the proper handling and storage of all glassware, containers, and laboratory equipment. Safety glasses, ear protection, back supports, rubber gloves, and protective aprons are the basic safety equipment provided to laboratory employees. An eyewash station, safety shower, and first-aid kit are easily accessible.

Acid-rinsing of sample containers must be done under the chemical-fume hood in the water-quality laboratory. Detailed safety-related procedures are explained in the "Chemical Hygiene Plan of the Northeastern Region Kentucky District Water-Quality Laboratory," which is maintained in the water-quality laboratory. This plan is designed to protect employees from overexposure to hazardous chemicals. No procedures take place in the laboratory without approval of the laboratory chief, and all chemicals must be stored in the certified chemical-storage cabinets provided.

REFERENCES CITED

Acceptable methods for the analysis and reporting of sediment data by the U.S. Geological Survey (USGS) are provided in USGS Techniques of Water-Resources Investigations publications; in internal technical memorandums of the USGS Water Resources Division, the Division's Office of Surface Water, and Office of Water Quality; and other publications. Reference is made to the following publications.

Fishman, M.J., and Friedman, L.C., eds., 1985, Methods for determination of organic substances in water and fluvial sediments: U.S. Geological Survey Techniques of Water-Resources Investigations, book 5, chap. A1, 709 p.

Friedman, L.C., and Erdmann, D.E., 1982, Quality assurance practices for the chemical and biological analyses of water and fluvial sediments: U.S. Geological Survey Techniques of Water-Resources Investigations, book 5, chap. A6, 181 p.

Guy, H.P., 1969, Laboratory theory and methods for sediment analysis: U.S. Geological Survey Techniques of Water-Resources Investigations, book 5, chap. C1, 58 p.

Knott, J.M., Sholar, C.J., and Matthews, W.J., 1992, Quality assurance guidelines for the analysis of sediment concentration by U.S. Geological Survey sediment laboratories: U.S. Geological Survey Open-File Report 92-33, 30 p.

Matthes, W.J., Sholar, C.J., and George, J.R., 1991, Quality-assurance plan for the analysis of fluvial sediment: U.S. Geological Survey Open-File Report 91-467, 31 p.

Tyler Industrial Products, 1976, Testing sieves and their uses: Cleveland, Ohio, W.S. Tyler Co., Handbook 53, 48 p.

GLOSSARY

The following are definitions of selected terms as they are used in this report; they are not necessarily the only valid definitions for these terms.

Accuracy—A measure of the degree of conformity of the mean value obtained by using a specific method or procedure with the true value. The concept of accuracy includes both bias (systematic error) and precision (random error) (Friedman and Erdmann, 1982).

Bias—A persistent positive or negative deviation of the mean value obtained by using a specific method or procedure from the true value. In practice, bias is expressed as the difference between the accepted true value and the mean value obtained by repetitive testing of a homogeneous sample (Matthes and others, 1991).

Precision—The degree of agreement of repeated measurements of a homogenous sample by a specific procedure, expressed in terms of dispersion of the values obtained about the mean value (Friedman and Erdmann, 1982).

Quality assurance—A term used to describe programs and the sets of procedures, including (but not limited to) quality-control procedures, which are necessary to assure data reliability. With regard to the analysis of fluvial sediment, the term includes practices used both by personnel outside as well as within the laboratory to assure the quality of laboratory data (Friedman and Erdmann, 1982).

Quality control—A term used to describe the routine procedures used to regulate measurements and produce data of satisfactory quality (Friedman and Erdmann, 1982).

Reference samples—Prepared sediment samples of known particle-size distribution or of a known weight. These samples are provided to laboratories or are produced in the laboratory as a part of the quality-assurance program.

Replicate sample—A second field-collected sample, collected using the same method of collection at the same location and at nearly the same time.

Sample blank—Samples consisting of deionized water that are inserted into the analysis routine. A sample blank verifies accuracy and precision of the laboratory balances and the quality of the deionized water and is a part of the quality-control program.

Split-sample—A part of a sample separately analyzed for comparison of results.

Standard solution—A fluid that is mixed to produce a specific value when it is tested with measurement instruments; it is used to check and calibrate the instruments.

Tare weight—The weight of a dry, clean, and empty sample container, crucible, or evaporation dish.