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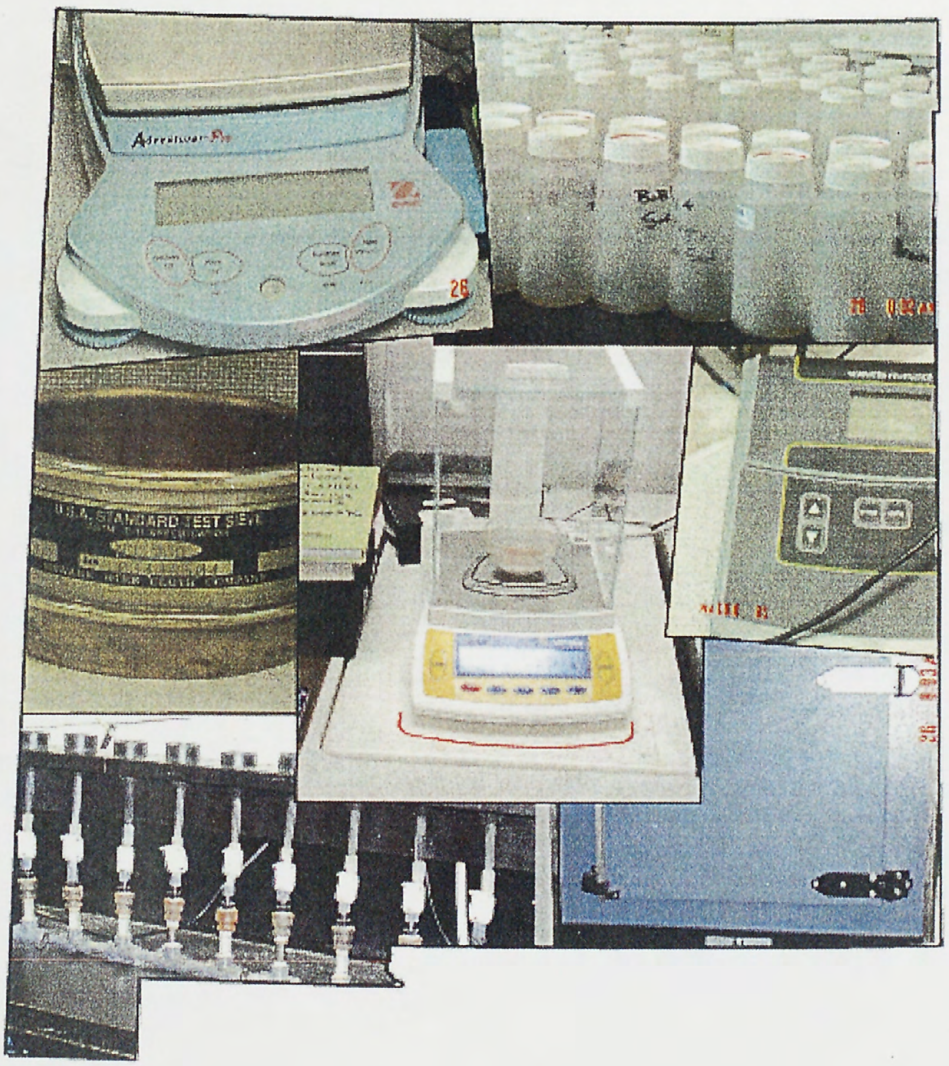
Quality-Assurance Plan for the Analysis of Fluvial Sediment by the U.S. Geological Survey New Mexico Water Science Center Sediment Laboratory



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U.S. Department of the Interior
U.S. Geological Survey

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By Jessica A. Stiles

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Conversion Factors and Abbreviations

SI to Inch/Pound

Multiply	By	To Obtain
	Length	
centimeter (cm)	0.3937	inch
millimeter (mm)	0.03937	inch
	Volume	
liter (L)	33.82	ounce, fluid (fl. Oz)
liter (L)	2.113	pint (pt)
liter (L)	1.057	quart (qt)
milliliter (mL)	0.03381	ounce, fluid (fl. Oz)
	Temperature	
degree Fahrenheit (°F)	°C=5/9 (°F–32)	degree Celsius (°C)
degree Celsius (°C)	°F=(1.8×°C)+32	degree Fahrenheit (°F)
	Flow rate	
liter per minute (L/min)	0.2642	gallon per minute (gal/min)
	Concentration (Mass/Volume)	
milligrams per liter (mg/L)	1.0	parts per million[1] (ppm)
	Mass	
gram (g)	0.03527	ounce, avoirdupois (oz)
milligram (mg)	35.27	ounce, avoirdupois (oz)
	Density	
gram per cubic centimeter (g/cm^3)		

[1]This conversion is true for:

$$\text{mg/L} = \text{Concentration (ppm)} = \frac{\text{Concentration} \times \text{Weight of Sediment} \times 10^6}{\text{Weight of Water-Sediment mixture}}$$

when the ratio of weight of sediment ($\times 10^6$) to weight of water-sediment mixture is between 0 and 8,000. If this ratio is greater than 8,000, the investigator is referred to Quality of Water Branch Technical Memorandum No. 72.10, table 1 and 2, for the correction conversion factor to be used in the formula (Edwards and Glysson, 1988).

Abbreviations

Specific conductance is given in microsiemens per centimeter at 25 degrees Celsius (µS/cm at 25°C).

Concentrations of chemical constituents in water are given in milligrams per liter (mg/L).

ppm – parts per million

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Abstract

This report describes laboratory procedures used by the U.S. Geological Survey New Mexico Water Science Center 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. Procedures for preserving sample integrity, calibrating and maintaining of laboratory and field instruments and equipment used to analyze samples, internal quality assurance and quality control, and validity of the sediment-analysis results also are described. The report includes a list of references cited and a glossary of sediment and quality-assurance terms.

Introduction

The U.S. Geological Survey operates sediment laboratories throughout the United States. Selected laboratories perform sediment analyses for suspended-sediment concentration, determination of percentage of sand and finer material, and complete particle-size analyses for suspended and bed-material sediment. The remainder of the laboratories analyze sediment samples only for suspended-sediment concentration and do only limited particle-size analysis. To ensure that all types of analyses performed in all laboratories are accurate and uniform, standard analytical procedures, as described by Guy (1969), must be followed and the procedures documented.

This report describes in context a quality-assurance (QA) plan for the U.S. Geological Survey (USGS) New Mexico Water Science Center Sediment Laboratory in Albuquerque, N. Mex., to assure that standard procedures are followed.

The USGS New Mexico Water Science Center Sediment Laboratory is operated with a staff of one full-time hydrologic technician (certified laboratory chief) and one additional part-time student contract employee. Members of the part-time staff generally are students in post-high school institutions, hired under special Federal hiring programs for students. About 15 percent of the operating funds of the laboratory are dedicated to quality control.

The reader is assumed to be familiar with basic sediment analytical techniques. This report sets forth the requirements for quality assurance and control as the user performs the various analyses in a sediment laboratory.

Sample Handling

The reliability of the analyses provided by the sediment laboratory is affected by the quality of samples received from the field. Information about a sample's collection location and the analysis requested also affect the quality of the data. It is critical that all information about the sample be with the sample for accurate login and sample analysis.

Sediment-Sample Bottle Label

The sediment-sample bottle label (fig. 1) is completed at the time of sample collection. A permanent marker is used to write on the label. Required label information supplied by the person collecting the sample includes the date, river, time (use military time), gage height (G. Ht.), station number (Sta.), and temperature (Temp.).

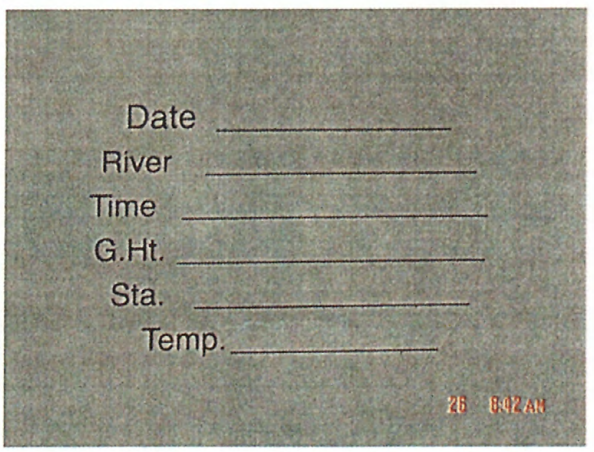

Figure 1. Sediment-sample bottle label.

Samples from an automatic sampler require the following information on the labels: river, station number, date the sample set begins, time for set, and sequence number 1 on the first bottle. If the bottles are kept together as a set, only the first and last bottles require both a sequence number and date. All other bottles require only a sequence number. A completed Field Form (fig. 2), with bottle numbers and corresponding sequence numbers, must accompany these samples.

New Mexico Sediment Laboratory Field Form

The New Mexico Water Science Center Sediment Laboratory has created its own unique form for submitting samples. Before samples are submitted to the laboratory, the Field Form (fig. 2) must be completed by the servicing technician during his or her visit to a sediment station. The form should be legible and include the information relevant to the sediment station's location, sample dates or sequence number, and analysis required. General comments about sample condition and pertinent remarks often are valuable to the laboratory personnel. The completed form provides basic information required by laboratory personnel to correctly identify each sample and, therefore, each set of samples must contain its own Field Form.

When shipping samples to other laboratories, a standard Sediment Laboratory Analysis Request (SLAR) form is used. It can be accessed online at *http://water.usgs.gov/osw/techniques/sediment/USGS_Sediment_Laboratories.html*.

New Mexico Sediment Laboratory Field Form (Field Form)

SEDIMENT SAMPLE FIELD LOG

ST.# _____ ST. NAME _____ RECORD # _____
CURRENT DATE _____ NAME _____ OFFICE _____ LAB # _____

*TYPE OF ANALYSIS: 1) CONCENTRATION 2) SAND / FINE BREAK 3) PARTICLE SIZE

BOTTLE #	DATE	TIME	*ANALYSIS REQUIRED	REMARKS	BOTTLE #	DATE	TIME	*ANALYSIS REQUIRED	REMARKS
1					15				
2					16				
3					17				
4					18				
5					19				
6					20				
7					21				
8					22				
9					23				
10					24				
11					25				
12					26				
13					27				
14					28				

X-SECTIONAL SAMPLE:
SAMPLE DATE _____ START TIME _____ END TIME _____
MEAN TIME _____ # BOTTLES ____ GAGE HT _____ TEMP _____
*ANALYSIS REQUIRED _____ LAB CONCENTRATION _____
REMARKS _____
IF TOTAL SIZE: ALSO INCLUDE
STREAM WIDTH _____ MEAN VELOCITY _____
MEAN DEPTH _____ INST. Q. _____
BED MATERIAL: TIME _____ # BED SECTIONS _____

X-SECTIONAL SAMPLE:
SAMPLE DATE _____ START TIME _____ END TIME _____
MEAN TIME _____ # BOTTLES ____ GAGE HT _____ TEMP _____
*ANALYSIS REQUIRED _____ LAB CONCENTRATION _____
REMARKS _____
IF TOTAL SIZE: ALSO INCLUDE
STREAM WIDTH _____ MEAN VELOCITY _____
MEAN DEPTH _____ INST. Q. _____
BED MATERIAL: TIME _____ # SECTIONS _____

Field Login Form.doc
4/27/2006

Figure 2. Sediment Laboratory Field Form.

Sample Storage

This section is divided into two major topics. The first topic discusses the instruments and equipment used in the laboratory. This includes the use of logbooks and the processes of documenting the calibration and maintenance of the instruments and equipment. The second topic describes the containers used in the field and laboratory. Described are various processes that can be used with these items to help ensure the validity of the sediment analysis.

Samples are stored in a cool, dark location when they are received at the laboratory. The samples are allowed to settle for 2 weeks and are analyzed within 60 days from the time they are received. If it is anticipated that a sample cannot be analyzed within this period of time, the weight of the water-sediment mixture of the sample is obtained and recorded to prevent errors because of evaporation. If algae growth is a problem in the sample, the growth normally occurs before receipt by the laboratory, and the laboratory chief is notified. Improper storage of samples before and after the samples are received by the laboratory can cause major difficulties and possibly complete loss of the sample. Some of the more common problems that occur during sample storage include growth of algae, dirt and grime accumulation on exterior of the sample bottle, evaporation of water, spillage or breakage of the sample, and loss of the field-sample label.

Instruments and Equipment

The instruments and equipment used in the laboratory that are discussed in this section are computers and computer software, balances, drying ovens, desiccators, equipment used in the evaporation and filtration analysis, sieves, and specific-conductance meters.

Computer Hardware and Software for Sediment Analysis

The information and data results for concentration and particle-size analyses of suspended-sediment are entered into the Sediment Laboratory Enviromental Data System (SLEDS) software program. Documentation for SLEDS software is available online at the USGS internal Web site: *http://eris.wr.usgs.gov/SedLab/framework.html*, accessed June 13, 2005.

Balances

Two types of balances are used in the USGS New Mexico Water Science Center Sediment Laboratory. The macro balance is used to weigh items weighing 100 g (grams) or more, such as sample containers with or without the water-sediment mixture. The precision of the macro balance is ±0.5 g (plus or minus 0.5 gram). The analytical balance is used to weigh items weighing less than 100 g, such as crucibles and evaporating dishes. The required precision of the analytical balance is ±0.0005 g. The analytical balance is mounted on an antivibration table to maximize stability during use. The analytical balance has a protective hood over the weighing pan to minimize air-current disturbances of the weighing pan. The balances are fitted with RS232 connectors to enable data transfer directly to a computer.

Balance calibrations are checked each day before use. Macro balances are checked with at least two Class S weights, traceable to the National Bureau of Standards (Friedman and Erdmann, 1982), in the range of weights of the anticipated samples. The macro balances are recalibrated if there is an error of more than ±0.5 g. Analytical balances are checked with at least two Class S traceable weights in the anticipated range of weights. The analytical balances are recalibrated if the measured weight deviates more than 0.001 g from the standard weight. The balance is serviced by a service representative if calibration does not bring it within required tolerances.

In addition to the daily checks by laboratory personnel, the laboratory chief checks the balances weekly with Class S weights to verify their accuracy. The balances are serviced and calibrated by a service representative using National Bureau of Standards traceable weights at least once a year. All balance checks, calibrations, and professional servicing for each balance are recorded in the instrument logbook. 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.

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 ranging from 85° to 103°C, ± 2°C. (See USGS Office of Surface Water Technical Memorandum no. 99.04, dated January 25, 1999, Guidelines from the 1998 sediment Laboratory Chiefs Workshop, online at *http://water.usgs.gov/admin/memo/SW/sw99.04.html*, accessed June 13, 2005.)

Oven temperatures are checked during each drying operation. The oven's internal thermometer readout is checked against the external digital reading to make sure the temperatures match. The values are recorded in each oven's logbook. Oven temperatures are checked twice during each operation. Some reduction in oven temperature may occur when drying large quantities of samples. This is acceptable as long as drying times are extended to allow for complete drying of sediment.

The oven's thermometer is checked semiannually at 80° and 110°C (American Society of Civil Engineers, 1975) against a calibration thermometer. The oven thermometers are replaced if they differ by more than 2°C from the calibration thermometer. These checks are also recorded in the oven's logbook.

Desiccators

Desiccator cabinets are used to store crucibles and evaporating dishes after removal from the drying oven to prevent the sample from reabsorbing moisture from the air during cooling. The desiccators are checked for moisture buildup each time they are used. A hygrometer mounted in the door of each desiccator indicates the humidity level of each desiccator cabinet. A desiccant with a color indicator is also used to monitor moisture, and it is replaced when the color indicator shows excessive moisture. The desiccant can be reused by drying in an oven until its original color is restored.

Twice a year the desiccant is removed, and the desiccator is washed with a laboratory detergent. The desiccant is dried and new desiccant or indicator is added as needed. The date of desiccator cleaning and desiccant change is recorded in the desiccator logbook.

Specific-Conductance Meters

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 specific conductance are measured in microsiemens per centimeter at 25°C (µS/cm). The precision of the specific-conductance meter used in the sediment laboratory is at least ±5 µS/cm. The probe used with the meter may be the plastic-immersion or flow-through type. Because of the fragile nature of glass-immersion probes, they are not recommended for use in the sediment laboratory.

Each time before use, a test sample is collected from the set of samples to be measured. The meter is then calibrated using a standard within the range of the expected set of sample values. This process is repeated with each new set of samples being measured. All calibrations are recorded in the specific-conductance meter logbook.

When the meter readings differ 5 percent 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 National Water Quality Laboratory (NWQL) in Denver, Colorado.

Specific-Conductance Standard Solutions

Specific-conductance standard solutions are stored in tightly capped containers to avoid contamination and evaporation. Extreme care is taken to avoid contamination. Once the solution is poured out of the original container, the solution is not poured back whether or not it is used. All standards are labeled with an expiration date and are not to be used after that time. Specific-conductance standards may be obtained from the USGS National Water-Quality Laboratory (NWQL), in Denver, Colorado.

Sieves

Sieves are generally used to determine the particle-size distribution of material that is larger than 0.062 mm. Two sets of 3-inch sieves are maintained for determining particle sizes according to the standard U.S. Geological Survey scale (Guy, 1969).

The sieves are inspected before each use for clogging of the screen with particles. If a sieve is estimated to have more than 25 percent of the surface area clogged with material, it is cleaned with an ultrasonic cleaner. A sieve that cannot be cleaned is replaced. The wire cloth must be taut in the sieve. When the wire cloth becomes baggy, the sieve is replaced. As long as the sieves retain a taut wire mesh without tears and are not clogged with material, the wire mesh will retain the integrity of the screen size (Tyler Industrial Products, 1976). The sieves are inspected after each analysis for tears in the wire cloth. Inspection is especially important in the finer mesh sieves. One sample in 20 is reanalyzed.

Decanting Equipment

The decanting equipment consists of a J-shaped decanting nozzle attached to rubber tubing that is connected to a vacuum system. To avoid disturbing the sediment, the decanting rate of samples is not to exceed 1 to 1.5 liters per minutes (L/min). A test is conducted twice a year to ensure that the decanting rate is not exceeding the proper rate; if it is outside the limits, the pump is adjusted.

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 counters specifically designed for this purpose.

Deionized Water

Deionized (DI) water is used in the laboratory to prevent the addition of dissolved solids when the sediment sample is rinsed into a crucible or evaporating dish or during wet-sieving. DI water is produced by passing tap water through a mixed cation-anion exchange resin. The DI water system is furnished through a local Albuquerque company. The specific conductance of DI water at 25°C must not exceed 10 µS/cm (microsiemens per centimeter). The specific conductance of the DI water is tested daily to determine if the water is suitable for use. If it is found unsuitable, the company is notified and the tanks are exchanged promptly. All readings are recorded in a DI water logbook.

Hand-held spray bottles, containing DI water used for rinsing samples, require periodic cleaning with deionized water and laboratory detergent to remove organic buildup. The specific steps in cleaning the storage containers may vary according to their size and shape. The bottle is agitated and allowed to soak for 5 minutes. This procedure is repeated as needed. The bottle is rinsed several times with deionized water to remove soap residue.

Sample Containers

Sample containers are of two types: containers for field use and laboratory containers. The sample containers for field use will be discussed first and laboratory containers discussed second.

Field Sample Containers

Sediment samples are collected in many types of sample containers, depending on the type of sampler used. The 1-pt glass "milk" bottle and 1-L plastic bottle are the containers most frequently used in USGS New Mexico Water Science Center Sediment Laboratory for depth-integrating and point samplers. All the automatic samplers used in this laboratory use a standard size plastic container.

Empty sample bottles are weighed on the macro balance. The tare weight, to the nearest 0.1 g, is marked on sample bottles before the bottles are available for field technicians to use. The weight is etched on a glass bottle with an etching tool or jack frost, a chemical glass-etching solution, and marked on a plastic bottle with paint or a permanent waterproof marker.

Glass Bottles

When samples have been analyzed, the empty glass bottles are first washed with tap water in a low-residue general-purpose laboratory detergent, by using a brush and scouring pad. The low-residue detergent removes oil, grease, and other hard-to-remove material. A thorough rinse in tap water followed by a thorough rinse in DI water, removes detergent and any remaining residue. If clouding or buildup of material is detected, the bottles are then rinsed with a 5-percent HCL (hydrochloric acid) solution to remove this. The HCL rinse is followed by a thorough rinsing in DI water to remove all residues left behind from the HCL solution. Once the bottles have been thoroughly washed, they are stacked upside down on a bottle rack and air dried. The dried bottles are inspected for cleanliness and rewashed if necessary before packing into field cases. The glass bottles are checked for chipped glass; if chips are found, the bottle is discarded. Five percent of the glass bottles are weighed to confirm their tare weights. If there is a difference of more than ±1 g on any bottle from the set, another 3 percent of the bottles are weighed. If any bottle from this set is determined to have a difference of more than ±1 g, all containers from that group are retared.

Plastic Bottles

Plastic bottles, depending on shape and style, generally are more difficult to clean. Most plastic containers are opaque, which causes some difficulty in visual detection of material on the inside of a container. Once the samples have been analyzed, the empty plastic bottles go through the same washing process as the glass bottles. The only difference would be the type of bottle brush used to wash them. Because of their odd shape, a bendable brush is used to make sure all hard-to-reach surfaces are properly cleaned. If, after washing with a low-residue laboratory detergent, a buildup is detectable, the bottle is also rinsed with a 5-percent HCL in the same manner as the glass bottles. Plastic bottles are examined for cracks that may cause leakage and are discarded when necessary. Tare weights are obtained on all plastic bottles before placing them in the proper field cases. If any corrections are needed, a large X is placed through the existing tare weight and the proper weight is then legibly marked on the containers, using permanent marker.

Laboratory Containers

Sediment samples are analyzed in two types of containers, depending on the analysis to be performed. The first type discussed is the evaporating dishes and the second, filtration crucibles.

All glassware and crucibles are handled with tongs, cotton or Nitril gloves, or rubber finger guards to avoid contamination by moisture, dirt, or oil.

Evaporating Dishes

Evaporating dishes are made of Pyrex glass and are used to weigh sediment in samples requiring the evaporation analytical process. The dishes made of Pyrex glass can be placed in the oven for the drying of the sediment sample. The dishes are washed manually by using a scrub pad or a brush, in a low-residue detergent, followed by a thorough rinse in tap water and then DI water. If a residue is apparent, the dishes are rinsed in a 5-percent HCL (hydrochloric acid) solution to remove the residue. A thorough rinsing in DI water follows to remove all residues left behind from the HCL solution. Dishes are then placed on drying racks and air dried.

Dishes not in use are stored in closed drawers. Dishes are weighed to the nearest 0.0001 g with the analytical balance, to obtain their tare weights before analysis. Once tared, the dishes are stored in a laboratory rack and covered with a clean, lint-free cloth to prevent contamination when they are not in the drawers.

Filtration Crucibles

Coors porcelain and glass crucibles are used in the sediment laboratory. The crucibles are fitted with Whatman #934-AH glass-fiber filters that allow for 1.5-µm retention of suspended solids. The filter allows passage of

clear liquid and dissolved solids; the sediment collects on the filter disk. The crucibles are used for samples estimated to have sand concentrations less than 10,000 parts per million (ppm) and clay concentration less than 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 warm water and a low-residue detergent with a scrub pad to remove any deposit buildup. Crucibles are thoroughly rinsed with tap water, placed under vacuum, and seated with glass-fiber filters.

Filters are seated by placing a glass-fiber filter centered in the crucible and applying vacuum. DI water is filtered through the crucible. The DI water aids in seating of the filter and helps remove loose glass-filter fibers. While the filter is under vacuum, the filter is checked for air escaping around its perimeter. If air is detected, the vacuum is turned off, the filter is adjusted and rewetted with DI water, and the vacuum reapplied. The filter is replaced if it does not seat properly.

The crucibles with properly seated filters are placed in a tray and oven-dried 3 hours to overnight at 103°C, ±2°C (Matthes and others, 1992). The crucibles are immediately placed in a desiccator cabinet to cool at least 3 hours before weighing. Each crucible, with filter, is weighed to the nearest 0.0001 g with the analytical balance, to obtain a tare weight prior to analysis. 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 defective.

Quality Assurance of Analytical Procedures

The USGS New Mexico Water Science Center 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 the computer program SLEDS, as follows: mg/L = Concentration (ppm) = Concentration

$$\frac{\text{Weight of sediment} \times 10^6}{\text{Weight of water} - \text{sediment mixture}}$$

A linear conversion automatically takes place within the SLEDS software to report data results in mg/L and, at higher limits, also in ppm.

Login of Samples

Samples received at the laboratory are separated into two groups on the basis of the information received on the field log form (fig. 2). Samples are grouped by concentration analysis and sand/fine analysis.

Concentrations

The procedures followed for concentration determination are listed in this section. These procedures focus on quality control (QC) and include specifics not described in Guy (1969).

Two ASTM D3977-97 (2002) methods used for the analysis of suspended-sediment concentrations are the evaporation method and the filtration method.

The evaporation method is used when the concentration of the water-sediment mixture exceeds 10,000 mg/L of sediment that is mostly sand and 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 concentration may be required. A dissolved-solids correction needs to be applied when the concentration is greater than 2,300 mg/L, and the concentration of sediment is less than 200 mg/L (Guy 1969).

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.

Login of Samples for Concentration Analysis

Upon receipt in the laboratory, samples and Field Forms are examined for sample condition and requested analysis type. When the laboratory receives the samples, the samples are checked for damage during transport, damage is noted on the form, and the collector is notified if damage occurred. The forms are used by the laboratory to verify that all samples have been received. The samples are then sorted by station and arranged chronologically so that the sample with the oldest date is the first to be analyzed. They are then logged into the sample logbook.

The login procedures described are used in conjunction with the Sediment Laboratory Environmental Data System (SLEDS), the INGRES-based sediment program supported by the Office of Surface Water (OSW). Documentation for this program is maintained at the Cascades Volcano Observatory (CVO) laboratory in Vancouver, Washington.

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. The samples are logged in chronologically, when possible. Login of a concentration sample consists of entering the station number and name for the sample, the sample field label information, measurement

of laboratory specific conductance if requested by the customer, 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 database tables. Login information is printed and compared with information on the Field Forms. Mislabeled or missing samples are noted on the Field Forms. The collector is contacted if sample information cannot be resolved in the laboratory.

Five percent of the samples analyzed are QC sample blanks. The laboratory chief is responsible for providing sample blanks. Results of the sample blanks provide quantitative information about computer-program performance and efficiency.

A login summary report is printed after the login portion of the analysis is complete. 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.

Specific conductance is measured for all concentration samples so that a determination of dissolved solids can be applied if required by a particular sample. The methods and their quality-control measures are outlined below.

Data Entry of Concentration Analyses

Login inputs sample information from the Field Form and sediment-sample bottle label. Procedures during login include the following:

- Bottles are checked with corresponding bottle data on the Field Form (fig. 2).
- Bottles are assigned a unique lab number that will be kept throughout the analysis procedure. When a collector provides a sample record number, that record number will be recorded in the sample remarks when the sample is logged in.
- Information from the sediment-sample bottle label (fig. 1) is checked against the information on the Field Form and the bottle tare weight is entered into the computer; by pressing the print key on the scale, the weight is electronically transferred into the SLEDS screen. 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.
- Login summary reports are printed and reviewed by the laboratory technician before proceeding.

Sample bottles are moved to the decanting table to allow sediment to settle. Samples remain on the decanting table for 1 week before proceeding with the decanting process. Decanting is delayed longer if the samples appear cloudy, and an extended settling period may be considered. Some samples may never be decanted if the native water remains turbid. Samples may be processed before the 1-week period, but the whole sample would be processed.

If the samples are left for more than 2 weeks, they are placed in an unlit storage closet or covered with an opaque blanket to prevent algae growth.

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

- Crucibles and evaporating dishes are numbered with unique identification numbers for use in the laboratory.
- Crucibles are used in the filtration method.
- Evaporating dishes may be used with samples having net weights greater than 0.1 g.
- 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 print key on the scale, the weight is electronically transferred into the SLEDS screen.
- Crucible or dish weights and container identification numbers must be tared and available in SLEDS for all samples before proceeding to sample processing.
- A worksheet of the dish file report is printed for use during analysis of the samples.
- During final analysis of data entry, the containers with the dried sediment are weighed. They are removed from the desiccator and placed on a stable table. 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 to the balance by the use of tongs or cotton or nitrile gloves or rubber finger guards to avoid contamination by moisture, dirt or oil.

Quality-Control Measures for Data Entry of Concentrations

- Field Forms (fig. 2) are compared with data entered into the computer to confirm sample documentation.
- The analytical balance is connected to the computer, and the data are transferred electronically. The 0.0-g empty weight display is checked between each weighing.
- The weight displayed on the balance is compared with the weight shown on the computer screen.

- Tongs, cotton or nitrile gloves, or rubber finger guards are used to handle any glassware.
- Desiccator door is tightly closed between the removing of trays.
- 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 requires supplies and equipment, the determination of sampling method (filtration or evaporation), calculation of dissolved-solids corrections, and QC measures. Equipment and methods are described in Guy (1969), with the exception of QC measures. The following list highlights the key components of each category for the analysis of samples for concentration.

Supplies and Equipment

- Coors porcelain or Pyrex glass crucibles
- 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 1.5 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 noted and written on the lab analysis form. All sediment is washed from the sample container with DI 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 completely before obtaining gross weights.

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 trays. 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. The dish-identification number is checked and written on the lab analysis form. All sediment is washed from the bottle into the dish by using DI 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. To avoid any loss of sediment from boiling, the dishes are dried at 85° to 95°C until all visible water has evaporated. With the removal of all visible water, samples are dried for an additional 3 hours at 103°C, ±2°C, or overnight. Containers are then cooled completely in a desiccator, at least 3 hours, before obtaining gross weights.

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 water 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. The gross weight of the dish and solid residue is calculated and recorded. The net weight of the residue is calculated and recorded. The dissolved-solids correction (C) is computed as follows:

$$C = \frac{\text{Net weight of residue} \times \text{sample volume (after decanting)}}{25 \text{ mL (pipet volume)}}$$

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 are replicated when drying and desiccating samples.
- Label information, bottle tare weight, and crucible identification are compared with corresponding data on the worksheet for accuracy.
- Precalculated system checks and blanks 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 0.062-mm mesh sieve, and sand fraction refers to particles large enough to be retained on such a sieve.

Data Entry for Sand/Fine Analyses

The SLEDS software program is used to store and compute the data and maintain a record of analytical status of samples.

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

- The Field Form is compared with sample data entered into 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.
- The weight displayed on the balance is compared with the weight shown on the computer screen.
- Tongs, cotton or nitrile gloves, or rubber finger grips are used when handling glassware.
- Desiccator door is tightly closed between removal of each tray of evaporating dishes or crucibles.

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 QC measures. SLEDS 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 at the CVO.

Supplies and Equipment

- A 0.062-mm mesh sieve
- Coors porcelain or glass crucibles
- 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

ASTM D3977-97 (2002) Method C, wet-sieving filtration, is used to separate sand and coarser material from finer material.

Thoroughly wet the surface of the sieve and place in a Pyrex evaporating dish.

The sample is washed onto the sieve with DI water. The screen is rinsed with a gentle stream of water (gravity flow) to wash the particles through and into the assigned dish. A hand oscillator can be used to help with the separation process.

The sample may require more than one rinse. The screen stays immersed in DI water during the rinsing procedure. The sample is rinsed until no visible fines pass through the screen.

The sieve is then rinsed with DI water on the sides and underneath to remove any fine particles that may have adhered to the sieve.

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 SLEDS 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 thoroughly checked 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 percent of the samples analyzed by the laboratory will be QA samples inserted by the analyst or the laboratory chief. These samples consist of blank samples. Blank samples are prepared from DI water and will be distributed throughout the set of concentration-analysis samples. These samples will be clearly marked QC samples. Analyses from the QC samples are reviewed by the laboratory chief; corrective actions are taken as required. The notes of these corrective actions are retained in the QC logbook.

The USGS New Mexico Water Science Center 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 Field Quality Assurance Program (NFQA), Branch of Quality Systems. (See USGS OSW Technical Memorandum no. 98.05, dated March 2, 1998, A National Quality Assurance Program for Sediment Laboratories Operated or Used by the Water Resources Division, online at *http://water.usgs.gov/admin/memo/SW/sw98.05.html*, accessed June 13, 2005.)

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. The hardcopy of sample results is kept on file in the laboratory.

Data Procedures

The results of the analyses are reported on computer-generated forms and are reviewed for errors before submitting them to the laboratory chief. The remarks section includes comments concerning the sample, such as the presence of algae, unusual quantities of sand and sediment, or unidentified material observed in the samples.

Results of the QC samples are supplied to the customers annually. If problems arise with regard to sample transfer, copies of the field log forms are retained until the analyses are complete. The laboratory chief is notified as soon as a problem is identified. All problems are resolved and documented before results of analyses are marked for completion.

Computer Procedures

Documentation for the SLEDS software is available online at the USGS internal Web site: *http://eris.wr.usgs.gov/SedLab/framework.html*. Only the system administrator, 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 final analysis of the program.

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:

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.

Each laboratory employee is provided with a copy of the "USGS New Mexico Water Science Center Sediment Laboratory Student/Employee Guidebook."

Each employee reads this QA plan to help provide a general understanding of the laboratory operations.

Each employee determines concentrations for 2 to 4 weeks under the supervision of the laboratory chief.

The work of the employee is reviewed carefully by the laboratory chief.

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 Data Chief, supervisor.

Equipment Checks

A logbook is maintained for each balance, meter, and oven.

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. 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 (1992).

2. A QA/QC manual specific to the USGS New Mexico Water Science Center 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 and analysis of QA/QC samples; necessary corrective actions would be logged if needed.

4. This 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 backup measures are used. Hard copies are maintained in the laboratory for three complete water 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 reenter the files into the computer system, if necessary. The computer files are archived every other night. Only the 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 USGS New Mexico Water Science Center Safety Coordinator. They each receive a copy of the USGS New Mexico Water Science Center "Emergency Procedures" handbook for reference, as needed. Laboratory safety and hazard-communication training is provided to all laboratory employees.

The laboratory chief instructs laboratory personnel in the proper handling and storage of all glassware, containers, and laboratory equipment. Safety glasses, ear protection, nitrile 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 bottles must take place under the fume hood. The safety-related procedures are explained in the "Chemical Hygiene Plan of the USGS New Mexico Water Science Center 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 USGS are provided in USGS Techniques of Water-Resources Investigations publications, in internal technical memoranda of the USGS Water Resources Discipline, the Discipline's Office of Surface Water and Office of Water Quality, and other publications. Reference is made to the following publications.

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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.

A

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).

B

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, 1992).

P

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

Q

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).

S

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.

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.

T

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