QUALITY ASSURANCE FOR LABORATORY ANALYSIS OF SUSPENDED-SEDIMENT SAMPLES BY THE U.S. GEOLOGICAL SURVEY IN MONTANA

By John H. Lambing and Kent A. Dodge

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CONVERSION FACTORS AND ABBREVIATED WATER-QUALITY UNITS

Multiply	By	To obtain
centimeter (cm)	0.3937	inch (in.)
gram (g)	0.03527	ounce
gram per cubic centimeter	0.03613	pound per cubic inch
kilogram per square centimeter (kg/cm^2)	28.96	inch of mercury
liter (L)	0.2642	gallon (gal)
liter per minute (L/min)	0.2642	gallon per minute
milliliter (mL)	0.06102	cubic inch
millimeter (mm)	0.03937	inch (in.)
Watt (W)	0.001340	horsepower (hp)

Temperature can be converted from degrees Celsius (°C) to degrees Fahrenheit (°F) by the equation:

$$°F = 9/5\ (°C) + 32$$

Abbreviated water-quality units used in this report:

µS/cm	microsiemens per centimeter at 25 °C
mg/L	milligrams per liter
ppm	parts per million

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ABSTRACT

This report describes the policies and procedures used by the Montana District sediment laboratory of the U.S. Geological Survey to assure the quality of suspended-sediment data. Suspended-sediment concentration and percentage of sediment finer than 0.062 millimeter (sand-silt separation) for stream samples are routinely analyzed by standard methods. Quality-control practices and documentation are used to identify and minimize errors for the various types of equipment and procedures used for sample processing and reporting of analytical results. Data are systematically evaluated by technical review and verification prior to final approval and release of suspended-sediment data.

INTRODUCTION

Suspended-sediment samples are routinely collected from streams and analyzed for concentration and particle-size distribution by the U.S. Geological Survey (USGS) in response to various data-collection objectives. The procedures used to collect these samples are described in reports by Guy and Norman (1970) and Edwards and Glysson (1988). Laboratory theory and methods of sediment analysis are described by Guy (1969); although that report is used as a primary guide to standard analytical procedures, it does not specifically address quality assurance.

Quality-assurance practices used by sediment laboratories of the USGS depend on the size of the sediment program, the objectives of data collection, and the methods used by individual laboratories. Because of the recent emphasis on verification of quality in all aspects of data collection and analysis, the Montana District is formally documenting the quality-assurance plan for operation of its sediment laboratory. This report describes the policies and procedures used to assure the quality of results from the analysis of about 7,000 suspended-sediment samples annually by the Montana District sediment laboratory in Helena. Quality assurance is described for laboratory equipment and supplies, sample management, sample analysis, and data management. The quality-control practices used to identify and minimize errors are described for the various equipment and procedures used for sample processing and the reporting of analytical results. The procedures may be applicable to other laboratories having production capabilities similar to those of the Montana sediment laboratory.

QUALITY ASSURANCE PROGRAM

Quality assurance is defined as a comprehensive plan of operation that specifies the measures used to produce data of known quality. These measures include quality-control procedures that are systematically applied to identify, quantify, and control errors in order to evaluate the accuracy and precision of the analytical data.

The assurance of data quality on a continuing basis is achieved by a management program that includes developing an operational design, acquiring necessary equipment and expertise to perform laboratory operations, using standard USGS analytical methods, and implementing quality-control procedures to systematically review and evaluate the analytical results. This management program is implemented by assigning responsibilities for the various components of laboratory operations to qualified personnel who have received adequate training. Data quality is verified by documenting the results of numerous quality-control checks integrated throughout all phases of sample processing.

The Montana District supports quality-assurance objectives by designating discipline specialists who provide technical oversight to operations within their respective areas of expertise (Moreland, 1991, p. 12). This staffing organization includes a Sediment Specialist, who is responsible for developing sediment programs and maintaining the technical adequacy of laboratory operations. Operational guidelines adapted from formal publications and internal agency technical memoranda are applied in accordance with sediment policies of the USGS.

Within the Montana sediment laboratory, the quality-control system contains operational checks and documentation that enable the accuracy and precision of the data to be evaluated. Quality-control practices are implemented systematically throughout the operational sequence required to process suspended-sediment samples and report data. Quality control for equipment and procedures includes actions ranging from equipment checks and simple visual observation to systematic, documented test results. Unacceptable errors that are identified through systematic checks are promptly evaluated for corrective action. If the corrective action requires a change in procedure, the operational guidelines are changed accordingly. Procedural changes that implement non-standard methods require approval from the USGS Office of Surface Water. Quality control, therefore, incorporates both standard guidelines and appropriate modifications to meet the objectives of maintenance and improvement of data quality.

Personnel Responsibilities

All personnel involved in sediment operations are responsible for quality assurance within the scope of their duties. The Sediment Specialist is responsible for the overall evaluation and approval of the quality of sediment data within the Montana District. Implementation of any procedural change requires approval by the Sediment Specialist. The Sediment Specialist is assisted by a senior hydrologic technician, who supervises daily laboratory operations, establishes analytical priorities, and reviews all analytical results and quality-control data generated by the laboratory. The laboratory supervisor consults with the Sediment Specialist to resolve problems identified by quality-control checks. Sample management, analysis, and data entry are performed by one or two hydrologic technicians or their assistants. These technicians also check equipment to verify acceptable operation.

Training

Implementation of quality assurance begins with adequate training of laboratory personnel to develop necessary expertise. The training required to accurately and efficiently analyze sediment in the laboratory is provided by qualified staff having previous experience and by formal USGS training courses. Laboratory manuals detailing procedures, equipment operation, and data-documentation requirements are provided to assist laboratory analysts. Supervision of new analysts is provided for every phase of sample processing and data entry until the analyst has demonstrated the ability to independently and correctly perform all procedures. The USGS Office of Surface Water also requires that each USGS sediment laboratory have at least one analyst certified by proficiency training to perform analyses to agency standards. Proficiency training is provided by the Office of Surface Water.

Documentation Requirements

All analytical quality-control measurements are documented in laboratory "quality-control logbooks" for each major component of sediment analysis. In addition, data that document the performance of analytical equipment are entered routinely into the logbooks. Records of equipment calibration, cleaning, and adjustments are entered in the logbooks at the time of servicing.

The information used in the analytical processing of suspended-sediment samples is recorded on several forms. Some forms are used to enter data manually, whereas others are computer-generated. The forms are designed to allow systematic entry of all pertinent data necessary to analyze, track, and report sample data. All final forms are stored in station files as a permanent record of data processing.

All quality-control records are periodically reviewed by the laboratory supervisor. These reviews are performed to identify subtle, but systematic, errors that might not be detected by casual observation. Persistent deviations of quality-control data from original characteristics are investigated to identify the possible source of variation. The trends are brought to the attention of the Sediment Specialist and tests are designed to isolate the source of error. Evaluations of test data are used to assess the need for adjustment or servicing of equipment, or modification of analytical procedures.

Laboratory Safety

Instructions and safety precautions for a wide range of activities that occur in the District water-quality laboratory, of which the sediment laboratory is a part, are described generally in a laboratory safety handbook by Skinner and others (1983) and specifically in the Montana District Chemical Hygiene Plan. These manuals describe various laboratory procedures, hazards, safety precautions, and chemical storage requirements. All personnel who routinely or periodically work in the laboratory are required to read and sign the handbook, which is prominently displayed in the laboratory. Laboratory restrictions and emergency procedures are identified on signs placed on walls. An emergency safety shower, eyewash fountain, and acid-spill cleanup kit are also located in the laboratory. Personal-safety apparatus such as rubber gloves, goggles, aprons, and a chemical-fume hood are provided. Operation of the shower, eyewash fountain, and hood exhaust fans are checked periodically and inspections are documented on safety inspection sheets. Two fire extinguishers, mounted on the walls, are checked regularly by building inspectors of the General Services Administration. All chemicals are stored in metal cabinets, clearly marked with identifying labels, and dated. A list of chemicals currently in stock is stored in a fireproof cabinet and is updated annually. A listing of Material Safety Data Sheets is located at the entrance to the laboratory and is readily accessible to all laboratory personnel.

QUALITY ASSURANCE OF LABORATORY EQUIPMENT AND SUPPLIES

The equipment and supplies used in the sediment laboratory must meet operational specifications and perform reliably to allow for accurate and efficient sample processing. The equipment and supplies used by the Montana sediment laboratory are described in the following sections, along with associated quality-control procedures used to maintain and evaluate performance.

Laboratory Equipment

The weighing, processing, and analysis of sediment samples require a wide variety of equipment. Electronic balances, ovens, conductivity meters, vacuum pumps, and laboratory-grade thermometers are obtained from commercial sources. More specialized equipment, such as decanting or filtering devices, generally are assembled or fabricated to conform to processing requirements of individual laboratories. Storage shelves and processing benches might also require custom fabrication. A computer system for data entry and storage is preferable for data management and retrieval, especially for large numbers of analyses and maintenance of historic data files.

Description and Use of Equipment

The ability of commercially available equipment to meet the technical requirements of sample processing is evaluated prior to purchase. This equipment is tested and calibrated before its initial use to ensure proper performance. Manuals describing equipment operation and addresses of manufacturer representatives are provided for each set of commercial equipment. Specially fabricated equipment is designed and tested for technical adequacy prior to utilization in sample processing.

Electronic balances

Two types of balances, macro and analytical, are used to determine the mass of sediment samples and containers. (To conform to traditional usage, the word "weight" is subsequently used in place of the technically correct term "mass.") The balances are electronic and provide digital readout. The balance specifications encompass the expected range of weight with an acceptable level of accuracy and precision. Weight is displayed in grams.

The macro balance is used to weigh items of 100 g or more. Such items typically include sample bottles (tare weight) and the sample (water-sediment mixture). The macro balance used in Montana is capable of determining weight ranging from 0.1 to 2,000 g, with a precision of ± 0.05 g.

The analytical balance is used to weigh items of 100 g or less. These items typically include sediment containers (crucibles and evaporation dishes) used in laboratory analysis of sediment weight. The analytical balance used in Montana is capable of determining weight ranging from 0.0001 to 162 g, with a precision of ± 0.0001 g. This balance is equipped with an internal counterweight balance for automatic calibration. The analytical balance is electronically linked to the laboratory computer terminal to enable automatic transfer of balance readings into computer storage, thus eliminating keyboard transcription errors (fig. 1).

Figure 1.--Analytical balance electronically connected to laboratory computer terminal.

Each balance is mounted on a sturdy table to minimize vibrations that could affect the weight readings. This mounting procedure is especially important for the very sensitive analytical balance. In Montana, the analytical balance is mounted on a sand-filled, marble-top table. This balance has a protective hood with sliding plexiglass panels surrounding the balance pan to provide a dust-free environment and to eliminate air currents during the weighing process. Balance doors are always closed before recording a weight. After a container is placed on the pan, a green light on the balance control panel is illuminated until vibrational stability is achieved. Weights are recorded only after the green light goes out. Balances also need to be located in an area of minimal vibration (restricted foot traffic) and where humidity and temperature are not subject to excessive change.

Ovens

Two electric ovens are used to dry the sediment, the sediment containers, and various types of laboratory apparatus. Both ovens are equipped with internal temperature sensors for monitoring oven performance.

The convection oven is used to dry crucibles containing filtered sediment. Because temperatures in excess of about 110 °C may break down molecular hydration bonds of some clay minerals, the convection oven is operated at a maximum temperature of 105 °C. This oven is equipped with a simple on/off switch and contains a laboratory-grade thermometer mounted in the roof of the oven for external monitoring of temperature. The capacity of this oven is about 150 crucibles.

The mechanical-convection oven is controlled with a timer, an alarm system, and air-circulation blowers. This oven, which is used to dry sediment in evaporation dishes, is operated at about 92 °C. Because evaporation dishes may contain 100-250 mL of sample, temperatures must be maintained at less than the boiling point of water (96 °C at about 4,000 ft altitude) to prevent splattering of samples and resultant loss of sediment or cross-contamination of samples. The oven alarm system sounds when a pre-set temperature threshold is reached, thereby indicating a temperature approaching the boiling point. The threshold temperature is typically set at 95 °C. The capacity of this oven is about 100 dishes (fig. 2).

Figure 2.--Mechanical-convection laboratory oven for drying sediment in evaporation dishes.

Conductivity meters

A laboratory-grade conductivity meter is used to measure specific conductance of suspended-sediment samples in order to estimate the concentration of dissolved solids within the water phase of the water-sediment mixture. This measurement is necessary to correct for the additional weight that is not part of the particulate material originally suspended in the stream. Specific conductance is measured in samples that are processed by evaporation (rather than filtration) where the solid-phase residue contains precipitated salts. The meter is also used in general laboratory practice to verify the purity of the deionized water supply that is used for various water-quality activities. The electronic conductivity meter in the Montana sediment laboratory is capable of measuring specific conductance ranging from 1 to 100,000 µS/cm.

Decanting equipment

Decanting equipment is used to remove most of the overlying clear water of the water-sediment mixture after settling, without removing any of the settled sediment. The apparatus consists of a J-shaped nozzle made of a firm material such as copper inserted into flexible tubing and connected to a vacuum system. The hose system is suspended above a workbench on which the sediment samples are placed for processing (fig. 3). The decanted water moves through outflow tubing into a waste-water jug having a capacity of about 38 L (10 gal). The vacuum pump used in this system is operated by a 124 W (1/6 hp) motor.

Figure 3.--Vacuum hose system and workbench for decanting water from suspended-sediment samples.

Filtering equipment

Filtering equipment is used to draw a sediment sample through crucibles having perforated bottoms. A glass-fiber filter is placed on the bottom of the crucible to allow the passage of water but not sediment. The crucibles are seated in rubber holders mounted in a fabricated crucible manifold (fig. 4). The bottom of each holder is connected by a portal tube to the vacuum system described previously. Each portal has a separate valve to control the in-line vacuum to individual crucibles. Sample water vacuumed through crucibles moves through outflow tubing into the same waste-water jug used for decanting the supernatant. A separate manifold is used for each of two sizes of crucibles used in the Montana sediment laboratory.

Calibration and Maintenance of Equipment

Equipment that operates properly and within specified limits of calibration is essential for producing reliable analytical results. Equipment is systematically checked using reference standards to verify acceptable accuracy. Equipment that fails to meet accuracy standards is either calibrated or promptly serviced to achieve conformance with standards. Calibration of equipment is performed according to guidelines of the manufacturer and the USGS. Maintenance of equipment to assure proper operation is systematically performed by adhering to maintenance schedules recommended by manufacturers.

Figure 4.--Manifold for vacuum filtration of suspended-sediment samples through crucibles.

All equipment checks, calibration, cleaning, and maintenance are documented in appropriate logbooks as part of routine quality control. A logbook is kept for performance checks and calibration measurements for each electronic balance, oven and oven thermometer, and conductivity meter. The date and analyst's initials are recorded for each log entry, followed by the performance data and comments for each item checked. The logbooks are periodically reviewed to evaluate equipment performance and to implement corrective actions, if necessary. In addition, manufacturer's operation manuals, maintenance schedules, and service forms provided by professional service representatives are stored in equipment files for each respective instrument. These documents are stored at each instrument's location for immediate access.

Electronic balances

The macro and analytical balances are calibrated each day prior to use by automatic internal balance controls. The calibration procedure is verified by weighing a series of analytical reference weights recommended by the balance manufacturer. This check provides an independent test of the internal counterweight balance used for automatic calibration. For each daily calibration check, the operator uses at least three weights that encompass the extremes and midpoint of the expected range. All weight measurements are recorded in a logbook for each balance. If errors in the weight reading of the balance exceed prescribed error limits, sensitivity adjustments outlined in the manufacturer's equipment manual are performed. If these adjustments fail to correct the error, the balance is internally cleaned and calibrated by the manufacturer's service representative. Regardless of balance performance, both balances are cleaned annually and then calibrated with American Society for Testing and Materials Class 1 weights by a service representative. The date and type of service performed are documented in the logbook for each balance.

During sample processing, balance readings may drift slightly as a result of sensitivity to humidity and temperature changes, vibrations, or static electricity. The analyst is responsible for closely monitoring any tendency for the balance to drift from a zero reading when the pan is empty. To ensure that particulate material is not causing drift, the pan is cleaned periodically with a fine-hair brush. To minimize the effect of persistent drift, the balance is re-zeroed by a reset button on the balance control panel after every 10 weighings, or more frequently if drift exceeds 0.0002 g. Drifting of more than 0.0004 g may indicate buildup of static electricity or moisture absorption from the air. Static electricity in the typically dry environment of Montana is controlled by placement of the sediment container on a wooden platform prior to placement in the balance. A plastic platform on the balance pan also aids in minimizing static drift. Moisture absorption is minimized by weighing containers promptly after removal from desiccators. If the drift becomes excessive (greater than about 0.0005 g), the balance is internally recalibrated. If the drift cannot be corrected, the balance is serviced by the manufacturer's representative.

Ovens

Oven temperature is read twice during each day of operation and recorded in the logbook for each oven. Oven thermometers are checked twice annually at their normal operating temperature (90-105 °C), with results recorded in the logbook for each oven. The mercury-bulb thermometer of the crucible oven is checked by replacing it with a 76-mm immersion calibration thermometer of known accuracy (plus or minus 0.1 °C). The readings are compared and recorded. The digital thermometer of the evaporation oven is checked by placing a full-immersion calibration thermometer on the center drying shelf. The results of these tests are recorded in the oven logbooks. Thermometers are replaced if temperature comparisons vary by plus or minus 2.0 °C.

Conductivity meters

Conductivity meters are checked for calibration each day of use with standard solutions of known specific conductance. Two standards are used to encompass the expected range of sample specific conductance. Linearity of slope is checked at least weekly with three standards. The meter is recalibrated by adjustment dials if more than a 3 percent difference exists between the known value of the standard solution and the displayed value of the meter. If calibration does not bring the displayed value within an acceptable error range, the sensor is cleaned and the meter is checked with another standard solution of equal or similar specific conductance to verify the exceedance of calibration limits. If the meter is in error, the sensor is replaced and the meter is re-tested. All standard solutions used for calibration checks have a clearly marked expiration date that indicates freshness of the solution. Standard solutions are discarded at the prescribed expiration date.

Laboratory Supplies

A variety of laboratory supplies is needed to process and analyze suspended-sediment samples. Supplies that are selected must meet technical and durability requirements of performance. Some of the items are disposable and purchased on a regular basis. Ordering a sufficient supply of these items by anticipating depletion rates ensures an adequate stock for uninterrupted analytical work. Other items are reusable, and therefore can be cleaned multiple times without significantly degrading their original characteristics. Cleaning procedures for all types of sediment containers used in Montana are documented in the Montana District Laboratory Safety Handbook and are posted at the primary cleaning station.

Sample Bottles

Standard suspended-sediment samplers used by the USGS accommodate different sizes and types of sample bottles (Edwards and Glysson, 1988, p. 19). The most common types of sample bottles used in Montana are the pint glass "milk" bottle and the quart glass "mayonnaise" bottle.

The glass sample bottles are reusable and are cleaned prior to collection of a water sample. The cleaning removes any residue that may remain after the analytical process. Sample bottles are cleaned by soaking in a water bath with a laboratory detergent, motorized brushing of internal and external surfaces, and rinsing with tap water. Washed bottles are turned upside down in their wire carrying cases to air dry. After drying, the bottles are turned upright and capped, and are then ready for field use. At this time, the bottles also are inspected for chips, cracks, or excessive residue. Bottles with large chips or cracks are discarded to prevent breakage in the field. Bottles with visible residue or staining that requires additional cleaning are rinsed with a 10-percent solution of hydrochloric acid, using appropriate safety apparatus such as goggles and protective gloves. After acid rinsing, the bottles are rewashed and rinsed with tap water.

Glass sample bottles may be used for many years; therefore, bottle weights to the nearest 1 g (without labels, tags, or caps) are permanently etched into the surface as a record of tare weight. Bottles are regularly checked to verify the accuracy of the etched tare weight. Five percent of each batch of processed sample bottles is randomly selected and reweighed on the macro balance after cleaning and drying. If the measured weight of any bottle differs by more than 2 grams from the weight etched on the bottle, the new tare weight is etched on the bottle and the old tare weight is nullified. All bottles that require acid rinsing are weighed to verify the tare weight, with corrections made as necessary. If a chip is discovered on a bottle, the bottle is reweighed and the new tare weight is etched.

Crucibles and Filters

Crucibles are small porcelain or glass vessels that are thermally resistant and have a perforated base that allows the passage of water. Two sizes and types of crucibles are used by the Montana sediment laboratory. The porcelain crucibles have a base diameter of 2.4 cm and a capacity of about 25 mL. The porcelain crucibles have a base with an array of uniform perforations. The pyrex-type glass crucibles have a base diameter of 4.25 cm and a capacity of about 50 mL. The glass crucibles have a base composed of an integrated fritted-glass disk. All crucibles are individually numbered with high-temperature paint or indelible marker for identification.

Both types of crucibles are cleaned after completion of analysis by first removing the filter with forceps. The porcelain crucibles are soaked in a laboratory-grade detergent, lightly brushed, then rinsed with tap water. The glass crucibles with the fritted-glass dish in the base are soaked in warm tap water and lightly brushed to remove any residue. The glass crucibles are periodically cleaned with an ultrasound water bath to remove particles that may become lodged in the fritted-glass maze. If residues are not successfully removed by ultrasound, the crucibles are soaked in a 10-percent solution of hydrochloric acid.

Filters are seated on the bottom of the crucible to retain sediment during the filtration of samples. The only filter type authorized by the USGS for suspended-sediment analysis is the Whatman glass-microfiber filter No. 934-AH[1] or equivalent (C.W. Boning, U.S. Geological Survey, written commun., 1991). These filters do not have a specific pore diameter, but instead filter sediment by means of sample passage through a tortuous path with an estimated effective pore size of 0.0015 mm (H.E. Jobson, U.S. Geological Survey, written commun., 1992). Filters are always placed in crucibles with the rough side facing the sample to prevent fiber loss during filtration.

After the crucibles have been cleaned, new filters of the correct diameter are placed in the crucibles with forceps. The filters are pre-rinsed with about 25 mL of distilled water and placed under vacuum. The vacuum serves to seat the filters and helps remove loose fibers. The crucibles and filters are oven-dried for about 2 hours at 105 °C to remove any moisture and allowed to cool for about one hour.

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[1] The use of trade or product names is for identification purposes only and does not constitute endorsement by the U.S. Geological Survey.

Crucibles are then transferred to a desiccator to remain moisture- and dust-free until needed for future use. Because the weights of the individual filters placed in the crucibles may vary, a new tare weight for each crucible plus filter is determined for every analytical episode. Crucible tare weights are recorded to the nearest 0.0001 g.

Evaporation Dishes

Evaporation dishes are small bowls made of thermally resistant glass that have a funneled lip for pouring. Evaporation dishes used in the Montana sediment laboratory have capacities of either 140 or 290 mL. The dishes are individually numbered with high-temperature paint or indelible marker for identification.

Evaporation dishes are cleaned by soaking in warm tap water or a soap solution, lightly brushing to remove sediment, and rinsing with tap water. The dishes are then oven-dried for 2 hours at 105 °C. After cooling for about one hour, the dishes are transferred to desiccators.

Sieves

Sieves are shallow metal pans having a bottom consisting of a screen with mesh openings of a standardized diameter. Sieves are used to separate sediment on the basis of particle-size diameter. Sediment particles with a median axis larger than the mesh openings are retained on the screen, whereas smaller sediment particles will pass through the screen. A 3-in. diameter sieve with a 0.062 mm mesh is used by the Montana sediment laboratory (fig. 5) to separate sand from fine sediment (silt and clay).

Figure 5.--Suspended-sediment sample poured through sieve to separate sand and fine sediment.

The sieve is inspected for particles clogging the screen after each sample is processed. Rinse water is used to flush any remaining sediment from the screen to the final sample container to ensure that all sediment from a sample has been transferred. This rinse generally constitutes adequate cleaning prior to processing of the next sample. Occasionally a few particles may become lodged in the mesh openings of the screen and cannot be removed by rinsing. When particles become lodged in the mesh openings, the sieve is gently cleaned with a nylon brush or immersed in an ultrasound bath. The wire-cloth mesh is also inspected prior to each use to ensure that it is taut and has no tears. A sieve is replaced if it cannot be completely cleaned, is no longer taut, or has become torn.

Desiccators

Desiccators are used to store crucibles and evaporation dishes after they are removed from the drying oven to maintain a constant sample weight. Storage in desiccators prevents the absorption of moisture by filters, sediment, or container surfaces, and also prevents the deposition of airborne particulates.

Desiccation is achieved by a desiccant placed at the bottom of the desiccator. The desiccant is granular calcium sulfate, which has a large moisture-absorption capacity. Because the desiccants used contain a moisture indicator that changes color as moisture saturation is approached, desiccant color is checked prior to each use. If the desiccant color has changed, the desiccant is either replaced or recycled according to manufacturer's instructions. Recycling is accomplished by oven-baking the desiccant at a prescribed temperature to restore the original absorption capacity and color.

Desiccators are inspected prior to each use to ensure that the seal between the jar and lid is airtight. If a seal is not airtight, the old sealant grease is removed with a paper towel and new grease is applied. All desiccators are cleaned and re-greased annually. With the desiccant removed, a laboratory-grade detergent is used to clean the entire desiccator. New grease is applied to the jar rim and lid to maintain an airtight seal. The desiccant also is replaced annually when the desiccator is cleaned.

A logbook is maintained that lists all the desiccators and the dates of cleaning and replacement of desiccant. The date of the most recent cleaning and desiccant replacement is also marked on the outside of each desiccator for easy reference.

Distilled Water

Distilled water is used in sediment-laboratory operations for rinsing the sediment from sample bottles into crucibles and evaporation dishes, seating filters in crucibles, cleaning conductivity-meter sensors, rinsing sieves, and preparing test samples. An electric still is used which produces about 4 L of distilled water per hour from tapwater. The criterion for acceptable quality of distilled water in the Montana District is a specific conductance of 4 µS/cm or less. If this criterion is exceeded, the still is disassembled and cleaned according to manufacturer's instructions.

As part of the overall Montana District quality-assurance program for water-quality activities (Knapton and Nimick, 1991), distilled water is tested monthly for specific conductance. Results of specific-conductance tests are stored in quality-control files of the District Data Management Unit.

QUALITY ASSURANCE OF SAMPLE MANAGEMENT

Sample management includes the handling, treatment, and documentation that is required to manage sediment samples after collection and prepare them for laboratory analysis. The quality of sediment data is largely dependent on thorough documentation of sample identity for the purpose of tracking the sample from the time of collection through the analytical sequence. Sample integrity is maintained by strictly adhering to established procedures for sample processing. All pertinent sample information is documented on at least two independent forms to enable cross-checking of sample identity and field information.

Field Documentation

The documentation of samples begins at the field sampling site by clear and complete labeling of pertinent information on the sample-bottle tag promptly after sample collection. Such information includes the station name, station identification number, date and time of collection, gage height, water temperature, sampling method, sampling points of a multiple-bottle set, and collector's initials. The same information is entered on the standard water-quality field form (fig. 6) utilized by Montana District personnel. Observers who are contracted to collect samples for the Montana District also document sample-collection information onsite using a standard form (fig. 7). Supporting information regarding the sample collection and remarks on hydrologic or water-quality conditions are also reported on the field form.

Shipment and Storage

After sample collection, the tightly capped bottles are placed in secure carrying cases for shipment from the field to either a USGS office or the observer's residence. Samples are typically transported in the field vehicle used for sampling trips. During winter, samples that are transported in field vehicles are protected from freezing by means of vehicle heaters. For overnight storage in the field, bottles are either protected by electrical hookups to vehicle heaters or by transferring the bottles to a heated inside location. After transport from the field, samples are stored indoors--preferably in a cool, dark location--to prevent evaporation, freezing, or algae growth that could degrade sample integrity. Samples stored at an observer's residence are picked up by USGS personnel about every 6 weeks during routine site visits and taken to the field office for storage until samples are transported to the District Office in Helena. Personnel of the District Office return samples collected in their area of operation directly to the District Office upon completion of a field trip.

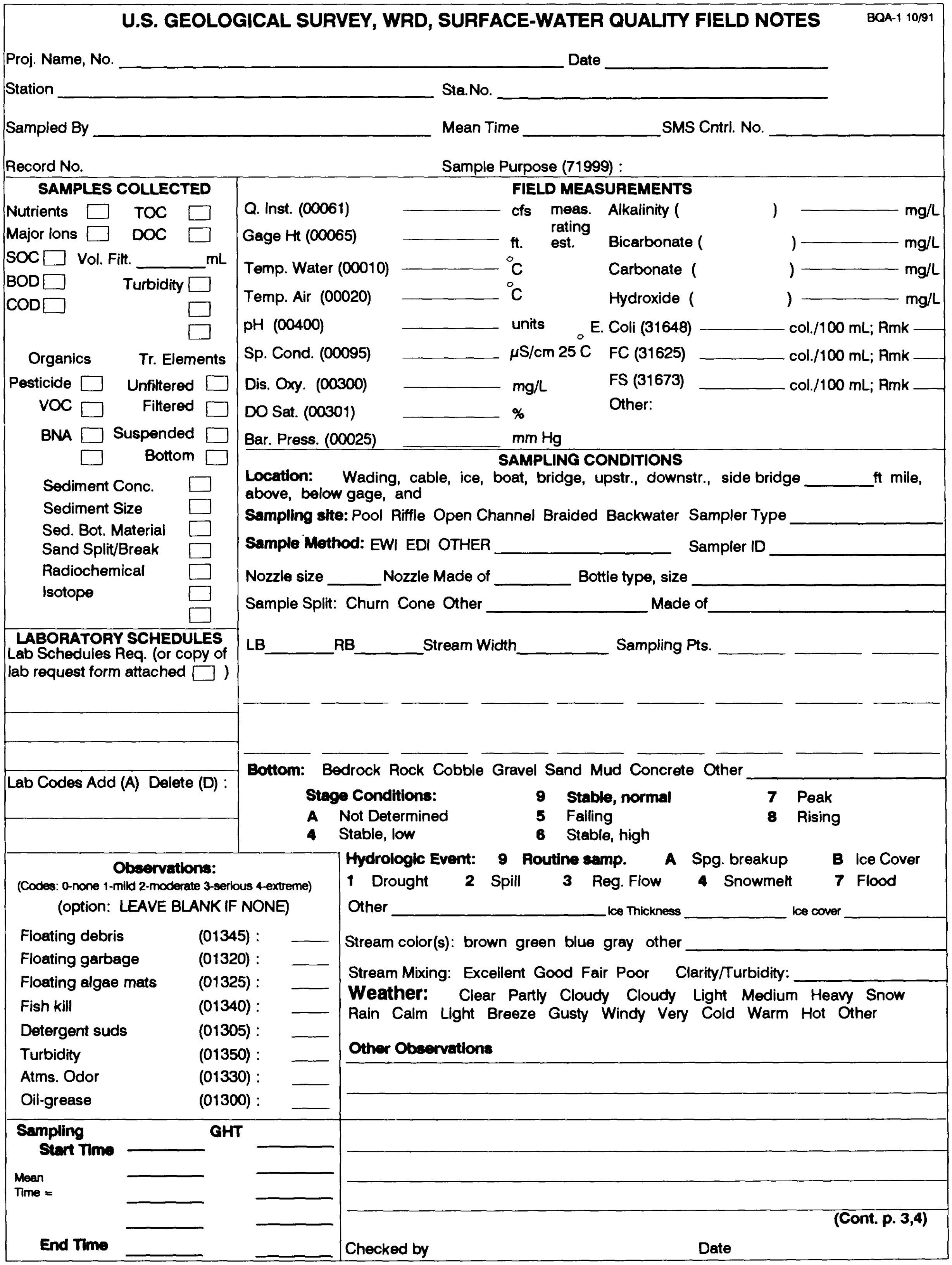
Sample Inventory

Upon arrival at the District Office, samples are immediately taken to a storage area for initial inventory. Samples are cross-checked with sample documentation on copies of field forms to assure that no bottles are missing. A filing system is maintained to document receipt of all samples. Bottles are inspected for breakage, loose caps, spillage, or missing labels. Samples that cannot be positively identified or whose integrity is known to be degraded are removed from the processing sequence, and the conditions are noted on the field and laboratory forms. To facilitate processing, the samples are sorted according to station and sample type and arranged in chronological order.

QUALITY ASSURANCE OF SAMPLE ANALYSIS

Sample analysis includes all the laboratory procedures used to document, prepare, and treat the samples during analytical processing. Also included are various quality-control tests that indicate the accuracy and precision of the analytical procedures.

Two types of sample analysis are performed by the Montana sediment laboratory--suspended-sediment concentration and particle-size distribution (fig. 8). Samples collected by observers from a single vertical in a stream on a daily or frequent basis typically are analyzed only for concentration. Samples collected periodically by USGS personnel from multiple verticals in a stream cross section are analyzed for both concentration and particle-size distribution.



U.S. GEOLOGICAL SURVEY, WRD, SURFACE-WATER QUALITY FIELD NOTES BQA-1 10/91

Proj. Name, No. ____________ Date ____________
Station ____________ Sta.No. ____________
Sampled By ____________ Mean Time ____________ SMS Cntrl. No. ____________
Record No. Sample Purpose (71999) :

SAMPLES COLLECTED

Nutrients ☐ TOC ☐
Major Ions ☐ DOC ☐
SOC ☐ Vol. Filt. ______ mL
BOD ☐ Turbidity ☐
COD ☐ ☐
☐

Organics Tr. Elements
Pesticide ☐ Unfiltered ☐
VOC ☐ Filtered ☐
BNA ☐ Suspended ☐
☐ Bottom ☐

Sediment Conc. ☐
Sediment Size ☐
Sed. Bot. Material ☐
Sand Split/Break ☐
Radiochemical ☐
Isotope ☐
☐

LABORATORY SCHEDULES
Lab Schedules Req. (or copy of lab request form attached ☐)

Lab Codes Add (A) Delete (D) :

FIELD MEASUREMENTS

Q. Inst. (00061)	______	cfs meas.	Alkalinity ( )	______	mg/L
Gage Ht (00065)	______	ft. rating est.	Bicarbonate ( )	______	mg/L
Temp. Water (00010)	______	°C	Carbonate ( )	______	mg/L
Temp. Air (00020)	______	°C	Hydroxide ( )	______	mg/L
pH (00400)	______	units	E. Coli (31648)	______	col./100 mL; Rmk ____
Sp. Cond. (00095)	______	µS/cm 25 °C	FC (31625)	______	col./100 mL; Rmk ____
Dis. Oxy. (00300)	______	mg/L	FS (31673)	______	col./100 mL; Rmk ____
DO Sat. (00301)	______	%	Other:		
Bar. Press. (00025)	______	mm Hg			

SAMPLING CONDITIONS

Location: Wading, cable, ice, boat, bridge, upstr., downstr., side bridge ______ ft mile, above, below gage, and
Sampling site: Pool Riffle Open Channel Braided Backwater Sampler Type ____________
Sample Method: EWI EDI OTHER ____________ Sampler ID ____________
Nozzle size ______ Nozzle Made of ______ Bottle type, size ____________
Sample Split: Churn Cone Other ____________ Made of ____________
LB ______ RB ______ Stream Width ______ Sampling Pts. ______ ______ ______

Bottom: Bedrock Rock Cobble Gravel Sand Mud Concrete Other ____________

Stage Conditions:
A Not Determined
4 Stable, low
9 **Stable, normal**
5 Falling
6 Stable, high
7 Peak
8 Rising

Hydrologic Event: 9 **Routine samp.** A Spg. breakup B Ice Cover
1 Drought 2 Spill 3 Reg. Flow 4 Snowmelt 7 Flood
Other ____________ Ice Thickness ______ Ice cover ______

Stream color(s): brown green blue gray other ____________
Stream Mixing: Excellent Good Fair Poor Clarity/Turbidity: ____________
Weather: Clear Partly Cloudy Cloudy Light Medium Heavy Snow Rain Calm Light Breeze Gusty Windy Very Cold Warm Hot Other

Other Observations

(Cont. p. 3,4)

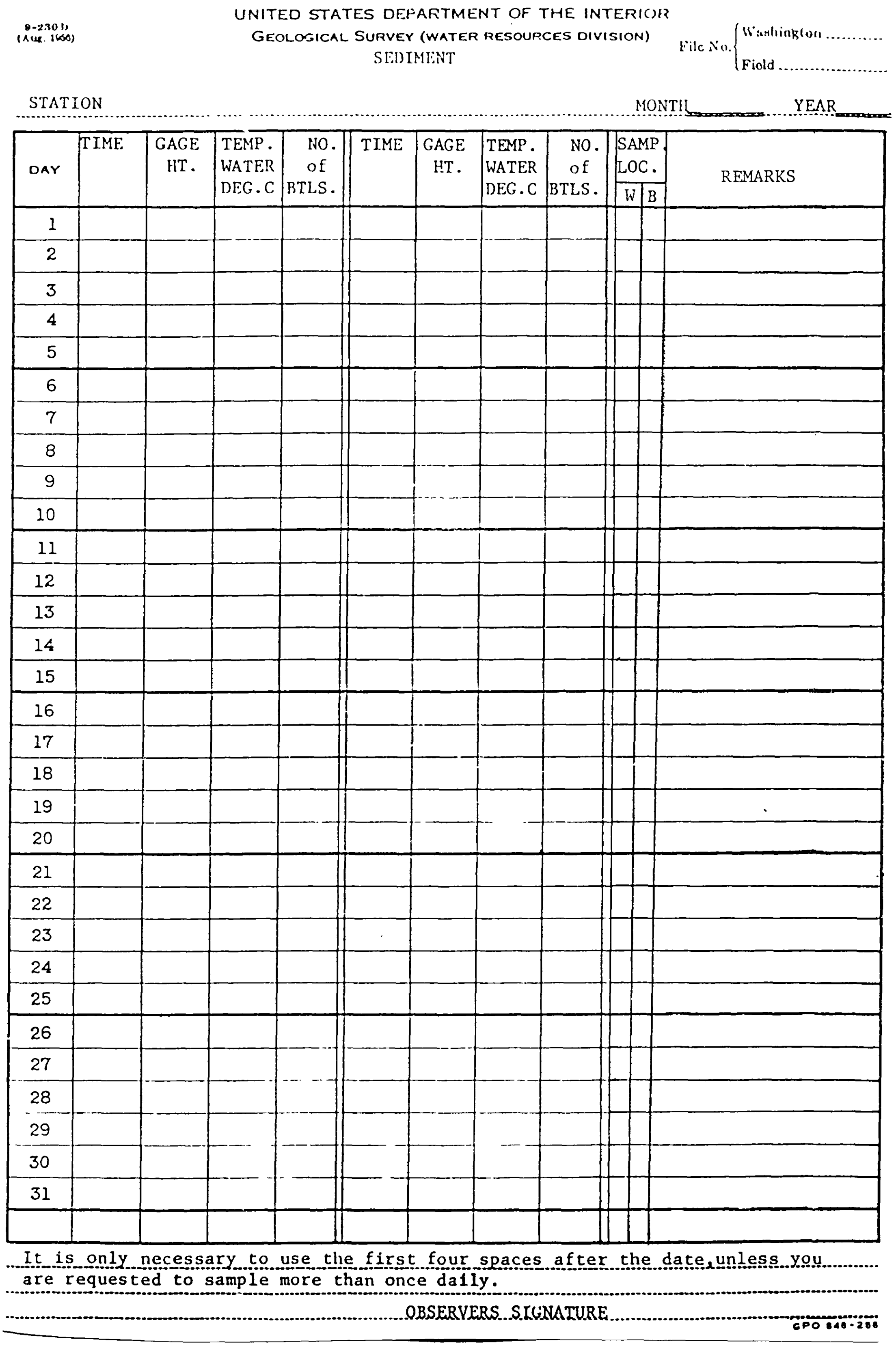
Checked by Date

Observations:
(Codes: 0-none 1-mild 2-moderate 3-serious 4-extreme)
(option: LEAVE BLANK IF NONE)

Floating debris	(01345) :	____
Floating garbage	(01320) :	____
Floating algae mats	(01325) :	____
Fish kill	(01340) :	____
Detergent suds	(01305) :	____
Turbidity	(01350) :	____
Atms. Odor	(01330) :	____
Oil-grease	(01300) :	____

Sampling Start Time	______	GHT ______
Mean Time =	______	______
	______	______
	______	______
End Time	______	______

Figure 6.--Example of field form for documentation of water-quality sample (reduced).



9-230 b
(Aug. 1966)

UNITED STATES DEPARTMENT OF THE INTERIOR
GEOLOGICAL SURVEY (WATER RESOURCES DIVISION)
SEDIMENT

File No. Washington ....
Field ....

STATION .... MONTH ________ YEAR ______

DAY	TIME	GAGE HT.	TEMP. WATER DEG.C	NO. of BTLS.	TIME	GAGE HT.	TEMP. WATER DEG.C	NO. of BTLS.	SAMP. LOC.		REMARKS
									W	B	
1											
2											
3											
4											
5											
6											
7											
8											
9											
10											
11											
12											
13											
14											
15											
16											
17											
18											
19											
20											
21											
22											
23											
24											
25											
26											
27											
28											
29											
30											
31											

It is only necessary to use the first four spaces after the date, unless you are requested to sample more than once daily.

.... OBSERVERS SIGNATURE ....

GPO 846-286

Figure 7.--Example of field form for documentation of observer sample (reduced).

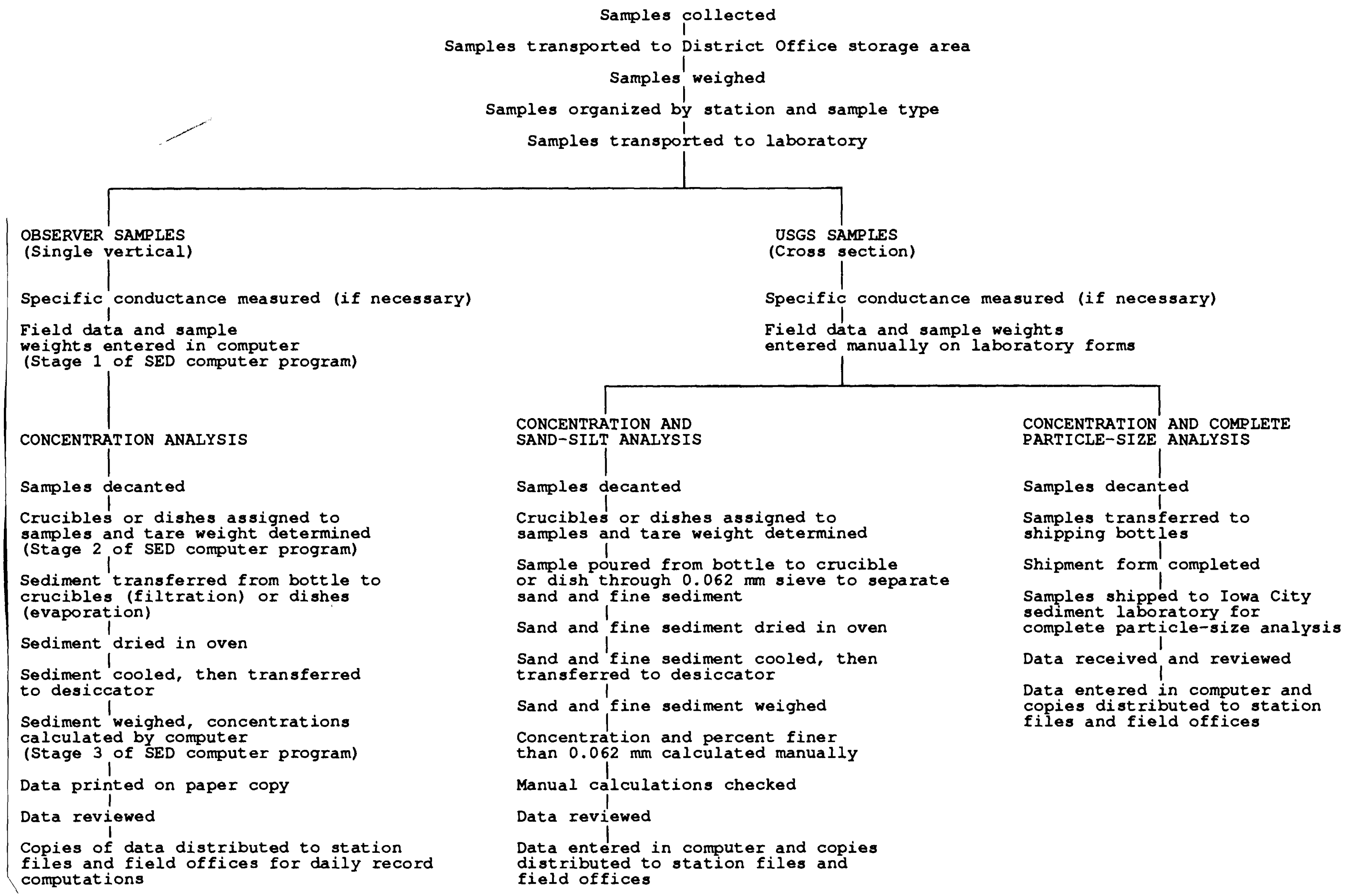


Figure 8.--Operational sequence for the analysis of suspended-sediment samples in the Montana District. [Abbreviations: mm, millimeters; USGS, U.S. Geological Survey]

Laboratory Procedures

A variety of laboratory procedures are used to analyze suspended-sediment samples depending on the study objective, type of sample, and condition of the sample. Sediment analysis consists largely of weight determinations made at various stages in a sequence of steps that separates the sediment from the original water-sediment mixture of the sample. The primary reference of standard methodology for sediment-laboratory operations is Guy (1969).

Sample Weight Determination

The initial step in the analytical sequence is weighing the sample prior to any physical laboratory treatment. Samples are weighed within 2 weeks of receipt at the District Office to minimize any potential evaporation that would decrease the weight of the water in the water-sediment mixture. This step is performed for all samples, regardless of sample type and subsequent analytical treatment.

Before samples are weighed on the calibrated macro balance, dirt or foreign material is wiped from the outside of the sample bottle. The first weight measurement recorded in the analytical sequence is the "sample gross weight," which includes the weight of the water-sediment mixture plus the weight of the glass bottle. To obtain this value, the combined weight of the attached bottle cap and bottle tag is subtracted from the total weight (the weight of the glass bottle is subtracted in a subsequent step). An average combined weight of 10 g for a bottle cap and tag, as determined by multiple weighings, is used as a tare correction. This tare correction is entered into the balance control panel and is automatically subtracted from the total weight of the sample. This eliminates the need to remove caps and tags from each bottle before weighing. The "sample gross weight" is recorded in bold notation on the sample bottle tag in a consistent location.

Sample Preparation

Following determination of the sample gross weight for each bottle, the samples are brought from the storage area to the laboratory for analytical preparation. At this step, the samples are visually inspected for sediment quantity to determine whether filtration or evaporation is required. The determination is made by an experienced analyst based on the depth of accumulated sediment in the bottom of the sample bottle and the likelihood of clogging a filter. If the analyst decides that the sample is to be evaporated, specific conductance is measured to provide an estimate of the mass of dissolved solids that will be retained as solid residue after evaporation. If a sufficient quantity of sediment is present in a sample for a site where complete-particle-size data is needed, it is sent to the USGS Iowa District sediment laboratory in Iowa City for a complete particle-size analysis.

A computer program (SED) developed by the Iowa District is used to enter, compute, and store sample information and analytical results for samples collected by observers at daily sediment stations. Documentation of the SED program is available in the Sediment Notes User Manual (S.C. Noble, U.S. Geological Survey, written commun., 1986) from the Iowa District Office.

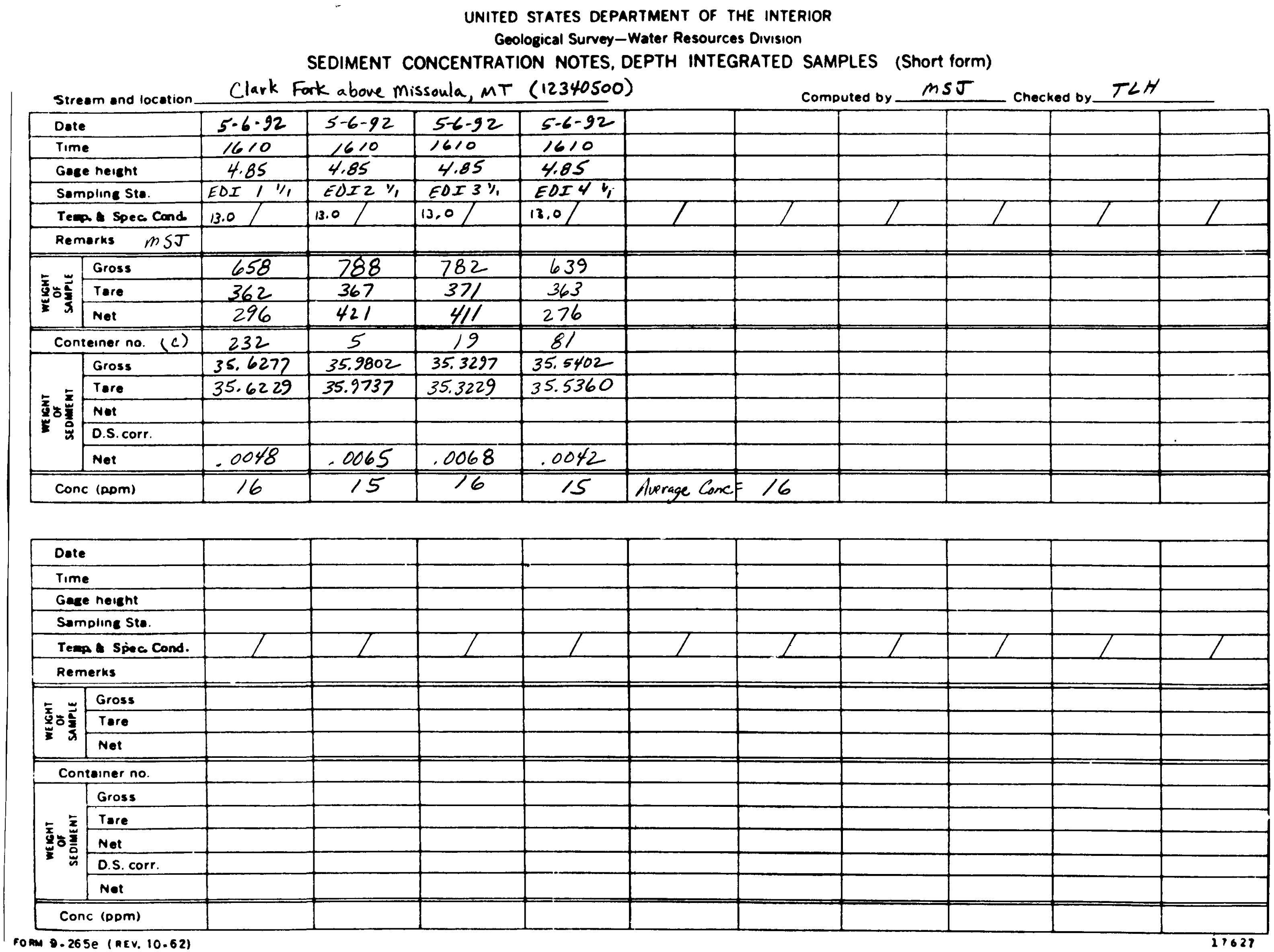
Data entry is performed in stages that correspond to the physical processing of the samples. The SED computer program is formatted into three stages: Stage 1, entry of field data and sample weights; Stage 2, assignment of containers to samples and entry of tare weights; and Stage 3, entry of sediment weights and automatic calculation of concentration. An example of data output from the SED program is shown in figure 9. Similar information is manually recorded on laboratory data sheets for cross-sectional samples collected periodically by USGS personnel (fig. 10). Because cross-sectional samples have more variable analytical requirements than observer-collected samples, manual data entry provides greater flexibility for recording modifications in analytical processing.

After the appropriate method of analysis is selected, information for individual sample bottles is initially entered, either by computer or manually. The initial data entry (Stage 1 of SED program) includes the stream and location, sample

```
  MONTANA U.S.G.S. SEDIMENT NOTES PROGRAM 5.1     FILE: MSBY.91          PAGE   5

  STREAM AND LOCATION   12-07-91 MUSSELSHELL RIVER @ MOSBY (06130500)
---------------------------------------------------------------------------------
 DATE                  07-13-91     07-13-91     07-15-91     07-15-91     07-16-91
 TIME                      1212         1212         0728         0728         0803
 GAGE HEIGHT               3.83         3.83         3.70         3.70         5.39
 SAMPLE STATION             1-2          2-2          1-2          2-2          1-2
 TEMP                      26.0         26.0         23.5         23.5         23.0
 SPEC. COND.                  -            -            -            -         1530
 REMARKS                      C            C            C            C            E
 D.S. CORR.                                                                  0.0325
 SAMPLE NET               310.0        294.0        375.0        393.0        368.0
 SEDIMENT NET            0.0656       0.0684       0.0581       0.0690       3.2432
 MGPL                       212          233          155          176         8860
 PPM                                                                         8813.0
 REMARKS                      -            -            -            -            -
---------------------------------------------------------------------------------
 DATE                  07-16-91     07-16-91     07-16-91     07-18-91     07-18-91
 TIME                      0803         2016         2016         0627         0627
 GAGE HEIGHT               5.39         4.19         4.19         3.64         3.64
 SAMPLE STATION             2-2          1-2          2-2          1-2          2-2
 TEMP                      23.0         25.5         25.5         22.0         22.0
 SPEC. COND.               1530         1580         1580         1770         1770
 REMARKS                      E            E            E            E            E
 D.S. CORR.              0.0325       0.0337       0.0337       0.0380       0.0380
 SAMPLE NET               368.0        303.0        350.0        379.0        348.0
 SEDIMENT NET            3.2332       4.5482       5.2648       0.1697       0.1347
 MGPL                      8830        15200        15200          448          387
 PPM                     8785.8      15010.7      15042.4
 REMARKS                      -            -            -            -            -
---------------------------------------------------------------------------------
 DATE                  07-20-91     07-20-91     07-22-91     07-22-91     07-24-91
 TIME                      1231         1231         1612         1612         1836
 GAGE HEIGHT               3.70         3.70         3.54         3.54         3.12
 SAMPLE STATION             1-2          2-2          1-2          2-2          1-2
 TEMP                      27.0         27.0         27.0         27.0         27.5
 SPEC. COND.                  -            -            -            -            -
 REMARKS                      C            C            C            C            C
 SAMPLE NET               339.0        349.0        348.0        310.0        317.0
 SEDIMENT NET            0.1044       0.0848       0.0740       0.0737       0.0550
 MGPL                       308          243          213          238          174
 PPM
 REMARKS                      -            -            -            -            -
---------------------------------------------------------------------------------
 DATE                  07-24-91     07-26-91     07-26-91     07-28-91     07-28-91
 TIME                      1836         0755         0755         0808         0808
 GAGE HEIGHT               3.12         3.07         3.07         3.06         3.06
 SAMPLE STATION             2-2          1-2          2-2          1-2          2-2
 TEMP                      27.5         22.5         22.5         22.5         22.5
 SPEC. COND.                  -            -            -            -            -
 REMARKS                      C            C            C            C            C
 SAMPLE NET               300.0        315.0        379.0        307.0        326.0
 SEDIMENT NET            0.0461       0.0571       0.0669       0.0474       0.0521
 MGPL                       154          181          177          154          160
 PPM
 REMARKS                      -            -            -            -            -
---------------------------------------------------------------------------------
```

Figure 9.--Example of output from the SED computer program for concentration analysis of observer samples.



UNITED STATES DEPARTMENT OF THE INTERIOR
Geological Survey—Water Resources Division
SEDIMENT CONCENTRATION NOTES, DEPTH INTEGRATED SAMPLES (Short form)

Stream and location Clark Fork above Missoula, MT (12340500) Computed by MSJ Checked by TLH

Date		5-6-92	5-6-92	5-6-92	5-6-92						
Time		1610	1610	1610	1610						
Gage height		4.85	4.85	4.85	4.85						
Sampling Sta.		EDI 1 1/1	EDI 2 1/1	EDI 3 1/1	EDI 4 1/1						
Temp. & Spec. Cond.		13.0 /	13.0 /	13.0 /	13.0 /	/	/	/	/	/	/
Remarks MSJ											
WEIGHT OF SAMPLE	Gross	658	788	782	639						
	Tare	362	367	371	363						
	Net	296	421	411	276						
Container no. (c)		232	5	19	81						
WEIGHT OF SEDIMENT	Gross	35.6277	35.9802	35.3297	35.5402						
	Tare	35.6229	35.9737	35.3229	35.5360						
	Net										
	D.S. corr.										
	Net	.0048	.0065	.0068	.0042						
Conc (ppm)		16	15	16	15	Average Conc =	16				

Date											
Time											
Gage height											
Sampling Sta.											
Temp. & Spec. Cond.		/	/	/	/	/	/	/	/	/	/
Remarks											
WEIGHT OF SAMPLE	Gross										
	Tare										
	Net										
Container no.											
WEIGHT OF SEDIMENT	Gross										
	Tare										
	Net										
	D.S. corr.										
	Net										
Conc (ppm)											

FORM 9-265e (REV. 10-62) 17627

Figure 10.--Example of laboratory form for concentration analysis of USGS cross-sectional samples (reduced).

collection date, collection time, gage height, sampling station (includes sample type and cross-section location), water temperature, specific conductance (if required), and sample gross weight. The container type (C for crucible, E for evaporation dish) is entered in the remarks field to indicate the analytical method that will be used. After entry of field data and sample gross weight, the "sample tare weight" (bottle weight that is etched in the glass) is subtracted from the sample gross weight to obtain a "sample net weight" (weight of water-sediment mixture).

The next step in sample preparation is assigning containers (either crucibles or evaporation dishes, depending on analytical method) to each sample bottle or set of sample bottles (Stage 2 of SED program). These containers are identified with a unique number, which is recorded with the sample information for the corresponding bottle. As the containers are assigned, their weight is determined with the analytical balance and recorded either by computer entry or manually on laboratory analysis forms as the "sediment tare weight." The tare weight for crucibles is determined with clean filters in place. After the tare weights of the crucibles or evaporation dishes have been determined, the containers are either used immediately or stored in desiccators until needed. All containers are handled with tongs to prevent moisture or finger oils from adhering to the container surface.

The next step in sample preparation is arranging the sample bottles on the processing bench by station and chronological order. The samples are allowed to sit for a minimum of 24 hours to allow all sediment to settle to the bottom of the bottle. After complete settling, most of the overlying clear water is removed to facilitate subsequent processing. The water can be removed because all the sediment is retained at the bottom of the bottle and the original weight of the sample (sample net weight) previously has been determined and recorded. A J-shaped tube connected to a vacuum hose is inserted into each bottle and held carefully at or near the bottom of the bottle without disturbing the sediment. The water is decanted at a withdrawal rate of about 1.0 L/min (0.3 gal/min) under a vacuum pressure of 0.5 kg/cm^2 (15 in. of mercury). The decanted water is drained into a large glass collection jug, which is observed for turbidity before disposal. Vacuum withdrawal proceeds until the water is decanted to the level of the J-tube entry port, which is about 25 mm (1 in.) above the bottom of the bottle. Because the entry port of the J-tube is above the bottom and slow withdrawal prevents turbulence, all of the original sediment and about 30 mL of sample is retained in the bottle after decanting. Final separation of sediment from the remaining water-sediment mixture is achieved by one of the following methods.

Filtration Method

The filtration method is used to analyze samples having a suspended-sediment concentration of less than about 300 mg/L. The advantage of this method is that water passes through the filter but sediment is retained; thus, any dissolved solids present in the water also are removed. As a result, drying and weighing of the sediment can proceed directly without any correction for the weight of dissolved-solids residue. The disadvantage of the filtration method is that a large quantity of sediment, especially fine sediment, can quickly clog the filter and prevent the passage of water. Crucible size to be used is determined on the basis of sediment content.

Prior to filtration, a computer or manual laboratory form containing the initial sample information is retrieved. The decanted samples are arranged on a processing bench in the sequence indicated on the laboratory form. Previously assigned crucibles are matched to the proper sample bottles by identification numbers and are arranged sequentially in the crucible manifold. A final cross-check between container and bottle is made to verify a correct match.

The crucibles are firmly seated in the rubber holders of the manifold and the vacuum pump is started. Each crucible is rinsed with distilled water to wet the filter. The vacuum line to the first crucible in the sequence is then opened by an in-line valve. A glass funnel attached to a movable ring-stand is positioned over the first crucible. The sample is prepared by first dispersing the sediment that has settled in the bottom of the bottle by vigorous agitation with a rubber-tipped stirring rod. The stirring rod is rinsed with distilled water while in the bottle to remove any attached sediment and then placed in the next bottle in the sequence. With the sediment in the first bottle dispersed, the bottle contents are slowly poured into the glass funnel to drain into the first crucible that is under vacuum. To prevent overfilling of the crucible, the sample is poured at a rate that does not exceed the filtration rate. As the sample is filtered through the crucible, the bottle and glass funnel are rinsed thoroughly with distilled water to flush all remaining sediment into the crucible. When rinsing is complete, the vacuum line to the first crucible is closed and the line to the second crucible is opened. The same procedure is repeated for each bottle and crucible in the manifold.

After the entire manifold of crucibles has been processed, the crucibles are placed in chronological sequence in a drying tray. Rubber finger tips are used for secure and clean handling during the transfer. The next set of crucibles is inserted into the manifold, verified for cross-match, and the above procedures repeated. When the drying tray is full, the tray of crucibles is placed in the convection oven for 3-4 hours at 100-105 °C.

After oven drying, the crucibles are allowed to cool for about one hour. Hot crucibles are not placed into desiccators because air expansion caused by heating could force the lid off the desiccator. The cooled crucibles are placed in desiccators for a minimum of 4 hours before final weighing to prevent moisture accumulation from ambient humidity. The crucibles are stacked in reverse chronological order from the bottom of the desiccator to allow subsequent removal from the top in proper chronological order to facilitate data entry. It is important that warm crucibles not be placed in the analytical balance because the heat will cause erratic readings.

Evaporation Method

The evaporation method is used to analyze samples having a large quantity of fine sediment that cannot be easily filtered (generally more than 300 mg/L). Use of the evaporation method requires that a correction be made for the weight of dissolved solids that precipitate as a solid residue as the water evaporates. Knowledge of the dissolved-solids concentration and the volume of sample after decanting (30 mL) enables calculation of the weight of dissolved solids present in the decanted sample.

The method used by the Montana sediment laboratory to estimate a dissolved-solids correction is to measure the specific conductance of each sample prior to processing by the evaporation method. A set of regression equations relating dissolved-solids concentration to specific conductance has been developed using data from recent chemical analyses for sites throughout the State. Separate equations are used for each of two categories of drainage basin physiography and predominant water types based on major ion composition: (1) streams with a substantial part of the basin draining mountainous terrain, and (2) streams with a substantial part of the basin draining semiarid prairies. The dissolved-solids concentration, in milligrams per liter, is estimated by regression from the specific conductance of the sample. This concentration is then converted to a dissolved-solids correction, in grams (per 30 mL of sample), by multiplying the concentration by a units conversion factor of 0.00003. The equations currently (1993) used to compute a dissolved-solids correction are:

Mountain drainage:

$$DSC = 0.00003\,[(0.654 \cdot SC) - 11.8] \tag{1}$$

Prairie drainage:

$$DSC = 0.00003\,[(0.742 \cdot SC) - 59.1] \tag{2}$$

where:

DSC = Dissolved-solids correction, in grams, in a decanted sample volume of 30 mL, and
SC = Specific conductance, in microsiemens per centimeter at 25 °C.

Both equations are statistically significant at $p = 0.001$, display strong correlation (equation 1, $R^2 = 0.99$; equation 2, $R^2 = 0.93$), and have a well-balanced residual distribution. Documentation of regression statistics is available in the Montana District Office in Helena.

The equations are stored in the SED computer program to allow automatic calculation of the dissolved-solids correction from the stored value of specific conductance. The dissolved-solids correction is subsequently subtracted from the weight of the dry sediment plus residue. The calculations are performed manually for data entered on the laboratory analysis forms. The equations are periodically updated, if necessary, using recent data from chemical samples.

Similar to the filtration method, the sample bottles and corresponding evaporation dishes are arranged in sequence on a processing bench. A laboratory form of sample information is retrieved and used to verify the cross-match of bottle to container. A glass funnel is positioned over the evaporation dish. The settled sediment in the bottle is dispersed by vigorous agitation with a rubber-tipped stirring rod, which subsequently is rinsed with distilled water to remove any attached sediment. The bottle contents are poured slowly into the glass funnel and drained directly into the dish. The bottle and funnel are rinsed thoroughly with distilled water to drain any residual sediment into the dish. Care is necessary to prevent splashing or overfilling. Samples having excessive quantities of sediment that require large volumes of rinse water are split into two dishes, with results mathematically combined.

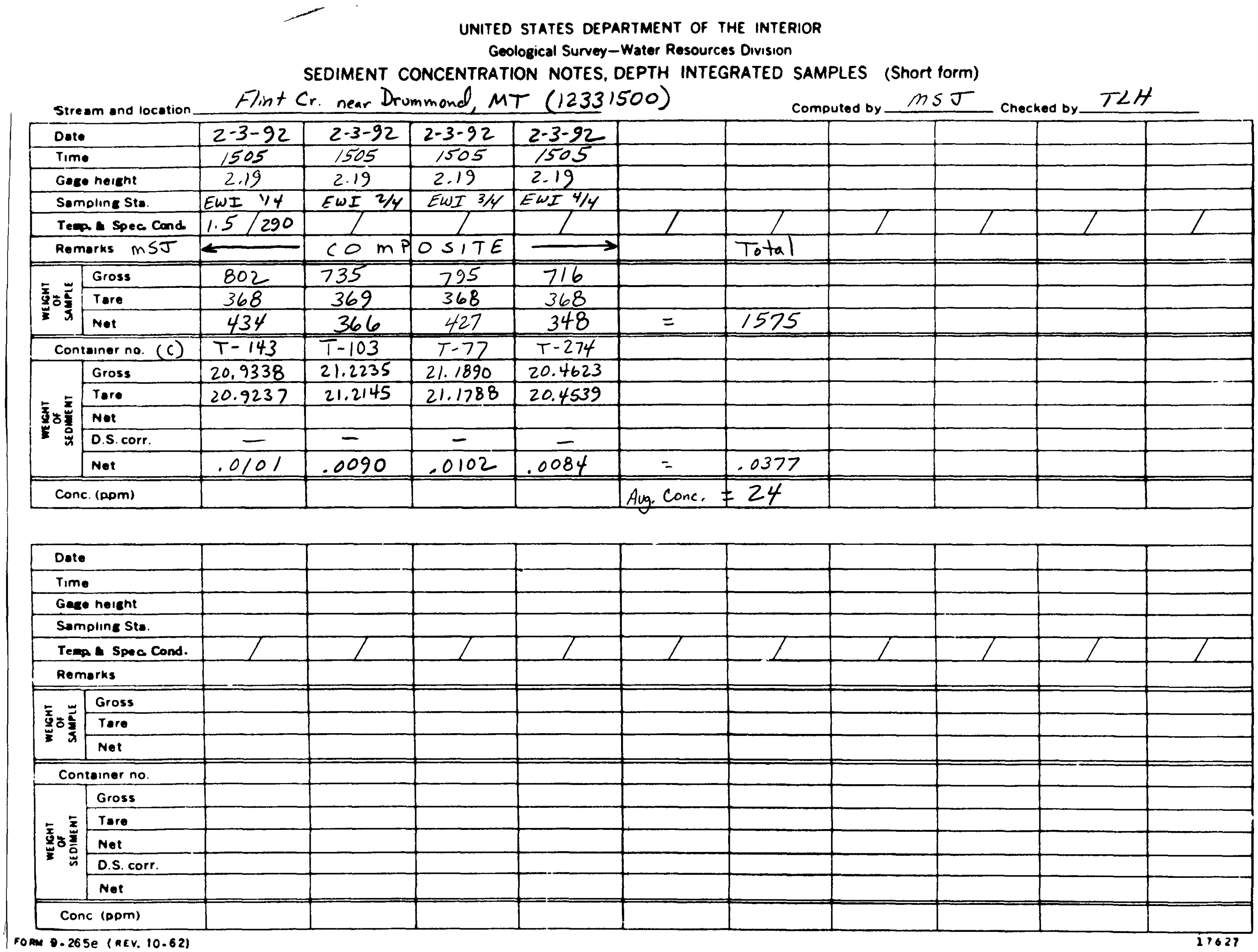
After sample transfer from bottle to dish is complete for a sequence, the dishes are immediately placed in the mechanical oven at 92-95 °C until all visible moisture has evaporated (up to 24 hours for samples with large quantities of fine sediment). After a pre-set drying time, the oven automatically shuts off. The samples are dried for an additional hour at 105 °C and then allowed to cool for about 1 hour while in the oven. When cool, the dishes are transferred to desiccators for a minimum storage time of 4 hours prior to weighing.

Composited Samples

The average concentration of suspended sediment in the cross section of a stream is determined by collecting depth-integrated samples at multiple verticals across the stream according to procedures described by Edwards and Glysson (1988). The procedures provide a volume of sample from each vertical that is proportional to the flow, thereby allowing individual bottles from multiple verticals to be composited and analyzed collectively.

Each bottle of a multi-bottle cross-sectional sample may be analyzed individually for concentration if the "equal-discharge increment" (EDI) sampling method was used (Edwards and Glysson, 1988, p. 57). Concentration analysis of each bottle indicates the horizontal variability in concentration across the stream. The average cross-sectional concentration is obtained by mathematically averaging individual concentrations from all the bottles (fig. 10). Bottles of a cross-sectional sample set obtained by other sampling methods are not analyzed individually, but rather are physically composited to determine total sample and total sediment weights for all the bottles combined. The average cross-sectional concentration for these samples is calculated from the ratio of total sediment to total sample weight (fig. 11).

Visual inspection of sediment quantity is used as a guide to determine the number of crucibles or evaporation dishes needed to composite multi-bottle sample sets. For samples having very small sediment quantities, the contents from several or all bottles of a cross-sectional sample can be filtered through one or two crucibles without substantial clogging of the filter. Samples having a larger



UNITED STATES DEPARTMENT OF THE INTERIOR
Geological Survey—Water Resources Division
SEDIMENT CONCENTRATION NOTES, DEPTH INTEGRATED SAMPLES (Short form)

Stream and location Flint Cr. near Drummond, MT (12331500) Computed by MSJ Checked by TLH

Date		2-3-92	2-3-92	2-3-92	2-3-92						
Time		1505	1505	1505	1505						
Gage height		2.19	2.19	2.19	2.19						
Sampling Sta.		EWI 1/4	EWI 2/4	EWI 3/4	EWI 4/4						
Temp. & Spec. Cond.		1.5 / 290	/	/	/	/	/	/	/	/	/
Remarks mSJ		←	COMP	OSITE	→		Total				
WEIGHT OF SAMPLE	Gross	802	735	795	716						
	Tare	368	369	368	368						
	Net	434	366	427	348	=	1575				
Container no. (C)		T-143	T-103	T-77	T-274						
WEIGHT OF SEDIMENT	Gross	20.9338	21.2235	21.1890	20.4623						
	Tare	20.9237	21.2145	21.1788	20.4539						
	Net										
	D.S. corr.	—	—	—	—						
	Net	.0101	.0090	.0102	.0084	=	.0377				
Conc. (ppm)						Avg. Conc. =	24				

Date											
Time											
Gage height											
Sampling Sta.											
Temp. & Spec. Cond.		/	/	/	/	/	/	/	/	/	/
Remarks											
WEIGHT OF SAMPLE	Gross										
	Tare										
	Net										
Container no.											
WEIGHT OF SEDIMENT	Gross										
	Tare										
	Net										
	D.S. corr.										
	Net										
Conc (ppm)											

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Figure 11.--Example of laboratory form for concentration analysis of composited USGS cross-sectional samples (reduced).

quantity of sediment or predominance of fine material might require a separate crucible or evaporation dish for each bottle. Samples having very large sediment quantities, which are processed only by the evaporation method, might require splitting the contents of one bottle into two or more dishes to accommodate the extra rinse water. Regardless of the number of containers used to composite bottles of a cross-sectional sample, the container identification numbers and tare weights are clearly shown along with the corresponding sample-collection information for each sample bottle processed. Thorough documentation of the compositing process enables tracking of the sample treatment and accurate calculation of concentration.

Particle-Size Analysis

Particle-size analysis involves separating the sediment in a sample on the basis of particle size, followed by weighing the sediment in each size class. In the Montana sediment laboratory, the distribution of particle size is determined by the proportion of sediment weight in each size class relative to the total sediment weight.

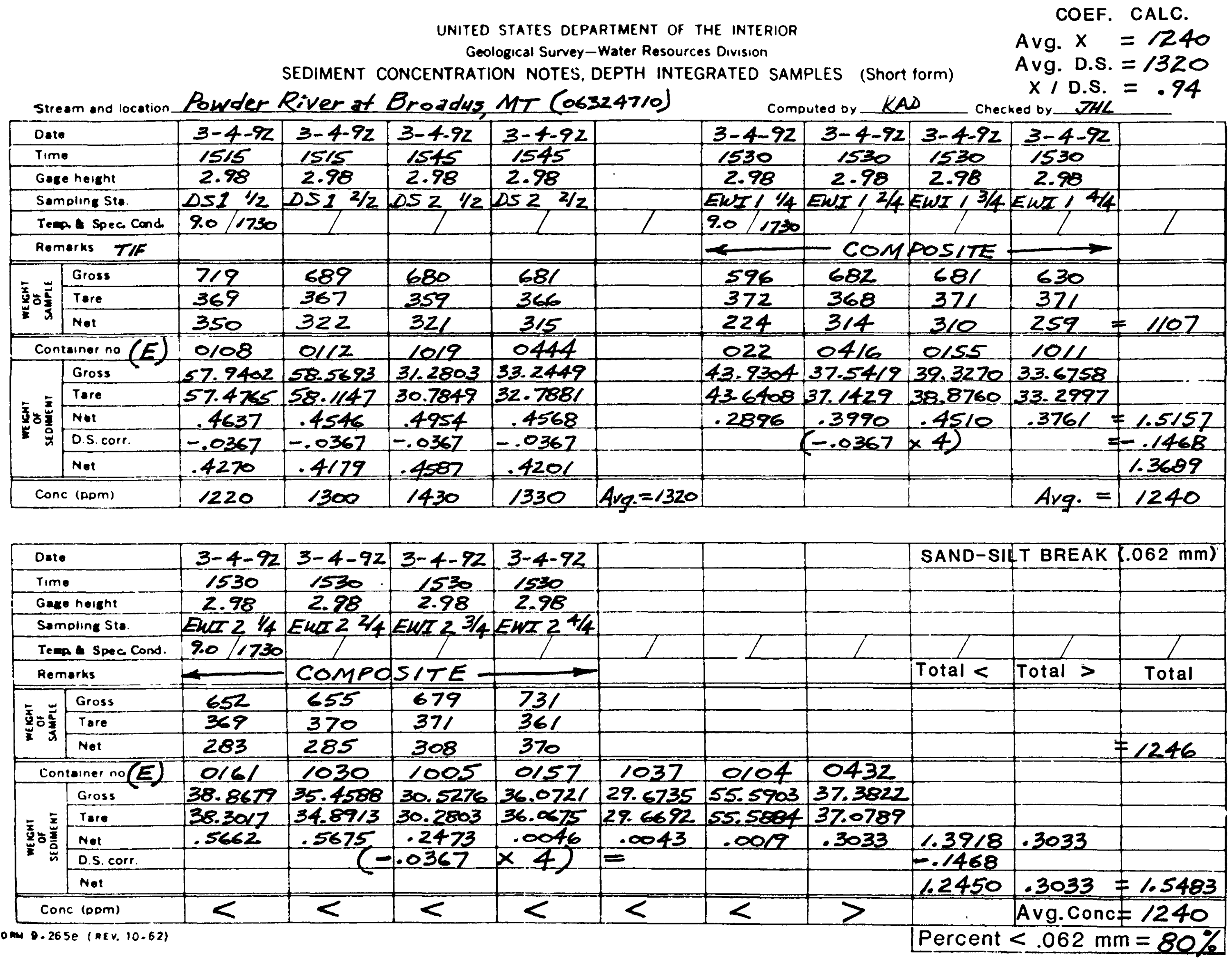
The particle-size analysis done by the Montana sediment laboratory is the sand-silt separation. This analysis determines the proportion of suspended sediment in two size classes--the coarse fraction with particle diameters greater than 0.062 mm (sand-size and larger), and the fine fraction with particle diameters less than 0.062 mm (silt-size and smaller). The particle diameter of 0.062 mm represents the transition between the two fractions and is, by convention, termed the "sand-silt break." Particle-size analysis is typically performed on cross-sectional samples, although single-vertical samples having unusually large concentrations occasionally are analyzed for particle-size distribution.

Similar to the concentration analysis, containers are assigned to the sample set and their identification numbers and tare weights are recorded. The containers are further identified on the laboratory form as to whether they will receive the fine (less than 0.062 mm) or the coarse (greater than 0.062 mm) fraction. Judgment by experienced laboratory personnel is required to ensure that enough containers are assigned for each fraction. A verification of bottle-to-container cross match is made before size processing begins.

To determine the proportion of sediment in each of the two size classes, the sediment is separated physically by pouring the sample through a 3-in. sieve having mesh openings of 0.062 mm. This size separation is incorporated in the regular sequence of steps for determining concentration; however, additional documentation is required to record the partitioning of sediment by size class among the crucibles or evaporation dishes (fig. 12).

The two size classes are separated by inserting a 0.062 mm mesh sieve into the glass funnel through which the sample is poured (fig. 5). The sample is slowly poured through the sieve and drained into the first container assigned to the "less than" 0.062 mm fraction. The sample bottle is thoroughly rinsed, with all rinse water also drained through the sieve into the same container. The sieve retains all sediment particles greater than 0.062 mm diameter. The glass funnel with the sieve is subsequently moved to the next container assigned to the "less than" 0.062 mm fraction and the next bottle of sample is poured. The process is repeated until all sample bottles have been poured and rinsed through the sieve and into the "less than" containers. The sediment on the screen is gently rinsed with distilled water to ensure that all material finer than 0.062 mm is flushed through the screen. At this point, all coarse sediment from the entire sample set resides on top of the sieve screen. The screen is lifted and the interior surface of the funnel is thoroughly rinsed with distilled water into the last "less than" container to remove any residual fine sediment.

The final step in the separation is to transfer the coarse sediment to the container assigned to the "greater than" 0.062 mm diameter fraction. This transfer is accomplished by moving the glass funnel with the sieve over the "greater than" container. The sieve is then inverted inside the glass funnel so that the coarse sediment is on the underside of the screen. The coarse sediment is backflushed



UNITED STATES DEPARTMENT OF THE INTERIOR
Geological Survey—Water Resources Division
SEDIMENT CONCENTRATION NOTES, DEPTH INTEGRATED SAMPLES (Short form)

COEF. CALC.
Avg. X = 1240
Avg. D.S. = 1320
X / D.S. = .94

Stream and location Powder River at Broadus, MT (06324710)
Computed by KAD
Checked by JHL

Date		3-4-92	3-4-92	3-4-92	3-4-92		3-4-92	3-4-92	3-4-92	3-4-92	
Time		1515	1515	1545	1545		1530	1530	1530	1530	
Gage height		2.98	2.98	2.98	2.98		2.98	2.98	2.98	2.98	
Sampling Sta.		DS1 1/2	DS1 2/2	DS 2 1/2	DS 2 2/2		EWI 1 1/4	EWI 1 2/4	EWI 1 3/4	EWI 1 4/4	
Temp. & Spec. Cond.		9.0 / 1730	/	/	/	/	9.0 / 1730	/	/	/	/
Remarks T/F							← COMPOSITE →				
WEIGHT OF SAMPLE	Gross	719	689	680	681		596	682	681	630	
	Tare	369	367	359	366		372	368	371	371	
	Net	350	322	321	315		224	314	310	259	= 1107
Container no (E)		0108	0112	1019	0444		022	0416	0155	1011	
WEIGHT OF SEDIMENT	Gross	57.9402	58.5693	31.2803	33.2449		43.9304	37.5419	39.3270	33.6758	
	Tare	57.4765	58.1147	30.7849	32.7881		43.6408	37.1429	38.8760	33.2997	
	Net	.4637	.4546	.4954	.4568		.2896	.3990	.4510	.3761	= 1.5157
	D.S. corr.	-.0367	-.0367	-.0367	-.0367			(-.0367 x 4)			= -.1468
	Net	.4270	.4179	.4587	.4201						1.3689
Conc (ppm)		1220	1300	1430	1330	Avg.=1320				Avg. =	1240

Date		3-4-92	3-4-92	3-4-92	3-4-92				SAND-SILT BREAK		(.062 mm)
Time		1530	1530	1530	1530						
Gage height		2.98	2.98	2.98	2.98						
Sampling Sta.		EWI 2 1/4	EWI 2 2/4	EWI 2 3/4	EWI 2 4/4						
Temp. & Spec. Cond.		9.0 / 1730	/	/	/	/	/	/	/	/	/
Remarks		← COMPOSITE →							Total <	Total >	Total
WEIGHT OF SAMPLE	Gross	652	655	679	731						
	Tare	369	370	371	361						
	Net	283	285	308	370						= 1246
Container no (E)		0161	1030	1005	0157	1037	0104	0432			
WEIGHT OF SEDIMENT	Gross	38.8679	35.4588	30.5276	36.0721	29.6735	55.5903	37.3822			
	Tare	38.3017	34.8913	30.2803	36.0675	29.6692	55.5884	37.0789			
	Net	.5662	.5675	.2473	.0046	.0043	.0019	.3033	1.3918	.3033	
	D.S. corr.		(-.0367 x 4)			=			-.1468		
	Net								1.2450	.3033	= 1.5483
Conc (ppm)		<	<	<	<	<	<	>		Avg.Conc	= 1240

Percent < .062 mm = 80%

FORM 9-265e (REV. 10-62)

Figure 12.--Example of laboratory form for particle-size analysis of USGS cross-sectional samples (reduced).

into the "greater than" container by thoroughly rinsing the sieve and funnel with distilled water. The containers are then transferred to the oven for drying as described for the concentration analysis.

Size classes in addition to the two fractions of greater than or less than 0.062 mm can also be determined. Complete particle-size distribution is typically analyzed only for samples having large suspended-sediment concentrations, because the analytical procedures generally require a substantial quantity of sediment (1 g or more). Complete particle-size distribution is not analyzed by the Montana sediment laboratory, but rather, the samples are sent to the Iowa District sediment laboratory, which is equipped to routinely perform multiple size-class separations. Quality assurance for complete particle-size analysis is implemented according to policies and procedures of the Iowa District, as described by Matthes and others (1992).

Samples determined to have a quantity of sediment that is sufficient for a complete size analysis are decanted as described previously, then transferred to small shipping bottles. The original bottle tag containing the field information is attached to the shipping bottle. The sample net weight (sample gross weight minus bottle tare weight) is recorded on the tag and labeled "sample net weight." All field information and sample weights are manually recorded on a laboratory-analysis form which is retained in the Montana sediment files as backup documentation. Prior to shipping in special padded cartons, a shipment form that contains sample-collection information and specific analytical requests is completed (fig. 13). A copy of the shipment form is enclosed with the sample bottles, and the original is kept in the Montana sediment files.

Sediment Weight Determination

The final step of the analytical process is determining the weight of the dry sediment (Stage 3 of SED computer program). After the sediment has been oven-dried, the containers are removed from the ovens, cooled, and transferred to desiccators for storage until final weighing. Weighing can begin when the containers have been desiccated for at least 4 hours.

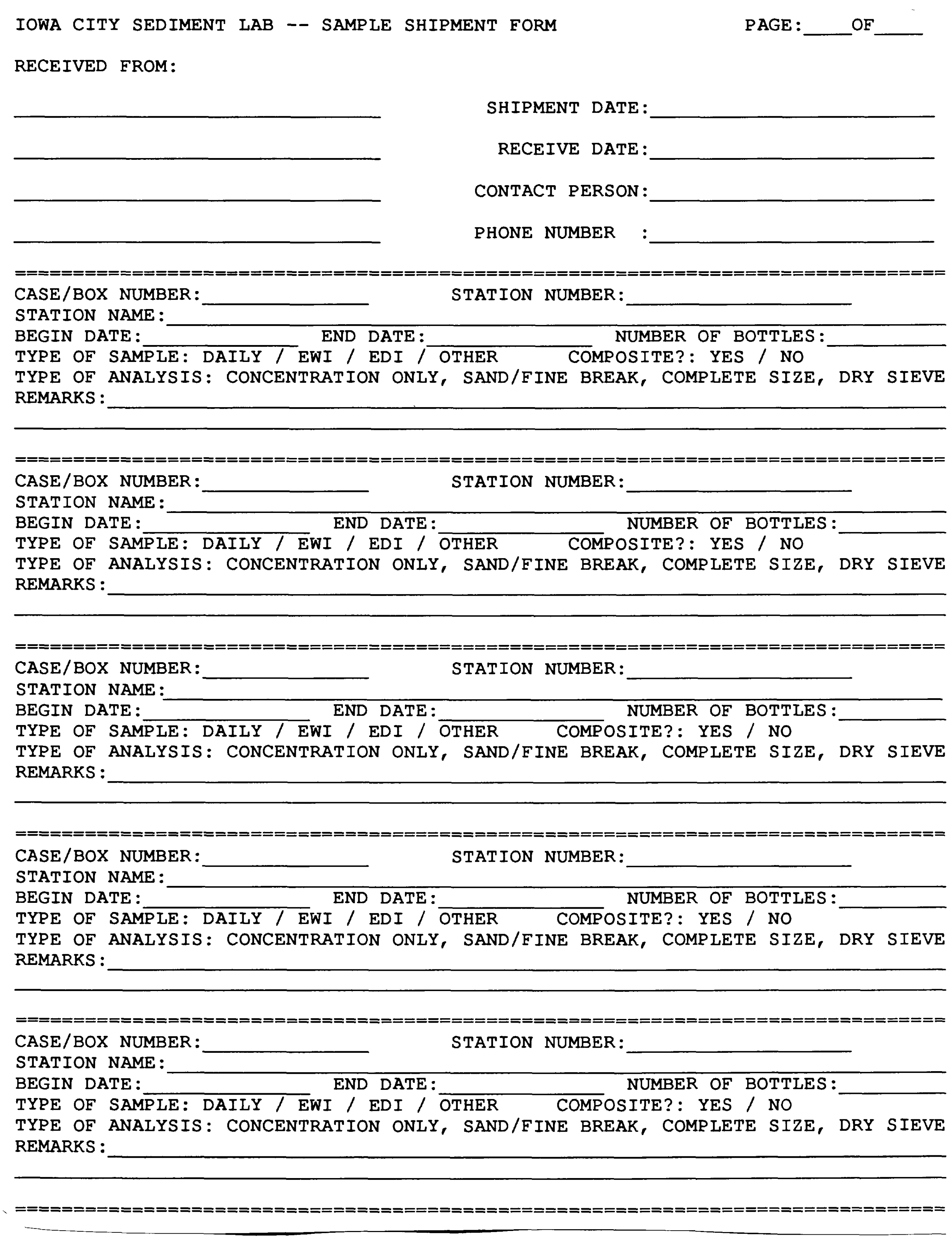
The containers are placed on the balance pan sequentially to maintain chronological order for daily samples. Proper cross-match is verified to ensure that the weight of the current container is recorded in the correct column identified by the container identification number. The weight of the container plus dry sediment (and residue for evaporated samples) is recorded as "sediment gross weight." After this weight is recorded, the container is removed and placed in a holding tray. Containers are not washed for reuse until the corresponding data have been reviewed and accepted as satisfactory by the laboratory supervisor. Reweighing may be needed to verify questionable results.

The "sediment tare weight" (container weight) and weight of dissolved-solids residue, if applicable, are subtracted from the "sediment gross weight" to obtain the "sediment net weight," which is the weight of the dry sediment. These subtractions are done automatically for data entered by computer, and manually on the laboratory analysis form.

Calculation of Results

After the weight of dry sediment has been determined, the suspended-sediment concentration can be calculated. Suspended-sediment concentration, which is reported, by convention, in milligrams per liter, is calculated by dividing the sediment net weight, in grams, by the sample net weight, in grams, and multiplying by 1,000,000. This calculation is performed automatically by computer program, or manually for data entered on laboratory-analysis forms. Significant figures reported for concentration are one for 1-9 mg/L, two for 10-99 mg/L, and three for concentrations equal to or greater than 100 mg/L.

The analytical procedures for determining concentration use the measurement of sample weight rather than volume, so the reported concentrations actually represent



IOWA CITY SEDIMENT LAB -- SAMPLE SHIPMENT FORM PAGE:____OF____

RECEIVED FROM:

______________________________ SHIPMENT DATE:______________________

______________________________ RECEIVE DATE:______________________

______________________________ CONTACT PERSON:______________________

______________________________ PHONE NUMBER :______________________

==

CASE/BOX NUMBER:______________ STATION NUMBER:__________________
STATION NAME:____
BEGIN DATE:______________ END DATE:______________ NUMBER OF BOTTLES:__________
TYPE OF SAMPLE: DAILY / EWI / EDI / OTHER COMPOSITE?: YES / NO
TYPE OF ANALYSIS: CONCENTRATION ONLY, SAND/FINE BREAK, COMPLETE SIZE, DRY SIEVE
REMARKS:____
____

==

CASE/BOX NUMBER:______________ STATION NUMBER:__________________
STATION NAME:____
BEGIN DATE:______________ END DATE:______________ NUMBER OF BOTTLES:__________
TYPE OF SAMPLE: DAILY / EWI / EDI / OTHER COMPOSITE?: YES / NO
TYPE OF ANALYSIS: CONCENTRATION ONLY, SAND/FINE BREAK, COMPLETE SIZE, DRY SIEVE
REMARKS:____
____

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CASE/BOX NUMBER:______________ STATION NUMBER:__________________
STATION NAME:____
BEGIN DATE:______________ END DATE:______________ NUMBER OF BOTTLES:__________
TYPE OF SAMPLE: DAILY / EWI / EDI / OTHER COMPOSITE?: YES / NO
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REMARKS:____
____

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CASE/BOX NUMBER:______________ STATION NUMBER:__________________
STATION NAME:____
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TYPE OF SAMPLE: DAILY / EWI / EDI / OTHER COMPOSITE?: YES / NO
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REMARKS:____
____

==

CASE/BOX NUMBER:______________ STATION NUMBER:__________________
STATION NAME:____
BEGIN DATE:______________ END DATE:______________ NUMBER OF BOTTLES:__________
TYPE OF SAMPLE: DAILY / EWI / EDI / OTHER COMPOSITE?: YES / NO
TYPE OF ANALYSIS: CONCENTRATION ONLY, SAND/FINE BREAK, COMPLETE SIZE, DRY SIEVE
REMARKS:____
____

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Figure 13.--Example of form for documentation of sample shipment.

a sediment weight:sample weight ratio of parts per million. This unit of measure is equivalent to the weight:volume ratio of milligrams per liter at concentrations less than about 8,000 ppm. At concentrations of 8,000 ppm or greater, a correction for change in specific weight is applied in accordance with factors listed in table 1. Correction factors are interpolated automatically by the SED computer program. This correction converts concentration values in parts per million to milligrams per liter and is performed automatically for data entered by computer and manually for data entered on laboratory-analysis forms.

Table 1.--Factors for converting suspended-sediment concentration from parts per million to milligrams per liter[1]

[The values of C are based on the assumption that the density of water is 1.000 g/cm^3 plus or minus 0.005 g/cm^3, the range of temperature is 0-29 degrees Celsius, the specific gravity of sediment is 2.65, and the dissolved-solids concentration is less than 10,000 ppm. Abbreviation: g/cm^3, grams per cubic centimeter; ppm, parts per million]

Suspended-sediment concentration range (ppm)	Conversion factor (C)	Suspended-sediment concentration range (ppm)	Conversion factor (C)
0- 7,990	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		

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[1] W.H. Durum, U.S. Geological Survey, written commun., 1972.

The results of particle-size analyses are reported as percent of total sediment weight finer than an indicated particle diameter, in millimeters. The sand-silt size separation performed by the Montana sediment laboratory is reported as percent of suspended sediment finer than 0.062 mm. Other size classes analyzed by the Iowa sediment laboratory are reported in the same manner for each size separation performed.

The percent of sediment finer than 0.062 mm is calculated by adding the sediment net weights from all bottles of a sample set to determine a total sediment net weight (fig. 11). The weight of the sediment fraction finer than 0.062 mm is determined by adding the individual sediment net weights for each of the "less than" containers. The combined sediment net weight of the fine sediment fraction (minus the dissolved-solids correction, if applicable) is then divided by the total sediment net weight of the sample set and multiplied by 100 to obtain a value in percent.

Analytical Quality Control

Analytical procedures are designed to produce data of known quality. Documentation of procedural checks during the analytical process is used to verify that procedures were properly implemented. These records of quality control are reviewed systematically to identify sources of error. Supplemental tests of analytical quality control performed on various types of reference samples provide a measure of the magnitude of error to assess the accuracy of analytical results.

Laboratory analyses are performed on a variety of test samples to identify analytical results that may indicate potential error. These test samples provide insight to consistent bias resulting from systematic procedural errors. The type and magnitude of error provide guidance in modifying procedures to correct the error. Inconsistent variability resulting from random procedural errors is less easily identified and controlled. These errors are controlled in a general manner by adequate training and supervision of laboratory analysts and by a prompt review of analytical results by the laboratory supervisor. Results of all quality-control analyses are recorded in a laboratory "quality-control" logbook, and periodically reviewed by the laboratory supervisor.

Standard Reference Samples

A program implemented by the USGS Office of Surface Water is designed to determine the bias and precision of concentration analyses performed by sediment laboratories of the USGS. Standard reference samples of known sediment quantity are distributed by the USGS Branch of Quality Assurance to all USGS sediment laboratories. Multiple sediment mixtures are prepared that contain particle sizes ranging from clay to sand. The weight of the sediment in each sample is known to the nearest 0.0001 g. A water-sediment mixture is quantitatively prepared from the reference material, and then analyzed by standard procedures. An interlaboratory comparison of results is used to estimate the bias and precision of results.

Evaporation Blanks

Evaporation blanks are used to document the volume of sample evaporated during field storage. Sample integrity and subsequent analytical results can be affected if an excessive quantity of water evaporates during storage, thereby decreasing the original sample volume. The effect of sample evaporation is a decrease in the sample net weight which could result in an erroneous increase in concentration. This effect is most likely to occur in observer-collected daily samples that are stored at an observer's residence for a maximum period of 6 weeks before being brought to the field office or District Office. Evaporation is minimized by using tight-fitting bottle caps, keeping bottles in a cool location during storage, and promptly weighing samples within 2 weeks after arrival at the sediment laboratory.

Evaporation blanks consist of a tightly capped sample bottle containing 300 mL of distilled water. The sample gross weight is determined, and one blank per 120 bottles is placed in the cases of empty sample bottles supplied to the observer during site visits. The evaporation blank is therefore exposed to the same ambient temperature and humidity conditions as the suspended-sediment samples collected and stored by observers. After the samples are retrieved from the observer's residence and brought to the laboratory, the evaporation blanks are reweighed to determine the magnitude of any weight loss. The weight of water evaporated can then be used to evaluate the effect on concentrations and determine if adjustments need to be made to the sample net weights measured in the laboratory. All evaporation data are recorded in a quality-control logbook and reviewed by the laboratory supervisor.

Sample Blanks

Samples of distilled water are analyzed with regular suspended-sediment samples to document the probable magnitude of systematic processing and weighing errors. Weight might be gained from the transfer of sediment between bottles owing to incomplete rinsing of the decanting J-tube, stirring rod, or funnel. Weight gains might also result from incomplete oven drying or subsequent moisture absorption after removal of containers from the oven. Weight might be lost by detachment of filter fibers during vacuum filtration.

Two distilled-water sample blanks are analyzed for every table of samples (120 bottles). One blank is placed at the beginning of the bottle sequence as an initial reference, and one is placed at the end to optimize detection of cross-contamination. A 300-mL volume of distilled water is measured and poured into a glass sample bottle to represent a typical sample volume. The sample gross weight is measured as previously described, and all processing steps are performed as if an actual sample were being analyzed. Both filtration and evaporation methods are used in the tests, depending on the type of procedure that is used at the time of processing.

After drying, the weight of the container (and filter if applicable) is determined for the distilled-water sample blanks and compared to the original tare weight. The criterion for an acceptable deviation from a zero net weight change is a positive or negative value of 0.0003 g (equivalent to 1 mg/L in a 300-mL sample). If the net weight change of the sample blanks exceed the equivalent of 1 mg/L, which represents the minimum reporting level, the balance calibration is checked. If the balance is correctly reading a value in excess of 0.0003 g, analytical procedures and previous results are reviewed to evaluate the need for corrective actions.

Filter Blanks

Filter blanks are used to identify systematic errors resulting specifically from fiber loss of filters during vacuum filtration of samples processed through crucibles. Whereas sample blanks attempt to identify errors collectively for a series of processing steps, filter blanks define the probable magnitude of error associated with a single step in the analytical process. Potential loss of filter fiber is minimized by pre-rinsing and seating filters in crucibles with distilled water under vacuum. This seating procedure is performed after the cleaning process and is designed to remove loose fibers prior to drying and obtaining a crucible tare weight.

Two filter blanks are processed for every table of samples (120 bottles) when the filtration method is used. All data are recorded in a quality-control logbook. A container tare weight (with filter) is determined for each test crucible. A volume of 50 mL of distilled water is poured into the crucible and vacuumed through the filter. The crucible is then dried in the same manner as routine samples. After drying and cooling, the crucibles are placed in desiccators. Final weights are obtained and recorded, and compared to the original tare weights. Consistent weight losses of 0.0003 g or greater indicate a potential problem that requires evaluation for corrective action.

Comparison of Methods

To assess the comparability and precision of results obtained by both the filtration and the evaporation methods, replicate samples having a known identical sediment concentration are analyzed independently by each of the methods. Because several aspects of processing differ between the two methods, the resulting concentrations are compared to each other to detect significant differences and to provide a measure of precision. The results are also compared to the theoretical concentration, which is based on the known sample volume and sediment weight. Methods are compared twice annually.

For the methods-comparison test, three sets of four clean, glass sample bottles are weighed to verify that the etched tare weights are correct. A volume of 300 mL of distilled water is added to each bottle. Identical weights of sediment are premeasured on dry, tared containers for each of three ranges of sediment weight. Sediment weights of 0.0010, 0.0300, and 0.1000 g are used to provide small, medium, and large concentrations of filterable sediment that represent a typical environmental range. The premeasured sediment is carefully transferred to the bottles. Sample gross weights are then measured and the three sets of test samples are processed as regular samples by the two methods. Two bottles of each sediment weight are processed by each method to provide duplicate results within and between methods. Because distilled water is used, test samples processed by the evaporation method do not require a dissolved-solids correction.

After drying and determining the sediment net weight, concentrations are calculated for each test sample. The concentrations determined by each method are considered to be of acceptable equivalence if they are within 5 percent of each other and the theoretical concentration. Concentrations of duplicate samples within each method are considered to have acceptable precision if they are within 5 percent of each other. If concentrations differ by more than 5 percent, another set of test samples is processed. If the second set of test samples results in unacceptable comparability or precision, procedures are reviewed to evaluate possible corrective actions.

Decant-Filtrate Analysis

The overlying clear water that is decanted from the sample bottles after the sediment has settled to the bottom is collected in a large glass jug and disposed of when the bottle is full. If settling is complete within the sample bottles and decanting is done carefully, this decanted water will contain no sediment or only a negligible quantity. Sample water that is vacuumed through crucible filters during the filtration process also is drained to the same jug. Improperly seated filters at the base of crucibles could allow sediment to pass by the sides of the filter. To verify that sediment is not being lost during the decanting or filtering process, test samples of the water removed by vacuum withdrawal are periodically analyzed. Such testing is done twice annually or when the decant-filtrate water is unusually cloudy.

Duplicate samples of about 300 mL are poured from the collection jug after thorough agitation into tared glass sample bottles. The sample gross weights are measured and the samples are processed by the filtration method. To optimize detection of minor quantities of sediment lost during processing, the entire undecanted sample volume is filtered. The crucibles are dried and the sediment net weight is determined. Concentrations determined for decant-filtrate samples are considered to be acceptable if they do not exceed the minimum reporting level of 1 mg/L (0.0003 g per 300 mL of sample).

Interlaboratory Comparisons

Duplicate sets of cross-sectional suspended-sediment samples provide an opportunity to compare results between the Iowa and the Montana sediment laboratories. Although sampling variability is inherent in duplicate field samples, a comparative data base can identify gross inconsistencies in results. Periodically, one set of a duplicate field sample is sent to the Iowa sediment laboratory for a complete particle-size and concentration analysis; the other sample set is retained in the Montana sediment laboratory for a concentration analysis. This procedure enables comparison of concentrations from both sediment laboratories.

In addition to concentration comparisons between duplicate sample sets, particle-size analytical results for the less-than 0.062-mm size class are compared periodically. For selected samples, a sand-silt size separation is performed on both sets of a duplicate sample. A 0.062-mm sieve separation is performed in the Montana sediment laboratory for one set of the duplicate sample set. A complete particle-size analysis, which includes the 0.062-mm separation, is performed on the other set by the Iowa sediment laboratory. Comparison of the percentage of sediment finer than 0.062 mm determined by each laboratory enables an evaluation of comparability.

Laboratory Reviews

All USGS sediment laboratories periodically undergo a review of facilities and operations. The review is conducted by a team of sediment specialists designated by the USGS Office of Surface Water. The review is designed to identify deficiencies in procedures and to provide recommendations for correcting problems or improving quality. Quality-control logbooks are reviewed to evaluate documentation procedures and the performance of laboratory equipment.

QUALITY ASSURANCE OF DATA MANAGEMENT

Determination of sediment concentration and particle size requires numerous tabulations of data and mathematical calculations. The accuracy of the laboratory data is systematically checked and the values are technically reviewed to assure that the data are reasonable for the prevailing hydrologic conditions and site characteristics. Analytical results in computer data bases and report manuscripts are verified prior to public release of sediment data.

Laboratory Data Files

All data generated during the sequence of steps from sample collection through analysis are stored in various files that permit the tracking, retrieval, storage, and transmittal of data. The files consist of computer-storage systems and original manual documents. Computer data are retrieved as paper copy and filed for backup.

Data entered in the computer during laboratory analysis are stored in files generated by the SED program of the USGS. These data are retrievable by a record-identification number. Paper copies are stored in the Montana District in both the water-quality station files and the sediment files. Copies of manual documents for daily sediment stations are mailed to the Montana District field office that is responsible for the daily record calculations. All original manual documents are stored in the District files for backup. All documents in station files are microfiched and archived about every 10 years for historical storage.

Tracking of the analytical progress of samples collected at daily sediment stations is documented by a sediment-data distribution form (fig. 14). This form is used to record sample-collection dates, sample type, and particle-size analysis. Samples sent to the Iowa sediment laboratory are identified by dates of shipment and receipt of analytical results. Subsequent distribution of laboratory data to the sediment station files, Data Management Unit, field office, and cooperating agencies is noted for verification of data transmittal.

The potential loss of data from either the paper or computer file necessitates that backup documentation be adequate to enable recovery of the data. Backup typically consists of making paper copies of all computer and laboratory data forms and storing these copies in at least two independent files. Copies, rather than original documents, are mailed from the laboratory in response to data requests.

Data files entered into computer storage are subject to damage or loss during power failure or equipment malfunction. Losses are minimized by routinely making temporary computer copies of data files during various stages of processing. Temporary computer files are deleted after final files are stored and backed up with

DISTRIBUTION RECORD OF SEDIMENT DATA

Sample date(s)	Sample type (Obs, X-sect, BM)	Helena S-S	Iowa CPS		Copies to District Sed. files	Copies to ADP	Mailing Date	
			Sent	Received			Field Off.	Coop.

Observer field sheets sent		
Month	FO	Dist.

Figure 14.--Example of form for documentation of sediment-data distribution (reduced).

paper-copy retrieval. Data losses are also minimized by regular computer system backups that transfer data to magnetic tape.

Data Review

The quality of suspended-sediment data generated by laboratory analysis of samples is maintained by following the procedures described in this report and by performing a systematic review of analytical results prior to data release. The systematic review includes mathematical checks of all manual calculations and semiannual checks of computer-program algorithms. Although quality-control measures integrated into the analytical sequence are designed to minimize systematic errors, they cannot prevent all random errors. Therefore, additional review of the data in a hydrologic context can identify questionable values that need to be either further evaluated or eliminated. For any unusual values, the sediment container is inspected to corroborate any obvious discrepancies. If visual inspection is inconclusive, the containers are reweighed to verify original results. Remarks on field forms that note the sampler's observation of stream conditions also are inspected for further verification. If adequate verification of unusual values is not possible, the concentration is not approved and is not entered in the data base.

Manual calculations of concentration and particle-size distribution made by the laboratory analyst are verified by an independent checker. The verification applies to all sample data that are recorded manually on laboratory-analysis forms. The individual mathematical calculations needed to determine the final results are noted directly on the laboratory forms as being either correct or incorrect. If corrections are not necessary, the checker initials the form and routes it to the laboratory supervisor. If corrections are necessary, the original value is crossed out and the correct value is written above the original before the form is initialed and routed to the laboratory supervisor.

Calculations performed by computer algorithm in the SED program are periodically checked to ensure that automatic computations are correct. On a quarterly basis, actual sample data for selected samples are mathematically processed using a calculator to determine concentration, dissolved-solids corrections, and conversion from parts per million to milligrams per liter. Results of these checks are compared to computer-generated values to verify algorithm accuracy, and are filed in the laboratory quality-control logbook. Discrepancies between computer-generated and calculator results are brought to the attention of the District Computer Specialist, who evaluates the extent of erroneous data and, if necessary, corrects the program code.

The final step prior to approval of the laboratory data for subsequent daily record calculations or publication is technical review by either the laboratory supervisor or the District Sediment Specialist. Concentration and particle-size data for all samples are reviewed to verify completeness, proper computational procedure, adequate documentation of departures from routine analytical procedures, consistency of results for duplicate samples, and hydrologically reasonable values. Approved data are entered in national computer files of the USGS. The data are retrieved from the files after entry and verified for proper transcription by comparison to original records.

Publication

Sediment data that have been technically reviewed and approved for release are processed through the Montana District Data Management Unit for publication in the report series "Water Resources Data, Montana" (U.S. Geological Survey, issued annually). Laboratory data published in this report include instantaneous concentration and particle-size distribution for periodic cross-sectional samples collected by USGS personnel. Laboratory data for samples collected by observers are stored in the SED program files. These data are used in a computational process described by Porterfield (1972) to determine daily mean suspended-sediment concentrations at daily sediment stations. These daily mean values are published rather than the instantaneous sediment concentrations of the observer's samples. Values approved for publication are verified for correct transcription in the report manuscript as a final step before printing of the report.

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