QUALITY ASSURANCE/QUALITY CONTROL MANUAL

National Water Quality Laboratory

By J.W. Pritt and J.W. Raese, Editors

U.S. GEOLOGICAL SURVEY

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CONVERSION FACTORS AND ABBREVIATIONS

Multiply	By	To obtain
centimeter	3.94×10^{-1}	inch
micrometer	3.94×10^{-5}	inch
milliliter	2.64×10^{-4}	gallon

Degree Celsius (°C) may be converted to degree Fahrenheit (°F) by using the following equation:

$$°F = 9/5\,(°C) + 32.$$

ABBREVIATIONS AND ACRONYMS

ASR	Analytical Services Request form
ASTM	American Society for Testing and Materials
BAS	Branch of Analytical Services
COC	chain of custody
DODEC	U.S. Department of Defense Environmental Contamination program
LIMS	Laboratory Information Management System
NIST	National Institute for Testing and Materials
NWIS-I	National Water Information System-I
NWQL	National Water Quality Laboratory
QA	quality assurance
QA/QC	quality assurance/quality control
QAU	Quality Assurance Unit
QC	quality control
QWSU	Quality Water Services Unit, Ocala, Fla.
SOP	Standard Operating Procedure
USEPA	U.S. Environmental Protection Agency
USGS	U.S. Geological Survey
WATSTORE	Water Data Storage and Retrieval System
WRD	Water Resources Division

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ABSTRACT

Quality-control practices for the operation of the U.S. Geological Survey's National Water Quality Laboratory are described. These practices specify how samples are preserved, shipped, and analyzed, and how quality is assured at the Laboratory. This manual is a revision of the 1992 Quality Assurance/Quality Control Manual and documents the practices that are currently (1995) used at the Laboratory.

1.0 INTRODUCTION

The National Water Quality Laboratory (NWQL) produces analytical data for the U.S. Geological Survey (USGS) to establish the trend, fate, and effects of environmental inorganic constituents and organic compounds in water, sediment, and biological tissue. Increasing concerns about water quality and about the possible effects of toxic chemicals at trace and ultratrace levels have contributed to the need for impartial, objective, and independent data, which can be used to assess impact in the aquatic environment.

To meet these needs as a service laboratory, the NWQL provides expertise in a variety of analytical methods. Data need to reflect the true identification and quantification of analytes in environmental matrices if they are to be interpreted correctly. The general quality-assurance (QA) policy and procedures for analytical activities performed by the NWQL are outlined in this manual.

1.1 Purpose and Scope

The purpose of this manual is to identify and document practices and standard operating procedures (SOPs) for those activities of the NWQL that affect quality of data. The primary objectives are as follows:

- To provide NWQL personnel and customers with general descriptions of quality practices from the time of receipt of sample to reporting of results.
- To describe the QA practices and performance of the NWQL. Following these practices ensures that personnel are trained to operate instruments that are properly calibrated, controlled, and maintained.

Although NWQL quality-control activities and assessments are primarily directed by the Quality Management Program, which reports directly to the chief of the NWQL, a successful quality-management program requires active and enthusiastic participation by all Laboratory employees.

External quality-assurance review is a responsibility of the Branch of Technical Development and Quality Systems.

1.2 Mission Statement

The NWQL fulfills analytical requirements of the Water Resources Division by analyzing environmental samples for inorganic, organic, and radiochemical constituents. The NWQL strives to provide quality results in a timely, cost-effective manner. To meet established quality objectives and to support District water-quality investigations, the NWQL provides assistance for project planning and data interpretation. The NWQL also develops new analytical methods and sample-collection procedures as needed by the Division.

1.3 Quality Policy

The NWQL is committed to providing quality environmental-analytical services to USGS. An extensive QA program has been implemented to ensure the production of scientifically sound, legally defensible data of known and documentable quality. The effectiveness of this program relies on clearly defined objectives, well-documented procedures, and management support.

1.4 Organizational Structure

The chief, Branch of Analytical Services (BAS), (1) directs, manages, and coordinates a number of programs in support of water-quality analytical functions of the USGS; (2) plans and implements research activities leading to modification and development of analytical procedures, and a comprehensive quality-management program; and (3) oversees the daily operations of the NWQL, including inorganic and organic chemistry and support functions. An organization chart is shown in figure 1.

The Quality Management Program oversees the quality-assurance functions for the NWQL through the Quality Assurance, Biological Quality Assurance, U.S. Department of Defense Environmental Contamination program (DODEC), and Radiochemistry and Stable Isotope Units. The Quality Assurance Unit (QAU) carries out operations related to monitoring and improving the quality of NWQL analytical programs through audits, data reviews, customer support and communications, and training. Additionally, the QAU coordinates and maintains the NWQL certifications for various Federal and State environmental regulators who participate in the Federal-State Cooperative program. The remaining units in the Quality Management Program oversee the preparation and maintenance of outside laboratory contracts, monitoring the quality of contract laboratories through audits and data review, and providing customer support, communication, and documentation. In addition, the Biological Quality Assurance Unit performs special project-related analytical work on request. Moreover, the Radiochemical and Stable Isotope Unit determines gross alpha, gross beta, uranium, and radon at the NWQL.

The Methods Research and Development Program (1) conducts supportive basic and problem-oriented research in analytical and environmental chemistry to improve the basis for field investigations and laboratory measurement techniques; (2) evaluates and tests new technology in analytical methods for potential use in the USGS water-quality programs; (3) coordinates analytical-methods research and development throughout the USGS; (4) consults with USGS personnel on new methods of sample-collection processing and analysis and sets priorities for research needs.

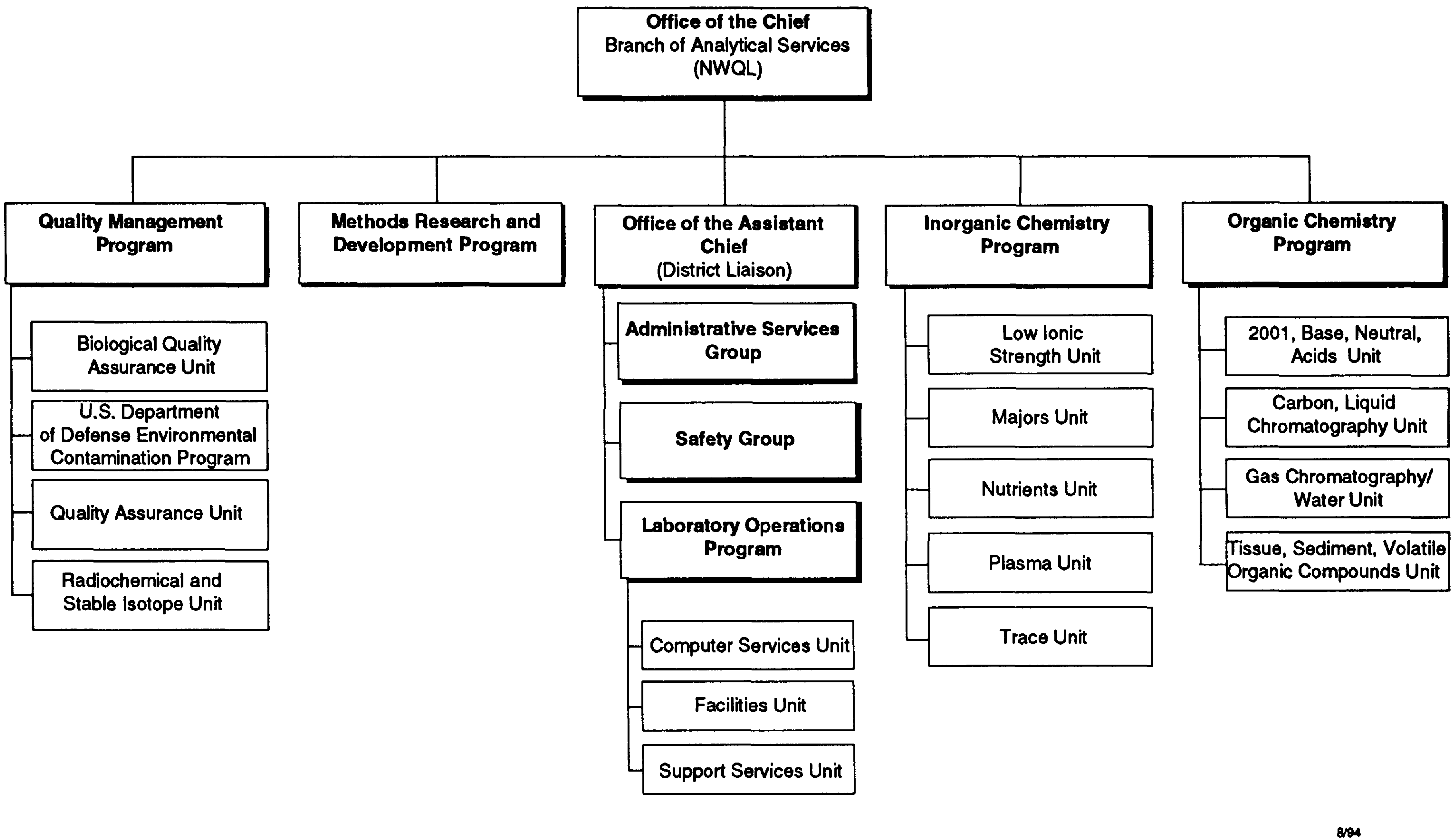

Figure 1.– Organizational chart of the National Water Quality Laboratory.

The assistant chief of the Branch is the District liaison for hydrologic programs and additionally oversees the following:

The Administrative Services Group provides a range of administrative support to management of the NWQL, including activities related to financial and fiscal analysis, budget and accounting, personnel management, procurement, office services, space and facilities, travel, and payroll.

The Safety Group (1) designs and administers programs for health and safety, chemical hygiene, hazard communication, environmental compliance, medical surveillance, indoor air-quality monitoring, hazardous waste management, emergency response, and training; (2) counsels management on matters of safety; and (3) manages security for NWQL grounds and facilities.

The Laboratory Operations Program provides support to the Branch chief through the management of facility operations and oversees the Computer Services and Support Services Units.

The Computer Services Unit (1) maintains the NWQL computer systems; (2) maintains the computer data base and software applications designed to operate QC programs and bill customers; and (3) transfers analytical data to customers.

The Support Services Unit performs the daily-support activities required at the NWQL through (1) receipt and distribution of equipment and orders; (2) maintenance of a warehouse stocked with required chemicals and supplies; (3) cleaning of laboratory glassware; (4) distribution of field QA supplies used for the collection of water-quality samples; and (5) receipt, verification, and distribution of all samples sent to the NWQL for analytical services.

The Inorganic and Organic Chemistry Programs (1) systematically carry out operational laboratory activities for analysis of inorganic constituents and organic compounds in water, sediment, and biological-tissue samples; (2) provide general consultation and scientific advice to USGS personnel and cooperators throughout the Nation on the determination of inorganic constituents and organic compounds, methodology, and automation techniques; and (3) define problems and develop and implement proposals to meet future analytical needs.

1.5 Quality-Assurance Publications

This manual describes the current (1995) QA practices and standards of the NWQL and supersedes the QA/QC Manual by Pritt and Raese (1992). This manual will be reviewed every 2 years and updated as needed to reflect the changes in quality-assurance and quality-control practices, organization, staffing, and data-management system at the NWQL. Pratt (1994) describes the analytical methods commonly used by the NWQL and provides guidance for selecting appropriate analytical methods in project planning, particularly for cooperative projects involving U.S. Environmental Protection Agency or State-regulatory requirements. The USGS also has many other publications that describe quality-assurance practices for sample collection and laboratory analysis. Selected references and a brief explanation of their content follow.

Janzer (1985) and Erdmann (1991a, 1991b) explain the Standard Reference Water Sample program and describe the laboratory-review process. In addition, biannual Open-File Reports describe the USGS analytical evaluation program for Standard Reference Water Samples for trace constituents, major constituents, nutrients, mercury, and low ionic-strength analytes (Long and Farrar, 1994a, 1994b). Friedman and Erdmann (1982) describe QA practices for chemical and biological analyses of water and fluvial sediments.

Also included are references to several administrative memoranda that deal with analytical methods or water-quality policy. In practice, such documents are not cited in USGS publications because they are not available to all readers. However, in this case, the editors of this report will make available copies of all cited memoranda. Readers who need copies of any technical memoranda are requested to write to the chief of the NWQL, providing the number and subject of the memorandum, or browse the NWQL Home Page on the World Wide Web (http://wwwnwql.cr.usgs.gov). For the convenience of the reader, all memoranda cited in this manual are listed as follows:

- Water Resources Division Memorandum 82.28, January 21, 1982
Subject: WATER QUALITY--Acceptability and Use of Water-Quality Analytical Methods
Author: L.B. Laird
- Water Resources Division Memorandum 91.09, November 28, 1990
Subject: PUBLICATIONS--Quality Systems Terminology
Author: V.R. Schneider
- Office of Water Quality Technical Memorandum 92.06, March 20, 1992
Subject: REPORTS--Report of Committee on Sample Shipping Integrity and Cost
Author: J.D. Broadus
- National Water Quality Laboratory Technical Memorandum 92.01, March 25, 1992
Subject: TECHNOLOGY TRANSFER--Availability of Equipment Blank Water for Inorganic and Organic Analyses
Author: P.F. Rogerson
- Office of Water Quality Technical Memorandum 93.05, January 21, 1993
Subject: PROGRAMS AND PLANS--Evaluation of Capsule Filters
Author D.A. Rickert
- National Water Quality Laboratory Technical Memorandum 94.07, February 10, 1994
Subject: QUALITY CONTROL--Description and Use by Districts of Laboratory QC Sample Information in Organic Determinations
Author: M.W. Sandstrom

2.0 GENERAL LABORATORY PRACTICES

This section includes a discussion of analytical methods, method validation, training, safety, and generally used materials and equipment. Also included is a description of data integrity and stability.

2.1 Analytical Methods

The Water Resources Division (WRD) has long maintained a policy of requiring analytical methods to be thoroughly documented, characterized for capability, reviewed, approved, and published prior to use (Fishman and others, 1994). These methods are categorized as follows: official, accepted, and special. The WRD official methods are validated, including precision and accuracy data, externally reviewed, and published in the Techniques of Water-Resources Investigations or Open-File Reports series of USGS publications. Provisional approval for forthcoming "official" methods may be given by the NWQL chief if the methods have been validated, including precision and accuracy data, and externally reviewed. These provisional methods are used while manuscripts are prepared for publication. At the time provisional status is given, a time limit will be set by the NWQL chief for publication of the report.

Accepted methods are those published by water-analysis authorities such as American Society for Testing and Materials (ASTM) (1994), U.S. Environmental Protection Agency (1982, 1988, 1992a), and Greenburg and others (1992). The NWQL demonstrates and documents proficiency to use non-USGS published methods. Results from accepted methods may be stored along with results collected from official methods (including provisional status) in the National Water Information System-I (NWIS-I) and in Water Data Storage and Retrieval System (WATSTORE), and published in USGS reports.

Methods that do not fall in the aforementioned categories or are experimental are called special methods. Special methods occasionally are requested by customers who have unique project requirements. Special methods are not covered by the same rigorous QA/QC practices described for official or accepted methods. Therefore, special methods may not have the level of validation as for official or accepted methods. Results from special methods will not be stored in the NWIS-I or WATSTORE data bases. Documentation and development records of special methods are kept by the analytical program that provided data for the project. The customer using the data is responsible for validating results obtained from special methods.

2.2 Method Validation

The USGS outlines policy for validating methods in WRD Memorandum 82.28 (L.B. Laird, U.S. Geological Survey, written commun., 1982), and subsequent revision by M.J. Fishman (U.S. Geological Survey, written commun., 1987) and B.E. Jones (U.S. Geological Survey, written commun., 1990). A standard operating procedure (SOP) describes procedures for documenting the following characteristics: method precision and accuracy; method detection limit; known and possible interferences; sample preservation and storage requirements; stability of reagents and standards; instrument performance; safety information; and method comparability to superseded methods, if any. Initially, for each method validation, a protocol is developed that describes the scope and approach for documenting method performance. Approved written methods then are developed into detailed SOPs that analysts follow.

2.3 Training

Supervisors are responsible for assuring that employees receive orientation, safety, and skills training. General training also is provided to expand the employees' capabilities in areas not directly related to the job. Topics such as employee relations, technical writing, and motivation are available to employees with their supervisor's approval. After general orientation, employees are introduced to the safety officer, and arrangements are made for any safety equipment that the new employee might need.

The Safety Group provides safety orientation to new employees within their first 30 days of hire, followed by continuing safety education. The employee orientation covers general safety issues, emergency procedures, standard-safety operations, the NWQL's chemical-hygiene plan, hazard communication, hazardous-waste management, waste disposal, the location of safety equipment, and a tour of the NWQL. General safety issues include a wide range of topics. Employees are informed of procedures on materials handling, transportation of chemicals, and hazardous waste-disposal procedures. Additional procedures specific to an employee's work group or unit are discussed by the supervisor. Employees are encouraged to continue safety training by completing annual classes in cardiopulmonary resuscitation and standard first aid. Training in the use of fire extinguishers is provided by the Arvada, Colorado, Fire Department.

Technical skills training is conducted from SOPs, published methods, and operation manuals. Supervisors document the following: trainer, trainee, type of analytical-method training, dates of training, and proficiency testing. Documentation is retained by the supervisor.

2.4 Safety

The Safety Group directs worker health and safety, waste management, security issues, and training. These responsibilities include air sampling, environmental compliance, and the testing of safety equipment at regular intervals. In addition to SOPs, Skinner and others (1983) discuss laboratory safety.

Air-sampling surveys are conducted as needed. Areas containing chemicals that pose serious health risks are monitored (1) annually, (2) on request, or (3) in potential exposure situations. Inhalation hazards, carcinogens, toxic materials, or other health hazards are monitored.

Safety equipment is tested at regular intervals. Safety showers and eyewashes are tested biannually. Fire extinguishers are checked annually and serviced as needed. Fume hoods are inspected annually for face-velocity measurement, hood adjustments, and for marking sash positions. Ventilation recommendations are made to improve engineering controls and systems.

Waste-disposal requests are processed by the Safety Group and conducted according to the SOP on waste management. This SOP complies with U.S. Environmental Protection Agency and State of Colorado hazardous-waste regulations. The disposal process is started by providing the Safety Group with a request for disposal. The request form requires information on the contents, concentration, pH, weight or volume, and source of the waste. The waste is segregated by physical characteristics, chemical reactivity, and waste stream. If necessary, the waste is pretreated.

The safety officer maintains a contract for disposal of radiochemical waste. Waste produced by the Radiochemical and Stable Isotope Unit is disposed of through this contract. Radiochemical waste is collected in a designated-waste barrel. A detailed list of waste contained in this barrel is maintained in the Radiochemical and Stable Isotope Unit files and is supplied to the Safety Group when the barrel is ready for disposal.

Visitors sign in and out of the building at the lobby, where they are provided with identification badges and safety glasses and any other personal-protective equipment necessary for entry into laboratory areas. Information on hazardous materials and chemical usage in the facility is provided. Minors will not be admitted to the NWQL without an adult escort.

Entry to the NWQL is by magnetic access card or keypad 24 hours a day. The cards are issued and maintained by the Safety Group. The NWQL is provided with security officers by the Federal Protective Service.

Documentation of the safety programs is maintained by various departments. Records of safety showers, fire extinguishers, eyewashes, emergency lights, fume-hood testing, waste manifests, and disposal requests are maintained by the Safety Group. Safety training documents are kept by Administrative Services Group and by the Safety Group.

2.5 General Laboratory Materials and Equipment

2.5.1 Deionized, Distilled, and Organic-Free Water

Two large-capacity, deionizing-water systems supply water that has a minimum resistivity of 12 megohms-centimeter to laboratory areas and the glassware-washing facility. Both deionizing systems are computer monitored for resistivity and water volume. The quality of water is further improved in the inorganic laboratory area by Millipore[1] or equivalent systems that supply reagent water with a minimum resistivity of 16.7 megohms-centimeter. The reagent water is used for preparing calibration standards, chemical reagents, and sample dilution.

Organic-free water (ASTM Type II) is prepared by carbon filtration followed by distillation. The system is located in the organic chemistry laboratory. Volatile organic-free water is prepared in small quantities from deionized water by boiling followed by nitrogen purge. Organic-free water and volatile organic-free water are tested at regular intervals by personnel in the organic chemistry laboratory.

2.5.2 Analytical Balances

Analytical balances are used for accurate weighing of samples, reagents, and calibration standards. The balances are cleaned and certified annually by a contract service technician. In addition, each balance is checked for calibration with class S weights certified by the National Institute for Standards and Technology (NIST). Calibration, maintenance, and use are documented in logbooks. Balances that fail calibration checks are labeled "out-of-calibration" and dated. An out-of-calibration balance is not used until repaired and re-certified by the contract-service technician.

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

2.5.3 Refrigerators and Freezers

Temperatures of refrigerators and freezers used for storing samples and reagents are monitored each workday. The temperature is recorded on a log sheet attached to each refrigerator and freezer. Log sheets are maintained in the laboratory section responsible for the refrigerator or freezer. Temperature is maintained nominally at 4°C with a tolerance range of ± 2°C for refrigerators. Temperature of freezers used for biological tissue and sediment samples is maintained at -10°C or lower. If the temperature exceeds these limits, the temperature-set point is readjusted until the temperature is within acceptable limits. Thermometers used to monitor refrigerator and freezer temperatures are verified against thermometers calibrated by NIST.

2.5.4 Ovens

Ovens are checked for temperature accuracy by use of an independently calibrated mercury thermometer. General-purpose thermometers are used for oven-temperature monitoring. Thermometers calibrated by NIST are used to verify accuracy of these general-purpose thermometers. Thermometers that are out of calibration are replaced. A temperature log is kept for each oven during use.

2.5.5 Glassware

Class A volumetric glassware is used for analytical work. Volumetric flasks and pipets are dedicated to specific uses to prevent cross contamination. Fixed and variable-volume micropipets often are used to prepare calibration standards and dilute samples. Micropipets regularly are calibrated with analytical balances or with an automatic calibrating spectrophotometer. Glassware is washed according to SOPs.

2.5.6 Fume Hoods

Laboratory-fume hoods are inspected and certified annually by a contract-industrial hygienist. Inspection reports are filed in the Safety Group. Maintenance personnel inspect and maintain hoods as needed. Roof-mounted blower assemblies are inspected every 6 months.

2.6 Data Integrity and Stability

2.6.1 Data Entry and Validation

Most of the analytical data produced by the NWQL are transferred electronically from the laboratory instruments to the in-house Laboratory Information Management System (LIMS) computer. Remaining analytical data are hand-entered by Computer Services Unit personnel. Manually entered data are verified through a "double-entry" routine designed to minimize keyboarding errors. Reports of analytical data that have been entered into the LIMS are produced daily and reviewed by appropriate personnel to ensure accurate data transfer.

2.6.2 Data Reporting

Analytical data for routine determinations are compiled on the LIMS computer in subgroups (subsamples), and, when completed, are released and electronically transferred to the Colorado District PRIME computer for distribution to the customer. The customer is responsible for retrieving and entering these data into the NWIS-I and WATSTORE data bases.

2.6.3 Computer, Data-Base Security, and Software Validation

Data backups on the in-house LIMS computer are as follows:

- The data base is backed up twice-daily Monday through Friday and is kept for 1 week.
- Work files are backed up once-daily Monday through Friday and are kept for 1 week.
- Data base and files are archived for off-site storage on the first Thursday and Friday of each month.

In addition to the aforementioned backups, transaction logs run 24 hours a day, 7 days a week, recording changes to the LIMS data base. These logs can be used in case of a system crash to roll the data base forward since the last backup. The data base and systems on the PRIME and Data General computers are backed up according to a written schedule.

Both computer systems reside in controlled-access areas that are environmentally and electronically monitored. The computer rooms have combination locks that are changed routinely. Data bases have controlled access which vary according to individual needs. Employees receive computer security-awareness training. Software that affects analytical results -- either vendor supplied, developed in-house, or other software -- is tested and compared to other current methods for producing data.

3.0 SAMPLE MANAGEMENT

Sample containers, field reagents, and sample handling are discussed in the following section.

3.1 Sample Containers and Field Reagents

3.1.1 Sample containers

Sample containers are processed (cleaned) and stored at the Ocala Quality Water Service Unit (QWSU) and are supplied to USGS field offices upon request to QWSU. The type of container to be used is specified in the NWQL Services Catalog (Timme, 1995, p. 7). The NWQL does not accept samples in containers other than those specified by WRD. Containers received from suppliers are quality checked by QAU according to standard procedures. Statistically based sampling procedures are used to obtain a sample from a shipment of containers (Grant and Leavenworth, 1988). Contamination testing is by the lowest detection methodology for constituents normally determined for a specific container (Timme, 1995). Records of container testing are maintained by the QAU. The NWQL also issues certificates of analysis on all containers that have undergone QC testing at the NWQL, and the appropriate certificates are shipped with the requested items.

3.1.2 Preservatives

Sample-preservation chemicals in ampules are supplied to USGS field offices by the QWSU. The types of preservatives and their correct use are described in the NWQL Services Catalog (Timme, 1995, p. 15). Preservatives are prepared by contract suppliers in accordance with WRD specifications. Preservatives are checked for quality prior to distribution in accordance with written SOPs. Records of accepted preservatives are maintained by the QAU. The NWQL issues certificates of analysis for all preservatives that have been tested.

3.1.3 Organic-Matrix Spike

Organic field-matrix spikes are a type of project-submitted QA sample that provides accuracy and precision information for samples analyzed for organic compounds. The data-quality objectives for each field project described in the project QA plan determine the number and type of matrix spikes. Organic compound-spike solutions, fixed-volume micropipets, and instructions for their use are available from NWQL. The spike solutions of organic compounds are prepared in numbered lots by commercial suppliers, according to WRD specifications. Each lot of the spike solution is randomly sampled and analyzed prior to acceptance and distribution. Test-data files are maintained by the QAU.

3.1.4 Field-Equipment Blanks

Field-equipment blanks are a type of project-submitted QA sample that provides information about the validity of samples collected by documenting that samples have not been contaminated or biased during collection and handling. The data-quality objectives of each field project described in the project QA plan determine the number and type of field-equipment blanks. Procedures for obtaining and using field-equipment blanks are described in NWQL Technical Memorandum 92.01 (P.F. Rogerson, U.S. Geological Survey, written commun., 1992). (See section 1.5 of this manual for access to memoranda.) Different types of water to be used for field-equipment blanks are available through the NWQL or the QWSU Laboratory in Ocala, Florida, depending on the types of sample analyses. These types are ASTM Type I water (for inorganic use), ASTM Type II water (organic-free), and volatile organic compound-free water. The water is prepared in numbered lots either in the NWQL, the QWSU Laboratory in Ocala, or by commercial suppliers. Each lot of the equipment blank water is tested according to SOPs prior to acceptance and distribution. Test-data files are maintained by the QAU.

3.1.5 Filters

Filters for processing samples collected in the field are supplied through the QWSU Laboratory in Ocala. The type of filter to be used for organic determinations is described by Sandstrom (1995). The type of filter to be used for inorganic determinations is described in the Office of Water Quality Technical Memorandum 93.05 (D.A. Rickert, U.S. Geological Survey, written commun., 1993). Each lot of filters is tested, as specified in SOPs, prior to acceptance and distribution. Test-data files are maintained by the QAU.

3.1.6 Sample Documentation and Shipping Requirements

When field-office personnel collect water samples, samples are placed in the appropriate bottles with the necessary preservatives, and an Analytical Services Request (ASR) form is submitted (fig. 2). All requests for analysis must be submitted using this form or a computerized version of the form. Writing requests for analytical work directly on the bottles is not acceptable. However, labeling for potentially hazardous samples should be indicated directly on the bottle. The following categories must be filled out when requesting sample analysis: station identification, State, District-user code, project account number, beginning date of sampling, schedules, field and laboratory codes, exact time of sampling for radon-222, milliliters of sample filtered for suspended organic carbon or chlorophyl A, and special handling for potentially hazardous samples. Additional information that will help the customer to receive improved service from the NWQL includes the following: name and phone numbers of shipper and project chief, number of bottles and packages shipped, bottle types, and identification of special samples. Specifications for shipping containers, preparing samples for shipment, preparing ASR forms, and packaging of samples are described in Office of Water Quality Technical Memorandum 92.06 (J.D. Broadus, U.S. Geological Survey, written commun., 1992).

3.2 Sample Handling

3.2.1 Log-In

Samples and the accompanying ASR forms are received by the Support Services Unit. Each sample and associated ASR form are assigned a unique laboratory-identification number that encodes the date of receipt and a serial number. The identification number is printed on barcode labels that are affixed to sample containers. A sample-type barcode label [filtered acidified (FA), for example] also is placed on the bottle. The various types of containers received for each sample are recorded, and each container is routed to the appropriate laboratory section for analysis. Temperature-sensitive samples are logged and refrigerated within 30 minutes of unpacking. If temperature-record cards are supplied with the samples, the temperature is recorded and returned to the sender. Irregularities detected by the Support Services Unit in sample shipping and preservation are noted on the ASR form.

U.S. GEOLOGICAL SURVEY - NATIONAL WATER-QUALITY LABORATORY
ANALYTICAL SERVICES REQUEST FORM

SMS CONTROL NO	NWIS RECORD NO	LABORATORY ID	LAB USE ONLY	SAMPLE SET

STATION NAME ________ *|__|__|__|__|__|__|__|__|__|__|__|__|__|__|__| STATION ID OR UNIQUE NO.

FIELD OFFICE ________ *PHONE NO. ________ *PROJECT CHIEF ________ FIELD SAMPLE ID ________ SITE TYPE ________

*|__|__| STATE *|__|__|__| DISTRICT/USER |__|__|__| CNTY *|__|__|__|__|__|__|__|__|__| PROJECT ACCOUNT

	YEAR	MONTH	DAY	TIME
*BEGIN DATE:	|1|9|__|__|	|__|__|	|__|__|	|__|__|__|__|
END DATE:	|1|9|__|__|	|__|__|	|__|__|	|__|__|__|__|

SCHEDULES, FIELD, AND LABORATORY CODES

SCHEDULE 1:________ **SAMPLE MEDIUM:________ **SAMPLE TYPE:________
SCHEDULE 2:________ GEOLOGIC UNIT:________ **HYDRO EVENT:________
SCHEDULE 3:________ **ANALYSIS STATUS:________
SCHEDULE 4:________ **ANALYSIS SOURCE:________
SCHEDULE 5:________ **HYDRO CONDITION:________

	A/D		A/D		A/D		A/D
CODE:____	____	CODE:____	____	CODE:____	____	CODE:____	____
CODE:____	____	CODE:____	____	CODE:____	____	CODE:____	____
CODE:____	____	CODE:____	____	CODE:____	____	CODE:____	____
CODE:____	____	CODE:____	____	CODE:____	____	CODE:____	____

FIELD VALUES

LAB/P CODE	VALUE	RMK	LAB/P CODE	VALUE	RMK	LAB/P CODE	VALUE	RMK
__21/00095	____	____	__51/00400	____	____	___2/39086	____	____
1171/00452	____	____	1170/00453	____	____			

+COMMENTS (Limit to 138 characters):________

LOGIN COMMENTS:________

SHIPPED BY:________ PHONE NO.:________ DATE:___|___|___

BOTTLE TYPES (PLEASE FILL IN NO. OF TYPES SENT)

___FA	___RU	___FU	___FAM	___RAM	___RCC	___FCC	___FAB	___CU
___RA	___RAH	___CN-	___RCB	___TOC	___DOC	___SOC	___COD	___VOA
___CHY	___O&G	___GCC	___RCA	___FCA	___PHENOL	________OTHER		

CUSTOM/SPECIAL SAMPLE APPROVED BY:________ APPROVAL NO.________
PROGRAM/PROJECT: ___NPDES ___NAWQA ___DRINKING WATER FILL IN OTHER________
POSSIBLE HAZARDS________

Revised 11/94 +Comments to be stored by the laboratory *Mandatory for acceptance for laboratory analysis
**Mandatory for storage in WATSTORE/NWIS

Figure 2. – National Water Quality Laboratory Analytical Services Request Form.

The ASR form is routed to Computer Services Unit, which enters sample information and analytical requests into the LIMS data base. After entry in the data base, the information on the ASR is converted daily to a series of NWQL workload requests. During log-in, each bottle received is recorded on an inventory form. Copies of these forms are distributed to the analytical units and to QAU for verification.

Radiochemical and stable isotope samples are received by the Support Services Unit and entered into the LIMS data base. The Radiochemical and Stable Isotope Unit maintains a separate data base to track analyses performed by the various contract laboratories.

3.2.2 Sample Control/Chain of Custody

Customers can arrange for the desired chain-of-custody (COC) level required for the project prior to submission of samples. Base-level COC for samples received at NWQL require no further documentation other than the ASR form. The samples are securely housed in an access-controlled and locked laboratory. If project requirements specify more rigorous COC, second and third levels are available. The second level is used to document sample transfer from the sampling site to the NWQL via a customer-supplied COC form. The completed form is returned to the customer after receipt and inspection of samples. The third level of COC precisely documents and controls samples in the Laboratory at all times from receipt until disposal. When samples arrive at the NWQL, receiving personnel inspect the shipping container, verify samples with the information on the ASR forms, document discrepancies, and sign and return the COC form accepting receipt of the samples. A "Y" in the hazard-code field on the ASR form notifies log-in personnel to use the third level of Laboratory COC process. The tag form for the U.S. Environmental Protection Agency (USEPA) is completed, if present, and a bar-code label is affixed to the tag and the sample containers. Samples are released by log-in personnel to the COC sample custodian, who maintains control of samples in a locked and controlled-storage area. Samples are checked out only after Laboratory personnel complete an internal COC form.

3.2.3 Sample Storage and Disposal

Samples that require chilling are stored in refrigerators in laboratory areas and in the sample warehouse. Refrigerators are kept at $4^{o}C \pm 2^{o}C$ except where otherwise indicated. Sediment and tissue samples are stored in freezers at $-10^{o}C$ or lower.

Samples analyzed for inorganic constituents except nutrients are disposed no earlier than 180 days after receipt. Analyzed nutrient samples are disposed no earlier than 30 days after receipt. Extracts of samples analyzed for selected organic compounds are archived. Contact the Organic Chemistry Program chief for information about which schedules have extracts archived and stability of extracts. In all cases, samples are disposed according to procedures defined by the Safety Group.

Samples for analysis of most radiochemical and stable isotope constituents are stored until they are shipped to the appropriate commercial or USGS laboratory. Nitrogen-15 samples are stored in a Support Services Unit refrigerator until shipment. Contract samples are shipped in accordance with the SOP for sample shipping and receiving. A list of samples showing date mailed and requested analyses is forwarded, in a separate envelope, to the contract laboratory. A copy of the list is kept in the Radiochemical and Stable Isotope Unit files.

3.2.4 Holding Times

The USEPA defines holding times for regulated inorganic constituents and organic compounds. Depending on environmental regulation, the legal holding-time requirements change (Wagner and Yogis, 1992). For projects that demand holding-time requirements that meet USEPA regulations, arrangements can be made with the NWQL before samples are submitted for analysis.

The average turnaround times for NWQL sample submission for summer 1994 are listed in table 1. Turnaround time is defined as the number of days between date sample was logged by the NWQL and date sample data were released from LIMS. This turnaround time does not include time for data review and reanalysis. The turnaround times represent actual performance of the NWQL and neither represent goals nor affect the data quality.

Table 1. -- *Average turnaround times for types of analysis and matrix*
[N/A, not applicable]

	Type of matrix (days)		
Type of analysis	Water	Sediment	Biological tissue
Trace elements only	30	[1] 90-180	[2] 45-60
Major ions only	30	N/A	N/A
Nutrients only	14	21	N/A
Acid-base/neutral extractables	[3] 30-90	[4] 180	N/A
Pesticides	[5] 30-60	[6] 180	[7] 180
Volatile organic compounds	14-30	N/A	N/A
Combined trace elements, major ions, nutrients	30	N/A	N/A
Radon (samples analyzed within 24 hours)	7	N/A	N/A
Gross alpha/beta and uranium	30	N/A	N/A

[1] Based on schedule 2400 (performed by USGS Branch of Geochemistry).
[2] Based on schedule 2200.
[3] Based on schedule 1383.
[4] Based on schedule 2502.
[5] Based on schedule 1324.
[6] Based on schedule 2501.
[7] Based on schedule 2101.

3.2.5 Sample Safety

Analysts are alerted to potential hazards associated with any samples by use of special handling and status codes printed on workfiles. Potentially hazardous samples must be labeled as such on the bottles and documented on the ASR form by customers.

4.0 INORGANIC ANALYSIS

Inorganic analysis is organized as follows under documentation, reagent preparation, sample preparation and storage, instrument calibration and maintenance, inorganic QC, and data reporting.

4.1 Documentation

Routine analyses are performed according to methods and associated SOPs described by Brown and McLain (1994), Damrau (1993), Faires (1993), Fishman (1993), Fishman and Friedman (1989), McLain (1993), and Patton and Truitt (1992). Published methods are distributed throughout the NWQL and are available to analysts.

Logbooks are used by analysts to record analytical information and instrument conditions. Logbooks are reviewed by supervisors or senior analysts. Several different types of logbooks are kept: instrument maintenance, analytical conditions, special samples, and calibration. Changes to an SOP are approved by the program chief and then are documented and recorded prior to implementation. Current SOPs are available to analysts. The SOPs are filed in the appropriate unit and in the QAU. Original analytical data such as strip-chart recordings are stored at the NWQL for 3 years with the date of analysis for reference and then archived.

4.2 Reagent Preparation

Reagents are prepared according to methods and associated SOPs described by Brown and McLain (1994), Damrau (1993), Faires (1993), Fishman (1993), Fishman and Friedman (1989), McLain (1993), and Patton and Truitt (1992). Reagents and standard solutions are labeled to indicate date last prepared. Labels include preparer's name and concentration of reagent. Standard solutions are prepared at least annually. Standard solutions are cross checked using alternative methods to verify accuracy to within 2 percent of the desired concentration. Documentation for standard solutions is filed in the unit. Reagents and standard solutions are made with reagent water (see section 2.5.1). Class A volumetric glassware is used to prepare calibration standard solutions and critical reagents. Chemicals are labeled with date upon receipt. An annual inventory is prepared and filed by the Safety Group.

4.3 Sample Preparation and Storage

Samples requiring treatment prior to analysis are prepared according to Brown and McLain (1994), Damrau (1993), Faires (1993), Fishman (1993), Fishman and Friedman (1989), McLain (1993), and Patton and Truitt (1992) with supplemental instructions provided in SOPs. Dilutions are made volumetrically, and in cases where automatic dilutors are used, the dilutors are calibrated monthly. Refer to section 3.2.3 for sample storage and disposal.

4.4 Instrument Calibration and Maintenance

Instruments are calibrated prior to analysis. Depending on instrumentation, calibration curves might contain as few as one calibration standard (inductively coupled plasma–atomic emission spectrometry) or as many as seven calibration standards (atomic absorption spectrophotometry). Data-acceptance criteria are developed on the basis of instrument performance and calibration-curve characteristics and are stated in SOPs for each method.

Instruments are maintained by NWQL analytical personnel. Routine and specialized maintenance are recorded in the instrument-maintenance logbook, which is monitored by the section chief or designated alternate. Occasionally, a factory-trained service technician may be called to maintain or repair an instrument.

4.5 Inorganic Quality Control

Quality-control samples are analyzed at a minimum of 1 in every 10 samples. These QC samples include at least one of the following: Standard Reference Water Samples provided by the Branch of Technical Development and Quality Systems, duplicate samples, standard solutions, blanks, or spikes. The QC data are reviewed following completion of an analysis set. First the analyst checks correlation coefficients for calibration curves. Acceptable correlation coefficients vary depending on the analysis, with most of the analytical methods required to meet a correlation coefficient of at least 0.99. If Standard Reference Water Samples are available, the analyst reviews results to ensure that they fall within 1.5 standard deviations of the mean value. The analyst checks blank, spike, duplicate, and standard solutions to be sure they fall within acceptance criteria. If all data-acceptance criteria in SOPs are met, then the analytical data are acceptable. The analytical instrument is shut down and recalibrated if QC samples fail to meet acceptance criteria. Quality-control charts, produced monthly from Standard Reference Water Sample data, are reviewed by the analyst and unit supervisors to ensure continued acceptable performance for each analytical method.

4.6 Data Reporting

Upon acceptance of analytical data by the analyst, results are submitted to Computer Services Unit. Original analytical data are stored at the NWQL for 3 years and then archived.

5.0 ORGANIC ANALYSIS

Organic analysis is organized as follows under documentation, reagent preparation, sample preparation and storage, instrument calibration and maintenance, organic quality control, and data reporting.

5.1 Documentation

Routine analyses are performed according to methods and associated SOPs described by Brenton and Arnett (1993), Burkhardt and others (1995), Fishman (1993), Foreman and others (1995), Leiker and others (1994), Lindley and others (1994), Markovchick and others (1994), Rose and Schroeder (1995), Sandstrom and others (1992), Werner and Johnson (1994), Wershaw and others (1987), and Zaugg and others (1995). Published methods are distributed throughout the NWQL and are available to analysts. SOPs are centrally filed in the unit supervisor's office, where they are dated and approved by the program chief; copies also are filed in the QAU office. Where possible, original data are stored on magnetic media, and printed copies are filed in archival boxes.

Laboratory notebooks are used by each analyst to record analytical data and instrument conditions. Logbooks are reviewed quarterly, initialed, and dated by unit chiefs for accuracy, completeness, and timeliness.

Analytical data (for example, date of extraction, concentration, and cleanup), chromatograms, report sheets, copies of ASR forms, and preparation notes are filed by set. A set is a batch of samples, a blank-control sample, and a spike-control sample. Sets are prepared for analysis, filed by schedule number, and indexed by Julian date.

Quality-control charts are maintained for several compounds defined by the U.S. Environmental Protection Agency (1992b, 1992c) Primary and Secondary Drinking-Water Regulations, and for surrogates. Charts are reviewed by unit supervisors, chemists, and sample-preparation analysts.

5.2 Reagent Preparation

Reagents are prepared according to methods and associated SOPs described by Brenton and Arnett (1993), Burkhardt and others (1995), Fishman (1993), Foreman and others (1995), Leiker and others (1994), Lindley and others (1994), Markovchick and others (1994), Rose and Schroeder (1995), Sandstrom and others (1992), Werner and Johnson (1994), Wershaw and others (1987), and Zaugg and others (1995).

Reagents are labeled with date prepared and preparer's initials. Reagents are stored in containers and in an appropriate atmosphere to reduce adsorption of water or contaminants. Storage times for reagents are listed in published reports or in SOPs. An annual inventory is prepared and filed by the Safety Group.

Class A volumetric glassware is used in the preparation of standard solutions and in sample dilution where volume exceeds 10 milliliters. Sample preparation glassware is washed and heated to 450^{o}C to remove residual organic compounds.

Pesticide residue-grade quality, or better, solvents are purchased to produce chromatograms with minimal interference. Each lot of solvent is tested to ensure that organic compounds are not interfering at method-detection levels. Solvents are pre-concentrated in accordance with sample-preparation methods before testing. Distilled water used for blanks and spikes is checked for purity using methylene chloride and hexane extraction techniques. Maintenance records for distillation are kept by the Facilities Unit.

Reagents are purchased in the highest purity when certified reagents are not available. Ultrapure reagents are used to prepare stocks when solutions are unavailable or concentrations are inadequate. Stock-solution concentrations are corrected for purity when less than 97 percent.

5.3 Sample Preparation and Storage

First, water samples are transferred from the Support Services Unit, where they are logged, to sample preparation. The samples are extracted within 7-working days. Samples are preserved with extraction solvent, or in the case of phenoxy-herbicide samples, with concentrated nitric acid. Samples are prepared according to methods and associated SOPs described by Brenton and Arnett (1993), Burkhardt and others (1995), Fishman (1993), Foreman and others (1995), Leiker and others (1994), Lindley and others (1994), Markovchick and others (1994), Rose and Schroeder (1995), Sandstrom and others (1992), Werner and Johnson (1994), Wershaw and others (1987), and Zaugg and others (1995). The National Water-Quality Assessment program (NAWQA) sediments are transferred from the Support Services Unit to organic preparation and immediately frozen at -10°C. The non-NAWQA sediments are transferred from the Support Services Unit to organic preparation and immediately refrigerated at 4°C. All sediment samples are prepared when there are enough samples to form a set.

Samples awaiting preparation are refrigerated in original containers. Sample extracts also are refrigerated at 4°C. The entire water sample is consumed in the extraction process except for samples analyzed for carbon, methylene blue active substances, phenols, sediments, and volatile compounds. Sample extracts are retained until the sample has cleared internal review.

5.4 Instrument Calibration and Maintenance

Instruments are calibrated by analysis type according to the appropriate SOP. For gas-chromatographic methods in which a mass spectrometer is not used as a detector, at least three external standards are used for calibration. Internal standards are used in all gas-chromatographic procedures in which a mass spectrometer is the detector. Instruments are calibrated after maintenance or replacement of columns, and after previous calibration or check samples exceed the acceptance range described in the individual SOPs.

Newly prepared standard solutions are verified against existing standard solutions or against vendor-supplied standard solutions, or both. The acceptance criteria for satisfactory verification are listed in all methods or SOPs. Standard solutions are accepted when they meet appropriate specifications.

Maintenance is required when instrument performance does not meet specifications described in the SOP or on a schedule recommended by the vendor. Maintenance records are kept in a logbook. Routine maintenance is handled by the analyst. Most instruments are under contract for major service.

5.5 Organic Quality Control

A detailed discussion of organic QC sample information is available in NWQL Technical Memorandum 94.07 (M.W. Sandstrom, U.S. Geological Survey, written commun., 1994). (See section 1.5 of this manual for access to memoranda.) Each new lot of reagents is tested for contamination prior to use. The reagents are taken through the appropriate procedure and repurified if results show interference on the chromatograms. Method blanks (see Glossary) are prepared for each sample set and processed in the same manner as the samples. If contamination is observed in the method blank, a reagent-only blank is prepared to determine the contamination source. Once the source of contamination is determined, corrective action is taken. Examples of corrective action include repurification or replacement of the reagent.

Reagent spikes (see Glossary) are prepared for each sample set and processed in the same manner as the samples. The reagent-spike solutions are used to verify the method-performance accuracy for each sample set. Over time, analysis of many reagent-spike solutions also provides method-performance precision. Control charts of reagent spike data are used by the NWQL to indicate trends, variability, and precision of analyses.

Operator QC includes the following where applicable: previous calibrations, method-detection-level verifications, calibration-check samples, system performance-check samples, tuning criteria, method blanks, reagent spikes, and surrogate-compound recoveries.

5.6 Data Reporting

Data are reported according to procedures described in SOPs. The information is manually transcribed onto preprinted data-report forms. Another analyst independently checks the data prior to entry into the computer data base. Data are mailed to the customer when it is not possible to enter the information into NWIS-I or when QC criteria are not met and data qualifiers need to be supplied.

Data are not reported when the sample was ruined during preparation or analysis. When surrogate recovery does not meet acceptable criteria, as described in the appropriate method or SOP, the customer is notified that the data need to be qualified.

6.0 RADIOCHEMICAL AND STABLE ISOTOPE ANALYSIS

Radiochemical and stable isotope analysis is organized as follows under contracts and in-house analysis.

6.1 Contracts

The Radiochemical and Stable Isotope Unit contracts with commercial and other USGS laboratories for radiochemical and stable isotope determinations.

6.1.1 Documentation

A copy of each commercial contract and USGS laboratory agreement is kept on file in the Radiochemical and Stable Isotope Unit. A notebook containing information regarding radiochemical QC sample preparation is maintained. The information recorded in this notebook includes the USEPA radioisotope standard that was used to prepare the QC sample, the initial decay date, and the radioactivity. A notebook is maintained for radiochemical QC samples that are sent to contract laboratories. The notebook contains the expected and actual concentrations of the QC samples, reanalysis results, and sample duplicates. A notebook is maintained for each stable isotope, which contains methods of QC sample preparation, expected and actual concentration of the QC sample, and reanalysis results. Moreover, QA manuals from commercial and other USGS laboratories that are used to analyze radiochemical and stable isotope samples are kept on file in the Radiochemical and Stable Isotope Unit.

6.1.2. Radiochemical and Stable Isotope Contract Determinations

Commercial and other USGS laboratories are reviewed to ensure compliance with contracts. Contract laboratory results are monitored using the USEPA cross-check program. Blind QC samples are prepared and submitted for most constituents in a wide range of concentrations using USEPA standards, deionized water, and reagent-grade nitric acid.

Duplicate samples provided by the USGS Districts are submitted to the contract laboratory. Every shipment of samples contains between 5 and 10 percent QC samples (except carbon-14 for which there are no available QC samples). The QC samples consist of a mix of blind duplicate and prepared QC samples. Reanalyses are obtained for QC samples that do not agree within the limits defined by the contract (2 standard deviations). If the QC sample indicates a bias in a particular constituent, further analyses are halted pending resolution of the bias. Reanalyses are requested for samples analyzed in that batch of samples showing a bias.

6.2 In-House Analysis

Currently (1995), the Radiochemical and Stable Isotope Unit analyzes radon (^{222}Rn), uranium, gross alpha, and gross beta at its in-house laboratory.

6.2.1 Documentation

A logbook that contains information regarding every sample analyzed by the Radiochemistry and Stable Isotope Unit is kept beside the instruments. Analysts write the sample-identification number, collection date, analysis date, background levels, and standard concentrations in the logbook. Each instrument has a maintenance logbook that details problems, date of maintenance, and action taken. Logbooks contain monthly plots depicting background levels, standard concentrations, and standard deviations of those measurements for each instrument. Copies of SOPs are kept in the unit chief's office and beside the instruments. The USEPA's radium (^{226}Ra) standard-data sheets are filed. The sheets are dated as to when the standards are received by the unit and when the standard ampule is opened.

6.2.2 Reagent Preparation

Reagents and standard solutions are prepared according to methods described in SOPs. Bottles of reagents and standard solutions are dated when they are received and when they are opened. The ^{226}Ra calibration standard is provided by the USEPA. Radiological standards are inventoried monthly. This list is kept in the unit files with a copy given to the NWQL Safety Group and the Central Region Radiological Safety Committee, which oversees the licensing of nuclear materials at the Laboratory.

6.2.3 Sample Preparation

The 20-milliliter liquid scintillation vials with poly-seal caps are used to prepare samples. Since ^{222}Rn has a half-life of 3.82 days, samples are placed in the counters the same day that they arrive at the NWQL.

6.2.4 Instrument Calibration and Maintenance

Each liquid scintillator is calibrated daily using an internal cesium-137 source and a tritium or a carbon-14 standard. The calibration ensures that the counting window will have the optimum efficiency for radon samples. The liquid scintillators are maintained by a service contract that includes emergency service and preventive maintenance, with all information recorded in a logbook.

6.2.5 Radiochemistry Quality Control

A blank is analyzed after every 10 samples. For radon the USEPA ^{226}Ra standards (three of varying activities) are analyzed once a day on each instrument. For gross alpha and gross beta, a 100-minute background count is obtained for every detector each working day. The background count is used for calculating results for each day. The background results are recorded in the instrument logbooks. Each month the control charts of the background counts are updated. The criteria for acceptable background levels are less than or equal to 2 counts per minute for gross beta and less than or equal to 0.1 count per minute for gross alpha. If background counts are greater than these acceptable levels, samples will not be analyzed on the specific instrument and detector until corrective action brings the background level to within acceptable limits.

For gross alpha and gross beta determinations, efficiency checks are performed daily on each instrument that is used for analysis. Efficiencies are recorded in the instrument logbooks. Monthly control charts are prepared using the efficiency data. Instrument counting efficiencies are acceptable if the efficiency is not greater than $\pm$ 2 sigma of the average value. If the efficiency exceeds this level, the standards are recounted. If the second efficiency count exceeds $\pm$ 2 sigma, a different standard is analyzed. If the second standard also fails the acceptance criteria, the instrument is recalibrated. Samples are not analyzed until the problem is resolved and efficiencies are within acceptable limits.

A minimum of 1 in 10 samples is randomly selected for a duplicate analysis and positioned near the beginning and ending of the sample-analysis set. The duplicate results must be less than or equal to $\pm$ 2 sigma on the basis of the counting statistics calculated for the sample. If the results are outside the limits, then corrective action is taken. As part of problem resolution some samples in the set may need to be reanalyzed.

Quality control for uranium determinations includes analysis of either a QC sample or duplicate in every batch of 5 to 15 samples. An independent laboratory checks 5 percent of the samples analyzed by the NWQL, serving as an interlaboratory verification. The Radiochemistry and Stable Isotope Unit participates in the USEPA cross-check program five times a year and in the U.S. Department of Energy QA program two times a year.

6.2.6 Data Reporting

Analytical results are independently reviewed by a chemist or an analyst who did not perform the measurement. The result and the calculated error are entered into NWIS-I. Computer entries are checked to ensure accuracy. Original data are kept on file for 3 years at the NWQL and then archived.

7.0 LABORATORY QUALITY ASSURANCE

Quality assurance at the NWQL includes programs for documentation, data validation, and internal and external blind sampling. External-evaluation studies as well as external-audit programs are included in this section.

7.1 Documentation

Records of data undergoing review are kept in the QAU until the data have been approved for release. SOPs and revisions are filed with the QAU. Currently (1995), data records are permanently archived. The NWQL data bases are retained on disk and tape.

7.2 Data Validation

The NWQL uses several QA checks to validate sample data. Sample-data review and QA checks are enhanced by using a computer program (fig. 3). When analytical determinations are completed for a sample, the computer program compares the data against acceptable limits. Sample data that pass the limits of the computer program are released to the customer. The QA checks of inorganic-sample data are performed according to practices described by Friedman and Erdmann (1982, p. 103-108).

When specified major cation and anion constituents are present, ion balance is checked. A difference of percentage is calculated and compared to an acceptance curve described by Friedman and Erdmann (1982, p. 104). Constituents that apply to the USEPA's Drinking-Water Regulations also are checked (U.S. Environmental Protection Agency, 1992b, 1992c). If the computer checks produce a warning flag, the sample data are rejected and a status report is prepared for review by a member of the QAU. Further action, such as data verification or reanalysis of the sample, might be necessary.

In order to return sample data quickly to the customer, nutrient samples are reviewed within the Nutrients Unit. The data values from the Organic Chemistry Program are validated by a verification printout. The Computer Services Unit sends a printout of the values to the analyst, who in turn checks to make sure these values were entered correctly. The customers can make inquiries for reanalyses and data verification about sample data, when sampling site and sampling history indicate an erroneous result. Results are sent back to customers via USGS geomail.

7.3 Blind-Sample Program

The QAU administers a QA assessment program based on results of blind reference and blank samples submitted to the Inorganic and Organic Chemistry Programs. Sources of reference water samples for the internal blind-sample program primarily are Standard Reference Water Samples supplied by the Branch of Technical Development and Quality Systems. Commercially prepared reference materials and standards supplement the blind samples where Standard Reference Water Samples do not exist. Blanks are prepared from reagent water with a minimum resistivity of 16.7 megohms-centimeter. Blind samples are prepared and preserved in the same manner as the environmental samples they represent. Frequency of blind sample and blank insertions for each analytical method is proportional to sample load.

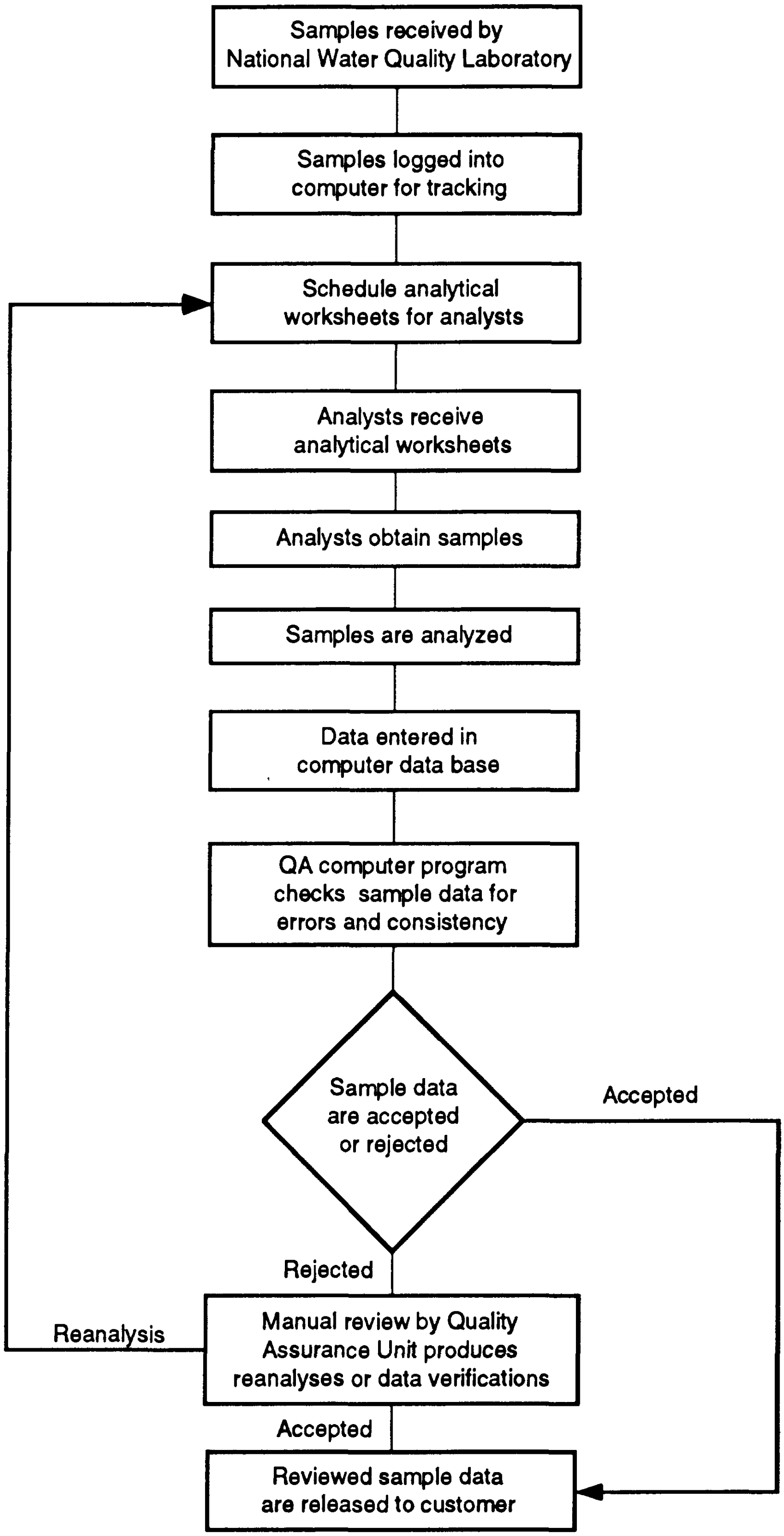

Figure 3.– Flowchart showing inorganic samples and data review at the National Water Quality Laboratory.

A computerized data base is used to track, monitor, and record results of the blind samples. Results from the Inorganic Chemistry Program blind samples usually are returned to the QAU the day after analysis. This schedule allows the QAU to review results quickly and respond with corrective-action reports to the appropriate program unit if a result is outside the acceptable range. Acceptable ranges for blind samples generally are 1.5 times the standard deviation calculated from data from the Branch of Technical Development and Quality Systems Standard Reference Water Samples. Ranges for commercially available reference materials are calculated on the basis of certificates of analysis. Monthly, the QAU produces QC charts for use in comparison with Inorganic Chemistry Program QC. The data from these comparisons are the basis for monthly Quality Assurance Unit/Inorganic Chemistry Program meetings, resulting in continuous quality improvement of sample analysis and QC systems.

7.4 External Blind-Sample Program

The Branch of Technical Development and Quality Systems, which is independent of the NWQL, submits blind samples that are prepared by mixing various Standard Reference Water Samples. The Branch of Technical Development and Quality Systems monitors not only the Inorganic Chemistry Program, but also the NWQL support functions such as sample logging and quality assurance. Results of these assessments are regularly published by the Branch of Technical Development and Quality Systems (Maloney and others, 1993, for example). The Branch of Technical Development and Quality Systems provides an interactive computer online service for retrieval and analysis of results from the external blind-sample program (Lucey, 1990).

7.5 External Evaluation Studies

The NWQL participates in a number of evaluation studies, as follows:

U.S. Environmental Protection Agency Water-Supply study -- 2 per year. Determination of low-level concentrations of organic compounds and inorganic constituents, in water.

U.S. Environmental Protection Agency Water-Pollution study -- 2 per year. Determination of high-level concentrations of organic compounds and inorganic constituents, in water.

Canadian Center for Inland Water Samples -- 4 per year. Determination of trace-level concentrations of inorganic constituents, in water.

Branch of Technical Development and Quality Systems -- 2 per year. Determination of low- and medium-level concentrations of inorganic constituents in water samples.

National Oceanic and Atmospheric Administration -- 1 per year. Determination of low-level concentrations of organic compounds and inorganic constituents in biological tissues and sediments.

Results of these studies are reviewed by staff and management. Any questionable results are investigated, and, if necessary, corrective action is taken.

7.6 External Audit Programs

External agencies and customer organizations audit the NWQL to assess analytical and quality programs. The Branch of Technical Development and Quality Systems annually reviews the NWQL. The Colorado Department of Public Health and Environment triennially audits NWQL analytical and QA activities that correspond to the USEPA's Drinking-Water Regulations. Occasionally, organizations involved in USGS cooperative programs also review the NWQL. Recommendations from the various audits help to improve the quality of service.

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9.0 GLOSSARY

Reporting the results of analyses of water and fluvial-sediment samples requires the use of a number of terms that are based on the combination of physical phases sampled (water or sediment) and analytical methods used. These terms are defined below although not all are used in this manual. Selected terms are included for background information. Definitions are taken from Fishman and Friedman (1989) and from V.R. Schneider (U.S. Geological Survey, written commun., 1990).

Accuracy. A measure of the degree of conformance of the values generated by a specific method or procedure with the true value. The concept of accuracy includes both bias (systematic error) and precision (random error) (Fishman and Friedman, 1989, p. 5).

Analyte. A constituent, trace element, property, radionuclide, or organic compound being determined in an analysis.

Bias. Systematic error that is manifested as a consistent positive or negative deviation from the known or true value. It differs from random error which shows no such deviation.

Blank solution. Solution that is free of the analyte(s) of interest. Such a solution would be used to develop specific types of blank samples.

Blind sample. A sample submitted for analysis whose composition is known to the submitter but unknown to the analyst.

Certified reference material. A reference material, for which one or more property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body.

Class A. A precision grade for volumetric glassware, whose design and tolerances generally conform to the National Institute of Standards and Technology requirements.

Class S. A specification of weighing standards, whose design and tolerances conform to the National Institute of Standards and Technology requirements.

Duplicate analysis. The analysis or measurement of the variable of interest performed as identically as possible on two subsamples of a sample.

External standard. A mixture of compounds of interest (analytes to be determined) prepared in a suitable organic solvent and diluted to approximate environmental residue concentrations; used for calibrating and checking detector response prior to instrumental analysis. External standards establish response and retention factors necessary for quantitative analysis when internal standard or standard addition methods are not used.

Filtered. Pertains to the constituents in a representative water sample that pass through a 0.45-micrometer membrane filter for inorganic analysis or a 0.7-micrometer glass fiber filter for organic analysis. This is a convenient operational definition used by Federal agencies that collect water data. Determinations of "dissolved" constituents are made on subsamples of the filtrate.

Internal standard. A compound similar in physical and chemical properties to the analyte in the sample; added to the final extract just prior to instrumental analysis. Internal standard responses are incorporated into quantitative analytical calculations, thus serving to normalize data to a known amount of a common reference. Internal standard materials must be chosen carefully; they must exhibit proper chromatographic behavior and yet must not occur either naturally or as a result of pollution. When using mass sensitive detectors, internal standards may be chosen from stable heavy isotope analogs of analytes of interest. Other types of gas and liquid chromatographic detectors require other kinds of compounds. An internal standard will correct for the biases associated with the instrumental determinative step in an analytical procedure.

Matrix. The predominant material comprising the sample to be analyzed. The matrices most often analyzed by the NWQL are water, soil/sediment, and biological tissue.

Method blank. An analyte-free matrix carried through the entire sample preparation and analytical procedure.

Method-detection limit. The minimum concentration of a substance that can be identified, measured, and reported with 99-percent confidence that the analyte concentration is greater than zero; determined from analysis of a sample in a given matrix containing analyte.

Precision. The degree of similarity among independent measurements of the same quantity, without reference to the known or true value (V.R. Schneider, U.S. Geological Survey, written commun., 1990).

Quality assurance (QA). Those planned or systematic actions necessary to provide adequate confidence that a product or service will satisfy given requirements for quality.

Quality control (QC). The operational techniques and the activities used to fulfill requirements of quality.

Quality management. That aspect of the overall management function that determines and implements the quality policy.

Reagent spike. A synthetic matrix fortified with known concentrations of all, or a representative selection of, the method analytes.

Recoverable from bottom material. Pertains to the constituents extracted from a representative sample of bottom material. Complete extraction generally is not achieved, and thus the determination often represents less than the total amount (that is, less than 95 percent) of the constituent in the sample. To achieve comparability of analytical data, laboratories performing such analyses would have to use equivalent extraction procedures, because different extraction procedures are likely to produce different analytical results.

Reference material. A material or substance one or more properties of which are sufficiently well established to be used for the assessment of a measurement method or for assigning values to materials.

Rounding. A consistent procedure is followed in rounding off numbers to n significant figures. Digits to the right of the nth digit are discarded. If the first of the discarded digits is greater than 5, add 1 to the nth digit. If the first of the discarded digits is less than 5, leave the nth digit unchanged. If the first of the discarded digits is 5 and the following digits are zero, round off to the nearest even number. If the 5 is followed by any of the digits 1 through 9, add 1 to the *n*th digit. In presenting numerical data, give only those digits that convey actual information. The last digit should represent the uncertainty in the data (Hansen, 1991, p. 119).

Sample. A representative part of a larger whole; a finite part or subset of a statistical population.

Spike sample. A sample to which known concentrations of specific analytes have been added in such a manner as to minimize the change in the matrix of the original sample.

Standard operating procedure (SOP). A written document that details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. It may be a standard method or one developed by the user.

Standard reference material. A certified reference material produced by the U.S. National Institute of Standards and Technology.

Standard Reference Water Sample (SRWS). A noncertified reference water produced by the U.S. Geological Survey Branch of Technical Development and Quality Systems to evaluate laboratories that perform analytical services for USGS and to provide a supply of QC material for laboratories.

Surrogate. A compound similar in physical and chemical properties to the analytes of interest; added to the sample upon receipt in the laboratory (or, ideally, at the time of field sampling). A surrogate is not used as an internal standard for quantitative measurement purposes. Surrogates may be added to every sample to provide quality control by monitoring for matrix effects and gross sample-processing errors. They should not occur naturally or be present in polluted water samples. Also called "surrogate spike."

Suspended, recoverable. Pertains to the constituents extracted from the suspended sediment that is retained on a filter. Complete extraction generally is not achieved, and thus the determination represents something less than the "total" amount (that is, less than 95 percent) of the constituent present in the suspended phase of the sample. To achieve comparability of analytical data, laboratories performing such analyses would have to use equivalent extraction procedures, because different extraction procedures are likely to produce different analytical results. Determination of "suspended, recoverable" constituents is made either by analyzing portions of the material collected on the filter or, more commonly, by computing the difference between (1) dissolved and (2) total recoverable concentrations of the constituent.

Suspended, total. Pertains to the constituents of the suspended sediment that are retained on a filter. This term is used only when the analytical procedure ensures measurement of at least 95 percent of the constituent determined. Knowledge of the expected form of the constituent in the sample, as well as of the analytical methodology used, is required to determine when the results should be reported as "suspended, total." Determinations of "suspended, total" constituents are made either by analyzing portions of the material collected on the filter or, more commonly, by computing the difference between (1) dissolved and (2) total concentrations of the constituent.

Total. Pertains to the constituents in a representative water-suspended-sediment sample. This term is used only when the analytical procedure ensures measurement of at least 95 percent of the constituent present in both the dissolved and suspended phases of the sample. Knowledge of the expected form of the constituent in the sample, as well as of the analytical methodology used, is required to judge when the results should be reported as "total." (Note that the word "total" does double duty here, indicating both that the sample consists of a water-suspended-sediment mixture and that the analytical method determines all of the constituent in the sample.)

Total in bottom material. Pertains to constituents in a representative sample of bottom material. This term is used only when the analytical procedure ensures measurement of at least 95 percent of the constituent determined. Knowledge of the expected form of the constituent in the sample, as well as of the analytical methodology used, is required to judge when the results should be reported as "total in bottom material."

Whole water, recoverable. Pertains to the constituents in solution after a representative water-suspended-sediment sample is digested (usually using a dilute acid solution). Complete dissolution of particulate matter often is not achieved by the digestion treatment, and thus the determination represents something less than the "total" amount (that is, less than 95 percent) of the constituent present in the dissolved and suspended phases of the sample. To achieve comparability of analytical data, equivalent digestion procedures would be required of all laboratories performing such analyses, because different digestion procedures are likely to produce different analytical results.

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