QUALITY ASSURANCE/QUALITY CONTROL MANUAL

National Water Quality Laboratory

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

U.S. GEOLOGICAL SURVEY

Open-File Report 92-495

Denver, Colorado
1992

U.S. DEPARTMENT OF THE INTERIOR

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CONVERSION FACTORS

Multiply	By	To obtain
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:

$$°F = 9/5\ (°C) + 32$$

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ABSTRACT

Quality-control practices are established for the operation of the U.S. Geological Survey's National Water Quality Laboratory. These practices specify how samples are preserved, shipped, and analyzed in the Laboratory. This manual documents the practices that are currently (1992) used in 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 analytes in water, sediments, and biological materials. Heightened concerns about water quality and about the possible effects of toxic chemicals at trace and ultratrace levels have contributed greatly to an increased demand for impartial, objective, and independent data, which can be used to assess the impact of these analytes in the aquatic environment.

To meet these needs as a service laboratory, the NWQL provides expertise in a variety of analytical methods applied to a variety of matrices (see Glossary). 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) 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, standardized, and maintained. In addition, methods used are within USGS accepted levels, and data are verified prior to reporting.

Although the QA program of the NWQL is directed by the Quality Management Group, which reports directly to the chief of the NWQL, a successful quality-management program requires active and enthusiastic participation by employees. External review of this program is a responsibility of the Branch of Quality Assurance.

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 high quality results in a timely, cost-effective manner. To meet established quality objectives and to support District water-quality investigations, the NWQL provides project planning and data interpretation assistance. 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 high 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. For its effectiveness, this program relies on clearly defined objectives, well-documented procedures, and management support.

1.4 Organizational Structure

The chief, Branch of Analytical Services, (1) directs, manages, and coordinates a number of programs in support of water-quality analysis 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 organic and inorganic analyses and support functions. The assistant chief of the Branch in the chief's office is the District liaison for hydrologic programs. An organization chart is shown in figure 1.

The Laboratory Operations chief (1) provides support to the Branch chief in the management of day-to-day Laboratory operations; and (2) oversees the operation of the Technical Services Unit, Automated Data Processing, and Facilities Unit.

The Technical Services Unit performs the operational support activities related to sample receipt, warehousing, supplies, and building and laboratory space requirements.

The Automated Data Processing Unit (1) operates and enhances a complex computer facility for sample analyses, quality control (QC), billing applications, and office management support; and (2) transfers analytical data to District offices.

The Safety Office (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;

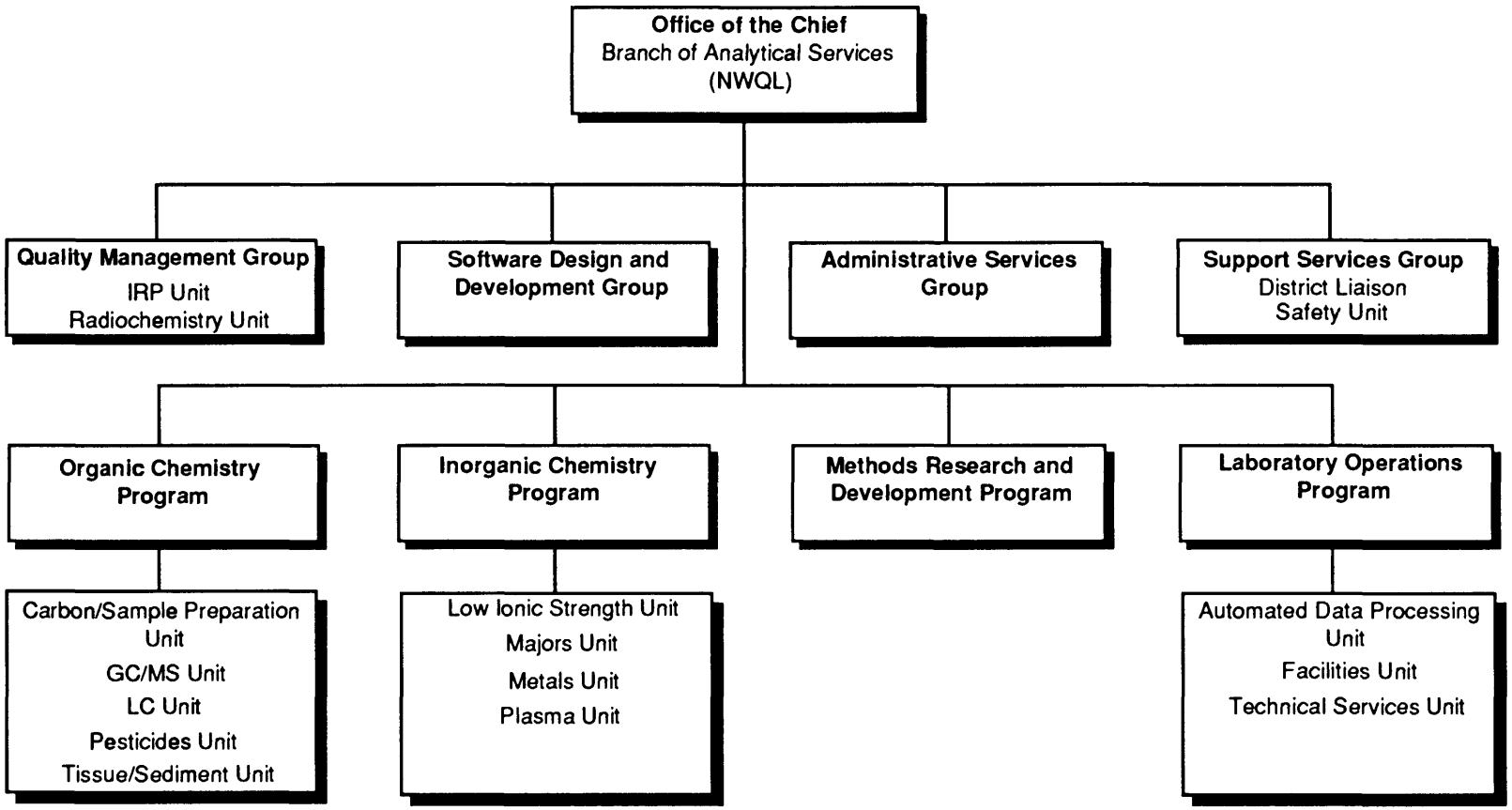

Figure 1. -- Organizational chart of the National Water Quality Laboratory.

(2) counsels management on matters of safety; and (3) manages security for NWQL grounds and facilities.

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; and (4) provides general consultation and scientific advice to laboratory and USGS personnel on new methods of sample-collection processing and analysis, development, and determination of priority for new research needs.

The Organic Chemistry Program (1) systematically carries out operational laboratory activities for organic analysis of water, sediment, and water-related medium samples; and (2) provides general consultation and scientific advice to USGS personnel and cooperators throughout the Nation on the determination of organic compounds, methodology, and automation techniques.

The Inorganic Chemistry Program (1) systematically carries out operational laboratory activities for inorganic analysis of water, sediment, and water-related medium samples; (2) provides general consultation and scientific advice to USGS personnel and cooperators throughout the Nation on the determination of inorganic compounds, methodology, and automation techniques; and (3) defines problems, and develops and implements proposals to meet future analytical needs for inorganic analyses.

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

The Quality Management Group (1) carries out operations related to the development and implementation of quality-assurance programs at the NWQL; (2) through the Radiochemistry Unit, oversees the preparation of contracts for radiochemical and stable isotope analyses not currently performed at the NWQL; (3) interacts with USGS scientists for the use and interpretation of NWQL and contract laboratory data; and supports the Installation Restoration Program.

1.5 Other Quality-Assurance Publications

This manual describes the current (1992) QA practices and standards of the NWQL. It supersedes the interim QC manual by Jones (1987). This manual will be reviewed every 2 years and updated as needed to reflect the rapid change and development of the organization, staffing, and data-management system at the NWQL. The USGS also has many other publications that describe QA in the laboratory and onsite sample collection for NWQL 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 unpublished reports describe the USGS analytical evaluation program for Standard Reference Water Samples for trace constituents, major constituents, nutrients, mercury, and low-ionic strength analytes. Friedman and Erdmann (1982) describe QA practices for chemical and biological analyses of water and fluvial sediments.

Also included are references to several unpublished memoranda that deal with analytical methods or water-quality policy. In usual 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 are requested to write to the editors or to the chief of the NWQL, providing the title and number of this Open-File Report and the number and subject of the memorandum. For the convenience of the reader, all memoranda cited in this manual are listed as follows:

- Water Resources Division Memorandum No. 82-28, January 21, 1982
  Subject: WATER QUALITY--Acceptability and Use of Water-Quality Analytical Methods

- Office of Water Quality Technical Memorandum 91.09, August 27, 1991
  Subject: REPORTS--Filtration of Water-Sediment Samples for the Determination of Organic Compounds

- Office of Water Quality Technical Memorandum 92.06, March 20, 1992
  Subject: REPORTS--Report of Committee on Sample Shipping Integrity and Cost

- National Water Quality Laboratory Technical Memorandum 92.01, March 25, 1992
  Subject: TECHNOLOGY TRANSFER--Availability of Equipment Blank Water for Inorganic and Organic Analyses

2.0 GENERAL CONSIDERATIONS

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

2.1 Analytical Methods

The NWQL uses proven documented methods for most analytical work. These methods are categorized as follows: USGS approved or interim approved methods, non-USGS published standard methods, and custom methods. The USGS approved methods are validated, including precision and accuracy data; are externally reviewed; and are published either as Techniques of Water-Resources Investigations (TWRI) or Open-File Reports (OFR). Interim approved methods are validated, including precision and accuracy data, and are internally reviewed. These interim methods are used while OFRs are prepared for publication.

Non-USGS published standard methods include U.S. Environmental Protection Agency methods, Standard Methods for the Analysis of Water and Wastes, and American Society for Testing and Materials (ASTM). Before a non-USGS published standard method is used, the NWQL first demonstrates and documents an ability to use these methods according to published criteria. Data collected from USGS approved and interim approved methods, and non-USGS published standard methods may be stored in the Water Data Storage and Retrieval System (WATSTORE) and published in annual data reports of the USGS.

Custom methods occasionally are requested by customers who have unique project requirements. Custom methods do not fall in the aforementioned categories or are experimental methods, which have not been validated to the level required to be classified as an approved or interim approved method. Custom methods are not covered by the usual QA/QC practices described in this manual. The NWQL works with the customer to define the QA/QC requirements. Therefore, chemical determinations by custom methods are not stored in the WATSTORE data base. Documentation and development records of custom methods are kept by the analytical program responsible for providing data for the project.

2.2 Method Validation

The NWQL follows USGS policy on validation of approved methods as outlined in Water Resources Division (WRD) Memorandum 82.28 (L.B. Laird, U.S. Geological Survey, written commun., 1982), and in revisions by M.J. Fishman (U.S. Geological Survey, written commun., 1987). B.E. Jones (U.S. Geological Survey, written commun., 1990) provides a more flexible approach to method validation that accommodates organic analytical methods in addition to inorganic analytical methods. Method validation accounts for and documents, at a minimum, the following characteristics: known and possible interferences; method precision; method accuracy, bias, and recovery; method detection level, 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 the method performance. Approved methods are developed into detailed analyst instructions called SOPs.

2.3 Training

Supervisors are responsible for assuring that employees receive orientation, safety, and skills training. General training 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 officer provides safety orientation to new employees within their first 30 days of hire, followed by continuing 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 section are discussed by the section 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 trainer, trainee, type of analytical method trained on, dates of training, and results from evaluation samples. Documentation is retained by the supervisor.

2.4 Safety

The Safety Office directs worker health and safety, waste management, and security. These responsibilities include air sampling, environmental compliance, and the testing of safety equipment at regular intervals. In addition to SOPs, Skinner and others (1983) discusses 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 maintenance is performed as needed. Fume hoods are inspected annually for face-velocity measurement, hood adjustments, and for marking operational sash positions. Ventilation recommendations are made to improve engineering controls and systems.

Waste requests are processed by the Safety Office and conducted according to the SOP on waste. 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 Office 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 generated by the Radiochemistry 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 Radiochemistry Unit files and is supplied to the Safety Office when the barrel is ready for disposal.

Visitors sign in and out of the building at the lobby. Visitors are provided with safety glasses or any other personal protective equipment necessary for entry into laboratory areas. Information on the hazard communication standard and chemical usage in the facility is provided. Minors will not be admitted to the NWQL without an adult escort.

Entry to the NWQL between 5:30 p.m. and 6:00 a.m. (Mountain Standard Time) is by magnetic access card only. The cards are issued and maintained by the Safety Office. 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 Office. Safety training documents are kept by Administrative Services Group and by the Safety Office.

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-cm to laboratory areas and the glassware-washing facility. One deionizing system is 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-cm. The reagent water is used for preparing calibration standards, chemical reagents, and sample dilution. The second deionized water system supplies water for the glassware washing facility.

Two separate water systems are used by the NWQL to prepare organic-free water (ASTM Type II). One system is in the organic laboratory and the other is in the glassware-washing

[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.

facility. Both systems use carbon filtration followed by distillation. Both organic-free water systems are monitored by the organic laboratory personnel at regular intervals. Volatile organic-free water is prepared in the laboratory from deionized water that has been boiled and purged by nitrogen.

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; 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 certified by the contract service technician.

2.5.3 Refrigerators

Refrigerators used for storing samples and reagents are monitored each workday. The temperature is recorded on a logsheet attached to each refrigerator. Logsheets are maintained in the laboratory section responsible for the refrigerator. The stabilized temperature is maintained nominally at 4°C with a tolerance range of ±2°C. If the stabilized temperature exceeds these limits, the temperature set point is readjusted until the temperature is within the acceptable range.

2.5.4 Ovens

Ovens are checked for temperature accuracy by use of an independently calibrated mercury thermometer. General-purpose thermometers are used for most temperature-monitoring applications. Thermometers calibrated by the National Institute for Standards and Technology are used to verify accuracy of general-purpose thermometers. A temperature log is kept for each oven.

2.5.5 Glassware Specifications

Volumetric glassware used for analytical work is class A. Volumetric flasks and pipettes are dedicated to specific uses to prevent cross-contamination. Fixed and variable volume micropipettes often are used to prepare calibration standards and dilute samples. Micropipettes regularly are calibrated with analytical balances. Glassware is washed according to SOPs.

The SOPs define the cleaning and preparation of laboratory glassware and containers for onsite sampling. Specific SOPs include: (1) washing-machine operations and inspection, (2) cleaning of laboratory organics and inorganics glassware, (3) preparation of field-sampling containers, and (4) oven and dryer operations.

2.5.6 Fume Hoods

Laboratory fume hoods are inspected and certified annually by a contract industrial hygienist. Inspection reports are on file in the Safety Office. 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 inorganic analysis data generated by the NWQL is transferred electronically from the laboratory instruments to the in-house Laboratory Information Management System (LIMS) computer. Currently (1992) organic data and some inorganic data are hand-entered by data-entry personnel. A daily report is printed and distributed within 24 hours to the appropriate analyst for verification of the hand-entered data.

2.6.2 Data Reporting

Analytical data for routine determinations are compiled on the LIMS computer in logical subgroupings (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 these data using the National Water Information System (NWIS-I) software and entering the data into the WATSTORE data base.

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 computer 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 need. 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 of data generation.

3.0 SAMPLE MANAGEMENT

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

3.1 Sample Containers and Onsite Reagents

3.1.1 Sample containers

Prepared sample containers are supplied to USGS field offices, on request to the NWQL. The type of container to be used is specified in the NWQL Services Catalog (Pritt and Jones, 1989). The NWQL does not accept samples in containers other than those specified by the NWQL. Containers received from suppliers are checked according to SOPs by the Quality Management Group. 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 type as described by Pritt and Jones (1989). Records of container testing are maintained by the Quality Management Group. Accepted containers are cleaned by the Technical Services Unit according to written SOPs.

3.1.2 Preservatives

Sample-preservation chemicals in ampules are supplied to USGS field offices by the NWQL. The types of preservatives and their correct use are described in the Services Catalog (Pritt and Jones, 1989). Preservatives are prepared by contract suppliers in accordance with NWQL specifications. Preservatives are checked for quality prior to dispensing onsite in accordance with written SOPs. Records of accepted preservatives are maintained by the Quality Management Group.

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 micropipettes, 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 the Methods Research and Development Program specifications. Each lot of the spike solution is randomly sampled and analyzed prior to acceptance and distribution. Test-data files are maintained by the Quality Management Group.

3.1.4 Onsite Equipment Blank Water

Onsite 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

onsite project described in the project QA plan determine the number and type of field equipment blanks. Procedures for obtaining and using onsite blank water are described in NWQL Technical Memorandum 92.01 (P.F. Rogerson, U.S. Geological Survey, written commun., 1992). Different types of water to be used for equipment blanks are available through the NWQL or the USGS Laboratory in Ocala, Florida, depending on the types of sample analyses. These types are organic-free water, volatile organic compound-free water, and ASTM Type I water (for inorganic use). The water is prepared in numbered lots either in the NWQL, the USGS Laboratory in Ocala, Florida, or by commercial suppliers. Each lot of the equipment blank water is tested according to SOPs. Test-data files are maintained by the Quality Management Group.

3.1.5 Filters

Filters for processing samples for organic determinations are supplied through the Laboratory in Ocala, Florida. The type of filter to be used for each determination is described in the Office of Water Quality Technical Memorandum 91.09 (M.W. Sandstrom, U.S. Geological Survey, written commun., 1991). Quality-control checks are performed on lots of filters to ensure freedom from contamination. Test-data files are maintained by the Quality Management Group.

3.1.6 Sample Documentation

When field office personnel collect water samples, samples are placed in the appropriate bottles with the necessary preservatives, and an Analytical Services Request Form provided by the NWQL is filled out. This form was updated as of April 1992 (fig. 2). The following categories must be filled out for acceptance for NWQL analysis: station identification, state, District user code, project account number, and beginning date of sampling. Additional information that will help the District to receive improved service from the NWQL includes the following: schedules, field and laboratory codes, name and phone numbers of shipper and project chief, number of bottles and packages shipped, bottle types, identification of special samples, and exact time of sampling for radon-222.

3.1.7 Shipping Requirements

The Committee on Sample-Shipping Integrity and Cost has addressed the need for samples to arrive intact at the NWQL and to meet the holding times for certain analytes. The Office of Water Quality Technical Memorandum 92.06 (J.D. Broadus, U.S. Geological Survey, written commun., 1992) provides recommendations to District offices for shipping samples to the NWQL. Included in the memorandum are specifications for shipping containers, preparing samples for shipment, preparing log forms (for example, Analytical Services Request Forms) for shipment, and packaging of samples.

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

SMS CONTROL NO. | NWIS RECORD NO. | 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 ACCT

* BEGIN DATE: 19 | END DATE: 19 | YEAR | MONTH | DAY | TIME

SCHEDULES, FIELD AND LABORATORY CODES

SCHEDULE 1: _____
SCHEDULE 2: _____
SCHEDULE 3: _____
SCHEDULE 4: _____
SCHEDULE 5: _____

** SAMPLE MEDIUM: _____
GEOLOGIC UNIT: _____
** ANALYSIS STATUS: _____
** ANALYSIS SOURCE: _____
** HYDRO CONDITION: _____

** SAMPLE TYPE: _____
** HYDRO EVENT: _____

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		
___/___			___/___			___/___		
___/___			___/___			___/___		

+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	____RC	____FC	____FAB	____CU
____RA	____RAH	____S=	____CN-	____RCB	____TOC	____DOC	____SOC	____COD
____VOA	____CHY	____O&G	____PHENOL	____GCC	_____ OTHER			

CUSTOM/SPECIAL SAMPLE APPROVED BY: _____ APPROVAL NO. _____

PROGRAM/PROJECT: _____ NPDES _____ NAWQA _____ DRINKING H_2O FILL IN OTHER _____

POSSIBLE HAZARDS _____

Revised 04/92 +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.

3.2 Sample Handling

3.2.1 Log-In

Samples and the accompanying Analytical Services Request Forms are received by the Log-in Section. Each sample and its request form are assigned a unique laboratory identification number that encodes the date of receipt and a serial number. The identification number is printed on barcoded 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 Log-in Section in sample shipping and preservation are noted on the Analytical Services Request Form.

The Analytical Services Request Form is routed to Automated Data Processing, which enters sample information and analytical requests into the LIMS data base. After entry in the data base, the information on the Analytical Services Request is converted daily to a series of NWQL workload requests. During log-in, each bottle received is recorded on a cover sheet. Copies of these cover sheets are distributed at the end of each day to the analytical sections.

Radiochemical and stable isotope samples are received by the Log-in Section and entered into the LIMS data base. The Radiochemistry Unit maintains a separate data base to track analyses performed by the various contract laboratories.

3.2.2 Chain-of-Custody

Samples having chain-of-custody forms are signed for by Log-in personnel. The signed forms are returned to the customer. Samples then are made available for analysis by the NWQL.

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 a stabilized temperature of 4°C ±2°C except where otherwise indicated.

Samples for analysis of inorganic constituents except nutrients are properly discarded within 180 days of receipt. Analyzed nutrient samples are discarded within 30 days of receipt.

Organic samples are stored in the organic laboratory until extraction and analysis are completed. After extraction, the samples are disposed of according to procedures defined by the safety and environmental officer.

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 the Log-in area 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, under separate cover, to the contract laboratory. A copy of the list is kept in the Radiochemistry Unit files.

3.2.4 Holding Times

Holding time is defined as the number of days between date of sampling and date of analysis (table 1). The maximum holding times--required to be met by the NWQL--are in the parameter-code dictionary in the PRIME and LIMS computers. In some instances, such as during peak load time, it may become necessary to proceed with an analysis even though the holding time has been exceeded.

Table 1. -- *Maximum holding times for samples*

Type of sample	Number of days	
Acidity, alkalinity, pH, and specific conductance	7	
Nutrients	8	
Anions and low-level major ions	28	
Trace metals and major cations	42	
Dissolved and total solids	42	
Phenols, total	14	
Volatile organic chemicals	14	
Acid-base/neutral extractables:		
Extraction	2	working days after receipt
Chromatography	45	
Pesticides:		
Extraction	2	working days after receipt
Chromatography	45	

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 are labeled as such.

4.0 ORGANIC ANALYSIS

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

4.1 Documentation

Routine analyses are performed according to methods described by Wershaw and others (1987) or by Sandstrom and others (1992) using associated SOPs. 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 supervisor; copies also are filed in the Quality Management Group office. Where possible, raw 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, initialed and dated by unit chiefs for accuracy, completeness, and timeliness.

Analytical data, chromatograms, report sheets, copies of Analytical Service Request Forms, and preparation notes are filed by set number. A set is a batch of samples, a blank-control sample, and a spike-control sample. Sets are indexed by Julian day in which the sets are formed for analysis. Sample data include dates of extraction, concentration, clean-up, and chromatograms, and are filed with the set. Sets are filed by analytical schedule.

Quality-control charts are maintained for several compounds defined in the U.S. Environmental Protection Agency (1990a) Primary and Secondary Drinking-Water Regulations, and for surrogates. Charts are reviewed by unit chiefs, chemists, and sample preparation analysts.

4.2 Reagent Preparation

Reagents are prepared according to methods described by Wershaw and others (1987), or by Sandstrom and others (1992) using associated SOPs. Reagents are labeled with date prepared and preparers' 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 the TWRI or bench methods manual.

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°C to remove residual organics.

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 the distillation unit are kept by the Technical Services Unit.

Certified solutions are purchased in the highest purity available. High-purity materials are used to prepare stocks when solutions are unavailable or concentrations are inadequate, and are corrected for purity when less than 97-percent pure.

4.3 Sample Preparation and Storage

Water samples arriving in the organic preparation unit are extracted within 2 working days. Water samples arriving on Friday will be extracted the following Monday, and samples arriving on Saturday will be extracted by Tuesday. Samples are prepared according to methods described by Wershaw and others (1987) and by Sandstrom and others (1992) using associated SOPs. Sediment samples are refrigerated upon receipt and are weighed and extracted within 2 working days.

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 at a minimum are retained until the sample has cleared internal review.

4.4 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 range described in the individual SOPs.

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

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.

4.5 Organic Quality Control

Distilled-water blanks are prepared for each sample set and processed in the same manner as the samples. When an extract from the blank water interferes with an analysis, a reagent-only blank is prepared to determine the source of the interference. If distilled water causes

interference, the water is redistilled or is extracted with the appropriate solvent, prior to its use as a blank. If a reagent is the source of contamination, then a new lot is tested or the reagent is repurified as specified in the appropriate method or SOP, and re-tested.

Each new lot of reagents is tested for contamination prior to use. In addition, they are tested during use. The reagents are taken through the appropriate procedure and re-purified if results show interference on the chromatograms.

Spikes containing analytes of interest are prepared with distilled water and included with each sample set. The spike solutions are checked against analytical standards prior to use to verify components and concentrations. Results of the spike are recorded and stored with the sample set.

Sample results are verified on two dissimilar columns except for gas chromatography/mass spectrometry determinations. Response factors are calculated for both columns. The smaller value of the two columns is reported to allow for interference peaks. Extracts are not analyzed on two columns because mass spectra are definitive for gas chromatography/mass spectrometry methods. Extracts are diluted and reanalyzed when concentrations exceed calibration ranges as specified in the appropriate method or SOP.

Operator QC includes the following: previous calibrations, detection-level verifications, calibration check samples, system performance check samples, tuning criteria, set blanks, set spikes, and surrogate recoveries, where applicable. Field blanks, spikes, and replicates can be part of the overall QA, if project personnel submitting samples request such analyses.

4.6 Data Reporting

Data are checked independently by another analyst prior to submittal to Automated Data Processing. Values are manually transcribed onto preprinted data-report sheets. Data are mailed to the customer when it is not possible to enter the data into NWIS-I or when certain QC criteria are not met.

Results are not reported when surrogate recovery is zero or 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.

5.0 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.

5.1 Documentation

Routine analyses are performed according to methods described by Fishman and Friedman (1989) or by Patton and Truitt (1992) using associated SOPs. 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 section supervisors or senior analysts. Several different types of logbooks are kept: instrument maintenance, line conditions, special samples, and calibration. Current SOPs are available to operators. Changes to an SOP are approved by the section supervisor, and then are documented and recorded prior to implementation. SOPs are filed in the section and in the Quality Management Group. Raw analytical data such as strip-chart recordings are stored at the laboratory for up to 3 years with the run date for reference.

5.2 Reagent Preparation

Reagents are prepared according to methods described by Fishman and Friedman (1989) or by Patton and Truitt (1992) using associated SOPs. Reagents and standard solutions are labeled to indicate date last made. 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 section. Reagents and standard solutions are made with reagent water. Class A volumetric glassware is used for the preparation of calibration standard solutions and critical reagents. Chemicals are labeled with date upon receipt. An annual inventory is mandated by the Safety Office, and a copy of the storage plan is filed in the Safety Office.

5.3 Sample Preparation and Storage

Samples requiring treatment prior to analysis are prepared according to Fishman and Friedman (1989) or by 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.

5.4 Instrument Calibration and Maintenance

Instruments are calibrated prior to each analytical run. Depending on instrumentation, calibration curves might contain as few as one calibration standard (inductively coupled plasma-atomic emission spectroscopy) or as many as seven calibration standards (atomic absorption spectroscopy). Data-acceptance criteria are developed on the basis of instrument

performance and calibration curve characteristics and are stated in SOPs for each analytical line.

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.

5.5 Inorganic Quality Control

Quality-control samples are run at least once every 10 samples. These QC samples include at least one of the following: Standard Reference Water Samples provided by the Branch of Quality Assurance, duplicate samples, standard solutions, blanks, or spikes. The QC data from analytical lines are reviewed following completion of a run. First the analyst checks correlation coefficients for calibration curves. Acceptable correlation coefficients vary depending on the analytical line, with most of the analytical lines 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 that they fall within acceptance criteria. If all data-acceptance criteria in SOPs are met, data on this analytical run are determined to be acceptable. Failure of the QC samples to meet acceptance criteria, established and published in SOPs, results in shutdown of the analytical line and re-calibration. Quality-control charts, produced monthly from Standard Reference Water Sample data, are reviewed by the analyst and section supervisors to ensure continued acceptable performance for each analytical line.

5.6 Data Reporting

Upon acceptance of analytical data by the analyst, results are submitted to Automated Data Processing. Raw analytical data are stored at the laboratory for up to 3 years.

6.0 RADIOCHEMICAL/STABLE ISOTOPE ANALYSIS

Radiochemical/stable isotope analysis services are organized as follows under contracts and in-house analysis.

6.1 Contracts

The Radiochemistry Unit contracts with commercial and 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 Radiochemistry Unit. A notebook containing information regarding radiochemical QC sample preparation is maintained. The information recorded in this notebook includes the U.S. Environmental Protection Agency 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 which are sent to the contract laboratory. The notebook contains the expected and actual values of the QC samples, rerun results, and QC sample duplicate values. A notebook is maintained for each stable isotope, which contains methods of QC sample preparation, expected and actual value of the QC sample, and sample rerun results. Moreover, QA manuals from commercial and USGS laboratories that are used to analyze radiochemical and stable isotope samples are kept on file in the Radiochemistry Unit.

6.1.2. Radiochemical and Stable Isotope Contract Determinations

Commercial laboratories are reviewed to ensure compliance with contracts. Radiochemistry staff monitors contract laboratory results using the U.S. Environmental Protection Agency cross-check program. Blind QC samples are prepared and submitted for most constituents in a wide range of values using U.S. Environmental Protection Agency 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 a minimum of 10-percent QC samples (except carbon-14 for which there are no available QC samples). The 10 percent consists of a mix of blind duplicate and prepared QC samples. Reruns are obtained for QC samples that do not check 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. Reruns are requested for samples run in that batch of samples showing a bias.

6.2 In-House Analysis

Currently (1992), the only in-house analysis performed by the Radiochemistry Unit is radon (^{222}Rn) by liquid scintillation counting.

6.2.1 Documentation

A logbook that contains information regarding every ^{222}Rn sample analyzed by the Radiochemistry Unit is kept beside the instruments. The logbook notes the sample identification number, collection date, analysis date, blank value, and standard values. Each instrument has a maintenance logbook that details problems, date of maintenance, and result. Logbooks contain monthly plots depicting blank values, standard values, and standard deviations of those values for each instrument. A copy of the SOP for the analysis of ^{222}Rn by liquid scintillation is kept in the unit chief's office and beside the instruments. The U.S. Environmental Protection Agency'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 was opened.

6.2.2 Reagent Preparation

A mineral oil-based scintillation cocktail is the only reagent used and does not require further preparation. The reagent bottles are dated when they are received and when they are opened. The ^{226}Ra calibration standard is provided by the U.S. Environmental Protection Agency. Standards are prepared in accordance with the SOP. Radiological standards are inventoried monthly. This list is kept in the section files with a copy given to the NWQL safety officer. Semiannually, a copy is sent to the Radiological Advisory Committee.

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 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.

6.2.5 Radiochemistry Quality Control

A blank is run after every 10 samples. The U.S. Environmental Protection Agency ^{226}Ra standards (three of varying activities) are run once a day on each instrument. Samples are analyzed in duplicate.

6.2.6 Data Reporting

Raw data generated by sample analyses are entered into the radon calculation computer program. Results are calculated for ^{222}Rn in picocuries per liter. Computer entries are checked to ensure accuracy. Sample results that do not check with their duplicate results as

specified in the SOP are held until the appropriate USGS District is notified. The average result (in picocuries per liter) and the calculated error of the duplicate samples are entered into the NWIS-I system. The appropriate USGS District is sent a copy of the actual values and calculated errors. Printouts of results are kept on file in the NWQL.

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 Quality Management Group office until the data have been approved for release. SOPs and revisions are filed with the Quality Management Group. Currently (1992) data records are permanently filed. 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 U.S. Environmental Protection Agency's Drinking-Water Regulations also are checked (U.S. Environmental Protection Agency, 1990a; 1990b). 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 Quality Management Group. Further action, such as data verification or reanalysis, might be necessary for the sample.

In order to return sample data quickly to the customer, nutrient samples receive QA checks within the nutrient analytical section. The data values from the Organic Program are validated by a verification printout. Automated Data Processing 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 reruns and data verification about sample data, when sampling site and sampling history indicate an erroneous result.

7.3 Blind-Sample Program

The Quality Management Group administers an internal QA system of blind samples and blanks to assess the Inorganic Program's QC. Sources of water samples for the internal blind-sample program primarily are Standard Reference Water Samples supplied by the Branch of Quality Assurance. Commercially prepared reference materials and standards supplement the blind samples where Standard Reference Water Sample preparations are

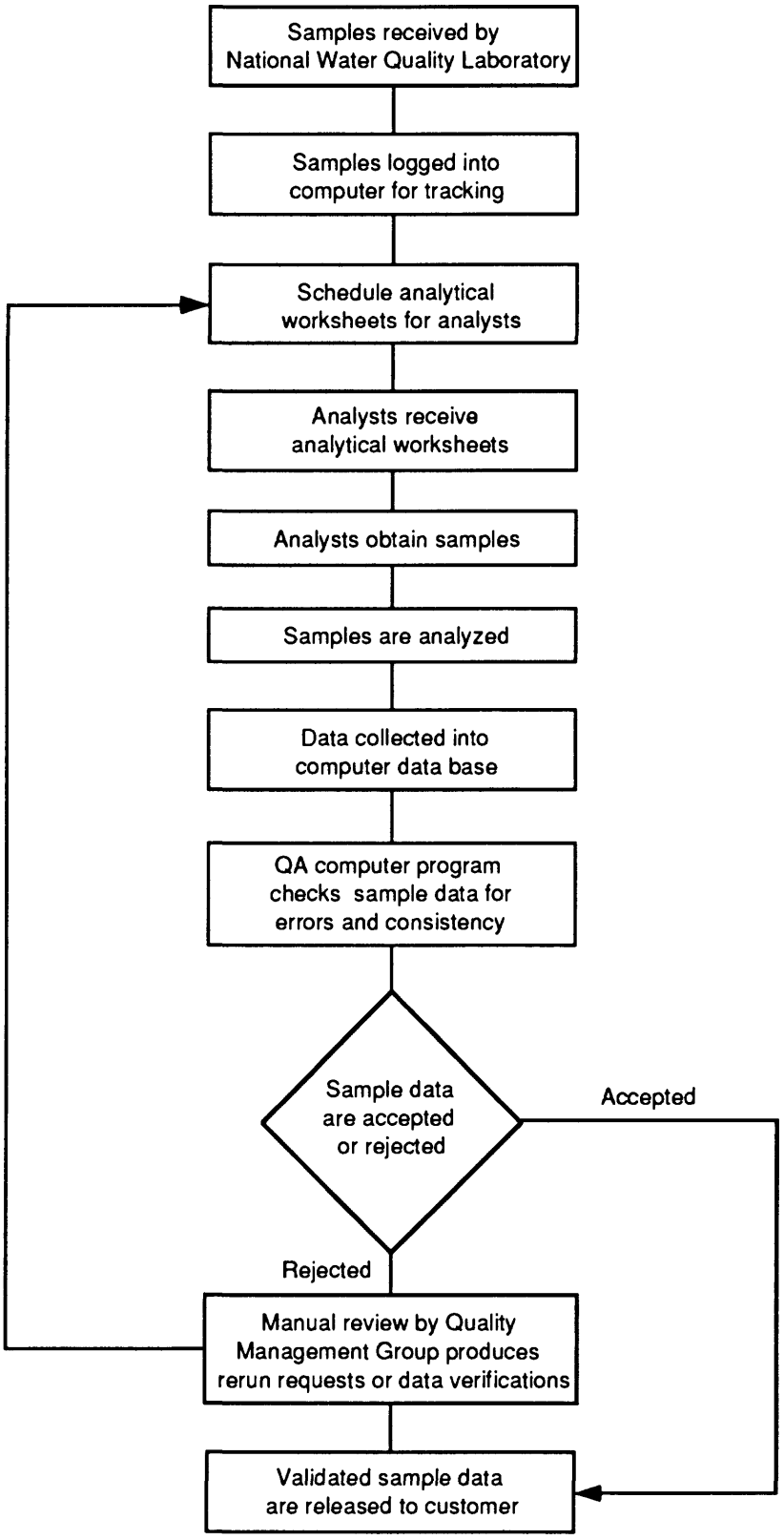

Figure 3.-- Flow of inorganic samples and data review through the National Water Quality Laboratory.

lacking. Blanks are prepared from reagent water with a minimum resistivity of 16.7 megohms-cm. Blind samples are prepared and preserved in the same manner as the onsite samples they represent. Frequency of blind sample and blank insertion for each analytical method is proportional to sample load and increases as sample loads increase.

A computerized data base is used to track the blind samples, monitor, and record results. Results from the blind samples usually are returned to the Quality Management Group one day after analysis. This allows the Quality Management Group to review results quickly and respond with corrective-action reports to the appropriate inorganic sections, 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 Quality Assurance Standard Reference Water Samples. Ranges for commercially available materials are calculated on the basis of certificates of analysis. Monthly, the Quality Management Group produces QC charts for use in comparison to Inorganic Program QC. The data from these comparisons are the basis for monthly Quality Management Group/Inorganic Program meetings, resulting in continuous quality improvement of sample analysis and QC systems.

7.4 External Blind-Sample Program

The Branch of Quality Assurance, which is independent of the NWQL, submits blind samples that are prepared by mixing various Standard Reference Water Samples. The Branch of Quality Assurance monitors not only the organic and inorganic analytical programs, but also the NWQL support functions such as sample Log-in and Quality Management Group areas. Results of these assessments are regularly published by the Branch of Quality Assurance (Lucey, 1989, for example). The Branch of Quality Assurance 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 and inorganic constituents.

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

Canadian Center for Inland Water Samples -- 2 per year. Determination of trace-level concentrations of inorganic compounds.

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

Heidelberg College Study -- 1 per year. Determination of pesticide compounds in water samples.

Results of these studies are reviewed by staff and management. Any questionable values are investigated, and, if necessary, corrective action is taken. Results are sent to the USGS Districts, informing them of the level of performance at the NWQL.

7.6 External Audit Programs

External agencies and customer organizations offer several audits to assess analytical and quality programs at the NWQL. The Branch of Quality Assurance performs annual laboratory reviews. The U.S. Environmental Protection Agency performs triennial audits of NWQL analytical and QA activities that correspond to the Agency's Drinking-Water Regulations. Occasionally, organizations involved in USGS cooperative programs review the NWQL. Recommendations from the various audits help to improve the quality of service that the NWQL provides.

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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 sediments) and analytical methods used. These terms are defined below. 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).

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.

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 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 analysis 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.

Limit of detection. 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.

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 *n*th digit are discarded. If the first of the discarded digits is greater than 5, add 1 to the *n*th digit. If the first of the discarded digits is less than 5, leave the *n*th 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 which 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.

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.

*U.S. GOVERNMENT PRINTING OFFICE:1992-774-207/60018