Quality Management System, U.S. Geological Survey National Water Quality Laboratory

By Thomas J. Maloney, Editor

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National Water Quality Laboratory
Quality Management System

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CONVERSION FACTORS

Multiply	By	To obtain
centimeter (cm)	0.394	inch (in.)
micrometer (µm)	3.94×10^{-5}	inch (in.)
millimeter (mm)	0.0394	inch (in.)
nanometer (nm)	3.94×10^{-8}	inch (in.)
microgram (µg)	3.53×10^{-8}	ounce, avoirdupois
liter (L)	0.2642	gallon (gal)
liter (L)	0.338	ounce, fluid
cubic meter (m^3)	35.31	cubic foot (ft^3)
cubic meter per second (m^3/s)	35.31	cubic foot per second (ft^3/s)

Temperature in degrees Celsius (°C) may be converted to degrees Fahrenheit (°F) as follows:
$°F = (1.8 \times °C) + 32$

Specific conductance is given in microsiemens per centimeter at 25 degrees Celsius (µS/cm at 25°C). Concentrations of chemical constituents in water are given either in milligrams per liter (mg/L) or micrograms per liter (µg/L).

NOTE TO USGS USERS: Use of hectare (ha) as an alternative name for square hectometer (hm^2) is restricted to the measurement of small land or water areas. Use of liter (L) as a special name for cubic decimeter (dm^3) is restricted to the measurement of liquids and gases. No prefix other than milli should be used with liter. Metric ton (t) as a name for megagram (Mg) should be restricted to commercial usage, and no prefixes should be used with it.

ABBREVIATIONS

>	greater than
<	less than
ASR	analytical services request form
ASTM	American Society for Testing and Materials
BQS	Branch of Quality Systems
BDT	Business Development Team
CCV	continuing calibration verification sample
CO	contracting officer
COR	contracting officer's representative
COC	chain of custody
DIAR	U.S. Department of the Interior's Acquisition Regulations
DOI	U.S. Department of the Interior
DQI	data-quality indicators
DQO	data-quality objective
"E"	estimated code
FAQ	frequently asked questions
FAR	Federal Acquisition Regulations
FOIA	Freedom of Information Act
F_σ	F-pseudosigma
FY	fiscal year
GALP	good automated laboratory practices
GSA	General Services Administration
HEPA	high efficiency particulate air
ID	identification
IDC	initial demonstration of capability
IRL	interim reporting level
ISO	International Organization for Standardization
IT	Information Technology Section
LC	laboratory code
LCS	laboratory control sample
LIMS	Laboratory Information Management System

LRL	laboratory reporting level
LT–MDL	long-term method detection level
ME	marginal exceedance
MDL	method detection limit
MRDP	Methods Research and Development Program
MRL	minimum reporting level
MSDS	material safety data sheet
MT	Management Team
NARA	National Archives and Records Administration
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
NIST	National Institute of Standards and Technology
NWIS	National Water Information System
NWQL	National Water Quality Laboratory
pCi/L	picocurie per liter
PE	performance evaluation
QA	quality assurance
QAPP	Quality Assurance Project Plan
QAS	Quality Assurance Section
OBSP	Organic Blind Sample Program
QMS	Quality Management System
QA/QC	quality assurance/quality control
QC	quality control
SHE	Safety, Health, and Environmental Compliance Section
SOP	standard operating procedure
SRS	standard reference sample
URL	uniform resource locator
USEPA	U.S. Environmental Protection Agency
USGS	U.S. Geological Survey
WRD	Water Resources Discipline

Quality Management System, U.S. Geological Survey National Water Quality Laboratory

By Thomas J. Maloney, Editor

1.0 INTRODUCTION

1.1 Scope

The U.S. Geological Survey (USGS) National Water Quality Laboratory (NWQL) established a Quality Management System (QMS) to include all operations associated with its internal management and extending as far as practicable toward the field-sampling component and the data user. Because the NWQL is not directly responsible for field-sampling or the final transfer of data to the USGS National Water Information System (NWIS), the QMS cannot include all aspects of the sampling, analysis, and data-management elements of an environmental program.

All personnel associated with the NWQL, including Federal and non-Federal employees, are bound by the requirements set forth in the policies, processes, procedures, and standard operating procedures (SOPs) included or referenced in this document.

The National Environmental Laboratory Accreditation Program (NELAP), which accredits the NWQL, implements the standards of the National Environmental Laboratory Accreditation Conference (NELAC). The standards incorporate both analytical testing of environmental samples and the laboratory accreditation process (Energy Laboratories, Inc., 2003). The sections, appendixes, and other information in this document are generally arranged according to the contents in Chapter 5, Quality Systems (National Environmental Laboratory Accreditation Conference, 2001) to facilitate the audit process. Management and technical requirements are presented in the text, supported by appendixes containing information on method holding times (Appendix A), quality control (QC) and quality assurance (QA) requirements (Appendix B), radiochemistry (Appendix C), the code of ethics (Appendix D), and a glossary (Appendix E).

1.1.1 Foundational Principles

Safety and health. Protect our most valuable asset, our people, through engineering controls, procedures, training, and visibility of the safety program to promote safe work practices and habits at all times.

Quality. Seek to provide the most consistent, cost-effective data of known quality that a laboratory can produce, providing scientists, planners, policy-makers, and decision-makers with sound, impartial data.

Ethical science and business practices. All laboratory science and business practices are performed to ethical standards. The NWQL staff understands the consequences of unethical behavior and actions. Using training and observation, the NWQL Management Team (MT) actively promotes the prevention of unethical, fraudulent, or questionable practices throughout the laboratory's technical, financial, and personnel operations.

Environmental stewardship and compliance. The NWQL participates in, and contributes to, investigations of the natural environment and its resources, and complies with all applicable laws and regulations in its operations. The NWQL seeks opportunities to improve its environmental stewardship as follows: to reduce or eliminate hazardous materials; to prevent accidental releases to the environment through continuous monitoring and training of Federal and non-Federal employees; and to use the expertise and knowledge to reach students, professionals, and groups interested in protecting the environment.

Science excellence through innovation and change. Endeavor to be a leader in laboratory pursuits and operations as evidenced by the NWQL publications, analytical products, quality of analytical data, and overall mission support to the USGS and the U.S. Department of the Interior (DOI).

Customer service. Use our talents, skills, knowledge, and passion to serve the USGS mission and assist our customers to achieve their scientific goals and objectives.

Driving force. Be a respected water-quality laboratory offering data of known quality, innovation, best practices, consultation, long-term stability, and leading-edge science in analytical chemistry and biology.

1.2 Quality

The purpose of the NWQL QMS is to establish and execute a set of management policies, procedures, and practices that together provide a foundation for producing analytical results that are consistent and meet the data-quality needs of our customers.

The NWQL operates under a philosophy of "mission first," meaning that the mission of the USGS is the driving focus of the laboratory's efforts and purpose for being. The primary mission of the NWQL is to support USGS programs that require long-term, consistent, analytical chemistry and biological data of known quality for use in national assessment and trends analysis. The NWQL also provides research capabilities, expertise, and consultation to the USGS with primary emphasis on the Water Resources Discipline (WRD). To this end, the laboratory uses its three most important strengths — people, quality, and safety — to provide consistent, high-quality analytical data, new analytical methods, contributions to scientific journals, and expert consultation to customers throughout the USGS.

1.3 Ethics Program

The NWQL, through its management, Federal and non-Federal (including contractors) employees, is committed to maintaining a sound and active ethics program. The NWQL MT recognizes its technical and fiduciary responsibilities and the role model it must establish that allows all personnel to express their concerns openly and freely. The MT further commits itself to provide adequate resources for promoting and maintaining ethical behavior by all Federal and non-Federal employees.

The NWQL ethics program, established by management, was developed to ensure that the commitment to ethics is functional and accountable. The primary components of the program include an ethics coordinator, the DOI ethics policy (accessed July 13, 2005, at URL http://www.doiu.nbc.gov/orientation/ethics.html), the NWQL Code of Ethics (Appendix D), the draft DOI Code of Scientific Conduct and Commentary, and training. The draft Code of Scientific Conduct and Commentary is included in Appendix D and may also be accessed at URL http://internal.usgs.gov/director/draftscidm.doc.

Ethics coordinator. The NWQL ethics coordinator is responsible for organizing and tracking ethics training for all Federal and non-Federal staff members. Additionally, the coordinator serves as a channel for staff members to report ethical concerns freely and to ensure that these concerns are brought to the attention of laboratory management.

NWQL ethics policy. The NWQL uses the draft DOI, Manual, Chapter 3: Code of Scientific Conduct and Commentary, as the basis for its ethics policy (see Appendix D). It establishes a uniform policy to govern the conduct of science for DOI and its Bureaus, and applies to all NWQL Federal and non-Federal staff engaged in conducting, supporting, or managing scientific activities on behalf of DOI.

Although the NWQL ethics training program predates this draft departmental code, the DOI policy provides a strong foundation, based upon broader Federal codes and policies relating to government research misconduct and standards of official conduct. The MT fully endorses, adheres to, and enforces all aspects of this DOI policy.

NWQL code of ethics. The draft DOI Code of Scientific Conduct and Commentary contains an Ap-

pendix entitled "The Code of Scientific Conduct." The MT fully endorses, adheres to, and enforces all aspects of this code. The NWQL produced its own Code of Ethics that focuses on specifics related to laboratory operations to supplement the DOI code (see Appendix D).

1.4 Safety and Health

The NWQL is committed to providing a safe and healthful environment for Federal and non-Federal employees, contractors, and visitors through a program of compliance with the Occupational Safety and Health Act, Executive Order 12196 (Occupational Safety and Health Programs for Federal Employees), and all applicable Federal, State, and local regulations. The primary basis and requirements for the NWQL Health and Safety Program are in the handbook (U.S. Geological Survey, 2002a).

The NWQL MT is responsible and accountable for creating and maintaining a safe working environment through effective health and safety programs, regular inspections and assessments of the workplace, staff training, and guidance and support to safety efforts. Everyone is responsible for working safely by following established safety and operating rules and procedures, maintaining an awareness of potentially hazardous situations, reporting all unsafe conditions to supervisors, and reporting all accidents or incidents that result in, or could have potentially resulted in, personal injury or property damage.

The Safety Program applies to all activities and operations of the NWQL, as well as the Federal and non-Federal employees, volunteers, contractors, and visitors that it serves. The program includes the following elements:

1. The development of organizational policy, plans, programs, directives and rules, and interpretation of safety and health policy and procedures, to include management and personnel accountability, establishment of councils, committees, and working groups that address safety, health, and environmental issues and the designation of appropriate personnel and financial resources for program implementation.

2. Ensure personnel awareness of, and accessibility to, applicable policy, documents, codes, regulations, and program standards.

3. Annual evaluations of program elements, which includes personnel and financial resources, to provide management with information on program effort and effectiveness and to establish short- and long-term goals for program enhancement and implementation.

4. Conduct operational and facility surveys, inspections, evaluations, and staff visits for the purpose of identifying hazards within the workplace and determining the level of organizational compliance with standards.

5. Hazard identification and abatement for organizational reporting and correction of unsafe or unhealthful working conditions and identification/correction of workplace hazards through job hazard analysis.

6. Investigate, report, and analyze accidents, providing necessary corrective actions and tracking corrective measures through abatement to prevent recurrence.

7. Identify, develop, coordinate, schedule, and conduct required training.

8. Standard and regulatory compliance assistance, awards and recognition programs, and development of safety and health promotion/awareness plans.

9. Industrial Hygiene/Occupational Medicine studies to include hearing conservation, respiratory protection, personal protective equipment, and laboratory safety.

10. Ensure that NWQL facilities and operations are compliant with established fire safety practices and policies.

11. Ensure that operators of motorized vehicles are identified and trained and that equipment is maintained in safe working condition.

12. Provide equivalent safety protections for non-Federal employees, contractors, concessionaires, volunteers, and visitors.

13. Provide specialized program assistance and coordination.

1.5 Environmental Compliance

The NWQL complies with applicable environmental regulations, including, but not limited to, the Comprehensive Environmental Response, Compensation, and Liability Act; the Resource Conservation and Recovery Act; and Executive Order 13148 (Greening the Government Through Leadership in Environmental Management). The primary basis for the NWQL Environmental Compliance Program is the handbook (U.S. Geological Survey, 2002b).

All USGS Federal and non-Federal employees are responsible and accountable for complying with environmental rules and regulations that apply to their duties, maintaining a general awareness of all applicable USGS environmental policies and goals, applying environmentally safe practices and pollution prevention to daily operations, and reporting all unsafe and/or unhealthful conditions that may negatively affect the environment. Their responsibilities include properly collecting and storing laboratory wastes and complying with NWQL's wastewater discharge requirements.

The NWQL closely monitors all discharges and has developed a strong culture not to discharge anything from a chemical process into the sewer lines. The NWQL has obtained variances from the local wastewater district to neutralize acidic waste and routinely performs tests to ensure that the waste meets discharge requirements prior to disposal.

The preferred method of environmental protection identified in the USGS handbook (2002b) is eliminating or controlling a source of contamination. Laboratory personnel must identify ways of eliminating or minimizing contaminants and, where possible, incorporating them at the earliest stages of planning, designing, and implementing analytical methods (U.S. Geological Survey, 2004). U.S. Geological Survey (2004), National Water Quality Laboratory Policy Memorandum 04-01, *Pollution prevention and waste minimization policy*, requires each USGS employee to minimize waste and prevent pollution in all laboratory activities. It also requires that as SOPs are reviewed, any means of reducing or eliminating the amounts and types of waste should be examined. Reducing the volume of standards or reagents prepared or substituting less hazardous chemicals are other possibilities.

The Environmental Compliance Program applies to all activities and operations of the NWQL, as well as the Federal and non-Federal employees, volunteers, contractors, and its visitors. The program includes the following elements:

1. Provide safe and healthful working conditions to protect Federal and non-Federal employees and the visiting public from injuries and illnesses, and property from accidental damage.

2. Include environmental protection and compliance as an integral part of every operation.

3. Ensure Federal and non-Federal employees are aware of, and have reasonable access to, applicable documents, codes, regulations, and program standards.

4. Conduct annual evaluations of program elements, inclusive of personal and financial resources, to establish short- and long-term goals for program enhancement and implementation.

5. Conduct operational and facility evaluations for identifying current and potential noncompliance areas within the workplace.

6. Take appropriate steps to prevent a noncompliance situation from arising or determine actions to be taken to correct an area of noncompliance.

7. Identify, develop, coordinate, schedule, and conduct required training to the appropriate audiences.

8. Ensure contractor, concessionaire, volunteer, and other non-Federal employees working or performing duties in the NWQL comply with the environmental rules and regulations.

2.0 MANAGEMENT REQUIREMENTS

2.1 Organization

2.1.1 Laboratory Management

The USGS NWQL, also known as the Branch of Analytical Services, reports directly to the Chief, Office of Water Quality, who in turn reports to the Associate Director for Water, the head of the Water Resources Discipline (WRD) in Reston, Va. The USGS is a bureau within the U.S. Department of the Interior.

The NWQL's management structure provides clear lines of authority and responsibility to help ensure timely, informed decision-making. The laboratory is comprised of sections, each with its own manager and subordinate supervisors as required (fig. 2.1). All Section Chiefs report directly to the Laboratory Chief. The NWQL Management Team (MT) includes the Laboratory Chief, Assistant Chief, and all Section Chiefs. The NWQL Management Group includes the MT plus all subordinate supervisors.

The laboratory is operated primarily with Federal employees. However, non-Federal, contract staff are used to provide additional support and capabilities for various laboratory operations. Federal employees are prohibited from supervising contract personnel. Contract employees are administered by the government contracting officer (CO), who is assisted by an appointed contracting officer's representative (COR) in the laboratory. The contractor is responsible for all task recruitment and task assignments for its employees.

Throughout this QMS, the terms "staff" and "personnel" refer collectively to all persons, Federal, or non-Federal, who are responsible for adhering to the requirements set forth by the QMS. These terms should not be confused with the actual employer of a particular individual.

2.1.2 Management Roles and Responsibilities

Statements that describe the mission and function of each section of the laboratory ensure adequate coverage of all management functions and responsibilities. These broad management areas are further specified in a management structure and functional responsibilities document. The latest version of this analysis is entitled *FY02 management structure and functional responsibilities, final revision: 21 December 2001* (National Water Quality Laboratory, written commun., 2001).

All Section Chiefs are accountable for the specific mission and functional elements specified.

Office of the Chief. Includes the Chief and Assistant Chief. The Assistant Chief is delegated the full authority of the Chief in his absence.

Mission. Responsible for overall management and technical and administrative leadership of the NWQL.

Functions. The Office of the Chief, NWQL, is responsible for providing mission critical analytical support services to WRD and other USGS components. Mission support is expected to be of high quality using efficient business practices to provide cost-effective products and services that meet the requirements of customers.

Priorities, budgets, and capital expenditures are developed within the NWQL to optimize human and material resources for routine and nonroutine support requirements. The Office of the Chief is ultimately responsible for maintaining a safe working environment for all personnel and ensuring the quality of all products and services.

Administrative Section

Mission. Provide administrative direction, management, and coordination to management, and Federal and non-Federal employees.

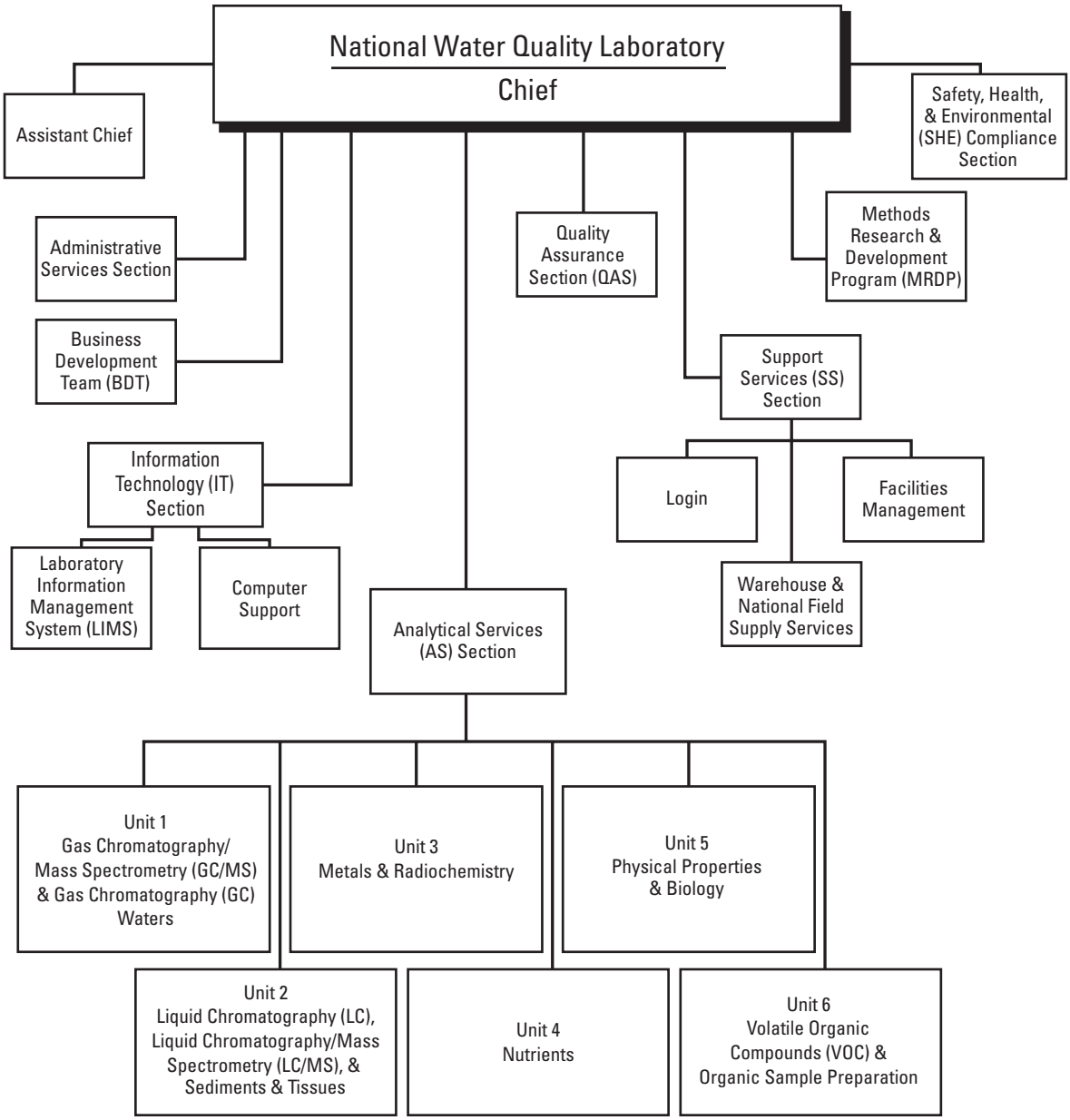

Figure 2.1. Organization of the National Water Quality Laboratory.

Functions.

- Financial and accounting management. Commitments, obligations, and expenditures that include processing requisitions, bankcard purchases, and convenience checks; preparing travel authorizations and vouchers, purchase orders and contracts; initiating and authorizing vendor payments; and reconciling monthly and annual financial reports.
- Budget management. Provide centralized budget and expenditure controls based on fiscal-year budget estimates; initiate program funding levels from allocated and Working Capital Fund customer income. Initiate internal voucher converting customer income into cost-center cash flow. Reconcile financial records throughout the fiscal year for both current and active prior fiscal years.

- Personnel management. Process personnel actions; provide guidance on position descriptions, employee relations, awards, training, and performance plans. Provide assistance and guidance to the supervisor. Process time and attendance records for 150 Federal employees.

Analytical Services (AS) Section

Mission. Provide the long-term, high-quality analytical chemical and biological data that meet the needs and requirements of NWQL's customers.

Functions. To offer a wide range of quality analytical services to support an integrated approach to water-quality monitoring by NWQL's customers. Provide organic and inorganic analytical services, in-house radiochemical analytical services, biological taxonomic services and population estimates, and develop custom services proposals. Provide customer data services and consultation.

Business Development Team (BDT)

Mission. Clarify roles and responsibilities within the NWQL. Define customer expectations. Improve internal and external communication. Implement a customer feedback program. Reduce response times for problem resolution. Investigate new markets and products.

Functions. To serve as the primary laboratory contact for customers and to manage all primary customer communications, including online publications and data base. Focus on customer needs and satisfaction with services offered by the NWQL. Develop effective communication pathways and techniques to improve focus and consistency. Assist customers with concerns, data tracking, and provide analytical expertise to customers requesting assistance in developing project plans. Develop enhancements for the NWQL communication tools, including the web sites and newsletter.

Information Technology (IT) Section

Mission. Provide computer infrastructure and software support, assure data integrity and security, and provide continuous quality improvement through state-of-the-art technology and practices.

Functions. Plan, coordinate, install, and maintain the Information Technology infrastructure at the NWQL, which includes the Laboratory Information Management System (LIMS), analytical data bases, and National Water Information System (NWIS) interface. Research advanced software and hardware technology. Make recommendations concerning IT innovations and data security, and their possible effect on the laboratory environment. Serve and advise the NWQL on compatible software and hardware and develop and deploy corporate and custom software required to support the laboratory and its staff.

Methods Research and Development Program (MRDP)

Mission. Develop, test, evaluate, and implement new methods and improve existing methods for chemical analysis of surface and ground water, and sediments for the NWQL and USGS.

Functions. Apply newly developed methods in national and regional water-quality studies in collaboration with other scientists. Provide consultation on the design of experimental programs, including sampling protocols, and the quality assurance/quality control (QA/QC) of data produced by analytical methods. Provide new methods development and implementation, method improvement, and specialized support for NWQL Analytical Services and to meet the mission requirements of other customers, such as national programs. Manage technical support allocations and discipline goods expenditures, support QA/QC functions, consult on the selection and acquisition of scientific instrumentation, and provide USGS-wide technical training. Interact with the scientific community through attendance and presentations at technical meetings, publication and review of journal articles, and participation in committees.

Quality Assurance Section (QAS)

Mission. Develop and operate a comprehensive quality system to maintain laboratory accreditation, document the quality of NWQL data, and ensure the integrity of data produced by the laboratory.

Functions. Ensure programs and procedures are in place to determine and quantify the bias and variability in analytical methods. Oversee the quality management system for the NWQL, including administration of performance evaluation studies, internal audits, long-term method-detection-level project, and blind blanks. Provide specialized radiochemical and stable isotope analytical support through analytical contracting. Process standard operating procedures (SOPs). Various SOPs are cited throughout this document; copies of specific procedures are available on request.

Safety, Health and Environmental (SHE) Compliance Section

Mission. Manage and direct processes, policies, and procedures to ensure a safe and healthy work environment for Federal and non-Federal employees, and provide proper disposal of all laboratory hazardous waste. Ensure that the NWQL procedures, techniques, hazardous materials, and waste streams are in compliance with pertinent rules and regulations.

Functions. Responsible for the safety and occupational health of Federal and non-Federal employees, the safe operation of the laboratory, and all environmental compliance requirements associated with laboratory operations and hazardous waste storage and disposal. Operational responsibilities include hazardous waste management, sewer monitoring and compliance, required regulatory activities, accident and incident coordination, fire safety, and security. The SHE oversees emergency response coordination and manages health and safety, environmental compliance, and employee training and certification. The SHE follows the required Federal regulations (U.S. Geological Survey, 2002a) and NWQL guidelines.

Support Services (SS) Section

Mission. Provide logistical, sample processing, warehousing, supplies, and facilities support to NWQL analytical functions, and sample collection supplies to WRD field operations.

Functions. Provide logistical and facilities support to all NWQL functions. Provide critical sample receipt and login functions for the NWQL. Provide supply support to WRD field activities. Determine existing support requirements, specify facility needs, define future logistical and facility needs, and provide ongoing communication with customers.

2.2 Quality System

To meet the mission requirements of the USGS effectively, the NWQL adheres to high standards of quality in the operations within the laboratory. This includes all aspects of the testing services that make up the laboratory's core business and all of the critical elements that support this operation.

The NWQL QMS is designed primarily for internal processes, but also extends to field operations. This includes provision of quality-assured field-sampling supplies and sample transport guidance, recognizing that an effective quality system needs to be based upon a strong field-laboratory partnership.

The QMS also includes the end user of data products by providing post-delivery consultation, data checks, and sample reanalyses for customers.

NWQL MT accepts full responsibility and accountability for the quality of the products the laboratory delivers, including analytical data, research and development, consultation services, and fiscal operations. Management recognizes that statements regarding quality are insufficient to keep pace with the pressures of workload demands, and seeks innovative methods and opportunities to communicate to maintain high visibility and underscore quality. This visibility and value is conveyed and owned by every worker in the laboratory and with external groups that provide critical support services, such as acquisition.

Completely analogous to the safety culture of the laboratory, every manager, supervisor, and Federal and non-Federal employee has the authority to "pull-the-cord" and stop the analytical process, should they consider it to be out of analytical control or if any other situation occurs that they believe can or will affect the quality of the final data product.

2.2.1 Elements of the Quality Management System

The NWQL uses multiple internal and external systems and processes to assess its daily operations and long-term data product quality. The laboratory also seeks to improve these processes through review, outside consultation, and customer feedback. Some of these basic elements include the following:

Quality control (QC)

- continual online QC
- secondary data reviews

External quality assurance (QA)

- double-blind and standard reference sample programs administered by the USGS Branch of Quality Systems that is independent of the NWQL and its management structure
- external performance evaluations
- external audits

Internal QA

- internal blind-blank assessments
- research chemists assist by overseeing analytical processes and resolving problems
- internal audits
- continuous assessment of reporting levels using the internal long-term method-detection level (LT–MDL)

Communication

- participation in national committees and professional societies
- active contract management for subcontracted work
- active interaction with customers, including development of Quality Assurance Project Plans

Other

- ethics program

2.3 Managing Records

2.3.1 Types of Records

The NWQL maintains records that support its management and technical policies, confirm that procedures have been followed, and provide support for the technical interpretations, judgments, and discussions concerning laboratory results. These records, particularly those that are anticipated to be used as evidentiary data, provide the historical evidence needed for later reviews and analyses. SOPs identify the records necessary to meet this commitment.

The storage and retention of records is in compliance with Federal record retention requirements found in USGS General Records Disposition Schedule 432-1-S1 and the WRD Schedule 1400 series (U.S. Geological Survey, 2003a). Records to be maintained are sufficient to reconstruct laboratory activities that produce analytical data. Records include, but are not limited to, documentation of facilities, equipment, analytical methods, all aspects of sample handling and data verification.

Records are legible, recorded in indelible ink, identifiable, and retrievable, and protected against damage, deterioration, or loss.

Records maintained on-site. Records that are stored or produced by computers or personal computers have paper copies or electronic backup copies. These laboratory records usually are maintained near

the analytical laboratory or the analysts' workstation until final analysis and data submission. These records may be stored for several months or up to 1 year. Types of laboratory records include, but are not limited to, the following:

Standard operating procedures (SOPs). Any revisions to laboratory procedures are written, dated, and initialed by the supervisor and Chief, QAS, and distributed to all affected individuals to ensure implementation of changes.

Laboratory notebooks. A record of the method and other information pertinent to the conduct of analytical tests.

Calibration records. The date of analysis, initials or signature, frequency, conditions, standards, and reference materials.

Sample management. A record of procedures to which a sample is subjected while in the possession of the NWQL is maintained. These include records pertaining to the following:

- sample preservation, including appropriateness of sample container and compliance with holding-time requirement;
- sample identification, receipt, acceptance, or rejection and login;
- sample storage and tracking for shipping receipts, transmittal forms, and internal routing and assignment records, where possible;
- sample preparation and analysis documents; and
- disposal of hazardous samples, including the date of sample or subsample disposal and name of the responsible person.

Unprocessed data. The unprocessed data and calculated results for samples are maintained in laboratory notebooks, logs, bench sheets, electronic files, or other sample-tracking or data-entry forms as appropriate for the method. Instrumental data information is stored in a computer file or a paper report. These records may include the following:

- laboratory sample identification code;
- date of analysis;
- instrument identification and instrument operating conditions/characteristics;
- analytical method used and sample preparation information, including sample aliquots processed, cleanup, and separation protocols;
- manual, automated, or statistical calculations;
- confirmatory analytical data, when required to be performed;
- the analyst's or operator's initials or signature;
- unprocessed data and calculated results for all QC samples; and
- source and lot numbers of standards and reagents for traceability.

Correspondence. Correspondence (paper and electronic) for Quality Assurance Project Plans and NWQL proposals pertinent to a project, usually a written agreement between USGS Water Science Center and NWQL personnel, and communications regarding the progress of the project, are kept in accordance with U.S. Geological Survey (2003a) general records disposition schedule 432-1-S1, chap. 100, ¶102–02, Project Case Files.

Deviations. Deviations from established methods are documented in appropriate SOPs or individual data packets.

QA records from activities performed by QAS.

- QA program summaries,
- LT–MDL assessments,
- in-house audit findings,

- blind blank program studies,
- QMS,
- radiochemistry and stable isotope contracts,
- files of all changes to each method that could affect data quality,
- analysts' training records, and
- demonstration of capability for each analyst.

Administrative records.

- personnel qualifications, experience, and training records
- log of names, initials, and signatures for all individuals who are employed at the NWQL.

Electronic data. Electronic data are stored on secure servers or password-protected personal computers and backed up on a regular schedule. Data are stored so that they are protected from damage and destruction.

1. Required signatures on records and documents. All documented entries are signed or initialed by the Chief of the section creating or approving the records or documents for official use. The reason for the signature or initial is clearly indicated in the records, such as prepared, reviewed, or approved by.

2. Correcting documentation errors. Any documentation errors are to be corrected by drawing a single line through the error so that it remains legible and is initialed by the responsible individual, along with the date of change.

A correction is written next to the error accompanied by the reason for the change. The person who performed the instrumental analysis (or that person's supervisor) signs or initials the report printouts.

Correction fluid, self-stick removable notes, pencils, erasures, and obliteration of information are prohibited on analytical data records.

2.3.2 Types of Quality Documents

The NWQL has controlled and uncontrolled documents. The only controlled document is the QMS. A controlled document is used primarily by laboratory staff. Uncontrolled documents consist of SOPs, various published analytical methods, manuals for instruments and other equipment, and log books.

When copies of a controlled document are circulated outside the laboratory or may be downloaded by customers from the USGS-visible web site, the copies are designated as uncontrolled documents.

2.3.2.1 Controlled Documents

QMS. The QMS describes the NWQL quality system policies and their implementation as is within the laboratory. The QMS is updated as needed to reflect changes in the laboratory's processes, operations, or procedures, and to be in compliance with current National Environmental Laboratory Accreditation Conference (2001) (NELAC) guidelines for quality systems, Chapter 5 (effective July 2003).

2.3.2.2 Uncontrolled Documents

SOPs. These documents describe how a method is performed at the NWQL and include all the written directions necessary to perform methods within the laboratory. They supplement and expand the information contained in the selected method, and provide a working document in which all details are specified.

All SOPs follow one of two standard formats that are described in QUAX0001.3, *Writing and approving standard operating procedures (SOP) at the National Water Quality Laboratory*. The latest revision of all SOPs can be viewed on the NWQL intranet by accessing URL http://wwwnwql.cr.usgs.gov/nwql/sop/sops.html and selecting an operational heading. The intranet site is only accessible within the NWQL. The latest revision of an SOP always supersedes a reference in this document. An SOP may be revised or updated between revisions by amendment report. USGS employees requiring SOPs for project development or to prepare quality assurance project plans with coop-

erators can request copies of SOPs by contacting the NWQL. See section 2.5.5 for other information on quality assurance projecct plans.

All SOPs are assigned an alphanumeric identifier that identifies the functional group writing or using the procedure, a sequence number, and a version number. The appropriate Section Chief is responsible for ensuring that all SOP documentation is complete and current, and that the SOP is followed in the laboratory.

The master copy of each SOP is filed in the QAS master SOP file. Supervisors distribute additional copies of an SOP to the analyst(s) who will be performing the work. Supervisors are responsible for ensuring that these analysts are trained against each SOP. Documentation of such training is maintained in an individual's employee training file.

Paper copies reside in the laboratory where the method is used; the master list of SOPs is maintained by the QAS. Upon revision, the latest version is approved by the Chief, Analytical Services, and updated on the web. An electronic mail or memorandum is sent to all NWQL staff to ensure the updated version is changed in their documentation.

Analytical methods. Copies of published or approved methods are used to perform analytical tests at the NWQL.

Equipment manuals. The manufacturer's manuals associated with each piece of equipment or instrument are kept in the laboratory, near the instrument to which it applies. The manuals contain general information, such as instrument identification information, maintenance requirements, calibration schedules, operating procedures, and safety information.

Log books. Each piece of equipment has a log book located near it. Content of instrument log books may include instrument maintenance records, calibration, and analysis information. Analytical results can be reconstructed from the log books, data packets, and QC data. The information documented in log books for nonanalytical equipment is specified in the relevant SOP. Entries must be accompanied by an analyst's initials or signature, and the date.

2.3.3 Procedures for Document Control

Document control ensures that personnel have access to current policies and procedures at all times. Quality documents that are placed under a controlled distribution include, but are not limited to, the QMS.

Control is maintained by initially distributing the documents to staff members who must be aware of or follow the information or procedures. The QMS is distributed to all staff; electronic copies of SOPs reside on the NWQL intranet web site in read-only format.

Subsequent revisions or updates to the QMS are first made only to the QAS copy until the next version is published, when they become effective. Controlled copies are clearly identified as such, and a controlled copy distribution list is maintained with the name of each staff member who received a copy along with their control copy number. The QAS maintains records of controlled distribution. A file is kept of all controlled documents and the following information is required for every controlled document:

1. Document control procedures allow for adequate documentation and control of specific documents. These procedures use a unique identification system that allows for tracking, training, documentation, and traceability of official copies, and the time period the procedure or document is in force.

To ensure the QMS remains a controlled document, the original official version of the QMS and copies are identified. The NELAC title page of each controlled copy includes a stamp (identifier) that, when filled in, shows that the document is controlled copy X of Y, initialed and dated by the Chief, QAS, in red ink. This enables an analyst to identify and use the current or acceptable update or version of the QMS.

2. The QMS Distribution Form is prepared for a version of the QMS. The form includes the QMS stamp or identifier, control number, individual receiving the QMS, and the issue date.

2.3.4 Storage and Disposal of Records

Storage and disposition standards for all laboratory records are identified in the General Records Schedule developed in accordance with 44 U.S.C. Chapter 33, *Basic laws and authorities of the National Archives and Records Administration* (NARA) (U.S. Code of Federal Regulations, 2005). Usually, administrative files have short retention periods, less than 3 years. Program subject files may be needed by the agency for 30 years or more, and may have archival value as well. Administrative records are maintained separately from program records, such as laboratory analytical data. USGS protocols have established that laboratory data should be kept for 30 years.

It is the responsibility of supervisors in each laboratory unit to use the correct disposition schedule to ensure that all records are stored in a secure manner and are easily retrievable.Records disposition schedules are identified in the USGS general records disposition schedule 432-1-S1 containing the WRD mission-specific records disposition schedule series 1400 (U.S. Geological Survey, 2003a).

Paper copies of administrative and analytical records are kept in the unit or section for about 1 year. The records are then boxed and moved to the NWQL records staging area on the first floor of Building 95, for a period of 3 additional years. Records that need to be retained for a longer period are then transferred to the Federal Records Center. All records are disposed of after a period of 30 years unless otherwise specified by legal or other written agreements.

Electronic records disposition criteria are being developed at the agency and NARA but have not been completed. At the present time, paper files are considered as the official record. Electronic files of analytical results are available on individual analytical instruments and on the LIMS. Final concentrations for analytes are stored in the LIMS. The current LIMS includes results for the past 10 years. Migration of data to new systems is a fundamental consideration that is addressed when operating systems are upgraded.

As a Federal laboratory, all analytical data produced at the NWQL are accessible by the public through the Freedom of Information Act (FOIA). Requests by a third party for access to any records must be accompanied by a FOIA request. The Chief, QAS, is the FOIA control point.

2.4 Acquisition

2.4.1 Acquisition Requirements

The internal web site providing guidance on processing acquisition requirements can be found at URL http://internal.usgs.gov/ops/acquisition/acqguide.html.

The technical expert is responsible for observing all NWQL policies and procedures when performing a review that can lead to an acquisition action. This ensures the following:

1. The requirements are adequately defined, documented, and understood. The requirements include chemical and laboratory supplies, service and maintenance of laboratory equipment, and equipment acquisition.

2. Depending upon the acquisition requirement and dollar amount, an acquisition may require a statement of work prepared by the technical expert in accordance with established acquisition requirements.

3. For acquisitions that fall into the category of contracts, the NWQL has the capability and resources to meet the requirements through established acquisition procedures. Bureau procurement offices assign a contracting officer (CO) for the contract process, and the laboratory assigns a contracting officer's representative (COR) to sit as the chairman of the Technical Evaluation Committee and technical representative of the contract once awarded. The COR is responsible for accepting the service and authorizing vendor payment.

4. A contract or purchase order is a written agreement that outlines the laboratory requirements.

5. The technical expert is responsible for submitting the appropriate acquisition documents to the Administrative Section.

2.4.2 Acquisition Request and Award

1. Acquisition requests are submitted through the appropriate process to the Administrative Section.

2. Submission of an acquisition request to the Administrative Section determines the appropriate acquisition method, which is based upon dollar amount and acquisition requirements.

3. Acquisition methods range from bankcard, convenience check, purchase order and/or contract, depending upon the requirement and dollar amount.

4. The guidelines for procurement include the following:

a. Federal Acquisition Regulation (FAR);

b. Department of the Interior's Acquisition Regulations (DIAR); and

c. established Bureau policies in conjunction with the method of procurement, dollar limitations, and procurement authority.

5. Acquisition documentation may include some, but not all of the identified documents, depending upon acquisition method: requisition, statement of work, sole-source justification, or vendor quote.

2.4.3 Acquisition Payment Documentation

The packing slip and the acceptance signature of item(s) are required before payment of the vendor invoice is processed. The documents from paragraph 2.4.2(5) above are reviewed and combined with the invoice and receiving report, which completes the acquisition package. Certification, approval, and processing of payment are accomplished in accordance with established Prompt Pay Act and Bureau-established guidelines.

2.4.4 Subcontracting Analytical Services

USGS and NWQL policy requires that all contract laboratories, where applicable, be accredited by NELAP. This requirement is included in all Statements of Work, which form the Solicitation and Request for Proposals. The NWQL subcontracts part of its radiochemical analyses. The following procedure is required for subcontracting analytical services:

1. The acquisition of services and/or goods is based on requirements developed by the technical expert.

2. The acquisition package contains a requisition (DI1 form), statement of work, period of performance, and estimate of cost.

3. The Contracting Office issues a Request for Proposal – Contract Officers in accordance with FAR and DIAR policies.

4. The duties of the Technical Evaluation Committee Chairman and the contracting officer's representative (COR) are usually performed by the technical expert.

5. The Technical Evaluation Committee rates and ranks all proposals from applicants.

6. The process concludes with the best and final offer and final award of the contract.

2.4.4.1 Financial Management of Subcontracts

The Administrative Section of the NWQL handles the financial management of subcontracts as follows:

1. Upon receipt of vendor's invoice, a cover memorandum is prepared for the COR's acceptance signature.

2. Once the COR's signature and acceptance is received on the cover memorandum, the invoice and

cover memorandum are forwarded to the contracting officer for approval.

3. When the cover memorandum and invoice are returned to the Administrative Section, they are processed for payment in the Federal Financial System.

4. The Administrative Section maintains an on-going payment log sheet that reflects the amount obligated for the contract and each subsequent invoice processed against the obligation.

5. At the end of each fiscal year, any outstanding obligations are de-obligated.

6. If the COR determines that the services and/or goods are unacceptable, the cover memorandum is so annotated and forwarded to the contracting officer to notify the vendor of reasons for the 'not acceptable' notices and the nonprocessing of the invoice for payment until deficiencies are corrected.

2.4.5 Automated Billing Records

At sample login, billing information is recorded in the Laboratory Information Management System (LIMS) by userid (state code), USGS Water Science Center account number (a four-digit cost center code, and alphanumeric project number), sample quantity, and laboratory test. LIMS produces weekly reports of sample login, costs, and monthly billing files. These files interface with the USGS's Federal Financial System, and are the basis for billing customers and receiving payments.

Customer-service web site. The NWQL has a customer-service web site that provides frequently asked questions (FAQs), a billing schedule, and 'How to' instructions for new users. This site is accessible on the NWQL USGS-visible intranet web site at URL http://nwql.cr.usgs.gov/usgs/billing/index.cfm.

2.4.6 Receipt of Supplies

The Support Services Section receives supplies in the laboratory warehouse and examines packing slips to determine package contents and to identify the ordering group. Unit supervisors are responsible for assessing and approving the quality of supplies before they are stocked and used. Acceptance testing procedures are documented and records are maintained within the appropriate group. Quality assurance of supplies is described in SOP QX0347.0, Acquisition, quality assurance, and sale of field supplies.

All original supply containers include an expiration date. If an expiration date is not provided by the manufacturer, the date is 5 years after receipt or as determined by the NWQL.

2.5 Customer Service

Communication is the foundation of customer service at the NWQL. Direct communication with a customer via the telephone or electronic mail is used to obtain additional information needed to resolve sample login problems or answer questions.

Information on the availability and quality of routine analytical services, administrative support, field supply information, and method development is available on the NWQL USGS-visible intranet site. The LIMS or NWQL services catalog is the primary means of announcing routine services. The Sample Status, USGS Water Science Center Rerun Request, and Update/Reload pages each allow customers to find out information about samples they submitted. The Customer Billing Report page provides financial information to USGS Water Science Center customers and is accessible on the NWQL USGS-visible intranet site at URL http://nwql.cr.usgs.gov/usgs/billing/index.cfm.

Broad-based notifications and information on nonconformance and corrective actions taken by the laboratory are released to customers electronically using the NWQL's Rapi-Note system. Technical Memoranda are issued when more detail or discussion is necessary. Copies of all Rapi-Notes and Technical Memoranda are posted on the NWQL USGS-visible intranet site.

The NWQL resends data to a customer with appropriate qualifiers upon the completion of an evalua-

tion of nonconforming laboratory work. These qualifier codes indicate the type of nonconformance. A list of these qualifier codes is maintained and described on the USGS National Water Information System (NWIS) internet site at URL http://phoenix.cr.usgs.gov/www/rmk_qual.html.

2.5.1 Service to Customer

The NWQL provides customer service to its customers using LabHelp and LabLogin electronic mailboxes. Trained staff from the Business Development Team handle LabHelp customer inquiries. The Support Services Section is responsible for LabLogin, which monitors sample login inquiries. There also is a toll-free telephone number (1-866-ASK-NWQL) for customers needing personal or immediate assistance.

2.5.2 Complaints

The Business Development Team uses the LabHelp electronic mailbox to track and respond to questions, requests, and complaints from customers. The LabHelp mailbox also receives a copy of correspondence from NWQL laboratory scientists that was not originally sent to customer service for resolution. This enables tracking of the resolutions of all requests and complaints. Requests and complaints are assigned to general categories for tracking upon receipt. The categories and types of requests and complaints are reviewed for timeliness of response. This information is forwarded to the Management Team, and included as part of the annual quality system review.

2.5.3 Sample Login Customer Service

The NWQL provides the LabLogin electronic mailbox to send and receive inquiries regarding sample submittal, receipt, and login. This mailbox is set up to provide direct communication between field activities and the NWQL Login staff. The mailbox is similar to LabHelp and is monitored by several staff within the Support Services Section. The NWQL notifies customers when samples are received and logged into the data base at the laboratory. LabLogin sends an electronic mail message to each USGS Water Science Center contact, collector, and Water-Quality Specialist, notifying them of the arrival and condition of the sample(s) received each day.

2.5.4 Requests for Nonstandard Work

A proposal process, described in SOP AX0075.2, *Proposal and contract process at the NWQL,* is used for requests for nonstandard work. The SOP describes copying appropriate instrument analysis reports, priority sample processing or analyzing samples in a specific order, using custom methods, designing custom methods, or modifying analytical variables. The Business Development Team works with the customer to develop a proposal that is ready for review and approval by the Laboratory Chief and the customer.

If the NWQL is unable to perform the work requested by a customer for any legitimate reason, the work is declined. For example, the analytical method(s) or reporting levels requested are unattainable or final data cannot be delivered in the desired timeframe.

A custom proposal is not effective and work does not begin until it is signed by the customer and the Laboratory Chief or a designee. Electronic signatures are valid. The original proposal is kept in the Administrative Section.

The Project or Task Officer at the NWQL is responsible for informing the customer of deviations from the custom proposal. An analyst is responsible for informing the Project or Task Officer of problems or departures from the scope of work.

Proposals may be modified through written agreement of all parties. Modifications are effective once all parties have signed the modified proposal.

2.5.5 Quality Assurance Project Plans

Many USGS Water Science Center cooperators require a Quality Assurance Project Plan (QAPP) prior to implementation and agreement to perform work. A QAPP documents the planning, implementation, and assessment procedures for a particular project, as well as any specific QA and QC activities.

It integrates the technical and quality aspects of the project to provide a "blueprint" for obtaining the type and quality of environmental data and information needed for a specific decision or use. The U.S. Environmental Protection Agency offers an internet site with detailed information on how to prepare QAPPs at URL http://www.epa.gov/quality/qapps.html.

The USGS Water Science Center staff initiate QAPPs, but include the NWQL in discussions to address laboratory-related services. The NWQL requires the Laboratory Chief to sign off on all QAPPs requiring its analytical services. Laboratory information typically required in a QAPP includes the following:

- method references,
- detection and reporting levels,
- bias and variability criteria,
- holding times, and
- QA and QC requirements for the project.

USGS Water Science Center customers are asked to contact the Chief, QAS, for assistance and information required in the preparation of QAPPs. A proposal may be necessary to address nonstandard work to meet cooperator requirements.

2.5.6 Chain of Custody

The NWQL is a secure facility on the campus of the Denver Federal Center. Routine sample delivery by mail or overnight delivery service to the NWQL satisfies the sample integrity requirements of the majority of studies conducted by USGS Water Science Centers. Requirements for chain of custody (COC) are described in SOP QX0030.2, *NWQL chain of custody*.

Routine procedures include email notification to Water Science Center personnel at the end of each business day to acknowledge receipt and login of the samples. The email also addresses any problems with the structural integrity of the sample containers and the shipping container. More information regarding this notification system and an example of the electronic report may be found in NWQL Rapi-Note 02-017, *New sample receipt notification system*, accessible on the NWQL USGS-visible intranet web site at URL http://wwwnwql.cr.usgs.gov/USGS/rapi-note.html.

The NWQL offers formal COC for studies requiring it. COC processing is recommended for projects that may possibly be associated with future litigation and for projects that are expected to have a high level of scientific scrutiny. See SOP QX0030.2, *NWQL chain of custody* for requirements. Under the rules of COC, all COC samples are kept in someone's custody (physical possession or view) at all times or kept in a designated locked, limited-access room or locked refrigerator until needed. All transfers of custody are documented and the process by which each analyst removes an aliquot from a bottle is also documented.

SOP QX0030.2, *NWQL chain of custody*, addresses requirements on the part of customers and laboratory staff to conduct this type of sample handling. All COC procedures and analyses performed by the NWQL are prearranged through a proposal procedure as described in SOP AX0075.1, *Proposal process*, involving an NWQL Project Coordinator and USGS Water Science Center personnel.

2.6 Control of Nonconforming Environmental Testing

Nonconforming work occurs whenever the results or any other aspect of a test do not conform to procedures established for that work or requirements established with the customer. Occasionally, it is necessary to allow exceptional departures from established procedure. This is permissible with approval of the unit supervisor and documentation of the sample(s) affected by the departure in the LIMS.

NWQL personnel are responsible for documenting a nonconformance. A nonconformance is reported to the supervisor in charge of the work, who reviews it and documents the corrective action to be taken. The evaluation and corrective action consider the imme-

diacy of the actions to be taken and the acceptability of the nonconforming work. An analyst, supervisor, or representative of QAS may stop work at any time until the noncomformance is resolved.

When an evaluation of a nonconformance indicates the possibility of a recurrence, the Chief, QAS, is notified and the corrective actions are documented.

2.7 Corrective Action

Corrective action is required when a nonconformance or other departure from NWQL policies or procedures is identified. QC samples are used for ongoing corrective action of analytical processes. Conditions adverse to quality are identified by a number of other processes, including the following:

1. results of performance evaluation studies,
2. internal audits,
3. internal and external QA programs,
4. customer complaints, and
5. the annual quality system review.

The goal of corrective action is not only to eliminate such events, but also to reduce repetition of the causes. The identification and resolution of any nonconformance or departure from NWQL policies and procedures include the following:

1. analysts and supervisors using the available features in LIMS to review and chart QC data to monitor process performance;
2. realizing that QC data outside the acceptance limits or exhibiting a trend are evidence of unacceptable error in the analytical process;
3. initiating prompt corrective action to determine and eliminate the source of the error;
4. not reporting data until the cause of the problem is identified and corrected or qualified; and
5. maintaining records of all out-of-specification events, the causes determined, and the corrective action(s) taken.

If a condition adverse to quality is identified, a proposed corrective action is prepared and submitted for approval by the Chief, QAS. The proposed corrective action requires an evaluation of the need for action(s) to prevent any recurrence of the problem. The corrective action is based upon a determination of the root cause, assigns responsibility for the action, and includes completion dates.

The QAS verifies that corrective actions were completed. Corrective actions are tracked to determine their effectiveness. Subsequent audit(s) may be performed to confirm that the corrective actions have been implemented and are effective.

2.7.1 Technical Corrective Action

Corrective action begins with the analyst, who is responsible for knowing when the analytical process is meeting acceptable performance requirements. The analyst initiates corrective actions when a QC check exceeds the acceptance limits or exhibits trending. If the problem is not corrected by the actions, the analyst should report the out-of-specification event to the supervisor. Such events include the following:

1. QC outliers,
2. holding-time failures,
3. loss of sample,
4. equipment malfunctions, and
5. evidence of sample contamination.

In addition, assessments of method performance from various internally and externally administered QA programs are used to evaluate technical performance. When unsatisfactory performance is evident from these evaluations, corrective actions are required.

Internal QA functions used for these technical assessments include the long-term method detection level (LT–MDL) sample submission program, the blind blank project, and field supply quality-assurance samples. Analytical results from the blind blank project are evaluated on a continual basis and results are provided to the analytical units several times a month to help in their assessment of ongoing operations. Control charts and statistical summaries of these QA programs are used to develop the control criteria for analytical operations. Annual meetings with QAS and analysts establish updated control criteria.

The Branch of Quality Systems (BQS) administers two external QA functions — the Inorganic Blind Sample Project and the Organic Blind Sample Project. Control charts and detailed summary-statistic information are routinely prepared by BQS staff. Concerns arising from these QA programs are discussed at meetings coordinated by the BQS project chiefs. Analytical Services Section, QAS, and Method Research and Development Program staff attend these meetings to resolve technical issues.

BQS prepares QA summary reports that describe analytical methods that have indicated a bias or higher than expected variability. These reports list comments from the NWQL that may include corrective actions to be addressed. The QA summary reports are available on the BQS internet site at URL http://bqs.usgs.gov/bsp/mainpage.html.

2.7.1.1 Performance Evaluation

The NWQL participates in the following external performance evaluation (PE) studies:

1. USGS Branch of Quality Systems standard reference sample (SRS) inter-laboratory comparison for inorganic analytes,

2. New York State Department of Health potable and nonpotable waters for inorganic and organic constituents,

3. Environment Canada for low-level determinations of inorganic and selected organic constituents,

4. National Research Council (Canada) for trace metals in tissues and sediments, and

5. National Institute of Standards and Technology (NIST) for organic contaminants in marine tissues and sediments.

Additional PE studies include intermittent participation in Discharge Monitoring Report – Quality Assurance; Chesapeake Bay Blind Audit for particulate nutrients, chlorophyll, carbon; and other performance evaluation studies upon agreement with customers.

External PE results for the NWQL are available at URL http://nwql.usgs.gov/Public/perf_hdr.html.

PE samples are logged in when feasible and analyzed as regular environmental samples. Samples are not analyzed multiple times unless that is a normal procedure for that method. For NELAP certification samples, analysts and the Laboratory Chief are required to sign a statement attesting that the laboratory followed the proficiency testing provider's instructions for preparing and analyzing the test sample as they would an environmental sample.

All analytes that receive an unsatisfactory rating or are flagged for the laboratory to investigate must be investigated and corrective actions must be implemented based on the results of the investigation.

Any environmental data produced during the time that the analytical work was deemed unsatisfactory also must be reviewed to determine if the sample(s) must be reanalyzed or the data otherwise qualified.

Analytical units investigate the problem, identify the probable cause(s), and determine corrective actions required. A written report is submitted to the Chief, QAS, that describes the investigation and the corrective actions that have been implemented. The results are presented to the Laboratory Chief. The reports are kept in the NELAC files in the QAS office with the study results and may (if applicable) be posted to the performance evaluation web site. Investigation and corrective actions must be completed within 2 weeks of notification from QAS.

Refer to NWQL SOP QX0077.1, *Performance evaluation (proficiency testing) studies and accreditation at the NWQL*, Attachments 11 and 12, for specific guidance on failed PE studies and criteria for investigating and preparing written responses.

2.7.1.2 Internal Audits

The QAS is responsible for performing internal audits of all laboratory processes to verify that current activities follow published methods, approved SOPs, and the QMS (see section 2.8.1). Refer to NWQL SOP QX0084.1, *Conducting internal audits of current laboratory activities at the NWQL*, for further information. A major function of these audits is to ensure that policies and objectives of the QMS are documented in unit SOPs, have been communicated to unit personnel and understood by them, and have been fully integrated into the unit workflow.

Audit reports are provided to the Chief, QAS, the Laboratory Chief, the Section Chief, the Unit Supervisor, unit personnel, and the audit team within 2 weeks of the audit. Unit Supervisors respond to audit reports within 30 days and identify corrective actions on how audit deficiencies will be addressed. Audit report responses are documented and tracked by the lead auditor.

2.7.2 Preventive Action

Communication. The most effective preventive action is accurate, timely communication that travels vertically and horizontally throughout the organization. The NWQL strives to communicate on technical, administrative, and human relations levels through a variety of automated systems, broadcast electronic mail messages to management and the entire workforce, and through quarterly Town Hall meetings with all Federal and non-Federal employees. Customer's comments and suggestions also contribute to the information the laboratory needs to avoid problems and rectify those that are recurring.

One of the single most important items in preventive actions is providing timely, pertinent information to the entire workforce and soliciting its participation and ownership in all aspects of laboratory operations. The NWQL conducts quarterly Town Hall meetings open to all its Federal and non-Federal employees. The primary purpose of these meetings is to convey and receive information between management and staff. Town Hall meetings routinely open with items of health and safety followed by items associated with quality. All personnel in attendance are encouraged to participate in an open environment of discussion. In addition, the Laboratory Chief maintains an “open door policy” for personnel who do not feel comfortable discussing a topic in a more public forum.

To underscore the importance of communication, it is a stated expectation that all NWQL managers and supervisors actively, accurately, and in a timely manner communicate important aspects of laboratory operations to all staff. This expectation is clearly defined in the critical performance elements included in the performance plans of laboratory managers.

The laboratory actively participates in USGS-sponsored field training courses, providing both instructors and laboratory space.

Administrative actions. Administrative preventive actions are handled through dissemination of USGS guidance and regulations, internal NWQL policies, and through appropriate reviews. Issues that have been discovered, lessons learned, and items anticipated that could have adverse effects are routinely discussed at the weekly Management Team meeting. The meeting is an open forum for discussion and dissemination of current and anticipated technical items that could affect product quality.

Customer interactions. Customer interaction is encouraged prior to the submission of any samples to the laboratory. Issues of sample matrix concerns, one-of-a-kind samples, or other unique concerns can be addressed with subsequent followthrough upon receipt. The Business Development Team within the laboratory operates a “LabHelp” web site that deals with customer issues. As the primary conduit for customer interaction, it is in a position to identify and communicate any trends that might appear from a customer perspective. Recurring issues can be identified. thereby leading to a determination of root cause.

Laboratory information management system (LIMS). The LIMS monitors samples through receipt and login, work assignment, results entry by a manual process or a data-capture utility, QC testing, and release of results to customers. Use of the LIMS at the NWQL is documented in a user manual (White and Macke, 2003). Researchers can access a history of results and verify QC information through a web-based reporting procedure. Data are delivered electronically to customers through secure procedures. Some features of LIMS that contribute to preventive actions include the following:

1. maintenance of sample inventory;

2. sample history and tracking;

3. template-based system to prepare analyses that displays available samples, necessary QA and QC samples, and continuing calibration verification samples;

4. templates reflect how test is to be performed;

5. quality-control charts;

6. analysts' access to LIMS restricted by 'need to know' or use;

7. use of data-capture utilities that minimize acceptance of improperly formatted or inaccurate data components;

8. automatic data logic checks;

9. displaying information and data to analysts and supervisors in real time;

10. certain error corrections;

11. access to corporate data base restricted to LIMS and selected IT personnel;

12. reports and flags data out of specification;

13. billing information; and

14. weekly reports of sample login, costs, and monthly billing files.

2.8 Reviews

2.8.1 Internal Audits

The QAS conducts internal audits to verify that laboratory operations comply with the QMS. These audits are not limited to analytical lines within the Analytical Services Section, but include Support Services, Quality Assurance, Information Technology, and to a certain extent, Administrative Services. Each year, internal audits cover all technical aspects of laboratory activities, with an audit of each operation, including analytical methods, login and other support services, QA functions, and IT processes. The audits are conducted throughout the year, typically once every 3 to 4 weeks. This process allows for scheduling, researching and preparation, interviewing staff, and preparing the audit report.

Internal audits are conducted under the guidance and direction of the Chief, QAS. Trained and qualified personnel who are independent of the audited activity implement these audits. The audit team interviews laboratory personnel, observes procedures, and examines records and documentation.

The audits verify that current laboratory activities follow published methods, approved SOPs, and the QMS. A major function of these audits is to ensure that policies and objectives of the QMS are documented in unit SOPs, have been communicated to and are understood by unit personnel, and have been fully integrated into the unit workflow. The audit program also includes monitoring data integrity on a quarterly basis, examining a sample request from receipt of the sample at the NWQL through data release and retrieval by the customer.

Nonconformances to documented procedures are identified in the audit reports as deficiencies requiring corrective action.

Audit reports are provided to the Chief, QAS, the Laboratory Chief, the Section Chief, the unit supervi-

sor, unit personnel, and the audit team within 2 weeks of the audit. Unit supervisors respond in writing to audit reports within 30 days and identify corrective actions to be addressed. Audit report responses are documented and tracked by the lead auditor. The internal audit process, including resolution of problems discovered in the audit, is described in detail in NWQL SOP QX0084.1, *Conducting internal audits of current laboratory activities at the NWQL.*

2.8.2 Quality Management System Reviews

As required by NELAC Standard, rev. 16, section 5.4.14 (National Environmental Laboratory Accreditation Conference, 2001), and NWQL SOP QX0398.0, *NWQL quality systems reviews*, the quality system is reviewed annually by the MT and supervisory personnel, and led by the Chief, QAS. The review is a documented examination using specified criteria to provide objective feedback about the laboratory's quality system and considers, but is not limited to, the following actions:

1. suitability of policies and procedures;
2. suggestions from managers and supervisors;
3. outcomes of recent internal audits;
4. corrective and preventive actions;
5. assessments by external bodies;
6. results of interlaboratory comparisons and performance evaluations;
7. changes in the volume and type of the work;
8. customer feedback;
9. complaints;
10. previous management reviews; and
11. other relevant factors, such as quality-control activities, resources, and staff training.

The Chief, QAS, notifies management and supervisory personnel before a review. During the review the information is discussed and critical issues are given priority. The outcome of the review is a list of critical issues for management to resolve during the next year. The Chief, QAS, is responsible for distributing the list of issues to the MT for resolution.

Prior to the next annual review, the Chief, QAS, summarizes the status of the issues identified in the previous review, the actions taken, any problems encountered, and the outcome.

2.9 Software

The NWQL Information Technology (IT) Section strives for uniformity in its application of good automated laboratory practices throughout the laboratory.

Commercial products. Commercial software, such as electronic mail, statistical, and basic office applications such as word-processing, and spreadsheet programs, are used wherever possible throughout the laboratory. Installation and maintenance is provided by IT support services.

Software, including routines and macros that perform basic calculations, may be validated by hand calculations or other methods that produce known results. Large complex programs are verified and validated over their full range of functions. The documentation associated with the software includes an explanation of its functional requirements, algorithms and formulas used, and any testing performed.

Many analytical instruments now include data processing systems and proprietary software. The software that controls laboratory equipment must be installed according to the manufacturer's instructions and verified to be operating correctly.

Laboratory information management system (LIMS). The IT Section chose to customize StarLIMS software from LIMS USA to serve as its LIMS. StarLIMS software has been certified for ISO 9001:1994 *Quality management system specification* and Institute of Electrical and Electronics Engineers Standards.

StarLIMS uses a multiple-tier, data driven architecture, in which the StarLIMS application, the NWQL business rules, and the Oracle relational data base are each maintained in separate tiers. Each tier is separately validated using documented methods to ensure that the design, operation, performance, and installation meet NWQL requirements.

The LIMS tracks samples through login, work assignment, results entry by a manual process or by a data-capture utility, QC test, and release of results to customers. Use of LIMS at the NWQL is documented in a user manual (White and Macke, 2003). Additions and changes or corrections to data values are restricted and recorded in audit trails. Researchers can access a history of results and verify quality-control information through web-based reporting procedures. Data also are delivered electronically to customers through secure procedures verified with checksums.

The NWQL must adhere to policies and procedures defined by the U.S. Department of the Interior and the USGS regarding data security, software development processes, and quality of information. These procedures and policies are located on the USGS IT Security web page (accessible at URL http://internal.usgs.gov/gio/security/index.html) and the System Development Life Cycle web page (accessible at URL http://internal.usgs.gov/gio/irm/sdlc.html).

In addition to the data base, the IT Section has designated space available on a network appliance to store LIMS unprocessed data, which is backed up nightly to a remote site as well as to magnetic media, which are stored in safes according to procedures both on site and off site These procedures are an integral part of the Continuity of Operations Plan (accessible on the NWQL intranet web site at URL http://www nwql.cr.usgs.gov/safety/safety_documents.html).

3.0 TECHNICAL REQUIREMENTS

3.1 Personnel

There are three components associated with the recruitment of positions: position classification, qualification, and performance requirements for Federal employees.

3.1.1 Federal Employees

3.1.1.1 Classification Standards

Establishes the position series, title, and grade according to the level of difficulty and supervision required. Position descriptions are written by the technical expert (for example, the supervisor) in the established classification format for review and classification approval by the servicing Personnel Office. Classification standards are established by the Office of Personnel Management and can be accessed at URL http://www.opm.gov/fedclass/html/gsseries.asp.

3.1.1.2 Qualification Standards

The servicing Personnel Office determines a person's qualifications for a series and grade level based upon education and/or experience identified in the specific qualification standard. Documentation required by the Personnel Office includes a resume and official college transcripts. Qualifications are established by the Office of Personnel Management and can be found at URL http://www.opm.gov/qualifications/sec-iii/a/num-ndx.htm.

Qualified personnel perform tasks associated with the production of data at the NWQL. Minimum qualifications and training requirements are established for each position associated with data production.

3.1.1.3 Performance Plan and Rating Requirement

Management creates Employee Performance Appraisal Plans (EPAP) for all positions that manage, perform, or verify work associated with the laboratory. Annual performance plans for Federal employees, with one interim rating, are prepared and rated by the technical expert (for example, supervisor). The EPAPs contain at most five critical elements, with evaluation based on the following performance standards: exceptional, superior, fully successful, minimally successful, and unsatisfactory.

The Department of the Interior established performance plan and rating guidelines. The guidelines may be found at URL http://www.doi.gov/hrm/guidance/370dm430hndbk.pdf.

3.1.2 Contract Employees

The contract establishes the education and experience requirements for each task assignment. Task assignments are reviewed annually for each contract employee assigned to a task, and an annual consolidation of the year's performance is written by the contracting officer's representative and provided to the contracting officer . The contract clearly indicates training requirements for ethics, safety, and laboratory practices.

3.1.3 Training

3.1.3.1 Technical Training and Initial Demonstration of Capability

Data produced by the NWQL meet established data-quality objectives. This happens when analyses are performed using good laboratory practices and following relevant SOPs, and the analyst performing the procedure has been properly trained and has demonstrated proficiency with the analysis. The NWQL provides SOP training that is documented on a training checklist for each method SOP that the analyst is assigned to perform. This checklist is followed for each trainee analyst by the supervisor or the lead chemist assigned to train the individual.

A standard training checklist is accessible on the NWQL intranet forms page at URL http://wwwnwql.cr.usgs.gov/pub/WORDDOCS/NWQL-Trng.doc. Analysts are issued a set of training materials (for example,

method reference, SOPs, QMS document) and receive training by the supervisor or another experienced analyst. Progress is monitored with frequent performance reviews, QC check samples, performance audits, and bench sheet reviews.

A training file for each analyst is kept in the Records Management Office. These training files include information indicating the analyst's position classification, an initial demonstration of capability (IDC), and continuing demonstration of proficiency for each analyst. Position classification is based on the Federal Office of Personnel Management standards, and each classification has a specific set of minimum qualifications, as described above in section 3.1.

The IDC is performed for each analyst and instrument. An analyst's IDC includes a demonstration of the ability to achieve a low background level for the analysis, the bias and variability required by the method, and satisfactory performance on an unknown sample as ongoing proficiency test results. The IDC is repeated when there is a change in analyst, test method, or instrument. Standard IDC certification and data compilation spreadsheet forms are available on the NWQL intranet forms page at URL http://wwwnwql.cr.usgs.gov/pub/WORDDOCS/NWQL-IniCert.doc and http://wwwnwql.cr.usgs.gov/pub/WORDDOCS/NWQL-Cpbilty.xls.

If spikes are not applicable (for example, pH or total suspended solids), QC samples can be used for the IDC. The laboratory retains all associated supporting data necessary to reproduce analytical results summarized in the IDC certification statement. Four aliquots of the sample are analyzed concurrently or over a period of days. Mean recovery and standard deviation for each variable of interest are calculated in the units used for reporting to customers. The resulting mean recovery and standard deviation must meet the acceptance criteria for the method. If there are no mandatory criteria in the method, either reference or laboratory generated limits are used.

Analysts do not independently process environmental samples until an IDC for all variables of interest meet acceptance criteria. If one or more of the test variables do not meet the acceptance criteria, the problem is corrected followed by repeated analysis of the four aliquots or at least for those that failed to meet criteria.

Marginal exceedance, described in section B.1.1.2 of Appendix B, does not apply to IDCs.

3.1.3.2 Continuing Demonstration of Capability

The laboratory performs an annual demonstration of capability for each analyst to ensure that each technical Federal or non-Federal employee demonstrates ongoing proficiency for the tests performed by the technical employee. The NWQL significantly expanded its scope of accreditation request for National Environmental Laboratory Accreditation Conference (NELAC) nonpotable water certification in July 2003. Generally, methods that were included in the expanded scope of accreditation have been used at the NWQL for many years with analysts trained years ago. A continuing demonstration of capability has been used to document analyst performance for these methods. When new analysts are trained or as new equipment is brought on line, initial demonstration of capability documentation is prepared.

Ongoing proficiency is checked by ensuring that the training of personnel is kept up to date by the following:

1. Evidence on file that demonstrates that each employee has read, understood, and is using the latest version of the laboratory's in-house SOP and all other documentation that relates to his/her job responsibilities.

2. Training courses or workshops on specific equipment, analytical techniques, or laboratory procedures shall all be documented.

3. A certification that the technical personnel have read, understood, and agreed to perform the most recent version of the test method (the approved method or SOP), and documentation of continued proficiency by at least one of the following once per year:

a. acceptable performance of blind sample (single blind to the analyst);

b. another initial demonstration of method performance;

c. successful analysis of a blind performance sample on a similar test method using the same technology (documentation required for only one of the test methods);

d. at least four laboratory control samples analyzed either concurrently or over a period of days, with acceptable levels of bias and variability.

4. Continuing demonstrations of capability (such as laboratory control and matrix spike samples) are performed.

5. Initial demonstration of capability is completed each time there is a significant change in instrument type, personnel, or test method.

6. All continuing demonstrations of capability are documented through the use of certification statement found on the NWQL intranet forms page at URL http://wwwnwql.cr.usgs.gov/pub/WORDDOCS/NWQL-ContCert.doc.

7. Marginal exceedance, described in section B.1.1.2 of Appendix B, may be applied to continuing demonstration of capabilities (section D.1.1.2.1e in National Environmental Laboratory Accreditation Conference, 2003a). This allows a certain number of analyte data points in a laboratory control sample to be between three and four standard deviations from the mean without initiating a corrective action. A marginal exceedance may be used only for analytical systems with a specified list of 11 or more analytes.

3.1.3.3 Ethics

All Federal and non-Federal personnel are required to attend New Employee Ethics training within the first 6 months of employment at the NWQL. Additionally, annual data integrity refresher training is mandatory for all analytical personnel. Training records are maintained to ensure that each employee has attended the required training and understands his/her ethical responsibilities and reporting mechanisms. The Ethics Coordinator works with each employee to complete his/her *NWQL Training Record* form. This is filed in the employee's training file, which is located in the laboratory records technician's office, room 1140.

Training for new employees addresses three areas: personal ethics, corporate (government) ethics for Federal employees, and professional ethics that includes data integrity. Personal ethics training highlights the USGS *Guiding Principles* of respect, communication, collaboration, accountability, and encouragement.

Corporate (government) training includes guidelines on accepting gifts from outside sources; acceptance of travel and related expenses; gifts between employees; outside employment; political activity; misuse of official position, time, and equipment; negotiating for non-Federal employment; and post-employment issues.

Professional ethics training includes, but is not limited to, data integrity topics such as laboratory fraud and inappropriate laboratory practices, for example, falsifying or fabricating data, misrepresentation of quality-control (QC) or calibration data, improper spiking procedures, improper instrument clock setting/recording, method deviation, and other breaches of ethical behavior. The training covers the need for honesty and full disclosure in analytical reporting. The training includes ethically challenging scenarios that give personnel the opportunity to gain insight through discussion. It instructs personnel on ethics expectations for business conduct and makes them aware of the effect of inappropriate practices. The training addresses ethical and legal responsibilities of Federal and non-Federal employees, including the potential punishments and penalties for improper, unethical, or illegal actions.

The Ethics Coordinator determines specific data integrity training annually. Appropriate training topics and courses are scheduled to meet the annual refresher training requirement.

3.1.3.4 Safety Training

All Federal and non-Federal employees receive annual safety training to meet Occupational Safety and Health Act, Colorado Department of Public Health and Environment (1998), and U.S. Geological Survey (2002a) requirements. Training includes instruction on the elements of the Occupant Emergency Plan and the Chemical Hygiene Plan, and hazard communication information. Some staff members receive specialized safety training, such as emergency response hazardous material spill containment and cleanup, first aid/cardiopulmonary resuscitation, automated external defibrillator response, and fume hood training. Federal employees who drive on official business must complete defensive-driver training every 3 years.

All new Federal and non-Federal employees receive orientation training in the NWQL Safety, Health, and Environmental Compliance Program, usually within the first week of employment.

3.1.4 Medical Surveillance

The NWQL provides occupational medical surveillance to Federal and non-Federal employees who are occupationally exposed to hazards (U.S. Geological Survey, 2002b). The NWQL has contracted with the Public Health Service, Division of Federal Occupational Health, to provide this service. The surveillance program includes preplacement or baseline, periodic, and exit exams.

3.2 Accommodation and Environmental Conditions

The NWQL is the primary laboratory used by the USGS for the analysis of organic and inorganic constituents in samples of ground water, surface water, river and lake sediments, aquatic plant and animal tissues, and atmospheric precipitation collected in the United States and its protectorates. The NWQL shares Building 95 at the Federal Center in Denver, Colo., with the National Research Program, the Branch of Quality Systems, and the Office of Water Quality. The building has 10,876 square meters of laboratory and office space, and occupies an 8.3-hectare plot.

All Federal and non-Federal employees enter by magnetic access card. Visitors sign in and out of the building at the main entrance, where they are provided with “visitor” identification badges and safety glasses. Visitors are accompanied by a Federal employee at all times. Minors are not admitted without an adult escort.

3.2.1 Facility

The new NWQL at the Federal Center was ready for occupancy in March 1999. The facility represents the efforts of the architects and staff to ensure that safety, health, and environmental concerns were incorporated, incompatible laboratory processes were not located near one another, and that design features, such as the layout of shelves and cabinets, special air and power requirements, the need for walls between laboratories and workstations, location and types of fume hoods, and any other special needs, were addressed satisfactorily.

Important safety and design features of the NWQL facility include the following:

1. The two wings each passes about 2,000 cubic meters per minute (m^3/min) (70,000 cubic feet per minute, ft^3/min) of air through high efficiency particulate air (HEPA) filters to provide the building with clean, conditioned air. This air is not reused. It is exhausted after one pass. During the exhaust process, the air is passed through a heat exchanger to capture some of the heating or cooling effect, but the air is not reused. Clean air moves from office spaces through laboratory areas, up and out. This minimizes exposure to exhaust air.

2. The low-level nutrients laboratory is closed to minimize ammonia contamination.

3. The preparation area for organic samples has full-length hoods to enclose the liquid-liquid extraction process completely.

4. Individual snorkel ventilation is provided for many instruments to ensure that all exhaust is vented to the outside.

5. Access to the chain-of-custody area is controlled by key card. Access to the computer server room and radiochemical standards laboratory is controlled by cyberlocks. Access to entry may be tracked for safety and security purposes.

6. The sample/waste disposal area is near the loading docks for easy access. This area has individual alcoves for different types of waste. Each alcove has a dam at the bottom of the alcove door to ensure that spills can be contained. Each alcove has features for the chemicals contained within; explosion-proof lights and/or special air handling when needed.

7. The floor in the waste disposal area has been sealed to prevent spills from contaminating the floor or the ground under the floor.

8. Automatic doors from the loading dock allow personnel to move incoming samples and packages easily and safely to the areas where they are unpacked. Automatic doors also are placed in the major corridors to allow personnel to easily and safely move chemicals, samples, and equipment.

9. Closed laboratories have glass in the doors and often have extra windows for workers' safety and allow visitors to tour the laboratory without having to enter work areas.

10. The deionized water system continually circulates water through the building. This prevents any portion of water from becoming “stale” or developing biological activity to degrade its quality. Return water is treated again before being placed in the storage tank.

11. The central corridor was designed to move samples and chemicals into and from laboratory areas.

12. The central corridor in each laboratory wing has room for chillers and refrigerators to reduce noise and clutter within the analytical laboratories.

13. The central corridor has safety showers and spill kits.

14. Each wing has several small conference rooms to allow work units to meet close to where they work. This allows analysts to check instruments during meetings and minimize lost instrument time.

15. Servers are in a room with special power and temperature requirements, and an uninterrupted power supply to keep IT equipment operational.

16. The building has controlled access.

17. A special floor sink was installed in the sample login area so the login staff could easily dispose of the plastic and ice from the unpacked coolers. The ice can melt over night and the plastic bags can be disposed of in the morning.

18. A computer controls air flow in the fume hoods. As more fume hoods are used, variable speed motors increase air flow to ensure laboratory safety.

19. An emergency generator for critical systems is installed in the event of a power loss.

20. Automated external defibrillators (specialized devices designed to recognize and treat certain lethal heart rhythms during cardiac arrest) are in place, with 18 trained staff members available.

Isolating incompatible areas. The laboratory in which volatile organic compounds are analyzed is isolated from the preparation area and laboratories for organic analyses. Laboratory design and other considerations included the following:

1. The laboratory analyzing volatile organic compounds is in a separate laboratory wing (the South wing) from where organic samples are routinely processed (the North wing) to minimize contamination of the volatile analysis area.

2. Each wing has a separate air-handling system.

3. Because the prevailing wind is from the west, the air for both wings has intake vents on the west side of the building and outlets on the east side to minimize potential contamination from laboratory exhaust.

4. The North wing is set slightly east of the South wing, in part, to reduce the chances of North-wing exhaust from entering the South-wing intake.

5. Entry/exit alcoves prevent volatile compounds from entering while doors are open for access to the laboratory.

6. The temperature in the laboratory in which volatile organic compounds are analyzed is kept lower than other laboratory areas.

3.2.2 Air and Surface Monitoring

The NWQL contracts with the Public Health Service to provide industrial hygiene services. Air monitoring and quantitative exposure evaluations are performed to assess levels of airborne contaminants in accordance with Occupational Safety and Health Administration Permissible Exposure Limits, National Institute of Occupational Safety and Health Recommended Exposure Limits, or American Conference of Governmental Industrial Hygienists' recommendations.

Monitoring is performed at various times and locations for airborne contamination of lead, mercury, formaldehyde, methylene chloride, ethyl acetate, and more. Laboratory fume hoods and work benches are tested for contaminates to protect the employee and prevent sample contamination. Surveys are done for noise hazard levels and exposure.

3.2.3 Fume Hood Testing

Engineering controls are used to the greatest extent possible to either eliminate or reduce respiratory hazards to acceptable levels. To ensure that fume hoods are in proper working order, average face velocities of all fume hoods are determined at least annually. This measurement is documented on labels attached to the fume hood. Hoods found to be not working properly are removed from service until adjustment or repairs can be made. An added benefit of a properly working fume hood is the reduced likelihood of contamination of samples from airborne particles.

3.2.4 Maintenance, Repair, and Replacement

USGS is responsible for maintenance, repair, and replacement of USGS-owned and/or programmatic equipment, fume hoods, and corresponding equipment. Cleaning of fume hoods, exhaust fans, and exhaust ductwork is the responsibility of the USGS.

The General Services Administration (GSA) is responsible for operation and maintenance of the building operating equipment, as well as daily operation and maintenance of the building and appurtenances. GSA performs normal maintenance of the blower motors in the penthouse supporting the fume hoods. Any other work assumed by USGS personnel should be described in writing and agreed to by the GSA community Business Center Manager.

3.2.4.1 USGS Equipment Maintenance Responsibilities

The NWQL is responsible for maintenance of the

1. deionized water system;

2. acid-neutralization system and acid waste lines;

3. agency-operated and -owned equipment (including fume hoods);

4. USGS-installed fire extinguishers;

5. perchloric and radioisotope systems;

6. Kjeldahl system;

7. house nitrogen, argon gas, and compressed air systems (does not include the air compressors);

8. specialized filters within laboratories (carbon and HEPA);

9. plumbing within laboratories; and

10. walk-in cooler.

For maintenance or building concerns during a regular work week between the hours of 6:30 a.m. to 3:00 p.m., call Building 95 Facility personnel on cell/office phone; a list of those phone numbers is attached to every phone throughout Building 95. In addition, because individuals, contractors, and their telephone numbers may change, the NWQL maintains a current list of GSA contacts, GSA contractors, and USGS contacts on a white board in Room 1135 of Building 95.

Cell phone numbers are more appropriate for calls after work hours and on weekends, . All work requests require a Facility work order form available in room 1135 with proper signatures and priorities. Any operation or maintenance not covered on the above list must be requested through GSA Building Service and the Mega Center.

Any GSA Service requests outside regular working hours, holidays, and weekends must go to the Mega Center. During regular work hours and on a regular workweek, the calls must go to Building Services. These numbers are as follows:

Federal Center contact

Denver Federal Center Switch Room
303-236-7998

GSA contacts

Mega Center
303-236-6709

Building Services
1-888-999-4777

Ground Maintenance
(Bruce Johnson)
303-236-8000 x5556

GSA Telephone Support
(Greg Stock)
303-236-6023

GSA Building Management Specialist
(John Paxton)
303-236-8000 x2714

GSA Customer Service Specialist
(Doug Baughman)
Cell: 303-941-1306
303-236-8000 x2630

GSA contractors

Custodial Service
(Loretta Montez)
Cell: 303-944-0459
303-231-9040

Telephone Support
(Premises Support Group)
303-238-4636

USGS contacts

Central Region Space & Facilities
(Gregg Schuster)
303-236-9180

Building 95 Facilities

Andy Martinez
Cell: 303-435-8486
303-236-3701
and
Will Lanier
Cell: 303-472-8082
303-236-3710

3.2.4.2 NWQL Housekeeping Procedures

The NWQL Safety and Facilities Sections conduct combined orientations on safety and good housekeeping procedures for new arrivals of housekeeping and maintenance personnel to the NWQL. Safety and the minimal use of contaminating cleaners, polishers, deodorizers, solvents, and paints in the building are emphasized. How to handle spills of any kind, the disposal of used containers, liquid disposal, and the disposal locations are described.

Janitorial service for offices and common areas is provided by contractor through GSA. Requests for custodial service in the laboratories is handled by special request submitted by supervisors. Requests are submitted using form NWQL SSS.1, which is accessi-

ble on the NWQL intranet web site at URL http://www nwql.cr.usgs.gov/nwql/Forms/admin.html.

Because not all housekeepers speak English, a translator describes the procedures and regulations. Communication with GSA enables the NWQL to receive prior notification when spraying of pesticides, herbicides, snowmelt chemicals or any product that could be harmful to laboratory operations is planned. GSA notifies the NWQL 24 hours prior to performing any work in the facility.

Housekeeping and maintenance personnel are required to maintain copies of Material Safety Data Sheets (MSDS) of all cleaners and polishers used in the building, and in their work area. A copy of all MSDS used in the building is provided to the Safety Office. Any cleaners used by the janitorial staff or solutions used by maintenance personnel must be approved by the NWQL before use.

3.3 Environmental Test Methods and Method Validation

3.3.1 General

The USGS Water Resources Discipline requires that its methods for sampling and analysis be described, validated, documented, reviewed, approved, and published prior to routine, widespread use. This is necessary for the maintenance of long-term records of water resources and a well-documented data base of water-resource information, so that environmental trends can be observed.

3.3.2 Sources of Methods

USGS Office of Water Quality Technical Memorandum 98.05 provided the criteria for the acceptance and use of the two types of water-quality analytical methods — approved and unapproved (U.S. Geological Survey Office of Water Quality Technical Memorandum 98.05, 1998a). Approved methods include those classified as official and methods accepted and published by recognized agencies and organizations.

Approved methods.

Official. Methods that are approved by the USGS Office of Water Quality and published in the Techniques of Water-Resources Investigations series, Water-Resources Investigations Reports, Open-File Reports, or Fact Sheets, and Methods and Techniques.

Accepted. Methods require validation at the NWQL before they can be implemented routinely, but publication in one of the above USGS formats is unnecessary. Accepted methods also can be performed at the NWQL on a limited basis with a custom proposal. Such methods are already accepted and published by the U.S. Environmental Protection Agency, the American Society for Testing and Materials Annual Book of ASTM Standards, or in Standard Methods for the Examination of Water and Wastewater (American Public Health Association, 1998).

Only approved methods are used for routine production analysis at the NWQL. Unapproved methods may be used on a limited basis, but require submission of a custom proposal (NWQL SOP AX0075.2, *Proposal and contract process at the NWQL*). Data produced using new, unapproved methods or using research methods may not be published in USGS Publication Series data reports or in USGS Water Science Center annual data reports. In addition, the data may not be released to the public in publicly accessible data bases. Data provided to the public from unapproved methods or research methods must be accompanied by a method description that documents the method and the quality of the data reported.

3.3.3 Standard Operating Procedures (SOPs)

The Office of Water Quality requires that all methods used on a routine basis at the NWQL include an approved SOP. The available list of current SOPs can be viewed on the NWQL intranet web site at URL http://wwwnwql.cr.usgs.gov/nwql/sop/sops.html. SOPs receive an initial supervisory review, and are reviewed and approved by NWQL personnel (as determined by the NWQL Management Team), the Section or unit supervisor and the Chief, QAS.

All SOPs follow one of two standard formats described in NWQL SOP QUAX0001.3, *Writing and approving standard operating procedures (SOP) at the National Water Quality Laboratory*. SOPs describe how to prepare samples for analysis, prepare standards and reagents, set up and calibrate instrumentation, interpret data, and report data. Information concerning specific grades of materials used in reagent and standard preparation, appropriate glassware and containers for preparation and storage, and labeling and recordkeeping for stocks and dilutions are included. SOPs also describe essential laboratory operations and techniques that are not addressed elsewhere. These may include, but are not limited to, procedures for cleaning glassware, and operation of analytical instruments, balances, pipetting techniques, and type and use of volumetric glassware.

SOPs that affect an analyst's duties are provided when he/she is assigned the duty. Analysts are trained in accordance with these SOPs. SOPs are reviewed, revised, or updated as needed, at least every 3 years. Necessary changes to an SOP that occur between its periodic reviews are submitted as amendment reports.

3.3.4 Demonstration of Capability

Before new methods, either developed by the NWQL or from other sources, are implemented, the capability of NWQL personnel to perform them must be confirmed. Additional information is contained in sections 3.1.3.1 and 3.1.3.2. A demonstration of capability is also completed each time there is a change in instrumentation type, method, or personnel. (See SOP MX0015.2, *Guideline for method validation and publication at the National Water Quality Laboratory*.)

3.3.5 NWQL-Developed Methods

The Methods Research and Development Program (MRDP) at the NWQL is the primary developer of water-quality methods for chemical analysis within the USGS. The NWQL Analytical Services Section also may develop methods with MRDP consultation and/or participation. Methods developed by Analytical Services must meet the same criteria as above, including review and approval, before they can be implemented.

A laboratory providing data to the USGS may request approval of a new water-quality analytical method from the Office of Water Quality by providing documentation, including a final draft of the water-resources report and an SOP from the originating laboratory. (See SOP MX0015.2, *Guideline for method validation and publication at the National Water Quality Laboratory*.) The originating laboratory must also be approved by the Water Resources Discipline (U.S. Geological Survey, 1998b).

The Office of Water Quality reviews and approves new analytical methods, and requires submission of a final draft of the method report and the SOP.

3.3.6 Nonstandard Methods

Unapproved methods may be used on a limited basis but require submission of a proposal (NWQL SOP AX0075.2, *Proposal and contract process at the NWQL*). The term custom analysis includes approved and unapproved methods. An important distinction is whether or not the method is currently implemented at the NWQL although a proposal may still be required if the laboratory's current capacity for a particular analysis is exceeded by a customer's request. Proposals are required for all nonroutine analyses, including new method development, method modifications, and other projects requiring time and resources of NWQL staff beyond what is routinely available.

3.3.7 Validation of Methods

The experimental work required to validate a method for approval follows a standard format. Occasionally, the technical characteristics of a method require that the standard format be modified. The participation of all involved, including the MRDP, the Analytical Services and QA Sections, and the external sponsor (if present), in agreeing to a work plan, allocating resources, and reviewing progress is the preferred approach. Arriving at a consensus on the exact data requirements for validation and periodic status reviews before development begins enhances communication and promotes efficiency. If appropriate, periodic written (electronic mail) status reports to all participants are recommended.

To be approved as an official USGS method of analysis by the Office of Water Quality, data for the following characteristics must be evaluated and reported: bias and variability (the preferred terms used by the NWQL and the USGS Branch of Quality Systems), method detection limit, comparability to superseded methods, interferences, sample preservation requirements, stability of reagents and standards, instrument performance, and safety information. (See SOP MX0015.2, *Guideline for method validation and publication at the National Water Quality Laboratory*, for details.)

All new or revised methods, if the latter have a change in the chemistry, must be published in an approved USGS publication series report. The series of choice is Techniques and Methods. Official water-quality analytical methods have been published in the Techniques of Water-Resources Investigations series, Open-File Reports, or Water-Resources Investigations Reports. Method-development projects require extensive interpretation of experimental results. If new analytes are added to an existing approved NWQL method, an Open-File Report or Fact Sheet is sufficient to document the changes.

At the time the method report is prepared, an SOP also must be written. The content of an SOP differs from the content of the method report. The SOP contains summary, scope and application, references, safety issues, comments, holding times, instrumentation, reagents and standards, procedure, calculations, QA/QC requirements, reporting, and archiving sections. The SOP should be reviewed and updated periodically, typically once every 3 years. Changes may be made to an SOP between its review cycles by amendment report. For details, see the most recent version of the SOP on SOPs [NWQL SOP QUAX0001.3, *Writing and approving standard operating procedures (SOP) at the National Water Quality Laboratory*].

The Office of Water Quality must approve the USGS method and requires an approved SOP before implementation of the method. The method report also must be approved by the USGS Director's office before publication.

3.3.8 Estimation of Uncertainty of Measurement

To be approved as an official USGS method of analysis by the Office of Water Quality, the bias and variability of data (the preferred terms used by the NWQL and the Branch of Quality Systems) must be evaluated and reported. To determine constituents in water, bias and variability must be measured in three different sources (matrices): (1) laboratory reagent water, (2) ground water, and (3) surface water. In some instances, specific water-quality variables, such as specific conductance or dissolved organic carbon, may be known to have substantial effects on the performance of analytical methods. Specific consideration should be given to selecting water sources that span expected ranges of those key variables.

Bottom material, water-suspended sediment, and biological tissues are analyzed similarly, that is, measurements made in a reagent blank matrix; and different matrices need to be representative of the expected environmental samples.

Each sample must be spiked with each constituent determined in the method. Two concentrations must be used at a minimum. A "low" spike must be within 5 to 10 times the estimated detection limit. The "high" spike must be near the upper one-third of the applicable concentration range. At least seven replicate spikes at each concentration level must be analyzed (10 or more spikes are preferred) for a total of 42 to 60 or more determinations. For methods that require minimal sample preparation, spikes must be analyzed over at least 3 days, with no more than three replicates analyzed on any single day. It is preferred that these data be produced over a longer time than 3 days and that no more than one replicate be determined per day.

A summary of determined concentrations must be reported, and the bias and variability must be computed and reported for each concentration level using accepted statistical figures of merit, such as standard deviation, percent recovery, and percent relative standard deviation. Alternatively, the median, F-pseudosigma (F_σ), percent recovery, and percent relative F-pseudosigma may be preferred. If a method

is performed on a custom basis prior to approval, it is suggested that set spike, blank, and surrogate information for these custom analyses be included in the method approval documentation.

3.4 Equipment

3.4.1 Analytical Support Equipment

Analytical support equipment includes balances, thermometers, refrigerators and freezers, ovens, glassware, and fume hoods. If quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume, then the equipment must be maintained and its performance documented. Support equipment is maintained in proper working order, and records of all activities, including service calls, are kept.

The equipment is calibrated or verified at least annually, using National Institute of Standards and Technology (NIST) traceable references when available, for the entire analytical range. The results of calibration must be within the specifications required of the application for which the equipment is used. Failure to meet these specifications requires that the equipment be removed from service and clearly labeled as "Equipment Out of Service" until repaired. (See SOP OX0360.0, *Maintenance of equipment at the National Water Quality Laboratory.*) When returned to service, such equipment must be calibrated, verified, and the calibration documented.

The acceptability for use or continued use of such equipment is determined by needs of the analysis or application for which the equipment is being used. Temperature-measuring and mechanical volumetric-dispensing devices (except Class A glassware) are uniquely identified and checked for bias and documented in a log book.

Analytical balances. Analytical balances are used for accurate weighing of samples, reagents, and calibration standards. Prior to use on each working day, or on an as-used basis, balances are checked with NIST-traceable references in the expected-use range. The balances are cleaned and certified twice a year by a contract service technician. (See SOP QX0029.3, *Calibrating, operating, and maintaining balances.*)

The calibration of each balance is checked with Class I weights certified by NIST or a NIST-certified entity. Balances are verified as often as once per use for high-use balances or as infrequently as once per quarter for low-use ones. The calibration, maintenance, and use are documented in a log book. Balances that fail calibration checks are clearly labeled "Equipment Out of Service" and dated. An out-of-calibration balance is not used until it is repaired and re-certified by the contract-service technician.

Thermometers. Thermometers are verified annually against thermometers calibrated to NIST-traceable standards, following the procedure described in SOP QX0376.0, *Calibrating, maintaining, and adjusting thermometers*. Thermometers are replaced if they are out of calibration.

Refrigerators and freezers. An automated recording device continuously monitors the temperatures of most refrigerators and freezers. Temperatures of refrigerators and freezers not connected to the electronic monitoring system are monitored manually at a minimum of once each workday. Each section maintains the computer records for its refrigerator or freezer. Procedures are outlined in SOP QX0375.0, *Calibrating, recording, and maintaining the temperature monitoring program*.

Refrigerator temperatures are maintained nominally at 4°C with a tolerance range from 0.5°C to 6.5°C. Temperatures of freezers used for biological tissue and sediment samples are 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, or the equipment is repaired.

Ovens. Ovens are checked for temperature accuracy by use of a NIST-traceable thermometer. General-purpose thermometers or continuous automated recording devices are used for oven-temperature monitoring. A temperature log is maintained for each oven during use. (See SOP OX0375.0, *Calibrating, recording, and maintaining the temperature monitoring program.*)

Glassware. Class A volumetric glassware is used for analytical work where appropriate. Glassware is cleaned to meet the minimum contamination requirements of the method. Any cleaning and storage procedures that are not specified by a method are documented in laboratory records and SOPs.

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 are calibrated quarterly with analytical balances or with an automatic calibrating spectrophotometer. (See SOP QX0356.1, *Calibration of mechanical volumetric devices for the NWQL.*)

Fume hoods. Laboratory fume hoods are inspected and certified annually, and repaired as needed, by qualified personnel [as defined by the NWQL Safety Health and Environmental (SHE) Compliance Section]. Inspection reports are filed in the SHE office.

3.4.2 Calibration

3.4.2.1 Initial Calibration

The initial calibration of an instrument prior to use is documented. The documentation includes calculations, integrations, acceptance criteria, associated statistics, and other pertinent information sufficient to reconstruct the calibration. In all cases, the initial calibration is verified (as indicated in the published analytical method or approved method SOP) using an independently prepared (third-party) calibration verification solution other than the one supplied by the manufacturer (unless, as in some organic analyses, this step is not possible).

3.4.2.2 Continuing Instrument Calibration

All instruments used for environmental testing are uniquely identified. Continuing calibration procedures for a specific laboratory analytical instrument at minimum consist of a calibration of at least three standards or two calibration verifications throughout each analysis as described in the *Organic QA/QC guidance manual* (M.P. Schroeder, U.S. Geological Survey, written commun., 1999). An exception to this standard is instrument technology that uses only a zero and a single-point standard, such as inductively coupled plasma–atomic emission spectrometry. The SOP for each analysis performed describes the calibration procedures, frequency, acceptance criteria, and the conditions that require re-calibration.

All test methods or method SOPs provide specific details of instrument calibration and incorporate, where appropriate, the features identified below:

1. reagent blank used to establish calibration baseline;

2. use of the same preservation matrix for standards and samples;

3. adequate number of standards used to define calibration;

4. use of the low standard is addressed as appropriate for the method;

5. application of appropriate curve fit;

6. linearity of curve is established if relevant;

7. use of control sample, third-party check, where available, to verify calibration accuracy;

8. use of verification standard, continuing calibration verification or reference sample, to verify system stability; and

9. describe acceptance for calibration and process for dropping calibration points.

Sufficient unprocessed data records must be retained to permit reconstruction of the initial instrument calibration. Analytical methods and SOPs, where applicable, also specify whether the calibration is recovery corrected; that is, standards and samples undergo similar sample processing.

Log books. A sample log book is maintained for each instrument and analytical system, which can contain, but is not limited to, the following information: instrument identification, serial number, date of calibration, operator, calibration solutions with traceability, and analysis file information. The log books are kept with the instrument until, at a minimum, 1 year after completion, when they are transferred to the laboratory records technician for archiving.

Control charts. QC data are entered into control charts upon completion of analyses. These data are examined by supervisors or designated staff as data are produced. Suitability of data submission is based on historical or pre-determined acceptance criteria. QC data are available for control-charting functions when data are approved in LIMS.

Verification of calibration. All verifications of calibration are dated and labeled with, or traceable to, the analytical method, analyst, instrument, analysis date, analyte name, concentration, and response (or response factor). When instrument software allows for this capability, sufficient information is recorded and archived to permit reconstruction of the calibration. Acceptance criteria for calibrations comply with method requirements and are documented in the method reports and SOPs. Whenever practicable, equipment requiring calibration is to be labeled to show its calibration status, including the last and the next calibration dates.

Failure of initial calibration. All results are calculated on the basis of the response curve from the initial calibration or from subsequently adjusted analyses (see section 3.4.2.1). If the initial calibration fails, the analytical procedure is stopped and evaluated. For example, a second standard might be analyzed and evaluated or a new initial calibration curve might be established and verified. In all cases, the initial calibration must be acceptable (as indicated in the published analytical method or approved SOP) before reporting data for any affected samples.

Calibration verification checks. When an instrument is not calibrated on the day of analysis, a calibration verification check standard is analyzed at the beginning and at the end of each batch of about 10 samples. The concentration of this calibration check could vary on a regular basis and is described in the method SOP.

If a calibration check standard fails, and routine corrective action procedures fail to produce a second consecutive calibration check within acceptance criteria, then a new calibration curve is prepared.

Data calibration. Unqualified data may not be reported if they are greater than the highest calibration standard used (extrapolated). The sample must be diluted or concentrated, if possible, so the concentration falls within the calibration range. The observed concentration then is multiplied by the dilution factor.

The only exceptions to this rule are those constituents that cannot be reanalyzed; these data are qualified in the appropriate manner. In the case of concentrations that are reported less than the lowest calibration standard, the data also must be appropriately qualified (these two situations are qualified with an estimated or "E" code).

3.4.3 Preventive Maintenance

Log books are maintained for each major piece of equipment, with all reference materials applicable to the tests performed. These records include documentation on all routine and nonroutine maintenance activities and reference material verifications and may be specific to each analytical schedule or lab code. The records may include the following items:

1. name of the equipment;

2. manufacturer's name, type identification, and serial number or other unique identification;

3. date received and placed into service;

4. location;

5. condition when received (for example, new, used, reconditioned);

6. location of a copy of the manufacturer's instructions in the laboratory;

7. details of maintenance performed and all plans for preventive maintenance, including dates;

8. a history of any moves, damage, malfunction, modification, or repair of instruments;

9. identification of supporting software; or

10. traceability standards referenced to NIST standards.

A routine preventive maintenance program is used to minimize instrument failure and other system malfunctions. Designated Federal and non-Federal employees, and service contractors regularly perform routine scheduled maintenance and repair. Maintenance is documented in the instrument records.

Failure to meet specified acceptance criteria requires that the equipment be removed from service and clearly labeled as "Equipment Out of Service" until repaired. (See SOP OX0360.0, *Maintenance of equipment at the National Water Quality Laboratory*.)

If an analytical system fails because of mechanical or chemical problems and cannot produce environmental sample data for 10 working days, the unit supervisor must notify the Chiefs of the Laboratory, Analytical Services, QAS, and BDT by electronic mail no later than the 11th working day. The notification must identify the analytical procedure by lab code and provide a summary of the problem, details of repair or other corrective action(s) being taken, and the expected date of resolution. Before returning to service, any equipment must be calibrated and the new calibration curves verified and documented.

Prior to the return to service, the supervisor must notify the Chiefs of the Laboratory, Analytical Services, QAS, and BDT of the impending start-up of the instrument. The BDT Chief will notify customers of significant delays and expected resolution dates.

3.5 Measurement Traceability

See discussion in Section 3.4.1, Analytical Support Equipment.

3.5.1 Documentation and Labeling of Standards and Reagents

Records are kept for all standards and reagents, including the manufacturer/vendor, the manufacturer's Certificate of Analysis or purity (if supplied), the date of receipt, receipt analyst initials, recommended storage conditions, and an expiration date after which the material is not used unless it is verified.

Original solvents, reagents, reference materials, and standard containers provided by a vendor must be labeled with date of receipt, receipt analyst's initials, date opened, initials of the opener, and expiration date. An expiration date is the date after which the material may not be used unless confirmed as reliable. If no manufacturer-supplied expiration date is listed on the original container, contact the supervisor to determine the expiration date.

Records are maintained for solvents, reagents, and the preparation of standards. These records indicate the source of standard materials (NIST-traceable standards are preferred, if available), with reference to the method of preparation, date of preparation, expiration date, and preparer's initials.

All containers holding materials from vendors (working standards and their precursors tracing back to the vendor-purchased containers, and working reagents and their precursors tracing back to vendor-purchased containers) are marked with the contents and concentrations, date prepared, initials of the preparer, and expiration date.

All this information is recorded in a log book. These prepared reagents and standards bear an identifier and are linked to the documentation requirements given in this manual. For reagents and standards made on a daily basis, 'daily' may be used in lieu of a specific expiration date on the container and in supporting expiration documentation for those reagents and standards. Date of preparation must still be recorded for all reagents and standards. NWQL SOP IX0400.0, *Documenting and labeling standards, reagents, and reference materials*, provides additional detail.

In methods where the purity of reagents and solvents is not specified, analytical reagent grade or better is used. Reagents, gases, and solvents of lesser purity than those specified by the method are not to be used. The labels on the container are checked to verify that the purity of the reagents meets the requirements of the particular method. Such information is documented. The quality of reagent-water sources is monitored and documented to meet method-specified requirements.

3.6 Sampling and Sample Handling

Policies and practices for sample handling are described in this section. Further details are contained in SOPs specific to individual analytical systems.

3.6.1 Sample Collection

Samples are collected by NWQL's customers in accordance with protocols published in the National Field Manual (U.S. Geological Survey, variously dated). The National Field Manual provides guidelines for the collection of water-quality data:

1. establishes and communicates scientifically sound methods and procedures;

2. provides methods that minimize data bias, and, when properly applied, result in data that are reproducible within acceptable limits of variability;

3. promotes consistent use of field methods to produce nationally comparable data; and

4. provides citable documentation for USGS water-quality data-collection protocols.

"Formal training and field apprenticeship are needed to correctly implement the protocols and guidelines" described in the National Field Manual (Wilde and others, 1998, chap. A1, p. 4). Courses are offered through the USGS National Training Center in Denver.

Typical instruction includes the 2-week course "Field Water-Quality Methods for Ground-Water and Surface-Water" (QW 1028C), designed to introduce all Federal and non-Federal employees involved in collecting and processing samples to USGS methodologies for water-quality analyses; and the 5-day course "Quality-control sample design and interpretation" (QW2034), which introduces techniques for using quality-control samples in water-quality projects. The latter course is designed for hydrologists responsible for implementing water-quality data-collection programs and projects and interpreting water-quality data. Courses are offered in Denver, as well as in selected USGS Water Science Centers throughout the United States. Refer to the national and regional courses available through the USGS Office of Employee Development at URL http://training.usgs.gov/ntc/courses/Course_Info/Course_Catalog.cfm.

Requirements for shipping samples to the laboratory are documented in NWQL Technical Memorandum 02.04, Requirements for the proper shipping of samples to the National Water Quality Laboratory (U.S. Geological Survey, 2002c).

3.6.2 Field Supply Quality Assurance

The procedures identified in SOP QX0347.0, *Acquisition, quality assurance and sale of field supplies*, address the NELAC standard requirements "on the need to ensure that field supplies and consumable materials that affect the quality of environmental tests are not used until they have been inspected or otherwise verified as complying with standard specifications or requirements." This pertains to field supplies used to collect samples analyzed at the NWQL.

Responsibility for the quality assurance (QA) of individual field supplies to ensure fitness for use rests with the NWQL. The QAS determines the number of QA samples per lot according to the military specifications for new supplies (Grant and Levenworth, 1996). For items for which there are historical data for different variables, QA sampling is done in accordance with the ASTM Method E 122-99, Standard practice for choice of sample size to estimate a measure of quality for a lot or process (American Society for Testing and Materials, 2003).

Review the following questions to determine the requirements for field-supply QA:

1. Is this a new item purchased from any manufacturer?

2. Are there known or highly probable risks that the supply could contaminate a sample for the target analytes and in the range of analytical method?

3. Is a vendor's QA assessment available?

4. For blank contamination assessment, does the vendor certificate indicate that a supply is suitable for concentrations well below the reporting level for analytes and methods that samples will be used for?

5. For standards, does the certificate indicate that the concentrations are well within acceptance criteria established by MRDP or the analyst?

If the decision is made to perform a QA assessment on the field supply, then the QAS must ensure that methods with the smallest bias, or if necessary, the lowest detection levels, are used for the assessment. In some cases it may be desirable to use concentrated or diluted samples for QA assessment.

Certificates documenting the quality of field supply lots are posted on the NWQL USGS-visible intranet site as appropriate.

For information on organic spike solution QA, see URL http://wwwnwql.cr.usgs.gov/USGS/spike/spike.html.

For information on organic blank water QA, see URL http://wwwnwql.cr.usgs.gov/USGS/OBW/obw.html.

For information on inorganic field supplies, see URL http://wwwnwql.cr.usgs.gov/USGS/certificates/coverpage.html.

Although the Ocala Laboratory is closed, certificates are linked to the NWQL USGS-visible intranet site to retain the certification history for field supplies used to collect samples (URL http://wwwnwql.cr.usgs.gov/USGS/Ocala/OcalaCertificates.html). The link also is available on the inorganic field supplies page.

3.6.3 Sample Tracking

The NWQL uniquely identifies each sample for testing to ensure that there can be no confusion regarding identity. (See SOP TX0076.3, *Login Unit of the National Water Quality Laboratory*.) The sample login system includes individual identification of all sample containers. Subsamples and subsequent extracts and digestates are tracked by analysts.

A unique identification (ID) code is placed on each sample container. This is referred to as the laboratory ID and consists of the four-digit year, the three-digit Julian day, and the sequential four-digit number of login order for that day (for example, 20031920010), and a sequential bottle number. For samples containing more than one bottle type, each bottle receives the same unique number, and each container is labeled with its sample type.

For samples that are received with more than one bottle, each bottle receives a separate designation, such as 20031920010-1, 20031920010-2, and so on. This process minimizes possible bottle mix-up, based upon the use of one bottle in relation to the other. (See SOP IX0104.2, *Investigation and resolution of bottle mix-ups at the National Water Quality Laboratory.*)

3.6.3.1 Holding Chain-of-Custody Samples

Requirements for chain of custody (COC) are described in SOP QX0030.2, *NWQL chain of custody*. Other information on laboratory procedures for COC are included in section 2.5.6.

3.6.3.2 Holding Times

The NWQL tracks sample processing to meet the holding time to ensure data integrity. The holding time is the maximum allowable time after collection that samples may be held before analysis (or constituent isolation) and be considered valid or uncompromised (U.S. Environmental Protection Agency, 2005a). Isolation is achieved by concentration, digestion, solvent extraction, or other sample preparation. Methods with an isolation step often assign holding times to indicate

the maximum allowable time from the isolation process to instrumental analysis.

The holding time is determined by scientific assessment of recovery data in reagent matrix spikes and natural matrix spikes over time whenever possible. For multiconstituent methods, the holding time is determined for each constituent, but management processes may be established to achieve the holding time for the constituent with the shortest holding time.

If holding-time studies have not been conducted for a method, the NWQL relies upon its analytical experience or uses the holding time(s) established by the USEPA in 40 CFR §136.3, table II (U.S. Environmental Protection Agency, 2005a) for a similar analytical method. The best match of analyte or compound class from the USEPA listings will be selected to set the NWQL constituent holding times. Appendix A lists the holding-time data for all routine procedures conducted at the NWQL. Most holding times are tracked based on full calendar days. In the case of holding times less than a few days, steps are taken to track the holding time based on fractions of days.

The NWQL LIMS tracks time-critical processes for each sample, including time of collection, login date, sample preparation date, analysis date, and the release date to the customer.

3.6.4 Sample Receipt and Acceptance

The NWQL has a written sample acceptance policy that outlines the circumstances under which samples are accepted at login described in its SOP TX0076.3, *Login Unit of the National Water Quality Laboratory*, as well as other historical memoranda and SOPs. Data from any samples that do not meet this policy are noted in the data report that defines the nature and substance of the variation. This policy applies to all samples logged in at the NWQL. The policy requires or establishes the following:

1. proper, full, and complete documentation, including the station identification (which may be either a 15-digit number that reflects the latitude and longitude of the site where the sample was collected or an 8-digit number that was assigned earlier to a hydrologic unit, such as a gaging station or other site), the location, date and time of collection, medium code, project chief, account number, user code, collector's name, NWIS sample type, and any special remarks concerning the sample. The station identification must correspond to the identifier entered into the NWIS site file at a USGS Water Science Center.

2. unique identification of samples using durable labels completed in indelible ink;

3. use of correct sample containers, prescribed temperature, and chemical preservation (information in the NWQL services catalog available at the USGS-visible intranet site at URL http://nwql.cr.usgs.gov/usgs/catalog/index.cfm);

4. receipt within holding times (if holding times may be compromised, it is the responsibility of the customer to contact the NWQL for assistance);

5. obvious inadequate sample amount (further checks on volume are the responsibility of analysts);

6. procedures that are used when samples show signs of damage or contamination.

In the instances where all of the above criteria are not met, designated NWQL personnel contact the USGS Water Science Center to resolve the issue.

Upon receipt, the condition of the sample containers, including any abnormalities or departures from standard condition, is recorded in the "NWQL Login Comments" field on the analytical services request form (ASR), the tracking form accompanying all samples. Comments are transmitted to a customer service representative at the NWQL who resolves the question(s) for the customer. "No comment" from Login personnel on the ASR is the default, indicating that samples were received in proper condition. See section 2.5.3 for information on LabLogin procedures.

All ASRs are initialed by the person(s) unpacking the samples and entering the information into the LIMS. If there is any doubt as to a sample's suitability for testing (for example, broken bottles, chilled sam-

ples received warm, incorrect lab codes or schedules, and missing sample types), the cooler(s) is removed from the login process and placed into a "problem sample" refrigerator.

An NWQL representative then consults with the customer for further instruction before proceeding. If the USGS Water Science Center customer still wants the affected sample analyzed, analytical data produced from the improperly received sample (including chilled samples received warm) should not be entered into the NWIS data base. These data may be communicated by memorandum to the customer or released with a remark noting that the proper temperature was not maintained prior to analysis.

After samples are logged in, they are transferred to locations designated by the Analytical Services Section and remote from contaminating sources, such as chemicals and food. Samples are stored in a manner that satisfies NWQL requirements for safety, maintenance of sample integrity, and ease of retrieval by analysts. Further information is included in sections 4.6, "Delivery and storage of completed samples," in NWQL SOP TX0076.3, *Login Unit of the National Water Quality Laboratory*, and 4.2.2.1 in NWQL SOP QX0030.2, NWQL *Chain of Custody*.

3.6.4.1 Verification of Temperature and Chemical Preservation

Temperature verification. For samples with a specified temperature of 4ºC, the temperature is verified during login with a NIST-certified infrared thermometer and recorded into the LIMS. The acceptable temperature range is just above the freezing temperature of water to 6ºC. Samples that must be submitted frozen must arrive at a temperature no greater than -2ºC. The temperature of samples not required to be chilled is not taken and the nominal temperature of 25ºC is assigned. Samples designated as chilled that are collected and submitted to Login during the same day do not need to be at temperatures described above if there is evidence that the chilling process has begun, such as arrival on ice. The NWQL Login Unit refrigerates these samples immediately.

Chemical preservation verification. Verification of pH preservation is conducted by pouring off a small volume of water sample onto a pH test strip and inspecting visually. If the sample has not been properly preserved, the test strip is taped to the side of the sample bottle and a sample preservation corrective action form is prepared. The sample processing and data reporting corrective action that is taken depends on the sample type. Customers are contacted in all cases of improper preservation. Rapi-Note 03-029 describes the procedure, sample handling, and data reporting associated with this process. It is accessible at the NWQL USGS-visible intranet web site at URL http://www nwql.cr.usgs.gov/USGS/rapi-note/03-029.html.

The SOP TX0076.3, *Login Unit of the National Water Quality Laboratory*, presents the detailed procedure for conducting the preservation checks and Appendix 1 identifies the bottle types that require verification, identifies the responsible unit for conducting the verification, and indicates the corrective action to be taken for data processing. Samples for metals (nitric acid) and mercury (hydrochloric acid) can be preserved at the NWQL for subsequent analysis. Data for improperly preserved samples are qualified and a method specific comment is released with the data.

3.6.5 Delivery and Storage of Samples After Login

Upon completion of the login process and login preservation verification, the samples are delivered to predetermined locations throughout the laboratory. Bottle types are described in the NWQL services catalog, accessible at the NWQL USGS-visible intranet web site at URL http://nwql.cr.usgs.gov/usgs/catalog/index.cfm. Delivery and storage of samples is described in SOP TX0076.3, *Login Unit of the National Water Quality Laboratory.*

1. Samples are stored in the 30-day storage area for 30 working days (not calendar days), after which time they are moved into the 180-day storage area by the bottle warehouse custodian. They are stored numerically by tray and bottle type, then from left to right, top to bottom.

2. There is a marking board at the end of each shelf row noting the starting and ending bottle sequence for that row, allowing for ease of retrieval.

3. Bottles are maintained in 180-day storage area for 180 working days (not calendar days), after which time the bottles are removed by the Safety, Health, and Environment office for proper disposal.

4. The bottle storage custodian is responsible for keeping the storage area in proper sequence, clean, and neat; checking and replacing broken bottle caps; and replacing missing or damaged tray labels as needed. Empty trays are returned to Login and placed onto the tray storage bin for reuse.

3.6.6 Sample Disposal

Samples are disposed of as described in SOP SHEX0355.1, *Waste disposal at the National Water Quality Laboratory,* especially section 4Q, "Sample disposal procedures." Sample disposal is in accordance with Federal, State, and local regulations (Colorado Department of Public Health and Environment, 1995) and U.S. Geological Survey (2004), National Water Quality Laboratory Policy Memorandum 04-01, *Pollution prevention and waste minimization policy*. Depending upon the composition of the sample, disposal options include neutralization and passing through the ion exchange system before sewer discharge or shipment to a waste disposal facility that is approved and permitted by the U.S. Environmental Protection Agency.

3.7 Reporting the Results

3.7.1 Report Format and Contents

Data are reported according to NWQL guidelines, primarily as stated in the relevant published analytical method or approved SOP. Less-certain results than specified in these policy statements are qualified appropriately or deleted according to method-specific acceptance criteria outlined in the appropriate SOP. Details on detection limits and data quantitation are discussed in Childress and others (1999).

Routine data with a single result for an analyte are transmitted electronically and stored in the USGS National Water Information System (NWIS) data base. Custom or provisional data that do not yet have assigned parameter codes are transmitted by memorandum. These data are entered into the NWIS data base with data-quality indicators (flags) to show that the results are from a custom, provisional, or unapproved method.

Occasionally, multiple methods with overlapping analytes are requested for a single sample (same sample site/date/time/medium/type). When multiple results for the same analyte occur, the NWQL sends the most preferred value to the customer electronically because NWIS can only accept one result. All other results (the non-preferred ones) are sent via electronic mail to the customer. The process for determining which data are preferred is described in Hierarchical procedure used in selecting the ‘preferred’ method for organic analyses, accessible at URL http://wwwnwql.cr.usgs.gov/USGS/Preferred_method_selection_procedure.html.

All results are reported according to a strict format that allows for transmission and storage in NWIS. Additional paper reports are not standard and must be requested with specific needs (that is, specific report format, specific protocols, or specific QC samples) on a custom basis, as described in SOP AX0075.2, *Proposal and contract process at the NWQL.*

Any positively identified analyte may be reported, but the concentration uncertainty increases as the concentration is extrapolated lower than the lowest calibration standard or higher than the highest calibration standard. Data are reported according to four conventions (fig. 3.1):

1. The minimum reporting level (MRL) derives the reporting level from means other than using an LT–MDL study.

2. The laboratory reporting level (LRL) derives a reporting level and an LT–MDL value by using the LT–MDL study.

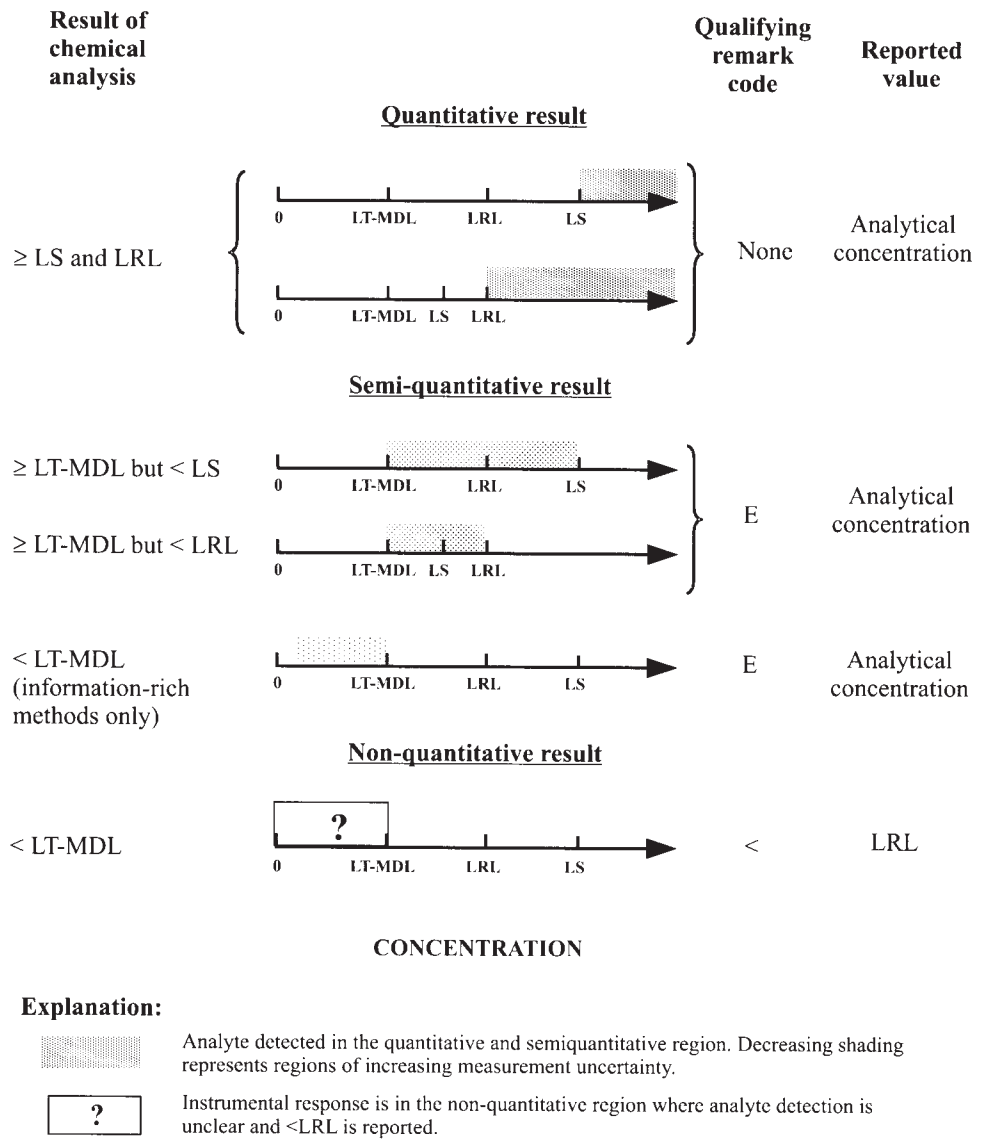

Figure 3.1. New low-concentration reporting conventions showing the reported value and associated qualifying remark code in relation to the long-term method detection level (LT–MDL), the laboratory reporting level (LRL), and the lowest calibration standard (LS). [>, greater than or equal to; <, less than; E, estimated] Source: http://water.usgs.gov/owq/OFR_99-193/images/fig10.html, Childress and others (1999).

3. The interim reporting level (IRL) uses an interim reporting level LT–MDL value that is derived from means other than the LT–MDL study.

4. Information-rich methods can use either the LRL or IRL convention, but add an additional low reporting level typically set at 1 percent of the LT–MDL value.

There are five ways for analysts to convey quantitative information:

1. report the concentration as found if the measurement is within the calibration range;

2. dilute the sample to within the calibration concentration range (ideally to the center of the calibration range) and multiply the resultant datum by the dilution factor if the measurement is higher than the highest calibrant;

3. estimate and qualify the concentration if the measurement is lower than the lowest calibration standard or less than the reporting level, and use appropriate value qualifier; or

4. censor detections at less than the reporting level.

Censoring detections at less than the minimum reporting level is the normal operating procedure for analytical laboratories, and the result returned for these data is "less than" the reporting level (LRL, MDL, or MRL).

Censoring values also are known as remark codes and are referred to as such in all NWIS documentation. Remark codes provide additional information about the magnitude (or absence) of a value. The remark code is almost always viewed with the value in the software to avoid misinterpretation of the value. Valid NWIS remark codes and descriptions can be found at the URL http://phoenix.cr.usgs.gov/www/rmk_qual.html.

The only exception to this standard is reporting data between the LRL and LT–MDL (data are qualified with an "E" code). "Less than" (<) the reporting level is the typical broad-spectrum analytical tool for stating nondetection of a constituent, or

5. qualify the concentration if the measurement is derived from an information-rich method (URL http://water.usgs.gov/owq/OFR_99-193/conventions.html#methods) and the measurement is less than the LT–MDL and greater than the lowest reportable value, which is typically set at 1 percent of the LT–MDL value.

Data from undiluted samples may not be reported if they are greater than the highest calibration standard used. The sample must be diluted so that the concentration falls within the calibration curve. The observed concentration then is multiplied by the dilution factor. The only exceptions to this rule are those constituents that cannot be reanalyzed; these data are censored in the appropriate manner ["E" coded or "greater than" (>)]

If a corrective action, such as the identification of defective measuring or test equipment, casts doubt on the validity of results given in any test report or amendment to a report, customers are notified in writing. Notification may be by individual electronic mail, USGS Office of Water Quality Technical Memorandum, or Rapi-Note.

3.7.2 Value Qualifiers and Data Quality Indicators

Value qualifier codes provide information about the process used to determine an analytical value and, often, the remark code associated with the value. Up to three value qualifiers can be stored with any single result. Valid NWIS qualifier codes, usage, and descriptions can be found at the URL http://phoenix.cr.usgs.gov/www/rmk_qual.html. The following qualifiers are currently in use:

1. A 'b' qualifier is added when the value falls below the lowest calibration standard but above the reporting level.

2. An 'n' qualifier is added when the value falls above the LT–MDL and below the LRL and the

method is using either the LRL, IRL, or information-rich conventions.

3. A ‘t’ qualifier is added when the value falls below the LT–MDL value and above the lowest reporting value for information-rich methods.

4. A ‘*’ qualifier is added for values determined from bottles that are supposed to be chilled, but were received warm or became warm at the laboratory. In this case, the values above the reporting level receive a remark code of ‘E’ as well.

5. A ‘+’ qualifier is added for values determined from nonmetals analyses on bottles that were improperly preserved.

6. A ‘d’ qualifier is added when a dilution greater than 1 is performed on an analysis. A dilution equal to 1 is no dilution at all.

7. An ‘m’ qualifier is added when the compound is identified as a highly variable compound when analyzed for the current method. These compounds are often referred to as ‘flakes.’

8. An ‘o’ qualifier is added when the value obtained is derived from a method that was not the method originally requested. These generally occur when a low-level method is requested and the value falls above the calibration range of the low-level method. Instead of diluting and re-analyzing on the low-level method, the request is transferred to another method.

9. A ‘c’ qualifier is added whenever a comment is released with the result.

Data-quality indicator (DQI) codes indicate the review status of a result, control the ability of a batch input program to overwrite a value, and affect the inclusion of a result in output. Valid NWIS DQI codes can be found at the URL http://phoenix.cr.usgs.gov/www/dqi.html.

The DQI codes used for methods are as follows:

1. I — currently in review;

2. S — presumed satisfactory; and

3. U — unapproved.

3.7.3 Data Release Batch File Format

Data release from the NWQL to the NWIS data system occurs daily and uses two files in batch file format. The format of the batch files can be found at the URL http://wwwnwis.er.usgs.gov/nwisdocs4_3/qw/QW-AppxF.pdf.

Precision (for NWIS output) and rounding at the NWQL. A seven-character decadal rounding array is stored for each parameter code and method combination. Each individual character corresponds with a decade of the actual value. The decadal significance for each byte of the rounding array is as follows:

1st	2nd	3rd	4th	5th	6th	7th
< 0.01	0.01 to < 0.1	0.1 to < 1	1 to < 10	10 to < 100	100 to 1,000	> or = to 1,000

An example of a corresponding rounding array for a parameter code and method combination is as follows:

1st	2nd	3rd	4th	5th	6th	7th
3	3	2	2	2	1	1

As an example, a result of 0.4567 with an associated rounding array of 3322211 lies within the 3rd decadal significance (0.1 to < 1, from above). Therefore, the rounding calculation will use the value in the 3rd digit of the rounding array, which is a 2 in this example. This is the number of significant digits allowed for a value that lies between 0.1 and < 1. The result of 0.4567 is therefore rounded to two significant digits, giving the final value of 0.46.

The number of significant digits, determined by the rounding array, is sent to NWIS as the precision code value, which is 2 in this example. Additional information on the NWIS precision codes is accessible at URL http://phoenix.cr.usgs.gov/www/rounding.html.

The precision differs from significance for values less than 1. Precision above 1 is the number of significant digits; precision for values less than 1 is the number of decimal places. For example, if we have a rounding array with a 1 in the 1st byte and a value of 0.005, the precision value would be 3 (that is, three decimal places). A fact sheet entitled "How can I round my results in QWDATA output?" is available at URL http://www nwis.er.usgs.gov/nwisdocs4_5/qw/QW-TipSheet5.22.pdf and explains how NWIS currently interprets the digit sent by the NWQL as "sigfig/precision" with each result.

In many instances the NWQL creates multiple results under the same parameter code for a single sample. Due to a limitation in the NWIS data structure, the NWQL is unable to send values for the same collection activity and method to NWIS in the batch format. In 1985 it was discovered that doing so overwrites any previously stored values in the NWIS system and therefore data are lost. The NWQL LIMS stores multiple values for the collection/method key because of schedules that contain overlapping parameter codes.

To alleviate the NWIS overwrite problem, the NWQL data release application determines when overlapping data exist and uses a release order to determine which results will be sent in the data release files and which results will be sent in the duplicate electronic mail. These electronic messages are sent to the Project Chief and the USGS Water Science Center contact.

The following information is included in the duplicate electronic mail: station identification, sample date, sample time, medium code, account number, laboratory identification, parameter code, method code, remark code, result, and precision. After the electronic mail is delivered, a delivery status is set to ‘E’ to identify the result as being sent by electronic mail.

Data release from the NWQL to the Organic Blind Sample Program (OBSP) occurs daily. The results to be released in the OBSP format are identified by the user code from the analytical services request. A header file with sample information, a result file, and a file of parameter information are provided in separate files that are named “qc_header”, “new_qc_results” and “new_qc_parm,” respectively. The output files are retrieved and processed into the OBSP data base.

3.8 Confidentiality and Sensitive Information

By USGS design, scientific information is protected and restricted, as appropriate, to the Federal and non-Federal employees or other authorized individuals who have a legitimate need to know. Data are transmitted only to the requesting USGS Water Science Center and to the public through NWIS after USGS Water Science Center review and approval, unless prearranged with the NWQL. NELAP-related records are made available to authorized accrediting-authority personnel or to other personnel as necessary.

Data produced using new, unapproved methods or research methods may not be published in data reports (U.S. Geological Survey, 1998a).

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Appendix A — Method Holding Times Used by the National Water Quality Laboratory

[Reference to the National Water Quality Laboratory as "NWQL" as a source for a holding time refers to holding times currently (2005) in practice at the laboratory that do not have a specific reference, but may be described in standard operating procedures (SOP) or other internal documents. Citations for analytical methods and holding times listed in Appendix A may be found online at the NWQL services catalog page on the NWQL USGS-visible intranet web site (accessed October 3, 2003) at URL http://nwql.cr.usgs.gov/usgs/catalog/index.cfm.

<, less than; °C, degrees Celsius; ACT/ULUG, agricultural chemical transport/urban land-use gradient; aquatic biota (populations), holding time reflects the time from arrival at the NWQL to stabilization with ethanol; ASF, automated segmented flow; BTEX, benzene, toluene, ethylbenzene, and xylenes; $CaCO_3$, calcium carbonate; CFR, Code of Federal Regulations; cICP–MS, collision/reaction cell inductively coupled plasma–mass spectrometry; DBCP, 1,2-Dibromo-3-chloropropane; DTH, depositional-targeted habitat (NAWQA); EDB, 1,2-Dibromoethane; EDTA, ethylenediamine tetraacetic acid; GC–ECD, gas chromatography–electron capture detection; GC–MS, gas chromatography–mass spectrometry; GC–MS–FS, gas chromatography–mass spectrometry–full scan; HPLC, high-performance liquid chromatography; IC, ion chromatography; ICP, inductively coupled plasma; ICP–MS, inductively coupled plasma–mass spectrometry; lab, laboratory; LC, laboratory code; Leco, instrument name; mm, millimeter; MRL, minimum reporting level; MS, mass spectrometry; MTBE, methyl tertiary-butyl ether; N, nitrogen; NAWQA, National Water-Quality Assessment Program; nm, nanometer; NO_2, nitrite; NO_3, nitrate; NPDES, National Pollution Discharge and Elimination System; NTRU, nephelometric, turbidity ratio units; OFR, Open-File Report; OI, Oceanography International; P, phosphorus; PAH, polycyclic aromatic hydrocarbon(s); PCB, polychlorinated biphenyl; PCN, polychlorinated naphthalene; RTH, richest targeted habitat (NAWQA); SiO_2, silicon dioxide; SPE, solid-phase extraction; SPE–HPLC–MS, solid-phase extraction–high-performance liquid chromatography-mass spectrometry; USEPA, U.S. Environmental Protection Agency; USGS, U.S. Geological Survey; UV, ultraviolet; WRIR, Water-Resources Investigations Report; µg/L, microgram per liter; µS/cm, microsiemens per centimeter at 25 °C]

Parameter–method code pair	LC/schedule	Description	Holding time[a]	Reference and source of holding time
		Aquatic biota (populations)		
LC2172	2172	300-count subsample with standard taxonomic assessment	14	Moulton and others (2000), NWQL
LC2174	2174	100-count subsample with standard taxonomic assessment	14	Moulton and others (2000), NWQL
LC2176	2176	qualitative visual sort with standard taxonomic assessment	14	Moulton and others (2000), NWQL
LC2892	2892	field-sorted, 100-count subsample with standard taxonomic assessment	14	Moulton and others (2000), NWQL
		Aquatic biota (plant pigments)		
various	3152	chlorophyll and pheophytin *a* in phytoplankton by fluorescence, USEPA Method 445.0	25[b]	Arar and Collins (1997)
various	3153	chlorophyll and pheophytin *a* in periphyton (RTH) by fluorescence, USEPA Method 445.0	25[b]	Arar and Collins (1997)
various	3154	chlorophyll and pheophytin *a* in periphyton (DTH) by fluorescence, USEPA Method 445.0	25[b]	Arar and Collins (1997)
		Aquatic biota (biomass)		
81354B	2190	phytoplankton biomass as dry weight, gravimetric	30	NWQL
81353B	2189	phytoplankton biomass as ash weight, gravimetric	30	NWQL

Parameter–method code pair	LC/schedule	Description	Holding time[a]	Reference and source of holding time
00573A	603	periphyton biomass as dry weight, gravimetric	30	NWQL
00572A	611	periphyton biomass as ash weight, gravimetric	30	NWQL
		Aquatic biota (trace elements in tissue)		
various[c]	various[c]	ICP, dry weight	180	NWQL
various[c]	various[c]	ICP–MS, dry weight	180	NWQL
various[c]	various[c]	cICP–MS, dry weight	180	Garbarino and others (in press)
49258B	6050	mercury by cold vapor atomic fluorescence, dry weight	180	NWQL
		Radiochemistry		
82303B	1369	radon	3	NWQL
		General inorganic chemistry and physical properties in water or sediment		
00080A	20	color reported in platinum-cobalt units	30	NWQL
00950B	31	ASF, ion-selective electrode, fluoride, filtered water	180	NWQL
00955C	56	colorimetry, ASF, molybdate blue, silica as SiO_2, filtered water	180	NWQL
00403A	68	electrometric electrode, pH, lab	30	NWQL
90095A	69	Wheatstone bridge, specific conductance, lab, whole-water recoverable	30	NWQL
90410A	70	electrometric titration, acid neutralizing capacity as $CaCO_3$, lab, whole-water recoverable	30	NWQL
38260A	96	methylene blue active substances, whole-water recoverable	10	NWQL
29801A	2109	electrometric titration, alkalinity as $CaCO_3$, lab, filtered water	30	NWQL
00340C	2144	chemical oxygen demand, Hach	30	NWQL
99872A	2187	nephelometric, turbidity ratio units as NTRU	30	NWQL
70300A	27	gravimetric, residue on evaporation at 180 °C, solids, filtered	180	NWQL
00515B	159	gravimetric, residue on evaporation at 105 °C, solids, filtered	180	NWQL
00500A	165	gravimetric, residue on evaporation at 105 °C, solids, total	180	NWQL
00530B	169	gravimetric, residue on evaporation at 105 °C, suspended solids	180	NWQL
00535A	49	gravimetric, volatile on ignition at 550 °C, suspended solids	180	NWQL
71865D	1202	colorimetry, ASF, ceric-arsenious, iodide, filtered water	180	NWQL
71870E	1246	colorimetry, ASF, fluorescein, bromide, filtered water	180	NWQL

Parameter–method code pair	LC/schedule	Description	Holding time[a]	Reference and source of holding time
71870F	1258	IC, bromide, low level (specific conductance < 100 µS/cm), filtered water	180	NWQL
00940I	1259	IC, chloride, low level (specific conductance < 100 µS/cm), filtered water	180	NWQL
00950D	1260	IC, fluoride, low level (specific conductance < 100 µS/cm), filtered water	180	NWQL
00945E	1263	IC, sulfate, low level (specific conductance < 100 µS/cm), filtered water	180	NWQL
00940J	1571	IC, chloride, filtered water	180	NWQL
00945G	1572	IC, sulfate, filtered water	180	NWQL
00680A	114	OI, organic carbon, total, whole-water recoverable	28	NWQL
00694A	2606	total particulate carbon, suspended	90	Zimmermann and others (1997)
00688B	2608	total particulate inorganic carbon, suspended	90	Zimmermann and others (1997)
00689B	2611	particulate organic carbon, suspended	90	Zimmermann and others (1997)
00681D	2612	organic carbon, dissolved, filtered water, Supor filter, sulfuric-acid preserved	28	NWQL
00681C	2613	organic carbon, dissolved, filtered water, glass fiber filter, sulfuric-acid preserved	28	NWQL
LC2607	2607	total particulate nitrogen, suspended	90	Zimmermann and others (1997)
00693A	133	Leco, total carbon, bottom material (sediment)	90	NWQL
49270E	2319	coulimetric, inorganic carbon, for NAWQA, bottom material (sediment)	90	NWQL
00686E	2321	coulimetric, inorganic carbon, bottom material (sediment)	90	NWQL
00556B	2125	oil and grease	28	NWQL
45501A	2126	total petroleum hydrocarbons	28	NWQL
32730B	2322	total recoverable phenols in water	28	U.S. Environmental Protection Agency (2005a), table II
50624B	2614	UV-absorbing organic constituents, 254 nm, filtered, glass fiber filter	8	NWQL
61726B	2615	UV-absorbing organic constituents, 280 nm, filtered, glass fiber filter	8	NWQL
59624C	2616	UV-absorbing organic constituents, 254 nm, filtered, Supor filter	8	NWQL
61726C	2617	UV-absorbing organic constituents, 280 nm, filtered, Supor filter	8	NWQL

Parameter–method code pair	LC/schedule	Description	Holding time[a]	Reference and source of holding time
		Nutrients		
00613F	1973	colorimetry, ASF, nitrite as N, filtered water	30	Patton and Gilroy (1999)
00671H	1974	colorimetry, ASF, phosphomolybdate, orthophosphate as P, filtered water	30	Patton and Gilroy (1999)
00631E	1975	colorimetry, ASF, cadmium reduction-diazotization, nitrite + nitrate as N, filtered water	30	Patton and Gilroy (1999)
00608F	1976	colorimetry, ASF, salicylate-hypochlorite, ammonia as N, filtered water	30	Patton and Gilroy (1999)
00613H	1977	colorimetry, ASF, nitrite as N, low level, filtered water	30	Patton and Gilroy (1999)
00671I	1978	colorimetry, ASF, phosphomolybdate, orthophosphate as P, low level, filtered water	30	Patton and Gilroy (1999)
00631G	1979	colorimetry, ASF, cadmium reduction-diazotization, nitrite + nitrate as N, low level, filtered water	30	Patton and Gilroy (1999)
00608H	1980	colorimetry, ASF, salicylate-hypochlorite, ammonia as N, low level, filtered water	30	Patton and Gilroy (1999)
00666D	1983	colorimetry, ASF, microkjeldahl digestion, phosphorus as P, filtered water	30	Patton and Gilroy (1999)
00665D	1984	colorimetry, ASF, microkjeldahl digestion, phosphorus as P, whole-water recoverable, acidified	30	Patton and Gilroy (1999)
00623D	1985	colorimetry, ASF, microkjeldahl digestion, ammonia + organic nitrogen as N, filtered water	30	Patton and Gilroy (1999)
00625D	1986	colorimetry, ASF, microkjeldahl digestion, ammonia + organic nitrogen as N, whole-water recoverable, acidified	30	Patton and Gilroy (1999)
00631F	1990	colorimetry, ASF, cadmium reduction-diazotization, nitrite + nitrate as N, filtered water, acidified	30	Patton and Gilroy (1999)
00608G	1991	colorimetry, ASF, salicylate-hypochlorite, ammonia as N, filtered water, acidified	30	Patton and Gilroy (1999)
00623E	1994	colorimetry, ASF, microkjeldahl digestion, ammonia + organic nitrogen as N, filtered water, acidified	30	Patton and Gilroy (1999)
00610E	2188	colorimetry, ASF, ammonia as N, whole-water recoverable	30	Patton and Gilroy (1999)
00666G	2331	low-level total phosphorus, filtered water	30	Patton and Gilroy (1999)
00666H	2332	low-level total phosphorus, filtered water, acidified	30	Patton and Gilroy (1999)
00665G	2333	low-level total phosphorus, whole-water recoverable, acidified	30	Patton and Gilroy (1999)
00668B	515	colorimetry, ASF, phosphomolybdate, phosphorus as P, bottom material (sediment)	180	NWQL
00611A	524	colorimetry, ASF, salicylate hypochlorite, ammonia as N, bottom material (sediment)	180	NWQL
00633A	513	nitrate + nitrite, bottom material (sediment), low demand	180	NWQL
62854A	2754	total nitrogen, filtered water, alkaline persulfate digestion	30	Patton and Gilroy (1999)

Parameter–method code pair	LC/schedule	Description	Holding time[a]	Reference and source of holding time
62854B	2755	total nitrogen, filtered water, acidified, alkaline persulfate digestion	30	Patton and Gilroy (1999)
62855A	2756	total nitrogen, filtered water, acidified, alkaline persulfate digestion	30	Patton and Gilroy (1999)
00666I	2757	total phosphorus, filtered water, alkaline persulfate digestion	30	Patton and Gilroy (1999)
00666J	2758	total phosphorus, filtered water, acidified, alkaline persulfate digestion	30	Patton and Gilroy (1999)
00665A	2759	total phosphorus, unfiltered water, acidified, alkaline persulfate digestion	30	Patton and Gilroy (1999)
		Trace elements, mercury, and arsenic species		
various[e]	various[e]	ICP, filtered and unfiltered water, acidified	180	U.S. Environmental Protection Agency (2005b)
various[e]	various[e]	ICP, bed sediment < 2 mm, digestate	180	U.S. Environmental Protection Agency (2005b)
various[e]	various[e]	ICP, potassium, low level (specific conductance < 100 µS/cm), filtered water, acidified	180	U.S. Environmental Protection Agency (2005b)
various[e]	various[e]	ICP–MS or cICP–MS, filtered and unfiltered water, acidified	180	U.S. Environmental Protection Agency (2005b)
various[e]	various[e]	ICP–MS or cICP–MS, sediment and soil < 2 mm, digestate	180	U.S. Environmental Protection Agency (2005b)
71890C	2707	cold vapor–atomic fluorescence spectrometry, organic plus inorganic mercury, filtered water, acidified	30	Garbarino and Damrau (2001)
71900C	2708	cold vapor–atomic fluorescence spectrometry, organic plus inorganic mercury, recoverable, unfiltered water, acidified	30	Garbarino and Damrau (2001)
various[e]	various[e]	arsenite and arsenate by ICP–MS or cICP–MS, filtered water, field separation, EDTA added	49 (cartridge) 120 (extract)	U.S. Geological Survey (variously dated) and Garbarino and others (2002, in press)
various[e]	various[e]	arsenite and arsenate by HPLC ICP–MS or cICP–MS, filtered water, lab separation, malonate/acetate, EDTA added, opaque bottle	90	Garbarino and others (2002, in press)
various[e]	various[e]	arsenic species (arsenite, arsenate, monomethylarsonate and dimethylarsinate) by HPLC ICP–MS or cICP–MS, filtered water, phosphate mobile, EDTA added, opaque bottle	90	Garbarino and others (2002, in press)
various[e]	various[e]	arsenic species (arsenite, arsenate, monomethylarsonate and dimethylarsinate) by HPLC ICP–MS or cICP–MS, filtered water, nitric acid mobile, EDTA added, opaque bottle	90	Garbarino and others (2002, in press)

Parameter–method code pair	LC/schedule	Description	Holding time[a]	Reference and source of holding time
		Organic methods (all matrices)		
10079	79	chlorophenoxy-acid herbicides, Dicamba and Picloram, whole-water recoverable	7[b]	NWQL
11304	1304	chlorophenoxy-acid herbicides, whole-water recoverable	7[b]	NWQL
11379	1379	organonitrogen herbicides (Triazine), filtered water	7[b]	NWQL
11389	1389	organonitrogen herbicides (Triazine), whole-water recoverable	7[b]	NWQL
12001	2001	herbicides (Triazine), filtered water, SPE-C18 (lab extraction)	7[d]	NWQL
12010	2010	herbicides (Triazine), filtered water, SPE-C18 (field extraction)	7	NWQL
12052	2052	glyphosate, aminomethylphosphonic acid, and glufosinate, filtered water, HPLC–MS, SPE	24	Lee and others (2002)
11306	1306	GC–MS, volatile organic compounds, soil fumigants, EDB and DBCP, whole-water recoverable	14	Munch (1995a)
11307	1307	34 regulated volatile organic compounds, whole-water recoverable, acidified	14	Connor and others (1998)
11380	1380	61 volatile organic compounds, reported at < 0.2 µg/L, whole-water recoverable	14	Connor and others (1998)
12020	2020	NAWQA, 85 volatile organic compounds, with nontargets, acidified, whole-water recoverable	14	Connor and others (1998)
12021	2021	NAWQA, 85 volatile organic compounds, whole-water recoverable	14	Connor and others (1998)
14054	4054	85 volatile organic compounds at MRLs, and identifications, whole-water recoverable, acidified	14	Connor and others (1998)
11383	1383	semivolatile compounds—chlorinated hydrocarbons, haloethers, nitroaromatics and isophorone, nitrosoamines, phthalate esters, polynuclear aromatics, and priority pollutant phenols	7[b]	NWQL
12502	2502	NAWQA semivolatile organic compounds in bottom material (sediment)	180	Furlong and others (1996)
11319	1319	organophosphate insecticides, whole-water recoverable	7[b]	NWQL
11402	1402	organophosphorus pesticides, filtered water	7[d]	NWQL
11403	1403	organophosphorus pesticides, unfiltered water	7[d]	NWQL
11404	1404	organophosphorus pesticides, bed sediment	7[d]	NWQL
11324	1324	organochlorine insecticides, with gross PCBs and gross PCNs	7[b]	NWQL
11398	1398	organochlorine insecticides, with gross PCBs and gross PCNs (low level)	7[b]	NWQL
11608	1608	organochlorine insecticides, with specific PCB aroclors (NPDES, USEPA Method 608)	7[b]	U.S. Environmental Protection Agency (2005a), pt. 136, app. A
11364	1364	GC–ECD, pesticides, PCB aroclors	7[b]	NWQL

Parameter–method code pair	LC/schedule	Description	Holding time[a]	Reference and source of holding time
12101	2101	NAWQA organochlorine pesticides and total PCB, tissue	90	NWQL
12501	2501	NAWQA organochlorine pesticides in bottom material (sediment)	180	Furlong and others (1996)
15504	5504	organochlorine insecticides and PCBs in bottom material (sediment)	180	Noriega and others (2004)
12504	2504	selected PAHs and total PCBs in bottom material (sediment) by GC–ECD	180	Noriega and others (2004)
12505	2505	selected PAHs in bottom material (sediment) by GC–MS	180	Noriega and others (2004)
12506	2506	alkylated PAHs in bottom material (sediment) by GS–MS	180	Noriega and others (2004)
11433	1433	GC–MS–FS, SPE, wastewater compounds	14	NWQL
11494	1494	base/neutral plus acid-extractable (combined extracts)	7[b]	NWQL
12002	2002	moderate-use pesticides and degradation compounds (lab extraction)	7[d]	NWQL
12011	2011	moderate-use pesticides and degradation compounds (field extraction)	7	NWQL
12003	2003	NAWQA ACT/ULUG, selected pesticides and degradation compounds	7	NWQL
12060	2060	HPLC–MS, SPE, polar pesticides and metabolites	7	NWQL
11378	1378	fuel compounds, BTEX, MTBE	14	Connor and others (1998)
12023	2023	NAWQA BTEX and fuel ethers	14	Connor and others (1998)
14024	4024	gasoline oxygenates and degradates	14	Rose and Sandstrom (2003)
14025	4025	gasoline oxygenates, degradates, and BTEX	14	Rose and Sandstrom (2003)

[a]The holding time is the maximum time in calendar days that a sample may be held prior to analysis and its analytical results still be cosidered valid.

[b]The holding time is based upon the arrival of the sample at the NWQL. Samples may be stabilized with a preservative upon arrival.

[c]Information on individual analytes may be found in the NWQL services catalog, accessible on the USGS-visible intranet page at URL http://nwql.cr.usgs.gov/usgs/catalog/index.cfm

[d]Samples received for some methods are extracted within 2 working days of receipt at the laboratory. NWQL SOPs OD0341.1, *Analysis of pesticides in water by SIM–GC–MS, LS 2002/2011*, and OD0053.4, *Automated preparation by method 2001: NWQL schedules 2001, 2002, 2010, and 2011*, may be useful references.

APPENDIX B — Essential Quality Control and Quality Assurance Requirements

B.1 Chemical Testing

B.1.1 Laboratory Quality Control Samples

The data acquired from quality control (QC) procedures are used to assess the quality of analytical data, to determine the need for corrective action in response to identified deficiencies, and to interpret results after corrective-action procedures are implemented. Each method SOP includes a QC section that addresses the minimum QC requirements for the procedure. The internal QC checks might differ for each individual procedure, but they include positive and negative controls that are described below.

The application, acceptance limits, and corrective actions for these QC checks are primarily derived from NWQL-generated historical analytical data, approved methods, Branch of Quality Systems regression equations, acceptance limits provided by suppliers of materials used to make QC samples, and SOPs. The LIMS allows analysts and supervisors access to QC data close to real time.

B.1.1.1 Negative Controls

Method blanks. Much of the following section is derived from the National Environmental Laboratory Accreditation Conference (NELAC) standards (National Environmental Laboratory Accreditation conference, 2003a).

A method blank consists of a defined,well-characterized matrix with minimal analyte interferences where possible, that is carried through the entire sample preparation and analytical procedure. All reagents are added in the same volumes or same proportions as they are added to the environmental samples. For most water samples, analyte-free reagent water is the synthetic matrix used as the method blank matrix.

For inorganic prcedures, the synthetic matrix used generally is the in-house deionized water followed by a final ultrapure deionizing and polishing that results in ASTM Type I reagent water (American Society for Testing and Materials, 2001). Cartridges to produce the Type I reagent water are replaced as necessary, and cartridge-holder labeled with the date of installation.

For organic procedures, reagent water is prepared by deionization and further purified by one or more of the following steps: distillation, filtration, boiling, purging with nitrogen, ultrapure deionizing and polishing, or ultraviolet irradiation. For organic tissue and sediment samples, sodium sulfate salt or scientific sand, or both, may be used as the synthetic matrix blank.

At least one method blank is required per batch of samples in each sample extraction or preparation test method. The result is one of the QC measures used to assess batch acceptance. The purpose of a method blank is to assess the preparation batch for possible contamination during preparation, processing, and instrumental analysis.

The method blank is processed along with, and under the same conditions, as the associated samples to include all steps of the analytical procedure. The treatment of organic blank data is described in the Organic QA/QC Guidance Manual (M.P. Schroeder, U.S. Geological Survey, written commun., 1999). The treatment of inorganic blank data is addressed in method SOPs.

The goal is no detectable contaminants. Nevertheless, each method blank must be evaluated critically as to the nature of possible contamination and the effects on the analysis of each sample within the batch.

If a method blank is contaminated, the source must be investigated and measures taken to minimize or eliminate the problem. Samples associated with a contaminated blank are evaluated as to the best corrective action for the affected samples (that is, reprocessing or assigning data-qualifying codes).

Multicomponent techniques have different procedures for troubleshooting because of insufficient

sample remaining or lengthy analysis time; these data are qualified in reporting to the customer.

B.1.1.2 Positive Controls

Laboratory control samples (LCS). The NWQL analytical systems analyze one or more of a series of reference samples called LCS. LCS include standard reference water samples, reagent spikes, certified reference materials, surrogate spikes, and continuing calibration verification (CCV) standards.

The LCS are used to evaluate the performance of the total analytical system, including all preparation and analysis steps. The number of LCS samples can vary and is either specified in the method or SOP. Data from the LCS are compared to established criteria, and, if found to be outside of the criteria, indicate that the analytical system is out of specification. Relevant LCS data are entered into control charts. Any affected samples associated with an out-of-specification LCS are reanalyzed or the results reported with appropriate data-qualifying codes.

One or more LCS are analyzed at a minimum of 1 per batch (set) of 20 environmental samples. Exceptions to this guideline would be for those analytes that have no LCS, such as total volatile solids and color. The in-bottle digestion (INBD) preparation process also is an exception. The digestion process performed at the NWQL is unique in that samples are digested in a closed container and never exposed to the possibility of cross-contamination. The INBD process can digest as many as 180 samples in one set and only one LCS is required.

Analyte concentrations in the LCS must be within the calibration range of the methods where possible. An LCS that is determined to be within the acceptance criteria effectively establishes that the analytical system is in control and validates system performance for the samples in the associated batch.

Samples analyzed along with an LCS determined to be “out of acceptance limit” are reprocessed and reanalyzed, or the data are reported with data-qualifying codes. Constituents permanently marked with an “E” code (remark code indicating an estimated concentration) are excluded from these requirements because they do not perform ideally in a given method; typically, they cause large standard deviations or frequent performance problems.

The National Environmental Laboratory Accreditation Conference (2003a), section D.1.1.2.1e, describes the procedures to be implemented when analytes in the LCS are outside control limits. For analytical systems determining many analytes in the LCS, it is statistically likely that some may be outside the control limits. This result might not indicate that the system is out of control; therefore, corrective action may not be necessary.

Upper and lower marginal exceedance (ME) limits can be established to determine when corrective action is necessary. A ME is defined as being beyond the LCS control limit (three standard deviations), but within the ME limits. ME limits are between three and four standard deviations from the mean. The number of allowable marginal exceedances is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and corrective action is necessary. This marginal exceedance approach applies to methods with more than 10 analytes.

The number of allowable marginal exceedances is as follows:

1. >90 analytes in LCS, five analytes allowed in ME of the LCS control limit;

2. 71–90 analytes in LCS, four analytes allowed in ME of the LCS control limit;

3. 51–70 analytes in LCS, three analytes allowed in ME of the LCS control limit;

4. 31–50 analytes in LCS, two analytes allowed in ME of the LCS control limit;

5. 11–30 analytes in LCS, one analyte allowed in ME of the LCS control limit;

6. <11 analytes in LCS, no analytes allowed in ME of the LCS control limit;

All analytical systems using marginal exceedance must have a written procedure in the method's analytical SOPs to monitor the application of ME allowance to LCSs to ensure random behavior, so that persistent systematic problems are not overlooked because of the ME procedure.

Marginal exceedance also applies to continuing demonstration of capabilities, but specifically not to initial demonstration of capabilities.

B.1.1.2.1 Standard Reference Samples

Standard reference samples (SRS) are prepared by the USGS Branch of Quality Systems to check and monitor inorganic analytical systems. These reference samples are produced from large, homogeneous quantities of water, bottled under strict aseptic conditions, and designed to minimize contamination sources. These references usually are composed of a natural matrix collected from different sources, such as snowmelt, streams, and ground water. They also include SRS prepared from certified reference standards and materials, spiked into reagent-grade ASTM Type I water.

B.1.1.2.2 Reagent Spikes

Another LCS is a controlled matrix known to be free of analytes of interest where possible, and subsequently spiked with known and verified concentrations of analytes. In some cases, it might not be reasonable or feasible to spike simultaneously all of the method analytes into a single LCS. Multicomponent mixtures, such as PCBs or toxaphene, might coelute or interfere with other analytes in a single organochlorine LCS.

Concerns regarding analyte cross-reactions, solubilities in the spiking solvent, and component availability from vendors also might limit the capability to prepare a single suitable LCS. In such cases, the LCS might be made from a subset of the entire method list intended to represent the method performance characteristics for all of the analytes. It also might be appropriate to prepare multiple LCSs per sample set.

The individual LCS is compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the NWQL determines internal criteria and documents the method used to establish the limits or uses customer-specified assessment criteria. For those test methods that have long lists of analytes, a subset of analytes is chosen. The analytes selected need to be representative of all analytes reported.

For organic analyses, the following criteria are used for determining the minimum number of analytes to be spiked; however, the NWQL ensures that all selected analytes are included in the spike mixture over a 2-year period:

- methods with 1 to 10 selected analytes: all analytes are spiked;
- methods with 11 to 20 selected analytes: at least 10 analytes or 80 percent of the total analytes are spiked, whichever is greater;
- methods with more than 20 selected analytes: at least 16 analytes or 60 percent of the total analytes are spiked, whichever is greater.

B.1.1.2.3 Surrogate Spikes

Surrogates most often are used in organic chromatography test methods and are chosen to represent the various chemistries of selected analytes in the method. Added prior to sample preparation/extraction, they provide a measure of recovery for each sample matrix. Often they are specified by the mandated method and are deliberately chosen because they are unlikely to be present as an environmental contaminant.

The number of surrogates used varies with each analytical method but generally is from one to four compounds. Typical surrogates are compounds that are isotopically labeled, fluorinated, or brominated. When used, surrogate compounds are added to all samples, standards, and blanks.

Results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the NWQL determines internal criteria and documents the method used to establish the limits. Surrogates outside the acceptance criteria must be evaluated for the effect indicated for the individual sample results.

Data-quality objective or other site-specific requirements may guide the appropriate corrective action. Results from analyses with surrogate recoveries outside the acceptance criteria must include appropriate data qualifiers, such as an "E." Surrogate data are provided to project personnel for their quality assurance (QA) use.

B.1.1.2.4 Certified Reference Materials

The LCS might consist of a commercially purchased medium containing verified concentrations of analytes known as standard or certified reference material.

In applicable methods, analyte recoveries are compared to criteria developed by the provider of the certified reference materials. Associated environmental sample data may be qualified, the samples re-prepared or re-analyzed, or both, according to specifications in the SOP.

B.1.1.2.5 Continuing Calibration Verification Standards

Continuing calibration verification (CCV) standards are solutions used in instrumental analysis to check instrument responses to the analytes (exceptions include multicomponent standards, such as toxaphene, or method-specific restrictions, such as fraction appropriate mixes) in relation to the calibration curve.

CCVs are prepared from the same materials and in the same manner as calibration standards. The concentration of the CCV is chosen to allow easy review by the analyst and typically is in the midrange of the calibration curve.

B.2 Quality Assurance

B.2.1 Field Blanks

Field blanks are submitted by field personnel using reagent-blank materials evaluated by the NWQL. Blanks may be used as QA samples to evaluate specific sources of contamination by field personnel. These include, but are not limited to, trip blanks, source solution blanks, equipment blanks, and field blanks.

B.2.2 Field Replicates

Typically, replicate samples are prepared on field sites by project personnel and submitted as QA samples by the project. In some cases, replicate samples are produced in the laboratory for use as laboratory QC samples where there is sufficient sample volume available (U.S. Geological Survey, 1994).

B.2.3 Matrix Spikes and Matrix Spike Duplicates

Sample-holding times, sample integrity, and sample storage space prohibit a routine practice of matrix spiking with given NWQL sample-handling practices. The NWQL does not require more than a single liter of water for analysis. For most organic analyses, the analytical methods require extraction of the entire sample. Therefore, no sample remains for preparing matrix spike samples. Matrix spiking may be requested on a custom basis. If such sample data are requested, a USGS Water Science Center customer needs to supply additional sample after making arrangements with the NWQL.

Field matrix spike samples also may be supplied by the customer if required by the project data-quality objectives (DQO). Matrix spikes and matrix spike duplicates indicate the effect of the sample matrix on the bias and variability of the results produced using the selected method. The information from these controls is matrix specific and normally would not be used to determine the validity of the entire batch. The frequency of the analysis of matrix-specific samples is

determined as part of a project-specified basis (for example, DQO) or as specified by the required method.

The components to be spiked are specified by the mandated test method or project DQOs. The results from the matrix spike/matrix spike duplicate primarily are used by oon-site personnel; they are designed to assess the bias and variability of analytical results in a given matrix and are expressed as percent recovery and relative percent difference.

B.2.4 Branch of Quality Systems Programs

The Branch of Quality Systems manages two projects to assess the NWQL that involve the use of SRSs or commercially purchased materials submitted blind to the NWQL. Data from these two programs are used to resolve technical corrective actions as described in Section 2.7.1, Technical Corrective Action.

The Inorganic Blind Sample Project primarily uses SRSs in various forms: unprocessed, diluted with deionized water, mixed in varying amounts with other standard water reference samples, or standards from commercial suppliers. This process produces a large number of unique samples for Branch of Quality Systems quality assessment of the NWQL and other laboratories used by the U.S. Geological Survey for inorganic analyses.

The Organic Blind Sample Project submits blind samples to the NWQL prepared from commercial sources; these blind samples are designed to assess the entire analytical range of most organic-analyte determinations. The primary purpose of these two projects is to produce an independent, third-party evaluation of the quality of data from the NWQL and most other laboratories that provide analytical water-quality results to the USGS.

B.3 Data Reduction, Review, Reporting, and Records

B.3.1 Data Reduction and Review

Sample analytical data are interpreted according to protocols described in the method SOPs. Available information used in the calculations (for example, unprocessed data, calibration files, tuning records, results of standard additions, sample response, and blank or background-correction protocols) is recorded to reconstruct the final result, if necessary.

Data are reviewed by a second analyst or supervisor (second-level review) to ensure that data interpretation is correct, QC is acceptable, and to detect transcription and dilution errors. Second-level reviews are performed by analysts or supervisors who performed the analyses or were trained in evaluating data on the system. The unit supervisor refers errors identified in the review process to individuals able to make the correction(s). These individuals vary with the error or problem detected, and may include the unit supervisor, senior personnel within Analytical Services or QAS, IT and BDT if the problem involves the LIMS, or the Administrative Section if the customer must be credited. Supervisors or designated senior personnel spot-check the second-level reviewers.

The results of all quality-control sample analyses are reviewed and evaluated before data are reported, as described in SOP QX0364.0, *Secondary data review at the National Water Quality Laboratory.*

B.3.2 Method Detection Limits and Levels

It is the policy of the NWQL to report the lowest possible concentrations of known quality to monitor environmental contamination of water. Most laboratory reporting levels (LRL) are based on long-term method detection levels (LT–MDL) designed to measure method variation over time. The LT–MDL and LRL data are available to analysts and data users (Childress and others, 1999). (Terms are defined in the Glossary, Appendix E.)

For establishing new analytical methods, initial method detection limits are determined by a method detection limit (MDL) study as described by the U.S. Environmental Protection Agency in its Appendix B (2005a). The spike concentration used to conduct the MDL study should be two to five times higher than the final MDL that is determined. This ideal spike

concentration often is difficult to estimate, especially for new instruments. The initial spike level can be set at two to five times the instrument detection limit (American Public Health Association, 1998). The MDL initially is determined for the analytes of interest in each method in ASTM Type I reagent water, scientific sand, or sodium sulfate, as appropriate for the method and matrix.

An MDL or LT–MDL study is not performed for any component for which spiking solutions are not available, such as total suspended solids, total dissolved solids, total volatile solids, pH, color, odor, temperature, dissolved oxygen, or turbidity. For these types of analytes, the MDL is the minimum reporting level (MRL). Because the definition of the MRL is not specific, an MRL can be set at a concentration acceptable to the data user and laboratory as long as reliable measurement is achieved (Childress and others, 1999).

B.3.3 Selectivity

Selectivity is the capability of a test method or instrument to respond to a selected compound or constituent in the presence of known or unknown substances.

1. Absolute and relative retention times help to identify components in chromatographic analyses.

2. A two-column confirmation is used in gas chromatographic analysis with nonspecific detectors. Mass spectrometers are used as specific detectors in other analyses. High-performance liquid chromatography determinations are confirmed with either mass spectrometers or by spectral photometers operating in the ultraviolet to visible range of wavelengths.

3. Acceptance criteria for mass-spectral tuning are contained in SOPs and published analytical methods.

4. Selectivity as specified in the method is assessed in method validation.

B.4 Constant and Consistent Test Conditions

The NWQL will install and operate instrumentation according to specifications included with manufacturers' documentation and recommendations. Such specification can include, but are not limited to, electrical power requirements, ventilation, gas flow and quality conditions, and temperature.

Glassware shall be cleaned to meet the sensitivity of the test method. Any cleaning and storage procedures that are not specified by the test method shall be documented in laboratory records and SOPs.

B.5 Biological Testing

Quality-control checks for processing benthic invertebrate samples are described in approved SOPs:

- BS0331.1, *Preparing benthic macroinvertebrate samples for processing*, accessible at URL http://wwwnwql.cr.usgs.gov/pub/.SOP/Word/Bio/biop0331.1.doc

- BS0332.1, *Qualitative visual sort method for processing benthic macroinvertebrate samples,* accessible at URL http://www nwql.cr.usgs.gov/pub/.SOP/Word/Bio/biob0332.1.doc

- BS0333.1, *Quantitative fixed-count method for processing benthic macroinvertebrate samples*, accessible at URL http://www nwql.cr.usgs.gov/pub/.SOP/Word/Bio/biob0333.1.doc

- BS0334.1, *Preparing benthic macroinvertebrate slides*, accessible at URL http://wwwnwql.cr.usgs.gov/pub/.SOP/Word/Bio/biop0334.1.doc

- BS0335.1, *Taxonomic identification of benthic macroinvertebrates*, accessible at URL http://wwwnwql.cr.usgs.gov/pub/.SOP/Word/Bio/biob0335.1.doc

Processing methods are described in Moulton and others (2000).

Interactions with taxonomic specialists at other organizations, including colleges, universities, and museums, are used to maintain accurate identification of taxa. A collection of confirmed specimens is maintained at the NWQL to ensure accuracy and consistency in identification among analysts.

APPENDIX C — Radon by Scintillation Counting

The analysis of radon is described in SOP RW0017.2, *Radiochemical analysis of radon-222, using the Beckman liquid scintillation system.*

C.1 Preparation of Blanks and Standards

Blanks are prepared with deionized water and scintillation cocktail.

A series of standards from near the minimum detectable concentration to above the usual sample levels is prepared with Ra-226 obtained from the USEPA, deionized water, and scintillation cocktail. The present range is 24 to 24,000 pCi/L.

New standards are analyzed and compared to the old standards before their acceptance and use as described in sections 4.2 (source materials) and 4.9.1 (verification of calibration standards) in NWQL SOP RX0413.0, *Preparation of standard materials for in-house radiochemical analysis*.

Both the blanks and standards are held in the dark for 30 days to allow for the decay of any residual radon in the water.

C.2 Analysis

Radon has a short half-life (3.82 days). Samples must be sent to the laboratory by overnight mail. They are placed in the counter the day they are received.

The two LS 5801 scintillation counters are calibrated with a tritium standard and the two LS 6000 counters are calibrated with a C-14 standard. Both standards are furnished by the manufacturer. These calibration checks are performed each day that samples are to be counted.

A blank and standard are counted followed by the samples. After every 10 samples a continuing calibration verification sample is analyzed. Blanks are used as background samples and are subtracted from the sample gross counts.

C.3 Calculation of Results

Results are corrected for decay back to the time of collection and reported with an accompanying counting uncertainty.

APPENDIX D — Code of Ethics

The U.S. Geological Survey (USGS) National Water Quality Laboratory (NWQL) follows the U.S. Department of the Interior (DOI) Code of Ethics. This document is part of the Departmental Management Series, Part 305, Departmental Science Efforts, Chapter 3: *Code of Scientific Conduct and Commentary* (draft, May 6, 2003). The full text of the draft is included in this appendix (U.S. Department of the Interior, 2003).

The DOI Code is followed by the USGS NWQL's Code of Ethics. This document is available for downloading on the NWQL intranet web site at URL http://wwwnwql.cr.usgs.gov/nwql/nwql_emp_coe.doc and is also reproduced in this appendix.

The USGS maintains an online Ethics Office, which is accessible on its employee intranet at http://internal.usgs.gov/ops/hro/ethics. Ethics regulations and statutes that govern the conduct of USGS employees is accessible at http://internal.usgs.gov/ops/hro/ethics/regulationstatutes.html.

Department of the Interior Departmental Manual

Series: Departmental Management
Part 305: Departmental Science Efforts
Chapter 3: Code of Scientific Conduct and Commentary (draft)
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305 DM 3

3.1 Purpose

This chapter establishes a uniform code and policy governing the conduct of science for the U.S. Department of the Interior (Department). The Code (see Appendix) describes ethical conduct for all Department employees who are engaged in conducting scientific activities on behalf of the Department and who, by virtue of those activities, are subject to Federal Policy on Research Misconduct. A Commentary (Appendix) explains and clarifies the basic elements of the Code. The Commentary is incorporated into the Code by reference.

3.2 Authorities

A. 5 U.S.C. §301 allows the head of an executive department to prescribe regulations for the conduct of its employees. 43 C.F.R. §20.501 requires that employees of the Department comply with all Federal statutes, Executive Orders, Office of Government Ethics and Office of Personnel Management regulations, and Departmental regulations. 43 C.F.R. 20.502 states that employees are required to carry out the announced policies and programs of the Department. 43 C.F.R. §20.502(a) provides that an employee is subject to appropriate disciplinary action if he or she fails to comply with any lawful regulations, orders or policies.

B. Federal Policy on Research Misconduct, 65 *Federal Register* (FR) 76260-76264, December 6, 2000.

C. "Standards of Official Conduct," President's Memorandum for the Heads of Executive Departments and Agencies, dated January 20, 2001 (can be found at <http://www.doi.gov/ethics>).

3.3 Goal

The Code is intended to establish a clear understanding of Departmental expectations and requirements regarding the conduct of scientific activities on its behalf. It is also intended to help employees meet the Secretary's stated policy to make decisions based on the best science available, and to conduct all scientific activities and information gathering with honesty, accuracy and integrity.

3.4 Policy

The Department is dedicated to preserving the integrity of scientific activities conducted on its behalf. The Department does not tolerate misconduct in the execution of scientific activities. The Department will take appropriate action to protect the public from the effects of inappropriate or inaccurate information

produced through scientific activities (see Information Quality Guidelines, <http://www.doi.gov/ocio/guidelines/515Guides.pdf>), and will investigate all allegations of such misconduct in a manner that assures, to the maximum extent of the law, the rights and privacy of any party against whom allegations have been made. The Department will take disciplinary action, up to and including removal from the federal service, against employees for noncompliance with this chapter, in accordance with Departmental personnel policies and the Department's Handbook on Charges and Penalty Selection for Disciplinary and Adverse Actions.

Specifically, this chapter directs that:

A. Employees must comply with the Code, which is attached as an Appendix.

B. Employees must comply with the Federal Policy on Research Misconduct, published at 65 FR 76260-76264 (December 6, 2000). The Federal Policy on Research Misconduct includes a definition of research misconduct, which is as follows:

"Research misconduct is defined as fabrication, falsification, or plagiarism in proposing, performing, or reviewing research, or in reporting research results.

- Fabrication is making up data or results and recording or reporting them.
- Falsification is manipulating research materials, equipment, or processes, or changing or omitting data or results such that the research is not accurately represented in the research record.
- Plagiarism is the appropriation of another person's ideas, processes, results, or words without giving appropriate credit.
- Research misconduct does not include honest error or differences of opinion."

C. Employees are required to comply with the Federal Policy for the Protection of Human Subjects, published at 56 FR 28012-28018 (June 18, 1991).

D. Employees are required to comply with the President's Memorandum for the Heads of Executive Departments and Agencies, dated January 20, 2001, entitled "Standards of Official Conduct."

E. In addition to the above policy statements, employees are reminded that they are required by 43 C.F.R. §20.501 and §20.502 to comply with all Federal statutes, Executive Orders, Presidential Memoranda, Office of Government Ethics and Office of Personnel Management regulations, and Departmental regulations and policies.

3.5 Scope

The policy adopted in this chapter applies specifically to the conduct of scientific activities. For purposes of this chapter, "scientific activities" has the same meaning as that in the definitions section of the Commentary in the Appendix. Administrative rules and laws pertaining to activities such as falsification of government documents, sexual harassment, civil rights, gifts, nepotism, disclosure of financial interest, conflict of interest or outside employment are neither altered nor diminished in importance by the existence of this Code.

A. The policy adopted in this chapter applies to all employees of the Department who engage in scientific activities and related work. Examples of employees to whom this policy applies include individuals who conduct scientific activities; professional and technical staff and their support staff; administrators and managers of scientific activities within the bureaus and offices of the Department; and individuals who compile and translate scientific information into documents used in the Department's decision processes.

B. Requirements for contractors have been developed and will be issued in final form at 48 CFR 1435.70 and 48 CFR 1452.235-1 after reconciliation of public comments. Similar requirements have been drafted to apply to other non-federal employees who do similar work on behalf of the Department. These requirements will be issued in the Code of Federal Regulations after reconciliation of public comments.

3.6 Process for Handling Allegations Of Misconduct for Violations of the Code Adopted by this Chapter

3.6.A. Employees

1. Findings of Misconduct in the Execution of Scientific Activities (Misconduct)

A finding of misconduct requires that:

- There is a significant departure from accepted practices of the scientific community for maintaining the integrity of the scientific/research record;
- The misconduct is committed intentionally, or knowingly, or in reckless disregard of accepted practices; and
- The allegation is established by a preponderance of evidence.

Each bureau and office shall establish procedures to inquire into and investigate allegations or suspicions of misconduct. These procedures are to be consistent with Federal Policy on Research Misconduct published at 65 FR 76260-76264 (December 6, 2000), Departmental policy, and the Department's Handbook on Charges and Penalty Selection for Disciplinary and Adverse Actions (Handbook).

2. Process

Disciplinary action for confirmed misconduct by employees of the Department of the Interior will be administered in accordance with Departmental personnel policies and the Handbook. Supervisors generally should attempt to select the penalty they believe necessary to correct the misconduct and to discourage repetition.

In a case where there is a serious breach of Code, as well as repeated violations of a less serious nature, supervisors may propose and decide on more serious penalties, up to and including removal from the federal service. In any situation where a supervisor is considering a formal disciplinary action, the supervisor should consult with his or her Human Resources office immediately. All phases of the response of each Bureau and Office to an allegation of misconduct, from inquiry to confirmation to adjudication and appeal, shall be consistent with the guidelines and principles in the Federal Policy on Research Misconduct (65 FR 76260-76264, December 6, 2000), Departmental policy, and the Handbook.

3.6.B. Contractors

The policy that has been developed for contractors sets out a process for handling allegations of violations that will be issued in final form at 48 CFR 1435.70 and 48 CFR 1452.235-1 after reconciliation of public comments. The policy that has been developed for other non-federal employees sets out a process for handling allegations of violations of the policy, and will be issued in the Code of Federal Regulations after reconciliation of public comments.

3.7 Legal Effects

This Code is intended to improve the internal management of the Department of the Interior. This Code and Commentary are not intended to, and do not, create any right or benefit, substantive or procedural, enforceable at law by any person against the United States, its agencies, its officers or employees, or any other person.

APPENDIX THE CODE OF SCIENTIFIC CONDUCT

To the best of my ability:

I will act in the interest of the advancement of science and contribute the best, highest quality scientific information for the Department of the Interior.

I will conduct, manage, judge, report, and communicate scientific activities and information honestly, thoroughly and without conflict of interest.

I will be responsible for the resources entrusted to me, including equipment, employees' time, and funds. I will be accountable for the prompt and accurate collection, use, and reporting of all financial resources and transactions under my control.

I will disclose the research methods to the local communities, Indian tribes, and other individuals whose interest and resource uses are studied; and respect the confidential and proprietary information provided by those individuals to the fullest extent permitted by law.

I will neither hinder the scientific and information gathering activities of others nor engage in dishonesty, fraud, deceit, misrepresentation, or other scientific, research or professional misconduct.

I will welcome constructive criticism of my scientific activities and information, participate in appropriate peer reviews, and critique others' work in a respectful manner amid objective scientific review.

I will be diligent in the creation, use, preservation, and maintenance of collections and data records; adhere to established quality assurance and quality control programs; follow the records retention policies of the Department; and comply with Federal law and established agreements related to the use, security, and release of confidential and proprietary data.

I will know, understand and adhere to standards of public information dissemination and the formal publication of scientific information and respect the intellectual property rights of others.

I will be responsible in all scientific activities for both the collection and interpretation of data I collect and the integrity of conclusions I present.

I will place quality and objectivity of scientific activities and information ahead of personal gain or allegiance to individuals or organizations.

Commentary to Explain and Clarify the Intent of the Basic Elements of the Code

A. Scientific excellence, integrity, and conflict of interest

Scientific honesty and integrity of Department employees are vital to the public interest and critical to conducting the Department's mission. Scientific activities provide data to inform many of the Department's decisions regarding the stewardship of our Nation's land, water and cultural resources. Employees subject to the Code must avoid conflicts of interest which occur when an individual's personal interest or gain interferes with the objectivity of his/her actions or judgments. They are obligated to be thorough in documenting their work to ensure that the details of their methods are described adequately enough to allow other scientists to critically evaluate or reproduce their results. They will use the best available and practicable practices, protocols, methodologies and technologies when conducting scientific activities as well as in the use and dissemination of scientific information.

This Code and Commentary do not suggest that it is unethical to use novel investigative approaches, employ unusual methods of analysis, exclude data points known to be faulty for identifiable material reasons, or interpret data in a new or unique way. However, novel methods and data modifications should be fully documented in the research record to avoid misinterpretation of any such departure from standard protocols or methodology.

B. Abuse of resources

Department employees will ensure appropriate use of resources in the conduct of scientific activities, including equipment, funding, staff time, and any privately owned or Federal property. Most importantly, employees will use resources wisely, efficiently, respectfully, and prevent abuse of cultural and natural resources (*e.g.*, archeological sites, historic structures, cultural landscapes, museum property, ethnographic resources and animals) during the conduct of scientific activities.

Employees must strive to select methods and materials that, to the best of current knowledge, do not adversely affect cultural resources or their future examination, scientific investigation, treatment or function. Animals used for research purposes are public resources, and employees will obey public laws concerning treatment of research animals. Public law (P.L. 99-198), The Food Security Act of 1985, and Federal regulation (9 C.F.R. Part 3) primarily apply to treatment of laboratory animals. Much Department research, however, involves animals in the wild; therefore, researchers should follow public law (including P.L. 99-198 as applicable) and regulations and consider, where appropriate, guidelines regarding treatment of wild animals published by professional wildlife or scientific societies.

C. Research involving human subjects

1. Department employees conduct scientific activities among groups, including but not limited to hikers and campers in National Parks, present-day ethnic or occupational communities, and Indian tribes to meet compliance concerns for planning, the Native American Graves Protection and Repatriation Act of 1990 (P.L. 101-601), historic preservation, and subsistence uses. Human subjects involved in scientific activities must be treated with professionalism and respect. To this end, the Department adopts the common rule published as Federal Policy for the Protection of Human Subjects (56 FR 28012-28018, June, 18, 1991).

Exempted from this rule is human subjects research involving the use of educational tests, survey procedures, interview procedures or observation of public behavior unless the information obtained is recorded in such a manner that human subjects can be identified, and/or disclosure could place subjects at risk of criminal or civil liability. Many information collections are regulated under the Paperwork Reduction Act of 1995 (5 CFR 1320), and Departmental procedures should be followed (381 DM 11, 12). Exempted from this rule is observation of human public behavior that involves no data collection from subjects.

2. Before initiating new scientific activities with any group, Department employees should be familiar with the laws, regulations, and policies (including those that are bureau-specific) governing privacy and freedom of information, ethnographic research guidelines, and types of release and consent forms, as provided information might not be protected from release.

Department employees will ensure that the research methods are made clear to participants, that permission is obtained to use interview materials, tapes, photographs, maps and any other materials, and that participants know the legal limits of confidentiality. Professional standards for non-invasive or non-destructive testing/sampling will be followed when studying cultural materials.

D. Hindering research and information-gathering activities; protection of proprietary and confidential information; engaging in dishonesty, fraud, deceit, misrepresentation; or other scientific, research, or professional misconduct

1. The scientific research of other employees is subject to being hindered by actions such as biased review of scientific proposals or manuscripts; physical disruption of another scientist's experiments, field surveys, or database; denial of reasonable access to resources or data needed by other scientists to perform their work; or failure to provide information that others need to duplicate scientific activities or verify their accuracy. Engaging in these activities is not tolerated by the Department.

Scientific staff will allow management and other employees appropriate access to research resources entrusted to them, unless doing so would compromise the scientific validity of their activities or substantially interfere with their performance. Employees are expected to understand existing rules and guidelines regarding the need to make data gathered with federal dollars accessible.

Reasonable judgments to delay public access depend upon individual circumstances when premature release would compromise validity or decision-making ability. Specifically, this applies to work in progress where data have not gone through a planned quality control/quality assurance protocol that is part of the research design. Therefore, this Code and Commentary do not attempt to provide specific guidelines for making such determinations.

2. Requirements related to use, security and release of proprietary data are sometimes covered by law, regulation or policy and may be established through an agreement with the originator of the data. These agreements are usually established on a case by case basis. Employees will adhere to these agreements to the extent permitted by law, or policy. It is clear, nonetheless, that the Code and Commentary prohibit denying other scientists reasonable access to published scientific information for the purpose of enhancing one's interests. Falsification and fabrication of data and results are not tolerated by the Department.

E. Participating in review processes and offering fair and unbiased opinions

Peer review is an important element in the range of management controls for the creation and use of scientific information. In some cases, external (to the Department) scientific review of scientific activities, information, inventory or monitoring data to be published or used in decision-making is essential. All employees must know, understand and adhere to Departmental guidelines (305 DM 4, in preparation) related to peer review of scientific activities. Open and honest debate is essential for the advancement of science, and peer review is an important part of that debate.

The peer-review process should be free of personal and professional jealousies, competitions, disagreements, and conflicts of interest. Reviewers should focus on the logical and scientific validity of the research findings, rather than personal feelings, or past or current interactions between the reviewer and the author/investigator. Authors/investigators should address reviewers' comments in a thorough manner, and should document appropriately how they responded to those comments. It is the responsibility of prospective reviewers to disqualify themselves, if the review cannot be done in an unbiased manner (5 C.F.R. 2635.502).

F. Integrity in the collection and preservation of data

1. Quality control and assurance, including protocols, standards, and methodologies, should be routinely established for activities pertaining to the conduct of scientific inquiry and the collection of data. Persons engaged in scientific activities and their managers must know and follow established programs, protocols, standards, and methodologies for the activities they conduct to inform Departmental decisions.

Preservation of collections and records created during the conduct of scientific activities is controlled by Federal law (44 U.S.C. chs.21, 29, 31, and 33) and Departmental regulations and policies (36 CFR §1228.1-1228.282; 381 DM 11, 12; 384 DM 2, 3, and 4). This is important for substantiating scientific activities and supporting subsequent decisions that are influenced by the results. Employees subject to this Code must follow these laws, regulations and policies. Collections made for retention include, but are not limited to, cultural objects in archeological collections and non-cultural research samples in geological samples and paleontological samples.

2. Documents that should be retained for the scientific record vary according to the nature of the study and include: study plans; primary data, such as laboratory notebooks, original data, metadata, and quality assurance/quality control information; and formal data sets and products. These items may be in any media, including printed and electronic media. Failure to retain data in accordance with law, regulations and policy is not tolerated by the Department.

G. Responsible authorship and dissemination of information

1. Authorship of a scientific product must be based on a major intellectual contribution (as part of conception, design, data collection, data analysis, or interpretation) *and* a significant contribution to its preparation (writing, reviewing, or editing). Authorship includes the responsibility for ensuring that the work reported meets scientific criteria and ethical standards.

2. Scientific knowledge is cumulative and is built on the contributions of numerous scientists over many years. Recognition of other contributors often takes the form of credits in a publication through an acknowledgment, citation, or co-authorship. Authors will cite or acknowledge any scientific work that substantially contributes to a scientific activity and its interpretation and result.

The Code prohibits plagiarism or theft of ideas, data, or unpublished findings. Plagiarism is research misconduct and is not tolerated by the Department. Departmental employees subject to this Code will acknowledge and, to the extent permitted by law, protect the intellectual efforts of others and the confidentiality of information provided by human subjects. When these considerations conflict with the regulations and guidance of the Council on Environmental Quality regarding the publication of information under the National Environmental Policy Act (NEPA), the regulations and guidance of the Council shall govern. Department employees preparing NEPA documents are covered by all other provisions of this Code.

3. The Code prohibits duplicative publication. Duplicative publication is not tolerated by the Department. This does not suggest that it is inappropriate to publish more than one manuscript based on a single scientific activity. In some cases, the same scientific activity may be of interest to separate audiences having different technical specialties or to journals having different readerships. Prior publication of portions of an original idea should always be referenced in later publications. Employees will accept professional responsibility associated with authorship and know that the interpretation and results of their work are used to inform important decisions in the public interest.

4. In order to support the Department's interest in providing for its decision making to be based on the best available science, the Code requires a scientific product to be subject to appropriate level of peer review. Public release of a scientific product without the appropriate level of peer review or without the inclusion of appropriate disclaimers could be considered misconduct.

5. Additionally, in support of the Department's interest in protecting its decision making, the Code prohibits changing conclusions, deletion of data, or knowingly omitting data from reports prepared for decision makers for purposes of misrepresentation or manipulation. These actions are not tolerated by the Department. They are violations of the Federal Policy on Research Misconduct because they wrongly characterize results and manipulate results so that research is not accurately represented. Scientific conclusions may only be changed in light of new data or new analyses. Additionally, scientists should not succumb to coercion to change data. Pressure or coercion to do so should be reported immediately to the respective supervisor or bureau ethics official.

DEFINITIONS

For purposes of this Code, the following definitions apply:

Conflict of Interest. A situation in which an individual's personal interest interferes with or could be construed to interfere with the objectivity of the individual's actions or judgments when conducting scientific activities. For purposes of this definition, the personal interest may include those of the employee's spouse and minor child. Possible conflict of interest situations may include those where actions or judgments are affected by opportunities for career advancement, concern for professional prestige, the influences of personal allegiances or animosities, or the pursuit of pecuniary gain.

Duplicative Publication. Republishing an original manuscript. Prior publication should always be referenced in later publications. Simultaneous publication of results, such as in a conference proceedings and a journal article, is not duplicative publication if both outlets and the author(s) have mutually agreed to the simultaneous publication of the results.

Research Misconduct (from the Federal Policy on Research Misconduct, 65 FR 76260, December 6, 2000). Fabrication, falsification, or plagiarism in proposing, performing, or reviewing research, or in reporting research results.

- Fabrication is making up data or results and recording or reporting them.
- Falsification is manipulating research materials, equipment, or processes, or changing or omitting data or results such that the research is not accurately represented in the research record.
- Plagiarism is the appropriation of another person's ideas, processes, results, or words without giving appropriate credit.
- Research misconduct does not include honest errors or differences of opinion.

Research Record. The record of data or results that embody the facts resulting from scientific inquiry, and includes, but is not limited to, research proposals, laboratory records, both physical and electronic progress reports, samples or other physical materials, abstracts, theses, internal reports, and journal articles.

Science. Knowledge obtained and tested through use of the scientific method. May also include the observation and classification of facts with the goal of establishing verifiable knowledge derived through induction and hypothesis.

Scientific Activities. Activities involving inventorying, monitoring, study, research, adaptive management or assessment that are conducted in a manner specified by standard protocols and procedures involving any of the physical, biological or social sciences, cultural resources scholarship, engineering or mathematics. Inspections for regulatory compliance and resulting records are not included because they are covered by separate requirements.

Scientific Product. A scientific product presents the results of scientific activities.

Scientific Record. The record of data or results that embody the information resulting from scientific inquiry, including but not limited to, research proposals, study plans, collections, samples and other physical materials, field and laboratory records, photographs, maps, progress reports, abstracts, theses and dissertations, oral presentations, internal reports, journal articles, and web sites. The scientific record includes all primary data, formal data sets, and published results. These may be in any media, including print and digital media.

U.S. Geological Survey National Water Quality Laboratory Employee Code of Ethics, 6 November 2003 (supplement to the U.S. Department of the Interior Code of Ethics)

The employees of the National Water Quality Laboratory are committed to ensuring the integrity of our data and meeting the quality needs of our customers. As an NWQL employee, I will strive to

- produce results that are technically sound and scientifically defensible;
- accurately and honestly represent the laboratory's capability for requested services;
- present services in an appropriate, honest, and forthright manner, with appropriate confidentiality;
- gain a clear and mutual understanding with our customers as to the extent and kind of services to be rendered;
- operate our facilities in a manner that protects the environment and the health and safety of

employees and the public, obeying all pertinent Federal, State, and local laws, and regulations;

- maintain high product and service quality;
- treat co-workers equitably, acknowledge their workplace contributions, and encourage opportunities for professional growth and development;
- recognize and respond to community concerns;
- deal openly, honestly, and fairly in all scientific, business, and financial matters with co-workers, customers, and the public;
- respect diversity in the workplace;
- present and promote the USGS and NWQL in a positive light; and
- follow the given ethical guidelines and seek guidance to resolve ethical issues.

APPENDIX E — Glossary

acceptance criteria — specified limits placed on characteristics of an item, process, or service defined in requirement documents (National Environmental Laboratory Accreditation Conference, 2003b)

accreditation — the process by which an agency or organization evaluates and recognizes a program of study or an institution as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one (National Environmental Laboratory Accreditation Conference, 2003b).

accuracy — the degree of agreement of a measured value with the true or expected value of the quantity of concern (Taylor, 1987); see "bias"

amendment report — a document used to update or revise the Quality Management System or a standard operating procedure (SOP) at the National Water Quality Laboratory

analyte — a substance being determined in an analysis

analytical services request (ASR) — an order form for analytical services that allows customers and the U.S. Geological Survey National Water Quality Laboratory to identify samples and processing, and keep a record of analytical requests and any comments from the customer. An electronic version (E-ASR) also is available.

ASTM Type I reagent water — Type I grade of reagent water is prepared by distillation or other equal process, followed by polishing with a mixed bed of ion exchange materials and a 0.2-µm membrane filter. It should have a minimum resistance of 18.0 MΩ-cm at 25 ºC (American Society for Testing and Materials, 2001, p. 107).

attachment — a secondary document that is attached to and supports the procedures presented in the standard operating procedure (SOP). An attachment provides supplementary information that is not included within the body of the SOP but is necessary to correctly perform the procedure or clarify statements made within the SOP. Attachments must be referenced within the text of the SOP. Attachments go through the same review procedures as the rest of the SOP and are part of the SOP. Examples of attachments are manifold diagrams, computer instructions, acceptance ranges for specific quality-control samples, forms required to complete a procedure, and formatting instructions. Attachments must be posted on the NWQL intranet web site with the corresponding SOP.

audit — a systematic evaluation to determine the conformance to quantitative specifications of some operational function or activity (National Environmental Laboratory Accreditation Conference, 2003b)

backlog — not the preferred term; see “sample inventory”

bias — systematic error inherent in a method or measurement system. The error can be positive [for example, contamination or spectral interference] or negative [for example, analyte loss or signal suppression] (Taylor, 1987). It differs from random error, which shows no such consistent or systematic deviation. Bias is the preferred term used by the NWQL and USGS Branch of Quality Systems.

blank — a sample that has not been exposed to the analyzed sample stream to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results (National Environmental Laboratory Accreditation Conference, 2003b).

blind sample — a subsample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst’s or laboratory’s proficiency in the execution of the measurement process (National Environmental Laboratory Accreditation Conference, 2003b).

calibrate — to check, adjust, or determine by comparison to a standard, accessed October 31, 2005 at URL http://www.thefreedictionary.com/calibrate

calibration — the set of operations that establishes, under specified conditions, the relation between values or quantities indicated by a measuring instrument or system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. In calibration of support equipment, the values realized by standards are established through the use of reference standards traceable to the International System of Units.

In calibration according to test methods, values realized by standards are typically established using refer-

ence materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using equipment calibrated or verified to meet specifications (National Environmental Laboratory Accreditation Conference, 2003b).

calibration curve — the graphic relation between the known values, such as concentrations, of a series of calibration standards and their analytical response (National Environmental Laboratory Accreditation Conference, 2003b)

calibration standard — a solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The calibration solutions are used to calibrate the instrument response with respect to analyte concentration (National Environmental Laboratory Accreditation Conference, 2003b, in part). Fig. 3.1 (p. 3.20 in the text) uses LS as an abbreviation to identify the lowest calibration standard.

certified reference material (CRM) — a reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation that is issued by a certifying body (National Environmental Laboratory Accreditation Conference, 2003b)

chain of custody (COC) — an unbroken trail of accountability that ensures the physical security of samples, data, and records. May also be referred to as legal chain of custody. A chain-of-custody form is a record that documents the possession of the samples from the time of collection to receipt in the laboratory and generally includes the number and types of containers, the mode of collection, collector, time of collection, preservation, and requested analyses ((National Environmental Laboratory Accreditation Conference, 2003b).

checksum — an error-detection process in which each transmitted message is accompanied by a numerical value based on the number of bits in the message. The receiving station then applies the same formula to the message and verifies the accompanying numerical value is the same. The transmission protocol applies an additional checksum to the data containing the U.S. Geological Survey National Water Quality Laboratory-produced checksum.

conformance — an affirmative indication or judgement that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements (National Environmental Laboratory Accreditation Conference, 2003b)

continuing calibration verification standard (CCV) — a standard solution used in instrumental analysis to check instrument stability in relation to the calibration standard curve. Prepared from the same materials in the same manner as the calibration standards. Concentration of the CCV should be chosen to allow easy review by the analyst and is typically in the midrange of the calibration curve.

controlled document — a document that is maintained and updated. Controlled documents are formally approved and their distribution is traceable to enable changes to be made. A controlled document meets one or more of the following criteria (U.S. Department of Energy, 2003):

- is prepared, reviewed, modified, and approved using established implementing documents, and is subject to controlled distribution and a defined process for change;
- users require a copy of the current version of a controlled document at the site where the work is to be performed and must perform the work in accordance with the controlled document;
- could adversely affect project or program activities if used in its noncurrent version;
- specifies technical or quality requirements or prescribes activities affecting quality (that is, implementing documents); or
- a supervisor or chief has requested it to be controlled.

control limit — statistically derived values used to get acceptable ranges for quality-control samples analyzed in conjunction with environmental samples

corrective action — action taken to eliminate the cause(s) of an existing nonconformity, defect, or other undesirable situation to prevent recurrence (National Environmental Laboratory Accreditation Conference, 2003b)

custom analysis — a term applied to any analysis that requires a proposal. This includes modifications to an approved, operating U.S. Geological Survey National Water Quality Laboratory (NWQL) method or implementation of a non-NWQL approved method. Also included are analyses using methods that have not been completely evaluated applying USGS guidelines for developing approved methods and for which the validation has not been completed. Proposals are required for all nonroutine analyses, including new method development, method modifications, and other projects requiring time and resources of NWQL staff beyond what is routinely available.

data — a carrier of information and knowledge, having no real meaning, and lacking any relational connection or interpretation. It can exist in any form, and need not be usable.

data integrity — the ability to maintain and/or preserve a piece of data or information, and to recreate a piece of data or information should accidental loss occur

data packet — a compilation of analytical data needed to reconstruct an analysis. Contents of the data packet are based upon the standard operating procedure (SOP) for the specific method and may include calibration information, analytical sequence/sample pour-up protocol, instrumental method, quality-control summaries, unprocessed (area counts and signal readings) and processed data for reagent spikes, reagent blanks, continuing calibration verification (CCV) standards, standard reference materials (SRM) , and certified reference materials (CRM).

data reduction — the process of transforming unprocessed data by arithmetic or statistical calculations, standard curves, concentration factors, and collation into a more usable form (National Environmental Laboratory Accreditation Conference, 2003b)

data release — the digital or other product release of approved data from the U.S. Geological Survey National Water Quality Laboratory to the customer's site or address. These locations are usually pre-defined and/or found with the "analytical services request" form.

data review — the process of validating and/or approving data after analysis or processing. Once approved the data are flagged as ready to be released to the customer.

demonstration of capability — a procedure to establish an analyst's ability to perform a test with acceptable accuracy (National Environmental Laboratory Accredication Conference, 2003b)

detection limit — see "method detection limit"

dissolved — refers to constituents that exist in true chemical solution in a water sample; a convenient operational definition used by agencies that collect water data, the term "dissolved" commonly refers to constituents in a representative water sample passed through a 0.45-micrometer (µm) filter membrane for inorganic analysis or a 0.7-µm glass fiber filter for organic analysis (Franceska Wilde, U.S. Geological Survey, written commun., 1995)

document control — the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity is performed (National Environmental Laboratory Accreditation Conference, 2003b)

double-blind sample — a sample submitted to evaluate performance with concentration and identity unknown to the analyst

field blank — blank prepared on-site by filling a clean container with deionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (National Environmental Laboratory Accreditation Conference, 2003b)

filtered — pertains to constituents in a water sample passed through a filter membrane of specified pore diameter, most commonly 0.45 micrometer (µm) or less for inorganic analytes and 0.7 µm for organic analytes (Franceska Wilde, U.S. Geological Survey, written commun., 1995)

F-pseudosigma (F_σ) — a nonparametric, resistant measure of data spread defined as the interquartile range of the data divided by 1.349 (Hoaglin and others, 1983). F_σ and the standard deviation of the data will be nearly equivalent if the data have a near-normal distribution.

good automated laboratory practices (GALP) — principles and practices to ensure data integrity in automated laboratory operations; see *Good automated laboratory practices: Principles and guidance to regulations for ensuring data integrity in automated laboratory operations with implementation guidance*, accessed October 31, 2005, at URL http://www.epa.gov/irmpoli8/pdfs/2185galp.pdf

good laboratory practices (GLP) — general guidelines or formal regulations for performing basic laboratory operations or activities that are known or believed to influence the quality and integrity of the results; see *Good laboratory practice standards*, title 40, pt.160, accessed October 31, 2005, at URL http://ecfr.gpoaccess.gov/cgi/t/text/text-idx?c=ecfr&sid=20d03188867b0e2ecf824e19133e3c1e&tpl=/ecfrbrowse/Title40/40cfr160_main_02.tpl

hazardous samples — samples considered to contain high concentrations of contaminants and may have deleterious effects on human health or the environment

hazardous waste — a solid, liquid, or contained gaseous material that is no longer used or that no longer serves the purpose for which it was produced and could pose dangers to human health and the environment after it is discarded (Colorado Department of Public Health and Environment, 1995)

holding time (maximum allowable holding time) — the maximum time(s) that a sample may be held prior to analysis and its analytical results still be considered valid (U.S. Environmental Protection Agency, 2005a)

information-rich methods — classified as organic methods that use either mass spectrometric or photodiode array ultraviolet/visible spectroscopic detection. These methods have additional qualifying information that allows enhanced analyte identification (Childress and others, 1999).

initial demonstration of analytical capability — a procedure to establish the ability of the laboratory to produce acceptable bias and variability, which is included in many of the USEPA's analytical test methods. In general the procedure includes the addition of a specified concentration of each analyte (using a QC check sample) in each of four separate aliquots of laboratory pure water. These are carried through the entire analytical procedure and the percentage recovery and the standard deviation are determined and compared to specified limits (in part from U.S. Environmental Protection Agency, 2005a).

instrument detection limit (IDL) — the concentration equivalent to a signal from an analyte of interest, which is the smallest signal that a particular instrument can distinguish from background noise. The IDL may be used for statistical data analysis and comparing the attributes of different instruments (American Public Health Association, 1998). It is determined on samples that have not gone through any sample preparation steps (West Coast Analytical Services' web site, accessed 8 November 2005, at URL http://www.wcaslab.com/TECH/DETLIM.htm.

interim reporting level (IRL) — a temporary reporting level used for new or custom methods when long-term method-detection-level data are unavailable and a laboratory reporting level has not yet been established (U.S. Geological Survey, 2003b)

internal standard — a known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the bias and variability of the applied analytical test method (in part from National Environmental Laboratory Accreditation Conference, 2003b)

laboratory — a body or organization that calibrates and/or tests (National Environmental Laboratory Accreditation Conference, 2003b)

laboratory code (LC) — a 1- to 4-digit code in the NWQL services catalog, always preceded by the letters "LC," that uniquely represents a variable determined by a particular method of chemical analysis. For example, iron by atomic absorption and iron by inductively coupled plasma have different LCs. The NWQL services catalog is available on the USGS-visible intranet web site at URL http://nwql.cr.usgs.gov/usgs/catalog/index.cfm

laboratory control sample (LCS) (however named, such as laboratory fortified blank or spiked blank) — a sample matrix free from the analytes of interest spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes. It is generally used to establish intralaboratory or analyst-specific bias and variability or to assess the performance of all or a part of the measurement system (National Environmental Laboratory Accreditation Conference, 2003b).

laboratory information management system (LIMS) — a centralized data management and storage system that maintains data security, and data and information integrity. The system gathers or receives data from other analytical systems, and enforces quality-control measures, operating procedures, and business rules to process information and ensure data of known quality.

laboratory records technician — the individual responsible for the life cycle of data management, including accessioning, disposition, reference, retrieval, arrangement, description, records appraisal, and records preservation

laboratory reporting level (LRL) — equal to twice or more the annually determined long-term method detection level (LT–MDL). The LRL controls false negative error. The probability of falsely reporting a nondetection for a sample that contained an analyte at a concentration equal to or greater than the LRL is predicted to be no more than 1 percent. The LRL concentration will be reported with a "less than" (<) remark code for samples in which the analyte was not detected.

The National Water Quality Laboratory (NWQL) collects quality-control data from selected analytical methods on a continuing basis to determine LT–MDLs and establish LRLs. These LT–MDLs and LRLs are reevaluated

annually on the basis of the most current quality-control data, and may, therefore, change (Childress and others, 1999).

Note: In several previous U.S. Geological Survey NWQL documents (Connor and others, 1998; U.S. Geological Survey NWQL Technical Memorandum 98.07, 1998c), the LRL was called the nondetection value or NDV—a term that is no longer used.

long-term method detection level (LT–MDL) — a detection level derived by determining the standard deviation (or F-pseudosigma) of a minimum of 24 method detection limit (MDL) spike-sample measurements over an extended period. LT–MDL data are collected continually to assess year-to-year variations in the LT–MDL. The LT–MDL controls false positive error. The chance of falsely reporting a concentration at or greater than the LT–MDL for a sample that did not contain the analyte is predicted to be less than or equal to 1 percent (Childress and others, 1999).

marginal exceedance (ME) — laboratory control sample (LCS) data that are beyond the LCS control limit of three standard deviations around the mean, but within the ME limits of three and four standard deviations around the mean. If a large number of analytes is in the LCS, it is statistically likely that a few will be outside control limits. This may not indicate that the system is out of control and corrective action may not be necessary. Upper and lower ME limits can be established to determine when corrective action is necessary (National Environmental Laboratory Accreditation Conference, 2003a).

matrix — the component or substrate that contains the analyte of interest; the substrate of a test sample. For purposes of batch and QC requirements determination, the following matrix distinctions shall be used: aqueous—any aqueous sample excluded from the definition of drinking water (National Environmental Laboratory Accreditation Conference, 2003b).

matrix spike (spiked sample, fortified sample) — prepared by adding a known mass of specified analyte to a specified amount of matrix sample for which an independent estimate of specified analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency (National Environmental Laboratory Accreditation Conference, 2003b).

matrix spike duplicate (spiked sample/fortified sample duplicate) — a second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte (National Environmental Laboratory Accreditation Conference, 2003b)

matrix spike solution — a solution of known composition added to actual samples of water to monitor effects of sample storage and shipment and the bias and variability of laboratory analysis

method — a way to perform an analysis that has been peer-reviewed, approved, and published or standardized that may produce data for one to many analytes; see "test method"

method blank — a clean sample (matrix similar to the batch of associated samples and is free of the analyte of interest) processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the analytical procedures, and iin which no target analytes or interferences are present at concentrations that affect the analytical results for sample analyses (National Environmental Laboratory Accreditation Conference, 2003b)

method detection limit (MDL) — the minimum concentration of a substance (an analyte) that can be measured and reported with 99-percent confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. At the MDL concentration, the risk of a false positive is predicted to be less than or equal to 1 percent (U.S. Environmental Protection Agency, 2005a).

minimum reporting level (MRL) — smallest measured concentration of a constituent that may be reliably reported by using a given analytical method (Timme, 1995). It is the "less than" value reported when an analyte either is not detected or is detected at a concentration less than the MRL. See the discussion at http://water.usgs.gov/owq/OFR_99-193/minimum.html for further information.

National Environmental Laboratory Accreditation Conference (NELAC) — a cooperative association of states and Federal agencies, formed to establish and promote acceptable performance standards to operate environmental laboratories. The standards cover analytical testing of environmental samples and the laboratory accreditation process.

Input to the process from the private sector is obtained through a variety of mechanisms including open semiannual meetings, participation in NELAC committees, and through the Environmental Laboratory Advisory Board (ELAB). The ELAB is a federally chartered advisory

committee with a balanced representation of the private sector that provides advice to the U.S. Environmental Protection Agency and NELAC. The goal of NELAC is to foster production of environmental laboratory data of known and acceptable quality on which to base decisions concerning public health and environmental management (Energy Laboratories, Inc., 2003).

National Environmental Laboratory Accreditation Program (NELAP) — the National Environmental Laboratory Accreditation Program (NELAP) implements the standards developed by the National Environmental Laboratory Accredication Committee (NELAC). States and Federal agencies serve as accrediting authorities, with coordination facilitated by the U.S. Environmental Protection Agency to ensure uniformity. Accreditation by one NELAP accrediting authority is mutually recognized by other State and Federal accrediting authorities approved under NELAP (Energy Laboratories, Inc., 2003).

negative control — measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results (National Environmental Laboratory Accreditation Conference, 2003b)

NELAC standards — the protocol and/or requirements established by the National Environmental Laboratory Accreditation Conference (NELAC). The plan of procedures for consistently evaluating and documenting the ability of laboratories performing environmental measurements to meet nationally defined standards (National Environmental Laboratory Accreditation Conference, 2003b).

NWQL — National Water Quality Laboratory

performance audit — the routine comparison of independently obtained quantitative measurement system data with routinely obtained data to evaluate the proficiency of an analyst or laboratory (National Environmental Laboratory Accreditation Conference, 2003b)

performance evaluation (PE) — proficiency testing by evaluating a laboratory'sperformance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source (National Environmental Laboratory Accreditation Conference, 2003b) and determining a laboratory's calibration or testing performance by means of interlaboratory comparisons. The results of PE samples show whether or not a laboratory has an analytical bias, if the bias is continuing, and/or if the bias is increasing. If results are not within the control limits, a sample is analyzed again.

Test samples are prepared by spiking known concentrations of select analytes into a well-characterized matrix. Typically, PE samples are made in a single matrix such as an aqueous, solid, or oil matrix. PE samples can be distributed as single- or double-blind samples. PE samples are used to determine a laboratory's accuracy as it relates to the execution of a particular analytical method (Forman and Vitale, 1999).

A PE program provides controlled and standardized environmental samples to participating laboratories for analysis, reporting of results, statistical evaluation of the results in comparison to peer laboratories, and the collective demographics and results summary of all participating laboratories (National Environmental Laboratory Accreditation Conference, 2003b).

pH — the logarithm of the reciprocal of the hydrogen-ion concentration in gram atoms per liter. It is numerically equal to 7 for neutral solutions and increases with increasing alkalinity and decreasing with increasing acidity. The pH scale ranges from 0 (acidic) to 14 (basic). Each unit of change represents a ten-fold change in hydrogen ion concentration.

positive control — measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects (National Environmental Laboratory Accreditation Conference, 2003b)

precision — the degree of mutual agreement characteristic of independent measurements as the result of repeated application of the process under specified conditions (Taylor, 1987) or a measure of the degree of agreement among replicate analyses of a sample, usually expressed as the standard deviation (American Public Health Association, 1998); see "variability"

preservation — refrigeration and/or reagents added at the time of sample collection or later to maintain the chemical and/or biological integrity of the sample (National Environmental Laboratory Accreditation Conference, 2003b)

procedure — a process, method, or instrument used and documented by an approved standard operating procedure (SOP); a specified way to carry out an activity or process (National Environmental Laboratory Accreditation Conference, 2003b)

protocol — a detailed written procedure for field and/or laboratory operation (for example, sampling, analysis) that must be strictly followed (National Environmental Laboratory Accreditation Conference, 2003b)

QAS — Quality Assurance Section

Quality Assurance (Project) Plan (QAPP) — a formal document describing the detailed quality-control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved (National Environmental Laboratory Accreditation Conference, 2003b)

quality assurance (QA) — a set of operating principles that, if strictly followed during sample collection and analysis, will produce data of known and defensible quality. That is, the accuracy of the analytical result can be stated with a high level of confidence. It is a definitive program for laboratory operation that specifies the requirements that will produce data of known bias and variability. Included in QA are quality control and quality assessment (American Public Health Association, 1998).

In a more general context, QA is an integrated system of activities involving planning, quality control, quality assessment, reporting, and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence (National Environmental Laboratory Accreditation Conference, 2003b).

quality control (QC) — a set of measures within a sample analysis methodology to assure that the process is in control (American Public Health Association, 1998); the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users (National Environmental Laboratory Accreditation Conference, 2003b)

quality management system (QMS) — a document stating the quality policy, quality system, and quality practices of an organization. This also may be called a Quality Assurance Plan or a Quality Plan.

Note: The document may include by reference other documentation relating to the laboratory's quality arrangements (National Environmental Laboratory Accreditation Conference, 2003b).

quality system — a structured, documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system is the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance and quality control (National Environmental Laboratory Accreditation Conference, 2003b).

range — the difference between the minimum and the maximum of a set of values

raw data — not the preferred term; see "unprocessed data"

reagent blank (method reagent blank) — a sample consisting of reagent(s), without the specified analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps (National Environmental Laboratory Accreditation Conference, 2003b)

reagent spike — a synthetic matrix fortified with known concentrations of all, or a representative selection of, the method analytes. The synthetic matrix usually is the same as the method blank, for example, organic-free water or sodium sulfate. For the purpose of interpreting the corrective action guidelines described in this document, a reagent spike failure is defined as an out-of-control recovery for any relevant spiked analyte (U.S. Geological Survey National Water Quality Laboratory Technical Memorandum 94-07, 1994).

reference material — a material or substance, one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials (National Environmental Laboratory Accreditation Conference, 2003b)

reference standard — a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived (National Environmental Laboratory Accreditation Conference, 2003b)

replicate analyses — the measurements of the variable of interest performed identically on two or more subsamples of the same sample within a short time (National Environmental Laboratory Accreditation Conference, 2003b)

requirement — denotes a mandatory specification; often designated by the term "shall" (National Environmental Laboratory Accreditation Conference, 2003b)

sample — one or more portions of a liquid, gas, or solid material (including biological tissues) taken in an unbiased manner from a batch, lot, process stream, or on-site from the environment and considered to be representative of the whole for subsequent testing to determine the chemical, physical, mechanical, or other quality charac-

teristics of the material, or combination thereof (modified from American Society for Testing and Materials, 2000)

sample inventory — all samples in the laboratory that are recognized, verified, and tracked in the laboratory information management system (LIMS). Two types of samples are identified, including samples within their holding time(s) and samples whose holding time(s) have expired.

sample preparation — any pre-defined test or process performed on a sample before analysis. This may be done by personnel at a U.S. Geological Survey (USGS) Water Science Center at the time of sample collection or employees at the USGS National Water Quality Laboratory after login of samples, or both.

schedule — a collection of one or more methods; not the preferred term; see "method" or "suite"

selectivity — the capability of a test method or instrument to respond to a selected compound or constituent in the presence of known or unknown substances (National Environmental Laboratory Accreditation Conference, 2003b)

sensitivity — the capability of a test method or instrument to discriminate between measurement responses representing different levels (for example, concentrations) of a variable of interest (National Environmental Laboratory Accreditation Conference, 2003b)

single blind sample — the laboratory knows that the sample submitted is a test sample, but does not know its true concentration or results

spike — a known mass of specified analyte added to a blank sample or subsample; used to determine recovery efficiency or for other quality-control purposes (National Environmental Laboratory Accreditation Conference, 2003b)

spiked sample — see "matrix spike duplicate"

standard — the document describing the elements of laboratory accreditation that have been developed and established within the consensus principles of the National Environmental Laboratory Accredication Conference (NELAC) and meets the approval requirements of NELAC procedures and policies (National Environmental Laboratory Accreditation Conference, 2003b)

standard operating procedure (SOP) — a document that describes the analytical methods to be used in the laboratory in sufficient detail that a competent analyst unfamiliar with the method can conduct a reliable review and/or obtain acceptable results.

Where applicable, an SOP may include title of referenced, consensus test method; sample matrix or matrixes; method detection level (MDL); scope and application; summary of SOP; definitions; interferences; safety considerations; waste management; apparatus, equipment, and supplies; reagents and standards; sample collection, preservation, shipment, and storage requirements; specific quality control practices, frequency, acceptance criteria, and required corrective action if acceptance criteria are not met; calibration and standardization; details on the actual test procedure, including sample preparation; calculations; qualifications and performance requirements for analysts (including number and type of analyses); data assessment/data management; references; and any tables, flowcharts, and validation or method performance data. At a minimum, validate a new SOP before use by first determining the MDL and performing an initial demonstration of capability using relevant regulatory guidelines (American Public Health Association, 1998).

standard reference material (SRM) — certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization, and characterized for absolute content, independent of analytical test method (National Environmental Laboratory Accreditation Conference, 2003b)

standard reference sample (SRS) — a sample that is prepared to check and monitor inorganic analytical systems. These samples are prepared from large, homogeneous quantities of water, bottled under strict aseptic conditions, and designed to minimize contamination sources. These references usually are composed of a natural matrix collected from different sources, such as snowmelt, streams, and ground water. They also include SRS prepared from certified reference standards, spiked into reagent-grade ASTM type I water.

suite — a collection of tests, analytes, variables, or any combination that is defined by the U.S. Geological Survey National Water Quality Laboratory as a routine (a test or suite of tests that can be obtained directly from an Analytical Services Request form) or a customer-defined request

supervisor — individual designated as being responsible for a particular area, unit, or category of scientific analysis.

This responsibility includes direct day-to-day supervision of technical employees; supply and instrument adequacy and upkeep; quality assurance/quality control duties; and ascertaining that technical employees have the required balance of education, training, and experience to perform the required analyses (National Environmental Laboratory Accreditation Conference, 2003b).

supervisory review — a review of a standard operating procedure (SOP) by a unit supervisor, who evaluates the SOP for technical accuracy, use of the appropriate document format, and completeness. The supervisor reviews the SOP twice, once after it has gone through peer review but before it is passed to the Section Chief, and again, after the SOP is revised following the inter-Section reviews prior to final Section approval.

surrogate — a substance with properties that mimic the analyte(s) of interest. It is unlikely to be found in environmental samples and is added to them for quality-control purposes (National Environmental Laboratory Accreditation Conference, 2003b).

surrogate spike — compounds similar in physical and chemical properties to the compounds of interest in a given method that are added to all environmental samples, reagent spikes, method blanks, and other relevant QC samples for applicable methods. A surrogate is expected to behave similarly in the analytical process to at least some of the analytes (Wershaw and others, 1987, p. 4).

test — a technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process, or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate (National Environmental Laboratory Accreditation Conference, 2003b).

test method — a defined technical procedure for performing a test. It also may be the adoptation of a scientific technique for a specific measurement problem, as documented in a laboratory SOP or as published by a recognized authority (National Environmental Laboratory Accreditation Conference, 2003b).

traceability — the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons (National Environmental Laboratory Accreditation Conference, 2003b)

trip blank — a sample of analyte-free media taken from the laboratory to the sampling site and returned to the laboratory unopened (Wagner, 1992)

turnaround — the maximum time between login of a sample at the U.S. Geological Survey National Water Quality Laboratory and the availability of data on the World Wide Web. Turnaround is a variable-specific time that is defined by the laboratory to be shorter than the holding time to allow re-analysis if needed.

uncontrolled document — a working copy of a document that has been verified by the user as current or a field-controlled copy that contains identification of outstanding chanes to the controlled document and can be used on site (U.S. Department of Energy, 2003). If a document is uncontrolled, there is no assurance that it is the most recent or current version available. Uncontrolled copies can be copied and modified by users and are not considered to be official copies.

U.S. Environmental Protection Agency (USEPA) — the agency of the Federal government with the responsibility of protecting public health and safeguarding and improving the natural environment (that is, the air, water, and land) upon which human life depends (National Environmental Laboratory Accreditation Conference, 2003b)

unprocessed data — any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that is necessary to reconstruct and evaluate a report of the activity or study. Unprocessed data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies have been prepared (for example, tapes that have been transcribed verbatim and data verified accurate by signature), the exact copy or exact transcript may be submitted (National Environmental Laboratory Accreditation Conference, 2003b).

update — a minor reworking of an SOP. The changes will not affect data quality. Examples of an update would be a chemical disposal change, minor data-entry changes, or change in vendor that require minor modification of procedure without affecting data quality. Updates go through a limited review process prior to approval.

validation — the process of substantiating specified performance criteria; confirmation by examination and provision of objective evidence that the particular requirements

for a specific intended use are fulfilled (National Environmental Laboratory Accreditation Conference, 2003b)

variability — random error in independent measurements as the result of repeated application of the process under specific conditions. Variability can be statistically described by the standard deviation (standard error) (Taylor, 1990). All data contain some experimental error and individual measurements change or fluctuate within limits. Precision is a measure of variability in experimental data. Variability is the preferred term used by the NWQL and USGS Branch of Quality Systems.

verification — confirmation by examination and provision of evidence that specified requirements have been met

Note: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation, or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustments, to repair, to downgrade, or to declare obsolete. In all cases it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record (National Environmental Laboratory Accreditation Conference, 2003b).

volatile organic compound(s) (VOC) — a compound having high-vapor pressure and low-water solubility. VOCs are typically industrial solvents, constituents in petroleum-fuel products, or by-products produced by chlorination in water treatment.

waste — a solid, liquid, or contained gaseous material that is no longer used or no longer serves the purpose for which it was produced

whole water, recoverable (WWR) — pertains to the constituents in solution after an unfiltered 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. For inorganic determinations, digestions are performed in the original sample container to ensure digestion of material adsorbed on the container walls. 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 (Timme, 1995, p. 95).

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