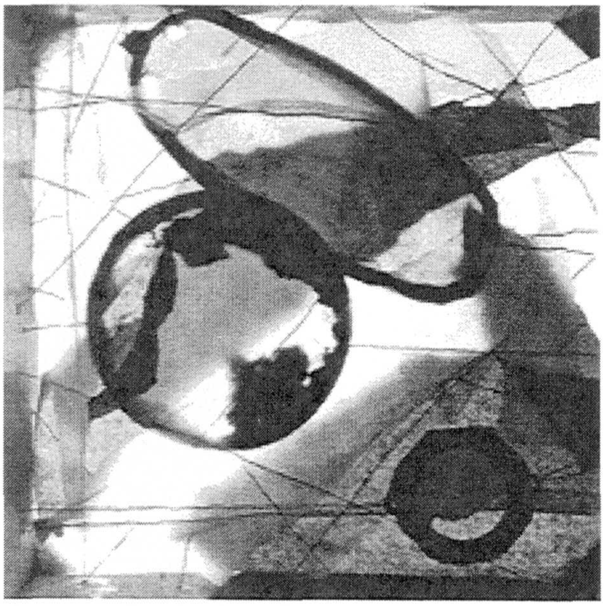
U.S. Department of the Interior
U.S. Geological Survey

Rec'd
11/30/98

Quality-Assurance/Quality-Control Manual for Collection and Analysis of Water-Quality Data in the Ohio District, U.S. Geological Survey

Water-Resources Investigations Report 98-4057



USGS
science for a changing world

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

Quality-Assurance/Quality-Control Manual for Collection and Analysis of Water-Quality Data in the Ohio District, U.S. Geological Survey

By D.S. Francy, A.L. Jones, D.N. Myers, G.L. Rowe, Michael Eberle, and K.M. Sarver

Water-Resources Investigations Report 98-4057

U.S. DEPARTMENT OF THE INTERIOR
BRUCE BABBITT, Secretary

U.S. GEOLOGICAL SURVEY
Thomas J. Casadevall, Acting Director

For additional information write to:

District Chief
U.S. Geological Survey
975 West Third Avenue
Columbus, Ohio 43212-3192

Copies of this report can be purchased from:

U.S. Geological Survey
Branch of Information Services
Box 25286, Building 810
Denver, CO 80225-0286

Columbus, Ohio
1998

CONTENTS

FIGURES

TABLES

CONVERSION FACTORS AND VERTICAL DATUM

Multiply	By	To obtain
inch (in)	25.4	millimeter
inch (in)	0.254	micrometer
foot (ft)	0.3048	meter
square foot (ft^2)	0.09290	square meter
mile (mi)	10.609	kilometer
pint, U.S.	0.4732	liter
quart, U.S. (qt)	0.9464	liter
gallon, U.S. (gal.)	3.7854	liter
foot per second (ft/s)	0.3048	meter per second
square mile (mi^2)	2.590	square kilometer
gallon per minute (gal/min)	3.785	liter per minute
million gallons per day (Mgal/d)	3,785	cubic meter per day

Temperature: Temperature is given in degrees Celsius (°C), which can be converted to degrees Fahrenheit (°F) by use of the following equation:

°F=1.8(°C) + 32

Vertical datum: In this report, "sea level" refers to the National Geodetic Vertical Datum of 1929 (NGVD of 1929)—a geodetic datum derived from a general adjustment of the first-order level nets of both the United States and Canada, formerly called Sea Level Datum of 1929.

Abbreviated water-quality units used in this report: Chemical concentrations and water temperature are given in metric units. Chemical concentration is given in milligrams per liter (mg/L) or micrograms per liter (mg/L). Milligrams per liter is a unit expressing the concentration of chemical constituents as weight (milligrams) of chemical per unit volume (liter) of water. One thousand micrograms per liter is equivalent to one milligram per liter.

Specific conductance of water is expressed in microsiemens per centimeter at 25 degrees Celsius (uS/cm). This unit is equivalent to micromhos per centimeter at 25 degrees Celsius (mmho/cm), formerly used by the U.S. Geological Survey.

Other abbreviated metric units used in this report are the following:

g	gram
L	liter
mL	milliliter
mL/min	milliliter per minute

Concentrations of bacteria are given in colonies per 100 milliliters (col/100 mL) which is the same as colony forming units per 100 milliliters (CFU/100 mL)

Other abbreviations used in this report:

AAS	Atomic absorption spectrophotometry
ANC	acid-neutralizing capacity
APHA	American Public Health Association
ASR	Analytical Service Request Form
ASTM	American Society for Testing and Materials
BOG	U.S. Geological Survey Branch of Geochemistry
BQS	U.S. Geological Survey, Office of Water Quality, Branch of Quanlity Systems
BU	Biological Unit of the U.S. Geological Survey, National Water Quality Laboratory
DEM	digital elevation model
DLG	digital line graph
DNR	Department of Natural Resources
DO	dissolved oxygen
DOD	Department of Defense
DODEC	Department of Defense Environmental Contamination
DQO	data-quality objective
E. coli	*Escherichia coli*, an intestinal fecal-indicator bacterium
EDI	Equal-discharge increment (stream-water sampling technique)
EDTA	ethylenediaminetetraacetic acid or ethydimethyltetrasodium acetate
EIA	confirmation media (substrate agar) for enterococcus
EROS	Earth Resource Observation System
EWI	equal-width increment (stream-water sampling technique)
E_H	oxidation-reduction potential, expressed relative to the Standard Hydrogen Electrode
EPA	Environmental Protection Agency
GC	gas chromatography
GC/MS	gas chromatography/mass spectrometry

GF-AAS	graphite furnace-atomic absorption spectrophotometry
GWSI	U.S. Geological Survey, Ground-water Site Inventory
HIF	U.S. Geological Survey, Hydrologic Instrumentation Facility
HPLC	high performance liquid chromatography
IBW	inorganic-free blank water
ICP	inductively coupled plasma
ICP-MS	inductively coupled plasma emission spectroscopy-mass spectrometry
LFPM	linear feet per minute
MBAS	methylene blue active substances
mF	membrane filter method
MPN	most-probable number
mTEC	membrane filter media for *Escherichia coli*
NA-MUG	nutrient agar-4-methylumbelliferyl-B-D-glucuronide for *E. coli*
NAWQA	U.S. Geological Survey National Water-Quality Assessment Program
NWQL	U.S. Geological Survey National Water Quality Laboratory, Arvada, Colorado
NFQA	National Field Quality Assurance Program
NIST	National Institute of Standards and Technology
NRCS	U.S. Department of Agriculture, Natural Resources Conservation Service
NRP	U.S. Geological Survey, Water Resources Division, National Research Program
NWIS	National Water Information System, U.S. Geological Survey
OBW	organic-free blank water
OEPA	Ohio Environmental Protection Agency
OSW	Office of Surface Water
OWQ	U.S. Geological Survey, Office of Water Quality
PCB	polychlorinated biphenyls
PVC	polyvinyl chloride
QA	quality assurance
QA/QC	quality assurance/quality control

QC	quality control
QWSU	U.S. Geological Survey, Quality of Water Services Unit, Ocala, Florida
SOP	standard operating procedure
SPiN	Schedules, Parameters, and Network Records of the National Water Quality Laboratory
SRWS	Standard Reference Water Sample
USEPA	U.S. Environmental Protection Agency
VOC	volatile organic compound
USGS	U.S. Geological Survey
WATSTORE	Water Data Storage and Retrieval System, U.S. Geological Survey
WRD	Water Resources Division

Quality-Assurance/Quality-Control Manual for Collection and Analysis of Water-Quality Data in the Ohio District, U.S. Geological Survey

by D. S. Francy, A. L. Jones, D. N. Myers, G. L. Rowe, Michael Eberle, and K. M. Sarver

1.0 Abstract

The U.S. Geological Survey (USGS), Water Resources Division (WRD), requires that quality-assurance/quality-control (QA/QC) activities be included in any sampling and analysis program. Operational QA/QC procedures address local needs while incorporating national policies. Therefore, specific technical policies were established for all activities associated with water-quality projects being done by the Ohio District. The policies described in this report provide Ohio District personnel, cooperating agencies, and others with a reference manual on QA/QC procedures that are followed in collecting and analyzing water-quality samples and reporting water-quality information in the Ohio District.

The project chief, project support staff, District Water-Quality Specialist, and District Laboratory Coordinator are all involved in planning and implementing QA/QC activities at the district level. The District Chief and other district-level managers provide oversight, and the Regional Water-Quality Specialist, Office of Water Quality (USGS headquarters), and the Branch of Quality Systems within the Office of Water Quality create national QA/QC polices and provide assistance to District personnel.

In the literature, the quality of all measurement data is expressed in terms of precision, variability, bias, accuracy, completeness, representativeness, and comparability. In the Ohio District, bias and variability will be used to describe quality-control data generated from samples in the field and laboratory. Each project chief must plan for implementation and financing of QA/QC activities necessary to achieve data-quality objectives. At least 15 percent of the total project effort must be directed toward QA/QC activities. Of this total, 5-10 percent will be used for collection and analysis of quality-control samples. This is an absolute minimum, and more may be required based on project objectives.

Proper techniques must be followed in the collection and processing of surface-water, ground-water, biological, precipitation, bed-sediment, bedload, suspended-sediment, and solid-phase samples. These techniques are briefly described in this report and are extensively documented. The reference documents listed in this report will be kept by the District librarian and District Water-Quality Specialist and updated regularly so that they are available to all District staff.

Proper handling and documentation before, during, and after field activities are essential to ensure the integrity of the sample and to correct erroneous reporting of data results. Field sites are to be properly identified and entered into the data base before field data-collection activities begin. During field activities, field notes are to be completed and sample bottles appropriately labeled and stored. After field activities, all paperwork is to be completed promptly and samples transferred to the laboratory within allowable holding times.

All equipment used by District personnel for the collection and processing of water-quality samples

is to be properly operated, maintained, and calibrated by project personnel. This includes equipment for onsite measurement of water-quality characteristics (temperature, specific conductance, pH, dissolved oxygen, alkalinity, acidity, and turbidity) and equipment and instruments used for biological sampling. The District Water-Quality Specialist and District Laboratory Coordinator are responsible for preventive maintenance and calibration of equipment in the Ohio District laboratory.

The USGS National Water Quality Laboratory in Arvada, Colo., is the primary source of analytical services for most project work done by the Ohio District. Analyses done at the Ohio District laboratory are usually those that must be completed within a few hours of sample collection. Contract laboratories or other USGS laboratories are sometimes used instead of the NWQL or the Ohio District laboratory. When a contract laboratory is used, the project chief must first obtain written approval of the Chief, Office of Water Quality. The work of the contract laboratory is subject to ongoing review throughout the project by the USGS-WRD Branch of Quality Systems in the Office of Water Quality.

Finally, data collected are monitored for bias and variability, checked for errors, validated, and stored to facilitate retrieval and use by District personnel and others. Performance and system audits are done by the Ohio District to provide independent evaluation of the quality of data collected. The audits are done at approximately the 10-, 40-, and 70- percent stages of project completion and at other times deemed appropriate by the District Water-Quality Specialist. If a significant condition that adversely affects data quality is noted, project personnel must promptly identify, report, and correct conditions. In addition, each project chief is required to maintain a file of project QA/QC activities. The District Water-Quality Specialist will write an annual report to District management on all QA/QC activities in the District.

2.0 Introduction

The U.S. Geological Survey (USGS), Water Resource Division (WRD), collects, analyzes, interprets, and disseminates information about the quality of water in our nation's streams, lakes, and aquifers. As an earth-science agency, the USGS has a reputation for collecting accurate data and producing factual and impartial interpretive reports (Schroder and Shampine, 1992). Many sample-collection techniques for subsequent analyses of physical, chemical, and biological qualities of water and sediments are documented and have become standard, but new ones are implemented regularly. Within the WRD, the Office of Water Quality (OWQ) and in particular, the Branch of Quality Systems (BQS) within the OWQ, provides protocols, policies, and guidance on how to conduct a quality-assurance/quality-control (QA/QC) program; however, the specific technical policies and operational QA/QC procedures that address local needs while incorporating national policies are developed and implemented at the District, discipline, and project levels.

2.1 Purpose and scope

This report describes current policies of the USGS and Ohio District Office for the collection, storage, processing, analysis, and disposition of data from samples of water, aquatic biota, and solid-phase material. These samples are collected for subsequent analysis of chemical, biological, and (or) physical properties. Biological-, sediment-, and water-quality procedures are described in this report and will all be referred to as "water-quality." The policies described within this report were established for all activities associated with water-quality projects undertaken by the Ohio District. These policies provide Ohio District personnel, cooperators, and others with a reference manual on QA/QC procedures that are followed in collecting and analyzing water-quality samples and reporting water-quality information in the Ohio District.

2.2 Description of water-quality projects

Water-quality projects in the Ohio District can be divided into three main categories (table 2.2-1): (1) projects that are investigations of water-quality constituents and the relation of these constituents to hydrologic variables and processes, (2) projects that are assessments of current conditions or trends in water quality, and (3) projects that assess or evaluate water-quality conditions and are guided by protocols set by other government organizations. Often, the USGS accepts QA/QC or method guidelines of other governmental organizations when such guidelines have been established to support a mission of water-resource management.

Projects that are oriented toward the processes that affect the physical, chemical, or biological qualities of water and sediment comprise most of the water-quality studies done in the Ohio District in a typical year. Many of these projects have a research component while meeting the specific needs of the cooperator(s). Some projects include monitoring of several processes to examine the effects of these processes on an ecosystem.

Long-term programs, such as the National Water-Quality Assessment Program (NAWQA), are directed by WRD management on the national level and implemented in Ohio and elsewhere to assess water quality. These are national programs for which occurrence and distribution of water-quality trends and current conditions are a focus. Other studies are done within the Ohio District to assess water quality (usually on a shorter term than National programs) relating to a specific need from a cooperating agency or other entity.

Several studies that assess water quality and are driven by protocols and requirements set by regulators have been done in cooperation with the Department of Defense (DOD), such as site evaluations and geophysical studies and smaller projects at local DOD-operated facilities. Certain projects are driven by protocols and requirements set by the U.S. Environmental Protection Agency (USEPA), the Ohio Environmental Protection Agency (OEPA), or others. Often, protocols other than standard USGS methods must be followed and strict chain-of-custody procedures must be implemented. The reporting requirements also may be different, including very detailed documentation of quality-control data. The other agency protocols usually enhance the legal aspects of data collection and only rarely diminish the technical quality of the resulting data.

Table 2.2-1. Categories and characteristics of water-quality projects

Water-quality project type	Objectives and characteristics
Process-oriented studies	Experimental designs, research investigations; regional, statewide, or local
Assessment studies	Network designs; national, regional, statewide, or local
Assessment studies guided by the protocols of other agencies	Site-specific investigations

3.0 Quality-assurance/quality-control objectives and activities

Quality assurance/quality control is defined as all those planned or systematic actions necessary to provide adequate confidence that a product or service will satisfy given objectives for quality. Quality-assurance (QA) elements focus on procedures used to control immeasurable components of a project and include project work plans, protocols specifying sampling and processing procedures, maps locating field sites, and books containing equipment maintenance and calibration records. Quality-control (QC) data are the data generated to estimate the measurable components of quality in the processes used for obtaining environmental data. These processes include operational techniques and activities in the field and in the laboratory. Quality assurance/quality control is the responsibility of all those involved in project work; however, QA/QC is implemented at the management level (Schroder and Shampine, 1992).

3.1 Quality-assurance/quality-control objectives for data in terms of bias, variability, representativeness, comparability, and completeness

Quality-control data in the Ohio District will be used to provide estimates of bias and variability. These are the terms recommended for use by the OWQ (Schertz and others, 1998) because they are consistently defined in the literature and address most data-quality objectives in WRD water-quality projects and activities. Quality-assurance elements are used to describe representativeness, comparability, and completeness of data. Other terms are defined in this section and are included for comparisons to the literature.

> Bias is a systematic error inherent in a method or caused by some artifact or idiosyncrasy of the measurement system. Bias may be either positive (from contamination) or negative (from loss or degradation), and multiple sources of bias may contribute to the net bias (Taylor, 1987). Bias is evaluated through the use of field and laboratory blanks, spikes, or standard reference samples.
>
> Variability is the degree of variation in independent measurements as the result of repeated application of the measurement process under specified conditions. Variability is dependent on the sample matrix, data-collection methods, analytical method, and analytical concentration relative to the method detection limit. Variability is evaluated through the use of field and laboratory replicate samples.
>
> Precision is the agreement among independent measurements of the same quantity, without reference to the known or true value. Precision is a measure of repeatability or reproducibility, and it is evaluated most directly by comparing multiple measurements of the same parameter on the same sample under the same conditions. Precision is evaluated through the use of field and laboratory replicate samples.
>
> Accuracy is defined as the degree of agreement of a measured value with the true or the accepted value for that quantity. An accurate method is one that provides precise and unbiased results within acceptable limits (Taylor, 1987).
>
> Representativeness is how well data describe the chemical composition or the biological or physical conditions in the environment at a point or period in time. Representativeness of samples is ensured by (1) collection of samples from locations typical of the site of interest, (2) use of approved sampling methods and equipment, (3) use of appropriate sample-preservation techniques, (4) use of appropriate analytical methods, and (5) adherence to appropriate sample-holding times.
>
> Comparability is a characteristic that represents degree of agreement between results from one sample, sampling round, site, laboratory, project, or study stage to those from another. Comparability is achieved by using processes that yield results of known and similar quality. Procedures used to ensure data comparability include (1) using standard methods for sample collection, processing, and analysis, (2) providing training in standard methods to be employed, (3) using traceable standards for calibration, and (4) reporting results from similar matrices in consistent units.
>
> Completeness of the data is determined by comparing the amount of valid data obtained from the measurement system, either field or laboratory, with the amount of data expected to be obtained under normal conditions. If completeness goals are not met, an attempt is made to re-sample for the characteristics of interest.

3.2 Quality-control samples

Quality-control data are used to determine the magnitude of measurement variability, adjust the measurement process, and aid in interpreting environmental data. Without QC data, sample data cannot be adequately interpreted because the variability associated with the sample data are unknown (Horowitz and others, 1994). The types of field and laboratory QC samples used in the Ohio District are detailed in the following paragraphs and summarized in table 3.2-1. Many of these QC sample types are from Horowitz and others (1994) and WRD Memorandum 91.09. [1]

3.2.1 Field quality-control samples

As a check on the quality of field activities (including sample collection, processing, shipment, and handling), field QC samples are collected periodically and analyzed with project environmental samples. The level of error associated with each step of the collection, processing, and preservation of samples can be measured by use of QC samples. Details on how to collect field QC samples are outlined in Horowitz and others (1994).

Field replicates are a set of environmental samples, collected and analyzed in a manner such that the samples are thought to be virtually identical in composition. They are used to estimate variability for some part of the sample collection and analysis process. Replicate is the general term for two or more samples, whereas duplicate is the term for two samples. Field replicates are either split replicates or concurrent field replicates. Split replicates are subsamples of an already collected and processed sample and are used to determine the analytical variability for various constituents in an environmental sample matrix. Concurrent field replicates are two samples taken as closely together in time and space as possible. Sequential field replicates are two replicates taken one right after the other. Concurrent and field replicates are collected, processed, and preserved separately and provide the user with a measure of sampling and analytical variability.

Blanks are used to identify sampling and analytical bias caused by contamination from equipment, supplies, and ambient environmental conditions. A blank solution is free of the analyte(s) of interest. Inorganic-free blank water (IBW) and organic-free blank water (OBW) may be purchased from the Quality Water Service Unit (QWSU) of the USGS in Ocala, Fla. A field blank is a blank solution used to determine potential contamination that can occur through all stages of sample collection, processing, preservation, transportation, and handling. A field blank is generated under actual field conditions and at least one blank is collected during each sampling trip. An equipment blank is similar to a field blank except it is used to determine potential contamination from the equipment cleaning process. An equipment blank is processed in the relatively controlled environment of an office or laboratory and is collected before project field activities begin and at least annually thereafter. Sequential blanks are used to assess potential contamination from each step in sample collection, processing, and handling. Sequential blanks are a series of blank samples (sampler blank, splitter blank, pump blank, preservation blank) collected in sequential order after each step in the generation of field or equipment blanks. A source solution blank verifies the composition of the blank solution and is collected in a relatively protected area. Sequential and source solution blanks are collected along with field and equipment blanks and are submitted to the laboratory if contamination is found in the equipment or field blank. A trip blank is a blank solution used to determine contamination from migration of compounds into the sample from the air. A trip blank is put in the same type of bottle used for an environmental sample and kept with the environmental sample bottles before, during, and after sample collection. Typically, trip blanks are analyzed for volatile organic compounds. An ambient blank is a blank solution used to

---

[1] Throughout this report, in addition to published reports, technical memorandums from the WRD, OWQ, and NWQL will be included with the reference documents. All official technical memorandums can be accessed on the World-Wide Web at http://wwwoper.er.usgs.gov/memos.

determine potential contamination from environmental sources. It is put in the same type of bottle used for an environmental sample, kept with the set of sample bottles before sample collection, and opened at the site and exposed to ambient conditions.

Field matrix spikes are environmental samples fortified in the field with known concentrations of some or all of the analytes of interest. These are used to determine bias of analyte recovery in a sample matrix and degradation of the analyte during shipping and storage.

Table 3.2-1. Summary of the types, definitions, and purposes of quality-control samples

Type of sample		Definition	Purpose
Field quality-control samples			
Field replicate		Environmental samples, collected and analyzed so that they are virtually identical in composition	Sampling and analytical variability
	Split	An already collected and processed sample split into two or more samples	Analytical variability
	Concurrent	Two replicates taken as closely together as possible in time and space; samples are collected, processed, and preserved separately.	Sampling and analytical variability
	Sequential	Two replicates taken one right after the other	Sampling and analytical variability
Blanks		A sample that contains a blank solution, free of the analyte(s) of interest	Sampling and analytical bias caused by contamination from equipment, supplies, and ambient environmental conditions
	Field blank	Blank solution processed through all stages of sample collection, processing, preservation, and handling under field conditions	Contamination from equipment, supplies, and the environment
	Equipment blank	Blank solution processed through all stages of sample collection, processing, preservation, and handling in a controlled environment	Contamination from equipment and supplies
	Sequential blank	Blank solution collected after each step in the generation of a blank sample	Contamination from step(s) in field sampling and sample processing
	Source solution blank	Blank solution collected in a protected area	Verifies the composition of the blank solution
	Trip blank	Blank solution put in the same type of bottle and kept with the environmental sample	Contamination from migration of compounds into the sample from the air
	Ambient blank	Blank solution put in the same type of bottle as the environmental sample and opened at the site	Contamination from ambient environmental conditions
Field matrix spike		Environmental sample fortified in the field with known concentrations of the analyte(s) of interest	Bias of analyte recovery and degradation during shipping
Laboratory quality-control samples			
Laboratory replicate		Environmental sample split into two or more subsamples in the laboratory	Variability of the analytical method
Laboratory blank		Blank solution carried through the sample preparation and analytical procedures	Laboratory bias from contamination
Reagent spike		Blank solution fortified with known concentration of the method analyte(s)	Laboratory bias of analyte recovery
Laboratory matrix spike		Environmental sample fortified with known concentrations of the analyte(s)	Laboratory bias with (if any) matrix interferences
Surrogate		Compounds similar in properties to the analytes of interest and added to environmental samples	Monitor matrix effects on analyte recovery
Standard reference sample		Sample with known concentration of the analyte(s)	Bias of the analytical procedure

3.2.2 Laboratory quality-control samples

Laboratories routinely analyze various QC samples to estimate the quality of analytical procedures, determine the need for internal corrective action, and interpret results after corrective action is implemented. Results of these samples can be obtained from the laboratory if desired; selected QC results typically are provided with the analytical data.

There are several common types of laboratory QC samples. A laboratory replicate is a single environmental sample, split into two or more replicates in a controlled laboratory environment and used to assess the variability of the analytical method. A laboratory blank is a blank solution carried through the entire sample preparation and analytical procedure used to evaluate bias from laboratory contamination. A reagent spike is a blank solution fortified in the lab with known concentrations of some, or all, of the method analytes. The reagent spike is used mainly to assess bias of organic analyses in reagent water. A laboratory matrix spike is an environmental sample fortified in the laboratory with known concentrations of some, or all, of the method analytes of interest. Matrix spikes are used to assess the extent of matrix interferences and to evaluate bias for specific sample matrices. Surrogates are compounds that react in analysis similarly to the analytes of interest, but are not typically found in environmental samples. Samples fortified with surrogates are used commonly in organic analyses and do not interfere with the analysis of the analytes of interest. The surrogate compounds are added to environmental samples immediately before sample preparation (Wershaw and others, 1987) and are used to monitor matrix effects on analyte recovery. Standard reference samples are samples that are certified reference materials and have known concentrations of the analytes of interest. Standard reference samples are used to assess the analytical process and provide insight into bias from calibration procedures or instrument drift.

3.3 Organization and responsibility

The diagram in figure 3.3-1 shows general relationships among personnel and organizational units responsible for QA/QC of water-quality data collected in the Ohio District. Project chiefs obtain approval for QA/QC activities from the District Water-Quality Specialist. The District Water-Quality Specialist, with assistance from the Regional Water-Quality Specialist and OWQ, in particular the Branch of Quality Systems (BQS), is responsible for creating QA/QC policies, obtaining reference materials, providing assistance with their use, and providing oversight on project QA/QC activities. The Laboratory Coordinator provides support to the District Water-Quality Specialist in organizing water-quality records and maintaining supplies and equipment. The project chief has the primary responsibility for assuring that QA/QC procedures are implemented by the project support staff and for monitoring results; however, the District Chief and other managers have the oversight for assuring the technical quality of all district products. Therefore, communication between all concerned parties is of critical importance.

3.4 Development of project-specific objectives and minimum requirements for quality-assurance/quality-control activities

3.4.1 Planning quality-assurance/quality-control activities

Each project chief must plan ahead for implementation and financing of QA/QC activities. During the project proposal phase, the objectives and levels of QA/QC activities are projected. Although these levels may change between the project proposal phase and ongoing parts of the project, a thorough examination of QA/QC objectives is required in the project proposal phase for reasonable estimation of project costs. Costs associated with QA/QC activities are not limited to data-collection activities; these costs also include data analysis, processing, and reporting.

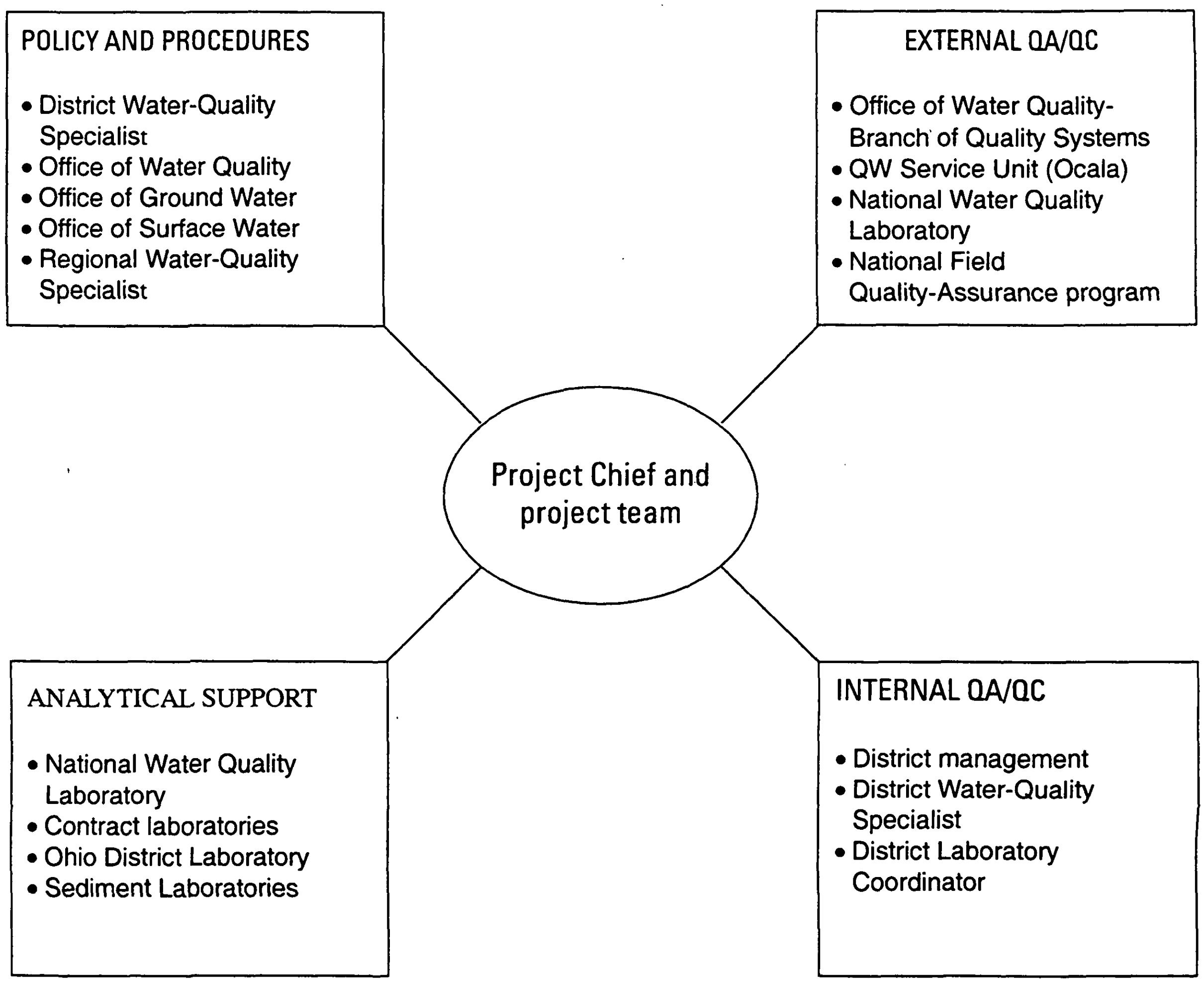


Figure 3.3-1. Guidance, support, and review components of quality-assurance/quality-control activities (QA/QC) for Ohio District water-quality projects.

The number and types of QC samples are established during the project proposal phase in consultation with the District Water-Quality Specialist. Various statistical techniques can be used to determine QC sample size requirements (Walpole and Myers, 1985; Noether, 1987). Careful examination of the following questions also will help evaluate QC sample needs:

1. Why are the data being collected, and how will the data be used?
2. What level of potential contamination from sampling and analytical procedures will be assessed by use of blanks?
3. How will the data on replicate samples be used to establish acceptable limits of variability?
4. How will the data from standard reference samples and spike samples be used to assess acceptable levels of bias for each analyte?
5. How will the types of QC samples be distributed during the phases of the project?
6. What other variables in the project (such as flow, season, and land use) will affect the distribution of QC samples?

3.4.2 The project work plan

When the project begins, the QA/QC objectives and implementation plan must be included in the project work plan prepared by the project chief. The work plan includes discussions of the types, number, and objectives of QC samples, the process for deciding when QC samples are needed, how the QC data are to be evaluated, and actions to be implemented on the basis of QC results (Shampine and others, 1992). QA elements are listed in the project work plan and include references for standard protocols, nonstandard protocols that need to be documented, and training requirements. QA/QC activities in the project work plan are reviewed and approved by the District Water-Quality Specialist before the project is implemented. Guidance on integrating QA/QC into the project work plan is provided by Shampine and others (1992).

Data-quality objectives (DQO's) are to be considered when developing the project work plan (Shampine and others, 1992; and USEPA, 1987 and 1994). DQO's are qualitative and quantitative statements developed to specify the quality of data needed from a particular data-collection activity to support a specific decision. DQO's are established before data collection by evaluating the project objectives, hypotheses to be tested, time and resource constraints on data-collection activities, methods available for collecting and analyzing the data, and end uses of the data. Acceptance criteria are defined for bias, variability, representativeness, comparability, and completeness as part of the DQO's. Each topic in the work plan will have a statement describing activities needed to ensure that data obtained will meet DQO's of the project.

DQO's and the types of QC samples and QA elements can be dynamic and may change as new information is obtained. For example, early in the project, QC samples are used to validate sample collection and analytical methods (or identify the need to adjust methods), provide early detection of problems in data interpretation, and document data quality. After the validity of field and laboratory procedures is documented, the number and types of QC samples may be reduced.

3.4.3 Implementation and minimum requirements for quality-assurance/quality-control activities

The effectiveness of a project work plan and implementation of DQO's requires that all members of the project team be aware of QA/QC objectives and the types of samples required. Personnel must be properly trained in the rationale and proper methodologies for collection and analysis of data. Written protocols or references outlining all phases of data collection and analysis, including QA/QC activities, are required. These written instructions are incorporated in the project work plan and are the standard operating procedure in all phases of the project. Accordingly, communication of all project issues between project chief, project personnel, District Water-Quality Specialist, and the laboratory is crucial.

Each project chief is required to maintain a file of project QA/QC activities. This file will contain, at a minimum, (1) the project work plan containing QA/QC objectives and implementation plan and DQO's, (2) written protocols or references for project activities, including those references contained in this report (3) dates and results of any QA/QC audits conducted by the District Water-Quality Specialist (see section 9.1), and (4) the project QA/QC checklist provided by the District Water-Quality Specialist.

The USGS, WRD, requires that QC samples and QA elements be included in any sampling and analysis program because without this information, the quality of collected environmental data can neither be qualified nor evaluated (Horowitz and others, 1994). Accordingly, at least 15 percent of the total project effort must be directed toward QA/QC activities. Of this total, 5-10 percent are used for collection and analysis of QC samples. This is an absolute minimum, and more may be required based on project objectives.

4.0 Quality-assurance/quality-control requirements in the collection and processing of samples

In order to achieve project DQO's and QA/QC requirements, proper techniques must be followed in the collection and processing of samples. Considerable information is available about the techniques and procedures that are used in the collection and processing of surface-water, ground-water, biological, precipitation, sediment, and solid-phase samples. Because of rapid changes in technology, new and improved methods are continuously being developed. Therefore, the methods listed in this and other sections of this report are briefly described and heavily referenced. The reference documents described in this report will be kept in the District library and by the District Water-Quality Specialist for distribution to District staff, as requested. These documents will be updated regularly by the District Water-Quality Specialist.

4.1 Surface-water samples

Procedures and references for collecting and processing surface-water samples are described in the following sections and summarized in table 4.1-1.

Table 4.1-1. Summary of methods for collection and processing of surface-water samples

	Type of method	Method reference
	Site selection and sampling frequency	
All surface waters	Random, stratified random, cluster, and systematic sampling	Averett and Schroder, 1994, p. 21-29
	Site selection methods	Averett and Schroder, 1994, p. 30-34
Streams	Site selection methods	Edwards and Glysson, 1988, p. 45-47 Ward and Harr, 1990, p. 2 Wilde and others, 1998a
Lakes and reservoirs	Dispersed random and transect sampling	Ward and Harr, 1990, p. 15-16

Table 4.1-1. Summary of methods for collection and processing of surface-water samples—Continued

	Type of method	Method reference
	Representative samples and collection techniques	
Streams	Single vertical EDI and EWI	Edwards and Glysson, 1988, p. 49-76
	Isokinetic sampling	Ward and Harr, 1990, p. 2-9
	Dip (grab)	Martin and others, 1992
	Point	Wilde and others, 1998d
Lakes and reservoirs	Point	Ward and Harr, 1990, p. 15-23
	Water-temperature profiles	Averett and Schroder, 1994, p. 14-18
	Cleaning and avoiding contamination	
Inorganics	Detergent, tap water, hydrochloric acid, deionized water	Horowitz and others, 1994, p. 11-12, 41-44, and 52-56 Wilde and others, 1998c
Most organics	Detergent, deionized water, methanol, and hexane	Sandstrom, 1995, p. 7 Wilde and others, 1998c
Organic carbon	Detergent and deionized water	Shelton, 1994, p. 13 Wilde and others, 1998c
Volatile organics	Detergent, deionized water, methanol	Shelton, 1997
	Sampling protocols	
Inorganics	Inorganic protocol	Horowitz and others, 1994
Organics	Organics associated with sediment	Horowitz and others, 1994
	Organics dissolved in water	Shelton, 1994 Wilde and others, 1998d
	Organics as a film on water's surface	Wershaw and others, 1987, p. 7-8
	Volatile organics	Wilde and others, 1998d Shelton, 1997
	Organic carbon	Shelton, 1994, p. 16 Wilde and others, 1998d
Radio-chemicals	Inorganic protocol	Horowitz and others, 1994
	Other protocols	Thatcher and others, 1977, p. 9-11 Wilde and others, 1998d
	Sampling devices	
EDI and EWI	Bridge sampling—US-D77TM Wading—US-DH81	Horowitz and others, 1994, p. 7-8 Edwards and Glysson, 1988, p. 7-16 Shelton, 1994, p. 10 Wilde and others, 1998b
	Transit rates for isokinetic sampling	Edwards and Glysson, 1988, p. 67-76
Single vertical and dip	US-D77TM US-DH81 Weighted-bottle sampler	Ward and Harr, 1990, p. 9-14. Shelton, 1994, p. 11 Wilde and others, 1998b
Point	Van Dorn Nansen-type Kemmerer-type Weighted-bottle sampler	Ward and Harr, 1990, p. 16-24 Wilde and others, 1998b
Automatic	Types and representativeness	Ward and Harr, 1990, p. 10-13 Edwards and Glysson, 1988, p. 32-43

Table 4.1-1. Summary of methods for collection and processing of surface-water samples—Continued

	Type of method	Method reference
	Compositing and splitting devices	
Inorganics	Churn splitter	Horowitz and others, 1994, p. 9 Ward and Harr, 1990, p. 34-39 Wilde and others, 1998e
	Cone splitter	Shelton, 1994, p. 12 and p. 18 Capel and others, 1995 Wilde and others, 1998e
Organics and sediment	Cone splitter	Ward and Harr, 1990, p. 36-39 Shelton, 1994, p.12 and p. 18 Capel and others, 1995 Wilde and others, 1998e
	Filtration devices	
Trace elements	Capsule filter	Horowitz and others, 1994, p. 13-14 Wilde and others, 1998e
Major ions, nutrients, or radiochemicals	Capsule filter Plate filter holders with cellulose membranes	Horowitz and others, 1994, p. 13-14 Ward and Harr, 1990, p. 51-52 Timme, 1995, p. 17-18 Shelton, 1994, p. 20 Wilde and others, 1998e
Organics	Glass-fiber or Teflon membrane or Teflon-capsule filters	Sandstrom, 1995 Timme, 1995, p. 18 Shelton, 1994, p. 23 Wilde and others, 1998e
Dissolved-organic carbon	Stainless steel or Teflon filter holders with silver membranes	Ward and Harr, 1990, p. 53 Timme, 1995, p. 18 Wilde and others, 1998e Shelton, 1994, p. 23
	Preservation	
All samples	Chilling, preservative, or no preservation required	Timme, 1995, p. 17-19 Horowitz and others, 1994, p. 17-19 Ward and Harr, 1990, p. 54-64

4.1.1 Site selection and sampling frequency

Deciding when and where to sample is the first step toward achieving project DQO's and QA/QC requirements. Averett and Schroder (1994) discuss how to determine frequency of sampling and design a surface-water sampling program based on the following sampling designs: simple random, stratified random, cluster, and systematic sampling. In deciding where to sample for stream, lake, and reservoirs studies, refer to Averett and Schroder (1994). For a complete discussion on the selection of streamwater sampling sites, refer to Edwards and Glysson (1988), Ward and Harr (1990), or Wilde and others (1998a). For example, locate streamwater sites near a gaging station because of the relation between water-quality characteristics and streamflow. In addition, avoid streamwater sites that may be affected by backwater conditions.

If the sampling site is not near an established gaging station, the site data must be entered into the USGS Ground-Water Site Inventory System (GWSI) (Mathey, 1989) as described in section 5.1.1. Once a streamwater site is established, carefully select a sampling cross section to ensure adequate mixing conditions (Edwards and Glysson, 1988; Ward and Harr, 1990). For lakes and reservoirs, sampling locations may be selected by two schemes described in Ward and Harr (1990): dispersed random sampling and transect sampling. All pertinent information about the site and sampling scheme are to be maintained in the site field folder, described in section 5.1.1.

4.1.2 Representative samples and collection techniques

Obtaining a sample of water that represents the characteristics of the stream, lake, or reservoir at a point in time is essential for meeting DQO's for water-quality project data. Details of representative sampling techniques of surface waters are described in Edwards and Glysson (1988), Ward and Harr (1990), and Wilde and others (1998d) and are summarized below. Review these references before beginning any surface-water sampling program.

Streams. If discharge weighted samples are needed for a project; for example, the distribution of target constituents is uneven or unknown in a cross section, a multiple-vertical sampling method must be used (Ward and Harr, 1990). The **equal-discharge-increment (EDI) method** (Edwards and Glysson, 1988) requires some knowledge of the distribution of streamflow in the cross section. In this method, samples are obtained from the centroids of segments having equal discharge increments. The transit rate at each centroid or vertical need not be constant; however, equal sample volumes are collected at each vertical. The **equal-width increment (EWI) method** requires that samples be taken at verticals equally spaced across the stream. The volume collected is proportional to stream discharge and is not the same at each vertical; however, the transit rate at all verticals must be equal. A complete discussion of EDI and EWI is given in Edwards and Glysson (1988).

In addition, if one is concerned with collecting samples representative of the water-sediment mixture, the sampler needs to be filled isokinetically; that is, water approaching the sampler must not change in velocity or direction as it enters the intake (Ward and Harr, 1990). To collect an isokinetic sample, it is very important that the correct sampler be used for the conditions at the sampling site and that the sampler be used correctly. Refer to Edwards and Glysson (1988) for details on how to collect an isokinetic sample.

If the physical properties and chemical-constituent concentrations of the streamwater are well mixed in the cross section, if only concentrations of dissolved constituents are to be determined, or if a depth-integrated sample is needed at a single location in the cross section, the single-vertical depth-integrated sampling method may be used to obtain a representative sample. In the **single-vertical method**, the sampler is lowered and raised through the water column at a uniform transit rate.

Dip (grab) sampling and **point-sampling methods** may also be used to obtain a representative sample in some cases. For example, dip samples are collected when high velocities (>10 ft/s), shallow channel depths, or excessive debris in the stream preclude the use of EWI or EDI methods. However, dip samples collected near the water surface may underrepresent concentrations of some sediment-associated constituents (Martin and others, 1992). In the dip-sampling method, a sample is collected below the surface of the water for some constituents (such as bacteria) to minimize collection of surface film and avoid contact with the streambed (Myers and Wilde, 1997). Alternatively, for volatile organics, a dip sample is collected at the surface of the water. The point-sampling method is used when project objectives include defining the water quality of one location in the stream. In the point-sampling method, water is collected from a fixed point in a vertical.

Lakes and reservoirs. The probability that a single sample of a lake or reservoir is representative of the whole body of water is slight; therefore, a sampling program must be carefully designed (Ward and Harr, 1990). No sampling program would be complete, however, without a good understanding of water-temperature profile (Averett and Schroder, 1994). In sampling lakes and reservoirs, the point-sampling method is commonly used at several locations and depths.

4.1.3 Cleaning and avoiding contamination

Considerable care must be taken to avoid contamination during sampling and processing. The two biggest sources of aqueous sample contamination are (1) improperly cleaned equipment and (2) atmospheric inputs, such as dirt and dust (Horowitz and others, 1994).

The selection of proper cleaning procedures is dependant on the characteristics of target constituents. For inorganic sampling, complete laboratory and field cleaning procedures for removal of contaminants and for avoiding contamination during sampling and processing are detailed in Horowitz and others (1994) and Wilde and others (1998c). Specifically, detergent and hydrochloric acid are used to remove trace inorganics during the cleaning procedure. To remove organics from equipment, use detergents and methanol in the cleaning procedures; however, the use of methanol must be considered on a case-by-case basis. For example, methanol is not used to clean equipment used in sampling for dissolved organic carbon (Shelton, 1994; Wilde and others, 1998c) or methylene blue active substances. For samples with relatively high concentrations of hydrophobic organic compounds, use an additional solvent such as hexane after the methanol rinse (Sandstrom, 1995).

Processing and preservation chambers are used to reduce or eliminate potential atmospherically derived contaminants. The chambers can be a permanently installed structure in the field vehicle or a portable unit composed of a nonmetallic frame and a disposable plastic cover. The use of processing and preservation chambers to reduce atmospherically derived contaminant inputs is highly recommended in processing and preserving all samples and is required for trace-element analysis (Horowitz and others, 1994; Wilde and others, 1998e). In collecting and processing samples for subsequent organic analysis, be aware of any potential sources of contamination such as exhaust from engine motors and pesticide applications in a nearby field.

4.1.4 Sampling protocols by target constituent

Surface-water samples are collected to determine the concentrations of inorganic (trace elements, nutrients, and major ions), organic, and radiochemical constituents. Processes that control concentrations of surface-water constituents are discussed in Hem (1989).

Samples for subsequent inorganic analyses. A protocol for the collection and processing of samples for low-level inorganic analyses in filtered water was developed by the OWQ to improve and ensure the quality of data produced by the WRD (Horowitz and others, 1994). That document provides a detailed description and explanation of procedures to follow in the preparation and use of sample collection and processing equipment for obtaining contaminant-free samples suitable for inorganic analyses at the microgram-per-liter level. The protocol (hereinafter called, "the inorganic protocol") is now the standard operating procedure (SOP) for collecting and processing samples for trace elements, nutrients, and major ions, even at reporting limits above the microgram-per-liter level. Any deviations from the inorganic protocol must be justified by the DQO's and must be verified by a higher number of QC samples than normally required.

Samples for subsequent organic analyses. The sampling technique for organic constituents will depend on the nature of the target organic compound and surface-water conditions. In sampling for organics of any type, sample equipment must be made of Teflon, stainless steel, aluminum, or glass; contact between the water sample and plastic is prohibited. For organics associated with sediment or dissolved in water, the inorganic protocol techniques (Horowitz and others, 1994) or collection techniques for representative samples (Shelton, 1994; Wilde and others, 1998d) may be used. For organics that are found as a film on the water's surface, a dip-sampling method is used. In collecting samples for volatile organics, sample bottles are filled to the top directly from the stream, lake, or reservoir, leaving a convex meniscus at the mouth (Wershaw and others, 1987; Wilde and others, 1998d). A newly developed volatile organic carbon sampler may also be used for collection of stream-water samples (Shelton, 1997). Samples must be collected so as not to degas the volatile organic of interest. In collecting samples for organic carbon analyses, collect the sample directly into a baked 250-mL amber glass bottle using a weighted-bottle sampler at a single midstream vertical (Shelton, 1994). Contact the appropriate National Water Quality Laboratory (NWQL) section chief about other organic analyses that may require special handling.

Samples for subsequent radiochemical analyses. Details of techniques for collection and treatment of samples for radiochemical analyses are given in Thatcher and others (1977) and Wilde and others (1998d). Requirements to be met in sampling water for the determination of radioactive constituents are generally the same as for other constituents. For example, although the inorganic protocol (Horowitz and others, 1994) has not been evaluated for its applicability to the collection of samples for subsequent determination of radiochemical constituents, it may be used with appropriate QA/QC checks. Contact the radiochemical coordinator at the NWQL when beginning a sampling program for radiochemical constituents.

4.1.5 Sampling and processing equipment and supplies

Sampling devices. The type of sampler used in collecting surface water depends on the characteristics of the target analyte(s) and surface-water conditions.

For sampling streams by use of EDI or EWI from a bridge, the US-D77TM or US-D95 samplers are recommended (Horowitz and others, 1994; Edwards and Glysson, 1988; Wilde and others, 1998b). The use of the US-D77TM and US-D95 samplers with rigid bottles are restricted to depths of 3 to 15 ft. and 2 to 15 ft., respectively, with velocities from 2.0 to 8.0 ft/s (Shelton, 1994). A 5/16- in. nozzle size is used with the US-D77TM and 1/4-in. or 5/16 in. nozzles may be used with the US-D95. For deeper waters, use the US-D77 or a frame-type sampler assembled with Teflon or Reynolds oven bags described in OWQ Technical Memorandum 83.08 (U.S. Geological Survey, written commun., 1983) and recommended in Horowitz and others (1994) and Shelton (1994). For collecting a depth-integrated or discharge-weighted sample under wadable conditions, use the US-DH81 sampler with 5/16-in. nozzles. The US-D77TM, US-D95, and US-DH81 samplers may be used for collection of surface-water samples for determination of inorganic, radiochemical, bacterial, and some organic constituents. All components that contact the water sample are coated with plastic or Teflon, minimizing the potential for contamination. In order to collect an isokinetic sample, one must adjust transit rates on the basis of stream velocity and nozzle size. A complete discussion of transit rates for the US-D77TM sampler is in Edwards and Glysson (1988). Tables of acceptable transit rates and nozzle sizes and minimum volumes that must be collected to ensure that an isokinetic sample is being collected can also be obtained from Wayne Webb (U.S. Geological Survey, written commun., 1997).

To collect a single-vertical or dip sample, a US-D77TM, US-D95, or US-DH81 may be used. One may also use an weighted-bottle sampler (a heavy plastic basket that adjusts to hold various sizes of uncapped bottles has been developed by the Ohio District and may be used in sampling for trace elements). Weighted bottles do not sample isokinetically and collect only quasi-depth-integrated samples (Ward and Harr, 1990). For sampling lakes or reservoirs or point samples in a stream, Van-Dorn type, Nansen-type, Kemmerer-type, or open-mouth water-sampling bottles may be used (Ward and Harr, 1990).

Automatic samplers are used to collect water samples at sites where conventional sampling is not practical or economical, such as at close intervals during runoff or at remote sites. Automatic samplers collect a sample at a single point in the stream vertical. Types of automatic samplers are discussed in Ward and Harr (1990) and Edwards and Glysson (1988). It is important to collect numerous hand samples to verify the representation of the automatic samples for depth-integration and cross-section representation (Ward and Harr, 1990). A detailed discussion of these concerns is given in Edwards and Glysson (1988).

Compositing and splitting devices. Water samples are composited and split when it is necessary to obtain a mixture from several subsamples, such as those collected by the EDI or EWI methods (Ward and Harr, 1990). Two types of splitters are recommended—the churn splitter and the cone splitter (Wilde and others, 1998e). For the collection of inorganic samples by use of the inorganic protocol, Horowitz and others (1994) dictate that the modified churn splitter be used; however, a later evaluation of laboratory and field protocols showed that a Teflon cone splitter can be used to produce contaminant-free subsamples for analysis of trace elements (OWQ Technical Memorandum 97.03). In another study, it was shown that the all-Teflon cone splitter can split a water sample for subsequent analysis of semivolatile organic chemicals and trace elements without contamination (Capel and others, 1995). Shelton (1994) reports that the all-Teflon cone splitter is the best available device for compositing and splitting water samples for analyses of major ions, nutrients, trace elements, pesticides, and sediment and that it is the only choice for splitting pesticide and sediment samples. When collecting samples for analysis of organic constituents, do not use the plastic churn splitter; a Teflon cone splitter is the recommended compositing device (Mark Sandstrom, U.S. Geological Survey, written commun., 1990). If only dissolved organic constituents are determined, a glass or Teflon compositing bottle may be sufficient. For dissolved inorganic constituents, a plastic bottle can be used in addition to either glass or Teflon compositing bottles.

Filtration devices. The choice of filtration equipment and filter type depends on the characteristics of the target analytes(s). A complete discussion of filtration devices is given in Wilde and others (1998e). The disposable capsule filter, made of plastic materials, is the required device in filtering water for subsequent determination of trace elements (Horowitz and others, 1994). Plate filter holders with cellulose-type filter membranes may be used in sampling for major ions, nutrients (Horowitz and others, 1994), or radiochemicals (Ann Mullin, U.S. Geological Survey, oral commun., 1995); however, capsule filters are recommended. In filtering water for organic analysis, glass-fiber or Teflon membrane or capsule filters are used (Sandstrom, 1995). In filtering a sample to be analyzed for dissolved or suspended organic carbon, silver membrane filters, stainless steel or Teflon filter holders, and a nitrogen-gas or hand pump filtration system are used (Ward and Harr, 1990). Regardless of the type of filtration equipment used, it is very important to precondition the filter membrane. For organic-carbon samples, the filter is preconditioned with 25 mL of organic-free deionized water (Shelton, 1994). For other organic samples, about 20 mL of organic-free water and 125 mL of sample water are used for preconditioning (Sandstrom, 1995). For inorganic samples, the filter is preconditioned with 1 L of deionized water followed by 25 to 50 mL of the sample water (Horowitz and others, 1994). Filtration procedures are described in detail by Horowitz and others (1994), Ward and Harr (1990), and Shelton (1994). All samples must be filtered into the appropriate bottle type. A complete listing of filtration requirements and bottle types may be obtained from the NWQL Services Catalog (Timme, 1995), which is also available on the World-Wide Web at http://wwwnwql.cr.usgs.gov/USGS.

Preservation. The final step in sample processing is preservation of the sample. Preservation techniques retard the chemical, physical, or biological changes that occur after the sample is removed from its source (Ward and Harr, 1990). Some sample types require no preservation, some need only to be chilled, and some require the addition of a preservative. Because preservation methods are constantly changing, consult preservation requirements in the current NWQL Services Catalog (Timme, 1995), also available on the World-Wide Web at http://wwwnwql.cr.usgs.gov/USGS.

4.2 Ground-water samples

The main goal of collecting ground-water samples is to obtain samples that accurately represent the water quality of the aquifer. However, ground-water quality tends to have significant spatial variations that make it difficult to characterize the water quality of an aquifer from only a few samples. Also, because ground water is usually isolated from the atmosphere, special techniques may be required to minimize oxidation or degassing of reduced or volatile chemical species. For some unstable constituents, field measurements are required to produce unbiased and representative data (Wood, 1976).

4.2.1 Site-selection criteria

One of the most important QA activities associated with ground-water sampling is selection of sampling sites (wells) that will provide samples capable of meeting water-quality data objectives set for the study. Types of wells that are suitable for use in various types of ground-water studies and associated selection criteria are discussed in Claasen (1982) and Lapham and others (1997). Criteria used to select wells for the NAWQA ground-water studies are discussed in Hardy and others (1989) and Lapham and others (1995). Well-selection criteria for projects involved in contaminant monitoring or regulatory response may have very specific well-selection criteria that are set by the USEPA, OEPA, or other agencies. The minimum well-selection criteria for any ground-water project done in the Ohio District includes sufficient documentation to (1) determine the hydrogeologic zone from which the ground water is being withdrawn and (2) ensure that materials and techniques used to construct the well are suitable for sampling the constituents of concern.

4.2.2 Site documentation

Wells selected for sampling are to be adequately documented (see section 5.1). This includes entering site data into the USGS Ground-Water Site Inventory (GWSI) (Mathey, 1989), as described in section 5.1.1 of this report. If a project objective is to relate ground-water quality to land use, then land-use and land-cover field sheets must be completed. Examples of land-use and land-cover forms used by the NAWQA program are found in Koterba and others (1995, p. 88-89). Wells selected as potential sampling sites must be visited before completion of the GWSI site file to verify that information obtained from drillers' logs or other sources is correct. Copies of well logs obtained from the Ohio Department of Natural Resources are filed with the original GWSI form and in project files. Before sampling, obtain permission to sample the well (use WRD form 90.034— "Permission to Sample") and arrange for site access. It is also recommended that photographs of the well site be taken to document well characteristics and local land-use practices near the well (Hardy and others, 1989; Lapham and others, 1995). Keep one set of photographs with the original GWSI form and a second set in the site file.

4.2.3 Well construction and development criteria

For some projects, new wells may be constructed. Selection of appropriate construction and development methods and casing and grout materials used will be project specific. It is important that the techniques and materials used do not affect the quality of samples with respect to the analytes of interest. For example, avoid using drilling techniques that introduce water (or other fluids) into the aquifer. A complete review of literature covering well-installation techniques is beyond the scope of this report. Instead, the reader is directed to the following sources and references contained therein: Aller and others (1991), Driscoll (1986), Dumouchelle and others (1990), Hardy and others (1989), Lapham and others (1995, 1997) and Ohio Environmental Protection Agency (1995). If new wells are to be installed, the District Ground-Water Specialist is to be consulted.

For new and existing wells, the materials to be used for the well casing and to seal the annular space surrounding the casing are to be evaluated for their potential effects on water quality (Claasen, 1982). Certain casing materials have been shown to sorb trace elements and volatile organic compounds, whereas some plastic casing materials and the compounds used to join casing sections may leach volatile organic compounds (Gillham and O'Hannesin, 1990; Lapham and others, 1997). Teflon and 316 stainless steel are rated highest with respect to maintaining sample integrity. However, because of high cost (and the poor strength characteristics of Teflon), most monitoring wells are constructed with 40 or 80 schedule polyvinyl chloride (PVC) casing (Driscoll, 1986; Aller and others, 1991; Lapham and others, 1997). PVC casing with threaded, not glued joints should be used to avoid contamination from adhesives used to glue the joints (Lapham and others, 1997). Common materials used to grout the annular space above the screened interval include bentonite, cement, and bentonite-cement mixtures. If the seal is not installed properly, grout materials can affect the pH and major ion chemistry of ground-water samples collected from the well. Sorption of volatile organic compounds and trace elements onto grout materials, such as bentonite, can also occur.

The length of the well screen open to the aquifer can also influence the representativeness of ground-water quality samples. Reilly and others (1989) have shown that substantial well-bore flow may occur in long-screened wells in some hydrogeologic settings. Similar results have been reported by other workers (Robbins, 1989; Martin-Hayden and others, 1991; Robbins and Martin-Hayden, 1991). Sharp variations in aquifer chemistry can occur over small vertical intervals. Therefore, unless the project goal is to examine the quality of ground water obtained from wells open to large intervals of the aquifer or supply wells with long or multiple screens, wells with short screens or open intervals are preferred for most ground-water-quality studies. Placement of the screen will depend on study objectives. If contaminants are being introduced at land surface, wells near the source are to be screened at, or just below the water table. Further downgradient, well screens may be placed at greater depths. Use of multiple-depth well clusters, although more expensive than use of a single well with a long screen or open interval, is recommended in most situations. Hydrogeologic features, such as the presence or absence of confining units or highly transmissive fracture zones in consolidated aquifers, can also affect where well screens are placed.

Newly completed wells must be developed to rid the casing and screen of fine-grained particulate matter (drilling mud and cuttings), as well as to remove fine-grained material from the sand and gravel pack around the well screen. Existing wells that have not been sampled recently may also require development because their screens may be clogged with bacteria, chemical precipitates, or fine sediments (Claasen, 1982). Techniques used for well development are described in Driscoll (1986), Aller and others (1991), and Lapham and others (1995, 1997).

4.2.4 Ground-water sampling equipment

Once wells are selected and documentation is completed, sampling equipment is selected and tested to ensure that representative ground-water samples can be obtained. Selection criteria for ground-water sampling equipment includes type of materials, cost, pump capacity (flow rate), equipment durability, and portability. Evaluate materials used in the sampling equipment with respect to possible chemical effects (leaching/adsorption and gas permeability) and physical effects (abrasion and flexibility under variable weather conditions). The two main classes of ground-water sampling equipment are bailers and pumps. Of these, bailers are generally less expensive and can be obtained in a variety of designs and materials appropriate for sampling most analytes. However, bailers are inconvenient for sampling wells with large purge volumes, and many researchers have questioned the ability of bailers to obtain representative ground-water samples, particularly with respect to trace elements and volatile organic compounds (Martin-Hayden and others, 1991; Robbins and Martin-Hayden, 1991; Barcelona and others, 1994). Hence, in most situations, the use of a pump to sample ground water is recommended.

Another class of ground-water samplers are discrete-depth samplers that are emplaced in the subsurface by cone-penetrometer testing rigs or drilling rigs (Edge and Cordry, 1989; Zemo and others, 1994). Devices used to collect discrete-depth samples are shielded by a retractable sleeve that is pulled back when the target zone is reached; depths up to 50 ft can be sampled by this technique at intervals as small as 1 ft. Sampling can be done at successively greater depths as the device is advanced downward. This method has been shown to be very cost effective for site characterization investigations where delineation of shallow contaminant plumes in unconsolidated aquifers is the primary objective (Zemo and others, 1994).

The decision of which pump to use for a particular project is made only after consultation with the District Ground-Water specialist or other personnel familiar with the use and operation of the various pump types. A list of pumps available for project work and their current condition is currently (1997) being compiled by District personnel and can be obtained from the District Ground-Water Specialist. For projects involved in long-term monitoring, the use of dedicated pumps and tubing in each monitoring well can result in significant savings and a reduced risk of cross-contamination between wells. Comparison of the ability of different types of ground-water sampling devices to deliver representative ground-water samples with respect to various analyte classes (organics and inorganics) has recently been reviewed by Parker (1994). This review article contains an extensive reference list that cites field and laboratory studies; short descriptions of the main findings of many of the pre-1990 papers cited by Parker (1994) are given in Dumouchelle and others (1990). Koterba and others (1995, p. 17) list low-discharge, submersible, portable pumps that are approved for use by the NAWQA program for sampling ground water for a wide variety of constituents including volatile organic compounds (VOC's), pesticides, major ions, nutrients, radionuclides, and selected trace elements. Tests of the recovery efficiencies of these pumps for various inorganic and organic constituents have been done by personnel of the Office of Ground Water (Franceska Wilde, U.S. Geological Survey, written commun., 1994).

4.2.5 Well-purging criteria

To ensure that representative ground-water samples are collected, it is necessary to purge stagnant water from the well bore and replace it with water freshly drawn from the surrounding aquifer. Before pumping, the water level of the well must be measured and the volume of water in the casing (including the screened interval) must be determined and recorded on a field sheet (see Koterba and others, 1995, p. 96, for example water-level measurement and purge form). The pump intake is placed near the top of the water column during purging and lowered to the top of the screened interval before the sample is collected. For shallow wells, the rate of pumping is to be as uniform as possible during purging and sampling and is to be done at a low flow rate. The purge and sampling flow rate recommended for the NAWQA program is about 0.1 gal/min (or 500 mL/min) (Koterba and others, 1995). Such low flow rates are recommended because higher flow rates can cause turbulence, degassing, entrainment of colloids and small particles, excessive drawdown, and mixing of water from different parts of the aquifer in the wellbore (Reilly and others, 1989; Powell and Puls, 1993; Barcelona and others, 1994; Koterba and others, 1995). All of the above-listed processes can lead to collection of nonrepresentative ground-water samples; use of the low flow rate during purging and sample collection minimizes these effects. Initial purging at a higher rate is acceptable for deeper wells whose purge volumes are high; however, before the sample is collected, the purge rate is to be gradually reduced to the recommended flow rate and selected water-quality characteristics are to be monitored for stability. Procedures for dealing with wells where standard purging procedures cannot be followed (including purging and sampling of low-capacity wells) are discussed in Koterba and others (1995).

Monitor temperature, pH, dissolved oxygen (DO) (unless H_2S is present) and specific conductance during purging and record values on field sheets at regular intervals (for example, 5-minute intervals). Turbidity measurements, if required by state or Federal regulatory agencies for the project, are also made at regular intervals. The NAWQA protocol requires regular turbidity measurements for any wells where ground-water samples are being collected for trace-element analysis (Koterba and others, 1995). Perform all measurements (except turbidity) in a flowthrough chamber that isolates ground water and probes from the atmosphere. The flowthrough chamber is attached as close to the pump discharge as possible to minimize temperature fluctuations and outgassing from the tubing. Descriptions of the basic design elements for flowthrough chambers are given in Hardy and others (1989). Flowthrough chambers are available from the Hydrologic Instrumentation Facility; alternatively, many multiple-parameter water-quality instruments offer accessory flowthrough chambers that attach directly to the instrument.

Determination of when purging is complete is based on volumetric criteria and stability of water-quality characteristics. The OWQ recommends that three to five casing volumes be removed (three minimum) and that three successive field measurements of selected water-quality characteristics are stable within the limits given in table 4.2.5-1 (Wilde and others, 1998d; Koterba and others, 1995).

Table 4.2.5-1. Stability criteria for well purging for selected water-quality characteristics

Characteristic	Units	Stability criteria
pH	Standard units	± 0.1
Specific conductance	Microsiemens per centimeter (µs/cm)	> 100 µs/cm, within 3% < 100 µs/cm, within 5%
Dissolved oxygen	Milligrams per liter (mg/L)	± 0.3 mg/L
Temperature	Degrees Celsius (°C)	± 0.2°C
Turbidity	Formazine or nephelometric turbidity units (FTU or NTU)	< 5 FTU or NTU

Of these criteria, demonstrated stability of the water-quality characteristics is the most important criterion, although it is recognized that natural variability in ground-water quality or well-bore hydraulic effects will prevent stabilization of some (or all) of these characteristics in some aquifers and wells. For these situations, use of the volumetric rule is recommended. Water-quality characteristics measured immediately before sample collection begins are to be recorded on the field sheets.

Recently, micropurging techniques have been developed that involve purging of a small percentage of the total casing volume by use of dedicated pumps at very low pumping rates (Kearl and others, 1994; Puls and Paul, 1995). These techniques have not been used by the Ohio District, but they have significant potential for use in studies where disposal of large volumes of contaminated purge water is an issue.

4.2.6 Ground-water sample collection and processing

Once purging is complete, the sampling device intake is lowered to just above the well screen (or the base of the cased interval in bedrock wells) and collection of ground-water samples can begin. Collect and preserve subsamples for constituents subject to volatilization or contamination first. USEPA and NAWQA protocols recommend that organic compounds (volatile and semivolatile organic compounds, organic carbon, phenols) be sampled for first, followed by collection of samples for dissolved and total inorganic analytes (trace elements, major ions, nutrients, alkalinity). These in turn are followed by collection of subsamples for cyanide, radon and other radionuclides, and isotopic and various environmental tracers such as chlorofluorocarbons (Koterba and others, 1995; Ohio Environmental Protection Agency, 1995; Wilde and others, 1998d). Samples for onsite determination of alkalinity are to be collected at the same time the sample for anions is collected.

Good field practices to be followed during sample collection and processing include the use of gloves and precleaned equipment and tubing, minimization of the number of sample-processing steps, decontamination of equipment after sampling, and collection of field blanks to assess overall ability to collect clean samples (Koterba and others, 1995; Wilde and others, 1998d). If possible, plan the field trip so that wells are sampled in the order of lowest to highest concentrations of target analyte(s). This strategy may involve sampling from upgradient to downgradient wells, from wells with low-ionic-strength water to high-ionic-strength water, or from pristine wells to contaminated wells. If wells are to be sampled several times as part of a regular monitoring program, use of dedicated pumps may be a practical and cost-effective alternative to use of a single pump that must be cleaned after each use and regularly checked for cross-contamination.

Detailed descriptions of processing techniques for ground-water samples is beyond the scope of this report, however, the reader is referred to Claasen (1982), Hardy and others (1989), OEPA (1995), and Wilde and others (1998e) for discussions of collection, filtration, and preservation techniques for ground-water samples. Protocols issued for ground-water sampling for the NAWQA program are described by Koterba and others (1995). In addition, sample-processing guidelines given in the surface-water inorganic protocol by Horowitz and others (1994) apply to ground-water projects that seek to interpret trace-element or nutrient data at the microgram-per-liter level.

4.3 Biological samples

Assessments of occurrence, distribution, and relative abundance of aquatic biota; the concentrations of fecal-indicator bacteria; and physical and chemical quality of stream habitats are important aspects of comprehensive water-resources investigations. Aquatic organisms such as algae, macroinvertebrates, and fish can be sampled for a variety of objectives including studies of biological water-quality conditions, human and aquatic ecosystem health, and species occurrence and distribution.

Many collecting and processing methods have been developed to fit a variety of sampling locations and objectives. A large number of scientific articles have been written about sampling and study design for projects involving algae, macroinvertebrates, and fish. Design of studies of aquatic biota and habitat requires careful thought and planning because the sampling error can often exceed the natural variability in populations, making data interpretation difficult or impossible. Averett (1973) and Rosenberg and Resh (1993) are excellent introductory references.

Collection methods vary primarily with the size of the stream (wadable or non-wadable) or lake. Tables 4.3.1-1 to 4.3.5-1 contain references to methods accepted within the USGS, other Federal agencies, the States of Ohio, Michigan, and New York, and academic institutions and scientific organizations. These methods are suitable in freshwater streams and lakes. The guidelines encompass methods for collection, processing, preservation, storage, analysis, and taxonomic identification and enumeration of algae, invertebrates, and fish in lakes and streams, and habitats of lakes and streams. For background information, the publications of Pennak (1991), Rosenberg and Resh (1993), and Hynes (1970) are recommended.

4.3.1 Algae

Algae are present in a variety of stream and lake habitats and microhabitats. The choice of the type of habitat to sample in streams (riffles, pools, runs) and lakes (littoral, or nearshore; limnetic, or open water; epilimnetic, or upper, warm, lighted, layer of open water; and hypolimnetic, or lower, cool, dark layer of open water) is related to project objectives. Classification of wetlands requires even finer determinations of aquatic and semiaquatic habitats (U.S. Fish and Wildlife Service, 1979, p. 13).

Periphyton and macroalgae are the most abundant type of algae in streams. Both qualitative and quantitative samples can be collected from many different stream habitats and microhabitats. Samples can be collected using artificial substrates as well. Microhabitats include but are not limited to periphyton growing on wood (epidendritic), on rooted and floating aquatic and semiaquatic plants (epiphytic), on rocks (epilithic), on sand (epipsammic), on mud (epipelic), and on other debris and detritus (epibenthic). Some periphyton are loosely attached to, or unattached but associated with, objects in the water. Littoral areas of lakes have abundant periphyton in several microhabitats. A variety of accepted sampling equipment and collection methods have been developed (table 4.3.1-1). A review of methods for collection of periphyton can be found in Wheeler and others (1979).

Phytoplankton, the algae suspended in water, are the most abundant type of algae in large rivers, natural lakes, ponds, and reservoirs. Sampling of these communities can be done by use of the same equipment used to collect point water-quality samples in standing water, such as a Kemmerer or Van Dorn sampler (table 4.3.1-1). Nets can also be towed through the water column to obtain a depth-integrated sample. Nets selected for use, such as the Wisconsin-style net, are designed and constructed to minimize drag at the net opening. Mesh-size of tow nets determines the size of phytoplankton that are sampled. Microplankton (smaller than 20 µm) are best sampled by use of a point sampler such as a Kemmerer or Van Dorn bottle (table 4.3.1-1).

The acceptable level of bias and variability necessary to achieve DQO's is determined by the project chief before collecting samples. Data quality is dependent on at least three factors: (1) method variability, (2) method bias, and (3) measurement of the variability of the numbers and species of algae in the study area. Estimates of method variability are based on replicate counts of algal cells, filaments, or colonies from a sample that has been split into subsamples. Bias is determined by repeatable and correct taxonomic identifications and counts among replicate or split samples. To assess bias and variability of counts and identifications, one in every 10 to 20 samples are collected, split into two or more equal parts, enumerated, and taxonomically identified. Replicates for species identifications and enumerations should agree within +/- 10-15 percent to ensure data quality.

Collection of a sufficient number of samples to adequately describe spatial variability of organisms in a study area is estimated through the collection and analysis of a predetermined number of samples from the sampling reach. For example, somewhere between 5 and 10 replicates may be needed to estimate statistical population parameters such as mean and median number of cells per square meter to within +/- 25 percent.

In most cases, taxonomic identification by the analyst can be made to the species level for algae. Verify questionable taxonomic identifications of algae with an independent expert. Specimens that have been verified by taxonomic experts can be used to develop a collection of reference or voucher specimens. These verified samples provide a taxonomic check on current work. Experts in species confirmation work must be identified early on in the planning phase of the project for budgeting and contracting purposes.

Table 4.3.1-1. Summary of methods of collection, processing, preservation, enumeration, and analysis of algae samples from streams and lakes in Ohio and adjacent states

[APHA, American Public Health Association and others; HPLC, high performance liquid chromatography]

Biological community type	Type of method	Method reference
	Sample collection methods	
Benthic periphyton	Natural substrates: from rocks and other hard surfaces, from vegetation, sand, silt, wood, and other microhabitats	Britton and Greeson, 1987, p. 131-132 Porter and others, 1993, p. 14-21
	Artificial substrates	APHA and others, 1992; p. 10-28 Britton and Greeson, 1987, p. 132 Porter and others, 1993, p. 22-24
Phytoplankton	Grab sample with a bottle or sampler: Van Dorn Sampler Kemmerer	APHA and others, 1992, p. 10-2 to 10-5 Britton and Greeson, 1987, p. 99-101
	Netted sample by towing net through the water column: Wisconsin style Standard style	APHA and others, 1992, p. 10-5 to 10-8 Britton and Greeson, 1987, p. 100-103
Macroalgae	Collection methods	Aloi, 1990 APHA and others, 1992, p. 10-41 to 10-47 Littler and Littler, 1985 Porter and others, 1993
	Primary productivity (growth rate)	
All algae	Standing-water methods: Dissolved oxygen method Carbon-14 method	APHA and others, 1992, p. 10-32 to 10-33 Britton and Greeson, 1987, p. 281
	Flowing-water methods	APHA and others, 1992, p. 10-33 to 10-39 Britton and Greeson, 1987, p. 285
	Sample processing, storage, and preservation methods	
All algae	Preservatives: glutaraldehyde, formaldehyde, Lugol's solution, glacial acetic acid, copper sulfate, other	APHA and others, 1992; p. 10-5 and 10-28 Britton and Greeson, 1987; p. 99 Porter and others, 1993; p. 27
	Sample preservation, and processing for chlorophyll and other pigments: Filter within 2 hours of collection. Chill at 1° -4° C before filtration. After filtration, freeze filters on dry-ice or in freezer for no longer than 6 months before analysis.	APHA and others, 1992, p. 10-17
	Storage and containers Glass or plastic	APHA and others, 1992; p. 10-17 Britton and Greeson, 1987; p. 99
	Biomass determination methods	
All algae	Biovolume	APHA and others, 1992, p. 10-13
	Sample processing and analysis for chlorophyll pigments (HPLC and spectrophotometric methods only)	APHA and others, 1992, p. 10-17 to 10-22 Britton and Greeson, 1987, p. 219-247
	Determination of biomass by gravimetric analyses (ash-free dry weight and seston)	APHA and others, 1992, p. 10-22 to 10-24, and 10-30 to 10-31 Britton and Greeson, 1987, p.127-129
	Enumeration methods	
All algae	Inverted microscope	Britton and Greeson, 1987, p. 143-144

Table 4.3.1-1. Summary of methods of collection, processing, preservation, enumeration, and analysis of algae samples from streams and lakes in Ohio and adjacent states—Continued

[APHA, American Public Health Association and others; HPLC, high performance liquid chromatography]

Biological community type	Type of method	Method reference
	Counting cell methods	APHA and others, 1992, p.10-13 to 10-16 Britton and Greeson, 1987, p. 105-108
	Light microscope-permanent slides	APHA and others, 1992, p. 10-10 to 0-12 Britton and Greeson, 1987, p. 141-142
	Taxonomic identification methods	
All algae	Diatoms	Britton and Greeson, 1987, p. 314- 317 Patrick and Reiner, 1966 Prescott, 1962
	Green, blue-green, golden-brown, brown, and yellow algae	Prescott, 1962, 1970 Smith, 1950 Taft and Taft, 1971 Whitford and Schumacher, 1973

4.3.2 Aquatic invertebrates

Aquatic invertebrates are organisms such as insect larvae, adult insects, and various classes of worms, mussels, clams, and snails that inhabit surface water for all or part of their life cycle. Collecting and processing samples containing these organisms for subsequent identification and enumeration can be time consuming and complex. Like other aquatic organisms, macroinvertebrate species prefer specific habitats and are associated with those habitats when sampled. In lakes, these habitats include limnetic, profundal (bottom), and littoral areas. In streams, these habitats include riffles, pools, runs, stream banks, island or bar edges, tree roots, and other woody debris (Meador and others, 1993a).

In most instances, macroinvertebrates that are benthic (live in or on the bed material and other habitats of streams) are insect larvae and freshwater clams and mussels. Insects and freshwater clams and mussels represent the largest biomass of invertebrates in streams and rivers. Zooplankton such as microcrustaceans, represent the largest biomass of invertebrates in lakes. Consider which component of the invertebrate community is most important for meeting project objectives.

The choice of the type of habitat from which to collect invertebrate samples from streams, wetlands, and lakes is related to project objectives (table 4.3.2-1). Within a habitat, samples can be collected from many different microhabitats. These microhabitats include but are not limited to different grain sizes such as clay, silt, sand, cobbles, gravels, and boulders or bedrock. Other microhabitats include living and dead wood, roots, stems, leaves and leaf particles, and other organic material such as rooted emergent and submergent aquatic plants and macroalgae (Cuffney and others, 1993a; Meador and others, 1993b).

Qualitative and (or) quantitative samples can be collected by use of artificial substrates as well as from natural substrates. A variety of nets, dredges, and other equipment can be used to collect stream macroinvertebrates (table 4.3.2-1) (Cuffney and others, 1993b; Ohio Environmental Protection Agency, 1989).

The sampling and identification methods must be closely matched to project objectives to obtain adequate assessment of macroinvertebrate communities. Often, many samples (more than five) must be collected to arrive at a good estimate of relative abundance (Rosenberg and Resh, 1993, p. 159).

As with algae, data quality for samples and analyses of macroinvertebrates is dependent on at least three factors: (1) method variability, (2) method bias, and (3) variability of the spatial distribution of organisms in the study area. The variability in the spatial distribution of macroinvertebrates in a study reach is estimated by the collection and analysis of a predetermined number samples from the same area. Averett (1973) provides an excellent discussion of the integration of study design, DQO's, and determining the number of samples to be collected. Determine the acceptable level of bias and variability necessary to achieve project objectives before sampling. For example, somewhere between 5 and 10 replicates may be needed to estimate statistical population parameters such as mean and median number of organisms per square meter to within +/- 25 percent.

Estimates of bias and variability in counting (enumeration) and identification can be made for macroinvertebrate samples. Cuffney and others (1993a) recommend splitting a single sample into different fractions for identification and enumeration. A similarity index value is computed on the basis of the differences between counts and identifications obtained from the various fractions (Cuffney and others, 1993b). For the NAWQA program, a minimum numerical value for an index of similarity of 0.90 between sample fractions is set as a DQO (Cuffney and others, 1993b, p. 60). The DQO's are set for projects by the project aquatic biologist in conjunction with the project chief.

In many cases, taxonomic identification cannot be made to the species level for macroinvertebrates because key morphological features of immature specimens are often undeveloped and unrecognizable. Consequently, identifications are often limited to order, family, or genus rather than species. Verify questionable taxonomic identifications with an independent expert. Specimens that have been verified by taxonomic experts can be used to develop a collection of reference or voucher specimens. These verified samples provide a taxonomic check on current work. Experts in species confirmation work must be identified early on in the planning phase of the project for budgeting and contracting purposes. For the NAWQA Program, all specimens are kept in a reference collection by the Biological Unit (BU) at the NWQL.

A Scientific Collectors permit is required to collect freshwater mussels and their shells in Ohio and most neighboring States including Indiana, Michigan, Pennsylvania, and New York. Application for a Scientific Collectors permit must be made to the Ohio Division of Wildlife or other similar agency in adjoining States. Because freshwater mussels are among the most diverse and endangered aquatic organisms globally and in North America, collecting threatened or endangered mussels requires special permission from the permitting agency. Avoid unnecessarily dislodging mussels from their habitats.

Methods are available for collection and processing of invertebrate tissue samples for analysis of hydrophobic substances such as polychlorinated biphenyls (PCB), DDT and its degradation products, and chlordane (Crawford and others, 1992). Although depuration of organisms (emptying of the digestive tract) is recommended for NAWQA samples (Crawford and others, 1992), it may not be necessary for all projects.

Sample sorting and processing of samples preserved with formalin or ethanol must be done under a hood or outside to ensure good ventilation.

Table 4.3.2-1. Summary of methods of collection, processing, preservation, enumeration, and analysis of invertebrate samples from streams and lakes in Ohio and adjacent states

[DNR, Department of Natural Resources; EPA, Environmental Protection Agency; APHA, American Public Health Association]

Biological community type	Type of method	Method reference (unless numbered, each type of method can be found in all references)
	Sample-collection methods	
Benthic macro-invertebrates	Natural substrates in streambeds, from vegetation, sand, silt, wood, and other microhabitats: Surber-type samplers (1, 2, 3) Box, drum, and stream bottom-type (2,3) Kick samples (1,2,3,4,5) Dip-net samples (4) Dredges (2,3) Corers (2,3)	(1) Bode and others, 1993, p. 13 (2) Britton and Greeson, 1987, p. 151 (3) Cuffney and others, 1993b (4) Michigan DNR, 1991, p. 13-14 (5) Ohio EPA, 1989, Procedure No. WQPA-SWS-3, Revision 6, Part A p. V-1-2 to V-1-5
	Artificial substrates: Multiplate sampling (1-4) Rock and basket sampling (2)	(1) APHA and others, 1992, p. 10-28 (2) Britton and Greeson, 1987, p. 155-158 (3) Bode and others, 1993, p. 10-11 (4) Ohio EPA, 1989, Procedure no. WQPA-SWS-3, Revision 6, Part A p. V-1-2 to V-1-5
	Drift nets	Britton and Greeson, 1987, p. 163
Zooplankton	Grab sample with a bottle or sampler	APHA and others, 1992, p. 10-2 to 10-5 Britton and Greeson, 1987, p. 117-120
	Netted sample by towing net through the water column	APHA and others, 1992, p. 10-5 to10-8 Britton and Greeson, 1987, p. 100-103
	Sample containers, processing, preservation and storage methods	
All invertebrates	Preservatives: formaldehyde, formalin, ethyl alcohol (1-4)	(1) APHA and others, 1992, p. 10-5 and 10-28 (2) Britton and Greeson, 1987, p. 99 (3) Cuffney and others, 1993b (4) Ohio EPA, 1989, Procedure WQPA-SWS-3, Revision 6, Part A p. V-1-2 to V-1-5
	Storage and containers	Britton and Greeson, 1987, p. 99
	Processing samples to remove detritus and sorting of organisms to order level before identification and enumeration	APHA and others, 1992, p. 10-64 to 10-65 Cuffney and others, 1993a Britton and Greeson, 1987
	Biomass and tissue analysis methods	
Macroinvertebrates	Drying and gravimetric method for biomass measurement	Britton and Greeson, 1987, p. 165-168
	Collection and processing for tissue analysis	Crawford and others, 1992
	Taxonomic identification and enumeration methods	
All Invertebrates	Enumeration	APHA and others, 1992, p. 10-10 to 10-16 Britton and Greeson, 1987, p. 141- 144 Ohio EPA, 1989, p V-1-11 to V-1-14
	Taxonomic identification (Additional references to monographs and other taxonomic publications are located in the method references)	APHA and others, 1992, p. 10-65 Britton and Greeson, 1987; p. 314- 317 Merritt and Cummins, 1984 Ohio EPA, 1989, p V-1-11 to V-1-14 Pennak, 1991 Thorpe and Covich, 1990

4.3.3 Fish

Fish are the most common vertebrates found in streams, rivers, and lakes. Sampling programs must be designed to collect fish in the specific habitats they prefer. These habitats include open water and littoral areas of lakes; stream riffles, pools, runs, stream banks, and island or bar edges; and tree roots and other woody debris (Meador and others, 1993a).

The design of sampling programs and reproducibility of results are interrelated because variability, bias, and representativeness are dependent on at least three factors: (1) the quality of the sample analysis (count, weight, and length of each specimen, (2) correct species identification, and (3) variability of the spatial distribution of organisms in the study area. Replicate samples are impractical to collect in assessments of fish communities.

The effectiveness of electrofishing technique is related to appropriateness of sampling equipment, size and depth of the stream, level of effort (time spent electrofishing), length of the stream sampled, number of habitats sampled, and power sent by the electrofishing unit and received by the fish. Unlike other methods, representativeness of sampling fish communities is done by measuring the time spent electrofishing: Spend a minimum of 1,300 to 1,600 seconds electrofishing in a reach that is approximately 500 ft in length; as much as 2,000 seconds may be necessary in slow-moving reaches or where there are woody snags and debris (Ohio Environmental Protection Agency, 1989).

Voltage emanating from the generator used for shocking fish must be adjusted according to water temperature and specific conductance to ensure a representative sample as well as reduce fish injury. The use of a control box is necessary to make the proper voltage adjustments to the generator (Burkhardt and Gutrueter, 1995). Discussions of level of electrofishing effort and recommended equipment for various sized streams are discussed in Ohio EPA (1989), Burkehardt and Gutrueter (1995), and Cuffney and others (1993a). Take extra care to minimize fish injury and mortality during collection, measurement, and identification: When electrofishing, keep live wells or holding tanks in boats and barges aerated and as cool as possible.

Sampling fish is a labor-intensive endeavor, and most identification and other measurements must be carried out in the field rather than the laboratory. It is essential that an ichthyologist be a part of the sampling team. Inexperienced individuals will not be able to identify the majority of fish to species level in the field. The result is unacceptable; that is, an unnecessarily large number of specimens must be taken, preserved, and transported back to the laboratory for taxonomic identification or verification. Field assistants and inexperienced personnel must be accompanied by an ichthyologist and field crew leader when sampling fish. For the NAWQA Program, the field-crew leader or ichthyologist must be certified through the U.S. Fish and Wildlife Service in safety and operation of the electrofishing unit.

Voucher specimens are collected and preserved to form a reference collection for future studies and for taxonomic verifications. The only traceable method to confirm fish to species level is through a reference collection and verification of species by an independent taxonomic expert. For the NAWQA Program, all voucher specimens are kept in a reference collection at the District Office, and verifications are done by local taxonomic experts or by contract laboratories with the Biological Quality Assurance Unit at the NWQL. Fish identifications done in the laboratory on specimens preserved in formalin or ethanol must be made under a hood or where ventilation is adequate.

When collecting samples for subsequent tissue analysis, proper precautions must be taken to ensure sample integrity (protecting the samples from extraneous contaminants in the environment). The proper type of material, such as Teflon, plastic, or glass, is determined by the nature of the analysis. Secure containers must be used to store and ship samples before analysis.

Table 4.3.3-1. Summary of methods of collection, field identification, enumeration, and preservation of fish from streams and lakes in Ohio and adjacent states

[EPA, Environmental Protection Agency; APHA, American Public Health Association and others]

	Type of method	Method reference (unless numbered, each type of method can be found in all references)
	Sample-collection, identification, and measurement methods	
Fish from wadable and non-wadable streams and lakes	Barge-mounted electrofishing technique with or without seine (4) Backpack electrofishing techniques (1,3,4) Boat-mounted electrofishing techniques (4): Straight electrode array Circular electrode array Voltage specifications (2)	(1) Britton and Greeson, 1987, p. 199-205 (2) Burkehardt and Gutreuter, 1995 (3) Meador and others, 1993a (4) Ohio EPA, 1989
Fish from wetlands and lakes	Nets (1,2): Fyke net Hoop net Seines (1,2,3) Traps (2)	(1) Britton and Greeson, 1987, p 202-205 (2) Meador and others, 1993a (3) Ohio EPA, 1989
Tissues and filets	Electrofishing Net Handling and processing	Crawford and others, 1992 Ohio EPA, 1989
All fish	Taxonomic identification	Kuehne and Barbour, 1983 Ohio EPA, 1989, p. V-4-31 and V-4-32 Smith, 1985. Trautman, 1981
	Biomass Length Weight	Britton and Greeson, 1987, p. 203-205 Meador and others, 1993a Ohio EPA, 1989
	Voucher specimens	Ohio EPA, 1989, p. V-4-31 and V-4-32
	External deformities, eroded fins, lesions, tumors (DELTS), and fish-disease measurements methods	
	DELTS and disease identification fish kills	Meador and others, 1993a Meyer and Barclay, 1990 Ohio EPA, 1989, p. V-4-16 to V-4-18
	External evidence of fish kill Preparing and transporting samples	Meyer and Barclay, 1990
	Sample containers, processing, preservation and storage methods	
All fish	Preservatives: formaldehyde, formalin, ethyl alcohol	APHA and others, 1992, p. 10-5 and 10-28 Britton and Greeson, 1987, p. 99 Meador and others, 1993a Ohio EPA, 1989, Procedure WQPA-SWS-3, Revision 6, Part A p. V-1-2 to V-1-5
	Storage and containers	APHA and others, 1992 Britton and Greeson, 1987, p. 99
	Collection and processing methods of samples for analysis of fish tissue	
All fish	Collection and processing of whole fish	Crawford and others, 1992 Ohio EPA, 1989
	Collection and processing of edible filets	Ohio EPA, 1989

A Scientific Collectors permit is required to collect fish in Ohio and most neighboring States, including Indiana, Michigan, Pennsylvania, and New York. Application for a Scientific Collectors permit must be made to the Ohio Division of Wildlife or other similar agency in adjoining States. Collecting in streams in which threatened or endangered fish species are present may require special permission from the permitting agency.

4.3.4 Fecal-indicator bacteria

Fecal-indicator bacteria are the most frequently sampled bacteria in streams, lakes, and aquifers. Fecal bacteria are mostly intestinal in origin, although some may be found in the environment. For the most part, fecal-indicator bacteria do not represent the natural bacterial communities in the environment. Fecal-indicator bacteria do not necessarily cause disease but are associated with the presence of intestinal pathogens in water. As such, fecal-indicator bacteria are measures of the sanitary quality of water. They are the only direct measure of fecal pollution by warm-blooded animals, including humans. Fecal-indicator bacteria are used to monitor ambient water quality for recreational, industrial, agricultural, and water supply purposes. The concentration of these bacteria indicate whether water is safe for body-contact recreation or consumption, and free from disease-causing (pathogenic) organisms.

Test methods for fecal bacteria are of two types, membrane filtration (MF) methods and most-probable number (MPN) methods (table 4.3.4-1). In most cases, the MF methods are preferred over the MPN methods because of better quantification and ease of use and interpretation. MPN methods are used if the sample contains sufficient suspended sediment to clog the membrane filter or when toxic substances are present in the sample. Field methods for the collection of samples for bacterial analyses are described in Myers and Wilde (1997). A QA/QC program for membrane-filter analysis is described by Bordner and Winter (1978) and by D.N. Myers (U.S. Geological Survey, written commun., 1994).

Five MF methods are used in the Ohio District Laboratory for presumptive identification and enumeration of bacteria in the groups: total coliform, fecal coliform, fecal streptococci, enterococci, and *Escherichia coli (E. coli).* Verification tests for these five methods are also done (American Public Health Association and others, 1992). Verification tests confirm that colonies resulting from the presumptive test are the desired test organism.

The suitability of surface water for body contact is best determined by use of membrane-filter methods for enumeration of *E. coli*, fecal coliform, enterococcus, and fecal streptococcus. Although all four indicators can be used to assess recreational quality, only *E. coli* has been demonstrated to be strongly associated with gastroenteritis in swimmers. For assessment of drinking water supply and source-water suitability, total coliform analysis coupled with the *E. coli* method on NA-MUG[2] media is recommended.

Descriptions and definitions of the tests and their use are given in APHA and others (1992); Britton and Greeson (1987); Bordner and Winter (1978); and Myers and Wilde (1997). Troubleshooting problems with test results and a QA/QC program for fecal-indicator testing is described in Bordner and Winter (1978); APHA and others (1992); and D.N. Myers (written commun., 1994).

Methods for the collection, preservation, storage, processing, and analysis of samples for fecal bacteria are listed in table 4.3.4-1. Collection methods vary depending on whether samples are obtained from lakes, streams, or wells; whereas preservation, storage, processing and analysis methods are similar for all types of samples once they are collected. All collection, processing, and analysis of microbiological samples must be done by use of sterile techniques. Sterile techniques and procedures are discussed in APHA and others (1992).

---

[2] Nutrient agar-4-methylumbelliferyl-β-D-glucuronide.

Table 4.3.4-1. Summary of methods of collection, processing, enumeration, and computation of fecal-indicator bacteria from surface water and ground water

[EDTA, ethydimethytetrasodiumacetate; mTEC, *E. coli* culture media; NA-MUG; confirmation media; °C, degrees Celsius; µm; micrometers; APHA, American Public Health Association and others; EIA, enterococcus confirmation media: USEPA, U.S. Environmental Protection Agency]

	Type of method	**Method reference (unless numbered, each type of method can be found in all references)**
	Sampling location and method	
Streams	Equal width increment (2,3,4) Equal discharge increment (2,3,4) Point sample (1,3,4) Single vertical samples (3,4)	(1) APHA and others, 1992, p. 9-18 to 9-20 (2) Edwards and Glysson, 1988 (3) Myers and Wilde, 1997
Lakes	Point samples	APHA and others, 1992, p.9-19 Bordner and Winter, 1978 Britton and Greeson, 1987 Myers and Wilde, 1997
Wells	Sample collected with pump (4,5,6) Sample collected with bailer (4,5) Sample taken from a tap (1,2,3,4)	(1) APHA and others, 1992, p. 9-18 to 9-19 (2) Bordner and Winter, 1978 (3) Britton and Greeson, 1987 (4) Myers and Wilde, 1997 (5) USEPA, 1977
	Containers, preservatives, and holding time	
Containers	Glass, plastic, teflon; autoclavable	APHA and others, 1992 Bordner and Winter, 1978 Myers and Wilde, 1997
Preservatives	Chilling (1° to 4°C) Sodium thiosulfate EDTA	APHA and others, 1992 Bordner and Winter, 1978 Britton and Greeson, 1987 Myers and Wilde,1997
Holding time	Samples filtered as soon as possible but not more than 6 hours after collection	APHA and others, 1992 Bordner and Winter, 1978 Britton and Greeson, 1987 Myers and Wilde, 1997
	Primary culture methods	
Membrane filtration (Filter-pore size)	Total coliform (0.45 or 0.7 µm) Fecal coliform (0.7 µm) Fecal streptococci (0.45 µm)	APHA and others, 1992, p. 9-54 to 9-57 Bordner and Winter, 1978 Britton and Greeson, 1987
	Escherichia coli: m-TEC procedure (0.45 µm) (1) NA-MUG procedure (from total coliform at either 0.45 or 0.7 µm) (2) Enterococci (0.45 µm) (1)	(1) USEPA, 1985 (2) USEPA, 1991
Most Probable Number (MPN)	Total coliform Fecal coliform Fecal streptococci	APHA and others, 1992, p. 9-45 to 9-51 Bordner and Winter, 1978 Britton and Greeson, 1987
	Incubation times and temperatures	
Total coliform	24 +/- 2 hours at 35.0° +/- 0.5°C	APHA and others, 1992, p. 9-45 Bordner and Winter, 1978 Britton and Greeson, 1987
Fecal coliform	24 +/- 2 hours at 44.5° +/- 0.2°C	APHA and others, 1992, p. 9-61 Bordner and Winter, 1978 Britton and Greeson, 1987, p.

Table 4.3.4-1. Summary of methods of collection, processing, enumeration, and computation of fecal-indicator bacteria from surface water and ground water—Continued

[EDTA, ethydimethytetrasodiumacetate; mTEC, *E. coli* culture media; NA-MUG; confirmation media; °C, degrees Celsius; µm; micrometers; APHA, American Public Health Association and others; EIA, enterococcus confirmation media: USEPA, U.S. Environmental Protection Agency]

	Type of method	Method reference (unless numbered, each type of method can be found in all references)
Escherichia coli (E. coli)	2 hours at 35.0° +/- 0.5°C; then 22-24 hours at 44.5° +/- 0.2°C. Urea broth for 20 minutes	USEPA, 1985
Fecal streptococci	48 +/- 2 hours at 35.0° +/- 0.5°C	APHA and others, 1992, p. 9-71 Bordner and Winter, 1978 Britton and Greeson, 1987
Enterococci	48 +/- 2 hours at 41.0° +/- 0.5°C on m-E media; then 20 minutes at 41.0°C on EIA media	APHA and others, 1992, p. 9-71 Bordner and Winter, 1978 USEPA, 1985
	Confirmation and verification methods	
Total coliform	Biochemical tests	APHA and others, 1992, p. 9-65 to 9-67 Bordner and Winter, 1978
Fecal coliform	Biochemical tests	APHA and others, 1992; p. 9-65 to 9-67 Bordner and Winter, 1978
Escherichia coli	Biochemical tests	USEPA, 1985; p. 9-65 to 9-67
Fecal streptococci	Biochemical tests	APHA and others, 1992; p. 9-71 Bordner and Winter, 1978
Enterococci	Biochemical tests	USEPA, 1985

Policies of the USGS and OWQ are that all media and cultures must be sterilized before discarding. The use of an autoclave for sterilization of cultures and media after use is required as described in OWQ Technical Memorandum 93.10.

4.3.5 Habitat

Evaluation of aquatic habitat in streams and lakes is an important component of biological-water quality investigations. Habitat can be assessed in streams at four scales: basin, segment, reach, and subreach (channel and flood plain), and in microhabitats within the channel and flood plain. At basin and segment levels, most habitat assessments are done from maps or digital coverages. At the reach, channel, and flood-plain levels, most habitat assessments are done on site. Although literature on the subject is extensive, several references are used in the Ohio District for habitat assessment: Ashmore and others (1988), Harrelson and others (1994), Meador and others (1993b), Newbury and Gaboury (1993), Palcsak (1996), Wolman (1954), and Yuzyk (1986). Table 4.3.5-1 contains information and references to habitat methods at all scales.

Digital coverages that are fully documented and maps that have been published are recommended for habitat assessment. Many digital coverages at the scales appropriate for basin and segment assessment are available over the internet or by ftp; for example, digital line graph (DLG) data for streams, digital elevation models (DEM), land use and land cover—from the USGS's EROS (Earth Research Observation System) data center; ecoregions and river-reach files—from USEPA; soils and drainage areas—from the Natural Resources Conservation Service (NRCS); potential natural vegetation—from the U.S. Forest Service; wetlands—from the U.S. Fish and Wildlife Service. Regional data are generally available from the State of Ohio and other nearby States. The Ohio District maintains a set of digital coverages that can be used for basin and segment assessment.

Quality-assurance elements related to field measurements and sampling should follow those prescribed in the referenced documents. The most common types of errors associated with field measurements are transcription errors and incomplete collection of information. Use of standardized forms for data collection eliminates many problems associated with incomplete data collection, and provides for consistent reporting of habitat characteristics. Data entries in field notes and computer files must be verified independently before publication and use.

Specimens of aquatic, riparian, and flood-plain vegetation are kept in an herbarium for reference. All vegetation identified to species must be verified by an independent expert when the species is in question.

Table 4.3.5-1. Summary of methods of habitat assessment at six spatial scales

[GIRAS, Geographical Retrieval and Analysis System; NATSGO, National Soil Geographic Data Base; STATSGO, State Soil Geographic Data Base; NRCS, Natural Resources Conservation Service; MOSS, Map Overlay Statistical System; NWS, U.S. National Weather Service; RF3, U.S. Environmental Protection Agency River Reach; EROS, Earth Resource Observation System; DLG, Digital Line Graph Data; USGS, U.S. Geological Survey; NOAA, National Oceanographic and Atmospheric Administration; PCS, Pollution Control System; TRI, Toxic Release Inventory]

Format	Type of assessment	Source of data or reference (unless numbered, each type of method can be found in all references)
	Basin level: Scale is 1:250,000 to 1:2,500,000	
Map and (or) digital coverage	Ecoregion (6) Physiographic province (1) Land use and land cover (11) Geologic type (3) Soil type (5) Potential natural vegetation (4) Climate and runoff (7) Hydrography (8) Digital elevation models (9) Point source discharges (10) Toxic release inventories (10)	(1) Fenneman and Johnson, 1946 (2) GIRAS (Anderson and others, 1976) (3) King and Beikman, 1974 (4) Kuckler (1970) (5) NATSGO, STATSGO, NRCS (6) Omernick, 1987 (7) NWS, USGS (8) USEPA RF3; USGS EROS DLG (9) USEPA site files; NOAA, PCS system (10) USEPA, TRI data base (11) USGS EROS data center
	Basin level: Scale is 1:24,000 (7.5 minute series topographic maps)	
Maps	Drainage area (1) Drainage density (2) Drainage texture (2) Drainage shape (2) Stream length (2) Basin relief (2) Storage (2)	(1) Meador and others (1993b) (2) District drainage-area maps
Digital coverage	Wetlands (1,2,3) Drainage area (4)	(1) Frayer and others (1983) (2) Dahl and Johnson (1991) (3) MOSS (4) District drainage-area maps
	Segment level: Scale is 1:24,000 to 1:100,000	
Digital coverage	Stream networks	DLG, USGS EROS data center USEPA RF3
Maps	Segment code (3) Stream length (3) Elevation (3) Sideslope gradient (3) Segment gradient (3) Channel sinuosity (1,2,3,5,6) Stream order (1,2) Downstream link (4) Water-management feature (3)	(1) Horton (1945) (2) Leopold and others (1964) (3) Meador and others (1993b) (4) Osborne and Wiley (1992) (5) Schumm (1963) (6) Platts and others (1983)
	Reach level: Scale is 1:12,000 to 1:24,000	
Maps	Stream geomorphic channel units (1,2) Reach conditions (2) Reach location (2) Stream reach analysis and survey design (3)	(1) Bisson and others (1982) (2) Meador and others (1993b) (3) Newbury and Gaboury (1993)
	Stream type (1) Velocity (2)	(1) Leopold and Wolman (1957) (2) Meador and others (1993b)
	Elevation and slope	Meador and others (1993b)

Table 4.3.5-1. Summary of methods of habitat assessment at six spatial scales—Continued

[GIRAS, Geographical Retrieval and Analysis System; NATSGO, National Soil Geographic Data Base; STATSGO, State Soil Geographic Data Base; NRCS, Natural Resources Conservation Service; MOSS, Map Overlay Statistical System; NWS, U.S. National Weather Service; RF3, U.S. Environmental Protection Agency River Reach; EROS, Earth Resource Observation System; DLG, Digital Line Graph Data; USGS, U.S. Geological Survey; NOAA, National Oceanographic and Atmospheric Administration; PCS, Pollution Control System; TRI, Toxic Release Inventory]

Format	Type of assessment	Source of data or reference (unless numbered, each type of method can be found in all references)
	Channel and flood-plain level	
Field measurements	Channel features (3): Length, depth, width (2,4) Geomorphic units (1) Slope (4) Aspect (4) Habitat features (4) Bar, shelf, island (4,6) Channel geometry and pattern (5) Channel hydraulics (5) Channel stability (5) Flow frequency (5)	(1) Bisson and others (1982) (2) Harrelson and others (1994) (3) Leopold and Wolman, (1957) (4) Meador and others (1993b) (5) Newbury and Gaboury (1993) (6) Yuzyk (1986)
	Bank features (4): Bank erosional type (1,2,3) Bank slope (1) Bank stability (1) Bank width (1) Bank angle (1) Bank height (1) Bank shape (4) Bank substrate (4)	(1) Hupp (1986) (2) Hupp and Osterkamp (1985) (3) Leopold and Wolman (1957) (4) Meador and others (1993b)
	Flood-plain features (1,2): Width (3)	(1) Hupp (1986) (2) Hupp and Osterkamp (1985) (3) Leopold and others (1964)
	Flood-plain vegetation inventory (2) Quarter-point method (1) Canopy angle (3) Bank vegetation stability (5) Bank woody vegetation (4) Aquatic and riparian vegetation (3) Permanent vegetation plot (3)	(1) Hupp (1986) (2) Hupp and Osterkamp (1985) (3) Meador and others (1993b) (4) Mueller-Dombois and Ellenberg (1974) (5) Platts and others (1983)
	Bed substrate (1) Grain size (3,4) Embeddedness (2)	(1) Meador and others (1993b) (2) Platts and others (1983) (3) Wolman (1954) (4) Yuzyk (1986)
	Photodocumentation (1) Diagrammatic mapping (2,3)	(1) Harrelson and others (1994) (2) Meador and others (1993b) (3) Newbury and Gaboury (1993)
	Microhabitat characterization	
Field measurements	Bed substrate type (3) Macrophytes (1) Woody debris (2)	(1) Biggs and others (1990) (2) Cuffney and others (1993b) (3) Hawkins (1985)

4.4 Precipitation samples

The QA/QC issues surrounding collection of precipitation (wet deposition) samples for subsequent chemical determinations are particularly diverse, owing to the diverse needs for and uses of these data. The purpose of any given precipitation-quality study must be clearly examined in order that the project plans adequately address QA/QC issues, particularly those of representativeness (siting and frequency of automatic sampling and field visits) and comparability (use of wet-only, bulk, or dry collection equipment, sample handling, and analytical techniques).

The USEPA's recommended QA/QC procedures and requirements for collection and chemical analysis of precipitation are given by Peden and others (1986). Guidance on field and data-analysis procedures and laboratory schedules to be used by the WRD are given in OWQ Technical Memorandum 81.07.

Considerations for determination of metals in precipitation are discussed in Vermette and others (1995). Methods of sampling and analyzing for numerous properties and constituents in wet deposition are discussed in a case study from Indiana (Willoughby, 1995). Detailed descriptions of the field procedures at a typical site are given in a videotape produced by the National Atmospheric Deposition Program/National Trends Network (1989), which can be borrowed from the Chief of the Hydrologic Surveillance Section in the Ohio District. A brief summary of general QA/QC considerations follows.

4.4.1 Site selection, collector design, and sampling frequency

For studies of regional or background precipitation quality, locate sites so that they are reasonably distant from traffic and other human activity that would disturb land or water surfaces. Avoid overhead obstructions (trees, powerlines); a general guideline is that the line-of-sight angle from the top of the collector to the nearest overhead obstruction should be 30° or less. Orient the collector parallel with prevailing wind direction. The generally accepted criterion for height calls for the top of the collector to be 6 to 12 ft above the ground.

Bulk collectors are constructed to accept dryfall in addition to wetfall, whereas wet-only collectors are designed to open up only during periods of precipitation. Bulk sampling is appropriate if the project goal is determination of total atmospheric input of constituents. Wet-only sampling is appropriate if the goal is to determine concentrations of dissolved constituents in the precipitation itself. Dry-deposition collectors are in use at a few locations nationwide; a dry-deposition program is administered by the USEPA.

For either type of collector, ensure that sample containers, funnels, liners, and tubing consist of inert, nonabsorbing materials that will not affect concentrations of ions in solution. Polyethylene or Teflon are suitable materials for subsequent determinations of major ions. Glass is acceptable if mercury is to be determined, but Teflon is recommended if other trace elements are of interest (Willoughby, 1995, p. 6). Cost is commonly a factor in the selection of materials for precipitation samples.

Weekly, daily, event, and within-event sampling frequencies are all common; the choice depends largely on the project objectives. Periods of greater than 2 weeks between retrievals of wet-deposition samples are not recommended because of the possibility of evaporation and sample degradation. For dry-deposition samples, monthly retrievals are recommended.

4.4.2 Site visits and sample handling

During the site visit, approach all samplers from the downwind side to help prevent contamination from dust or from clothing or hair. Do not touch collection surfaces when removing or installing the collection vessel. Bring new collection vessels to the sampling site in clean plastic bags and install them immediately after the bags have been removed.

Check sampler covers frequently to ensure proper operation. Check the reciprocating cover of wet-only samplers to ensure that a tight seal is maintained between the vessel rim and the cover. Clean the underside of the cover at least monthly with distilled-deionized water to prevent buildup of dirt that could contaminate the sample. During visits to dry-deposition collectors, check the actuating mechanism for moving the cover plate; adjust the mechanism, if necessary, to ensure that a minimal amount of wet deposition is being collected.

Remove wet-deposition samples carefully and cap them until they are split and field measurements are made. Measure specific conductance and pH with an electrode that is temperature compensated and calibrated by use of low-ionic-strength solutions (Busenberg and Plummer, 1987). Record calibrations in ink in bound notebooks, which are to be kept with the instrument. Use Gran titration for acidity measurements (Peden and others, 1986; method 305.6). (See also methods listed under section 6.1.1 of this manual.) After removal of samples and before installation of new sample bottles, first rinse all precipitation-catching surfaces and tubing with 5 percent (by volume) hydrochloric acid solution, then with distilled-deionized water, then with dilute Liquinox solution, and then again with distilled-deionized water.

Remove dry-deposition material from the collector by sequential rinsing with deionized water from a wash bottle of known volume (250 mL recommended) and alternate scrubbing with a spatula of inert plastic. Before rinsing the collector, use tweezers to remove leaves, twigs, and other large pieces of organic matter. Discard samples containing bird droppings.

QC procedures will depend on the needs of the project but generally consist of a suite of equipment blanks and replicate samples to ensure that the samples are not contaminated by the sampling equipment and to verify the analytical procedures of the laboratory, respectively.

Subsequent sample-handling procedures (splitting, preservation, and so on) and laboratory analyses will depend on the data needs of the project. For low-ionic-strength solutions such as precipitation, use of ion-balance or specific-conductance comparisons are required as verification of laboratory analyses.

4.5 Suspended-sediment, bed-material, and bedload samples

The approach to collection of suspended-sediment and bed-material samples differs somewhat from that of other water-quality work in that the Office of Surface Water (OSW), WRD, oversees QA of the Division's sediment programs. In the Ohio District, the District Sediment Specialist is the primary technical contact for QA/QC-related questions.

QA/QC aspects of sediment studies within the WRD are thoroughly discussed in Knott and others (1993). Some of the main points of these and other authors as applied to sediment studies in the Ohio District are summarized below.

4.5.1 Site selection

For site selection, project personnel must do a thorough reconnaissance and site analysis and prepare a complete station description before any data are collected. Depending on the objectives for data collection, project personnel must select the appropriate equipment, sampling frequency, and visiting frequency (including visits by observers) to maximize representativeness of the data within the constraints of project design.

4.5.2 Field methods for suspended-sediment sampling

Once site operations commence, the project chief must ensure that data are collected by use of acceptable field methods. Comprehensive reports by Edwards and Glysson (1988), Ward and Harr (1990), and Johnson (1997) are the most recent USGS publications on field methods for collection of suspended sediment. Ohio District personnel are encouraged to read these works before planning a sediment-related project and have access to them for subsequent reference. Sediment observers should be retrained in data-collection methods annually, and their performance needs to be appraised through checking of their records with each field visit by USGS personnel (Knott and others, 1993, p. 5).

Manual suspended-sediment samplers are used at all sediment-data-collection sites; automated samplers are used to supplement manual measurements at sites where manual measurements cannot efficiently be made at the desired frequency or in response to rapid changes in streamflow. Types of suspended-sediment samplers commonly used in the WRD are listed in table 4.5.2-1.

Measurements made with the manual depth- or point-integrating samplers frequently involve multiple verticals, so project personnel will have to choose between the EDI and EWI approaches for determining spacing of verticals; strict adherence to the steps outlined in these approaches is crucial for obtaining accurate sediment data. (See section 4.1.2 for further discussion of these approaches.) Once the spacing of verticals has been determined, project personnel must choose from among several samplers, container sizes, and nozzle sizes for making the measurement (tables 4.5.2-2 through 4.5.2-4).

Table 4.5.2-1. Types of suspended-sediment samplers used by the U.S. Geological Survey

Type of sampler	Available models	Capacity
Manual samplers		
Hand-held depth-integrating samplers	DH-48	Pint
	DH-59	Pint
	DH-75P, Q, H	Pint, quart, 2 liters (respectively)
	DH-76	Quart
	DH-81	Any size bottle with standard Mason threads (1 liter recommended)
Cable-and-reel depth-integrating samplers	D-74, D-74AL	Pint, quart
	D-77	3 liter
	D-95	1 liter
Cable-and-reel point-integrating samplers	P-61	Pint, quart
	P-63	Pint, quart
	P-72	Pint, quart
Hand-held point samplers	Van Dorn type	2.2 to 30.2 liters
	Kemmerer type	0.4 to 16.2 liters
Automatic samplers		
Pumping-type samplers	PS-69	All listed samplers can be configured to a range of sample sizes.
	PS-82	
	CS-77	
	Various commercial models	

Sampling procedures to be used with the Van Dorn- and Kemmerer-type samplers are given in Ward and Harr (1990).

If determination of sediment chemistry is a goal, then care must be taken to ensure that the samplers, sample bottles, and other accessories are constructed of the proper materials for the constituent(s) being sampled. See section 4.1 for guidelines for choice of equipment, materials, and subsequent sample handling and processing.

Table 4.5.2-2. Sediment sampler selection matrix

[All samplers are suitable for sampling suspended sediment. Sampler names shown in italics are also suitable for water-quality sampling if Teflon and (or) glass parts are used. ft/s, feet per second]

Depth (feet)	Maximum velocity in the vertical[a] (ft/s)		
	0 - 2[b]	>2 - 6.6	>6.6 - 8.9
0-2	*Open bottle* DH-48 DH-59 DH-75 DH-76 *DH-81*	DH-48 DH-59 DH-75 DH-76 *DH-81*	DH-48 *DH-81*
2-15	*Weighted bottle* DH-48 DH-59 DH-75(H/P/Q) DH-76 *DH-81* D-49[c] D-74 D-77 *D-77AL* P-61 P-63 P-72	DH-48 DH-59 to 5 ft/s DH-75(H/P/Q) DH-76 *DH-81* D-49[c] D-74 *D-77* *D-77AL* to 4 ft/s *D-77 Bag* P-61 P-63 P-72 to 5.3 ft/s	DH-48 *D-77* to 8 ft/s *D-77 bag* to 8 ft/s *DH-81*
>15	*Kemmerer* *Van Dorn* P-61 to 180 ft deep P-63 to 180 ft deep P-72 to 72 ft deep	P-61 to 180 ft deep P-63 to 180 ft deep P-72 to 5.3 ft/s and 72 ft deep *D-77 bag* *Frame-type bag sampler*[d]	*Frame-type bag sampler*[d]

[a] The maximum velocity reported for a sampler is the lesser of the maximum calibrated velocity and the maximum velocity at which the sampler is thought to be hydraulically stable. None of the samplers have been calibrated at velocities higher than 8.9 ft/s.

[b] None of the samplers collect isokinetic samples at velocities less than approximately 1.5 to 2 ft/s.

[c] Sampler is no longer being produced.

[d] Does not sample isokinetically at velocities less than approximately 3 ft/s.

Table 4.5.2-3. Sample container sizes for sediment samplers

[Shaded boxes indicate possible sample container sizes]

Sampler	Pint	Quart	1 liter	2 liter	3 liter
DH-48	■				
DH-59	■				
DH-75P	■				
DH-75Q		■			
DH-75H				■	
DH-76		■			
DH-81[a]	■	■	■		
D-49	■				
D-74	■	■			
D-74AL	■	■			
D-77					■
D-95			■		
P-50	■	■			
P-61	■	■			
P-63	■	■			
P-72	■	■			

[a] Can use any size bottle with standard Mason threads.

Table 4.5.2-4. Maximum depths for selected sample container sizes and nozzle diameters

[Depths are reported in feet. Nozzle diameters are reported in inches]

Sample container size	Maximum depths at sea level for indicated nozzle diameters[a]			
	1/8[b]	3/16	1/4	5/16
Pint	15	15	8.7	5.6
Quart	15	15	15	10.4
1 liter	15	15	15	12.7
2 liters	15	15	15	15
3 liters	15	15	15	15

[a] For rigid containers without pressure compensation.
[b] This diameter nozzle not recommended for field use.

4.5.3 Field methods for bed-material sampling

Bed-material sampling has traditionally focused on collection of sediments composing the top few inches of the streambed and having particle sizes of 40 mm or less; these constraints represent the physical limits of traditional bed-material-sampling devices (Edwards and Glysson, 1988; Ward and Harr, 1990). Small amounts of fine-grained materials to be analyzed for trace elements and organic contaminants can be sampled by use of a guillotine sampler or a Teflon scoop, sieved through nylon cloth, and shipped to the laboratory in a glass jar (Shelton and Capel, 1994, p. 16-18). Coarser bed materials can be inventoried by a grid-based pebble-count method (Wolman, 1954) or by a photographic (Zeiss) technique (Ritter and Helley, 1968). Recently, methods have been developed for quick freezing and removal of streambed-sediment cores containing the full range of sediment particle sizes; one such method is described by Palcsak (1996). Types of bed-material samplers used in the WRD are listed in table 4.5.3-1. A complete discussion of field methods for bed-material sampling can be found in Radtke (1997).

Certain bed-material samplers must be used with caution. Those with spring-loaded scoops can seriously damage fingers. The freeze-type samplers require use of high-pressure gases or liquids; moreover, the devices themselves become cold enough to freeze exposed flesh on contact.

Where to sample bed material in a given stream cross section depends on data-collection goals. Commonly, bed material is collected at the bottom of the same verticals used in discharge and (or) suspended-sediment measurements. To avoid collection of bed-material samples from an excessively disturbed streambed, field personnel should collect bed material before making other measurements, especially in wadable streams.

Table 4.5.3-1. Types of bed-material samplers used by the U.S. Geological Survey

Type of sampler	Available models	Use and limitations
	Samplers for collection of material finer than medium gravel	
Hand-held samplers	Guillotine or Teflon scoop	Wadable streams, fine material
	BMH-53	Wadable streams
	BMH-60	Low-velocity streams or lakes
	BMH-80	Wadable streams
	Ponar	Streams, fine materials
	Ekman	Streams or lakes; fine, uncompacted material
Cable-and-reel sampler	BM-54 Ponar Shipik Ekman Petersen	All types suitable for lakes and streams of reasonable depth, except at streamflows of very high velocity; generally best for fine sediments
	Samplers for collection of material coarser than medium gravel	
Freeze-type samplers	Single-tube or tri-tube	Selection of type depends on kind of bed-material analysis to be done. See Palcsak (1996) for description of one type and references to other types.

If chemical analyses are to be done, bed-material samples must be chilled to 4°C for holding and subsequently sieved to exclude particles larger than either 2 or 0.063 mm, depending on analytical needs. Metal sieves are used to process bed-material samples for determination of major cations, nutrients, chemical oxygen demand, volatile solids, moisture content, radioelements and isotopes, and organic constituents. Plastic screens are used to process samples for determination of trace elements. Details on processing, storage, and shipment of bed-material samples for chemical analysis are given in Ward and Harr (1990) and in Shelton and Capel (1994).

4.5.4 Field methods for bedload sampling

Bedload is sediment that moves by sliding, rolling, or bouncing along on or near the streambed. Thus, it is typically not represented either in suspended-sediment samples (which may be collected above much of the bedload) or bed-material samples. The Federal Interagency Sediment Project (FISP) sampler is currently (1996) the recommended sampler for use in USGS work. This sampler, which is similar to the Helley-Smith sampler commonly used in the 1970's and 1980's, consists of a frame to which an expanding nozzle and a collection bag are attached.

Determining how to sample a cross section and calculate bedload discharge involves complicated procedures that cannot be summarized here; details of these procedures are given in Edwards and Glysson (1988) and in Office of Surface Water Technical Memorandum 90.08. (Although the procedures in Edwards and Glysson refer specifically to the Helley-Smith sampler, they are applicable for use with the FISP sampler.)

4.5.5 Record keeping, data processing, and quality-assurance reports

Observers must record all necessary sample information on a summary form. Project personnel must record sample information, equipment maintenance, and other pertinent observations on a field-inspection form. Field notes and sample labels must be correct and complete, and ancillary measurements (streamflow, water temperature, stage) must be of acceptable accuracy (Knott and others, 1993, p. 4). A plan should be in place for project staff to interact frequently with observers and for sediment records to be compiled and entered into the appropriate data bases within a reasonable amount of time. A file documenting all activity at and analyses for each sediment station must be established and maintained.

Reports include annual QA/QC reports submitted by the District Chief to the Regional Hydrologist, reports resulting from periodic surface-water reviews by teams from outside the Ohio District, and annual QA/QC reports from all laboratories doing sediment work for the district.

4.5.6 Laboratory work

Virtually all of the Ohio District's sediment analyses are done by contract laboratories. Sediment laboratories used by the Ohio District must follow QA/QC procedures that are consistent with those of the USGS, as outlined in Office of Surface Water (OSW) Memorandum 98.05, Matthes and others (1991), and Knott and others (1992, 1993). QA goals include use of accepted methods, equipment, and reagents; target accuracies for the measured properties and methods; correct and complete entry of information on laboratory forms; and analysis of all samples within 90 days of receipt.

4.6 Solid-phase samples

Techniques for the collection and processing of solid-phase samples and associated QA/QC procedures are discussed below. Solid-phase samples include consolidated and unconsolidated core samples, mine spoil, and man-made solids such as fly ash. Although mine spoil and man-made materials are typically associated with surface deposits, the focus of this section is on samples collected from subsurface formations.

4.6.1 Site documentation

Perform a thorough reconnaissance and site analysis before any samples are collected. Also, consult records kept by the Ohio Department of Natural Resources, Division of Oil and Gas, and the Division of Water, to determine if any drilling logs exist for the area where subsurface sample collection is planned. Determine the location and elevation of borehole to third-order accuracy by use of standard surveying methods (Moffit and Bouchard, 1992).

During drilling, detailed lithologic logs are to be kept by the driller and the project chief. Core samples need to be clearly labeled with respect to the time and date of sampling, well or borehole number, depth interval represented, percent recovery over sampled interval, and types of chemical or physical analyses to be done on the samples. Results of any onsite chemical analyses of core samples (such as VOC screening) must also be documented on the drilling log. QA/QC procedures associated with such onsite analyses are to be clearly written up in the project QA/QC plan.

4.6.2 Sample collection and processing

Core samples of competent rock are collected by diamond rotary drilling, a technique in which a circular diamond-coated bit is used to bore through rock and obtain a solid core. Samples of cuttings obtained by mud-rotary or cable-tool methods are useful for lithologic characterization but are not to be used for chemical analyses because they can be mixed with cuttings from shallower parts of the borehole or with drilling fluids. Sediment samples from unconsolidated deposits are usually obtained by use of either the cable tool or hollow-stem auger drilling methods. Rotasonic drilling combines rotation and high-frequency vibration to advance the core barrel to depths approaching 250 ft in unconsolidated deposits (Wright and Cunningham, 1994). Detailed descriptions of the various drilling methods and types of samplers used to collect geologic materials in unconsolidated deposits are given in Ohio Environmental Protection Agency (1995) and Lapham and others (1997). Methods for collecting and processing microbial samples in unconsolidated deposits can be found in McMahon and others (1992) and Chapelle (1993; p. 211-231).

Once formation samples are collected, the sample must be processed or preserved. Most consolidated cores are placed in wooden or cardboard core boxes for storage, and samples for chemical, petrographic, or physical analysis are extracted from the cores at a later date. Sampling strategies for cores will vary by project objective; cores may be subdivided by lithology, presence of weathered or mineralized horizons, or hydrogeologic properties such as porosity. Sampling techniques may include grab sampling or compositing of selected intervals of the core. Unconsolidated sediment samples are placed in plastic (or Teflon) bags. Samples for chemical analysis are to be taken from the center of the core if possible; material in contact with the sampler is to be excluded to avoid contamination from drilling fluids, overlying strata, or ground water. Clay and other fine-grained core samples collected for permeability measurements are typically collected in thin-walled metal tubes (Shelby tubes). Samples collected in this manner are stored in the tubes, filled at both ends with sand (to prevent movement of the core material), capped, and sealed at both ends with tape or paraffin to prevent desiccation. Permeability samples collected by other methods (rotary drilling) are to be stored in plastic bags or wrapped in aluminum foil to prevent desiccation. If oxidation of reduced solid phases in the samples (for example, sulfides or organic carbon) is a concern, then core samples need to be chilled or frozen or placed in sealed containers flushed with nitrogen gas. If the study of microbial populations is the objective, subsamples collected from the center of the core must be chilled but not frozen (Chapelle, 1993). Submission of replicate core or sediment subsamples is recommended to permit assessment of laboratory variability.

QA activities associated with the collection and processing of core samples are designed to prevent contamination of samples and the environment by drilling equipment. The most important QA activity is thorough decontamination of drilling and sampling equipment. Decontamination of equipment is required to ensure that representative samples are collected and that cross-contamination between sites or vertical intervals of individual boreholes does not occur. A typical decontamination procedure would involve washing the equipment with a soap solution followed by rinses with clean tap water and distilled water. Rinses with methanol or dilute acid solutions prior to the tap-water and distilled-water rinses may be necessary, depending on project objectives. Steam cleaning also may be done, especially where organic contaminants are suspected.

Precautions also must be taken to ensure that materials used during the drilling process, such as drilling mud or water, do not introduce contaminants to the formation. If drilling mud or water are used during the drilling process, collect samples of these fluids and have them analyzed for constituents of concern. Also, submit several equipment blanks to assess the efficiency of equipment decontamination procedures. Descriptions of decontamination plans and procedures are given in Aller and others (1991), Ohio Environmental Protection Agency (1995), and Lapham and others (1995, 1997).

5.0 Handling and documentation of samples

Before, during, and after the sample is collected, proper handling and documentation are essential to ensure the integrity of the sample and the correct reporting of data results. The correct order of activities for handling and documenting samples is outlined in the following paragraphs.

5.1 Sample identification, labeling, documentation, handling, packaging, and shipping

5.1.1 Before field activities

The project team is responsible for all field preparations associated with handling and documentation of samples. Preparations for field activities are detailed in Wilde and others (1998a).

The station identification number (station ID) and site name are established by the project chief by consulting the existing list of GWSI sites referenced by county (located in the Information Officer's office) and the GWSI administrator. Procedures for establishing and naming sites have been documented by USGS Indiana District personnel (Martin and Cohen, 1994), and these same procedures are to be used in the Ohio District. Except at locations of established USGS gaging stations, station ID's are generally determined by the latitude and longitude of the site. General site data, including site location, station name, and the 15-digit station ID must be entered into the GWSI data base before water-quality samples are collected. If this information is not entered before water-quality samples are collected, the samples cannot be entered into the Ohio District water-quality data base (see section 8.1).

Preprinted bottle labels are made by the project team and must include the station ID, site name, and spaces for the date, time, and sample collector. If the NWQL is doing the analyses, the project team must determine laboratory codes and schedules that correspond to the target constituents and desired methods. A list of laboratory codes and schedules can be obtained from Timme (1995), from the NWQL Services Catalog, or the Schedules, Parameters, and Network Records (SPiN) on the World-Wide Web at http://wwwnwql.cr.usgs.gov/USGS. The project team should write up a field checklist; supplies are then assembled by project team members. Consult the above references or the Laboratory Coordinator for correct bottle types and write the bottle designations on the bottles. The sample bottles are organized into mesh bags containing the bottles needed for each sample site.

A water-quality site-field folder is prepared by the project team for each site. For all sites, at the minimum, the folder will contain the station description, maps to the site, field equipment checklist, field note forms, sampling and processing instructions, safety information, preprinted labels, and plots of expected field values, For surface-water sites, include a cross-section profile of the channel (stream). For ground-water sites, include permission documents for site access, well construction and driller's log, and water-level records. A complete list of data to be stored in site-file folders is found in Martin and Cohen (1994) or by consulting the District Water-Quality Specialist.

5.1.2 During field activities

Take clear, concise field notes at the time of sample collection on standard ground-water and surface-water field forms, available from the QWSU. If the standard forms are not used, it is the responsibility of the project chief to develop a project-specific form with approval from the District Water-Quality Specialist. Check field notes for errors and completeness before leaving the site. Write the date, time, and collector's initials on preprinted labels with waterproof ink. Compute alkalinity and acidity values in the field to ensure that the analysis was done correctly. After sample collection, store the tightly capped, labeled bottles as appropriate for the type of sample; for example, nutrients and organics must be stored in a refrigerator or cooler at 1 to 4°C (Timme, 1995). Make sure labels are securely affixed and protected with plastic so that they will not come off if stored in a cooler with ice.

Because the collection of water-quality data in the field is frequently hazardous, the safety of all field personnel is a primary concern. Field teams often work under extreme environmental conditions and may come in contact with waterborne and airborne chemicals and pathogens while sampling. Field work involves the transportation and use of equipment and chemicals. Beyond the obvious negative consequences of unsafe conditions on field personnel, such as accidents and personal injuries, the quality of the data also may be compromised when sampling teams are exposed to dangerous conditions (Schertz and others, 1998). Guidelines pertaining to safety in field activities are provided in Lane and Fay (1998) and Yobbi and others (1995); guidance can also be obtained from the District Safety Officer.

5.1.3 After field activities

Analytical Service Request Forms (ASR's) must be included with all samples sent to the NWQL. It is the responsibility of the project chief to ensure accurate and thorough completion of the ASR. Alternatively, if an outside laboratory is being used, the project chief may develop similar forms for use by the contract laboratory. (Alternative forms must be approved by the District Water-Quality Specialist.)

The following sample information on the ASR is mandatory: station ID, project account number, beginning date and time, state code, district user code, and requested schedules and/or labcodes. In addition, because all WRD water-quality data are stored in the USGS National Water Information System (NWIS) database, NWIS codes for sample medium, analysis status, analysis source, hydrologic condition, sample type, and hydrologic event are also mandatory. These codes are on the cover of ASR pads, on the supplementary page of field forms, and in the NWIS users manual on the World-Wide Webb at http://wwwnwis.er.usgs.gov/; they may also be obtained from the District Water-Quality Specialist. Include the project chief's name and phone number on the ASR; this information is not mandatory but helps NWQL personnel in the event of problems or questions. Alkalinity, acidity, pH, and specific conductance values are included on the ASR, if available, to aide NWQL personnel in completing QA/QC checks. Include information about the number and types of bottles sent at the bottom of the ASR. Enclose ASR's in water-tight bags before shipment. When shipping chilled samples, include a bottle filled with water in the cooler for a temperature check upon arrival at the laboratory and a return form for the laboratory to fill out. Include a return shipping label with the 11-digit project identification number (for example, 39-4439xxxxx) to expedite return of coolers and to ensure that return shipping costs are billed to the appropriate project. Check sample bottle labels against information on the ASR's to ensure that all information is consistent.

All of the effort and time spent in sample collection will be wasted if samples deteriorate between the time of collection and time of analysis. Maximum holding times for samples that have been properly preserved are given in Pritt and Raese (1995). Include 2 days for shipping when determining when to send samples based on maximum holding times. Generally, chilled samples are to be shipped within 24 hours after collection, and should be shipped from the field. The types of suitable shipping containers are discussed in Ward and Harr (1990), and recommendations to district offices for shipping samples to the NWQL are discussed in OWQ Technical Memorandum 92-06 and NWQL Technical Memorandum 95-04. Within 5 working days of shipping samples, sample information is to be entered into the appropriate data base, as described in the NWIS users manual and in section 8.1 of this report. Contact the District Water-Quality Specialist for instructions.

Table 5.1-1. Summary of activities and references for procedures for handling and documentation of samples

Activity	Reference
Before field activities	
Establish station identification number and enter site information into Ground Water Site Inventory (GWSI)	Martin and Cohen, 1994 Consult GWSI administrator Consult Information Officer
Determine laboratory schedules and supplies needed, assemble supplies	Timme, 1995, p. 17-74 National Water Quality Laboratory Services Catalog or Schedules, Parameters, and Networks (SPiN) Record on the World-Wide Web at http://wwwnwql.cr.usgs.gov/USGS Consult District Water-Quality Specialist
Assemble bottle types and use preprinted bottle labels	Timme, 1995, p. 9-11 SPiN Program Consult Laboratory Coordinator
During field activities	
Complete clear, concise field notes	Consult District Water-Quality Specialist
Compute alkalinity and acidity values	Field forms, available from Quality of Water Service Unit, Ocala, Florida
Store bottles as appropriate for type of sample	Timme, 1995, p. 17-19
Follow safety guidelines	Lane and Fay, 1998
After field activities	
Verify field alkalinity values	Consult District Water-Quality Specialist for current computer program and calculation procedures
Complete Analytical Service Request (ASR) Forms	Project chief in consultation with District Water-Quality Specialist NWIS users manual on the World-Wide Web at http://wwwnwis.er.usgs.gov/
Mail samples to the laboratory in appropriate containers and within appropriate holding times	Pritt and Raese, 1995 Ward and Harr, 1990, p. 66-67 Office of Water Quality Technical Memorandum 92-06 and National Water Quality Laboratory Technical Memorandum 95-04
Enter samples into the data base within 5 working days after shipping samples	NWIS users manual on the World-Wide Web at http://wwwnwis.er.usgs.gov/ Consult District Water-Quality Specialist

5.2 Chain of custody procedures and documentation

When chain of custody procedures are appropriate or required, project personnel must establish, maintain, and document the custody of field samples. A sample is considered to be in custody if it is (1) in your possession, (2) in your view, after being in your possession, (3) sealed or secured to prevent tampering after being in your possession, or (4) placed in a designated secure area after being in your possession. Sample chain of custody documentation includes (1) sample identification, (2) pertinent information on sample collection, (3) sample source, (4) preservative, (5) required analyses, (6) name(s) of sample collector, (7) time(s) it was in his/her possession, and (9) space for signatures of custodians (those individuals having sample custody).

Containers used for shipment of samples are sealed with custody seals, which are signed and dated by the shipper and covered with tape. Before the shipper is released from custody of the samples, laboratory personnel receiving the samples carefully examine the shipping container to ensure that opening or tampering with the container has not occurred. The completed chain of custody record form verifying sample custody from collection to receipt at the laboratory is usually included as part of the analytical results data package. A copy of the chain of custody documentation is kept permanently by all custodians. If chain of custody documentation is not maintained, sample results may not be allowable as evidence in legal proceedings.

Each laboratory generally follows its own internal chain of custody procedures for sample tracking. The project chief should refer to the District Water-Quality Specialist or laboratory personnel for more information on laboratory chain of custody procedures.

6.0 Operation, preventive maintenance, and calibration of field and laboratory equipment

All equipment used by District personnel for the collection and processing of water-quality samples is to be properly operated, maintained, and calibrated. For correct operation of any field or laboratory equipment, carefully follow the operating guidelines of the manufacturer. Calibration and maintenance records of field equipment are to be kept by the project personnel in equipment books; those records of District laboratory equipment are to be kept by the District Laboratory Coordinator. Calibration and maintenance records are to be recorded in bound notebooks, in ink, and are checked annually for completion and accuracy by the District Water-Quality Specialist.

6.1 Field-monitoring equipment

In the following sections, calibration and preventive-maintenance procedures are described for equipment and probes used to monitor chemical, physical and biologic properties of water. Field screening techniques (immunoassay and headspace screening by use of portable gas chromatograph) are also briefly described.

6.1.1 Chemical and physical properties

Chemical and physical properties of water that are unstable in a sample over time are measured in the lake or stream or at the wellhead being sampled. These properties include temperature, specific conductance, pH, DO, alkalinity, and turbidity. Less commonly measured water-quality properties that also are determined in the field include acidity, oxidation-reduction potential (E_H), iron speciation (Fe^{2+}/Fe^{3+}ratio), and the concentration of hydrogen sulfide (H_2S) in ground water. Description of the theoretical principles underlying the techniques for measuring pH, specific conductance, DO, and E_H are beyond the scope of this report; however, the reader is referred to Langmuir (1971), Bates (1973), Hem (1989), Wood (1976), Thorstensen (1984), Wells and others, (1990) and Wilde and Radtke (1998) for details. Detailed descriptions of principles and methodologies associated with onsite analysis of unstable water-quality properties can be found in Wood (1976) and Wilde and Radtke (1998).

A variety of equipment is available to measure unstable water-quality properties and constituents in the field. Proper maintenance and calibration of this equipment is necessary, and it is important to follow manufacturer's instructions to ensure that accurate and precise measurements are obtained. The District Water-Quality Specialist or other experienced District personnel should be consulted if project personnel need assistance with the selection or use of equipment. The Hydrologic Instrumentation Facility (HIF), which tests equipment for bias and variability under field and laboratory conditions, also can be consulted. HIF can be accessed on the World-Wide Web at their homepage: http://wwwhif.er.usgs.gov.

Both single-probe and multiprobe meters are available for field measurement of basic water-quality properties such as temperature, pH, specific conductance, DO, and E_H. Multiprobe meters allow determination of several water-quality properties simultaneously and are typically equipped with flowthrough chambers that are required for ground-water sampling (Koterba and others, 1995). Most newer instruments and probes are equipped with built-in temperature sensors that provide automatic temperature compensation for measurements of pH and specific conductance.

pH. Use commercially prepared buffers traceable to the National Institute of Standards and Technology (NIST) for calibration of pH meters. The buffers have pH values of 4.0, 7.0, and 10.0 at 25°C and are kept in the constant-temperature room of the Ohio District. Perform a two-point calibration of the pH meter using buffers that bracket the pH range in the sample and are at the same temperature as the sample. Most new pH meters offer auto-calibration and will automatically calculate the slope of the two-buffer calibration line as a percentage of the theoretical Nernstian slope. If the calculated slope deviates more than 5 percent from the theoretical slope, repeat the calibration or replace the electrode. On some meters, deviation of the slope outside of a specified range will yield an error message. Special calibration techniques are required for accurate measurement of the pH of low-conductivity waters (<100 µm/cm); these techniques are described in Busenberg and Plummer (1987).

Specific conductance. For calibration of specific conductance meters, standards are prepared from stock potassium chloride solutions to yield specific conductance values between 50 and 10,000 µs/cm. Standards come in 1-L bottles, are stored in the constant temperature room, and are prepared and quality assured by the QWSU. For many specific conductance meters, calibration consists of adjusting the observed response of the meter to that of the known standard. Calibrate with a standard whose specific conductance is near the median specific conductance value expected for the sample. After calibration with the mid-range standard, measurement of two additional specific conductance standards is recommended (without adjustment to the meter) to bracket the range of specific conductance values that might be observed in the field. For standards whose specific conductance value is greater than 100 µs/cm, the readings are to be within 3 percent of the standard value. For standards with specific conductance values less than 100 µs/cm, the readings are to be within 5 percent of the actual value. If these limits are exceeded, cleaning or replacement of the conductivity probe may be required. Note that some instruments have different conductivity probes for highly dilute or highly saline waters (see the operating manual for details). In addition, the temperature compensators of specific-conductance meters should be checked monthly for accuracy.

Dissolved oxygen. Dissolved-oxygen meters and probes are calibrated by adjusting the response of the meter until it matches that predicted for water-saturated air. The solubility of oxygen in water is dependent on the partial pressure of oxygen in air, dissolved-solids concentration (salinity), and temperature. The partial pressure of oxygen in air is dependent on barometric pressure, which varies as a function of elevation and local weather conditions. Hence, corrections for each of these parameters must be made before accurate DO measurements can be made. The only USGS-approved tables that give the solubility of oxygen as a function of barometric pressure, temperature, and salinity are available as an interactive program on the World Wide Web (http://hassrvares.er.usgs.gov/dotables.html). These data have been summarized in tabular form by Wilde and Radtke (1998).

The accuracy of the DO probe at concentrations near 0.0 mg/L is to be tested by immersing the probe in a freshly prepared sodium sulfite (Na_2SO_3) solution. The QWSU prepares a 2 mg/L sodium sulfite solution; however, be aware of expiration dates and discard the solution one month after opening. For DO measurements in flowthrough cells, water velocity past the DO probe is to be at least 1 ft/s, otherwise measurements could be biased low by diffusion effects (Hardy and others, 1989). For surface-water bodies having velocities less than 1 ft/s, use of a DO stirrer is recommended. Correct installation of DO membranes on probes and electrodes is critical to ensure that accurate and precise measurements are obtained. Replace wrinkled or loose membranes as soon as feasible. Membranes with air bubbles need to be reseated or replaced because air bubbles will cause biased measurements. New membranes require a relaxation period of several hours before they are ready for use. See the instrument instruction manual for details. Finally, because membrane permeability to oxygen and the solubility of oxygen are temperature dependent, it is extremely important to verify that the thermistor in the oxygen probe is giving an accurate temperature reading. This can be done by comparing the thermistor reading at two temperatures with that recorded by an NIST-traceable thermometer (Wells and others, 1990).

Check the accuracy of thermistors in DO meters (and all other temperature probes) on a regular basis against temperatures recorded by mercury thermometers certified accurate to ± 0.1°C by NIST. Several NIST-certified thermometers are kept in the Ohio District constant-temperature room. Also, check the accuracy of hand-held altimeters against the barometric pressure recorded by the mercury barometer in the constant-temperature room (see section 6.1). These altimeters are used to provide barometric pressure readings for DO pressure correction. The pressure difference between the altimeter and constant-temperature room barometer must be less than 5 mm Hg; differences greater than this indicate problems with the altimeter and could affect the accuracy of field DO measurements.

$\underline{E_H}$. E_H is measured with a platinum-redox electrode (see Wood, 1976, p. 18-22 for description of measurement procedure). The only way to test the accuracy of the platinum redox electrode is by testing its response against solutions of known E_H. The most commonly used test solution is a potassium ferrocyanide-potassium ferricyanide mixture known as Zobell's solution. Zobell's solution has an E_H of 430 millivolts at 25°C relative to the standard hydrogen electrode (Nordstrom, 1977). Zobell's solution in 1-L bottles can be ordered from the QWSU. Zobell's solution is stable for several months if stored in a cool, dry place in a tightly capped brown glass bottle. For accurate measurements, the ionic strength of the filling solution of the platinum electrode needs to be matched to that of the water to be analyzed (see redox electrode operating manual for details). Also, the platinum tip of the E_H probe is subject to corrosion and fouling, especially in waters that contain measurable concentrations of hydrogen sulfide. Clean and polish the tip of the electrode with a toothbrush and nonabrasive toothpaste or an emery cloth when the surface becomes dull or discolored or if the response of the platinum electrode to Zobell's solution deviates from the theoretical value by more than ±10 percent. After cleaning, refill the electrode with new filling solution. If the deviation still occurs, the electrode needs to be replaced.

Alkalinity. Alkalinity titrations are to be done by use of the incremental titration technique on filtered water samples (Koterba and others, 1995; Wilde and Radtke, 1998). Titrations can be done using a burette and standardized sulfuric acid or digital titrators that use cartridges filled with sulfuric acid of known normality. Cartridges must be purchased from the QWSU, because each lot of cartridges must pass a quality-assurance check at QWSU. These cartridges have expirations dates that need to be checked before use. The pH meter used for the alkalinity titration should be calibrated with the 4.0 and 7.0 buffers. The sample needs to be stirred with a battery-operated stirrer or stir bar. Titrate the sample immediately after it is collected to prevent excessive temperature changes or degassing of the sample. If degassing of CO_2 is excessive (as indicated by vigorous effervescence), a cap with openings for the stirrer, pH, and temperature probes can be placed over the sample container.

Alkalinity titrations done on unfiltered samples yield data on the acid-neutralizing capacity (ANC) of the sample as unfiltered sample may contain particulate matter that will react with the sulfuric acid titrant (for example, carbonates, iron hydroxides). This can lead to high bias and poor reproducibility in the titration results; hence, current NAWQA protocols require that alkalinity titrations only be done on filtered samples (Koterba and others, 1995). For Ohio District projects, all alkalinity titrations must be done on filtered samples unless there is a specific need to determine the ANC of the sample. For samples where the titration endpoint is difficult to determine (for example, low conductivity, low alkalinity rainwater, or anoxic or organic-rich ground water) use of the Gran-Function plot method to determine sample alkalinity is recommended (Wilde and Radtke, 1998).

Acidity. Acidity is also determined by use of the incremental titration technique on filtered samples; however, the titration is done with a strong base (sodium hydroxide) to an endpoint near pH 8.3 (Brown and others, 1970; American Public Health Association and others, 1992). Acidity determinations can be affected by the presence of dissolved gases such as CO_2 and H_2S and hydrolyzable metal ions such as Fe, Mn, and Al. Oxidation and (or) hydrolysis of polyvalent metal cations can constitute a large percentage of the overall acidity of some industrial-waste and acid-mine waters. For samples where minimal concentrations of hydrolyzable metals are suspected, titrations are to be done in the field as quickly as possible because degassing of CO_2 and H_2S will affect the acidity titration. If such samples cannot be titrated in the field, chilling the sample during transport to the lab will prevent most degassing. For metal-rich samples, determination of total acidity requires addition of hydrogen peroxide and heating of the sample to just below the boiling point to ensure oxidation and complete hydrolysis of all polyvalent metals (Brown and others, 1970; American Public Health Association and others, 1992). This can be done in the field if equipment for heating the sample is available. In contrast, acidity values of metal-rich samples, such as acid mine drainage, are likely to remain relatively unchanged because metal concentrations will not be affected by degassing of CO_2 and H_2S. Hence, acidity titrations of samples known to contain significant concentrations of dissolved metals can be done upon return from the field in the Ohio District laboratory.

Turbidity. Turbidity is a measure of the amount of light scattered or absorbed by suspended particulate matter in a water sample. Turbidity is measured by optical techniques using turbidimeters or field spectrophotometers. Calibration of spectrophotometers is usually accomplished by use of formazine standards. These standards are prepared by serial dilution of formazine stock solutions and can be used to construct calibration curves. The formazine stock solutions can be purchased from commercial sources. Calibration is to be done immediately after the standards are prepared from the stock solution because the standards will deteriorate. A minimum of three standards is to be used for calibration. Calibrate turbidimeters and spectrophotometers daily. Because daily preparation of formazine standards is impractical, commercially available gel standards are measured before each use of the instrument to ensure that the formazine calibration has remained stable.

Hydrogen sulfide is a common constituent in anaerobic (low DO) water. H_2S readily degases and is easily oxidized upon exposure to air; hence, H_2S must be determined in the field. H_2S concentrations can be determined by the methylene-blue colorimetric technique by use of a field spectrometer (Hach Company, 1993). The method is highly specific for H_2S but can be affected by sample turbidity or the presence of other reduced chemical species. Spectrophotometric techniques for determining the oxidation state of selected metals (for example, Fe^{2+}, Fe^{3+}, Cr^{3+}, Cr^{6+}) in wastewater or acid mine drainage in the field also are available (Hach Company, 1993).

A summary of recommended minimum calibration frequencies for meters and probes used for the most common field measurements are in Wilde and Radtke (1998) and are summarized in table 6.1.1-1.

Table 6.1.1-1. Minimum calibration frequencies for equipment used for onsite measurements of selected water-quality characteristics

Property	Calibration procedure	Minimum calibration frequency
Temperature	Check against NIST-certified thermometer.	Monthly or with each use
pH	Use two-buffer calibration with slope evaluation	Daily; more frequently if probe is in contact with water for long periods of time
Specific conductance	(1) Check accuracy against minimum of two specific conductance standards	(1) Daily
	(2) Check temperature compensator	(2) Before every field trip or monthly, whichever is longer.
DO	(1) Calibrate to water-saturated air	(1) Daily; more frequently if air temperature changes.
	(2) Check low-range performance with sodium sulfite solution	(2) Monthly; weekly if working in low dissolved oxygen environments
	(3) Check field barometer against Hg barometer in constant temperature room	(3) Before each field trip
E_H	Check accuracy against Zobell's solution	Daily
Turbidity	Check accuracy against minimum of three formazine or gel standards	Daily

The guidelines given in table 6.1.1-1 are minimum guidelines. Certain applications involving the sequential analysis of many samples (10 or more) may require more frequent calibration. DO meters, whose calibration is dependent on barometric pressure and temperature, may need to be recalibrated at each sampling site. For ground-water sampling where purging of large-volume wells may take several hours or more, continuous flow of water can cause drift in the response of some monitoring probes (pH and E_H especially). Therefore, recalibration of some probes may be required. Moreover, as probes get old or are fouled by turbid or contaminated samples, more frequent calibration (or replacement) will be necessary.

Calibration and preventive maintenance. Preventive maintenance on equipment and probes used for field analyses is to be done according to schedules suggested by the manufacturer. General equipment maintenance may involve cleaning of probes, meters, and connectors; replacement of meter or probe batteries; laboratory checks of internal calibration curves; or replacement of optical cells or spectrophotometer lamps. Probe and electrode care may involve replacement of filling solutions, cleaning of the bulb or sensing element, or replacement of the membrane for DO probes. Proper storage of pH and other probes is essential for maintaining good probe performance. With proper care and maintenance, many probes will provide accurate and precise readings for several years. Even with proper probe care and maintenance, however, one should always bring backup probes, if possible, for all critical measurements to be made in the field.

Standard solutions for pH and specific conductance are to be replaced on a regular basis. Note that both pH and specific conductance standards have expiration dates marked on the containers; do not use the standards if the expiration dates have passed. Buffers for pH calibration are fairly stable and may be reused if not grossly contaminated. Specific conductance standards, particularly those with specific conductance values of less than 500 µs/cm, are prone to contamination; the small volumes used during the calibration procedure are discarded. It is good field practice to bring an extra set of pH buffers and specific conductance standards. At minimum, these solutions are to be replaced on a weekly basis or at the beginning of each extended field trip. Zobell's solution will maintain its E_H value for several months if it is stored in a brown glass bottle (capped tightly between use) in a cool place. Calibration and maintenance procedures for water-quality monitors used to provide continuous measurements of specific conductance, pH, DO, and temperature are described by Gordon and Katzenbach (1983). This reference also provides guidelines concerning the selection, installation, use of monitoring systems, as well as methods for evaluating the quality of data obtained. Various technological advances have occurred since Gordon and Katzenbach's manual (1983) was written, and new methods of data acquisition, recording, and transmission have been developed (Katzenbach, 1988; 1990). Additionally, probes capable of detecting and quantifying nitrate and ammonia, various metals, and selected organic compounds in surface water and ground water in the field have been introduced. The accuracy and precision of these probes under field conditions are being evaluated by the USGS in cooperation with the HIF and various District offices. If such equipment is being considered for use during Ohio District projects, additional QA/QC measures will need to be taken to ensure that accurate and precise data are being collected. A QA/QC plan for such equipment is to be developed in consultation with the District Water-Quality Specialist and relevant HIF personnel.

Field screening. Field screening techniques are available to quickly determine the presence or absence of certain chemical constituents, or to quantify others. Immunoassay tests for pesticides are available from several manufacturers. These kits have been extensively tested in the field and laboratory by the WRD and have been shown to provide reliable results (Thurman and others, 1990; Thurman and others, 1992). The function of the tests is to provide a qualitative or semiquantitative screening to detect the presence or absence of a target chemical or chemical family (Paul Capel, U.S. Geological Survey, written commun., 1992). Immunoassay techniques for VOC's, polycyclic aromatic compounds, PCB, and other organic compounds also are available. Headspace screening for volatile organic compounds by portable gas chromatograph (GC) with a flame ionization detector (FID) allows for rapid screening for large numbers of constituents and samples. The GC is calibrated by use of standards obtained from the NWQL. Details on the field GC method and QA/QC requirements are given in USEPA (1986a), Brock, 1990, and Parnell, 1995. Onsite analysis and screening of a large number of inorganic compounds is also available and is usually done with a field spectrophotometer or colorimeter (Hach Company, 1993).

6.1.2 Aquatic biology and habitat

All equipment and instruments associated with biological sampling such as analytical balances, scales, microscopes, thermistors, meter sticks, tapes, or other measuring devices are required to be kept in good working condition. Maintenance is accomplished through (1) service contracts on fume hoods, autoclaves, and balances, (2) preventive maintenance such as cleaning and periodic replacement of parts, proper storage, and proper use, (3) calibration, (4) service and repair when instrument is operating outside normal range of readings, and (5) keeping records of these activities in bound notebooks, in ink.

Equipment and instruments used for biological work in the Ohio District laboratory are subject to the same guidelines as described in section 6.2. Field equipment and instruments such as meters for the measurement of unstable constituents—temperature, pH, specific conductance, and DO—are required to be in good working condition, to be calibrated before each use, and have calibration and record books as specified in section 6.0. Instruments or tools used for making measurements of streamflow or channel dimensions such as width, length, depth, and reach characteristics are required to be in good working conditions as specified in policies of the OSW.

Sample-collection devices such as sieves, nets, and seines must be free of holes or gaps that allow organisms and materials to escape. These devices must be inspected after each use and repairs made where and when necessary. Devices such as those used to collect samples from rock surfaces, streambeds, and other microhabitats must be periodically examined for defects and wear. Repairs must be made quickly so that equipment remains in good working condition. Equipment used in association with sampling fish, such as weighing scales and electrofishing equipment, must be kept in good working condition.

Microscopes used for the measuring and (or) counting organisms must be fitted with calibrated micrometers. Calibrations must be recorded in ink in bound notebooks and kept with the instrument. Microscopes must be cleaned periodically, covered to keep out dust, and stored in a safe, dry, location when not in use.

Ice in ice chests and preservatives such as formalin, glutaraldehyde, alcohol, iodine solutions, and copper sulfate solutions must be checked visually during storage to determine whether specimens are being adequately preserved. Look for visual evidence of physical decomposition of animal or plant specimens.

6.2 Ohio District laboratory equipment

The District Water-Quality Specialist and Laboratory Coordinator are responsible for preventive maintenance and calibration of general laboratory materials and equipment. Calibration and maintenance procedures must be documented in bound notebooks, in ink.

The analytical balances are checked and calibrated annually by a service contractor. The temperatures of the hot-air ovens, incubators, refrigerators, and freezers are checked quarterly and recorded in ink in bound notebooks, which are kept by the District Laboratory Coordinator. The performance of the autoclave is checked by noting the operating temperature and pressure of each run, using heat-sterilizing tape to identify supplies that have been properly sterilized, and testing the performance with spore ampules quarterly. The operating temperatures of microbiological aluminum-block incubators and water baths are checked annually and recorded on the outside of each incubator. During periods of heavy use, the temperatures are checked and recorded daily. The laboratory has one fume hood for handling hazardous materials and a laminar flow hood for bacteria work or for cleaning and storing equipment for the inorganic protocol. Fume-hood face velocities are determined at least once per year by a qualified inspector and recorded on stickers placed on the hood. The face velocities should range between 60 and 100 linear feet per minute (LFPM); however, velocities up to 150 LFPM are acceptable. Face velocities on the laminar flow hood are also checked once per year and recorded on a sticker on the hood; the high efficiency particulate air filter is replaced if the face velocity falls below 70 LFPM as recommended by the manufacturer (Nuaire, Inc., Plymouth, Minn.). Once per year, agar plates are exposed to air flow in the laminar flow hood for 1 hour. The plates are incubated at 35°C for 24 hours and examined for contamination.

The Ohio District maintains a constant-temperature room used for calibrating field equipment and storing calibration standards. The temperature is maintained at 22°C by a dedicated heating and cooling system. Also stored in the constant-temperature room are the NIST-certified thermometer and a reference barometer (Princo Instruments, Inc., Southampton, Pa.). The barometer is checked quarterly by applying an altitude-correction factor to the barometric pressure at the Port Columbus International Airport as measured by the National Weather Service. The accuracy of District laboratory thermometers is checked semiannually against the NIST-certified thermometer. Temperature and barometric-check data are recorded by the Laboratory Coordinator in a QC logbook.

The Ohio District produces deionized Type III water from city of Columbus tap water for various uses including (1) final rinsing of laboratory glassware, (2) rinsing of electrodes used in field measurements, (3) preconditioning membrane filters, and (4) preparing biological and chemical reagents. The deionizing system is rented from a local vendor and contains a carbon filter, a 5 micron pore size prefilter, and cation, anion, and mixed bed resin columns. A conductivity-detection device on the system indicates when the mixed bed column is spent; when the light shuts off, the system has shifted to the standby mixed bed column. Every 4 months, or when the light turns off, the cation and anion columns are changed, the standby mixed bed column is moved forward, and a new standby tank is installed by the vendor. The carbon filter is changed every year. A blank from the deionized water unit is submitted to the NWQL annually by the Laboratory Coordinator and analyzed for trace elements, low level major ions, low level nutrients, and methylene-blue active substances. Blanks of deionized water are tested for specific conductance, turbidity, and bacterial growth monthly. A log book kept by the District Laboratory Coordinator contains records of all the activities mentioned above.

7.0 Analytical methods

7.1 Ohio District Laboratory

Most of the physical and chemical analyses for Ohio District projects are done either by NWQL or by approved contract laboratories; analyses done in the Ohio District laboratory generally are those that must be done within a few hours of sample collection but cannot be done conveniently in the field. General upkeep of the Ohio District Laboratory is the responsibility of the Laboratory Coordinator. The following is a list of the types of analyses that are done in the Ohio District and references to the method(s) used for each.

- **Bacteriological determinations.** The methods used for analysis of fecal-indicator bacteria are those of the USGS, USEPA, and APHA and others and are referenced below. Analysis of fecal-indicator bacteria also can be done in the field (Myers and Wilde, 1997).
- **Total coliform bacteria.** Membrane-filter method (Britton and Greeson, 1987).
- **Fecal coliform bacteria.** Membrane-filter method (Britton and Greeson, 1987; Bordner and Winter, 1978).
- **Fecal streptococcus bacteria.** Membrane-filter method (Britton and Greeson, 1987; Bordner and Winter, 1978).
- **Enterococci.** Membrane-filter procedure (U.S. Environmental Protection Agency, 1985, method 1106.1).
- ***E. coli.*** Membrane-filter procedure (U.S. Environmental Protection Agency, 1985, method 1103.1 and U.S. Environmental Protection Agency, 1991, method 1105).

- **Aquatic-organism enumerations and aquatic-habitat evaluations.** See section 4.3 of this manual.

Miscellaneous chemical determinations and procedures. The following are miscellaneous analyses that have recently been done by Ohio District personnel.

- **Hydrogen sulfide.** Titrimetric, iodometric method for sulfide (Fishman and Friedman, 1989). A field method for determination of hydrogen sulfide is mentioned in section 6.1.1.
- **Immunoassays for 2,4-D and atrazine in water.** Benchtop analyzer procedures (Ohmicron, 1992).
- **Soil-moisture extraction.** Saturation extract procedure (Page and others, 1982).
- **Sediment.** Grain-size analyses by dry-sieve procedures (Guy, 1969). (Sediment-concentration analyses could potentially be done in the Ohio District Laboratory, but the setup is not in place at this time.)

In the future, it may be necessary for analyses other than those listed above to be done in the Ohio District laboratory to fulfill project requirements. The District Water-Quality Specialist and the Laboratory Coordinator must be consulted before any unlisted analysis or determination is planned for the Ohio District laboratory.

The District Laboratory Coordinator is also responsible for providing services or maintaining supplies and equipment used in water-quality studies. Project personnel are strongly encouraged to rely on the services and supplies provided by the Ohio District laboratory to promote standardization and QA of sample collection and analysis. These services include but are not limited to the following:

- Maintain supply of calibration solutions, filling solutions, membranes, and standards for field meters (DO, pH, specific conductance, temperature, barometric pressure).
- Prepare and ensure the quality of deionized water.
- Maintain supply of containers and preservatives.
- Store hazardous materials used in water-quality work (flammables and corrosives).
- Order Standard Reference Water Samples (SRWS) in support of contract laboratories.
- Order QA/QC standards, organic matrix spikes, inorganic and organic equipment blank water, bacteria media, and sterile buffered water for project work.
- Order and maintain a stock of other miscellaneous water-quality supplies, such as bottles and pump tubing (sources are QWSU and private suppliers).
- Maintain fume hoods, laboratory balances, scales, microscopes, drying ovens, incubators, autoclaves, and other equipment.

The Laboratory Coordinator is available to advise project personnel regarding miscellaneous supplies and services. Laboratory Safety information can be found in Skinner and others (1983).

7.2 National Water Quality Laboratory

The NWQL is the primary source of analytical services for most of the project work done by the Ohio District. Information about the NWQL, as well as technical memorandums, is available from the NWQL Home Page on the World-Wide Web (http://wwwnwql.cr.usgs.gov/USGS). The NWQL's work includes (1) inorganic and organic determinations in water, sediment, and tissue, (2) biological determinations, and (3) radiochemical and stable-isotope-ratio determinations. Most of the NWQL's work is determination of physical properties and chemical constituents of water samples and of suspended and bed sediments; of these two domains of chemical analysis, analyses of water samples are the more commonly requested by the Ohio District. Each method of chemical analysis at the NWQL is assigned a lab code; in addition, suites of complementary analyses are grouped into schedules, which also have unique numbers. Laboratory schedules are an efficient means of selecting groups of related constituents for analysis. Commonly used schedules and associated laboratory codes are published annually in the NWQL Laboratory Services Catalog. All methods of analysis used at the NWQL are published among several volumes of manuals (for example, Fishman, 1993; Fishman and Friedman, 1989; and Wershaw and others, 1987).

NWQL divides its chemical work into two broad programs (Pratt, 1994):

- **Inorganic Chemistry Program.** This program offers analyses for trace elements, major cations and anions, nutrients, residue on evaporation, specific conductance, pH, and turbidity. Methods include automated and semiautomated colorimetry; atomic absorption spectrophotometry (AAS), including flameless AAS methods such as cold vapor AAS and hydride AAS; graphite furnace atomic absorption spectrophotometry (GF-AAS); inductively coupled plasma emission spectroscopy (ICP); inductively coupled plasma emission spectroscopy–mass spectrometry (ICP-MS); and ion chromatography methods for low-ionic-strength solutions.

- **Organic Chemistry Program.** This program offers analyses for insecticides, herbicides, and semivolatile and volatile organic compounds, total and dissolved organic carbon, methylene blue active substances (MBAS), and polychlorinated biphenyls (PCB) and other chlorinated hydrocarbons in water, bed material, and tissue samples. Methods include gas chromatography (GC), gas chromatography/mass spectrometry (GC/MS), and high performance liquid chromatography (HPLC).

In addition to doing the above-mentioned analyses, NWQL oversees biological, radioisotope, and stable-isotope analyses, as well as Department of Defense Environmental Contamination (DODEC) program analyses, that are done by approved contract laboratories (see section 7.4 of this manual).

Because the NWQL is a large central laboratory with a long history of operation, QA/QC activities have been documented for many years. Bias and variability of the individual methods used by NWQL are discussed in manuals on determination of inorganic substances (Fishman and Friedman, 1989; Fishman, 1993), minor elements (Barnett and Mallory, 1971), and organic substances (Wershaw and others, 1987); reports on laboratory performance are available as USGS Open-File Reports, published annually. Current QC charts for most methods of analysis in the Inorganic Chemistry Program are available online to NWQL users. A comprehensive examination of USGS policy on and practice of QA/QC (which relates closely to the work of NWQL) is given in Friedman and Erdmann (1982); the QA/QC guidelines used by the NWQL itself are given in Pritt and Raese (1995). Information on QA/QC issues related to sample collection, handling, and shipment to NWQL; internal performance reviews done by NWQL; and biological and radiochemical analyses done outside of NWQL can be found in other sections of this report.

NWQL is recommended for use whenever possible. If an unusual method is needed for your project but is not listed in the Laboratory Services Catalog, ask the District Water-Quality Specialist to contact NWQL before considering use of an outside laboratory. The Methods Research and Development Program at NWQL may be able to adopt or customize a previously unused method to meet your needs. Custom schedules also can be put together by NWQL to coordinate nonstandard suites of analyses.

The QWSU provides supplies the Ohio District and other WRD offices with supplies such as calibration solutions for pH and specific conductance, media kits and supplies for microbiological analyses, filters, samplers, sample bottles, sample splitters (churn type), preservatives, inorganic-free blank water, and other standardized and quality-assured materials and supplies for water-quality work. DENSUPPLY is a unit within the NWQL that also provides some supplies for water-quality work (spike kits, organic-free blank water, solid phase extraction cartridges). Project personnel are strongly encouraged to use the services provided by these two service units to promote standardization and QA of sample collection and analysis.

7.3 Other U.S. Geological Survey laboratories

Besides the NWQL, several other USGS laboratories offer a broad spectrum of analytical services. Some of these laboratories offer specialized analytical services that are not available elsewhere and are supervised by individual researchers or research groups that belong to Geologic Division, the National Research Program, or individual Districts in the WRD. QA/QC plans for such laboratories may or may not have been published; it is up to the individual project chief, in consultation with the District or Regional Water-Quality Specialist, to determine whether the laboratory QA/QC program is sufficient to meet the DQO's of the project.

7.3.1 Geologic Division

Bulk chemical analyses of rock or sediment samples are available through the Branch of Geochemistry (BOG) in Denver, Colo. These include major and trace-element determinations, forms of carbon and sulfur, and specialized methods such as selective extraction analyses. Submission of samples to BOG laboratories is done through the BOG Laboratory Coordinator. General procedures for handling, sample processing, analytical methods, use of instrumentation, and QA/QC procedures followed by BOG laboratories are given in Arbogast (1990).

The Branch of Coal Geology also maintains extensive laboratory facilities for major- and trace-element determinations in coal, coal-bearing rock, and coal-combustion materials. In addition, numerous research laboratories offer specialized physical and chemical analyses of a wide variety of geologic materials. Microprobe and scanning electron microscope facilities, which are sometimes used for District projects, are also maintained by various research laboratories in the Geologic Division. After consultation with the District Water-Quality Specialist, these laboratories are contacted directly by the project chief to arrange sample submission and analysis.

7.3.2 National Research Program

Many researchers who work in the WRD National Research Program (NRP) operate laboratories that are potentially available for use by Ohio District personnel. These laboratories typically are used for nonroutine determinations of the chemical and isotopic properties of water-quality samples. Examples include low-level tritium analyses by the Isotope Tracers Project Laboratory in Menlo Park, Calif., and stable isotope determinations of oxygen, hydrogen, carbon, sulfur, and nitrogen by the USGS Stable Isotope Laboratory in Reston, Va. Samples submitted to these laboratories are submitted through NWQL. Other NRP laboratories, such as the USGS Reston Chlorofluorocarbon Laboratory, offer specialized chemical, biological, or physical analyses, but are not currently administered by NWQL. District personnel interested in using such laboratories should consult with the District Water-Quality Specialist and the NRP researcher in charge of the laboratory when making arrangements for sample submission and analysis.

7.3.3 District laboratories

Several Districts of the WRD have in-house laboratories that can be used by Ohio District personnel. Examples of these include parts-per-trillion level mercury determinations by the Low-Level Mercury Laboratory in the Wisconsin District, trace-element analysis of sediment samples by the Sediment Partitioning Research Laboratory in the Georgia District, mineralogic analysis of rock and sediment samples by X-ray diffractometry by the laboratory in the California District (Sacramento office), and microbiologic characterization of ground-water and sediment samples by laboratories in the North Carolina District. Ohio District personnel interested in using these and similar laboratories should contact those laboratories directly for information regarding procedures for sample submission, but the District Water-Quality Specialist must be kept informed about such contacts.

7.4 Contract laboratories

Contract laboratories are occasionally used instead of the NWQL for three principal reasons: (1) a state, local, or regional cooperating agency suggests use of a contract laboratory of its choice, (2) for certain projects, the cooperator mandates use of a contract laboratory for technical or policy reasons, (3) a specific unusual analysis or set of analyses is outside the scope of work done by the NWQL.

For three specific categories of analytical data—radiochemical (Contracts/Radchem), DODEC, and biological (Bio/QA)—the NWQL has designated coordinators to provide advice to districts who require the services of contract laboratories. Chiefs of projects involving any of these three categories should, after informing the District Water-Quality Specialist, contact the appropriate NWQL coordinator for guidance as soon as use of a contract laboratory is proposed.

In all other cases where a contract laboratory is to be used for a project, the project chief is responsible for notifying the District Water-Quality Specialist. The District Water-Quality Specialist will then notify BQS, by memorandum through the Regional Hydrologist, of annual contract-laboratory use. Each contract laboratory should be reviewed and approved by BQS before environmental samples are submitted. Once approval has been granted, the work of the contract laboratory will be subject to ongoing review throughout the data-collection, data-analysis, and report-production phases of the project. Details on the approval process and required documents are given in OWQ Memorandum 98.03 and by Erdmann (1991a, 1991b). A brief summary of requirements follows:

- **Use of approved analytical methods.** All methods used in support of a USGS project must be described in laboratory-techniques manuals by the USGS, the USEPA, or the American Society for Testing and Materials (ASTM) or in "Standard Methods for the Examination of Water and Wastewater" (American Public Health Association and others, 1992). Any method or modification not included in any of these works must be approved by the OWQ before it can be used with USGS samples.

- **Review and approval of the contract lab's QA/QC plan.** All laboratories must have an approved QA/QC plan provided to WRD customers upon request. In addition, all analytical methods used must have documented standard operating procedures (SOP's).

- **Records Management.** The project chief is responsible for keeping up-to-date copies of (1) QA/QC plans for the contract laboratory and (2) summaries of analyses of QC samples.

- **Analysis of performance-evaluation samples.** The contract laboratory needs to obtain a satisfactory rating for analysis of performance-evaluation samples before it begins processing environmental samples; however, a preferable alternative is for the laboratory to be already participating in the SRWS program (see below).

- **Preliminary technical review of the contract laboratory.** The BQS will determine what type of review is needed (whether onsite or by conference call). A representative of the BQS will do the review; project chiefs and district specialists are expected to participate and provide documentation.

- **Participation in the Standard Reference Water Sample Program.** The contract laboratory will be required to participate in this program by incorporating all relevant and available SRWS into the analytical procedures for the project. The project chief should also encourage the contract laboratory to participate in non-USGS sample-testing programs, such as the Performance Evaluation Program of the USEPA.

- **Quality-Control Samples and Blanks.** The contract laboratory will be expected to analyze applicable QC samples (duplicate, independent standard, spiked, reference, and split), as well as blanks.

- **Ongoing Technical Reviews.** Review of the contract laboratory's work will be done on a 3-year cycle by the BQS. Project chiefs and district specialists usually participate in these reviews.

Although the requirements for use of a contract laboratory are thoroughly documented (Erdmann, 1991a, 1991b; OWQ Memorandum 98.03), the activities involved in preproject approvals and ongoing review can be complex and time-consuming. It is imperative that the project chief contact the District Water-Quality Specialist and the appropriate section chief immediately if it becomes necessary or desirable to use a contract laboratory in support of an Ohio District project.

8.0 Management, validation, reporting, and storage of data

Data collected for hydrologic investigations are monitored for bias and variability, checked for errors, validated, and stored in such a manner to facilitate retrieval and use by District personnel and others. Data are managed and stored in paper files and electronic files. Currently, in the WRD, electronic water data are processed and stored in NWIS. Retrieval and updating of water-quality data stored in the local data base is done through the "QWDATA" software program described in the NWIS users manual on the World-Wide Web at http://wwwnwis.er.usgs.gov/. Paper files include "watlists" generated from NWIS, field notes, and copies of ASR's.

8.1 Management of data

It is the responsibility of the project team to see that sample information is entered into the appropriate data base as soon as possible after returning from the field. Regular samples and QC samples are entered into different data bases by use of QWDATA. For samples analyzed by the NWQL, the information from the ASR's and completed field sheets are used to enter samples into NWIS. Samples analyzed by other laboratories must also be entered into NWIS. Field measurements are to be entered at this time by use of an appropriate data base field form designed by the Project Chief and approved by the District Water-Quality Specialist. After entering the sample information, note the "record number" generated by the computer and record it on the field sheet or ASR. The ASR's and the completed field sheets are filed in site folders by the project chief or project personnel.

Analytical results from the NWQL are retrieved weekly from the NWQL data base into the District data base by the Water-Quality Database Administrator and reviewed by the District Water-Quality Specialist. Samples that have not been entered into NWIS or samples with incorrect site header information will be rejected by the District data base. A list of these rejected samples are distributed to the project chiefs, who must return any corrections to the Water-Quality Database Administrator as soon as possible.

Analytical results from laboratory retrievals and any field information entered by project personnel are contained on computer printouts called watlists. Quality-control checks are done by the NWIS software, and QC failure flags are listed on the watlists. The most common QC failure flags include (1) sizeable differences between field and laboratory values for pH, specific conductance, and alkalinity, (2) a cation and anion balance that is not within the allowable range, (3) a ratio of dissolved solids to specific conductance that is below 0.55 or above 0.86, and (4) a dissolved concentration of a particular metal species or organic carbon that is greater than the total concentration. A more detailed discussion of these QC failures and appropriate remedies is given in Friedman and Erdmann (1982) and in NWQL Technical Memorandum 93-02 (U.S. Geological Survey, written commun., 1993). It is the responsibility of the project chief to carefully review the watlists for failure flags, errors, and omissions in a timely manner and to take appropriate remedial steps. Errors or omissions in the data base may only be changed by the Water-Quality Database Administrator or District Water-Quality Specialist. If a laboratory error is suspected, rerun requests are sent to the NWQL by the project chief on appropriate rerun request forms obtained from the District Water-Quality Specialist. After review of the watlists by the project chief, the watlists are filed by project personnel in the site files.

8.2 Validation of data

When all of the analytical results are complete, or sooner if needed, validation of the data is done by the project team. Validation of the data is the process wherein the regular and QC sample results are examined for errors and checked for bias and variability. The project team must tabulate regular data and check for errors or omissions. Procedures for tabulating the data in NWIS are given in the NWIS users manual on the World-Wide Web at http://wwwnwis.er.usgs.gov/. If data from more than one sampling round at the same site is available, the project team should compare constituent concentrations in order to identify obvious decimal errors and sample mixups. If an analytical result is suspected to be incorrect, the project team may request a rerun from the laboratory. The project team must also tabulate QC data to facilitate careful review. These include data on blanks, field replicate samples, replicate and standard reference samples sent to participating laboratories, and field matrix spikes. The QC sample results are evaluated in conjunction with the regular samples to assess the quality of the data resulting from field and laboratory activities. For example, results from field blanks are examined for sampling bias, and replicate samples are examined for sampling and laboratory variability. Details of statistical techniques are described in Taylor (1987) and Helsel and Hirsch (1992).

Quality-control data on analytical results from the NWQL may also be used by District personnel to validate data. For inorganic QC data generated at the NWQL, the BQS operates a blind sample program, the results of which are available in QADATA, an interactive program that allows users to retrieve the results through the USGS national computer network. The monthly report generated by the QA program includes statistical tables, control charts, and precision plots. Details of the QADATA program, including methods for interpretation of program output, are described in Lucey (1990) and on the World-Wide Web at http://btdqs.usgs.gov/bsp/qadatanew.htm. Information on QADATA program publications can be obtained at http://btdqs.usgs.gov/bsp/Publications.html. Organic QA/QC data generated at the NWQL are available upon request by District personnel. These data include surrogate recovery for each environmental sample, blank data for each sample set, and reagent spike recoveries. Information on interpreting organic QC data can be found in NWQL Technical Memorandum 94.07.

8.3 Reporting and storage of data

Water-quality data are published in hydrologic-data reports or interpretative reports. A summary of USGS and WRD policies pertaining to the publication of data and interpretive reports is contained in the WRD publications Guide (Alt and Iseri, 1986). All non-proprietary water-quality data collected during the water year are published in the USGS Water-Data Report (known as the "annual report") or in individual project data reports. Hydrologic-data reports make water-quality data available to users, but without interpretations or conclusions. Interpretive reports include such USGS outlets as Circulars, Professional Papers, Fact Sheets, and Water-Resources Investigations Reports, as well as non-USGS outlets, such as scientific journals, books, and proceedings of technical conferences. The District Water-Quality Specialist, project supervisor, and outside technical specialists will provide guidance in assuring that each water-quality report meets the highest technical standards.

The project chief is responsible for ensuring that all project data are accurately listed in the annual report. A list of the order of reporting water-quality data and number of significant figures can be obtained from U.S. Geological Survey Administrative Report "Preparation of Water-Resources Data Reports" (U.S. Geological Survey, Reston, Va., written commun., 1976). After a data set is tabulated and checked for accuracy by project staff, it is given to another District employee for a thorough review. The reviewer must follow a specified checklist of review activities provided by the District Water-Quality Specialist.

All original data recorded in the field, analyzed and recorded in the laboratory, or received from other sources must be archived. Original data is defined by Hubbard (1992) as follows:

> Original data—from automated data-collection sites, laboratories, outside sources, and nonautomated field observations—are those data unmodified as collected or received, once in conventional units. Original data shall be preserved in this form no matter how modified later.

In addition, the WRD maintains that all data published in WRD reports or used to support scientific analyses leading to conclusions in these reports must be archived in electronic and in paper form (Hubbard, 1992). Details of minimum accessibility requirements for different types of data are given in Hubbard (1992).

While the project is active, all paper files (including the original watlists, field notes, and ASR's) are to be kept in the office of the project chief or project support staff, preferably in site-file folders. Any kind of alternate organizational structure for paper files must be approved by the District Water-Quality Specialist. Contents of the site-file folders are discussed in section 5.1.1. Electronic files are kept by the project chief and project support staff in a project subdirectory.

At the conclusion of the project, the project chief will organize and archive paper and electronic project files for storage in the District, the Federal Records Center in Dayton, Ohio, and the World-Wide Web. Archive procedures are continuously being modified in the Ohio District, and the project chief must contact the District Archive Coordinator for current standard operating procedures. However, the following paragraphs contain general rules that apply for archival of information for all water-quality projects.

Selected paper project files will be placed in a project file folder, which will be maintained by the Archive Coordinator and filed by project number. The majority of paper project files will be boxed and archived at the Federal Records Center. Electronic project files will be written to CD-ROM (two copies); one copy will be included in the project archive boxes, and the second copy will be maintained by the District Computer Subunit. Details regarding the kinds of project records to be archived are listed on the Ohio District internal World-Wide Web page. Records include but are not limited to the following: (1) a copy of the project proposal, (2) copies of important correspondence and all quarterly progress reports, (3) important maps, model runs, and other interpretive information, (4) a copy of the final report, (5) a list of project personnel and their duties, (6) a compilation of all data-collection sites used in the project, with site identification numbers, and summary statements of the types of data collected at each site, and (7) miscellaneous information such as watlists, field notes, and ASR's.

9.0 Performance and system reviews

Performance and system audits provide independent evaluations of the quality of data collected. They provide checks on QA/QC plan design; field procedures; equipment cleanliness, calibration, and repair; employee technical expertise; and laboratory reliability and reproducibility, as well as ongoing data management and analysis.

9.1 Ohio District

The District policy is to do technical reviews of a project at approximately the 10-, 40-, and 70-percent stages of completion. It is the responsibility of the project chief to provide management and the District QW Specialist with a list of review dates in the project work plan. During the technical reviews, as well as other times throughout the project deemed appropriate by the District Water-Quality specialist, project QA audits will be done. The District Water-Quality Specialist will review the completed project QA checklist and the file of QA activities maintained by the project chief (see section 3.4). Figure 9.1-1 shows these checks and balances in a flow chart of general project activities that take place from the proposal stage through the end of the project.

Suggestions and recommendations made by various QA/QC reviewers are kept in the project QA file, where they can be referred to easily. A record of the project chief's response to reviewers also will be kept in the file as part of the audit process. Periodically (usually every 3 years), all of the water-quality projects are reviewed by an OWQ technical review team; therefore, suggestions from reviewers can be implemented before the project reaches a critical analysis stage.

The District requires annual calibration checks by the District Laboratory Coordinator of all meters by review of calibration notebooks and of employee use and care of meters. A QA check on the bias and variability of all field equipment used in the Ohio District to determine pH, specific conductance, and alkalinity is made biannually by the National Field Quality Assurance (NFQA) program. This program is run by the BQS in conjunction with the QWSU and involves analysis of several water samples for pH, specific conductance, and alkalinity. Results of these analyses are rated on a satisfactory/marginal/unsatisfactory basis for each parameter and are used to assess the current condition of District field equipment as well as field analytical skills of District personnel.

9.2 National Water Quality Laboratory

The USGS National Water Quality Laboratory (NWQL) analyzes most water-quality samples submitted by project personnel. Quality Assurance at the NWQL includes programs for documentation, data validation, and internal and external blind sampling. The NWQL's internal QA/QC program is detailed in Pritt and Raese (1995). The NWQL participates in several evaluation studies as follows:

- U.S. Environmental Protection Agency Water-Supply study—2 per year.
  - Determination of low-level concentrations of organic compounds and inorganic constituents in water.
- U.S. Environmental Protection Agency Water-Pollution study—2 per year.
  - Determination of high-level concentrations of organic compounds and inorganic constituents in water.
- Canadian Center for Inland Water Samples—4 per year.
  - Determination of trace-level concentrations of inorganic constituents in water.
- Branch of Technical Development and Quality Systems—2 per year.
  - Determination of low- and medium-level concentrations of inorganic constituents in water.
- National Oceanic and Atmospheric Administration—1 per year.
  - Determination of low-level concentrations of organic compounds and inorganic constituents in biological tissues and sediments.

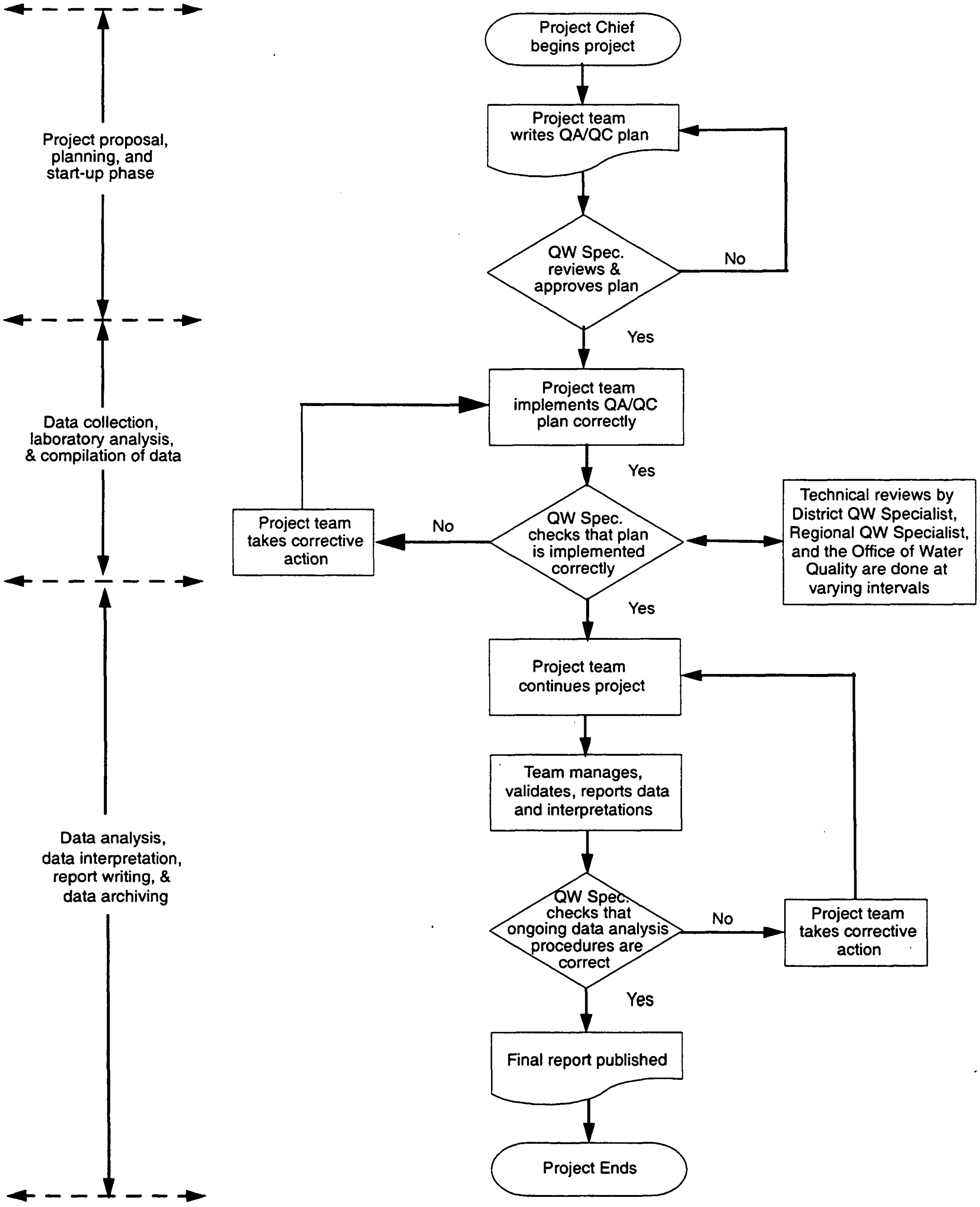


Figure 9.1.1. Summary of QA/QC procedures.

Results of these studies are reviewed by NWQL staff and management. Questionable results are investigated, and, if necessary, corrective action is taken (Pritt and Raese, 1995).

The NWQL also participates in the BQS's blind sample program, which is continuous. These external QA practices are in addition to the internal QA/QC measures employed routinely at the NWQL. A minimum of 10 percent of the samples sent to the lab are analyzed as internal QC samples (C.S. Gee, U.S. Geological Survey, written commun., 1995).

Project chiefs can review the laboratory's performance through the QADATA interactive program (see section 8.2).

9.3 Contract laboratories

All laboratories providing analytical services to the WRD (other than researchers providing their own) must be approved by the BQS through the District Water-Quality Specialist before they are put into service. Details of the approval requirements and ongoing review steps are given in section 7.4. This review and approval process ensures that all laboratories are capable of producing data of known and acceptable quality. Then, periodic checks and reviews are required to ensure that data quality is appropriately maintained. As part of the approval process, contract laboratories are reviewed in the following areas: use of appropriate, approved, and published methods, documented standard operating procedures, approved QA plan, types and amount of QC data and historical performance, participation in the Standard Reference Sample Project, scientific capability of personnel, and laboratory equipment. Office of Water Quality Technical Memorandum 98.03 and Erdmann (1991a, 1991b) describe the Division's policy on the use of laboratories providing analytical services for the WRD and the process necessary for approval. USGS Open-File Report 91-222 (Erdmann, 1991a) describes in detail the QA requirements for these laboratories.

The BQS publishes a bi-annual report that lists the results of laboratory performance in the SWRS program. The District Water-Quality Specialist will prepare a summary report for the District each year. It is the responsibility of the project chief to address any unsatisfactory results provided by a contract laboratory. All SWRS reports, an example of which is Farrar and Long (1996), are kept by the District Water-Quality Specialist.

Additional sample splits must be collected by project personnel if the contract laboratory does not have an internal blind standard reference water program. All of these checks provide assurance that data to be incorporated into a WRD project, regardless of the laboratory services provider, are of good quality. Erdmann (1991a) and OWQ Technical Memorandum 98.03 explain in detail the QA requirements for contract laboratories wishing to provide services to the WRD. Erdmann (1991b) contains a technical review of the performance of laboratories currently providing services to the WRD.

10.0 Corrective-action policy and procedures

When a significant condition that adversely affects data quality is noted at the project site or laboratory, the cause of the condition must be determined and corrective action must be taken to remedy the situation. All project personnel have the responsibility, as part of their normal duties, to promptly identify, report, and correct conditions adverse to quality.

Initiation of corrective actions may be warranted in the following situations, among others: (1) predetermined acceptance standards are not attained (that is, objectives for variability, bias, representativeness, comparability, and completeness), (2) equipment, instrumentation, or procedures are found to be faulty, (3) data compiled are determined to be erroneous, (4) QA requirements have been violated, (5) system and performance audits indicate the need, (6) management assessment indicates the need, or (7) laboratory/interlaboratory comparison indicates the need.

Possible corrective actions include modifying sample-collection methodology, reanalyzing samples at the laboratory, modifying equipment-cleaning procedures, resampling, accepting the problem but qualifying the data in the report, or rejecting data.

Documentation is maintained in the QA/QC file and includes a description of the adverse condition, the cause of the condition (if determined), the personnel who were notified regarding the condition, the corrective action taken, and results of corrective action.

Include a corrective-action strategy in the project work plan that outlines (1) predetermined limits for data quality that trigger corrective action if not met, (2) procedures for corrective action, and (3) identification of personnel responsibilities regarding corrective action (Shampine and others, 1992).

11.0 Quality-assurance/quality-control reports to management

The District Water-Quality Specialist is responsible for writing an annual report to District management on QA/QC activities in the District. This report will include the number of project QA/QC plans written, results of project QC samples analyzed, number of laboratory reruns requested, laboratory errors found by District personnel, results of the NFQA program, and performance of contract labs from the SRWS program.

12.0 References

Aloi, J.E., 1990, A critical review of recent freshwater periphyton field methods: Canadian Journal of Fisheries and Aquatic Science, v. 47, p. 656-670.

Aller, Linda, Bennett, T.W., Hackett, G., Petty, R.J., Lehr, J.H., Sedoris, H., Nielsen, D.M., and Denne, J.E., 1991, Handbook of suggested practices for the design and installation of ground water monitoring wells: Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Las Vegas, Nev., EPA/600/4--89/034, 221 p.

Alt, D.F., and Iseri, K.T., ed., 1986, WRD publications guide, v. 1. Publications policy and text preparation: U.S. Geological Survey, 429 p.

American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 1992, Standard methods for the examination of water and wastewater (18th ed.): Washington, D.C., American Public Health Association [variously paginated].

American Society for Testing and Materials, 1991a, Practice for determining suspended-sediment concentration in water samples: 1991 Annual Book of ASTM Standards, p. D3977-3980.

________1991b, Guide for sampling fluvial sediments: 1991 Annual Book of ASTM Standards, p. D4411-4484.

Anderson, J.R., Hardy, E.E., Roach, J.T., and Witmer, R.D., 1976, A land use and land cover classification system for use with remote sensor data: U.S. Geological Survey Professional Paper 964, 28 p.

Arbogast, B.E., ed., 1990, Quality-assurance manual for the Branch of Geochemistry, U.S. Geological Survey: U.S. Geological Survey Open-File Report 90-668, 184 p.

Ashmore, P.E., Yuzyk, T.R., and Herrington, R., 1988, Bed-material sampling in sand-bed streams: Ottawa, Ontario, Environment Canada, Sediment Survey Section, Method IWD-HQ-WRB-SS-88-4, 47 p.

Averett, R.C., 1973, Biological sampling and statistics, *in* Slack, and others, eds., Methods for collection and analysis of aquatic biological samples: U.S. Geological Survey Techniques for Water Resources Investigations, Book 5, Chapter A4, p. 6-26.

Averett, R.C., and Schroder, L.J., 1994, A guide to the design of surface-water-quality studies: U.S. Geological Survey Open-File Report 93-105, 39 p.

Barcelona, M.J., Wehrmann, H.A., and Vaarljen, M.D., 1994, Reproducible well-purging procedures and VOC stabilization criteria for ground-water sampling: Ground Water, v. 32, no. 1, p. 12-22.

Barnett, P.R., and Mallory, E.C., Jr., 1971, Determination of minor elements in water by emission spectroscopy: U.S. Geological Survey Techniques of Water-Resources Investigations, book 5, chap. A2, 31 p.

Bates, R.G., 1973, Determination of pH, theory and practice (2d ed.): New York, Wiley and Sons, 479 p.

Biggs, B.J.F., Duncan, M.J., Jowett, I.G., Hickey, C.W., Davies-Colley, R.J., and Close, M.E., 1990, Ecological characterization, classification, and modelling of New Zealand rivers—An introduction and synthesis: New Zealand Journal of Marine and Freshwater Research, v. 24, p. 277-304.

Bisson, P.A., Nielsen, J.L., Palmason, R.A., and Grove, L.E., 1982, A system of naming habitat types in small streams, with examples of habitat utilization by salmonids during low streamflow, *in* Armantrout, N.B., ed., Acquisition and utilization of aquatic habitat inventory information: American Fisheries Society, Western Division, p. 62-73.

Bode, R.W., Novak, M.A., and Abele, L.E., 1993, Twenty-year trends in water quality of rivers and streams in New York State based on macroinvertebrate data, 1972-1992: Albany, N.Y., New York State Department of Environmental Conservation, Stream Biomonitoring Unit, Bureau of Monitoring and Assessment, Division of Water, p. 7-16.

Bordner R., and Winter, J., 1978, Microbiological methods for monitoring the environment, water, and wastes: U.S. Environmental Protection Agency, EPA 600/8-78-017, 337 p.

Britton L.J., and Greeson, P.E., eds., 1987, Methods for collection and analysis of aquatic biological and microbiological samples: U.S. Geological Survey Techniques of Water-Resources Investigations, book 5, chap. A4, 363 p.

Brock, R.D., 1990, A protocol for on-site screening of volatile organic compounds using a portable gas chromatograph: U.S. Geological Survey Open-File Report 90-379, 15 p.

Brown, E., Skougstad, M.W., and Fishman, M.J., 1970, Methods for collection and analysis of water samples for dissolved minerals and gases: U.S. Geological Survey Techniques of Water-Resources Investigations, book 5, chap. A1, p. 39-41.

Burkehardt, R.W., and Gutreuter, Steve, 1995, Improving electrofishing catch consistency by standardizing power: American Journal of Fish Management. v. 15, p. 375-381.

Busenberg, Eurybiades, and Plummer, L.N., 1987, pH measurement of low-conductivity waters: U.S. Geological Survey Water-Resources Investigations Report 87-4060, 22 p.

Capel, P.D., Nacionales, F.C., and Larson, S.J., 1995, Precision of a splitting device for water samples: U.S. Geological Survey Open File-Report 95-293, 6 p.

Chapelle, F.H., 1993, Ground-water microbiology and geochemistry: New York, John Wiley and Sons, 424 p.

Claasen, H.C., 1982, Guidelines and techniques for obtaining water samples that accurately represent the water chemistry of an aquifer: U.S. Geological Survey Open-File Report 82-1024, 49 p.

Crawford, J.K., and Luoma, S.N., 1992, Guidelines for studies of contaminants in biological tissues for the National Water-Quality Assessment Program: U.S. Geological Survey Open-File Report 92-494, 69 p.

Cuffney, T.F., Gurtz, M.E., and Meador, M.R., 1993a, Guidelines for the processing and quality assurance of benthic invertebrate samples collected as part of the National Water-Quality Assessment Program: U.S. Geological Survey Open-File Report 93-407, 80 p.

Cuffney, T.F., Gurtz, M.E., and Meador, M.R., 1993b, Methods for collecting benthic invertebrate samples as part of the National Water-Quality Assessment Program: U.S. Geological Survey Open-File Report 93-406, 66 p.

Dahl, T.E., and Johnson, C.E., 1991, Status and trends of wetlands in the conterminous United States, mid-1970's to mid-1980's: Washington D.C., U.S. Fish and Wildlife Service, 28 p.

Driscoll, F.G., 1986, Groundwater and wells (2d ed.): St. Paul, Minn., Johnson Division, 1,089 p.

Dumouchelle, D.H., Lynch, E.A., and Cummings, T.R., 1990, A literature survey of information on well installation and sample collection procedures used in the investigation of ground-water contamination by organic compounds: U.S. Geological Survey Open-File Report 90-378, 60 p.

Edge, R.W., and Cordry, K., 1989, The HydroPunch—An *in situ* sampling tool for collecting ground water from unconsolidated sediments: Ground Water Monitoring Review, v. 9, no. 3, p. 177-183.

Edwards, T.K., and Glysson, G.D., 1988, Field methods for measurement of fluvial sediment: U.S. Geological Survey Open-File Report 86-531, 118 p.

Erdmann, D.E., 1991a, Quality assurance requirements for water-quality laboratories providing analytical services for the Water Resources Division of the U.S. Geological Survey: U.S. Geological Survey Open-File Report 91-222, 8 p.

________ 1991b, Technical review of water-quality laboratories providing analytical services for the Water Resources Division of the U.S. Geological Survey: U.S. Geological Survey Open-File Report 91-223, 19 p.

Farrar, J.W., and Long, H.K., 1996, Report on the U.S. Geological Survey's evaluation program for standard reference samples distributed in October 1995—T-137 (trace constituents), M-136 (major constituents), N-47 (nutrient constituents), N-48 (nutrient constituents), P-25 (low ionic strength constituents), and Hg-21 (mercury): U.S. Geological Survey Open-File Report 96-138, 106 p.

Fenneman, N.M., and Johnson, D.W., 1946, Physical divisions of the United States: U.S. Geological Survey, scale 1:7,000,000.

Fishman, M.J., ed., 1993, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of inorganic and organic constituents in water and fluvial sediments: U.S. Geological Survey Open-File Report 93-125, 217 p.

Fishman, M.J., and Friedman, L.C., eds., 1989, Methods for determination of inorganic substances in water and fluvial sediments: U.S. Geological Survey Techniques of Water-Resources Investigations, book 5, chap. A1, 545 p.

Frayer, W.E., Monahan, T.J., Bowden, D.C., and Graybill, F.A., 1983, Status and trends of wetlands and deepwater habitats in the conterminous United States, 1950's to 1970's: Fort Collins, Colorado State University, 31 p.

Friedman, L.C., and Erdmann, D.E., 1982, Quality assurance practices for the chemical and biological analyses of water and fluvial sediments: U.S. Geological Survey Techniques of Water-Resource Investigations, book 5, chap. A6, 181 p.

Gillham, R.W., and O'Hannesin, S.F., 1990, Sorption of aromatic hydrocarbons by materials used in construction of ground-water sampling wells, *in* Nielsen, D.M., and Johnson, A.I., eds., Ground water and vadose zone monitoring: Philadelphia, Pa., American Society for Testing and Materials Publication 1053, p. 108-122.

Gordon, A.B., and Katzenbach, M.S., 1983, Guidelines for use of water-quality monitors: U.S. Geological Survey Open-File Report 83-681, 94 p.

Gurtz, M.E., and Muir, T.A., 1994, Report on the interagency biological methods workshop: U.S. Geological Survey Open-File Report 94-490, 85 p.

Guy, H.P., 1969, Laboratory theory and methods for sediment analysis: U.S. Geological Survey Techniques of Water-Resources Investigations, book 5, chap. C1, 58 p.

_______ 1970, Fluvial sediment concepts: U.S. Geological Survey Techniques of Water-Resources Investigations, book 3, chap. C1, 55 p.

Guy, H.P., and Norman, V.W., 1973, Field methods for measurement of fluvial sediment: U.S. Geological Survey Techniques of Water-Resources Investigations, book 3, chap. C2, 59 p.

Hach Company, 1993, Procedures manual—DR/2000 spectrophotometer: Loveland, Colo., Hach Company [variously paginated].

Hardy, M.A., Leahy, P.P., and Alley, W.M., 1989, Well installation and documentation and ground-water sampling protocols for the pilot National Water-Quality Assessment Program: U.S. Geological Survey Open-File Report 89-396, 36 p.

Harrelson, C.C., Rawlins, C.L., and Potyondy, J.P., 1994, Stream channel reference sites—An illustrated guide to field technique: Fort Collins, Colo., U.S. Department of Agriculture, Forest Service, Rocky Mountain Forest and Range Experiment Station, General Technical Report RM-245, 62 p.

Hawkins, C.P., 1985, Substrate associations and longitudinal distributions in species of Ephemerellidae (Ephemeroptera: Insecta) from western Oregon: Freshwater Invertebrate Biology, v. 4, p. 181-188.

Helsel, D.R., and Hirsch, R.M., 1992, Statistical methods in water resources: New York, Elsevier, 522 p.

Hem, J.D., 1989, Study and interpretation of the chemical characteristics of natural water: U.S. Geological Survey Water-Supply Paper 2254, 263 p.

Horowitz, A.J., Demas, C.R., Fitzgerald, K.K., Miller, T.L., and Rickert, D.R., 1994, U.S. Geological Survey protocol for the collection and processing of surface-water samples for the subsequent determination of inorganic constituents in filtered water: U.S. Geological Survey Open-File Report 94-539, 57 p.

Horton, R.E., 1945, Erosional development of streams and their drainage basins—Hydrophysical approach to quantitative morphology: Bulletin of the Geological Society of American, v. 56, p. 275-370.

Hubbard, E.F., 1992, Policy recommendation for management and retention of hydrologic data of the U.S. Geological Survey: U.S. Geological Survey Open-File Report 92-56, 32 p.

Hupp, C.R., and Osterkamp, W.R., 1985, Upstream variation in bottomland vegetation distribution along Passage Creek, Virginia, in relation to fluvial landforms: Ecology, v. 19, p. 1-15.

Hupp, C.R., 1986, Upstream variation in bottomland vegetation patterns, northwestern VIrginia: Bulletin of the Torrey Botanical Club, v. 113, p. 421-430.

Hynes, H.B.N., 1970, The ecology of running waters: Toronto, Ont., University of Toronto Press, 555 p.

Johnson, G.P., 1997, Instruction manual for U.S. Geological Survey sediment observers: U. S. Geological Survey Open-File Report 96-431, 33 p.

Katzenbach, M.S., 1988, Economic comparison of two types of automatic water-quality monitors: U.S. Geological Survey Water Resources Investigations Report 87-4232, 24 p.

_________ 1990, Comparison of accuracy and completeness of data obtained from three types of automatic water-quality monitors: U.S. Geological Survey Water-Resources Investigations Report 89-4198, 65 p.

Kearl, P.M., Korte, N.E., Stites, Mike, and Baker, Joe, 1994, Field comparison of micropurging versus traditional ground water sampling: Ground-Water Monitoring & Remediation, v. 14, no. 4, p. 183-190.

King, P.B., and Beikman, H.M., 1974, Explanatory text to accompany the geologic map of the United States: U.S. Geological Survey Professional Paper 901, 40 p.

Knott, J.M., Sholar, C.J., and Matthes, W.J., 1992, Quality assurance guidelines for the analysis of sediment concentration by U.S. Geological Survey sediment laboratories: U.S. Geological Survey Open-File Report 92-33, 30 p.

Knott, J.M., Glysson, G.D., Malo, B.A., and Schroeder, L.J., 1993, Quality assurance plan for the collection and processing of sediment data by the U.S. Geological Survey, Water Resources Division: U.S. Geological Survey Open-File Report 92-499, 18 p.

Koterba, M.T., Wilde, F.D., and Lapham, W.W., 1995, Ground-water data-collection protocols and procedures for the National Water-Quality Assessment Program—Collection and documentation of water-quality samples and related data: U.S. Geological Survey Open-File Report 95-399, 113 p.

Kuckler, A.W., 1970, Potential natural vegetation, *in* The National Atlas of the United States of America: U.S. Geological Survey, p. 89-91.

Kuehne, R.A., and Barbour, R.W., 1983, The American darters: Lexington, Ky., University Press of Kentucky, 177 p.

Langmuir, Donald, 1971, E_H-pH determination, *in* Carver, R.E., ed., Procedures in sedimentary petrology: New York, Wiley and Sons, p. 597-634.

Lane, S.L., and Fay, R.G., 1998, National field manual for the collection of water-quality data—Safety in field activities: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A9 [variously paginated].

Lapham, W.W., Wilde, F.D., and Koterba, M.T., 1995, Ground-water data-collection protocols and procedures for the National Water-Quality Assessment Program—Selection, installation, and documentation of wells, and collection of related data: U.S. Geological Survey Open-File Report 95-398, 69 p.

__________ 1997, Guidelines and standard procedures for studies of ground-water quality—Selection and installation of wells, and supporting documentation: U.S. Geological Survey Water-Resources Investigations Report 96-4233, 110 p.

Leopold, L.B., Wolman, M.G., and Miller, J.P., 1964, Fluvial processes in geomorphology: San Francisco, W.H. Freeman, 522 p.

Leopold, L.B., and Wolman, M.G., 1957, River channel patterns—Braided, meandering, and straight: U.S. Geological Survey Professional Paper 282-B, p. 39-85.

Littler, M.M., and Littler, D.S., 1985, Ecological field methods—Macroalgae, *in* Littler, M.M., and Littler, D.S., eds., Handbook of phycological methods: New York, Cambridge University Press, 617 p.

Lucey, K.J., 1990, QADATA user's manual—An interactive computer program for the retrieval and analysis of the results from the external blind sample quality-assurance project of the U.S. Geological Survey: U.S. Geological Survey Open-File Report 90-162, 53 p.

Martin, G.R., Smoot, J.L., and White, K.D., 1992, A comparison of surface-grab and cross sectionally integrated stream-water-quality sampling methods: Water Environment Research, v. 64, no. 7, p. 866-876.

Martin, J. D., and Cohen, D.A., 1994, Policy and procedures for the management and archival storage of data collected for hydrologic investigations of the U.S. Geological Survey, Indiana District: U.S. Geological Survey Open-File Report 94-61, 36 p.

Martin-Hayden, J.M., Robbins, G.A., and Bristol, R.D., 1991, Mass balance evaluations of monitoring well purging, Part II, Field tests at a gasoline contamination site: Journal of Contaminant Hydrology, v. 8, no. 1, p. 225-241.

Matthes, W.J., Sholar, C.J., and George, J.R., 1991, Quality assurance plan for the analysis of fluvial sediment by laboratories of the U.S. Geological Survey: U.S. Geological Survey Open-File Report 91-469, 31 p.

Mathey, S.B., ed., 1989, National water information system user's manual, volume 2, chapter 4— Ground-water site inventory system: U.S. Geological Survey Open-File Report 89-587 [variously paginated].

McMahon, P.B., Chapelle, F.H., and Jagucki, M.L., 1992, Atrazine mineralization potential of alluvial-aquifer sediments under aerobic conditions: Environmental Science & Technology, v. 26, no. 8, p. 1556-1559.

Meador, M.R., Cuffney, T.F., and Gurtz, M.E., 1993a, Methods for sampling fish communities as part of the National Water-Quality Assessment Program: U.S. Geological Survey Open-File Report 93-104, 40 p.

__________ 1993b, Methods for characterizing stream habitat as part of the National Water-Quality Assessment Program: U.S. Geological Survey Open-File Report 93-408, 48 p.

Meyer, F.P., and Barclay, L.A., 1990, Field manual for the investigation of fish kills: U.S. Fish and Wildlife Service, Resource Publication 17, 120 p.

Merritt, R.W., and Cummins, K.W., 1984, An introduction to the aquatic insects of North America: Dubuque, Iowa, Kendall Hunt Publishing Co., 717 p.

Michigan Department of Natural Resources, 1991, Qualitative biological and habitat survey protocols for wadeable streams and rivers—GLEAS Procedure #51, Revised June, 1991: Michigan Department of Natural Resources, Surface Water Quality Division, Great Lakes and Environmental Assessment Section, 40 p.

Moffit, F.H., and Bouchard, H., 1992, Surveying (9th ed.): New York, Harper-Collins Publishers, 848 p.

Myers, D.N., and Wilde, F.D., 1997, National field manual for the collection of water-quality data—Biological indicators: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A7 [variously paginated].

Mueller-Dombois, D., and Ellenberg, H., 1974, Aims and methods of vegetation ecology: New York, John Wiley and Sons, 547 p.

National Atmospheric Deposition Program/National Trends Network, 1989, Every Tuesday morning—NADP/NTN deposition monitoring training for site operators: videotape, approx. 1 hour.

Newbury, R.W., and Gaboury, M.N., 1993, Stream analysis and fish habitat design, a field manual: British Columbia, Canada; Newbury Hydraulics, Ltd., 256 p.

Noether, G.E., 1987, Sample size determination for some common nonparametric tests: Journal of the American Statistical Association, v. 82, no. 398, p. 645-647.

Nordstrom, D.K., 1977, Thermochemical redox equilibria of Zobell's solution: Geochemica et Cosmochimica Acta, v. 41, p. 1835-1841.

Ohio Environmental Protection Agency, 1995, Technical guidance manual for hydrogeologic investigations and ground-water monitoring: Division of Drinking and Ground Waters.

_________ 1989, Biological criteria for the protection of aquatic life—Volume III, Standardized biological field sampling and laboratory methods for assessing fish and macroinvertebrate communities: Ecological Assessment Section, Division of Water Quality Planning and Assessment, Revision 6, WQPA-SWS-3, p. V-1-2 to V-4-43.

Ohmicron, 1992, Start-up manual for the RaPID Assay System: Newtown, Pa., Ohmicron Corporation [variously paginated].

Omernick, J.M., 1987, Ecoregions of the conterminous United States: Annals of the Association of American Geographers, v. 77, p. 118-125.

Osborne, L.L., and Wiley, M.J., 1992, Influence of tributary spatial position on the structure of warmwater fish communities: Canadian Journal of Fisheries and Aquatic Sciences, v. 49, p. 671-681.

Page, A.L., Miller, R.H., and Keeney, D.R., eds., 1982, Methods of soil analysis, Part 2, chemical and microbiological properties (2d ed.): Madison, Wis., American Society of Agronomy and Soil Science Society of America, 1,159 p.

Palcsak, B.B., 1996, Using the freeze-core method to collect streambed samples for determination of particle-size distribution: U.S. Geological Survey Open-File Report 95-466, 12 p.

Parnell, J.M., 1995, Screening for volatile organic compounds in soil and ground water by use of a portable gas chromatograph during field investigations at an Air Force installation in Ohio: U.S. Geological Survey Open-File Report 95-371, 15 p.

Parker, L.V., 1994, The effects of ground water sampling devices on water quality—A literature review: Ground Water Monitoring &Remediation, v. 14, no. 2, p. 130-141.

Patrick, R., and Reiner, C.W., 1966, Diatoms of the United States: The Academy of Natural Sciences of Philadelphia, Monograph 13, 688 p.

Peden, M.E, and others, 1986, Development of standard methods for the collection and analysis of precipitation: Cincinnati, Ohio, U.S. Environmental Protection Agency [variously paginated].

Pennak, R.W., 1991, Fresh-water invertebrates of the United States (3d ed.): New York, John Wiley & Sons, 628 p.

Platts, W.S.,.Megahan, W.F., and Minshall, G.W., 1983, Methods for evaluating stream, riparian, and biotic conditions: Ogden, Utah, U.S. Forest Service, General Technical Report INT-138, 70 p.

Porter, S.G., Cuffney, T.F., Gurtz, M.E., and Meador, M.R., 1993, Methods for collecting algal samples as part of the National Water-Quality Assessment Program: U.S. Geological Survey Open-File Report 93-409, 39 p.

Porterfield, George, 1972, Computation of fluvial-sediment discharge: U.S. Geological Survey Techniques of Water-Resources Investigations, book 3, chap. C3, 66 p.

Powell, R.M., and Puls, R.W., 1993, Passive sampling of ground water monitoring wells without purging—Multilevel well chemistry and tracer disappearance: Journal of Contaminant Hydrology, v. 12, p. 51-77.

Pratt, L.K., 1994, Using analytical services at the National Water Quality Laboratory: U.S. Geological Survey Open-File Report 94-26, 15 p.

Prescott, G.W., 1962, Algae of the western Great Lakes, with an illustrated key to the genera of desmids and freshwater diatoms: Dubuque, Iowa, William C. Brown Company, 977 p.

_________ 1970, The freshwater algae: Dubuque, Iowa, William. C. Brown Company, 348 p.

Pritt, J.W., and Raese, J.W, eds., 1995, Quality assurance/quality control manual—National Water Quality Laboratory: U.S. Geological Survey Open-File Report 95-443, 35 p.

Puls, R.W., and Paul, C.J., 1995, Low-flow purging and sampling of ground-water monitoring wells with dedicated systems: Ground Water Monitoring & Remediation, v. 15, no. 1, p. 116-123.

Radtke, D.B., 1997, National field manual for collection of water-quality data—Bottom-material samples: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A8 [variously paginated].

Reilly, T.E., Franke, O.L., and Bennett, G.D., 1989, Bias in groundwater samples caused by wellbore flow: Journal of Hydraulic Engineering, v. 115, no. 2, p. 270-276.

Ritter, J.R., and Helley, E.J., 1968, An optical method for determining particle sizes of coarse sediment: U.S. Geological Survey Open-File Report [unnumbered], 43 p.

Robbins, G.A., 1989, Influence of using purged and partially penetrating monitoring wells on contaminant detection, mapping, and modeling: Ground Water, v. 27, no. 2, p. 155-162.

Robbins, G.A., and Martin-Hayden, J.M., 1991, Mass balance evaluation of monitoring well purging, Part I, Theoretical models and implications for representative sampling: Journal of Contaminant Hydrology, v. 8, no. 1, p. 203-224.

Rosenberg, D.M., and Resh, V.H., 1993, Freshwater biomonitoring and benthic macroinvertebrates: New York, Chapman and Hall, 488 p.

Sandstrom, M.W., 1995, Filtration of water-sediment samples for the determination of organic compounds: U.S. Geological Survey Water-Resources Investigations Report 95-4105, 13 p.

Schertz, T.L., Childress, C., Kelly, V., Pederson, G., and O'Brien, M., in press, Workbook for preparing a district quality-assurance plan for water-quality activities: U.S. Geological Survey Open-File Report 98-98 [variously paginated].

Schumm, S.A.,1963, Sinuosity of alluvial channels on the Great Plains: Geological Society of America Bulletin, v. 74, p. 1089-1100.

Schroder, L.J., and Shampine, W.J., 1992, Guidelines for preparing a quality assurance plan for district offices of the U.S. Geological Survey: U.S. Geological Survey Open-File Report 92-136, 14 p.

Shampine, W.J., Pope, L.M., and Koterba, M.T., 1992, Integrating quality assurance in project work plans of the U.S. Geological Survey: U.S. Geological Survey Open-File Report 92-162, 12 p.

Shelton, L.R., 1994, Field guide for collecting and processing stream-water samples for the National Water-Quality Assessment Program: U.S. Geological Survey Open File-Report 94-455, 42 p.

Shelton, L.R., and Capel, P.D., 1994, Guidelines for collecting and processing samples of stream bed sediment for analysis of trace elements and organic contaminants for the National Water-Quality Assessment Program: U.S. Geological Survey Open-File Report 94-458, 20 p.

Shelton, L.R., 1997, Field Guide for collecting samples for analysis of volatile organic compounds in stream water for the National Water Quality Assessment program: USGS OFR 97-401, 14 p.

Skinner, E.L., Watterson, C.A., and Chemerys, J.C., 1983, Laboratory safety handbook: U.S. Geological Survey Open-File Report 83-13 [variously paginated].

Smith, C.L., 1985, The inland fishes of New York State: Albany, New York State Department of Environmental Conservation, 522 p.

Smith, G.M., 1950, The freshwater algae of the United States: New York, McGraw-Hill, 719 p.

Taft, C.E., and Taft, C.W., 1971, The algae of western Lake Erie: Bulletin of the Ohio Biological Survey, v. 4., 189 p.

Taylor, J. K., 1987, Quality assurance of chemical measurements: Chelsea, Mich., Lewis Publishers, 328 p.

Thatcher, L.L. Janzer, V.J., and Edwards, K.W., 1977, Methods for determination of radioactive substances in water and fluvial sediments: U.S. Geological Survey Techniques of Water-Resources Investigations, book 5, chap. A5, 95 p.

Thorpe, J.H., and Covich, A.P., 1990, Ecology and classification of North American freshwater invertebrates: New York, Academic Press, 911 p.

Thorstensen, D.C., 1984, The concept of electron activity and its relation to redox potentials in aqueous geochemical systems: U.S. Geological Survey Open-File Report 84-072, 45 p.

Thurman, E.M., Meyer, M., Pomes, M., Perry, C.A., and Schwab, A.P., 1990, Enzyme-linked immunosorbent assay compared with gas chromatography/mass spectrometry for the determination of triazine herbicides in water: Analytical Chemistry, v. 62, no. 18, p. 2043-2048.

Thurman, E.M., Goolsby, D.A., Meyer, M.T., Mills, M.S., Pomes, M.L., and Kolpin, D.W., 1992, A reconnaissance study of herbicides and their metabolites in surface water of the midwestern United States using immunoassay and gas chromatography/mass spectrometry: Environmental Science & Technology, v. 26, p. 2440-2447.

Timme, P.J., 1995, National Water Quality Laboratory 1995 services catalog: U.S. Geological Survey, Open-File Report 95-352, 120 p.

Trautman, M.B., 1981, The fishes of Ohio, with illustrated keys: Columbus, Ohio State University Press, 782 p.

U.S. Environmental Protection Agency, 1977, Sampling for organic chemicals and microorganisms in the subsurface—1977: Washington, D.C., EPA 600/2-77-176, p. 6.

________1982, Handbook for sampling and sample preservation of water and wastewater: Cincinnati, Ohio, Office of Research and Development, EPA 600/4-82-029, p. 251-254.

________1984, EPA requirements for quality assurance project plans for environmental data operations: Washington, D.C., Quality Assurance Management Staff, EPA QA/R-5.

________ 1985, Test methods for *Escherichia coli* and enterococci in water by the membrane filtered procedure: Cincinnati, Ohio, Environmental Monitoring and Support Laboratory, EPA 600/4-85/076, 24 p.

________ 1986a, Test methods for evaluating solid waste, physical/chemical methods: Washington D.C., U.S. Office of Solid Waste and Emergency Response, SW-846, EPA Method 3810, [variously paginated].

________ 1986b, Ambient water quality criteria for bacteria—1986: Washington, D.C., Office of Water Regulations and Standards, Criteria and Standards Division, EPA 440/5-84-002, 17 p.

________ 1987, Data quality objectives for remedial response activities—Development process: Washington D.C., Office of Emergency and Remedial Response and Office of Waste Programs Enforcement, EPA 540/G-87/003 [variously paginated].

________ 1991,Test methods for *Escherichia coli* in drinking water, nutrient agar with MUG membrane filter procedure: Cincinnati, Ohio, Office of Water, EPA 600/4-91/016, 2 p.

________ 1994, Guidance for the data quality objectives process: Washington, D.C., Quality Assurance Management Staff, EPA QA/G-4.

U.S. Fish and Wildlife Service, 1979, Classification of wetlands and deepwater habitats of the United States: Washington D.C., Biological Services Program, FWS/OBS-79-31, p. 13.

U.S. Geological Survey, 1977, National handbook of recommended methods for water-data acquisition [variously paginated].

________ 1989, Safety and environmental health handbook: Report 445-1-H [variously paginated].

Vermette, S.J., Peden, M.E., Willoughby, T.C., Lindberg, S.E., and Weiss, A.D., 1995, Methodology for the sampling of metals in precipitation—Results of the National Atmospheric Deposition Program (NADP) pilot network: Atmospheric Environment, v. 29, no. 11, p. 1221-1229.

Walpole, R.E., and Myers, R.H., 1985, Probability and statistics for engineers and scientists (3d ed.): New York, Macmillan, 639 p.

Ward, J.R., and Harr, C.A., 1990, Methods for collection and processing of surface-water and bed-material samples for physical and chemical analyses: U.S. Geological Survey Open-File Report 90-140, 71 p.

Wershaw, R.L., Fishman, M.J., Grabbe, R.R., and Lowe, L.E., 1987, Methods for the determination of organic substances in water and fluvial sediments: U.S. Geological Survey Techniques of Water-Resources Investigations, book 5, chap. A3, 80 p.

Wells, F.C., Gibbons, W.J., and Dorsey, M.E., 1990, Guidelines for the collection and field analysis of water-quality samples from streams in Texas: U.S. Geological Survey Open-File Report 90-127, 79 p.

Wheeler, J.B., Hoersch, H.M., McGlinchey, E.J., and Mahy, E., 1979, Methods and measurements of periphyton communities—A review: Philadelphia Pa., American Society for Testing and Materials, Special Technical Publication 690, 183 p.

Whitford, L.A., and Schumacher, G.J., 1973, A manual of freshwater algae: Raleigh, N.C., Sparks Press, 324 p.

Wilde, F.D., and Radtke, 1998, National field manual for the collection of water-quality data—Field measurements: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A6 [variously paginated].

Wilde, F.D., Radtke, D.B., Gibs, Jacob, and Iwatsubo, R.T., eds., 1998a, National field manual for the collection of water-quality data—Preparations for water sampling: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A1 [variously paginated].

________ 1998b, National field manual for the collection of water-quality data—Selection of equipment for water sampling: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A2 [variously paginated].

________ 1998c, National field manual for the collection of water-quality data—Cleaning of equipment for water sampling: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A3 [variously paginated].

________ 1998d, National field manual for the collection of water-quality data—Collection of water samples: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A4 [variously paginated].

________ 1998e, National field manual for the collection of water-quality data—Processing of water samples: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A5 [variously paginated].

Willoughby, T.C., 1995, Quality of wet deposition in the Grand Calumet River Watershed, northwestern Indiana, June 30, 1992–August 31, 1993: U.S. Geological Survey Water-Resources Investigations Report 95-4172, 55 p.

Wolman, G.M., 1954, A method of sampling coarse river-bed material: Transactions of the American Geophysical Union, v. 35, no. 6, p. 951-956.

Wood, W. W., 1976, Guidelines for the collection and field analysis of ground-water samples for selected unstable constituents: U.S. Geological Survey Techniques of Water-Resources Investigations, book 1, chap. D2, 24 p.

Wright, P.R., and Cunningham, W.L., 1994, Use of rotasonic drilling for field investigations of ground water at Wright-Patterson AFB, Ohio, in Proceedings of the Eight National Outdoor Action Conference, National Ground-Water Association, Minneapolis, Minn., May 23-25, 1994.

Yobbi, D.K., Yorke, T.H., and Mycyk, R.T., 1995, A guide to safe field operations: U.S. Geological Survey Open-File Report 95-77, 29 p.

Yuzyk, T.R., 1986, Bed material sampling in gravel-bed streams: Ottawa, Ontario, Water Survey of Canada and Environment Canada, Water Resources Branch, Inland Waters Directorate, Method IWD-HQ-WRB-SS-86-8, 63 p.

Zemo, D.A., Pierce, Y.G., and Gallinatti, J.D., 1994, Cone penetrometer testing and discrete-depth ground water sampling techniques—A cost-effective method of site characterization in a multiple-aquifer setting: Ground Water Monitoring and Remediation, v. 14, no. 4, p. 176-182.