

Part 4 –Data Collection: Field and Laboratory Methods

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I. Introduction

Sound water-resource management depends on the availability of reliable scientific data on which to base management decisions. To serve this purpose, data-collection and analysis activities should be governed by a clearly articulated monitoring design based on project objectives (Cogs 2 and 3 of the NWQMC monitoring framework, respectively). Thus, any scientifically valid water-quality investigation requires:

(1) data that accurately represent the water medium sampled under the intended spatial and temporal conditions, (2) use of appropriate methods that yield impartial and reproducible results, and (3) data of the type and quality to satisfy the purpose for which the data are collected (Keith, 1993; Horowitz, 1994; U.S. Geological Survey, 1997).

Documenting data quality is fundamental to the data-collection processes in the field and laboratory.

Documenting the quality of environmental data in monitoring programs is essential and integral to the entire data-collection process (ITFM, 1995a; Wilde et al., 1999; USEPA, 2000), and relates to how methods are selected, implemented, and quality-assured for water-quality-related field activities and laboratory analyses. Placing an emphasis on the quality of the data produced, rather than on a particular data-collection method, furthers comparability and interpretability of data within and among projects and fosters opportunities for collaboration and comparability among the scientific, regulatory, and land management sections of the federal, State, and private monitoring communities (Brass et al., 2000; NWQMC – Methods Board, 2001). This is of particular concern for long-term monitoring programs that seek to discern environmental patterns over time and across sampling locations, and for data sharing and synthesis over local, regional, and

national scales (Lenz and Miller, 1996; NWQMC – Methods Board, 2001; Wiley et al., 2003 (Dobson et al., 1999; Aas and Semb, 2001). Costly duplication of efforts often can be avoided when data-collection organizations use a standard practice for determining data comparability that is based on data quality. Emphasis on data quality also results in greater flexibility in methods selection and greater latitude in using and comparing new data-collection technologies as they become validated and available (NRC, 1995; Ward, 1996; Heinz, 2002).

Data comparability is the determination that water-quality monitoring data of known quality can be validly applied by entities other than the data originators, even if project objectives differ.

Recently, new tools and guidance have become available to help organizations: (a) determine the appropriate level of data quality needed for a given objective; (b) quantify or otherwise measure and document the quality of the data collected; and (c) select appropriate and compatible field and laboratory methods that will produce data of known and acceptable quality. The Methods and Data Comparability Board (Methods Board), under the auspices of the National Water Quality Monitoring Council, is engaged in interagency collaboration to identify, examine, recommend, and foster data-collection approaches and tools that facilitate collaboration among data-gathering organizations (see <http://wi.water.usgs.gov/pmethods/index.html> and <http://wi.water.usgs.gov/wicp/acwi/monitoring>). These tools are intended to improve the quality and transferability of data being collected, including data collected for purposes other than those for which the data were originally collected (Brass et al., 2000). This paper describes the use of some of these tools as it summarizes the major elements of data collection for water-quality monitoring projects; namely, the use of project objectives to determine field and laboratory methods; the development of sampling and quality-assurance plans to define data-collection tasks; and, the necessity for the absolute commitment of all project team members to the integrity of the data they collect.

II. Determining field and laboratory methods from project objectives

Accurate data, using the best possible methods, are useless if they do not address the study or program objectives. The strategy for collecting the data needed to meet the

goals of a water-quality monitoring project involves a process that is interwoven iteratively with the scientific approach developed for the project as whole, with particular emphasis on monitoring objectives (Cog 1) and study design (Cog 2) (Figure 1), but also including managing and interpreting the data (Cogs 4 and 5). Thus, the field and laboratory methods that should be used in a project will depend on the questions being asked, the decisions to be made based on the data collected, and the acceptable degree of risk in reaching an incorrect conclusion or decision.

To bring insight and intelligence to the monitoring effort, all project personnel should understand the purpose of the investigation and the study design. They also should understand project objectives sufficiently to determine when conditions exist that are adverse to fulfilling project objectives using pre-selected methods, and be cognizant of acceptable alternative approaches. Table 1 illustrates how data-collection and data-quality requirements vary, depending on the purpose of the project. Such requirements are addressed in the planning stages of the project, prior to monitoring activities.

One tool to help organizations establish appropriate sampling designs and to select appropriate data-collection methods is the **Data-Quality Objective (DQO)** process developed by the U.S. Environmental Protection Agency (USEPA, 2000; USDOE, 2000; Grumbly 1994; APHA, 2000; EPA Superfund program, USEPA, 1997a). The DQO process is a systematic, iterative, and customized planning framework through which project goals and objectives are articulated, appropriate types of environmental and quality-control data are determined, and tolerable levels of uncertainty are established that will support decisions (USEPA 1994, 2000; Crumbling, 2001). An example DQO might be: determine, to a 95% degree of statistical certainty, if there is a significant 50% change in average nitrate concentration over time at given sampling locations.

The DQO process can be useful in project design (Cog 2); for example, to help identify the geographic extent of the site and the distribution, frequency, and timing of sample collection. The objectives (often called DQOs) that follow from this decision process involves implementing the monitoring design under specific site conditions, which bears directly on representativeness of the data collected. The same method executed either at a

different type of field site or at a different time of day or season, may not perform with similar efficiency, precision, or bias. Measurements taken at disparate times over the course of a project could, in such cases, lead to non-comparable data and non-attainment of project data-quality objectives. The project DQO process dictates critical sample handling and laboratory method components such as the type of samples required (e.g., temporal or spatial composite, grab or isokinetic), and sample matrices to be tested (e.g., sediment, water, soil, air).

The DQO process also is used to define measurement-quality requirements, or acceptance criteria, for the data-quality indicators that are important to the project, such as sensitivity (e.g., the desired detection or quantification limit), selectivity (e.g., the specificity of the methods used to measure target analytes), and analytical accuracy and precision. Measurement requirements, in general, depend on the objectives of the monitoring program and must be defined *a priori* to avoid misleading information and, consequently, faulty data interpretation and ill-informed management decisions. For example, an emergency-response project may place emphasis on rapidity of data collection by using on-site field analyses, and have less need for precise laboratory methods (Table 1). A compliance monitoring program, on the other hand, will generally require precise, accurate laboratory methods to support compliance and enforcement actions and reduce the potential for false positive or false negative data (a false signal that a contaminant has been detected in the sample, or no signal, when, in fact, a contaminant is present).

To assist in identifying methods that fulfill study DQOs, the Methods Board has developed NEMI (www.nemi.gov), a web-based compendium that summarizes available performance information for laboratory and field methods.

If the goal is to select methods that can consistently yield the quality of data needed to fulfill the project objectives and measurement requirements identified through a DQO or similar process, it is useful to document performance characteristics by which methods can be objectively compared and the resulting data be realistically defined (Eaton and Diamond, 1999; NWQMC-Methods Board, 2001). Instead of using a prescriptive approach for selection of field and laboratory methods, a method-performance or

performance-based system (PBS) approach could allow for greater flexibility. Such flexibility can be important, for example, when a prescribed data-collection method is impractical or unsuitable at some field sites, but when similar data-measurement requirements and quality objectives are needed. A PBS approach could more easily allow use of new methods or new technologies (GAO, 2001). However, there are many outstanding implementation issues to be resolved in implementing a PBS, such as legal liability of laboratories and regulatory agencies, technical expertise needed of laboratory auditors, and appropriate methods verification procedures. Successful attempts using a PBS approach include: (a) National Environmental Laboratory Accreditation Committee (NELAC) laboratory standards for demonstrating method capability and documenting on-going performance (NELAC, 2000); (b) EPA's "streamlining" procedures for water methods (USEPA, 1997b); NOAA's Status and Trends Program (Cantillo and Laurenstein, 1998); EPA's solid waste methods program (USEPA, 1998); and (c) EPA's Alternate Testing Procedures (ATP) Program (USEPA, 1999). For either a PBS or a prescriptive method approach to data collection, critical review of the quality-control data generated using the method under various scenarios is mandatory to determine data quality and data comparability.

A PBS approach specifies documenting the quality of data obtained from a method without specification of the method itself (NWQMC-Methods Board, 2001).

III. Defining Data-Collection tasks: Sampling and Analysis (SAPP) and Quality-Assurance Plans (QAPP) Plans

The collection of scientifically defensible water-quality data depends not only on consistent implementation of appropriate methods based on project objectives, but also on clear instructions to the data collectors, meticulous documentation of the methods used, and data verification. Table 2 shows the general flow of specific tasks for developing an appropriate environmental monitoring effort, and illustrates the interdependence of the field and laboratory activities. Carefully prepared and peer-reviewed project plans provide a blueprint for implementing field and laboratory

activities and often include a sampling and analysis project plan (SAPP) and quality-assurance project plan (QAPP)(Koterba et al., 1995; Wilde et al., 1998; Bartholomay et al., 2003) These plans incorporate information and decisions from the DQO or other planning processes, and stipulate the appropriate field and laboratory methods to be used (USEPA, 2000). Development of these project plans is an essential tool in the conduct of any environmental investigation and is an indispensable reference, both for the team charged with collecting the data and often for later interpretation and assessment of results.

Although commonly treated as separate and independent operations, the field and laboratory components of the data collection process actually form a continuum in which the field and laboratory methods must be compatible with each other as well as project objectives. The evaluation of routine sampling and quality control methods, or development of new methods, is as important as the choice of analytical methods in terms of minimizing sample bias or interferences. A case in point is the increasing requirement for laboratories to lower the concentration levels at which constituents are analyzed, while the available field technology remains relatively static. Thus, mercury concentrations in water, for example, — capable of being analyzed in the parts per trillion — might be sampled using field methods and equipment that can only produce accurate results at the part per billion level. As laboratory method-detection levels decrease, sample vulnerability to contamination tends to increase exponentially, and, consequently, field sampling methods must be able to maintain sample integrity to accommodate the heightened analytical sensitivity.

Field Methods

A fundamental requirement for data collection activities involve an awareness and implementation of “good field practices;” for example, using standard procedures to prevent sample contamination, ensuring that data accurately reflect the characteristics of the sample collected, and integrating quality-control measures into all field activities (Wilde et al., 1999). Standard quality-assurance procedures need to be examined, refined, and often customized project by project, according to known and anticipated site

conditions, the resources and objectives of the investigation, and the data-quality requirements for the investigation. Thus, field methods must:

- Be capable of producing a sample without introducing a negative or positive bias to the data with respect to the detection level of the laboratory analytical method selected for the constituent(s) of interest;
- Incorporate quality-control measures to determine the range of variability to be associated with the sample data;
- Be thoroughly documented so that different methods can be compared with respect to the quality of the data they ultimately produce;
- Be consistently implemented, so that the data produced are comparable .

In order to define data-collection tasks for field personnel, a Sampling and Analysis and Quality-Assurance Project Plans (SAPP and QAPP) are best developed iteratively and as a team (Bartholomay et al., 2003). These plans should specify:

(a) the types and minimum amount of quality-control samples to be collected; (b) the qualifications and training needed by data collectors and other project personnel; and (c) a safety plan that specifies site-specific known or anticipated hazards.

In addition, a list of required minimum data elements, such as NWQMC's recommended core data elements, should be adopted and addressed in the sampling plan to enhance consistency and comparability among data sets and monitoring programs (NWQMC-Methods Board, 2001; NWQMC-Methods Board, 2002a).

Water Quality Data Elements for Chemical and Microbiological Data, (NWQMC-Methods Board, 2002a), provide a consensus-based list of core recommended metadata. Data elements are being developed for other types of methods and data (for example, macrobiological data).

When samples are collected from the field for laboratory analysis, they should be handled, preserved, and stored as indicated in the SAPP or QAPP and by the laboratory performing the analysis. Many monitoring and consensus-based standards organizations have excellent references on sample handling procedures for most analytes and types of media (e.g., ASTM 2000; APHA, 2000; USEPA, 2002; USGS, 1998). Unambiguous, accurate, and complete documentation is necessary for all data collected or recorded in

the field, including specific sampling locations and site conditions, date and time of collection, and other information as specified in the SAPP and QAPP.

Laboratory Methods

Laboratory methods should meet many of the same attributes listed above for field methods. Accurate and complete documentation of records is necessary. The project's sampling and QA plans should stipulate any non-routine laboratory sample-handling procedures in addition to the analytical methods selected. In particular, laboratory methods should:

- Be conducted in an independently accredited laboratory (NWQMC-Methods Board, 2002b)
- Meet the precision, accuracy, bias, sensitivity, and other data-measurement and data-quality requirements defined for the project in the matrices analyzed
- Incorporate on-going QA/QC protocols that document the quality of data generated (Pirkey, et al., 2003; Pritt et al., 1995)
- Be thoroughly documented and validated
- Be consistently implemented by trained analysts using appropriately calibrated equipment
- Ensure that sample holding times and preservation conditions are not exceeded.

Method performance in certain matrices (e.g. certain types of wastewater effluent, groundwater, leachates, or even some drinking waters high in dissolved solids) may be far different (poorer) than those same method characteristics based on laboratory reagent water or other relatively simple matrices (NWQMC-Methods Board, 2001; MDCB, 2002). Unless a laboratory conducts rigorous quality-control analyses on the matrix it is analyzing (which is now required in many newer compliance methods and should allow comparability to be assessed more easily), one cannot assume that the performance characteristics reported for the method have been achieved by a given organization or

The Methods Board and the NWQMC recommend the National Environmental Laboratory Accreditation Program (NELAP) as the preferred accreditation body for federal laboratory accreditation (NWQMC-Methods Board, 2002b).

program. Therefore, it is imperative that a laboratory archive permanent records of its on-going performance of a method.

IV. Data Integrity

Data integrity is critical to regulatory programs such as EPA's National Pollutant Discharge Elimination System (NPDES) program under the Clean Water Act because data accuracy is paramount to judging environmental compliance with applicable laws and for making correct management decisions. To regulatory authorities, data integrity issues are perhaps the most serious of all violations because such questions lead to the suspicion of fraud. To non-regulatory agencies, data integrity is equally important, since data generated is the basis for scientific and policy decisions that extend from local jurisdictions to the national level.

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| Data integrity is defined as data that are complete, accurate, and in compliance with appropriate regulations, policies and procedures. |
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The lack of data integrity refers not only to incorrect data but also to misleading or missing data, to statements that are incomplete, misleading, or withheld, and to initials or signatures that are misleading or not properly executed. These problems may lead to data being deemed unusable, suspicion of fraud, and valuable resources wasted.

A signature on any sampling or analysis record (e.g., field and laboratory notebooks, sample chain-of-custody forms) indicates that the signer has entered information or data accurately, reviewed the document, agree with its content, and has confirmed to the best of their ability its completeness and conformance to established standards or procedures. A signature also indicates that the signer has proper knowledge and training to review the document being signed. Such records should be independently checked and signed. Data-entry mistakes are a common cause of data-integrity loss, and should be remedied by a rigorously implemented system of independent checks and audits of hand-written or electronic data entries and computer-generated data tables or graphics.

V. Special Considerations and Future Directions

With the increasing awareness of potentially widespread health and environmental effects of various human-made chemicals and biological pathogens, there has been greater attention placed on monitoring new analytes and/or developing better technologies for detecting known contaminants at lower concentrations. For example, improved methods were developed to measure haloacetic acids and aldehydes in finished drinking water (USEPA, 1996) and sulfonylurea herbicides in ground and surface waters (Battaglin, et al., 2000). Application of microbial source-tracking techniques to monitoring programs is an area of expanding research (<http://water.usgs.gov/owq>). Many new biological analytes are being incorporated into mainstream monitoring programs; for example, methods have been developed for identifying and enumerating various viruses, protozoans, and other human pathogens previously considered rare or low priority human threats (Bushon, 2003; USEPA, 2002). Many of these require new technologies, necessitating extensive method development and validation. Validation of laboratory methods for new analytes is difficult because appropriate reference methods and reference materials are often lacking. A much wider array of reference materials, in different realistic matrices, will be needed to help validate new methods (NWQMC-Methods Board, 2001). Another challenge is demonstrating method comparability and performance criteria that meet regulatory objectives. Moreover, existing field methods and quality-assurance procedures for sample and data collection for these emerging analytes need to be evaluated and adjusted or new methods developed and tested.

The attention on homeland security, as well as other emergency situations, has also increased the demand for rapid methods and/or technologies that can measure analytes *in situ*. In many cases, private vendors are developing test kits or other rapid techniques, which analyze constituents that are generally measured using traditional laboratory methods (e.g., using immunoassay kits that measure certain pesticides or PCBs). A challenge in these cases is demonstrating method comparability and satisfactory method performance that meets regulatory objectives. The increasing usefulness of remote

sensing technologies to detect water quality patterns is another of the many areas that may change the way in which environmental data are collected in the future.

VI. Summary

Documentation of methods performance and data quality in data-collection activities for water-quality monitoring studies is the scientific foundation for collaboration and comparability among public and private-sector investigations. By documenting the quality of data obtained using given data collection methods, it is also possible for other organizations to determine whether those data are suitable for their use.

The selection of appropriate field and laboratory methods is driven by the need to ensure that project objectives are fulfilled. Determining a project's measurement-quality requirements is facilitated through a systematic process to determine data-quality objectives, and is implemented by active use of project-developed sampling and quality-assurance plans. Finally, confidence in the quality and impartiality of the data collected must be ensured through a rigorous system of quality-assurance protocols, training, performance and data audits, and peer review.

With the advent of more affordable and usable technologies at our disposal, data-collection methods will become more refined, resource-efficient, and, perhaps more automated. Regardless of the methods used, however, the basic principles of data collection outlined in this paper remain the same. Consistent use of these principles will improve the quality and usability of environmental data, thereby enhancing the NWQMC's goal of improving the quality of information used in environmental decision-making.

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Figure 1. Major elements and requirements of data-collection activities.

| Project planning: major elements | Major-element requirements |
|---|---|
| Identify objectives and design of monitoring project  | <ul style="list-style-type: none"> ▪ Study Objectives ▪ Monitoring Question ▪ Data-Quality Objectives (DQO) ▪ Measurement-Quality Objectives ▪ Sampling Design |
| Collect field samples and onsite data  | <ul style="list-style-type: none"> ▪ Sampling Plan (SAPPP) ▪ Field Certification and Training ▪ Documented protocols ▪ Field method performance ▪ Quality-Assurance Plan (QAPPP) ▪ Field-activities Safety Plan |
| Produce data from laboratory analyses  | <ul style="list-style-type: none"> ▪ Quality-Assurance Plan ▪ Method Comparability ▪ Laboratory Accreditation ▪ Reference Materials Availability ▪ Laboratory Method Verification |
| Manage Data | <ul style="list-style-type: none"> ▪ Required Metadata ▪ Data-Quality Documentation |

Table 1. Relationships among common data collection elements and various types of monitoring projects.

| Data Collection Elements | Selected Types of Monitoring Projects | | |
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| | Compliance Evaluations | Ambient Investigations | Emergency Investigations |
| <i>In situ</i> (field) measurement | Not necessary unless specified | Usually necessary | Preferred |
| Sampling design | Targeted | Statistical or targeted | Targeted |
| Definitive/confirmatory laboratory method | Routinely Required | Routinely required | Often required |
| Rapidity of analysis | Determined by regulatory holding times | Determined by analyte or project requirements | Important factor |
| Use of new, innovative, or advanced technologies (e.g., remote sensing) | As appropriate (slowly being incorporated) | Determined by project purpose, approach, objectives | Often encouraged |
| Quality assurance (QA) procedures; Quality control (QC) samples and metrics | Defined by regulatory programs, criteria, and measurement method | Fundamental requirements defined by organization. Additional QA/QC determined by the project or program systematic planning process | QA/QC provisions mandatory |
| Comparability | Uniform or comparable methods important | Currently, varies among programs and projects | Currently, comparability often unknown |
| Documentation | Defined by regulatory programs and criteria | Essential | Defined by program requirements |

Table 2. Generalized sequence of typical environmental monitoring project activities. [DQOs, Data-Quality Objectives; QA/QC, quality assurance and quality control; NEMI, National Environmental Methods Index; lab, laboratory; QAPP, quality assurance project plan; SAPP, sampling and analysis project plan]

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| (a) | Review project purpose, scope, and environmental framework in conjunction with the proposed project design (project design developed from DQOs, including QA/QC requirements and data use). Identify sample matrices, sample analytes, and field measurements. |
| (b) | Review field site information, including historical data; consider preliminary site assessment to refine sampling design and sample collection methods. |
| (c) | Review and identify appropriate available field data collection methods and quality control measures: e.g., using DQOs, through NEMI, other recommended references, or guidance sources, evaluate need for methods development to meet objectives. |
| (d) | Identify appropriate lab methods based on DQOs and associated field requirements; e.g., through NEMI. Evaluate need for methods development to meet objectives in consultation with the laboratory. |
| (e) | Select, order, and test appropriate field and lab equipment and supplies. |
| (f) | Document project protocols for field and laboratory activities, data entry, technical audits and peer review in planning documents, such as a SAPP or QAPP. SAPP basic elements: <ul style="list-style-type: none"> - Purpose of study, study design, DQOs, sampling locations, timeframe - Sampling schedule; sampling/QC methods, equipment, handling; safety plans - Laboratory methods, QC measures, accreditation/certification, rapidity of results to client QAPP basic elements: <ul style="list-style-type: none"> - Project management, problem definition, DQOs and QA protocols, training, accreditation/certification requirements - Overall design of measurement/data acquisition approach - Assessment and oversight procedures - Data validation and usability metrics |
| (g) | Identify ancillary data and collection methods. |
| (h) | Develop sampling schedule, work plan, sample- and data-management plans/protocols, safety plan, training plan, and schedule for technical review of data and data collection. Identify any accreditation/certification required for field and lab personnel and incorporate into project plans, as appropriate. |
| (i) | Communicate about field and lab methods to project personnel and provide each with project planning documents. |
| (j) | Communicate with the lab to ensure that project QC protocols will be incorporated in laboratory data collection tasks. Review lab and field data with respect to accomplishment of project objectives. |