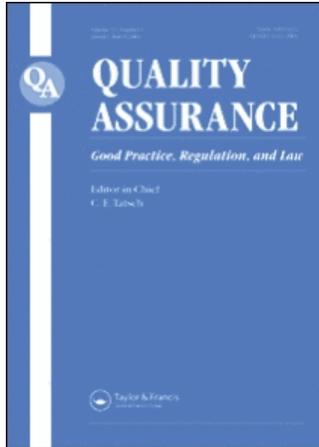


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ONE FISH, TWO FISH, WE QC FISH: CONTROLLING DATA QUALITY AMONG MORE THAN 50 ORGANIZATIONS OVER A FOUR-YEAR PERIOD

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ONE FISH, TWO FISH, WE QC FISH: CONTROLLING DATA QUALITY AMONG MORE THAN 50 ORGANIZATIONS OVER A FOUR-YEAR PERIOD

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EPA is conducting a National Study of Chemical Residues in Lake Fish Tissue. The study involves five analytical laboratories, multiple sampling teams from each of the 47 participating states, several tribes, all 10 EPA Regions and several EPA program offices, with input from other federal agencies. To fulfill study objectives, state and tribal sampling teams are voluntarily collecting predator and bottom-dwelling fish from approximately 500 randomly selected lakes over a 4-year period. The fish will be analyzed for more than 300 pollutants. The long-term nature of the study, combined with the large number of participants, created several QA challenges: (1) controlling variability among sampling activities performed by different sampling teams from more than 50 organizations over a 4-year period; (2) controlling variability in lab processes over a 4-year period; (3) generating results that will meet the primary study objectives for use by OW statisticians; (4) generating results that will meet the undefined needs of more than 50 participating organizations; and (5) devising a system for evaluating and defining data quality and for reporting data quality assessments concurrently with the data to ensure that assessment efforts are streamlined and that assessments are consistent among organizations. This paper describes the QA program employed for the study and presents an interim assessment of the program's effectiveness.

INTRODUCTION

The *National Study of Chemical Residues in Lake Fish Tissue* is a four-year, multi-million dollar effort led by EPA's Office of Water (OW) with extensive participation by 47 states, several tribes, each of the EPA Regional offices, and several EPA program offices. The primary

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objective is to estimate the national distribution of selected persistent, bioaccumulative, and toxic (PBT) pollutants in fish tissue from lakes and reservoirs of the continental U.S. However, results from the study will be useful to OW and other participants for a variety of other purposes, such as providing information to (1) fulfill objectives of the Clean Water Action Plan (CWAP), (2) support the EPA's PBT initiative, (3) answer important questions concerning the regional occurrence of fish tissue contamination, and (4) suggest specific areas in need of further study. Given the variety of potential uses for the data, the broad number of participants, the four-year duration, and the broad geographic range of the study, it was clear that a strong quality assurance (QA) program would be needed. Recognizing this, EPA OW managers included quality management activities throughout every phase of the study, beginning with the earliest planning phases. The result was a comprehensive QA program that addressed all aspects of study planning and implementation and that provides an effective means for real-time assessment and, where needed, improvement while the study is underway.

QA PROGRAM ELEMENTS

Planning

Collaborative Study Design

The Office of Water tasked a team of statisticians, biologists, and chemists from its Office of Science and Technology (OST) to work closely with experts from the Environmental Monitoring Assessment Program (EMAP) within EPA's Office of Research and Development (ORD) and with chemists in EPA's Office of Prevention, Pesticides, and Toxic Substances (OPPTS) to design the study. Together, the team developed a basic design framework and a draft list of possible target pollutants. The framework and proposed pollutant list were documented in a draft Study Design Document, along with an explanation of the team's rationale for the proposed design and target analyte list, issues considered by the team, and specific areas that the team felt should be further considered by a broader list of experts.

In October 1998, OW convened a two-day, invitation-only workshop to obtain peer input on the draft plan. More than 50 experts from federal, state, and tribal organizations (including EPA, NOAA, USGS, and state environmental, wildlife, and fisheries management agencies) were invited to participate. The draft study design document and a peer review charge were distributed in advance so that participants could arrive prepared with specific comments, questions, and concerns. After

describing the study objectives, resource limitations, draft design, and specific areas of concern, workshop participants were split into workgroups to consider four study design issues: (1) sampling design or approach; (2) pollutants of concern; (3) sampling methods; and (4) data management. The workshop concluded with workgroup presentations of their findings and recommendations for further consideration by OW study managers.

Use of the collaborative study design approach allowed OW to develop a study design that reflected lessons already learned by other organizations. At the same time, the design afforded an opportunity to build interest and gather support from the states, tribes, and Regions, on whom OW depended to collect study samples.

Use of Workgroup to Assist in Method Identification

In order to identify methods or techniques that would best meet OW's needs for the study, OW invited a group of recognized experts in the field of fish tissue analysis to participate in an Analytical Methods Workgroup. The workgroup was asked to assist in reviewing method development strategies and draft methods. Using guidelines suggested during the study design workshop, the workgroup sought to identify techniques that (1) minimized method development and validation costs; (2) yielded the lowest possible detection and quantification limits, and, where possible; (3) avoided the use of expensive or highly novel analysis techniques that could increase analytical costs. This process identified two existing methods that met study needs with no further testing, and several methods that required slight modification or testing to add target pollutants or optimize performance in tissue.

Selection of Methods with QC Elements That Support Quality Objectives of the Study

All of the methods used in the study, including those that were modified to meet study objectives, detail a comprehensive suite of laboratory QC elements needed to control and define the quality of results produced by each lab. These elements include: (1) required use of pure and traceable reference standards; (2) procedures for verifying that required detection and quantification levels are achievable by the laboratory; (3) procedures for demonstrating that the instrument is properly calibrated prior to and throughout sample analysis; (4) procedures for preparing, analyzing, and evaluating laboratory QC samples before analysis and during each shift to demonstrate the laboratory's ability to obtain precise and accurate results with the method; (5) use of either matrix spike samples or isotopically labeled standards to quantify recoveries of target analytes from tissue samples;

and (6) required analysis of blanks to demonstrate the absence of contamination.

Use of Approved Quality Assurance Project Plans (QAPPs) for Sampling and Analysis Activities

Two QAPPs were developed and approved by EPA to support this study. The *Quality Assurance Project Plan for Sample Collection Activities for a National Study of Chemical Residues in Lake Fish Tissue* (May, 2000) establishes data quality goals for all sample collection and handling activities and describes the QA/QC techniques employed by field teams and by the field support contractor to support those goals. (This sampling QAPP is available from EPA's website at <http://www.epa.gov/waterscience/fishstudy/sampling.htm>.) The *Quality Assurance Project Plan for Analytical Control and Assessment Activities in the National Study of Chemical Residues in Lake Fish Tissue* (September, 2000) establishes measurement quality objectives (MQOs) for laboratory data generated during the study and describes QA/QC techniques employed by laboratory and sample control contractor staff to ensure these MQOs are met. (This analytical QAPP is available from EPA's website at <http://www.epa.gov/waterscience/fishstudy/tissue.htm>)

Field Orientation/Training Program

Because the study design relied on a large number of state, tribal, and Regional sampling teams, the Office of Water conducted regional field orientation and training programs to ensure that personnel responsible for sampling activities within each organization understood the study objectives, were familiar with customized-paperwork developed to document sample collection activities, and were prepared to collect, document, and ship samples in accordance with the study design and the sample collection QAPP.

Implementation

Distribution of Study-Specific Sample Documentation and Sampling Kits

The study design calls for collection of fish samples by multiple teams from participating states, tribes, and EPA Regions. To date, more than 120 samplers representing 56 organizations have participated in the study. To ensure samples will be consistently documented by such a large and diverse group, several forms were custom-designed for the study. These forms include a Field Record Form to document information about each lake sampled and individual specimens collected

from the lake, a Sample Identification Label to accompany and identify each fish specimen, and Chain-of-Custody documentation. These forms are included in custom-made sampling kits prepared and distributed by EPA's sample control contractor. The kits also contain contaminant-free materials needed to store each specimen, a reference instruction sheet with contact phone numbers, and pre-completed forms needed to ship the specimens to the Sample Prep Laboratory for homogenization and compositing. Finally, sample Traffic Reports were created for use by the Sample Prep Lab to document each homogenized composite aliquot sent to either an Analysis Lab or to the Sample Repository for long-term storage.

Use of a Single Sample Prep Laboratory to Homogenize and Composite Samples

Many of the pollutants targeted in the study are being measured in the part per trillion or part per quadrillion range using state-of-the-art measurement techniques. For example, the quantification limit for mercury in tissue for this study is 2 ng/g, dioxin is being quantified at 0.1 ng/kg, and individual PCB congeners are being quantified at levels as low as 1 ng/kg. (Detection limits are even lower than these figures.) With monitoring levels this low for ubiquitous pollutants, it is critical to ensure that levels reported in samples reflect the true concentration of pollutants in samples and are not the result of contamination. Therefore, all sample processing activities (i.e., filleting, grinding, homogenizing, compositing, and aliquotting) are performed in a strictly controlled, clean laboratory and are associated with QC samples that will capture any problems with the sample prep processes.

Use of a Single Laboratory to Analyze a Given Pollutant throughout the Duration of the Study

As noted above, the study is statistically designed to determine the national distribution of pollutant residues in lake fish tissue. In any statistical analysis, lower measurement error translates to a higher level of confidence in final results. Nearly all of the QC measures described in this paper were designed to reduce sources of error. Use of a single laboratory to make all measurements for a given pollutant provided a rare opportunity to eliminate one source of error—inter-laboratory variability.

Prequalification of Laboratories

Prior to analyzing any samples collected in the study, each Analytical Laboratory was required to submit acceptable method detection limit (MDL) and initial precision and recovery (IPR) study results

generated in appropriate reference tissue matrix using the analytical method they would be using in the National Fish Tissue Study. MDL studies, which involve analysis of seven low-level (i.e., in the detection limit range) replicate samples, were to be conducted in accordance with the procedures given at 40 CFR 136, Appendix B. IPR studies, which involve preparing and analyzing four replicate reference standards spiked within the measurement range, were to be performed in accordance with the procedures given in each method.

Strong Communication Network

Routine contact with project staff and project participants is an integral aspect of the study design that has significantly contributed to the overall quality of data gathered in the study. The communication network employed in this study varies both in frequency (i.e., daily, weekly, monthly, annually) and medium (i.e., meetings, phone, email, fax), according to need. Highlights of this communication network include:

- Daily monitoring of sampling and laboratory activities: OW's contractor teams have been tasked with daily coordination and monitoring of sample collection, shipment and analysis activities. This monitoring has prevented unnecessary thawing of samples when shipping delays occurred and allowed OW to mitigate the impacts of deviations from the study design, thereby ensuring that limited study resources are used appropriately.
- Monthly project meetings: Each month, the study manager holds a meeting to discuss study status, schedules, and issues with other OW staff responsible for managing laboratory and data review activities and with the team of contractors responsible for daily tracking of activities. Depending on project activities, additional staff are brought into these meetings to facilitate planning and resolution of issues.
- Feedback to Study Participants: The OW Study Manager regularly communicates with study participants concerning the study status, issues, and concerns. Broad issues that affect all participants are disseminated via email. Examples include dissemination of progress reports, clarifications concerning the amount of dry ice needed for shipping, and requests to halt following the September 11, 2001, attacks. Specific concerns are discussed via telephone.
- Annual reporting of results: Analytical results, and associated data quality assessments, are being reported back to each state, tribe, and Region on an annual basis so that these organizations can evaluate their results and resolve any questions about their data prior to

public release. Public release of study results is delayed for six months to accommodate such reviews.

Implementation of Standardized Data Format

All data generated during the study are being compiled in a centralized, custom-developed database to ensure that results are reported to users consistently. The database allows for (1) eventual upload of results to the national STORET database system, (2) statistical manipulation of results, (3) export of results to user-friendly formats such as Excel spreadsheets, and (4) consistency in data format and nomenclature across laboratories and over time.

Assessment

Three Levels of Data Quality Assessment and Application of Standard Data Qualifiers

All analytical data generated in the study are subjected to three levels of review. First and as noted above, a pre-qualification review was performed on data submitted by each laboratory to demonstrate that the labs were qualified to prepare and/or analyze tissue samples collected during the study. Second, each submission of sample results is carefully scrutinized to verify that the samples were analyzed as directed and that supporting QC results demonstrated the quality of results generated. In evaluating these submissions, data reviewers employ a suite of standardized data qualifiers and abbreviated qualifier codes to consistently and accurately document the quality of all data generated so that both the primary data users (statisticians) in EPA Headquarters and secondary data users within Regions, states, tribes, and other organizations can make informed decisions regarding data use. A third level of data review is performed at the conclusion of each year and, ultimately, at the conclusion of the study, to determine if overall data quality supported study objectives.

Documentation of Data Quality in Annual QA Reports

With more than 50 different organizations interested in using data from this study, it is easy to imagine an unnecessary duplication of resources among organizations assessing data quality. To avoid this, OW thoroughly documented the procedures it was using to review the data, flag the data, and define data quality in a Quality Assurance Report for the National Study of Chemical Residues in Lake Fish Tissue: Year 1 Analytical Data. OW also documented all of its data quality findings in the report, and disseminated the reports to each study

participant, along with the reviewed, flagged, and qualified data. This report will be updated annually during the study.

Improvement

The study QA program recognizes that unanticipated challenges can arise and includes mechanisms to take corrective actions on specific situations and make programmatic changes that can minimize the potential for future problems of the same nature. For example, sample shipping instructions had to be modified to direct samplers to declare their shipments as having \$100 value after some Customs agents delayed shipping when they questioned how 75 lb coolers could be valued at less than \$10. A more dramatic example occurred when semi-volatile analysis activities started. Although preliminary testing suggested that a slightly modified version of Method 1625 would be capable of handling tissue matrices, the lipid contents encountered in the first batch of samples analyzed resulted in excessive interferences and the need for repeated reanalysis. Rather than allow such problems to result in excessive delays and reanalysis costs throughout the study, additional method modifications were developed and tested before initiating further analysis of study samples.

IS IT WORKING?

The first year of the study has been completed, and analytical activities for Year 2 are well underway. Data quality assessments from Year 1 indicated that:

- The Year 1 data set exceeded predefined study MQOs for precision, bias, sensitivity, and analytical completeness.
- 99.9% of the more than 100,000 field results gathered in Year 1 met all instrument calibration requirements.
- 98.8% of the Year 1 field sample results were not affected by blank contamination of any kind during the study.
- 99.7% of the Year 1 field sample results had no QC problems that would suggest sample matrix interferences.
- 99.5% of the Year 1 results had no QC problems that would suggest laboratory performance problems.
- 99.8% of the more than 100,000 sample results were determined within analytical holding times (even when re-analysis was required).