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Office of Water Quality Water-Quality Information Note 2004.04

Subject: Analytical Methods—Guidance for water-quality studies using research (unapproved) methods

Purpose: This Note provides guidance for planning and execution (including publication of results) of project activities that use research analytical methods (methods not approved under Office of Water Quality Technical Memorandum 98.05) to study water quality.

Scope: For the purposes of this discussion, research methods are analytical methods that do not meet the criteria of approved methods as outlined in Office of Water Quality Technical Memorandum 98.05 [http://water.usgs.gov/admin/memo/QW/qw98.05.html]. Research methods can include National Water Quality Laboratory (NWQL) custom methods as well as methods performed in laboratories of District researchers, National Research Program researchers, researchers from other USGS disciplines, and non-USGS research partners. Data generated using research methods require specific descriptions of the method and the quality of the resulting data if those data are to be published as part of USGS reports.

Background: Research methods are essential to the development of information on new and understudied water-quality issues and the general advancement of the scientific capabilities and contributions of the USGS. Essential goals in the development and use of research methods are that USGS data are relevant, accurate, defensible, and reproducible, and that USGS communicates information in an accurate, consistent, and objective manner. The discussion and recommendations made in this Note are intended to guide project activities to assure these goals are achieved.

Research methods often are used to analyze for new compounds that may start to appear in environmental water samples as contaminants and contaminant degradation byproducts. As such, there is a significant amount of uncertainty related to the compound characteristics, including transport properties, potential ecological and human health significance, chemical use and sources, and the related policy, licensing, and regulation. As a result, scientists investigating these chemicals must be familiar with a wide range of information regarding these contaminants, and increased attention must be given to the language used in publications presenting results of studies using research methods. This is particularly complicated in surveys that address a wide range of chemicals with different sources, uses, and characteristics.

Additional issues make interpretation and reporting of data from research methods more complicated. Some of these issues are:

- Research sometimes indicates that biological effects of some compounds may occur at lower thresholds than previously expected. For example, the levels of biological significance for some compounds, such as hormonally-active compounds, are in the sub-nanogram per liter (part-per-trillion) range, which is below our current reporting limits. There will be continuing need for lower detection levels to measure what may be biologically significant concentrations. This warrants additional attention, on a compound-by-compound basis, to the confidence in values measured below the reporting level and the decision as to whether they should be reported even for information-rich methods.
- By their very nature and purpose, research methods can change during their development and refinement. Reporting limits may be adjusted and compounds may be dropped due to poor performance or added based on new priorities or improved capabilities.
- Analysis of some sample matrices can have significant interferences from other organic compounds. Recent work on samples affected by municipal and animal-agricultural wastewaters are good examples of cases where such interferences occur. As a result, accurate identification and quantification of compounds can be affected in some samples, resulting in elevated reporting levels in just some samples. Furthermore, this suggests the increasing importance of the application of research methods to a wide range of sample matrices before method approval to better define the effects of, and appropriate response to, such interferences.
- Initially, quality-assurance data sets are sparse. Despite great pressure to analyze many samples, sample throughput for the research method is typically not as fast as a production method and the need for quality-control samples is even greater than long-standing production methods. Effective and consistent censoring of raw analytical data requires increased effort on quality-control samples, information from the research chemist on estimates of precision and bias from the lab, and field quality-control samples both for an individual project and for the broader set of samples analyzed in that chemist's lab.
- Research methods often do not have the capability to meet the large demand for analyses, as interest in a new contaminant or group of contaminants grows rapidly. The capability for production-scale analyses usually is not feasible until a method is approved for routine, production use at the NWQL or another approved lab.
- In many cases, research methods are developed in association with research plans for specific federal activities that have established research priorities. Popular demand for application of those methods may have objectives that differ from established research priorities and hard decisions need to be made related to the highest priority data that should be collected when activities may be limited by possible number of analyses and reasonable sample shelf-life and turn-around time.

Guidelines: For these reasons, the following recommendations are made:

The district or field office scientists leading the design and implementation of a field project using research methods is referred to as the project chief. The chemist that developed the research method and is analyzing samples from the field project is referred to as the research chemist.

- The research chemist should be contacted before a project activity utilizing their method is proposed to obtain their commitment to fulfilling the work outlined in the project work plan. Neither their participation in the project nor their sample analytical capabilities are to be included in the project work plan without that commitment.
- The research chemist, after making the commitment, should become a formal member of the project team, become involved in the project design, implementation, and report development, and serve as co-author of the resulting report.
- The research chemist will be responsible for fulfilling any commitments made for participation in additional project activities, which include, but are not limited to, sample analysis and the associated responsibilities as outlined herein.
- The research chemist will be responsible for including his/her commitments to other project activities in the work plan for their primary programmatic activity (for example, their National Research Program, Toxics Program, or NAWQA Program work plan).
- The research chemist will be responsible for prioritizing his/her commitments based on meeting the established research priorities for their primary programmatic activity (for example, the established research priorities of their National Research Program, National Water Quality Lab, or Toxics Program work plan).
- The project chief implementing field projects utilizing research methods will be responsible for consulting with the research chemist frequently during the course of the investigation regarding any changes in methods that may jeopardize the consistency of data and the reporting of that data. Issues that need to be discussed on a continuing basis include:
  - The status of the method.
  - Changes in methodology, reporting limits, compounds analyzed, etc. that may have occurred during the course of the project and how these changes may affect precision and bias of the measurements.
  - If changes have occurred, how to reconcile these over the existing data set.
  - Possible censoring of data based upon the most current laboratory quality assurance.
  - Possible censoring of data based upon field quality assurance from other studies.
- Similarly, the research chemist, once committed to participate in a project, will be responsible for communicating such issues (as listed in the previous bullet) to the project chief.
- The research chemist will be responsible for implementing a laboratory quality-assurance program and quality-control samples early in the method development process to help guide interpretation and use of data by other collaborators. In the absence of a long-term dataset suitable for establishment of a long-term method detection level, the laboratory quality-assurance program should be designed to document the method accuracy and precision at the reporting level identified by the research chemist. Data should not be reported below the level supported by that quality-control data. Even in cases where a long-term method detection level has been calculated, discretion should be taken as to how or whether data should be reported below that level, such as in cases where there is a very low frequency of detection and no detections above the long-term method detection level.

- The research chemist will be responsible for advising the project chief on implementation of a suitable field quality-assurance program, including quality-control samples, based on insight gained from other field activities.
- The project chief will be responsible for the design of a suitable field quality-assurance program, including quality-control samples, in consultation with the research chemist and any project leader of the federal research program that is leading development and testing of the research method.
- The project chief will be responsible for consulting with the research chemist on designing the appropriate number and type of quality-control samples needed for the project. Generally, a higher number and frequency of quality-control samples will be required for a research method than for a production method because of the issues mentioned above.
- It will be the mutual responsibility of both the project chief and the research chemist to define specifically any data qualifiers that need to be associated with data from a research method in the publication. The qualifier should not rely on a catchall phrase, such as estimated "E" value, but should specifically define the reason for qualifying data.
- It will be the mutual responsibility of both the project chief and the research chemist to collaborate and, if necessary, to contact the appropriate experts, including any other project leaders in the federal research program leading development and testing of the research method, on appropriate language for describing constituent transport, fate, effects, and any regulatory or policy implications in reports. Also, the research chemist must provide an appropriate description (preferably a citable published document) of the method and the quality of the data, which should be included in the report.
- Language must be included in the report that identifies the data as developed using research methods of the U.S. Geological Survey and explains that, as such, a description of the method and the quality of the data are presented in the published report or referenced in another published source. The description of data quality should include, at a minimum, analyses of lab spikes, lab blanks, spiked environmental waters, and field blanks, which together provide a qualitative description of precision and bias. An estimate of method detection limit should also be included.

Questions should be directed to the Chief of the Office of Water Quality.

WaQI Notes are archived on the internal Office of Water Quality web site, http://water.usgs.gov/usgs/owq/WaQI/index.html