CHEMICAL ANALYSES: HOW TO GET THEM DONE RIGHT (CUSTOMER-ANALYST INTERACTIONS)

M.D. Erickson, Environmental Research Division, Argonne National Laboratory, Argonne, IL 60439

ABSTRACT

The practical application of chemical analyses begins with a request for the analysis and concludes with provision of the requested analytical data. The key to successful execution of this activity is timely, professional communication between the requester and the analyst. Often chemical analyses are not satisfactorily executed, either because the requester failed to give adequate instructions or because the analyst simply "did what he or she was told." This article addresses the pitfalls of this interaction and provides suggestions for improving the quality of the analytical product through the requester-analyst interface. The request for and conduct of an analysis represents a contract for the procurement of a product (information about the sample); if both parties recognize and abide by this contractual relationship, the process generally proceeds smoothly. Note that the "customer" and "analyst" may be the same person, especially in a university research environment; these same issues hold even in this single-person interaction.

Chemical analysis is traditionally defined as the determination of what is in the sample and how much of that constituent. More rigorously, we also need to know if the analysis was conducted correctly, and we need documentation—in short, quality control. Before launching into a chemical analysis, it is important to establish the data quality objectives: what are we looking for, and how much error can we tolerate?

This presentation provides a practical tutorial on how to get chemical analyses done properly and cost-effectively. Suggestions for improving the quality of the analytical product through the requester-analyst interface are provided.

KEY WORDS

analytical, chemical analysis, procurement, management

INTRODUCTION

Chemical analysis is finding out "what is in the sample?" and "how much?" is there. The former is qualitative analysis; the latter is quantitative analysis. One must know what is in the sample before determining how much. Thus far, the definition is simple; when we try to define our quest, it becomes complicated. A typical (and irresponsible) request is for "everything." At the percent levels, this request may be appropriate; but as we descend into the trace and ultratrace level analyses, "everything" quickly becomes thousands of chemical species. Qualitative and quantitative analysis of thousands of constituents is clearly impractical. One common approach [1-4] is to define a "target analyte list," which limits the investigation to those analytes of significance; sometimes this list is based on technical merits (e.g., predicted synthesis products and by-products) or regulatory merits (e.g., the U.S. Environmental Protection Agency's priority pollutants). Another issue is how well do we

need to know the "how much?" Figures of merit such as accuracy, precision and limit of quantitation are the subject of extensive and ongoing technical and policy developments. Generally, improvements in figures of merit are achieved at rapidly escalating costs.

Emphasis on quality assurance (QA) in chemical analysis has increased dramatically in the past few years with the realization that data of unknown quality are virtually useless. The terms "QA" and "quality control" (QC) have often been used interchangeably or without discrimination (i.e., "QA/QC"). Quality assurance is generally defined as the program or structure within an organization that plans, designs and monitors the QC procedures and affirms the data quality in reports. Quality control is the term used to describe the activities in the QA program that control errors and define the data quality.

The QA program for a given laboratory, if it is already in place and operational, should need little adjustment for any specific analytical job; however, the QC must often be tailored to a specific analytical job, especially if it is in any way nonroutine. The first step in planning a QC program is to establish the data quality needed by the end user. At this point, the analyst and user must agree (or at least compromise) on the data quality objectives, specifically the degree of qualitative confidence, the limit of detection, the data confidence level (i.e., precision and accuracy), the validity of the method, and the amount of QC needed. Next, the analyst must design the QC program to fit these needs in the context of the analytical program. Finally, the analytical program is conducted. During this last phase, execution of all plans for QC is critical. The analyst must insert appropriate blanks and replicates. The QA staff must conduct audits. Management must insist on timely QA/QC reports so that corrective action can be taken. Last, and most important, the QC data and a data quality assessment should accompany the analytical report so that the user will know the data quality. The vast majority of peer-reviewed publications omit the last step, forcing the reader to speculate on the data quality.

PROCUREMENT AND EXECUTION OF CHEMICAL ANALYSES

Section I described the technical aspects of chemical analyses and discussed a few of the issues that the requester must consider in order to request the proper chemical analysis for his or her needs. This section focuses on how chemical analyses are procured and executed.

Chemical analyses begin with someone requesting the analysis and conclude with another person providing the requested analysis. The key to successful execution of this iteration is timely communication on a professional level. Often chemical analyses are not satisfactorily executed because either the requester failed to give adequate instructions or the analyst simply "did what he or she was told." The request for and conduct of a chemical analysis represents a contract for the procurement of a product (information about the sample); if both parties recognize and abide by this contractual relationship, the process generally proceeds smoothly.

Throughout this chapter, I use the monikers "requester" and "analyst" as if a single person is interacting with a single person. This assumption is not the real world. "Requesters" may be corporate purchasing agents working off of a scope of work, a sample-management office, a field-team leader, a project manager, a physician's office, or the analyst himself. The "analyst" that the requester communicates with may be a laboratory supervisor, a sample-receiving department, a salesperson for the laboratory, or the analyst himself. The "analyst" conducting the analyses is often a

team, where custody of the sample is passed from sample receiving to the extraction laboratory to the cleanup laboratory to the gas chromatography laboratory to the data-reduction person to the package-preparation person to the QC department for verification to shipping. Where a team of analysts is involved, the requester needs to have a central point of contact to minimize confusion and frustration. For the requester-analyst interface to work smoothly, it must function as if it is a one-to-one interaction; that is, the interface person has to know and communicate all of the required information as described subsequently.

Responsibilities of the requester

The requester must be sufficiently knowledgeable about both the overall problem and the chemical analysis to communicate a request to the analyst. In many cases, the general background of the problem (site conditions, regulatory background, origin of the sample, etc.) is important to the analyst. In all cases, the target analytes, the specific method, any regulatory reporting cutoff, the required limit of quantitation, custody requirements, QC requirements, reporting requirements and the schedule will be needed by the analyst. The end use of the data is important in planning the type of analysis and the level of data reporting. I have frequently heard analysts discuss the importance of a set of samples with respect to "this may be used in court," which is, unfortunately, all too often true. Conversely, many analyses that are presented in court are so poorly documented that their utility is seriously eroded, and the laboratory is perceived in a poor light. The detailed checklist presented in Table 1 may be modified as needed for specific organizations, projects and samples.

Responsibilities of the analyst

The analyst must ensure that he or she is sufficiently familiar with the sample(s) to conduct an appropriate analysis. Ask questions. If any of the information listed in Table 1 is not supplied by the requester and it could assist you in performing the analysis, ask for it. Communicate with the requester to clarify analytical objectives.

That the analyst communicate anticipated problems at the outset is important; for example, if meeting the requested detection limit will be a challenge, let the requester know, so that he or she is aware that there is some risk of not meeting the request. With complex, unusual, or previously unencountered matrices, communication is vital to ensure that matrix interferences (including chemical binding that precludes efficient and complete extraction) are known in advance and that the analysis can be planned accordingly.

When the analyses are reported, the analyst must document and report all deviations from the method and unexpected observations that may be of significance to the data user. Often copies of laboratory notebook documentation are sufficient and sometimes even necessary (readers are directed to Kanare [5] for a general background on preparation of laboratory notebooks). Discussing the data quality is especially important because many data users are not sophisticated enough to interpret the QC information (when it is provided) or to understand the inherent limitations of the method. Even when it is not specifically requested, providing a brief written explanation ("case narrative" in the terminology of some standard methods [4]) of the analysis, results and data quality may be an advisable policy.

To ensure success, the analyst must plan the analysis (e.g., availability of supplies, facilities, staff and instruments when needed), schedule the analysis to meet the requester's due date (preferably including a buffer for unanticipated problems and delays), take appropriate corrective action on problems encountered during the analysis,

and communicate with the requester when problems arise.

Procurement of chemical analyses

Specification of requirements

The requester must first determine the analyses to be requested, including the information listed in Table 1. The request must then be developed into a scope of work for a formal procurement or into a request for analysis for a less formal procurement. Where appropriate, this information must be communicated to the procurement organization.

Sample history
- General background on the problem
- Site conditions
- Regulatory background
- Origin of sample
- Safety issues (co-contaminants such as radionuclides or biohazards dramatically affect the execution and cost of analysis)
- Data use
    - Regulatory compliance
    - Litigation
    - Other

Analysis request
- Number of samples
- Receiving and storage requirements
    - Special handling considerations
    - Temperature
        - Room temperature
        - Cold room (4ºC)
        - Frozen
        - Other
- Custody requirements
- Target analytes
- Required method
- Limit of quantitation requested (specify units)
- Applicable regulatory reporting cutoff
- QC requirements (note any deviations from standard method, or develop in conjunction with analyst)

Reporting requirements
- Report results as individual analyte, "total mixture," by species, etc.
- Reporting units (micrograms per gram, micrograms per liter, etc.)
- Reporting basis (dry weight, lipid basis, etc.)
- Supporting documentation (specify format and order, or defer to standard method)
    - Case narrative or other text
    - Forms
    - Chromatograms
    - Notebook pages
    - Chain-of-custody records

Schedule
- Date (and mechanism) of sample's arrival at laboratory
- Date analyses are requested (do not request preliminary, unverified results via telephone)
- For large jobs, divide into batches, and determine schedules for batches

Cost
- Per-sample costs
- Additional costs for data packages, etc.
- Premium cost for expedited analyses
- Additional costs for data interpretation to users (e.g., testimony at trial)

Table 1. Analysis planning checklist.

Selection of a laboratory

Whether the procurement is formal or informal, the selection of the laboratory must be based on predetermined criteria. Cost is always a major consideration, but it should never be the only consideration. The stated ability to meet the request for analysis should be verified, preferably by verbal communication between the analyst and requester (i.e., the technical and not the contracting parties). If the organization is unfamiliar, investigate its reputation, capabilities and history through references, professional contacts and inquiries to the laboratory itself.

Selection of a customer

Laboratories should screen potential customers for both technical and business factors. If the customer cannot adequately articulate the request for analysis and the schedule, the laboratory can anticipate trouble with the analysis. If the request is beyond the laboratory's capabilities, the work should not be accepted. The laboratory must know and accept the final use of the data to avoid conflict of interest. The customer's history and capability to pay for the work should be investigated.

Execution of a contract

Whether a formal contract or an in-house analysis is requested, the parties need to agree on all factors concerning the analysis. These factors should be specified as fully as possible to avoid later disputes. When a contract for the analysis is in place, it may be changed as needed during the course of the analysis, subject to agreement by both parties. As always, communication and documentation are critical to a successful analysis.

Contract management

Once the parties agree to the contract, the work begins. The key to successful management (from the requester's viewpoint) and execution (from the analyst's perspective) is communication. Problems, schedule changes, delays, potential overruns and other relevant factors need to be communicated between the parties in a timely fashion and resolved before the problems get out of hand.

CONCLUSIONS

Chemical analysis, like other research activities, requires information, planning, communication and documentation. With sufficient amounts of each component, chemical analyses can be useful to the research project; with insufficient amounts of any one component, projects will suffer. This article has provided an overview of the considerations required to successfully analyze samples. With a clear objective and effective communications, the process need not be burdensome.

ACKNOWLEDGMENT

This work was supported by the U.S. Department of Energy, Assistant Secretary for Environmental Management, under contract W-31-109-Eng-38.

REFERENCES

1. American Society for Testing and Materials, Annual Book of ASTM Standards, Philadelphia, 1991.

2. U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd ed., Office of Solid Waste and Emergency Response, Washington, DC, 1986.

3. U.S. Environmental Protection Agency, Methods for the Determination of Organic Compounds in Drinking Water, EPA Report No. EPA/600/4-88/039, Washington, DC, 1991.

4. U.S. Environmental Protection Agency, U.S. EPA Contract Laboratory Program: Statement of Work for Organic Analy-

sis, OLM03.1, EPA Report No. EPA/540/R-94/073, Office of Emergency and Remedial Response, Washington, DC, 1994.

5. H.M. Kanare, Writing the Laboratory Notebook, American Chemical Society, Washington, DC, 1985.