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EPA

Guide to Laboratory Contracting

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1 INTRODUCTION

This document is intended to provide wastewater and drinking water treatment plants, states, industrial facilities, and other parties requiring analysis of environmental samples with guidance on laboratory contracting. It is based on laboratory contracting guidance developed specifically for the Office of Wastewater Management for use with pretreatment authorities and industrial users. This document provides guidance on the entire laboratory contracting process, from determining when to use a contract laboratory rather than perform in-house analyses to development of the analytical requirements and solicitation of the contract, to evaluation of laboratories and data review.

Regardless of the reason for the analyses, or the nature of the organization requiring laboratory services, the basic steps needed to ensure that reliable, useable data is generated in a timely manner are the same. These steps are outlined in the box below.

Critical Steps to a Successful Laboratory Contract

Step 1 – Clearly define your analytical needs

- ✓ Number and type of samples
- ✓ Applicable method(s)
- ✓ Quality control (QC) requirements

Step 2 – Develop a clearly defined contract

- ✓ Analytical and QC requirements
- ✓ Data deliverables
- ✓ Data turnaround
- ✓ Contract enforcement clauses

Step 3 – Solicit and award the contract openly and fairly

- ✓ Use standardized bid sheet
- ✓ Ensure laboratory is qualified before awarding the contract

Step 4 – Maintain communications with the laboratory after sample receipt

Step 5 – Thoroughly review the resulting data

Each of these general steps, as well as all of the other steps in between, are discussed in detail in this guide. By following the guidance in this document, you can obtain the data you need, when you need it, at reasonable costs and with minimal analytical problems.

2 WHEN SHOULD YOU OUTSOURCE ANALYTICAL SERVICES?

A fundamental decision when planning for laboratory analyses is whether the analyses should be performed by an in-house laboratory or whether the project should be contracted out to a commercial laboratory. Two factors impact whether the in-house laboratory can accept an analytical project: capacity and capability. An in-house laboratory must have: (1) the capacity to analyze the quantity of samples requested within the required time period, (2) the instrumentation expertise to perform the required analyses, and (3) staff qualified to perform the analyses.

If your laboratory does not have the capacity or the capability to handle the analytical requirements, you must decide whether it is appropriate to outsource the analyses or to increase the capacity or capability of the in-house laboratory. This decision is a function of time and cost.

The time required to increase the laboratory's capacity or capability will include the time required to identify and obtain the necessary equipment and instrumentation, the time necessary to bring instrumentation on-line, the time necessary to train staff in new procedures, and the time required to perform any necessary start-up tests and demonstrations of analyst capability. Each of these must be completed prior to project deadlines.

Determining the cost-effectiveness of increasing capacity or developing additional capability is a complex issue, however, the equation below provides a framework for this decision:

$$\frac{((I/5)+(F_t \times F_i)+(A(S)\times P)+(C\times T)+R+U)O}{N}=x$$

Where:

I = cost of required instrumentation

5 = years before instrument depreciation reaches 100%

F_i = square footage required for instrument and work area

F_t = square-foot cost of total floor space per year

A = number of analysts required to perform the analysis

S = annual salary of analysts

P = percentage of each analyst's time dedicated to the new capability (for example, 0.80)

C = number of hours of calibration and maintenance required per year

T = hourly cost of technician for maintenance, if not the primary analyst

R = annual cost of required reagents and other supplies, such as pressurized gas or liquid nitrogen

U = increase in annual utility cost to power the instrument and provide air conditioning

O = overhead factor for management (generally 1.2 - 2.0)

N = number of analyses per year anticipated over five years

Estimates for the annual number of hours of calibration and maintenance time (C) typically can be provided by instrument vendors or in-house staff with previous experience operating these instruments. In some cases, facilities may have instrumentation that is no longer in use, but may be serviceable. In such cases, the cost of required instrumentation (I) should be omitted from the equation. Similarly, the square footage required for the instrument and work area (F_i) and the cost per square foot of total floor space per year (F_t) should be modified to reflect only the added work area space needed to operate the already-housed equipment.

If $x \leq$ the average per-sample analysis cost at a commercial laboratory for the same analysis, you should consider expanding the your in-house laboratory capability accordingly, and begin analyzing such samples in-house. However, if $x \geq$ the average per-sample analysis cost at a commercial laboratory, you should consider contracting the analyses with a commercial laboratory. This guidance manual addresses the subsequent steps that should be taken to ensure delivery of quality data after the decision has been made to out-source the sample.

3 DEVELOPING AN ANALYTICAL CONTRACT

Although most organizations have established procedures and policies governing the purchase of services and supplies, these procedures seldom lend themselves to the purchase of analytical services, primarily due to the difficulty in defining the required services. This chapter provides a basic framework for defining the technical and contractual requirements associated with purchasing analytical services.

3.1 Defining the Project Parameters

Many laboratories have recognized the importance of customer service and employ staff who are trained to assist clients in defining specific requirements that meet their needs. Other laboratories, large and small, rely solely on the client to define these requirements. Still another group of laboratories, albeit a small group, perform analyses with little regard to the client's actual needs. One of the problems that arises when the client's requirements are poorly defined is the use of inappropriate methods. As detailed in Section 3.2, approved methods must be used for compliance monitoring purposes. It is not the laboratory's place to decide that an alternate method, even if it is an alternate EPA method, is "close enough." You are responsible for defining and ensuring that contract laboratories adhere to requirements that are consistent with your monitoring requirements. The first step in developing an analytical services contract is identifying the *who, what, why, when,* and *how* of the project. The remainder of this subsection provides guidance for defining these project parameters.

3.1.1 Client Information

Who is the name of the client, whether this is a single wastewater or drinking water plant or industrial facility, a utility or company that oversees several plants, or a third-party organization interested in generating original data. Client information should include specific contact points for use by the laboratory, including the following:

- The name of the person responsible for communicating with the laboratory regarding shipping delays and broken samples (a sample control contact)
- A technical contact for resolution of analytical questions or problems (a technical contact)
- An administrative contact for invoicing and payment (a billing contact)

Include with the contact name their address, telephone and fax numbers, and email, as these contacts often are in different locations.

3.1.2 Number, Frequency, and Matrix of Samples

What describes the samples to be analyzed, including:

- Number of "billable" samples and number of "unbillable samples." Billable samples (sample analyses for which the laboratory will be paid their per-sample cost) typically

include field samples, while unbillable samples (sample analyses for which the laboratory will *not* be paid) typically include internal laboratory quality control (QC) samples, such as blanks and ongoing precision and recovery (OPR) samples. Because the billable nature of some samples, such as matrix spike/matrix spike duplicate (MS/MSD) samples and field blanks is addressed differently by different organizations, you must clearly define which types of samples will be billable and which types will be unbillable, and provide the number of each type anticipated.

- Frequency with which samples will be sent to the laboratory (for example, five samples per week for eight weeks). This is particularly important for long-term projects, as it allows laboratories to accurately evaluate whether they have the capacity to analyze the samples and may result in volume discounts.
- Analyses required (for example, volatile organics, pesticides, or metals). Methods at 40 CFR Part 136 must be used for any National Pollutant Discharge Elimination System (NPDES) analyses unless otherwise specified in your NPDES permit. Drinking water utilities must use methods listed at 40 CFR Part 141.
- Sample matrices (for example, raw source water or finished drinking water, wastewater, sludge, or solids)

The number of samples, frequency of collection, and the types of analyses required are relatively straightforward issues, if properly defined, as noted above. However, special care should be taken to accurately and thoroughly describe the sample matrix because laboratories often attribute the inability to measure the concentration of a pollutant in a specific wastewater to "matrix problems." Most matrix interferences can be overcome with sufficient time, equipment, and procedures. Therefore, it is important to provide laboratory staff with as much advance information as possible concerning sample matrices so that the staff can adequately plan for analysis of any complex samples and avoid delays, unanticipated cost increases, or the generation of unusable data after samples are collected and shipped.

Examples of potential matrix problems include samples that contain high levels of organic compounds and samples that appear to be biphasic. Generally, the laboratory analyst should be responsible for evaluating specific matrix problems, developing solutions to matrix interference problems, and presenting recommended solutions to the client for approval. If, however, the client already is aware of matrix interferences and solutions that have been demonstrated to remove these interferences, the client should provide this information to the laboratory during project solicitation or, at the latest, when the samples are scheduled.

For compliance monitoring purposes, permittees must ensure that any modifications they make to improve method performance are legally acceptable. Most NPDES permits require the use of methods approved at 40 CFR Part 136, while drinking water monitoring requires the use of methods approved at 40 CFR Part 141. Many methods provide the analyst with the flexibility to modify the method as the analyst demonstrates that the method modifications produce results that are as good or better than those produced by the original method. If the method required for monitoring does not include such flexibility, it is incumbent on you to seek written permission for any and all method modifications from your permitting authority, state, or EPA Region. In doing so, you should work with the analytical laboratory and the permitting authority, state, or EPA

Region to identify the requirements necessary for demonstrating that the method modifications are appropriate.

The costs associated with removing matrix interferences vary from laboratory to laboratory and problem to problem, as do the costs of the basic analysis. The costs involved in modifying a given method to overcome a complex matrix problem and in validating the use of additional cleanup techniques could range between several hundred and several thousand dollars, depending on the complexity of the wastewater, the experience of the laboratory in resolving matrix interferences, and the flexibility of the method. These costs associated with known matrix problems should be addressed with the laboratories' bids.

3.1.3 Project Background

Why often is overlooked because personnel assume that all parties involved understand the purpose of the analysis. Providing information on why the analyses are required, such as stating that wastewater samples are to be analyzed for NPDES permit compliance monitoring or that samples are being analyzed by various methods to characterize a waste stream, will help the laboratory provide the data you want.

3.1.4 Project Schedule and Data Turnaround Times

When specifies the following dates:

- The date by which bids should be received from laboratories interested in the project (this is discussed in greater detail in Section 3.5)
- The approximate date that the samples will be shipped to the laboratory, including the means of shipment (such as overnight delivery, hand-delivery, or laboratory pick-up)
- The date the analytical results are required by the client (data turnaround time) (this is discussed in greater detail in Section 3.4.2 and 3.4.3)

The data turnaround time specifies the number of calendar days after the laboratory receives the sample that the results are to be received by the client. Common data turnaround times are 30 to 45 days after the laboratory receives the last sample. The data turnaround time can be specified on a method-specific basis, and often is a function of a reporting deadline under a permit. Because commercial laboratories typically operate in production mode, where samples are processed through a standard sequence of login, preparation, digestion/extraction, analysis, data production, and quality assurance (QA), you can reduce analytical costs by providing the laboratory with as much time as possible to provide the data. In addition, some laboratories may be willing to negotiate a discounted fee if you promise to accelerate payment within a certain number of days. Caution should be exercised with this option, however, as it may prevent you from thoroughly reviewing the quality of the data prior to payment. Turnaround times extending beyond 45 to 60 days rarely provides any significant cost savings.

3.1.5 Methodology

How is perhaps the most important question to be addressed in the analytical services contract, and specifies the required methodology, the quality assurance/quality control (QA/QC), and the reporting format. The analytical requirements must be very specific, and include the following:

- **Method source and number.** Methods approved for wastewater analyses are listed in Tables IA through ID at 40 CFR Part 136. Methods approved for drinking water analyses are listed at 40 CFR Part 141. Common method sources for wastewater and drinking water analyses include EPA methods, Standard Methods, American Society for Testing and Materials (ASTM) methods, and U.S. Geological Survey (USGS) methods. Other relevant method sources include EPA's solid waste methods (SW-846) and Association of Official Analytical Chemists (AOAC) methods. Selection of appropriate methods is discussed in detail in Section 3.2.
- **Holding time.** The holding time of a sample is the maximum amount of time that can elapse between sample collection and analysis, between sample collection and extraction, or between sample extraction and analysis, for a sample result to be considered valid. The sample holding time for each method is different, and some methods may have more than one holding time. The holding times always should be specified. Table II at 40 CFR Part 136 specifies the "Required Containers, Preservation Techniques, and Holding Times" for routine parameters. Samples should be analyzed as soon as possible after collection. Samples for many analyses are not stable for long after collection, and daily shipment of samples to the laboratory should be considered. Delays in sampling and sample shipment may necessitate specifying a "contract" holding time in the contract, based on the analytical holding time minus any time required for sample shipment.
- **Quality assurance/quality control.** To ensure that data are valid and not a result of contamination or improperly calibrated instruments, laboratories must take rigorous QA/QC steps when performing the analyses. Specific guidance for defining QA/QC requirements is provided in Section 3.3. To ensure that the proper QA/QC steps are being routinely followed, all data must be reviewed and validated, and the laboratory should be audited at a frequency commensurate with the length, scope, and importance of the project.
- **Deliverables and reporting format.** The laboratory also needs to know how the data are to be reported, what information to provide in addition to the results, and how many copies of the data package are required. Deliverables are discussed in greater detail in Section 3.4.1.

3.2 Selecting Appropriate Methods

The methods approved by EPA for nationwide use under the Clean Water Act (CWA) and Safe Drinking Water Act will satisfy most of your analytical needs. However, some industrial wastewaters may cause matrix-specific analytical problems, while other matrices may contain analytes of interest for which there are no EPA-approved methods. Sections 3.2.1 - 3.2.3 present information on methods generally applicable to analysis of wastewater and drinking water matrices. Exceptions to the information presented below may arise in specific permit situations; therefore, all permittees must be familiar with the terms and requirements of their individual permits.

3.2.1 Methods Approved for Nationwide Use in Wastewater and Drinking Water

EPA publishes test procedures for measuring regulated contaminants in wastewater and drinking water at 40 CFR Part 136 and 40 CFR Part 141, respectively. The methods approved for use at

40 CFR Parts 136 and 141 include EPA methods, Standard Methods, ASTM methods, USGS methods, and Department of Energy methods. Other resources for analytical methods can be found on the EPA Office of Water's Environmental Monitoring Methods Index database (Reference 1) and Methods and Guidance for Analysis of Water CD-ROM (Reference 2). These resources allow the user to search for methods by analyte, and provide information on detection limits, instrumentation, and applicable matrices. Additional information on applicable regulations and method guidance also can be found on these resources.

Wastewater plants should be aware that the results of any final effluent analyses that are conducted with test procedures approved at 40 CFR Part 136 must be reported with the data submitted in the permit-required monitoring report, even if those analyses were not required in the permit. Results of analyses conducted with non-approved methods or conducted on unregulated waste streams are not required to be reported (40 CFR Part 122.41).

The 40 CFR Part 136 methods are applicable to a wide range of industrial effluents and typically were used to generate the data necessary for developing the effluent guidelines promulgated by EPA. Despite this wide applicability, EPA recognizes that these analytical methods may fail to yield useful results when used on certain sample matrices. EPA is prepared to consider claims that the effluent is compliant in those instances in which the effects of the sample matrix make measurements difficult or impossible. However, all such claims must be supported by specific analytical data that demonstrates reasonable, but unsuccessful, attempts have been made to overcome matrix interferences.

3.2.2 Wastewater Methods Approved for Specific Industrial Categories

In addition to methods approved at 40 CFR Part 136 for all wastewaters, EPA promulgates methods to measure pollutants for specific industrial categories at 40 CFR Parts 405 - 471, along with that industry's categorical effluent limitations and guidelines. Because EPA's authority to promulgate wastewater methods is specified at Section 304(h) of the Clean Water Act, methods promulgated at 40 CFR Part 136 or at 40 CFR Parts 405 - 471 often are referred to as the "304(h) methods."

3.2.3 Other Methods

If no approved methods are applicable to the analytes of interest or the matrix, selection of non-approved methods may be necessary (note, however, that unapproved methods cannot be used when a comparable approved method is available unless a permit or state or EPA Region explicitly allows the use of such alternate methods). In such instances, appropriate QA/QC procedures must be performed and low level detection limits must be achievable as necessary to demonstrate compliance with applicable permit limits or maximum contaminant levels (MCLs). As mentioned above, the permitting authority must approve the use of these methods in advance.

3.2.4 Method Modifications

Many of the methods approved at 40 CFR Parts 136 and 141 provide flexibility to modify the method to improve method performance, reduce cost, or adapt the method to address more difficult matrices without prior approval. Example improvements include the use of additional cleanup techniques, alternative gas chromatography or liquid chromatography columns, and more

specific detectors. However, the modifications to these methods cannot result in any degradation of method performance, and the laboratory that analyzes the samples with a modified method must first demonstrate that the modifications result in performance equivalent to that of the reference method. At present, only some methods approved at 40 CFR Parts 136 and 141 provide this flexibility. Unless this flexibility is provided explicitly in the method, modifications to a methods must be approved through the Alternate Test Procedure program.

In October 1995, EPA proposed the use of several new and modified methods for monitoring inorganic pollutants at 60 FR 53988. Most of the methods included in that proposed rule provide laboratories with the flexibility described above. In March 1997, EPA proposed to expand and extend this flexibility to nearly all methods approved for use at 40 CFR Part 136 and 40 CFR Part 141. Details of this proposal, known as the streamlining initiative, are given at 62 FR 14976.

If methods approved at 40 CFR Parts 136 or 141 fail to yield acceptable results in a specific matrix, you should consider modifying an approved method to yield improved and acceptable performance. Similarly, if methods approved at 40 CFR Part 136 of 141 are not applicable to a specific wastewater or drinking water pollutant that you need to monitor, approved methods applicable to similar pollutants may exist. In such cases, these entities may wish to consider modifying an approved method to target the pollutant of interest. In all cases, the modifications must either be allowed in the approved version of the method through the flexibility described above, or must be approved by the permitting authority or state or EPA Region. Permittees seeking approval of method modifications are encouraged to use the streamlining proposal at 62 FR 14976 as a basis for initiating discussions with their permitting authority.

3.3 Determining Appropriate Quality Control Requirements

The ability to define the validity of your sample results is wholly based on the degree of QC data associated with your results. Therefore, you are strongly encouraged to use the guidelines provided in this section, or similarly developed standard protocols, to establish strict data quality requirements for the analyses performed by contract laboratories (use of these requirements for in-house analyses also is advised). These QA/QC requirements subsequently enable the end-users of the data to standardize data inspection and acceptance procedures. This minimizes differences that might otherwise result between data reviewers and laboratories.

A standardized QA/QC approach should take the form of performance specifications for each method and should contain the following elements:

- Established laboratory quality system
- Purity and traceability of reference standards
- Calibration Range
- Linearity of calibration
- Calibration verification
- Method detection limit (MDL), minimum level (ML), or quantitation limit
- Initial precision and recovery (IPR)

- Ongoing precision and recovery (OPR)
- Analysis of blanks
- Recovery of matrix spikes and labeled compound spikes
- Statements of data quality for recovery of spiked analytes or labeled compounds in samples
- Analysis of field duplicates

These elements are an integral part of many recent EPA methods. However, earlier methods may not specify some or all of these elements. As such, you should ensure that any QA/QC elements not specified in the method(s) required under the contract are specified in the contract itself. A summary of each of the elements is provided below. Guidance on assessing data using these QA/QC results is provided in Section 6. The analytical laboratory should be audited to ensure that QA/QC procedures are being implemented on a daily basis. The frequency of these audits should be commensurate with the length, scope, and importance of the project. Audit information on a laboratory may be obtained by contacting the QA Officers or inspection staff in state and EPA regional offices with jurisdiction.

3.3.1 Established Laboratory Quality System

Any laboratory that performs analyses to support permitting compliance and monitoring should be required to have an established quality system that is compliant with *ISO/IEC Guide 25: General Requirements for the Competence of Calibration and Testing Laboratories* (Reference 3). This document sets forth general QA/QC guidelines for laboratories to follow, including personnel, analysis environment, and equipment requirements, requirements for internal reviews and audits, and the other requirements specified in Sections 3.3.2 - 3.3.11. It is essential for laboratories to employ comprehensive quality systems throughout the duration of the contract to ensure data validity. The laboratory should implement the quality system, and should otherwise use safe handling procedures and employ accepted Good Laboratory Practices in all aspects of laboratory performance.

3.3.2 Purity and Traceability of Reference Standards

The accuracy of any non-absolute empirical measurement depends on the reference for that measurement. In determining pollutants in water or other sample matrices, laboratories must calibrate analytical instruments and analytical processes with a known reference material. Most of the methods approved at 40 CFR Parts 136 and 141 require that the standards used for calibration and other purposes be of known purity and be traceable to a reliable reference source. The ultimate source for reference materials is typically EPA or the National Institute for Standards and Technology (NIST).

3.3.3 Calibration Range

Instrument calibration is required to establish the relationship of analyte concentration to instrument response, and is subsequently used for the quantitative analysis of field samples. This relationship is determined by analyzing a series of reference standards at different concentration

levels (calibration points) which encompass the expected concentration range of field samples and the expected linear range of the analytical instrument.

Most EPA methods for organic pollutants specify a minimum of three calibration points. Newer methods for inorganic pollutants also specify a minimum of three calibration points. The lowest of these points is required to be at or near the MDL. The highest is required to be near the upper linear range of the analytical system, and the third point is approximately midway between the two. The lowest calibration point should never be greater than five times the MDL and should ideally be within three times the MDL. The results for the lowest calibration standard are the principal means by which to assure that measurements at levels near the MDL are reliable. The EPA Office of Water uses the lowest calibration standard as one means of defining the ML of quantitation.

The flexibility in selecting the levels of the calibration points in many EPA methods has led to a wide variety of calibration ranges as each laboratory may determine its own calibration range. Some laboratories establish a relatively narrow calibration range, such as a five-fold increase in concentration, because it makes it simpler to meet the linearity specifications of the method. Other laboratories choose wider calibration ranges in order to minimize the number of samples that have to be diluted and reanalyzed because the concentration of one or more analytes exceeds the calibration range. Understanding these differences is particularly important if a narrow concentration range results in increased costs of sample dilution or if the laboratory's concentration range prevents the laboratories from achieving the required detection or quantitation limits.

3.3.4 Linearity of Calibration

The relationship between the response of an analytical instrument to the concentration or amount of an analyte introduced into the instrument is referred to as the "calibration curve." An analytical instrument can be said to be calibrated when this relationship has been established. The ratio of the response of the instrument to the concentration of the analyte introduced into the instrument is called the response factor (RF), relative response factor (RR), or calibration factor (CF):

- Relative response (RR) for isotope dilution calibration
- Response factor (RF) for internal standard calibration
- Calibration factor (CF) for external standard calibration

A plot of instrument response and concentrations is generated, and the linearity of response is measured by the shape of the calibration curve. While the shape of calibration curves can be modeled by quadratic equations or higher order mathematical functions, most analytical methods recommend establishing a linear calibration. The advantage of the linear calibration is that the RF or RR represents the slope of calibration curve, simplifying calculations and data interpretation. The 1600 Series Analysis Methods contain specific criteria for determining the linearity of calibration curves determined by either an internal or external standard technique. When the applicable criterion is met, the calibration curve is sufficiently linear to permit the laboratory to use an average RF or RR, and it is assumed that the calibration curve is a straight line that passes through the zero/zero calibration point. Linearity is determined by calculating the relative standard deviation (RSD) of the RF or RR for each analyte and comparing this RSD to the specified limit.

The number of calibration points is dependent on the error of the measuring technique. Measurement technique error is determined by (1) calibrating the instrument at the ML of quantitation and a minimum of two additional points, and (2) determining the RSD of the RR, RF, or CF. For most analyses, such as the determination of semi-volatile organic compounds by extraction, concentration, and gas chromatography, the measuring instrument is calibrated, and sample preparation processes are excluded from the calibration process; for others, such as the determination of purgeable organic compounds by purge-and-trap gas chromatography, calibration encompasses the entire analytical process. Table 3-1 below gives the number of calibration points required depending on the calibration linearity.

Table 3-1. Minimum Number of Points Required for Calibration

Percent RSD[1]	Minimum Number of Calibration Points
0 - <2	1[2]
2 - <10	3
10 - <25	5
>25	7

[1]Percent RSD shall be determined from the calibration linearity test for replicate measurements at a fixed concentration.
[2]Assumes linearity through the origin (0,0). For analytes for which there is no origin (such as pH), a two-point calibration shall be performed. In almost no cases should only one calibration point be used. One calibration point most often leads to serious error.

The maximum RSD specification is applicable to calibration with three or more calibration points. Alternatively, a minimum correlation coefficient for the linear relationship may be specified, below which the calibration linearity is not acceptable. If the calibration curve is non-linear, a second order ($y = ax^2 + bx + c$) calibration curve may be used. Calibration functions higher than the second order are not allowed.

3.3.5 Calibration Verification

Calibration verification involves the analysis of a single standard, typically in the middle of the calibration range, at the beginning (and in some cases, at the end) of each analytical shift. The concentration of each analyte in the reference standard is determined using the initial calibration curve, and the results are compared with method specifications. This test is used to periodically verify that instrument performance has not changed significantly. Specifications for calibration verification are developed to define the allowable deviation of the RR, RF, or CF of the calibration verification standard from the mean RR, RF, or CF of the initial calibration; or in cases where the initial calibration curve did not meet linearity specifications, deviation from a prior calibration verification standard or a single point of the calibration curve.

3.3.6 Method Detection Limit, Minimum Level, or Quantitation Limit

The Minimum Level (ML) is defined as the lowest level at which the entire analytical system gives a recognizable signal and, in most instances, an acceptable calibration point. Procedures for determining an MDL are provided at 40 CFR Part 136, Appendix B. Most of the 40 CFR Part 136, Appendix A and 40 CFR Part 141 Subpart C methods contain MDLs, although few of the methods explicitly require laboratories to demonstrate their ability to achieve these MDLs. Laboratories that wish to practice any method on a routine basis should be required to demonstrate that they can measure pollutants at the MDL or the detection limit specified in the

method. Performance of an MDL study in accordance with the 40 CFR Part 136, Appendix B, procedure is one means of demonstrating such proficiency.

3.3.7 Initial Precision and Recovery

The IPR test is used as an initial demonstration of a laboratory's capability to produce results at least as precise and accurate as those of other laboratories. The IPR test is also used to demonstrate that a method modification will produce results as precise and accurate as results produced by the approved (reference) method. The IPR test consists of four aliquots of reagent water spiked with the analytes of interest and with either surrogate compounds, or for isotope dilution analysis, with the labeled compounds. The spike concentration of the target analytes in the spike solution may vary between one and five times the lowest concentration used to establish the calibration curve (such as one to five times the ML). The spiked aliquots are carried through the entire analytical process. The mean concentration (x) and the standard deviation (s) are calculated for each analyte and compared to the specifications in the method. The IPR test is performed by the laboratory before it uses a method or a method modification for analysis of actual field samples.

3.3.8 Ongoing Precision and Recovery

The OPR test, sometimes termed a "laboratory control sample," "quality control check sample," or "laboratory-fortified blank," is used to ensure that the laboratory remains in control during the period that samples are analyzed, and separates laboratory performance from method performance on the sample matrix. The test consists of a single aliquot of reagent water spiked with the analyte(s) of interest, which is carried through the entire analytical process with each batch of samples. Typically, the concentration of the target analyte(s) in the OPR sample is between one and five times the lowest concentration used in the calibration curve (such as one to five times the ML). The results of the OPR are compared with method specifications.

3.3.9 Analysis of Blanks

Blanks are analyzed either periodically or with each sample batch, and are analyzed to demonstrate that no contamination is present that would affect the analysis of standards and samples for the analytes of interest. Different types of blanks are analyzed to more precisely determine if and when contamination was introduced. The following are different types of blanks that may be required by the methods selected for analysis:

- **Initial and continuing calibration blanks (ICB/CCB).** These blanks are required for all calibrated instrumentation. Deionized distilled water that contains the same reagents as the prepared samples is analyzed after analysis of the calibration standard to demonstrate the absence of carryover from the standard into the sample.
- **Preparation blanks**. Deionized distilled water is carried through preparation and analysis, using the same sample preparation, reagents, and analysis methods used for field samples. Preparation blanks are prepared and analyzed with each sample set to demonstrate that contamination is not introduced during any of the sample preparation or analysis steps.
- **Blanks**. Blanks are required for titrimetric and gravimetric methods, and any other method which does not require instrument calibration or sample preparation. Deionized distilled water which is not prepared, but contains the same reagents as the prepared field samples,

is analyzed to determine if the method analyte or other interferences are present in the laboratory environment, reagents, or apparatus.

- **Trip blanks**. Trip blanks are generated by the sampler for volatile compounds and low level metals, such as mercury. These blanks consist of vials of water that accompany each sample shipment to determine whether contamination has occurred from permeation of volatile organic compounds or low-level metals during sample transportation.
- **Equipment blanks**. These blanks are sampler generated to determine contamination from compositor sampling line or tubing.

The types of blanks required for analysis is dependent on the requirements of each method, and the period or batch size for which these blanks are required is also defined in each method. QC acceptance criteria are given in most methods. Generally, the source of contamination in a blank analysis must be identified and eliminated before the analysis of standards and samples may begin. Samples analyzed with an associated contaminated blank must be reanalyzed and, for contaminated preparation blanks, reprepared.

3.3.10 Matrix Spikes and Labeled Compound Spikes

The non-isotope dilution methods require that laboratories spike the analytes of interest into a second aliquot of a field sample and analyze this spiked sample with the non-spiked field sample. The purpose of spiking the sample (often termed a matrix spike) is to determine if the method is applicable to the sample matrix in question. Most EPA methods were developed for the analysis of wastewater effluent or treated drinking water samples, and may not be appropriate for in-process samples. While many wastewater methods were tested using effluents from a wide variety of industries, samples from some sources may not yield acceptable results. It is therefore important to evaluate method performance in the sample matrix of interest.

If the recovery of the matrix spike is within the limits specified in the method, then the method is judged to be applicable to that sample matrix. If, however, the recovery of the spike is not within the recovery range specified, either the method does not work on the sample, or the sample preparation process is out of control. If the method is not appropriate for the sample matrix, then changes to the method are required. Matrix spike results are necessary in evaluating the modified method. If the analytical process is out of control, the laboratory must take immediate corrective action before any more samples are analyzed.

To separate indications of method performance from those of laboratory performance, the laboratory should prepare and analyze a QC check standard consisting of a spike of the analytes in reagent water. If the results for the QC standard are not within the range specified, then the analytical system must be repaired and the sample and spiked sample analyses repeated. If the recovery of this spike is within the range specified, then the analytical process is judged to be in control.

3.3.11 Statements of Data Quality for Recovery of Spiked Analytes or Labeled Compounds in Samples

EPA methods specify that after the analyses of five spiked samples, a statement of data quality is constructed for each analyte. The statement of data quality for each analyte is computed as the

mean percent recovery plus and minus two times the standard deviation of percent recovery for each analyte. The statements of data quality should then be updated by the laboratory after each five to ten subsequent spiked sample analyses.

For non-isotope dilution results, the statement of data quality can be used to estimate the true value of a reported result and to construct confidence bounds around the result. For example, if the result reported for analysis of phenol is 25 μg/L, and the statement of data quality for phenol is 70% ± 15% (i.e., the mean recovery is 70% and the standard deviation of the recovery is 15%), the true value for phenol will be in the range of 28 – 43 μg/L, with 95% confidence. This range is derived as follows:

$$\text{Lower limit} = [(25 \div 0.7) - (25 \times 0.3)] = [35.7 - 7.5] = 28\ \mu\text{g/L}$$
$$\text{Upper limit} = [(25 \div 0.7) + (25 \times 0.3)] = [35.7 + 7.5] = 43\ \mu\text{g/L}$$

Statements of data quality for isotope dilution methods are based on the recoveries of the labeled compounds. Using an isotope dilution method, the sample result has already been corrected for the recovery of the labeled analog of the compound. Therefore, for a reported result for phenol of 25 μg/L where the standard deviation of the labeled phenol recovery is 15%, the true value for phenol will be in the range of 21.25–28.75 μg/L, with 95% confidence, derived as follows:

$$\text{Lower limit} = [25 - (25 \times 0.15)] = 21.25\ \mu\text{g/L}$$
$$\text{Upper limit} = [25 + (25 \times 0.15)] = 28.75\ \mu\text{g/L}$$

3.4 Writing the Contract

Before writing a contract for any analytical services, consult with appropriate legal staff. A well-written contract will include the *who, what, why, when, how* issues outlined in Section 3.1, above. It also will address your right to review the data as needed, the timeliness of payment to the laboratory, and your ultimate right to determine that the work does not meet the requirements established in the contract. A general format for an analytical services contract is provided in Appendix A. Please note that the information requested in Appendices A and B may not be adequate for competitive, written solicitations to multiple laboratories; depending on the project, more information may need to be requested in order to ensure the laboratory will be able to meet the requirements of the analytical contract.

The best way to ensure that you get the required data within the required time period is to specify these requirements *in detail* in the contract. Combined with a careful analysis of the requirements discussed in Sections 3.1 through 3.3, a well-written contract can minimize or eliminate many common problems in procuring analytical services. It should enable the client to obtain technically sound, legally defensible, and timely analytical data to meet a variety of compliance monitoring needs. Once generated, the basic form of the contract should be viewed as a dynamic document that is routinely updated to clarify ambiguities that arise during its implementation. (Note: Active contracts typically require a formal contract modification that is approved by both sides before its terms can be changed; expired or closed contracts can be modified before they are re-issued.)

General issues that should be specified in the contract are detailed in Sections 3.4.1 through 3.4.5.

3.4.1 Deliverables

You must ensure that the laboratory provides data that can be easily reviewed and that includes non-quantitative information related to the analyses, such as descriptions of any problems encountered. Laboratories should be required to have the following data from samples analyzed available for review:

- Summary reports of all analytical results in hardcopy and electronic data format. The summary report should contain a summary of analytical results for all QC and field samples. For the IPR analysis, the spiking level, individual results of the four replicates, and the mean recovery and relative standard deviation of the four replicates should be reported. For the OPR, standard reference material (SRM)/quality control sample (QCS), and calibration verification analyses, the true (or expected) concentration of the QC sample, the measured concentration, and the percent recovery should be reported. For MS/MSD analyses, the background concentration of the field sample, the spiking level, the individual results of the MS and MSD analyses, the percent recovery for the MS and MSD, the average concentration found in the MS/MSD samples, and the RPD between the MS and MSD should be reported. The results for all other QC, including calibration and blanks, also should be reported.
- A list of the sample numbers analyzed and a run chronology.
- Copies of all raw data, including quantitation reports, strip charts, spectra, bench sheets and laboratory notebooks showing tare and sample weights, sample volumes, and other data that will allow the final results reported to be traced back to the analytical steps performed. Each data element should be clearly identified in the laboratory's data package.
- A written report that details any problems associated with the analysis of the samples.
- A detailed written description of any approved modifications to the procedures specified in the contract-specified method.

With the possible exception of electronically formatted data, EPA recommends that you require all of the above deliverables as part of the data submission by your contract laboratory(ies).

3.4.2 Data Turnaround Times

The required data turnaround must be stated clearly in the contract. Unless you can guarantee to the laboratory that the samples will arrive when the laboratory opens in the morning, the data turnaround time calculations should consider the day that the sample is received at the laboratory "day zero," and the following day as "day one." In addition to stating the time that the laboratory has to generate and deliver the data, it may be useful to assign some specific consequences to the possibility of late delivery. One approach is to assess a penalty of some percentage of the analytical price per day of lateness. In the past, EPA has used values of 1% or 2% per day after the due date that the data were delivered. Obviously, lateness penalties should not be assessed if the delays were due to changes in the requirements made after the samples were sent, or to the fact that the methods requested were not applicable to the samples. Many of the remedies to matrix problems cannot be expected to be carried out in the original turnaround time assigned to the sample unless those remedies were explicitly detailed and required in the contract (see Section 3.1.2). However, after you have established that your samples can routinely be analyzed by the

requested methods, lateness becomes an issue of laboratory management practices, not sample matrix.

If it is anticipated that some samples will have to be analyzed in a faster than normal turnaround time during the performance of the analytical contract, a cost for these shorter turnaround time samples should be negotiated prior to award of a contract. The bids should be broken out into time periods that apply to the turnaround needs of the project (i.e., 2-day turnaround, 5-day turnaround, 10-day turnaround, etc.).

3.4.3 Liquidated Damages and Penalties

In many cases, you should consider including penalty or damage clauses in your contracts as incentives to preclude laboratories from defaulting on the contract, submitting data late, or performing analyses improperly. Due to the nature of the services provided, it is often difficult to assess actual damages caused by improperly performed analyses. Liquidated damages often are used in many contracts in lieu of actual damages. Liquidated damages typically specify that, if the laboratory fails to deliver the data specified in the deliverables section of the contract, or fails to perform the services within the specified data turnaround time, the laboratory will pay a fixed, agreed, price to compensate the organization to whom the services should have been delivered. For example, some EPA contracts specify that the laboratory will pay, as fixed, agreed, and liquidated damages, 2% of the analysis price per calendar day of delay, to a maximum reduction of 50% of the analysis price.

If liquidated damages or penalties are involved, they should (1) be based on actual damage caused (in terms of cost) by each day of lateness, (2) be strong enough to discourage late delivery, and (3) be reasonable enough that they will not discourage laboratories from bidding. If liquidated damages or penalties will be applied to meet the required data turnaround time, this information should be included. The contract should specify that the laboratory will not be charged with liquidated damages when the delay in delivery or performance arises out of causes beyond the control and without the fault or negligence of the laboratory. It also may be necessary to limit damages to a certain dollar value or scope.

Other types of damages that should be considered and may be included in the contract include costs for resampling, fines incurred as a result of improperly conducted analyses, and administrative costs associated with the evaluation and processing of unacceptable data.

3.4.4 Reanalysis Costs

Every laboratory periodically produces data that are of little use for the intended purpose. While well-run laboratories will contact the client as soon as they identify the problem and work with the client to make the best of the situation, you still may find itself with no useful data and a deadline approaching. The contract should stipulate that the laboratory will reanalyze samples at no cost to the client if the problems are due to laboratory error. It also should state that the client has the right to inspect the results, and if they do not meet the requirements in the contract, the client has the right to reject the data, returning them to the laboratory without payment. Rejection of data should be based on sound technical review of the results. It also obligates the client to make no use of those results without making some payment to the laboratory.

3.4.5 Dilutions

The contract should discuss the instances in which dilutions of samples and reanalyses would be considered billable by the purchaser. Again, a laboratory should be prepared to do the job right the first time and not bill for reanalyses required due to their errors. In contrast, some samples may need to be diluted and reanalyzed in order to bring the results within the demonstrated calibration range of the instrumentation. This typically occurs when the concentration of pollutants in the sample turns out to be higher than projected by the organization issuing the contract. Dilutions also may be necessary when several pollutants are to be measured by a single method, and the concentrations of some pollutants are within the calibration range of the instrument but the concentrations of other pollutants are not. When this occurs, the laboratory ought to be paid for their efforts to dilute the sample as necessary to quantify all pollutants. Such reanalyses can be figured into the original price, inflating the per-sample price for all samples to account for the need to reanalyze some samples, or it can be broken out as a separate cost. For analyses involving an extraction or digestion as well as an analysis, it may be useful to specify the price for the extraction step and the analysis separately, as it may be acceptable to simply dilute and reanalyze the sample extract instead of diluting, re-extracting, and reanalyzing the entire sample.

3.5 Developing a Bid Sheet

After all project requirements have been established, you should develop a bid sheet to accompany the analytical requirements summary during the solicitation. The bid sheet allows laboratories to submit bids in the same format, making bid evaluations easier, and also clarifies the project. Bid sheets for analytical services typically are formatted as a chart, with analytical requirements along one axis and number of samples and prices along the other. An example of a bid sheet is attached as Appendix B.

The bid sheet should include the following information:

- Project identifier
- Space for laboratory identification information
- Day, date, and time of the bid deadline
- Estimated award date
- Laboratory period of performance (period of time during which the laboratory is obliged to resolve issues associated with analysis of the samples—generally six months after shipment of last sample)
- Required delivery date (data turnaround time and the basis of its calculation, such as from receipt of each sample or from receipt of last sample)
- Bid validity period (period of time during which bid prices are considered valid—generally 45 days after the bid deadline; if the project is awarded after this period, you must contact bidding laboratories to determine if bids need to be revised)
- Parameters to be analyzed (typically the type of analysis and/or method)
- Number of field samples to be analyzed for each parameter

- Number and type of billable QC samples (such as MS or SRM)
- Total number of samples (field samples plus QC samples)
- Columns for laboratories to submit per-analysis and total costs

Depending on the requirements of the project, additional information, such as qualification information (discussed in detail in Section 4.3) may need to be requested with the bid sheet to ensure that the laboratory will be able to meet the requirements of the analytical contract.

3.6 Estimating Costs

Before soliciting an analytical project, the anticipated cost of the work should be identified to ensure that the solicitation and procurement procedures are appropriate. Analytical projects typically are costed-out using per-sample analysis prices. The most common methods for estimating per-sample costs are: (1) reviewing current, published laboratory fee schedules for the same or comparable analyses, and (2) reviewing historic per-sample costs for the same or comparable analyses. Laboratory fee schedules are available by request from most commercial laboratories. Previous invoice and payment records at your plant or organization can be used to research historical costs. If your utility or company frequently outsources analytical work, it may be helpful to copy the per-sample prices from these records into a separate file for future use in estimating project costs and establishing the reasonableness of laboratory bid prices.

4 SOLICITING AND AWARDING THE CONTRACT

Procedures for soliciting and awarding contracts to perform analytical services can vary, depending upon the scope of the project and purchasing requirements within the organization that is issuing the contract. At one end of the spectrum are contracts that are awarded after placing a single phone call and obtaining a quote from a single laboratory. The opposite end of the spectrum are contracts awarded after a competitive solicitation and bidding process involving the distribution of a detailed project description and a formal bid sheet via fax or mail. Determining whether an analytical services request will be solicited on a casual basis, through a rigidly documented formal solicitation, or somewhere in between, depends on the following factors:

- **The nature of the analyses.** Projects for routine analyses for which laboratories have published fee schedules are less problematic to solicit than projects for experimental or esoteric analyses. Phone solicitations to local laboratories or laboratories nationwide typically can be used for routine analyses to confirm laboratory prices. If the purchasing organization's procurement policies allow, an award can be made after per-sample prices are confirmed over the phone with a laboratory.
- **The anticipated cost and the procurement system of the organization purchasing the analytical services.** If the anticipated cost of the project is minor and you do not have a highly structured procurement system, the most straightforward means of soliciting the project is to call one or more local laboratories, receive and evaluate the quotes, and award the work. However, if the anticipated cost of the project is substantial and/or the procurement system requires a competitive solicitation, enough laboratories should be solicited to ensure that at least three bids are received (a minimum of three bids is required to qualify as a competitively awarded contract according to the *Federal Acquisition Regulation (FAR)*). The project then can be awarded to the lowest of the three responsive, responsible bidders (Section 4.4).
- **Your knowledge of capable laboratories.** If you frequently outsource projects to the same laboratory or laboratories, solicitations to these laboratories generally will not require the submission of prequalification data or references. Projects that are solicited to laboratories that are previously unknown to you may warrant additional steps, such as those described in Section 4.3, to ensure that the laboratory is capable of performing the requested analyses.

Because of the relatively straightforward nature of phone solicitations of routine projects, the remainder of this chapter provides general guidelines for conducting competitive, written solicitations to multiple laboratories nationwide. Before implementing these procedures, you should consult with their legal or procurement departments to ensure that the procedures are consistent with those required within their organization.

4.1 Identifying Capable Laboratories and Transmitting the Requirements

Capable laboratories generally are defined as laboratories that have the instrumentation and expertise to perform the analyses you require according to the methods you specify. Thus, although a frequently used local laboratory may be perfectly capable of performing your routine wet chemistry or metals work, that laboratory may not be considered capable when you require samples to be analyzed for dioxins. Several laboratory indices are available as resources to enable you to identify laboratories to target in a solicitation, including the ASTM *International Directory of Testing Labs,* the *American Council of Independent Laboratories' Directory,* and the DynCorp *Directory of Environmental Testing Laboratories.* Each of these directories is readily available (see References 4 - 6).

After laboratories capable of performing the requested analyses are identified, a written bid package needs to be transmitted to them. This bid package should include the analytical services request and the bid sheet, at a minimum. The package also should include the required methods, if non-routine analytical methods are required, and a cover letter if any additional or introductory information needs to be provided to the laboratories. If possible, allow at least two weeks for the laboratories to submit bids. This deadline should be noted on the bid sheet.

Traditionally, solicitation packages of 10 pages or less were transmitted by fax, and mail or overnight services were used if the package was more than 10 pages. However, most laboratories now have email addresses, and transmitting solicitations via email is typically more efficient than faxing or mailing the package.

4.2 Evaluating Bids

After the laboratories have received the solicitation and submitted their bids, you must evaluate the bids to identify the laboratory that will be awarded the analytical services contract. Specific procedures for evaluating bids may vary, depending upon the requirements of the organization that is soliciting the contract. Therefore, it is recommended that the procedures used to evaluate the bids be communicated to all laboratories as part of the solicitation package.

One way to confirm the requirements will be met is to require the laboratories to submit a technical proposal with their bids. An example technical proposal request and technical proposal scoring sheet is provided in Appendix C. Generally, the bid evaluation process will focus on an evaluation and comparison of each laboratory's proposed cost and an evaluation of each laboratory's capability to meet the analysis requirements.

You should consult your legal department or purchasing department to identify any applicable requirements for evaluating competitive bids within their organization. In the absence of explicitly defined bid evaluation procedures, you may wish to follow the procedures outlined below. These procedures, which have been adapted from those published in the *FAR*, begin with evaluation of all bids received to identify the lowest responsive, responsible bid. A bid is considered responsive if the following criteria are met:

- The bid was submitted without contingencies or with acceptable contingencies

- The bid was submitted before the bid deadline
- The bid sheet (if required) contains no errors or omissions

The organization responsible for awarding the contract also should recalculate bid prices based on each laboratory's per-sample price to ensure that the bidding laboratories did not make any mathematical errors. If any incorrect calculations are identified, the laboratory should be contacted to confirm the corrected total bid price. In addition, you should ensure that there are no unacceptable contingencies associated with any of the bids (such as the use of an unacceptable method). After all bids have been checked for errors and contingencies, you can identify the lowest, responsive bidding laboratory for the project. If there is a question regarding a laboratory's ability to perform the work, you should perform a responsibility determination, as well (see Section 4.3).

If three or more responsive bids were received, then the low bid may be deemed reasonable based on the closeness of the bid prices to each other and current market conditions. If fewer than three bids were received, price reasonableness can be determined using bid prices submitted for comparable projects, price quotes from current laboratory fee schedules, or information requested from the laboratory, including a breakdown of costs or invoices to other clients for comparable work. The lower bid may be deemed unreasonable if it is significantly lower than the other bids, and may not be considered for award because it can indicate a lack of understanding of the requirements.

4.3 Conducting Responsibility Determinations

If the low-bidding laboratory is previously unknown to you, the importance of the project merits special effort to ensure that the awarded laboratory is capable of reliably performing the requested analyses, or you are required to follow the *FAR*, then a laboratory responsibility determination should be performed. The best means of confirming that a laboratory is capable of reliably performing an analytical requirement is to assess data recently produced by the laboratory using the same method on similar sample matrices. A less expensive approach is to rely on other information applicable to the analyses in question, such as performance evaluation (PE) sample results, federal or state certifications, and corporate references. Sections 4.3.1 through 4.3.4 provide guidance for using the laboratory's method performance data, PE sample results, certifications, or references to evaluate their capability.

If laboratory performance cannot be assessed based on existing data or references, another alternative is to require laboratories that bid on the project to analyze samples specific to the project and submit these results with their bids. Bids then are evaluated in terms of cost and performance. Laboratories that do not submit acceptable data are not qualified to perform work under the project, and can be eliminated from consideration for award. Section 4.3.5 provides additional guidance concerning the use of prequalification analyses as a means of evaluating laboratory capability.

For long-term, critical, or very costly projects, you should consider auditing the laboratory before an award is made. Section 4.3.6 provides guidance on conducting audits.

4.3.1 Method Performance Data

Many laboratories routinely use EPA-approved methods for analysis of wastewater and drinking water samples collected by their clients (including the methods approved at 40 CFR Parts 136, 141, and 405 - 471). In such cases, you can ask a laboratory to provide historical data that demonstrates the laboratory is capable of reliably analyzing the required sample matrices with the required methodology. Data requested should include results from all QC parameters required by the method, including results from calibration standards, blanks, initial and ongoing precision and recovery samples, and spiked matrix samples. You should request historical data generated within the past six months. Older data still may be relevant, but the laboratory should indicate any personnel, instrument, or facility changes that have occurred since the data were generated.

4.3.2 Performance Evaluation Sample Results

Several EPA and state laboratory programs send performance evaluation (PE) samples to laboratories that are part of their program on a periodic or regular basis to monitor laboratory performance. PE samples typically consist of a synthetic matrix spiked with concentrations of analytes known to the program office but unknown to the laboratory (single-blind samples). The program laboratories analyze the samples and report the results, and the program office compares these results to the true values of the PE samples. The program office or laboratories that participate in programs that issue PE samples should be able to provide you with the assessment of their latest PE sample results.

Several PE studies programs are administered by EPA in support of the Clean Water Act, the Safe Drinking Water Act, and Superfund:

- **Water Pollution (WP).** Laboratories in the WP program receive chemistry PE samples; the program tests laboratories' abilities to analyze for common surface water quality parameters and pollutants. The WP program supports more than 25 state wastewater and other environmental laboratory certification programs.
- **Discharge Monitoring Report Quality Assurance (DMRQA).** Laboratories in the DMRQA program receive chemistry and whole effluent toxicity PE samples. This national program is used by EPA and the states to ensure the quality of monitoring data submitted by more than 7,000 major NPDES permittees each year.
- **Water Supply (WS).** The Water Supply program includes chemistry, microbiology, and radiochemistry PE studies and supports the Safe Drinking Water Act.
- **Effluent Guidelines Program.** Laboratories awarded contracts to analyze samples for EPA's Engineering Analysis Division within the Office of Water's Office of Science and Technology are sent periodic PE samples for organics, metals, and wet chemistry analyses to monitor performance.
- **Contract Laboratory Program (CLP).** Laboratories in EPA's Contract Laboratory Program, which supports Superfund sample analyses, receive PE samples for organics and inorganics analyses on a quarterly basis.

Other PE programs administered by private associations also may be helpful in meeting your needs. The European Union, for example, has developed the QUASIMEME program to provide a means for private organizations interested in participating in laboratory performance studies. The program offers a wide variety of PE samples and intercomparison studies on a routine basis.

Information on this program is available by email at marlab.ac.uk, by phone at 044 (0) 1224295 352, and by fax at 0044 (0) 1224 295511.

In general, a laboratory not participating in a PE sample program or equivalent should not be considered for the contract. However, it is important to understand that PE sample results are only useful if the analyses are applicable to the project for which the laboratory is considered. A laboratory's ability to perform well on organics PE samples is not an indication of how reliable its metals laboratory is.

4.3.3 Certifications

You also can ask laboratories to supply a list of their current certifications, such as state certifications. In addition, information about laboratory certifications can be obtained through Internet searches or by telephone or email from the NPDES or drinking water staff or QA officer in the state or EPA regional office with jurisdiction over the certified laboratory.

State certification programs vary widely—some states certify for specific programs, such as drinking water monitoring, while others certify for specific methods or analyses. Certifications are particularly useful if they apply directly to the analyses you required, but also provide an indication of the overall standing of the laboratory. Most certification programs entail laboratory audits and PE sample analyses, and thus provide some assurance that the laboratory is generally capable of providing reliable analytical services. However, you should note that a state drinking water certification is no guarantee that a laboratory is capable of performing industrial wastewater analyses by methods not covered by that certification. Conversely, the absence of a certification is no guarantee that the laboratory is not capable of performing the analyses—a certification for that method or parameter simply may not exist.

Currently, guidance and standards for a national laboratory accreditation program are being developed through a state/EPA organized group known as the National Environmental Laboratory Accreditation Program (NELAP). Current information on NELAP and the National Environmental Laboratory Accreditation Conference (NELAC) is available on the Internet.

4.3.4 References

This means of establishing a laboratory's reliability and capability is, perhaps, the easiest. If you have not worked with a particular laboratory before, you can ask the laboratory to provide contacts and phone numbers of corporate or government clients for which the laboratory has performed services comparable to the project at hand. Questions to ask the references include:

- Did the laboratory provide data by the required due date?
- Were the data reviewed upon receipt to ensure that the laboratory performed the requested analyses according to the specified methods and with the required QA/QC ? (If the answer to this question is no, the reference is not likely to be capable of providing sufficient information to adequately assess the laboratory's capability.)
- Does the laboratory have a documentation system for sample control that retains accurate records of chain-of-custody; sample holding, handling, preservation, and analyses; raw data; QA/QC, and processed data? Have you audited this system?

- Were laboratory personnel easy to work with when problems arose during all phases of the project, including sample scheduling, sample analysis, and data review? If problems were noted during data review, was the laboratory prompt and responsive in addressing your concerns?
- Do you have any reservations in recommending this laboratory?

4.3.5 Prequalification Analyses

As noted above, prequalification analyses may be required if laboratory performance cannot be assessed based on existing data, certifications, or references. Two options are available regarding payment of prequalification analyses. The first is to require laboratories to provide prequalification data at no cost with their bids. Laboratories can recoup this cost if they are awarded the contract. This approach generally will not work if the project is small, and the laboratory has little incentive to provide prequalification data at no cost. If, however, the project entails analysis of a sufficient number of samples to justify a loss leader from the laboratory, this approach should be considered.

The second option entails payment for prequalification analyses. In such a situation, laboratories would bid on the project in two parts: one portion of the bid would apply only to prequalification analyses, while the balance of the bid would apply only to analysis of the real samples. The bids would be evaluated based on overall cost, and the laboratories with the lowest cost would be awarded contracts to perform only the prequalification analyses. After prequalification data have been submitted and evaluated, the lowest bidding laboratory with acceptable prequalification data would be awarded the contract to analyze the real samples during the balance of the project.

Prequalification analyses can take several forms, including:

- **Analysis of single-blind samples**. The best way of determining laboratory performance before award is requiring bidding laboratories to analyze samples that are spiked with the target analyte(s) at concentrations unknown to the bidding laboratories. Such samples are essentially identical in concept to the PE samples described in 4.3.2. You can either prepare their own single blind samples or you can purchase these samples from commercial vendors.

  Vendors typically carry several types of stock PE samples applicable to a variety of pollutants, matrices, and analytical methods. To ensure that laboratories are unable to "predict" the pollutants and associated concentrations in their PE samples, vendors offer PE samples that contain a minimum number of pollutants from a selected list (such as at least 7 of 10 listed metals), each of which will be present within a specified "range" (such as 1 - 50 μg/L). Vendors routinely prepare and distribute new batches in order to further protect the integrity of their PE sample program. Actual pollutants and pollutant concentrations in each batch are certified, and these "certified values" are provided to the organization that purchases and distributes the PE sample(s). You should purchase the PE sample that most closely matches their target pollutant list and concentration range.
- **Analysis of samples spiked at the laboratory**. A simpler, and potentially more cost-effective approach to the single-blind sample analysis scenario is to require laboratories to

spike samples in-house and provide the spiking levels and recoveries for evaluation. If this approach is chosen, it is recommended that the laboratory be required to spike and analyze four replicate samples so that both precision and accuracy can be assessed. The matrix used can include reagent water, effluent provided by the wastewater plant or source or finished water from the drinking water plant, or a representative matrix that can be selected by the laboratory. If the laboratory is permitted to select a matrix type for this analysis (such as municipal wastewater or ambient water), the data reported should include characterization data, such as turbidity, hardness, background concentrations of the unspiked sample, etc.

- **Analysis of a standard reference matrix.** A third, similar approach is analysis of a commercially available SRM. The SRMs should be chosen by the client, and can be purchased by the laboratory or purchased by the client and sent to the laboratory for analysis.
- **Analysis of method blanks and method detection limit studies.** If the project entails detection of analytes at very low levels, the laboratory(ies) awarded the project should be required to demonstrate that laboratory contamination does not exceed acceptable levels and demonstrate that they are capable reaching the low end of the detection range. The latter is accomplished by performing a method detection limit study according to the procedure at 40 CFR Part 136 Appendix B (essentially, analysis of seven replicate reagent water samples spiked with the analyte of interest at one to five times the method's estimated detection limit). A method blank analyzed with these MDL samples can be used to demonstrate freedom from contamination at low levels.

4.3.6 Laboratory Audits

The goal of a prequalification audit is to ensure that the laboratory has the capability and commitment to meet the program goals of timely delivery and high-quality analytical services. Audits may be announced, or an alternate technique to determining that a laboratory is capable of reliably performing the contract is to make an unannounced visit to the laboratory. Audits can focus on any or all of the following areas:

- Laboratory personnel qualifications
- Sample receiving and storage areas
- Sample preparation and analysis areas
- Instrumentation
- Laboratory quality assurance plan (QAP)
- Laboratory standard operating procedures (SOPs)

Although laboratory audits generally are specific to the project, general criteria are applicable to each of the above areas. The best approach to evaluating a laboratory, based on these criteria, is through the use of checklists. Examples of laboratory audit checklists are provided in Appendix D. These checklists should be modified as necessary to adapt them to the specific project.

Contact the appropriate state or EPA regional office via phone or email to obtain audit or inspection information about the laboratory. The Internet can be used to identify the appropriate state or EPA regional contact.

You have two options if a laboratory fails an audit: (1) the laboratory can be eliminated from consideration for the project, or (2) the laboratory can be provided the opportunity to correct the deficiencies identified in the audit and request a reevaluation. If the laboratory passes the audit, you can proceed to contract award.

4.4 Awarding the Contract

Contract awards typically should be made over the phone, then followed by a written contract for laboratory signature. Awarding the contract over the phone enables you to verify the scope of the analytical work and verify laboratory information. This information should include the name of the person assigned to receive the samples and the street address to which the samples will be shipped—overnight delivery services, such as Federal Express, will not accept samples with post office box addresses. Laboratory information also should include the name and address of the laboratory's administrative personnel that handle billing issues, as these may differ from the address to which samples are shipped.

5 TRANSPORTING SAMPLES AND COMMUNICATING WITH THE LABORATORY

After the analytical services contract is awarded, samples are collected and shipped to the laboratory. Although it is the laboratory's responsibility to contact the client if problems occur after sample receipt, you still should initiate communications with the laboratory periodically to monitor progress.

5.1 Transporting and Tracking Samples

You must ensure sample integrity from collection through data reporting to use the data for anything other than internal purposes. This includes the ability to trace possession and handling of the sample from the time of collection through analysis. The following items and steps will ensure that samples are processed accurately and that the data produced are defensible: sample labels, sample seals, field log books, chain-of-custody records, sample analysis request sheets, tracking of sample delivery to laboratory, receipt and logging of samples by the laboratory, and documentation of the sampling project from sample collection through sample analysis. This process of tracking samples is considered a "sample control system," and should be established as a documentation system for the laboratory.

- **Sample labels.** Sample labels always should be used to prevent sample misidentification. The sample number and required analyses should be stated clearly on the label. If space allows, the name of sampler, date and time of collection, and place of collection also should be included. Waterproof markers should be used to write on sample labels.
- **Sample seals.** When chain-of-custody is critical, sample seals can be used to detect any unauthorized tampering with samples up to the time of analysis. The seal should be attached in such a way that it is necessary to break it to open the sample container.
- **Field log book.** A field log book should be used to record all information pertinent to sample collection. The field log book should include the following: the purpose of sampling, the location of the sampling point, the name and address of the field contact, the producer of the material being sampled and address (if different from sampling location), the type of sample being collected (such as wastewater, drinking water, or biosolids), and, if the sample is a wastewater, the identification of the process producing the waste stream. In addition, the number of samples and volume of sample taken, the description of the sampling point and sampling method, the date and time of collection, and the sampling label number should be included. Other items that are useful to keep with the field log book are references such as maps or photographs of the sampling site, field observations and measurements, and signatures of personnel responsible for observations. Sampling situations vary, so no general rule can be given as to the information to be entered in the log book, but as much information as possible should be provided.
- **Chain-of-custody record.** The ability to trace possession and handling of a sample from the time of collection through analysis is referred to as chain-of-custody. A sample is

considered to be in an individual's custody if any of the following criteria are met: (1) the sample is in your possession or it is in your view after being in your possession, (2) it was in your possession and then locked up or sealed to prevent tampering, or (3) it is in a secured area. The chain-of-custody record is used as physical evidence of sample custody. The sampler completes a chain-of-custody record to accompany each sample or group of samples shipped from the field to the laboratory. The record includes the following: sample number, signature of sampler, date, time, and location of collection, sample type, signatures of persons involved in the chain of possession and inclusive dates of possession. The original signature copy of the chain-of-custody record is enclosed in plastic and secured to the inside of the container used for sample shipment. A copy of the custody record is retained for the sampler's file. The shipping containers are secured and custody seals are placed across the cooler openings. The laboratory representative who accepts the incoming sample shipment signs and dates the chain-of-custody record to acknowledge receipt of the samples. Custody procedures continue to be followed throughout the laboratory (from sample custodian to extracting specialist to analyst) until the sample is consumed or disposed of.

- **Sample analysis request sheet.** A sample analysis request sheet or traffic report should accompany the samples to the laboratory. The sampler should complete the field portion of this sheet with most of the pertinent information noted in the log book. The laboratory representative should complete the laboratory portion of this form, which includes: the name of the person receiving the sample, laboratory sample number, date of sample receipt, condition of samples upon receipt, and analyses to be performed.

- **Sample delivery to laboratory.** The samples should be delivered to the laboratory as soon as practicable. Commercial carriers often are the best method of shipment if the samples cannot be delivered to the laboratory the same day as collection. It also is advisable to use a carrier with the ability to track and provide the status of individual shipments. To facilitate return of the shipping containers, shippers should clearly mark the name and address of the return destination on the containers. The laboratory must be contacted every day they are to receive samples to confirm receipt of samples. Both you and the laboratory should document this confirmation.

- **Receipt and log-in of samples.** At the laboratory, the sample custodian receives the samples and should perform the following tasks with each sample: (1) inspect the condition of the sample, (2) inspect the condition of the sample seal (if present), (3) reconcile sample label information and seal against the chain-of-custody record, (4) assign a laboratory sample number, (5) log the sample in the laboratory log book, and (6) store the sample in appropriate storage conditions.

- **Documentation of sampling project from sample collection through sample analysis.** Documentation of the entire sampling project from sample collection through sample analysis, including any problems and resolutions that occur during the event, should be maintained.

- **Sample holding times.** Sample analysis results may not be valid if the prescribed holding times and other requirements for each parameter are not met. These requirements are listed in Table II of 40 CFR Part 136 for wastewater contaminants, as well as in Subpart C of 40 CFR Part 141 for many drinking water contaminants.

Samples should be packaged for shipment in compliance with the most current U.S. Department of Transportation, state, local, and commercial carrier regulations. All required government and commercial carrier shipping papers must be completed and shipment classifications made according to these regulations.

Waterproof, metal or hard plastic ice chests or coolers should be used for shipment. Inside the cooler, sample containers should be enclosed in clear plastic bags so that sample tags and labels are visible. Water and soil samples suspected to contain dioxin or highly toxic or reactive pollutants at high concentrations must be enclosed in a metal can with a clipped or sealed lid (paint cans typically are used). The outer metal can must be labeled with the number of the sample contained inside. Containers that do not fit into paint cans should be double bagged.

Ideally, shipping containers should be packed with noncombustible, absorbent packing material, such as vermiculite. The material should surround the sample bottles or metals cans containing sample to prevent breakage during transport. Earth or loose ice should never be used to pack samples; earth is a contaminant, and ice melts, resulting in container breakage.

The sampling and shipping conditions for each sample will depend on the analysis required for that sample, and will be specified in the method. When shipping with ice, the ice should be in sealed plastic bags to prevent melting ice from soaking packing material which, when soaked, makes handling of samples difficult in the laboratory. The Sample Analysis Request Sheet, chain-of-custody record and any other sample documentation accompanying the shipment must be enclosed in a waterproof plastic bag and taped to the underside of the cooler lid. Coolers should be sealed with custody seals in such a manner that the custody seal would be broken if the cooler were open. Shipping coolers must have clearly visible return address labels on the outside.

Samples should be shipped through a reliable commercial carrier, such as Federal Express, Emery, and Airborne Express, or equivalent if the samples cannot be delivered to the laboratory by the sampler on the day or day after the sampling occurs. The sampler should record the shipment tracking number (such as the airbill number) to resolve any problems associated with the sample if it is waylaid. Consideration also should be given to requesting the laboratory to pick up the samples.

5.2 Communicating with the Laboratory

You must maintain communications with the laboratory to confirm sample shipment receipt, timely analysis, and quality data. In addition, it is important that the laboratory is able to communicate immediately with the sampler or person responsible for the sampling event in case of sample shipment problems or analysis issues that may affect data quality.

Although phone communications currently are the norm, these communications ideally should be conducted via email. Email communications not only should provide virtually immediate responses, but also enables both the contracting party and the laboratory to maintain a written record of sample receipt confirmations, problem notifications, and problem resolutions. In addition, email communications reduce misunderstandings and miscommunications.

6 REVIEWING ANALYTICAL DATA

When reviewing data submitted by contract laboratories, you must ensure the test data include the QA/QC elements listed in the analytical method and in your contract (see Section 3.3);otherwise, the data can be considered noncompliant. As a result,. These supporting QA/QC results provide you with the simplest means of assessing the quality of your data.

In many of its early analytical programs, EPA relied upon laboratories to maintain records of the QA/QC data. This practice was cumbersome for the laboratories, because many of the QA/QC data were common to the analytical results for a variety of clients. Retrieving these data from the laboratory to resolve questions of permit compliance was time-consuming for the permittee and the permit writer. More importantly, this practice occasionally resulted in unscrupulous laboratories failing to perform the necessary QA/QC testing, or performing the QA/QC testing "after the fact" to satisfy an audit or data submission request. In particular, many laboratories did not perform the IPR test prior to practice of the method and did not perform a spike of the analytes into the sample matrix to prove that the method would work on a particular sample. Therefore, while the data provided by those laboratories may have been valid, there was no way to prove their validity.

Sections 6.1 through 6.11, below, provide guidance on evaluating sample data based on QA/QC data. A data inspection checklist is provided in Appendix E, providing a standardized format for the data review process and the documentation of findings.

6.1 Purity and Traceability of Reference Standards

Laboratories submitting analytical data must be able to trace the reference standards used in the analysis to EPA or NIST. The proof of this traceability is a written certification from the supplier of the standard. Documentation of the purity and traceability of the standards need not be provided with every sample analysis. Rather, it should be maintained on file at the laboratory and provided on request. When analyses are conducted in a contract laboratory, such documentation ought to be provided to the permittee the first time that a laboratory is employed for specific analyses and then updated as needed.

6.2 Calibration Range

The data reviewer must make certain that the calibration range encompasses the minimum level and that all measurements are within the calibration range of the instrument. Samples with analytes outside of the calibration range should be diluted and reanalyzed. The diluted sample results need only apply to those analytes that exceeded the calibration range in the initial analysis. In other words, it is acceptable to use data for different analytes from different levels of dilution within the same sample.

If data from an analysis of the diluted sample are not provided, limited use can be made of the data that are above the calibration range. The response of the analytical instrument to concentrations of analytes will eventually level off at concentrations above the calibration range. While it is not possible to specify at what concentration this will occur from the calibration data provided, it is generally safe to assume that the reported concentration above the calibrated range is a lower limit of the actual concentration. Therefore, if concentration above the calibration range is also above a regulatory limit, it is highly likely that the actual concentration would also be above that limit.

6.3 Linearity of Calibration

Linearity specifications vary from method to method, depending on the quantitation technique. Typical limits on the RSD are as follows:

- 15% for GC and HPLC methods
- 35% for analytes determined by the internal standard technique in GC/MS methods
- 20% for analytes determined by isotope dilution in GC/MS methods

If the calibration is not linear, as determined by the RSD of the response factor or calibration factor, the calibration curve, as opposed to the average response factor, must be used for quantitation. This means that a regression line or other mathematical function must be employed to relate the instrument response to the concentration. Properly maintained and operated lab instrumentation should have no difficulty in meeting linearity specifications for the EPA-approved methods.

Whatever calibration range is used, the laboratory must provide the RSD results by which one can judge linearity, even in instances where the laboratory is using a calibration curve. In instances where the laboratory employs a curve rather than an average response factor, the data reviewer should review each calibration point to assure that the response increases as the concentration increases. If it does not, the instrument is not operating properly, or the calibration curve is out of the range of that instrument, and data are not considered valid.. The analysis of samples should not proceed until linearity on that instrumentation is demonstrated.

6.4 Calibration Verification

Calibration verification results should be within method specifications. If any individual value falls outside the range given, system performance is considered unacceptable, and the laboratory may either recalibrate the instrument or prepare a new calibration standard and make a second attempt to verify calibration. If the laboratory was not able to verify calibration, the data should be evaluated to determine if it is usable with a qualification of high or low bias, or if the bias precludes use of the data.

6.5 Method Detection Limit, Minimum Level, or Quantitation Limit

Unless specific data gathering requirements require otherwise, the laboratory should report the concentration of all sample results that are at or above the ML. It should be noted that this ML is a sample-specific ML and, therefore, reflects any sample dilutions that were performed. If sample

results are reported below the ML, the data reviewer should require the responsible party to correct and resubmit the data, or if this course of action is not possible, the reviewer should determine the sample-specific ML and consider results below that level to be non-detects for regulatory purposes.

If sample results are reported above the ML, but are below a compliance level, then the data reviewer should consider the results to suggest that the pollutant has been detected but is compliant with the facility's permit (assuming that all QC criteria are met). If sample results are reported above a compliance level, the data reviewer must evaluate laboratory QC samples in order to verify that the level of pollutant is not attributable to analytical bias. In addition, the data reviewer must evaluate all blank sample results in order to determine if the level of pollutant detected may be attributable to contamination.

Although sample results are to be reported only if they exceed the ML, all blank results are to be reported, regardless of the level. This reporting requirement allows data reviewers the opportunity to assess the impact of any blank contamination on sample results that are reported above the ML.

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6.6 Initial Precision and Recovery

If the IPR data fail to meet the specifications in the method, none of the data produced by the laboratory can be considered to be valid. If the laboratory did not perform the start-up tests, the data cannot be valid, unless all other QC criteria have been met *and* the laboratory has submitted IPR (and associated instrument QC) data that were generated after-the-fact by the same analyst on the same instrument. If these conditions are met, then the data reviewer may consider the data to be acceptable for most purposes. NOTE: The inclusion of this alternative should not in any way be construed to sanction the practice of performing IPR analyses after the analysis of field samples. Rather, EPA believes that demonstration of laboratory capability prior to sample analysis is an essential QC component; this alternative is provided only as a tool to permitting authorities when data have already been collected without the required IPR samples. Once the problem has been identified, all responsible parties are expected to implement corrective action necessary to ensure that it is not repeated.

It is important to remember that if a change is made to a method, the IPR procedure must be repeated using the modified procedure. If the start-up test is not repeated when these steps are modified or added, any data produced by the modified methods cannot be considered to be valid. Such changes may involve alternative extraction, concentration, or cleanup processes; alternative GC columns, GC conditions, or detectors; or other steps designed to address a particular matrix problem..

6.7 Ongoing Precision and Recovery

The data reviewer must verify that the OPR sample has been run with each sample batch and that the applicable recovery criteria in the analytical method have been met. If the recovery criteria have not been met, the reviewer may use the following guidelines when making use of the data:

- If the concentration of the OPR is above method specifications but that analyte is not detected in an associated sample, then it unlikely that the sample result is affected by the failure in the OPR.
- If the concentration of the OPR is above method specifications and that analyte is detected in the sample, then the numerical sample result may represent an upper limit of the true concentration, and data users should be cautioned when using the data for enforcement purposes.
- If the concentration of the OPR is below method specification but that analyte is detected in an associated sample, then the sample result may represent the lower limit of the true concentration for that analyte.
- If the concentration of the OPR is below method specification and that analyte is not detected in an associated sample, then the sample data are suspect and cannot be considered valid for regulatory compliance purposes.

If the OPR standard has not been run, there is no way to verify that the laboratory processes were in control. In such cases, a data reviewer may be able to utilize the field sample data by examining the matrix spike recovery results (see item 9), the IPR results, OPR results from previous and subsequent batches, and any available historical data from both the laboratory and the sample site. If the matrix spike results associated with the sample batch do not meet the performance criteria in the methods, then the results for that set of samples cannot be considered valid. If the laboratory's IPR results and the matrix spike results associated with the sample batch in question meet the all applicable performance criteria in the methods, then the data reviewer may be reasonably confident that laboratory performance was in control during field sample analysis. This level of confidence may be further increased if there is a strong history of both laboratory performance with the method and method performance with the sample matrix in question, as indicated by additional OPR and matrix spike data collected from the laboratory and samples from the same site.

6.8 Analysis of Blanks

Unless the samples are still within analytical holding time and reanalysis is possible, there is no corrective action if unacceptable blank data are submitted with sample data. Therefore, the reviewer has several options in making use of the sample data. First, if a contaminant is present in a blank, but not present in a sample, then there is little need for concern about the sample result, though it may be useful to occasionally review the raw data for samples without the contaminant to ensure that the laboratory did not edit the results for this compound.

The second approach deals with instances where the blank contaminant is also reported in a sample. Some general guidance will help you determine the degree to which the contaminant is affecting sample results:

- If the sample contains the contaminant at levels of at least 10 times that in the blank, then the likely contribution to the sample from the contaminant in the laboratory environment is at most 10%. Since most of the methods in question are no more accurate than that level, the possible contamination is negligible.

- If the sample contains the contaminant at levels of at least 5 times but less than 10 times the blank result, the compound is probably present in the sample, but the numerical result should be considered an upper limit of the true concentration.
- If the sample contains the contaminant at levels below 5 times the level in the blank, there is no adequate means by which to judge whether or not the sample result is attributable to laboratory contamination. The results for that compound in that sample are then suspect.

There are two difficulties in evaluating sample results relative to blank contamination. First, the reviewer must be able to associate the samples with the correct blanks. The second difficulty involves samples that have been diluted. The dilution of the sample with reagent water or the dilution of the extract with solvent represents an additional potential source of contamination that will not be reflected in the results for the blank unless the blank was similarly diluted. Therefore, in applying the 10-times rule, the concentration of the sample is compared to the blank result multiplied by the dilution factor of the sample or sample extract. For instance, if 12 ppb of a contaminant are found in the blank, and the associated sample extract was diluted by a factor of 6 relative to the extract from the blank prior to analysis, then the sample result would have to be greater than 12×6×10, or 720 ppb, to be acceptable. Between 360 ppb and 720 ppb, the sample result would best be considered an upper limit of the actual concentration. Below 360 ppb, the sample result is not acceptable for compliance monitoring.

In most cases, the practice of subtracting the concentration reported in the blank from the concentration in the sample is not recommended as a tool to evaluate sample results associated with blank data. One of the most common problems with this approach is that blank concentrations are sometimes higher than one or more associated sample results, yielding negative results.

6.9 Matrix Spikes and Labeled Compound Spikes

When evaluating matrix spike results, the data reviewer must verify the following:

- An appropriate spike concentration was used
- The unspiked sample has been analyzed
- The spiked sample has been analyzed
- The recovery of the spike is within the range specified
- If the spike recovery is not within the range specified, a QC check standard has been analyzed
- If a QC check standard has been analyzed, the results are within the range specified

For isotope dilution analyses, the evaluation of the data is simpler because isotopically labeled analogs of the pollutants are spiked into each sample allowing recovery to be evaluated for every analyte in every sample, and because a QC check standard (termed the "ongoing precision and recovery standard," or OPR) is analyzed with each sample set.

If the recovery of a labeled compound spiked into the sample is not within the range specified in the method, and the results of analysis of the ongoing precision and recovery standard are within

the respective limits, the sample results are considered reportable, with qualification that the results may be biased. When labeled compound recoveries are outside of the method specifications, the problem may be related to the sample matrix. In these instances, the sample may be diluted with reagent water and reanalyzed. If the labeled compound recoveries meet the method specifications after dilution of the sample, then the results are acceptable, although the sensitivity of the analysis will be decreased by the dilution.

In instances where matrix spike or labeled compound recoveries are not within the specifications, it may still be possible to use the sample results for compliance monitoring purposes. In particular, if (1) the recovery of the spiked compound is above the method specifications and (2) the compound is not detected in the sample analysis, it is unlikely that the compound is present in the sample. This is because the factors that caused the analysis to over-estimate the concentration in the spiked sample would not likely have resulted in an under-estimate in the unspiked sample. For samples in which the compound is detected but the matrix spike or labeled compound recovery is above the method specifications, the concentration reported in the unspiked sample is likely an upper limit of the true concentration.

Unfortunately, for some sample matrices, even dilution will not resolve the problem, and for other matrices, the loss of sensitivity will preclude the use of the results for determining compliance. In these instances, additional steps need to be taken to achieve acceptable results.

6.10 Statements of Data Quality for Recovery of Spiked Analytes or Labeled Compounds in Samples

Many laboratories do not provide the data quality statements with the sample results, in which case the data reviewer must determine if the data quality statements are being maintained for each analyte and may need to obtain the data. If necessary, the reviewer can construct the data quality statement from the individual data points.

The lack of a statement of data quality does not invalidate results but makes some compliance decisions more difficult. If statements of data quality are not being maintained by the laboratory, there may be increased concern about both specific sample results and the laboratory's overall quality assurance program.

6.11 Field Duplicates

The field duplicate provides an indication of the overall precision associated with entire data gathering effort, including sample collection, preservation, transportation, storage, and analysis procedures. The data reviewer should examine field duplicate results and use the following equation to calculate the relative percent difference between the duplicate and its associated samples.

$$RPD = 200\frac{(|D1-D2|)}{(D1+D2)}$$

where:

D1 = concentration of the analyte in the field sample
D2 = concentration of the analyte in the duplicate field sample

If the analyte of interest was not detected in either replicate of the field sample, then the RPD will be zero. If the analyte was detected in each field sample replicate, but the results are highly disparate (indicated by a large RSD), the reviewer should apply the following guidelines when making use of the data:

- If the analyte was detected in each replicate and at similarly variable concentrations in the blank samples, then the field sample variability may be attributable to variable contamination, and the data may not be valid for regulatory compliance purposes.
- If the analyte was detected in each replicate at a concentration well above the regulatory compliance level, but was not detected in the associated blank samples, then it is likely that the sample results are not adversely affected.

Ideally, the RPD between field duplicates and MS/MSD samples will be close to zero. Any difference between the two duplicates is attributable to variability associated with the field sampling process.

7 REFERENCES

1 U.S. EPA Office of Water. *Environmental Monitoring Methods Index.* Version 2.0. National Technical Information Service (PB97-502637BDR), 1995

2 U.S. EPA Office of Water. *Methods and Guidance for Analysis of Water.* EPA 821-C-978-001. April 1997.

3 International Organization for Standardization (ISO) and International Electrotechnical Commission (IEC). 1990. *Guide 25: General Requirements for the Competence of Calibration and Testing Laboratories.*

4 American Society for Testing and Materials. 1998. ASTM *International Directory of Testing Laboratories.*

5 American Council of Independent Laboratories, Inc. 1992-1993. *Directory: A Guide to Leading Independent Testing, Research, and Inspection Firms in America.*

6 DynCorp. 1996. *Directory of Environmental Testing Laboratories.*

7 Code of Federal Regulations Title 40 Part 136—Guidelines Establishing Test Procedures for the Analysis of Pollutants

8 Code of Federal Regulations Title 40 Part 141—National Primary Drinking Water Regulations, Subpart C—Monitoring and Analytical Requirements

9 U.S. EPA. September 1994. *NPDES Compliance Inspection Manual,* EPA 300-B-94-014,
Chapter 4 (laboratory procedures).

APPENDIX A

ANALYTICAL SERVICES REQUEST FORM EXAMPLE

Analytical Services Request

Client Name:

Point of Contact (name, telephone and fax number, and email address):

Date of Request:

1. General description of analytical services requested:
2. Definition *and* number of samples involved (specify wastewater, groundwater, sludge, soil, etc.):
3. Purpose of analysis (NPDES, SDWA, RCRA compliance monitoring, etc.):
4. Estimated date(s) of sample collection:
5. Estimated date(s) and method of shipment:
6. Sampling/shipping contact (name and telephone number):
7. Holding times associated with analysis (specify number of days, or state "per method"):
8. Number of days after sample receipt that data are required:
9. Analytical method required (specify method number, source, and date, and attach copy where practical):
10. Special technical instructions (provide information on known problems, possible solutions, matrix effects, etc.):
11. Data reporting requirements (specify format of data, QA/QC reports, number of copies, etc.):
12. Sensitivity required (specify "per requested method," or list analyte names, CAS numbers, and quantitation limits required):
13. Quality control requirements (summarize QC operations specified in the referenced method, and any additional requirements):
14. Action required if QC limits exceeded (specify reanalysis, contacting client immediately, etc.):
15. Other (use additional sheets or attach supplementary information, as needed):

APPENDIX B

BID SHEET EXAMPLE

Bid Response for Analysis of Effluent and Marine Water Samples for Trace Metals

Laboratory name:	
Laboratory contact:	
Phone Number:	

Bid Deadline (Day, Date, Time) Friday, August 29, 1997, 5:00 pm EST	Liquidated damages will be assessed at a rate of 2% of the per-sample cost for each day that data is late
Estimated Award Date: September 17, 1997	Period of performance: From the date your bid price is accepted until June 13, 1998
Data deliverables due within 35 calendar days from receipt of last sample at lab	Bid prices listed below shall be valid for a period of 90 calendar days from the bid deadline date.

Parameter		Analyses		(A)	(B)	(A x B)
Method	**Matrix**	**Field Samples**	**MS**	**Total Analyses**	**Cost per Analysis**	**Total Cost**
1631	POTW effluent	14	1	15		
1631	Marine Water	4	1	5		
1632	POTW effluent	14	1	15		
1632	Marine Water	4	1	5		
1636	POTW effluent	12	1	13		
1637	POTW effluent	14	1	15		
1637	Marine Water	4	1	5		
1638	POTW effluent	14	1	15		
1639	POTW effluent	28	1	29		
1640	POTW effluent	14	1	15		
1640	Marine Water	4	1	5		
					Total Project Cost	

Note to bidding laboratories:

Bid sheets must be accompanied by the following prequalification criteria:
1. Method blank analysis results for each method.
2. Method detection limit study for each method and each matrix set (POTW and marine water).
   water) on which you submit a bid.

EPA will evaluate these data to identify laboratories qualified to participate in the study.

APPENDIX C

GENERAL LABORATORY AUDIT CHECKLIST EXAMPLE

General Laboratory Audit Checklist

Laboratory: _____

Audit dates: _____

Audit team: _____

_____

_____

Section 1: Quality Assurance Management Systems

1. Is there a quality assurance program plan (or equivalent) for the contract under which this work is being performed?

2. Are the staff familiar with the plan?

Strengths and weaknesses:

Section 2: Project Management Systems

1. Does a QAPP (or equivalent) exist for the project or work assignment?
2. Are the staff familiar with the plan?
3. Are all the specific elements in the QAPP included in the laboratory's QA plan?
4. Has the QAPP (or equivalent) been approved?
5. Have the requirements set forth in the QA plan been met?
6. Are deliverables on time?
7. Is sufficient coordination occurring between the laboratory and client project managers?
8. Are project files available?
9. Are software packages used by the laboratory for data reduction adequately described in project files?

Strengths and weaknesses:

Section 3: Laboratory Management Systems

1. Has sufficient laboratory space been allocated?

2. Have contamination-free areas been provided for trace-level work?

3. Are reagent-grade or higher purity chemicals used to prepare standards?

4. Is the following information documented for all reagents/standards used?

   a. Manufacturer

   b. Date of receipt

   c. Date opened

   d. Purity

   e. Lot number

5. Are notebooks being kept in accordance with good laboratory practice? Are laboratory notebooks controlled?

6. Have standard operating procedures (SOPs) been written where appropriate?

7. Do staff have copies of current SOPs? Are SOPs controlled documents?

8. Are staff performing operations according to SOPs?

9. Does documentation exist for standards preparation that uniquely identifies the reagents/solvents used and the method of preparation?

10. Does documentation exist for identification of standard preparer and date of standard preparation?

11. Are calibration standards validated prior to use?

12. Are standards replaced at the proper intervals?

13. Are samples subject to a chain-of-custody system?

    a. Are they uniquely identified?

    b. Is their storage documented and inventoried?

14. Are manufacturers' maintenance manuals available?

15. Are maintenance logs kept for lab equipment/instrumentation?

16. Is service on equipment instrumentation readily available?

17. Are replacement parts for equipment/instrumentation available?

18. Is the analytical balance located in an area free of drafts and rapid temperature changes?

19. Do balances have calibration stickers showing date of last certified calibration and date of next scheduled calibration?

20. Are records available for in-house calibration/checking balances?

21. Do micropipettes have logs indicating calibration checks performed in-house?

22. Do records exist for monitoring of laboratory water systems?

23. Is everyone aware of disposal plan? Is it adhered to?

24. Are glassware cleaning procedures adequate?

25. Are temperature logs available for freezers?

26. Are certified material standards used for all parameters such as material is available for?

Strengths and weaknesses:

Section 4: Data Management Systems

1. Are entries to logbooks signed, dated and legible?
2. Are changes to logbooks dated and initialed by the person who made them?
3. Can data be tracked from the project files?
4. Do the project files identify the specific pieces of instrumentation that were used?
5. Have lab data management systems been validated prior to use?
6. Are data manipulation procedures adequately described?
7. Are data (electronic and hardcopy) archived in a retrievable fashion?
8. Is there a projection/run tracking/filing system in place?
9. Is it possible to back-track and validate a final piece of data from it's beginning?
10. Are data periodically confirmed by independent (i.e. manual) reduction?
11. Are there written instructions for data receipt, storage, retrieval?
12. Are documents issued by the work assignment subject to a document control system?
13. Are data entered into the computer "checked at least three times by at least two people?
14. Are lab notebooks inspected by the group leader?
15. Is the inspection documented?

Strengths and weaknesses:

Section 5: Problem Resolution

1. Has a person been designated to follow-up on previously identified problems?

2. Has a time frame been stipulated for resolving problems?

3. Does documentation of the resolution of problems exist?

Strengths and weaknesses:

APPENDIX D

DATA REVIEW CHECKLIST EXAMPLE

Data Review Checklist

The following pages contain a data review checklist that may be used by data reviewers, laboratory personnel, and other parties to document the results of each data inspection in a standardized format.

Data Inspection Checklist

Summary Information			
1. Name of Reviewer: _____		Title: _____	
Required Samples		Sample Results Provided	
Sample Location or Sample ID	Analyte(s)	Sample Location or Sample ID	Analyte(s)

2. Method Used:

3. Total No. of analytical shifts per instrument (determined from analysis run log):

Instrument	No. of Shifts

4. Total No. of CCVs Required: (one for each 10 samples after the first 10 samples on each instrument)	_____	Total No. of CCVs Reported:	_____
5. Total No. of CCBs Required: (one for each CCV)	_____	Total No. of CCBs Reported:	_____
6. Total No. of Field Blanks Required: (one per site or per 10 samples, whichever is more frequent)	_____	Total No. of Field Blanks Reported:	_____
7. Total no. of Lab Blanks Required: (one per batch' per method/instrument)	_____	Total No. of Lab Blanks Reported:	_____
8. Total no. of OPR analyses Required: (one per batch per method/instrument)	_____	Total No. of OPR Analyses Reported:	_____
9. Total no. of MS/MSD samples Required: (one per 10% per matrix per site)	_____	Total No. of MS/MSD samples Reported:	_____
10. Total no. Field Duplicates Required: (one per 10 samples per site)	_____	Total No. of Field Duplicates Reported:	_____
11. Total no. of MDL results required: (one per method and per analyte)	_____	Total No. of MDL Results Reported:	_____

15. Initial Precision and Recovery (IPR)

a. Were IPR data reported for each analyte? ☐yes ☐no

b. Did all IPR aliquots meet required recovery criteria (x)? ☐yes ☐no

c. Did the standard deviation (s) of each IPR series meet the required criterion? ☐yes ☐no

d. If any item in a - c above was answered "no", document problem below.

Analyte	Ave. Result Reported (X)	RSD Reported	Affected Samples

16. Ongoing Precision and Recovery (OPR)

a. Were OPR data reported for each analyte, instrument, and batch? ☐yes ☐no

b. Did all OPR samples meet required recovery criteria (x)? ☐yes ☐no

c. If item a or b above was answered "no", document problem below.

Analyte	OPR Recovery (X) Reported	Shifts Missing OPR	Affected Samples

17. Continuing Calibration Verification (CCV)/Continuing Calibration Blank (CCB)

a. Were CCVs run prior to each batch of 10 samples on each instrument? ☐yes ☐no

b. Were all CCV results within the specified windows? ☐yes ☐no

c. Was each CCV followed by a CCB? ☐yes ☐no

d. Was each CCB free from contamination? ☐yes ☐no

e. If any item in a - d above was answered "no", list problems below:

Analyte	Affected Samples	Shift Missing CCV/CCB	Failed CCV/CCB ID

18.	Laboratory (Method) Blanks		
a.	Was a method blank analyzed for each instrument & sample batch?	☐yes	☐no
b.	Was each method blank demonstrated to be free from contamination?	☐yes	☐no
c.	If the answer to item a or b was "no", document problems below.		

Analyte	Affected Samples	Blank Concentration Reported	Shift Missing MB

19.	Field Blanks		
a.	Was a field blank analyzed for each 10 samples per site?	☐yes	☐no
b.	Was each field blank demonstrated to be free from contamination?	☐yes	☐no
c.	If the answer to item a or b was "no", document problems below.		

Analyte	Affected Samples	Blank Concentration Reported	Shift Missing FB

20.	MS/MSD Results		
a.	Were appropriate number of MS/MSD pairs analyzed?	☐yes	☐no
b.	Were all MS/MSD recoveries within specified windows?	☐yes	☐no
c.	Were all RPDs within the specified window?	☐yes	☐no
d.	Was appropriate corrective action (e.g., MSA for GFAA, serial dilution for ICP) employed on affected samples?	☐yes	☐no
e.	If the answer was "no" to items a - d above, document affected samples:		

Analyte	MS % R	MSD % R	MS/MSD RPD	Affected Samples

21.	Additional Information		
a.	Were Instrument Tune Data Provided?	☐yes	☐no
b.	Were equipment blanks demonstrated to be free from contamination?	☐yes	☐no
c.	Were statements of data quality provided?	☐yes	☐no
d.	Did field duplicate demonstrate acceptable precision?	☐yes	☐no

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