Report of the
Federal Advisory Committee
on
Detection and Quantitation Approaches and Uses
in
Clean Water Act Programs

Submitted to the

US Environmental Protection Agency

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ACKNOWLEDGMENTS

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Dear Administrator Johnson:

We are pleased to present to you the Final Report of the Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act Programs. This report responds to the charter from the US Environmental Protection Agency to "provide advice and recommendations on approaches for the development of detection and quantitation procedures and uses of these procedures in Clean Water Act programs."

Our Committee included balanced representation from states, industry, environmental laboratories, public utilities and the environmental community as well as EPA's Director of Engineering and Analysis Division. What brought the members of our Committee to the table and kept us hard at work for two and a half years was a common desire to improve federally-approved analytical procedures for determining Detection and Quantitation Limits and to reach agreement on the uses of the results.

We tackled difficult policy and technical questions. We agreed by consensus on many important issues and expect EPA will move these recommendations forward. We put other issues on the table which we all agreed are important but on which we could not reach consensus within the time available. In these cases, we have provided you with the full array of opinions on the Committee so you will have the benefit of our deliberations. We urge EPA to address these issues at the same time it considers our consensus recommendations.

We would like to thank the Office of Water for affording our Committee the opportunity to address these important issues and for providing significant resources for our work, including funds for the Pilot Study that were instrumental in developing for the Committee consideration of a single laboratory procedure for detection and quantitation. We also appreciate the outstanding support that EPA staff provided throughout our deliberations.

We respectfully request a formal response to our recommendations.

Sincerely,

Members, Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act Programs

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ACRONYMS

ACIL: American Council of Independent Laboratories
ASTM: American Society for Testing and Materials
ASIWPCA: Association of State and Interstate Water Pollution Control Administrators
ATP: Alternative Test Procedures
CFR: Code of Federal Regulations
CWA: Clean Water Act
DL: Detection Limit
DL_{lab}: Laboratory Detection Limit
DL_{nat}: National Detection Limit
DL_{per}: Permit Detection Limit
DMR: Discharge Monitoring Report
DNQ: Detected but not Quantified
DQI: Data Quality Indicator
DQO: Data Quality Objective
ELG: Effluent Limitation Guideline
FACDQ: Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act Programs
GLI: Great Lakes Initiative
ICIS: Integrated Compliance Information System
IDE: Interlaboratory Detection Estimate
IIAG: Inter-Industry Analytical Group
IQE: Interlaboratory Quantitation Estimate
ISO/IUPAC: International Organization for Standardization/International Union of Pure and Applied Chemistry
L_C: Critical value
LCMRL: Lowest Concentration Minimum Reporting Limit
L_D: Detection Limit
LTA: Long-Term Average
L_Q: Quantitation Limit
MCL: Maximum Contaminant Level
MDL: Method Detection Limit
ML: Minimum Level
MMA: Michigan Manufacturers Association
MQO: Measurement Quality Objective
MRL: Minimum Reporting Limit
NACWA: National Association of Clean Water Agencies
NELAC: National Environmental Laboratory Accreditation Conference
NPDES: National Pollutant Discharge Elimination System
OECA: US EPA's Office of Enforcement and Compliance Assurance
OGWDW: US EPA's Office of Ground Water and Drinking Water
OSW: US EPA's Office of Solid Waste
PCB: Polychlorinated Biphenyl

POTWs: Publicly-Owned Treatment Works
PQL: Practical Quantitation Level
PT: Proficiency Testing
QL: Quantitation Limit
QL_{lab}: Laboratory Quantitation Limit
QL_{nat}: National Quantitation Limit
QL_{per}: Permit Quantitation Limit
QL_{state}: State Quantitation Limits
RDL: Reliable Detection Limit
RSD: Relative Standard Deviation
SOC: Synthetic Organic Chemicals
SVOC: Semivolatile Organic Compound
TMDL: Total Maximum Daily Load
USGS: United States Geological Survey
WET: Whole Effluent Toxicity
WQC: National Water Quality Criteria
WQS: State Water Quality Standards
WQBEL: Water Quality Based Effluent Limits

EXECUTIVE SUMMARY

Introduction

Under the Clean Water Act, the US Environmental Protection Agency (EPA) is responsible for approving analytical procedures for monitoring wastewater pollutants. Detection (determining a pollutant's presence) and quantitation (determining the quantity of the pollutant) are significant issues for regulators, the regulated community, environmental laboratories that analyze wastewater for monitoring and compliance purposes, other agencies that must use EPA-approved analytical methods, and those who focus on human health and the environment.

By 2005, when EPA chartered the Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act Programs (Committee), concerns with the Method Detection Limit (MDL) procedure as published in 40 CFR Part 136, Appendix B were well characterized. The charge to the Committee was "to provide advice and recommendations on approaches for the development of detection and quantitation procedures and uses of these procedures in Clean Water Act programs."

Over a 30-month period, the Committee worked diligently on challenging policy and technical issues related to detection and quantitation. The Final Report details all of the Committee's recommendations and summarizes discussions of many important issues where consensus could not be achieved.

Procedure for Detection and Quantitation

Early in its work, the Committee reached agreement on 15 statements that accurately describe "What We Need A Procedure To Do." These statements were subsequently used as criteria for evaluating potential procedures for detection and quantitation. The Committee selected five procedures to test in a Pilot Study. When reviewing the Pilot Study results, the Committee agreed that the American Council of Independent Laboratories (ACIL) procedure included most of the elements that Committee members had said needed to be incorporated in a procedure. The Committee then revised the procedure, based on the Pilot Study results, to improve its performance, producing the DQ FAC Single Lab Procedure v2.4.

When the Committee voted on the DQ FAC Single Lab Procedure v2.4 as the proposed single laboratory procedure for determination of Detection and Quantitation Limits, the Committee did not reach consensus. However, the Committee did reach consensus on the following motion which supports the implementation of a new procedure:

> The Committee recommends that EPA act to develop an alternative to the current 40 CFR Part 136, Appendix B procedure. The results of the pilot study and evaluation of the ACIL modified procedure indicate that there are deficiencies in the current 40 CFR Part 136, Appendix B procedure that can and should be

corrected. The Single Lab DL QL Procedure v2.4 submitted contains elements that would be valuable to the agency in developing a new procedure.

Looking ahead to further work by EPA on procedure/s for detection and quantitation, the Committee recommended that a formal peer review of the procedure proposed for promulgation be undertaken and that a follow up pilot study be completed to confirm the performance of whatever procedure/s EPA proposes to promulgate.

Data Quality

The Committee approached the issue of data quality in two ways. First, the Committee reached agreement on Measurement Quality Objectives for purposes of the pilot testing single laboratory detection procedures study; however, the Committee was not able to reach agreement on universal Measurement Quality Objectives that would apply across the board for the use of quantitation for National Pollutant Discharge Elimination System (NPDES) permit compliance testing.

The Committee's second approach was to focus on the broader issue of Data Quality Objectives. In this area, the Committee reached consensus that EPA's Office of Water should, in all Clean Water Act programs, employ the Data Quality Objectives Process.

Uses of a Procedure for Detection and Quantitation

Initially, the Committee performed a preliminary review of where detection and quantitation may be used in most of the Clean Water Act programs and found potential differences in how these programs make use of Detection and Quantitation Limits. Time did not permit the Committee to fully evaluate the differences of all of the specific uses of detection and quantitation, let alone make specific recommendations, so a decision was made early on to focus instead on the use of Detection and Quantitation Limits in the NPDES permitting program. As a result, the Committee affirmatively decided to table discussion and recommendations on uses of Detection and Quantitation Limits in other Clean Water Act programs.

The Committee did fully discuss and vote on recommendations for the determination and use of Detection and Quantitation Limits in NPDES permitting and compliance processes, particularly in those situations where Water Quality Based Effluent Limits (WQBELs) are less than Quantitation Limits. Because of uncertainties surrounding data validity, these situations present a challenge in setting permit limits and conditions as well as in making compliance determinations. To address this challenge, the Committee fashioned a package of recommendations for regulated parties, EPA and states to use in applications such as data reporting, calculating monthly averages, and determining compliance. These recommendations are interlinked and were intended to represent the balanced package discussed by the Committee over the course of its deliberations. It was the Committee's intent that the recommendations of this section be implemented as a whole and not in a piecemeal fashion.

The Committee repeatedly affirmed that the various pieces of the Uses Document represented a package formed by give-and-take of the various competing interests and

individual Committee members. Committee members expected that the package would be voted on as a whole and in a single vote. Members assumed that either the entire package would be approved by consensus or the entire package would not be approved. Instead, five votes were taken on the package and some components of the package were approved by consensus while others were not. This voting process could have affected the outcome.

Some of the Committee's recommendations and majority opinions on uses of Detection and Quantitation Limits in the NPDES program are dependent on a national benchmark for quantitation, a National Quantitation Limit. The concept of a National Quantitation Limit was a key component of a "package of uses recommendations" that the Committee developed over many months. It was also intended to define the minimum level of acceptable performance by a laboratory analyzing wastewater for compliance determinations and to establish an important threshold for NPDES program compliance reporting when analyte WQBELs are below the capability of all approved methods.

The Committee offered several consensus recommendations related to National Quantitation Limits within the context of the complete uses package.

- The Committee recommended that National Quantitation Limits by analyte be promulgated in a table to be included in Part 122.
- The Committee recommended that EPA generate National Quantitation Limits as rapidly as possible.
- The Committee recommended that Quantitation Limits be promulgated only using a nationally promulgated approach yet to be defined.
- The Committee recommended that EPA have the latitude to promulgate a method without promulgating a Quantitation Limit for that method. As a new method is proposed without a promulgated Quantitation Limit, data (e.g., Single Laboratory Detection, Single Laboratory Quantitation, etc.) showing demonstrated method performance should be included in the method. The method should include a statement that performance levels are guidance and may not always be achievable.

The Committee recommended by consensus that EPA promulgate how National Quantitation Limits will be derived and a majority of the Committee suggested a number of criteria that could be considered when EPA proposes such a procedure. Finally, the Committee expressed a desire for EPA to promulgate new, more sensitive analytical methods.

Additional Consensus Recommendations

Procedure Verification

- The Committee recommended that EPA give additional consideration to increasing the frequency of QL verification and report its findings in the preamble of the *Federal Register* Notice and request specific comments on the final proposed frequency.

- The Committee recommended that during promulgation, EPA include and/or develop language to incorporate batch specific verification as an option in the procedure.

Implementation of a Procedure for all EPA Programs Referencing 40 CFR Part 136

- The Committee recommended that, to maintain consistency and minimize effects on the environmental laboratory community, EPA programs that reference the present Part 136 Appendix B procedure consider adopting a new procedure that would replace it.

Implementation Tools

- The Committee recommended that EPA develop guidance and outreach materials for stakeholders as EPA implements the FACDQ recommendations.
- The Committee recommended that EPA develop and implement guidance on the new procedures as well as a computer-based program to assist in calculating Detection and Quantitation Limits.

Measurement Quality Objectives

- The Committee recommended a ≤ 1% false positive rate be used for detection.
- The Committee recommended that for promulgated methods listed in 40 CFR Part 136 without established Measurement Quality Objectives, the initial Measurement Quality Objective for quantitation (upon implementation of the new quantitation procedure) be a specific false negative rate (≤ 5%) to be implemented through a multiplier of the Detection Limit, and that precision and accuracy for individual analytes/methods would be generated and promulgated, as the data to support those Measurement Quality Objectives becomes available.

Need for Data Comparability Assurance

- The Committee recommended that, during the Data Quality Objectives process, EPA give special attention to assuring that, at or near the National Quantitation Limit, the specific analytical method produces comparable results on split samples analyzed in different laboratories.

EPA Leadership Role in Clean Water Act Method Development

- The Committee recommended that EPA continue to act as the national lead for developing analytical methods and setting performance standards for Clean Water Act program analytical methods.

Targeting EPA Resources for Analytical Methods

- The Committee recommended that EPA dedicate and evaluate federal resources, and adjust those resources as necessary, to develop analytical methods with Detection/Quantitation Limits of sufficient quality to meet Clean Water Act data quality and program needs.

Great Lakes Initiative Compliance

- The Committee recommended that Committee recommendations not supersede the current Great Lakes Initiative provisions.

Conclusion

The Committee presented EPA with a number of consensus recommendations and where consensus could not be achieved, summaries of the Committee's discussions or decisions are provided. These recommendations are intended to help EPA improve the policy and science related to detection and quantitation in Clean Water Act programs, with a focus on the NPDES permitting process. Due to the fact that these are important issues and the Committee believes the recommendations and decisions could lead to improvements, we urge EPA to seriously consider all of the issues summarized in this report and implement the Committee's recommendations as soon as practical.

TO THE READER

This section provides an orientation to the information in this report and how it is presented.

Committee Decision-making and Votes
The Committee's ground rules defined consensus as agreement of all members, and conversely, consensus was the method of determining Committee agreement on issues. Members voted using one of three options: "agree," "disagree," or "not opposed." Consensus was defined as all members "agreeing" or "not opposed to" the decision. At Meetings 1 - 9, votes were tallied as totals for "agree," "disagree" or "not opposed." At Meeting 10 (September 19-21, 2007) when most Committee recommendations and decisions were finalized, the Committee agreed to display votes in the Final Report by caucus. Consequently, in this report, votes from Meetings 1 through 9 are given as total votes only, whereas votes from Meeting 10, 11, and 12 are given as both total votes and votes by caucus.

The Committee agreed by consensus to refer to Committee recommendations and decisions as follows:

- Recommendations and decisions approved by consensus are referred to as "consensus recommendations" and "consensus decisions," respectively. These votes are noted as "Approved By Consensus."
- Recommendations and decisions not approved by consensus are collectively referred to as "majority opinions" and, in one case, a "majority of the Committee voted not to recommend." These votes are noted as "Not Approved."

This report also refers to non-binding "straw polls" taken during Committee deliberations. These straw polls were taken as proposals were being developed to get a sense of Committee sentiment and to focus subsequent discussions.

In Committee decision-making, EPA voted as the Office of Water.

Majority – Minority Reports for Non-Consensus Decisions
At Meeting 10, the Committee agreed to provide majority and minority reports for non-consensus decisions; the majority and minority reports are presented following the decision to which they relate. Majority reports are followed by minority reports; the latter are indented for clarity.

Terms Used in the Report
A major focus of the Committee's work was to develop a recommendation on a detection and quantitation procedure or procedures to replace the procedures in 40 CFR Part 136, Appendix B. Over the course of its 30 months of work, the Committee used several terms to describe a procedure that could be used by a single laboratory to determine its

Laboratory Detection and Quantitation Limits. The single laboratory procedure developed and voted on by the Committee is consistently presented as the "DQ FAC Single Laboratory Procedure v2.4" (i.e., the American Council of Independent Laboratories (ACIL) modified procedure).

As the Committee developed a package of recommendations on uses, it proposed new concepts and terms to facilitate implementation. These terms (discussed in Chapters 3 and 4) include:

- National Quantitation Limit (referred to in many Committee documents as QL_{nat})
- Laboratory Detection Limit (referred to in many Committee documents as DL_{lab})
- Permit Quantitation Limit (referred to in many Committee documents as QL_{per})
- State Quantitation Limit (referred to in many Committee documents as QL_{state})

During Committee deliberations, the members adopted the convention of referring to analytical methods as "methods" and Detection or Quantitation Limit procedures as "procedures." This report continues that convention.

Public Notice and Comment

The Committee recognized that EPA could not commit to promulgate the recommendations of the Committee without the benefit of public notice and comment. Wherever "promulgate" appears in the Final Report, the Committee's assumption is that EPA will propose a rule consistent with the Committee recommendations and will fully consider public comments before deciding on its final actions.

CHAPTER 1 – PURPOSE OF THE COMMITTEE AND COMMITTEE PROCESS

1.1 Background

In 1999, several industry groups filed suit against EPA (Alliance of Automobile Manufacturers, et al. v. EPA, No. 99-1420, (D.C. Cir.)) and in October, 2000, the parties reached a settlement agreement that required EPA to assess procedures to determine Detection and Quantitation Limits under EPA's Clean Water Act (CWA) programs by November 1, 2004. Pursuant to this agreement, on March 12, 2003, EPA issued for public comment a draft report assessing various detection and quantitation procedures and a proposed rule amending EPA's Method Detection Limit (MDL) and Minimum Level (ML) definitions and procedures. The vast majority of the 126 comments EPA received in response to the *Federal Register* notices were critical of the conclusion of EPA's assessment and proposed revisions.

1.2 Situation Assessment

Rather than proceeding with the revisions, EPA decided to withdraw the proposed rule and contract with a neutral third party, Triangle Associates, Inc., to conduct a situation assessment. The purpose of the situation assessment was to obtain additional input on technical and policy issues related to detection and quantitation and to explore the feasibility and design of a stakeholder process.

As a result of the interviews conducted for the situation assessment, Triangle Associates recommended that a Federal Advisory Committee be formed to address detection and quantitation issues and concluded that the Committee stood a good chance of achieving consensus on revised detection and quantitation approaches and uses in Clean Water Act programs. Triangle also found, however, that many of the interviewed stakeholders believed that the process would only be successful with a strong commitment from EPA. To emphasize the need for this commitment, the assessment report recommended that EPA have a seat at the table.

1.3 Creation of the Committee

EPA accepted Triangle's recommendation and in May 2005 formed a Federal Advisory Committee under the Federal Advisory Committee Act. The two-year Charter for the Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act Programs specified that the purpose of the Committee was to provide advice and recommendations on approaches for the development of detection and quantitation procedures, and uses of these procedures, in Clean Water Act programs. The Committee initially consisted of 21 Committee members representing a diverse group of professionals from the following sectors: state government, environmental laboratories, regulated industry, public utilities, the environmental community, and EPA. (The Committee members, who were organized in caucuses, are listed on the report inner

cover). On May 30, 2007, the Committee's Charter was renewed to give the Committee additional time to complete its work.

1.4 Committee Process

The Committee met 12 times; the first meeting was held on June 21-22, 2005 and the last meeting was held on December 21, 2007. At the outset, a Technical Work Group was created to carry out assignments on technical issues. The Technical Work Group was tasked with preparing papers on definitions relevant to detection and quantitation, presenting concepts, proposing criteria for evaluating possible detection and quantitation procedures, recommending procedures for the Pilot Study for the Committee's consideration, designing the Pilot Study, evaluating Pilot Study results, preparing a Pilot Study report, and many other tasks of a technical nature. Over the course of the Committee's work, the Technical Work Group held 70 conference calls.

At the Committee's September 29-30, 2005 meeting, the Committee created a Policy Work Group. Its initial purpose was to 1) identify and define uses of detection and quantitation; 2) identify the existing situation for each use category and Data Quality Objectives for each type of use and user; and 3) pose policy issues that emerge from these assignments. Over time, the Policy Work Group was asked to identify issues, explore options, and draft documents to frame discussions of specific issues in advance of Committee meetings. At a Committee meeting, the Committee would then take up the document for decision-making, with the possibility of assigning subsequent tasks to the Policy Work Group for the next meeting. The Policy Work Group held 42 conference calls and one face-to-face meeting.

The composition of both Work Groups reflected balanced membership from the Committee's caucuses.

As the Committee's work progressed, the Committee gave specific assignments to the Technical Work Group and to the Policy Work Group to carry out before the next Committee meeting.

More information and summaries of Committee meetings and meetings of the Technical Work Group and the Policy Work Groups are available at www.epa.gov/waterscience/methods/det/index.html and in EPA's public docket, EPA-HQ-OW-2004-0041.

1.5 Definitions of Detection and Quantitation

IUPAC Definitions

In interviews conducted for the situation assessment, a number of parties had argued that EPA methods should adopt definitions of detection and quantitation (L_C, L_D and L_Q) that are consistent with or the same as those of the International Union of Pure and Applied Chemistry (IUPAC.) The Committee tasked the Technical Work Group with considering the adoption of the IUPAC definitions. While the Technical Work Group was in general agreement with the IUPAC definition concepts, it ultimately recommended against adoption because the definitions lack direction on how they could be implemented in the existing environmental monitoring program framework without major, costly changes and because EPA methods generally disallow blank subtraction.

Most members of the Technical Work Group also believed there were no practical ways to adapt the IUPAC definitions to accommodate commonly found situations where data are censored or not normally distributed or where variance is not constant.

In the end, the Committee chose to decouple its definitions, but not its concepts, from IUPAC and the subsequent calculation procedure and to develop a more general way to produce estimates with a statistical confidence that could be applied to a greater variety of measurement technologies and issues. The Committee did agree to incorporate the IUPAC definitions into the glossary (Decision #11A - Recommendation #1, "The FACDQ recommends adding the IUPAC L_C, L_D, and L_Q definitions into the glossary.").

Committee Definitions

Although the IUPAC conventions use three points, L_C, L_D, and L_Q, to define detection and quantitation, the Committee agreed to the use of two points to define detection and quantitation for a number of reasons. Two points are currently used by EPA (the MDL and ML) and these are conceptually equivalent to the Detection Limit and Quantitation Limit defined in the DQ FAC Single Laboratory Procedure v2.4.

1. The Committee determined it would be extraordinarily difficult to confirm a predicted value for L_D, requiring hundreds of spikes at very closely spaced intervals.
2. Use of a three level system would be very difficult to implement. Laboratory reporting systems do not generally have that capability, and there is no definition of how the three levels would be utilized by the data user.

The Committee agreed that while the concept of the L_D was important, it would be acceptable not to derive an L_D, on the condition that the false negative error rate at the Detection Limit was acceptable for results at the Quantitation Limit.

The Committee then agreed to "working definitions" of detection and quantitation including two layperson definitions and two statistical definitions of detection, as follows:

DETECTION LIMIT (DL) – LAYPERSON'S DEFINITIONS

1. **Detection Limit (DL)**: The minimum result which can be reliably discriminated from a blank (for example, with a 99% confidence level).
2. **Detection Limit (DL)**: The lowest result that can be distinguished from the blank at a chosen level, α, of statistical confidence.

DETECTION LIMIT (DL) - STATISTICAL DEFINITIONS

1. **Detection Limit (DL)**: Smallest measured amount or concentration of analyte in a sample that gives rise to a Type I error tolerance of alpha under the null hypothesis that the true amount or concentration of analyte in the sample is equal to that of a blank. (The alternative hypothesis is that the true amount or concentration of analyte is greater than that of a blank).
2. **Detection Limit (DL)**: The minimum observed result such that the lower 100 (1- α) % confidence limit on the result is greater than the mean of the method blanks.

Vote: 12 Agree, 7 Not Opposed, 0 Disagree, 1 Absent
Approved By Consensus

States: 4 Agree,
Labs: 2 Agree, 2 Not Opposed
Industry: 4 Agree
EPA: 1 Not Opposed
Public Utilities: 4 Not Opposed
Environmental Community: 2 Agree, 1 Absent
Meeting #10, Decision 11.B

QUANTITATION LIMIT (QL) - DEFINITIONS

1. **Quantitation Limit (QL)**: The smallest detectable concentration of analyte greater than the Detection Limit (DL) where the accuracy (precision & bias) achieves the objectives of the intended purpose.
2. **Lab Quantitation Limit (QL_{lab})**: The smallest detectable concentration of analyte greater than the Detection Limit (DL) where the accuracy (precision & bias) demonstrated by the laboratory achieves the objectives of the intended purpose.

Vote: 3 Agree, 16 Not Opposed, 0 Disagree, 1 Absent
Approved By Consensus

States: 4 Not Opposed,
Labs: 4 Not Opposed
Industry: 1 Agree, 3 Not Opposed
Public Utilities: 4 Not Opposed
EPA: 1 Agree
Environmental Community: 1 Agree, 1 Not Opposed, 1 Absent
Meeting #10, Decision 11.B

CHAPTER 2 – DATA QUALITY OBJECTIVES AND MEASUREMENT QUALITY OBJECTIVES

2.1 Introduction

The Committee recognized the importance of following a Data Quality Objectives process in developing performance and acceptance criteria for data to be used in detection and quantitation decisions. This process includes identification of appropriate Data Quality Indicators, defined as quantitative and qualitative measures of data quality attributes such as precision, accuracy, and representativeness. This process also includes the establishment of Data Quality Objectives, or qualitative and/or quantitative statements which, in the context of detection and quantitation decisions, define the appropriate type of data needed to achieve the required decision certainty. Finally, the process involves the selection of Measurement Quality Objectives, or specific quantitative measures of performance in relation to particular Data Quality Indicators, such as specific values for precision, bias, and false positive or false negative error rates.[1]

The Committee recognized that EPA has developed, through its Quality System program, a number of guidance documents related to environmental data quality, in particular in relation to a project-specific Data Quality Objectives process.[2] However, there has been less focus on applicability to more routine monitoring done as part of mandatory programs (e.g., National Pollutant Discharge Elimination System or NPDES compliance monitoring under the Clean Water Act). The Committee attempted to address Data Quality Objective and Measurement Quality Objective issues in the context of decision certainty in NPDES compliance, specifically as they relate to detection and quantitation.

This Chapter discusses the Committee's recommendations on some of these issues. Although the specifics of applying the Data Quality Objectives process to other aspects of Clean Water Act programs were not discussed, this Chapter presents discussions and recommendations regarding the application of the same principles and practices that were

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[1] More specific or detailed definitions of these key terms in the Data Quality Objectives Process utilized by EPA include the following:
Data Quality Indicators: quantitative and qualitative measures of principal quality attributes, such as precision, accuracy, representativeness, and sensitivity.
Data Quality Objectives: qualitative and quantitative statements that clarify study objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions.
Measurement Quality Objectives: "acceptance criteria" for the quality attributes measured by project data quality indicators. During project planning, measurement quality objectives are established as quantitative measures of performance against selected data quality indicators, such as precision, bias, representativeness, completeness, comparability, and sensitivity.
Source: US EPA, Guidance on Environmental Data Verification and Data Validation (*QA/G-8*), EPA/240/R-02/004, November 2002, http://www.epa.gov/quality/qs-docs/g8-final.pdf.

[2] See, for example, US EPA, *Guidance on Systematic Planning Using the Data Quality Objectives Process (QA/G-4)*, EPA/240/B-06/001, February 2006, http://www.epa.gov/quality/qs-docs/g4-final.pdf, and other documents available at http://www.epa.gov/quality/.

discussed in the context of NPDES compliance testing to other aspects of Clean Water Act programs. The Chapter continues with more detailed discussion of issues, consensus recommendations, and majority opinions related to Data Quality Objectives and Measurement Quality Objectives appropriate for NPDES permit compliance testing.

All detection and quantitation procedures considered by the Committee required the selection of one or more Measurement Quality Objectives (e.g., false positive rate, false negative rate, accuracy, and/or precision). In some instances, procedures were designed around a particular Measurement Quality Objective. For example, all procedures (for detection) considered by the Committee targeted a 1% false positive rate. In discussion, it was generally agreed that detection would not require specific accuracy or precision. As evidenced by the definition of quantitation adopted by the Committee (p. 4), it was agreed that at least accuracy and precision would be required for determining quantitation and that the Quantitation Limit must be greater than the Detection Limit.

Because the detection and quantitation procedures require that these Measurement Quality Objectives be addressed, it was appropriate for the Committee to discuss how Measurement Quality Objectives would be set or determined. Initial discussion on specific numerical values for many potential Measurement Quality Objectives indicated that Committee consensus could not be achieved. Thus, the Committee decided to consider broader or more general recommendations rather than trying to achieve consensus on specific numerical values. This approach led to the following proposed recommendations and majority opinions.

2.2 Recommendations and Decisions on Data Quality Objectives

The Committee recognized that the Charter directed the Committee to consider recommendations with respect to determination and use of detection and quantitation in Clean Water Act programs. The Committee considered and discussed the application of the Data Quality Objectives setting process as an appropriate process for determining what Measurement Quality Objectives and Data Quality Indicators would be suitable for different uses within Clean Water Act programs. The Committee determined that it would be appropriate to apply such a process (although it did not discuss the process in detail) to all components of Clean Water Act programs and made the following recommendation accordingly.

Data Quality Objective Recommendation

The Committee recommends that the EPA Office of Water use the *EPA Guidance on Systematic Planning Using the Data Quality Objectives Process* in all Clean Water Act programs.

Vote: 17 Agree, 0 Not Opposed, 0 Disagree, 1 Absent

Approved By Consensus

Meeting #7, Decision 3

In the Committee's discussion of this consensus recommendation it was made clear that the intent was not to require the Office of Water to follow the referenced document strictly or in all detail. Rather, the intent was to indicate that EPA should go through a Data Quality Objectives process that looks at decision uncertainty (e.g., the compliance decision), determine which Measurement Quality Objectives are appropriate, and derive Measurement Quality Objectives consistent with the decision uncertainty requirements. The Committee believes that the Office of Water's current approach does not incorporate an appropriate Data Quality Objectives process. Some members of the Committee believe that selecting a measurement technology and then targeting Measurement Quality Objectives consistent with that technology's historical performance is not an appropriate Data Quality Objectives process.

Establishing Data Quality Objectives for Decision-Making in Clean Water Act Programs

The Data Quality Objectives process is intended to assure appropriate decision-making certainty and, thus, is equally applicable throughout all aspects of Clean Water Act programs. Time did not permit detailed Committee discussions, but that does not imply application of a Data Quality Objectives process is not equally important in other aspects of Clean Water Act programs. The following proposed recommendation expands on the previous consensus recommendation and provides more detail and clarity on the intent of the Committee. The Committee considered the broader issue and voted on the following language.

The Committee recommends that EPA establish Data Quality Objectives (with indicators and Measurement Quality Objectives) for Clean Water Act programs where Detection/Quantitation Limits are used in decision making.

Vote: 15 Agree, 4 Not Opposed, 1 Disagree

Not Approved

States: 3 Agree, 1 Not Opposed
Labs: 1 Agree, 3 Not Opposed
Industry: 4 Agree
Public Utilities: 4 Agree
EPA: 1 Disagree
Environmental Community: 3 Agree
Meeting #10, Decision 5.G

Majority Report in Support of the Proposal

This proposal was developed to clarify the intent of the Committee regarding the Data Quality Objectives process recommended by the Committee. EPA's Data Quality Objectives guidance states that specific Data Quality Objectives, Data Quality Indicators and Measurement Quality Objectives should be adopted prior to beginning any study or data collection effort. Data Quality Indicators may include measures of data quality including, but not limited to, accuracy, precision, false positive and false negative rates, comparability, representativeness and completeness. For example, EPA should consider

adopting a Measurement Quality Objective for accuracy at the Quantitation Limit to define the quality of data at that limit, thereby determining actions that can be taken given the quality of that data. Data Quality Indicators are accompanied by corresponding Measurement Quality Objectives defining the limits of acceptability for each Data Quality Indicator.

The Committee did not reach consensus on which Data Quality Indicators and corresponding Measurement Quality Objectives should be recommended other than for the false positive rate at the Detection Level. However, a majority of the Committee does believe that EPA should evaluate the uses of data in all Clean Water Act programs and determine the quality of data required to meet those uses prior to making regulatory decisions where detection and quantitation are in question.

A majority of the Committee believe Measurement Quality Objectives may not be achievable by the performance of all current analytical methods and when the Measurement Quality Objective(s) are not achieved, the use of data for the intended purpose should be evaluated for use. The majority's intent was to consider method performance when adopting Measurement Quality Objectives and to modify the use of the data accordingly, when necessary, but not to allow analytical methods alone to define Measurement Quality Objectives for Clean Water Act programs.

For example, the Measurement Quality Objective for accuracy at the Quantitation Limit when determining compliance with a permit limit may be more rigorous than an analytical method can provide. The majority opinion, in this example, would require one or more modifications of how data are used or qualified including, but not limited to, adjustment of the Quantitation Limit to meet the MQOs, use of another acceptable method meeting the MQOs, collection of additional data (and addressing that data's uncertainty), or use of professional judgment to justify a basis for using the data as reported or selecting another approach.

The goal of the approach used to address when MQOs are not met is to reach the level of decision certainty that is required for the use (in this example, certainty in correctly concluding exceedance of a limit for compliance). If the level of decision certainty is insufficient, then the use is adjusted.

Minority Report in Opposition to the Proposal

EPA voted to disagree with the proposed recommendation, "Establishing Data Quality Objective's for Decision-making in Clean Water Act Programs," based on concerns about resources. The core recommendations of the Committee – pilot test the new single laboratory procedure, promulgate and implement new rules incorporating the single laboratory procedure, propose an algorithm for determining the National Quantitation Limit, and define the uses of detection and quantitation in compliance and enforcement of NPDES permits – will require significant EPA resources over the next several years. At this time, EPA cannot commit additional resources to several of the other recommendations of this

report, including the recommendation, "Establishing Data Quality Objectives for Decision-making in Clean Water Act Programs," until these core recommendations are implemented.

2.3 Recommendations and Decisions on Measurement Quality Objectives for Measurements Used in NPDES Compliance Testing

The Committee's discussions with respect to Measurement Quality Objectives focused on NPDES permitting and compliance testing.

Measurement Quality Objective for Detection – False Positive Rate

> The Committee recommends that a ≤ 1% False Positive rate be used for Detection.
>
> *Vote*: 17 Agree, 0 Not Opposed, 0 Disagree, 1 Absent
>
> Approved By Consensus
>
> Meeting #7, Decision 4.A

The Committee agreed by consensus with the general premise that detection should target a false positive rate not to exceed 1%. A 1% false positive rate is consistent with a number of approaches adopted for detection. Furthermore, all Detection Limit procedures considered in the Pilot Study were designed to implement this Measurement Quality Objective. The IUPAC definitions for detection (L_D) include control of false negatives (≤ 5%). The Committee agreed to ignore false negatives for detection but instead included them in the concept of quantitation as a condition of dropping L_D.

Measurement Quality Objectives for Quantitation for Promulgated Methods

> The Committee recommends that for promulgated methods in 40 CFR part 136 without established Measurement Quality Objectives, the initial Measurement Quality Objectives for quantitation upon implementation of the new quantitation procedure is a specific false negative rate (≤ 5%) to be implemented through a multiplier of the Detection Limit (determined by the DQ FAC Single Laboratory Procedure v2.4). The precision and accuracy Measurement Quality Objectives for individual analytes/methods would be generated and promulgated as the data to support those Measurement Quality Objectives become available.
>
> *Vote*: 17 Agree, 0 Not Opposed, 0 Disagree, 1 Absent
>
> Approved By Consensus
>
> Meeting #7, Decision 4.C

Throughout discussions of setting Measurement Quality Objectives, the problem arose of how any Measurement Quality Objective would apply retroactively to methods currently promulgated in 40 CFR Part 136. This recommendation attempted to outline a process that could be applied to existing Part 136 methods that would essentially characterize their performance and use that performance as the basis for establishing Measurement Quality Objectives that would be written into the analytical methods. Although the

Technical Work Group was charged with coming up with a procedure for turning the data that would be collected into Measurement Quality Objectives for the methods, time limitations prevented it from developing the requested procedure.

Decisions on Measurement Quality Objectives for Future Promulgation of Methods

Straw polls indicated that the Committee could not come to consensus on setting fixed Measurement Quality Objectives for quantitation in the context of NPDES permit compliance testing. A proposal was then put forth as a compromise that might be more acceptable to the majority of the Committee. There were several key components to this proposal. First, the scope was limited to future promulgation of methods in Part 136, thus setting aside the difficulties of applying any Measurement Quality Objectives to existing methods. Second, the Measurement Quality Objective would be targets. EPA could still promulgate the target if those Measurement Quality Objectives were not achieved; however, EPA would be required to provide a rationale for why it felt the needs of the Clean Water Act program justified promulgating a method that failed to meet the target. Third, there would be some bounds on the Measurement Quality Objectives where it would not be considered acceptable to promulgate the method in 40 CFR Part 136. This proposal was discussed and voted on in the following two decisions.

> The Committee recommends, for future method promulgation, that target Measurement Quality Objectives for Data Quality Indicators, such as precision, accuracy, Method Specified Qualitative Identification, and false negative error rates derived from the Data Quality Objectives process, be established for Quantitation Limits in Part 136. If the target Measurement Quality Objectives cannot be met, EPA may promulgate with rationale.
>
> *Vote*: 16 Agree, 2 Not Opposed, 1 Disagree
>
> Not Approved
>
> Meeting #8, Decision 2

Majority Report in Support of the Proposal

This proposed recommendation was a compromise to having a fixed set of Measurement Quality Objectives for NPDES permit compliance testing. It is entirely consistent with the Committee's consensus recommendation that EPA should use the decision uncertainty Data Quality Objectives process to establish Measurement Quality Objective goals (not limits). It allows flexibility for the Data Quality Objectives process to determine which Measurement Quality Objectives need to be set and which Data Quality Indicators are appropriate for a specific situation. Furthermore, it does not require EPA to set a single set of Measurement Quality Objectives. EPA could implement the proposed recommendation in a general sense or by allowing issues specific to the substance to be taken into account.

The proposal also acknowledges that there may be some circumstances where, despite EPA's best efforts, it may not be able to achieve the Measurement Quality Objective goals. In these circumstances, EPA would be required to provide a rationale that may

include how it attempted to achieve the goals, what performance it was able to obtain, and why the unique circumstances of the substance and/or threat to human health or the environment may warrant accepting analytical method performance less than the Measurement Quality Objective goals. The essence of the proposal is use of the decision uncertainty Data Quality Objectives process to establish Measurement Quality Objective goals and transparency when those goals cannot be achieved. The proposal was crafted to afford EPA as much flexibility as possible.

Minority Report in Opposition to the Proposal

EPA disagrees with the language that "…. target Measurement Quality Objectives …. be established for Quantitation Limits in Part 136. If the target Measurement Quality Objectives cannot be met, EPA may promulgate with rationale."

This proposed recommendation would establish Measurement Quality Objectives for analytical methods that might be used in a variety of environmental decision-making situations without regard to what decision error might be acceptable. EPA believes that this runs counter to the Data Quality Objectives process currently used by EPA which considers the intended use of an analytical result and examines the extent and nature of the uncertainty that should be allowed. EPA would have the burden to provide a rationale for a method's performance without the benefit of knowing the nature of the environmental decisions to be made with the analytical result. Despite this, EPA does agree that, in making enforcement and compliance determinations, the uncertainty in the data should be taken into account.

Target Measurement Quality Objective Bounds Decision

The Committee recommends that a single set of Measurement Quality Objective bounds be established for promulgated Part 136 methods that define Quantitation for Clean Water Act compliance and enforcement uses.

Vote: 7 Agree, 3 Not Opposed, 8 Disagree, 2 Absent

Not Approved

States: 3 Not Opposed, 1 Disagree
Labs: 3 Disagree, 1 Absent
Industry: 3 Agree, 1 Absent
Public Utilities: 4 Agree
EPA: 1 Disagree
Environmental Community: 3 Disagree
Meeting #10, Decision 7

Majority Report in Support of the Proposal

The need for Measurement Quality Objective bounds and this recommendation grew from a compromise that a majority of Committee members supported, stating that EPA should use the Data Quality Objectives process to set Measurement Quality Objective targets (as opposed to limits) for appropriate Measurement Quality Objectives for

NPDES permitting. If circumstances were such that EPA could not achieve those Measurement Quality Objective targets, it would be acceptable to propose the method, provided that it contained a rationale explaining the compelling need to use a method that failed to meet target Measurement Quality Objectives. However, the subject recommendation suggests, that for NPDES compliance testing, there should be some level of performance below which one could simply not consider the data quantitative and suitable for determining compliance. A majority of Committee members agreed or did not oppose that quantitation bounds should be established in the context of providing a floor and ceiling for Measurement Quality Objectives derived during the Data Quality Objectives process addressing NPDES permit compliance testing. Based on the Committee consensus definition of "Quantitation Limit," detection is stated as one such bound (Quantitation Limit > Detection Limit).

Qualitative identification criteria are also required by several Part 136 methods as a threshold to determine the presence of a specific analyte. A result that meets qualitative identification criteria is expected to pass a higher bar than detection. At quantitation the result must not only be detectable, but the false negative error rate and accuracy (precision and bias) must also be acceptable for the intended use of the data. It is also important that a quantifiable result be repeatable and verifiable in order to base regulatory decisions upon it. A majority of Committee members agreed or were not opposed to clear bounds for quantitation established by EPA for compliance and enforcement. The Committee definition of quantitation is based on the level at which accuracy and precision for the intended purpose are achievable. Presumably these would be criteria determined as target Measurement Quality Objectives during the Data Quality Objectives process.

Minority Report in Opposition to the Proposal

EPA and others disagreed with the proposed recommendation that "a single set of Measurement Quality Objective bounds be established for promulgated Part 136 methods that define Quantitation for Clean Water Act compliance and enforcement uses." EPA disagrees with this version of the bounds language because it would establish a Measurement Quality Objective floor (bound), below which no methods would be allowed to perform without even the off ramp of a rationale. EPA believes that this runs counter to the Data Quality Objectives process currently used by EPA which considers the intended use of an analytical result and examines the extent and nature of the uncertainty that should be allowed. Despite this, EPA does agree that in making enforcement and compliance determination, the uncertainty in the data should be taken into account.

One member of the Laboratory Caucus opposed the establishment of target Measurement Quality Objective bounds under the Clean Water Act because of the "universal" nature of the proposal. The spectrum of data use under the Clean Water Act is so broad that establishing universal bounds would lead to an abundance of instances where the "bounds" would be too broad or not stringent

enough for the intended use of the data. This would lead to data being used that are not of sufficient quality to support its use or the unnecessary rejection of data that does support its intended use. The concept of having "bounds" for objectives also seems to be somewhat of an oxymoron. This member agreed that an assessment of data quality and the Data Quality Objectives process are essential for proper decision-making under the Clean Water Act.

2.4 Measurement Quality Objectives for Clean Water Act Uses

The Committee also considered Measurement Quality Objectives in the broader context of Clean Water Act uses that may go beyond NPDES permitting. In those discussions, the Committee considered an approach that would set outer bounds for Measurement Quality Objectives but could not come to consensus on the specifics. The Committee then considered the following recommendation which, if implemented, would have EPA consider appropriate bounds further and then publish for public comment the Measurement Quality Objective bounds that it determines are appropriate.

Measurement Quality Objective Bounds

> The Committee recommends that EPA establish quantitative Measurement Quality Objective bounds for relevant Data Quality Indicators that define Quantitation for intended Clean Water Act uses. These bounds would be offered for public comment by EPA.
>
> *Vote*: 9 Agree, 7 Not Opposed, 1 Disagree, 1 Absent
>
> Not Approved
>
> Meeting #7, Decision 4.E

Majority Report in Support of the Proposal

A majority of Committee members agreed with or were not opposed to the general concept that there should be some outside boundary for Measurement Quality Objectives or Data Quality Indicators beyond which a method may not be suitable for a particular purpose. However, the Committee did not agree on specific values for Measurement Quality Objective bounds or even that universal bounds for all different Clean Water Act uses were appropriate.

However, just because the Committee could not resolve these questions within the time available does not imply that the Committee did not think these questions were not worth addressing. The proposed recommendation was intended to convey that sentiment and to encourage EPA to continue to try to find an acceptable process for establishing Measurement Quality Objective bounds for Clean Water Act purposes. The proposed approach does not imply any constraints on how this might best be accomplished and it does not imply any universal, fixed Measurement Quality Objective bounds. Because of the issues raised by Committee members during the discussions, the proposed recommendation goes on to indicate that EPA should present, for public comment, the

results of its final determinations with regard to the question of Measurement Quality Objective bounds for Clean Water Act programs

Minority Report in Opposition to the Proposal

EPA disagrees with the proposed recommendation that "a quantitative Measurement Quality Objective bounds be established" for the reasons described in our reply to the "*Target Measurement Quality Objectives*" decision (Meeting #8, Decision 2.) Under this approach, EPA would still have to provide a rationale for these bounds without knowing what type of environmental decision would be made with the analytical results. This runs counter to the Data Quality Objectives process currently used by EPA which considers the intended use of an analytical result and examines the extent and nature of the uncertainty that should be allowed. Despite this, EPA does agree that in making enforcement and compliance determination, the uncertainty in the data should be taken into account.

CHAPTER 3 – PROCEDURES FOR DETECTION AND QUANTITATION

3.1 Introduction

The principal charge to the Committee was to develop recommendations on approaches for determining Detection Limits and Quantitation Limits and their uses in Clean Water Act programs. After two and one-half years of Committee and Work Group activities involving deliberations, design and assessment of a Pilot Study, and production of numerous working documents, Committee members developed a clear understanding of the complexity of the scientific, science-policy, and policy issues involved with low-level analytical measurements in support of Clean Water Act programs. A central challenge confronting the Committee (and thus EPA) was in developing the framework for a program involving detection and quantitation that is both technically/statistically rigorous while being able to be practically implemented by regulatory agencies, regulated entities and laboratories, all within the broad purview of the Clean Water Act.

The Committee discussed three basic types of procedures for determining Detection and Quantitation Limits. Although a formal definition was never adopted, the Committee had extensive discussions regarding what was termed a single laboratory procedure. This is a procedure which is performed by a laboratory to determine the laboratory- specific Detection and Quantitation Limits. The second type of procedure the Committee discussed was an inter-laboratory procedure. The Committee added to the Glossary a definition for what constitutes an inter-laboratory procedure.[3] In simple terms, such a procedure involves distributing identical samples to multiple laboratories for analysis and then using the resulting data to calculate a single Detection and/or Quantitation Limit representative of the participating laboratories. The final type of procedure discussed was a multi-laboratory procedure. The Committee also added to the Glossary a definition for what constitutes a multi-laboratory procedure.[4] Such a procedure involves the pooling of single laboratory estimates of detection and/or quantitation to calculate a multi-laboratory estimate of the detection and/or quantitation capabilities of the

[3] The definition of an inter-laboratory procedure added to the Glossary by the Committee is as follows: A study where a centralized study design coordinator sends identical samples to multiple different laboratories for analysis. The resulting raw data are analyzed by the study design coordinator by a given procedure to provide estimates of L_C, L_D and/or L_Q. The laboratories would generate only data that would be submitted to the study design coordinator who would compile the data, evaluate it and generate an inter-laboratory L_C, L_D and/or L_Q.

[4] The definition of multi-laboratory procedure added to the Glossary by the Committee is as follows: A study where multiple laboratories individually perform a L_C, L_D and/or L_Q estimation procedure (usually using self selected spiking concentrations) and those individual estimates are summarized in some fashion (e.g. averaging, upper or lower confidence intervals) to characterize some measure of how well the analytical method performs in qualified laboratories. The multi-lab procedure study would include two steps. First, each individual lab would conduct the analysis and generate its unique L_C, L_D and/or L_Q level. Second, those levels would then be compiled from all laboratories, evaluated, and, based on criteria, used to propose multi-lab L_C, L_D and/or L_Q levels, where appropriate.

laboratories. Multi-laboratory or inter-laboratory procedures would be used to develop National Detection and Quantitation Limits.

During the deliberations of the Committee, the members adopted the convention of referring to analytical methods as "methods" and procedures for determining a Detection or Quantitation Limit as "procedures." This report continues that convention.

3.2 What The Committee Needs A Procedure To Do

Over the course of multiple Committee meetings, the Committee developed and agreed to the document, "What We Need A Procedure To Do." (See Appendix B.) This document contains 15 objectives, initially developed for use in the Pilot Study, to evaluate how well the procedures tested met the objectives.

Committee members also generally agreed that the pilot test was an opportunity to inform the Committee's final recommendations and that some of the objectives might be refined as a result of the Pilot Study data.

The 15 objectives of the document "What We Need A Procedure To Do" follow. The term "limit" is used generally to refer to Detection and Quantitation Limits since the Committee had not yet defined them:

1. Does the procedure provide an explicit estimate of bias at L_Q for limits that must be verifiable by labs at those limits?
2. Does the procedure provide an explicit estimate of precision at L_Q for limits that must be verifiable by labs at those limits?
3. Does the procedure provide an explicit false positive rate for L_C?
4. Does the procedure provide an explicit false negative rate at L_C for the true value at L_D or L_Q that must be observed in labs at L_C for the estimated values of L_D or L_Q?
5. Does the procedure provide that qualitative identification criteria defined in the analytical method are met at the determined Detection and Quantitation Limits?
6. Does the procedure adequately represent routine variability in laboratory performance?
7. Does the procedure perform on-going verification of estimates?
8. Is the procedure capable of calculating limits using matrices other than laboratory reagent grade water?
9. Does the procedure use only data that results from test methods conducted in their entirety?
10. Does the procedure explicitly adjust or account for situations where method blanks always return a non-zero result/response?
11. Does the procedure explicitly adjust or account for situations where method blanks are intermittently contaminated?

12. Is the procedure clearly written with enough detail so most users can understand and implement it?
13. Is the procedure cost-effective?
14. Does the procedure assess multi and inter-laboratory variability when data from more than one laboratory is used?
15. Is the procedure applicable to all users and test methods?

As part of the decision-making process, the procedures tested in the Pilot Study were subsequently evaluated according to how well they met the 15 objectives.

3.3 Additional Requirements Based on Contemplated Uses of Detection and Quantitation in Clean Water Act Programs

As the discussion of uses of detection and quantitation in Clean Water Act programs developed, other potential requirements for the single laboratory procedure became apparent. The requirements were not clear during the deliberations, but when the final Clean Water Act detection and quantitation use recommendations were identified, there were two requirements that the single laboratory procedure needed to meet.

One was to determine the lowest possible concentration that a laboratory could detect and/or quantify a substance. The other was to demonstrate that a Laboratory Quantitation Limit was below the Permit Quantitation Limit or other applicable limit.

Additional details of how these requirements fit into the overall NPDES permitting strategy developed by the Committee can be found in Chapter 4.

3.4 Pilot Study Design

The process proposed for the Committee's work included pilot testing any procedure/s recommended by the Committee to confirm that the procedure/s performed as expected before the Committee completed its recommendation on one or more procedures. However, a number of Committee members expressed concern over how they could decide among the candidate procedures without data on performance. The Committee decided to pilot test several candidate procedures to help inform its decision-making. Thus, the Committee selected procedures to pilot test, developed a study design for the Pilot Study and, to the extent possible within strict budget and time constraints, verified the performance of candidate detection and quantitation procedures.

3.5 Pilot Study

A total of 104 analytes were included in the Pilot Study and, of that dataset, 55 were evaluated during the assessment portion of the study.

The Committee affirmatively agreed to pilot test the following EPA-approved methods:

- 200.7 (Determination of Metals and Elements in Waters and Wastes by Inductively Coupled Plasma-Atomic Emission Spectroscopy),
- 300.0 (Determination of Inorganic Ions by Ion Chromatography - Method A),
- 625 (Base Neutrals and Acids by GC/MS),
- 608 (Organochlorine Pesticides and PCBs by GC/ECD), and
- 335.4 (Total Cyanide by Semi-automated Colorimetry).

These methods were selected to represent a cross section of measurement technologies that appear in 40 CFR Part 136 and provide a good test of the performance of the procedures.

To begin the process of recommending procedure/s for determining Detection and Quantitation Limits of an analytical procedure, the Committee charged the Technical Work Group with compiling a list of candidate procedures. The Technical Work Group used the framework provided by the document, "What We Need A Procedure To Do," to select procedures for further consideration. The resulting list of procedures is shown in Table 1 below. This list of procedures includes single laboratory procedures, inter-laboratory procedures, and procedures that, although written as single laboratory or inter-laboratory, could be easily modified and implemented as either single laboratory or inter-laboratory procedures. No multi-laboratory procedures were proposed. After reviewing the initial list, the Committee asked the Technical Work Group to narrow the list. The Technical Work Group accomplished this task by identifying candidate procedures that were more conceptual in nature and thus lacked a specific written procedure to implement them. These procedures, shown at the bottom of Table 1, were dropped from further consideration.

Table 1 Summary of Detection and Quantitation Procedures Considered by the Committee

Procedures	Detection	Quantitation	Pilot Tested
American Council of Independent Laboratories (ACIL) Proposed Procedures for Determining the Method Detection Limit and Quantitation Limit (ACIL procedure)	X	X	X
Proposed Procedures for Estimating the Limit of Detection, Consensus Group Committee I on Detection for Proposal to USEPA for Replacement of 40 CFR Part 136, Appendix B MDL Procedure (Consensus Group procedure)	X	X	
Determination of Detection Limits Using Laboratory QC, East Bay Municipal Utilities District (Laboratory QC procedure)	X		
Hubaux-Vos Detection Limit Procedure	X		X[5]
ASTM Interlaboratory Detection Estimate (IDE)	X		X[5]
EPA MDL, 40 CFR Part 136, Appendix B	X		
ASTM Interlaboratory Quantitation Estimate (IQE)		X	X[5]
EPA OGWDW Lowest Concentration-Minimum Reporting Level (LC-MRL) for Quantitation		X	X[5]
EPA Minimum Level		X	
Procedures Dropped from Further Consideration			
Water Research Centre Determination and Quantitation	X		
ISO/IUPAC	X	X	
IIAG Sensitivity Test & Full - Range Validation Study	X		
Office of Solid Waste (OSW) Quantitation Limit		X	
NELAC Uncertainty Calculations			
USGS LT-MDL			

The Committee decided to require the same Measurement Quality Objective targets for every chemical and analytical method studied, as most of the procedures allowed for some flexibility in selection of different Measurement Quality Objectives. These tests, performed over several weeks, used blanks and spiked samples that may have encompassed several different concentrations of the target analyte. The Measurement Quality Objectives recommended by the Technical Work Group and approved by the Committee for the Pilot Study were 20% RSD, 50% to 150% mean recovery range, and false positive and false negative rates of ≤ 1%.

[5] Procedures were pilot tested as both single laboratory and inter-laboratory procedures.

There was considerable discussion over whether to pilot test the ACIL procedure or the Consensus Group procedure. The procedures are very similar in many respects. The decision was made to pilot test the ACIL procedure with modifications based on the Consensus Group procedure, such as specifying the use of *K* instead of student *t* for censored methods. The ACIL procedure was further modified by changing some of the specified Measurement Quality Objectives in the procedure to match those selected for the Pilot Study. The ≤ 1% false positive criterion was already implemented in the ACIL procedure. The recovery criterion was changed to a mean of 50-150%; the standard deviation of spikes at the Quantitation Limit had to be < 20%; and the Quantitation Limit had to be at least a factor of two times the Detection Limit. These changes were implemented in Revision 5 of the ACIL procedure.

The Technical Work Group recommended and the Committee approved pilot testing of the five procedures noted in the last column of Table 1:

- American Council of Independent Laboratories (ACIL) Proposed Procedures for Determining the Method Detection Limit and Quantitation Limit, Revision 5
- Hubaux-Vos Detection Limit Procedure
- ASTM Interlaboratory Detection Estimate (IDE)
- ASTM Interlaboratory Quantitation Estimate (IQE)
- EPA OGWDW Lowest Concentration-Minimum Reporting Level (LC-MRL) for Quantitation

3.6 Committee Decision-Making Process on a Single Laboratory Detection and Quantitation Procedure

At the completion of the Pilot Study, the Committee determined that the ACIL single laboratory procedure performed as well as or better than the other procedures and met most of the objectives in the document, "What We Need A Procedure To Do." The Committee directed the Technical Work Group to further modify the ACIL procedure as indicated by the Pilot Study results and informed by concepts from the Consensus Group procedure and the Laboratory QC procedure for single laboratory uses. A sub-group of the Technical Work Group implemented that charge, resulting in the DQ FAC Single Laboratory Procedure v2.4, which was then considered by the Committee.

At the September 19-21, 2007 meeting, a straw poll of the Committee regarding a recommendation that EPA adopt the DQ FAC Single Laboratory Procedure v2.4 indicated that several Committee members had issues they needed to resolve before they could support the recommendation. These issues related to several questions about verification frequency (both with respect to the frequency of blank or spike sample analyses as well as the frequency that the Detection or Quantitation Limits are evaluated with respect to the blank or sample analyses), a change from mandatory to optional re-calculations, and providing a batch specific alternative for small laboratories that do not have Laboratory Information Management Systems. To optimize the probability of reaching consensus on a single laboratory procedure recommendation, the Committee first attempted to find an acceptable resolution to these concerns and/or possible revisions to the DQ FAC Single Laboratory Procedure v2.4 before bringing it to a vote. The

discussions regarding efforts to reach resolution of these issues are summarized in the next section followed by the final decision on the single laboratory procedure.

3.7 Recommendations and Decisions on Procedure Verification

Quantitation Limit Verification Frequency Decision

The DQ FAC Single Laboratory Procedure v2.4 procedure (and its ACIL predecessor) both had provisions for some level of verification. However, it was always understood that these provisions could be changed to whatever frequency the Committee agreed upon. Concurrent with the development of the DQ FAC Single Laboratory Procedure v2.4, the Policy Work Group discussed an appropriate verification frequency with a keen awareness of the need to maintain a balance between rigor and practicality, while recognizing that important regulatory decisions will be made based in part on the reliability of estimates of detection and quantitation. While the Policy Work Group did not come up with specific recommendations, Committee members agreed this issue needed to be addressed before voting on a single laboratory procedure. Thus, the Committee discussed and considered the following recommendation.

The Committee recommends that the following be adopted into the DQ FAC Single Lab Procedure v2.4:

Section 2.10 of the ACIL procedure specifies monthly Quantitation Limit verification spikes, evaluated on a quarterly basis. Section 2.2 of revised ACIL procedure specifies a minimum of quarterly Quantitation Limit verification spikes, evaluated on an annual basis. If we went to monthly Quantitation Limit verification spikes, evaluated annually this would provide a minimum of 24 Quantitation Limit spikes over a two year period to generate the long term estimate:

2.2 Continue to collect method blanks with each batch from which data were reported and Quantitation Limit spikes for every analyte ***analyzed at least monthly*** *(or four per 12 month period in separate batches spread across the time period during which analysis is conducted)* ***which ever is greater****. If multiple instruments are to be used for reporting data with the same Detection Limit and Quantitation Limit,* ***analyze two to six*** *Quantitation Limit spikes per instrument per twelve month period,* ***so that a minimum of 12*** *Quantitation Limit* ***spikes are generated each year.***

2.2.1. Evaluate your Detection Limits and Quantitation Limits at least every year using all of the spikes available in a 24 month period using the procedures described in the Sections below. All method blanks and Quantitation Limit spikes collected within a ***24*** *month period should be used for reassessing Detection Limits and Quantitation Limits, unless there is reason to believe that the Detection Limit or Quantitation Limit changed substantially at some point during that* ***24*** *month period. In that case the most recent data may be used for the reassessment, but not less than 20 method blanks and seven Quantitation Limit spikes per instrument.*

Note: Proposed language changes shown as **Boldface – Underline**

Vote: 4 Agree, 5 Not Opposed, 11 Disagree

Not Approved

States: 4 Disagree
Labs: 4 Disagree
Industry: 4 Agree
Public Utilities: 4 Not Opposed
EPA: 1 Disagree
Environmental Community: 1 Not Opposed, 2 Disagree
Meeting #10, Decision 6.E

Majority Report in Opposition to the Proposal

One State Caucus member and EPA believe that the frequency of verification specified in the DQ FAC Single Laboratory Procedure v2.4 represents a balance between rigor and cost. The proposed language changes (despite their intent) appear to require monthly verification analyses, regardless of how frequently laboratories perform analyses on actual samples. In addition, for multi-component analyses requiring the preparation of a

variety of verification samples (due to incompatibility of mixtures or concentration ranges) to evaluate the entire spectrum of analytes measured, costs of monthly verification testing could outweigh any benefits gained by generating a larger evaluation data set. It would be prudent to perform a cost-benefit analysis prior to requiring more frequent verification than originally specified in Version 2.4.

Minority Report in Support of the Proposal

One of the key criticisms of the 40 CFR Part 136, Appendix B MDL procedure has been that it was developed over idealized conditions (e.g., short -term, most likely with all laboratory procedures and instrumentation optimized for peak performance). The same criticism could be applied to laboratory accreditation proficiency testing, which is often done quarterly. By setting the verification equivalent to the common frequency of proficiency testing, it is highly likely that the verification will also be done under idealized conditions. One of the features the Committee caucuses agreed upon was the need for Detection Limit and Quantitation Limit estimates that reflect normal, routine operations. Increasing testing frequency to monthly would assure that laboratories could not run the verifications from idealized, non-routine conditions. Furthermore, in the third and subsequent years, quarterly testing would limit the Detection Limit and Quantitation Limit labs to eight measurements (e.g., quarterly testing for the last two years). Thus, although this would incorporate long-term variability (note this would only be true if the Detection Limit were recalculated annually, see next recommendation), the number of data points going into the estimate would only be minimally greater that the required seven replicates currently specified in the 40 CFR Part 136, Appendix B MDL procedure. Monthly testing would increase the number of replicates to 24, which would provide a much more robust estimate of the Detection Limit and Quantitation Limit.

Quantitation Limit Verification Frequency Recommendation

Because the previous proposal was not approved by consensus, the Committee considered a more general recommendation asking EPA to give this subject additional consideration and to publish its findings in the *Federal Register* for public review and comment.

The Committee recommends that EPA give additional consideration to increasing the frequency of Quantitation Limit verification and report its findings in the preamble of the *Federal Register* Notice and request specific comments on the final proposed frequency.

Vote: 11 Agree, 9 Not Opposed, 0 Disagree
Approved By Consensus

States: 1 Agree, 3 Not Opposed
Labs: 4 Not Opposed
Industry: 4 Agree
Public Utilities: 3 Agree, 1 Not Opposed
EPA: 1 Not Opposed
Environmental Community: 2 Agree, 1 Not Opposed
Meeting #10, Decision 6.F

The Committee discussed but could not come to consensus on the appropriate frequency for verification as evidenced in the majority/minority decisions and opinions described above. However, the Committee did come to consensus on the recommendation that EPA should give additional consideration to the appropriate frequency for verification of Detection Limits and Quantitation Limits and that it should specifically discuss the results of its deliberations in the preamble of the *Federal Register* Notice where the final procedure/s are proposed.

The Committee recommends that, as EPA considers the appropriate level of verification, it maintain a balance between rigor and practicality, while recognizing that important regulatory decisions will be made based in part on the reliability of estimates of detection and quantitation.

EPA may address specific issues/components of verification, including such aspects as:

- The details of how verification would be carried out,
- Steps for validation of initial Detection Limit and Quantitation Limit values (and indication of when new limits should be obtained – e.g., major changes to an instrument) as well as steps for verifying those limits on an ongoing basis,
- Description of the frequency of steps undertaken in the ongoing verification process (e.g., number of samples over a given period), and
- Implications of failure to meet verification criteria (e.g., invalidation of a set of samples run over a particular period).

Optional Batch Specific Verification Decision

One caucus expressed concerns over the resource burden that adoption of the DQ FAC Single Laboratory Procedure v2.4 would impose on small laboratories that do not have Laboratory Information Management Systems. To remedy this problem, they asked the Committee to consider an optional batch specific verification approach to be incorporated into the single laboratory procedure.

The Committee recommends that the following language be moved into the DQ FAC Single Lab Procedure v2.4:

Blanks and Quantitation Limit spikes in each batch

a. If the method blank exceeds the Detection Limit and a cause cannot be identified, raise the Detection Limit to the blank result for future analysis.
b. If the Quantitation Limit spike result (or Quantitation Limit spike times Quantitation Limit/spike level, if not spiking exactly at the Quantitation Limit) is less than the Detection Limit, elevate the Quantitation Limit by a factor of two and repeat the Quantitation Limit spike at the new Quantitation Limit. Repeat this until the Quantitation Limit spike is at or above the Detection Limit.
c. If the Quantitation Limit spike result is outside the average specified accuracy, elevate the Quantitation Limit by a factor of two and repeat the Quantitation Limit spike at the new Quantitation Limit. Repeat this until the Quantitation Limit spike meets the specified accuracy criteria.

Vote: 4 Agree, 9 Not Opposed, 7 Disagree
Not Approved

States: 4 Disagree
Labs: 3 Not Opposed, 1 Disagree
Industry: 4 Not Opposed
Public Utilities: 4 Agree
EPA: 1 Disagree
Environmental Community: 2 Not Opposed, 1 Disagree
Meeting #10, Decision 6.C

Majority Report in Support of the Proposal

The Industry Caucus and one member of the Laboratory Caucus do not oppose batch specific verification where appropriate. Although a majority of industry NPDES Clean Water Act related analyses are performed by larger commercial laboratories, many are performed by small labs that do not have Laboratory Information Management Systems. Therefore, the Industry Caucus supports an option to allow batch verification to reduce record-keeping requirements as long as false positive and false negative error rates are adequately controlled and the regulatory requirement would permit a high detection or quantitation level due to the implementation of, essentially, more stringent Measurement Quality Objectives for false positives and accuracy. The proposed specific batch verification procedure would meet these criteria.

The Public Utility Caucus recognizes that the proposed procedure is designed to predict a 1% false positive rate at the Detection Limit when results from unspiked blanks are normally distributed. However, this Measurement Quality Objective may not be clearly met when the method does not produce numeric results or numeric results are non-normally distributed. Another issue to consider is that the vast majority of laboratories analyzing samples for Clean Water Act compliance are small.

These are usually "in-house" laboratories that perform process control testing for the discharger, e.g. dairies, sugar refineries, power plants, military bases, and public utilities, and generally only perform tests for their own facility. Such laboratories may only produce a few unspiked blanks per batch and may only run one batch on a monthly or quarterly basis. This means that it could take many years to accumulate enough unspiked blank data to determine if the laboratory were actually achieving the intended Measurement Quality Objective for the Detection Limit. The data requirements for the procedure may also create data storage and retrieval system requirements for these laboratories that otherwise would not be required. Laboratory Reagent Blanks for all method-analyte combinations would have to be stored and then periodically reviewed.

The Public Utility Caucus also supports a proposal to allow laboratories to have two options for on-going verification. One option is to use the currently proposed procedure of storing Laboratory Reagent Blank and Laboratory Fortified Blank results. These laboratories would need to comply with Measurement Quality Objectives of an average 1% false positive rate (i.e., a result greater than the Detection Limit for Laboratory Reagent Blanks and some average recovery and precision for Laboratory Fortified Blanks, as yet unspecified). The other option is for a laboratory to comply with a more stringent set of Measurement Quality Objectives on a batch by batch basis. These laboratories would meet a 0% false positive rate for Laboratory Reagent Blanks, i.e., all Laboratory Reagent Blanks would be less than the Detection Limit for a given batch. These laboratories would need to run a single Laboratory Fortified Blank at or below the Quantitation Limit (but not above) with each batch and get a recovery within the Measurement Quality Objectives set at some future date. However, it would not be an average recovery over several batches but recovery for that single Laboratory Fortified Blank and batch. If the Measurement Quality Objective for average recovery is ±50%, the Measurement Quality Objective for the single batch would be ±50%.

The Public Utility Caucus and one member of the Laboratory Caucus believe that when the batch specific Measurement Quality Objectives are not met, corrective actions need to be taken and that the actions listed in a. b. and c. of the decision are appropriate for that purpose.

Minority Report in Opposition to the Proposal

EPA and one State Caucus member noted that the DQ FAC Single Laboratory Procedure v2.4 incorporated, at the request of the Committee, provisions to allow assessment and verification of precision and accuracy at the Quantitation Limit, should Measurement Quality Objectives for those Data Quality Indicators be developed. The proposed change to the procedure did not specify how precision could be assessed or verified at the Quantitation Limit based on available batch data only. While it may be reasonable to allow provision for batch-only verification for laboratories that do not have access to a database, the details of how to verify precision and accuracy requirements may need further refinement.

One Environmental Community Caucus representative was concerned about ambiguities in how vigorous laboratories would need to be in attempting to identify causes for a method blank exceeding a Detection Limit. A single contamination incident producing a single high blank value (or set of blanks in one or more batches) could potentially lead to establishment of a Detection Limit level that might be significantly above a level that could easily be achieved in many subsequent analyses, with sufficient attention to practices to minimize blank contamination. Because the proposed approach for addressing Detection Limit would also have implications for Quantitation Limit (i.e., raising it in cases where the Quantitation Limit spike result is less than Detection Limit), it would seem the overall approach could easily have a tendency to lead to ever-increasing Detection and Quantitation Limits, without sufficient incentive to identify and remedy causes of high blanks.

Batch Verification Recommendation

Although a specific recommendation could not be reached by consensus, the Committee did feel the concern warranted further consideration and thus proposed the following recommendation:

> The Committee recommends that during promulgation, EPA include and/or develop language to incorporate batch specific verification as an option in the procedure.
>
> *Vote*: 16 Agree, 4 Not Opposed, 0 Disagree
> Approved By Consensus
>
> *States:* 3 Agree, 1 Not Opposed
> *Labs:* 2 Agree, 2 Not Opposed
> *Industry:* 4 Agree
> *Public Utilities:* 4 Agree
> *EPA:* 1 Not Opposed
> *Environmental Community:* 3 Agree
> Meeting #10, Decision 6.D

Although the Committee could not come to consensus on how batch verification should be incorporated into the single laboratory procedure, it did agree that EPA should develop this concept further and incorporate it into the final procedure it proposes.

Detection Limit Verification and Recalculation Decision

Revision 5 of the ACIL procedure indicated that the laboratory was required to re-calculate its Detection Limit annually using the additional data generated during the year. This was changed in the DQ FAC Single Laboratory Procedure v2.4 to be optional. Because this change concerned some Committee members, the following recommendation was discussed and considered by the Committee.

The Committee recommends that the following be adopted into the DQ FAC Single Laboratory Procedure v2.4:
Section 1.9 of the ACIL procedure specifies annual recalculation of Detection Limit and then uses an F test to determine if the Detection Limit should be revised. Section 2.2.2 (now 2.4) of the DQ FAC Single Laboratory Procedure v2.4 allows optional recalculation of the Detection Limit, with no decision criteria provided. By making the recalculation of the Detection Limit optional it is possible that the false positive error rate using the parametric statistical test could be greater than 1%.

2.2.2 ***Recalculate*** *the Detection Limit using the formulas in 1.1.7. or 1.2.7.*

Note: Proposed language change shown as **Boldface –** Underline

Vote: 8 Agree, 10 Not Opposed, 2 Disagree

Not Approved

States: 1 Agree, 2 Not Opposed, 1 Disagree
Labs: 4 Not Opposed
Industry: 4 Agree
Public Utilities: 3 Agree, 1 Not Opposed
EPA: 1 Disagree
Environmental Community: 3 Not Opposed
Meeting #10, Decision 6.G

Majority Report in Support of the Proposal

Section 1.9 of the ACIL procedure stipulated an annual recalculation and reevaluation of the Detection Limit. This required use not only of the initial estimate data (collected over a relatively short period of time) but also of the subsequent quarterly data (censored methods) or all blank data (non-censored methods) that clearly represent more long-term, routine performance. One of the criticisms of the 40 CFR Part 136, Appendix B MDL procedure was that it reflected only extremely short-term performance. Nothing was learned in the Pilot Study to justify dropping the recalculation requirement. If the requirement is dropped, the Laboratory Detection Limit would be marginally better than the MDL because the laboratory would not be required to use any data beyond that used for the initial short-term estimate. If laboratory performance of the method over time changed (becoming better or worse), the Laboratory Detection Limit would not reflect the laboratory's current capability unless there were a mandatory (at least) annual recalculation using all available information.

In the DQ FAC Single Laboratory Procedure v2.4, the primary control of the false positive error rate (target ≤ 1%) is parametric calculation of standard deviation times a constant, performed during the initial calculation and annual recalculations of the Detection Limit and Quantitation Limit. The non-parametric test is intended to catch intermittent blank outliers that may fall outside of the parametric tolerance or confidence intervals. Because the intermittent blank check is set at the 5% level, it is possible that a false positive error rate of between 1% and 5% can occur if the annual parametric recalculation is not performed prior to applying the non-parametric test.

Minority Report in Opposition to the Proposal

The DQ FAC Single Laboratory Procedure v2.4 was refined and tested over the course of several months by a team from the Technical Work Group. Version 2.4 represents a careful balance of many factors, including rigor, cost effectiveness, practicality and function. EPA and one State Caucus member were concerned that there was no discussion of changing the wording in Section 2.2.2 of the v2.4 procedure amongst the Technical Work Group prior to the 10th Committee meeting, nor was there any justification presented at the meeting for doing so. At the very least, the rationale for the suggested change should have been presented along with an assessment or discussion of the ramifications associated with making recalculation of the Detection Limit mandatory every time verification is performed.

3.8 Decision on a Single Laboratory Procedure

Single Laboratory-Determined Detection and Quantitation Limit Decision

After trying to address the issues related to verification through the proposals discussed above, the Committee turned to a discussion and vote on the single laboratory procedure recommendation with those resolutions in mind.

The Committee recommends that EPA promulgate the DQ FAC Single Laboratory Procedure v2.4 recommended by the Committee for individual laboratories to determine their Detection and Quantitation Limits. The DQ FAC Single Laboratory Procedure v2.4 shall be used instead of the current MDL procedure in 40 CFR Part 136, Appendix B, for calculating all future Laboratory Detection and Quantitation Limits. The DQ FAC Single Laboratory Procedure v2.4 has the following two capabilities:

- Demonstrates the laboratories performance at a specified level.
- Determines the lowest possible value achievable by the laboratory while meeting the Measurement Quality Objectives.

Vote: 14 Agree, 1 Not Opposed, 5 Disagree
Not Approved

States: 4 Agree
Labs: 3 Agree, 1 Disagree
Industry: 4 Agree
Public Utilities: 2 Agree, 1 Not Opposed, 1 Disagree
EPA: 1 Disagree
Environmental Community: 1 Agree, 2 Disagree
Meeting #10, Decision 6.A

Majority Report in Support of the Proposal

The original ACIL procedure was modified prior to the Pilot Study to incorporate Pilot Study Measurement Quality Objectives for precision, bias and false negative protection.

It was also modified slightly to take advantage of some of the strengths of the Consensus Group Detection Limit procedure, which was similar to the ACIL procedure in many ways, so that both procedures would not need to be included in the Pilot Study. The most substantial modification was using a "*K*" factor in place of a student "*t*" factor for calculation of the uncensored Detection Limit. The ACIL procedure, with modifications indicated by the Pilot Study results and informed by concepts from the Consensus Group procedure and the Laboratory QC procedure, was recommended for a single laboratory Detection/Quantitation Limit procedure, (DQ FAC Single Laboratory Procedure v2.4). These modifications included Measurement Quality Objective flexibility while maintaining false negative protection, an optional procedure for the determination of the "lowest possible Quantitation Limit," and a procedure to protect against intermittent blank contamination.

A majority of the Committee voted in favor of EPA adopting the modified ACIL Single Laboratory Detection/Quantitation Limit procedure (DQ FAC Single Laboratory Procedure v2.4) to replace the 40 CFR Part 136, Appendix B (MDL) procedure and the minimum level (ML), because of its superior performance. The ACIL procedure, as demonstrated in the Committee Pilot Study, achieves or addresses all of the criteria that the Committee identified as critical for a single laboratory detection and quantitation procedure. The resulting DQ FAC Single Laboratory Procedure v2.4 is robust and achieves all of the Committee's objectives for a single laboratory procedure.

Procedure Performance

Overall, the ACIL procedure performed better in terms of achieving targeted false positive and false negative rates than other procedures under consideration in the Pilot Study. Some weaknesses of the procedure were identified, and a work group made several modifications to the procedure to address these weaknesses. As a result, the modified ACIL procedure (DQ FAC Single Laboratory Procedure v2.4) is stronger in the way that verification is performed and in the handling of non-normal data and intermittent blank contamination issues.

Comparison with "What We Need A Procedure To Do"

Early in the Committee process, the Committee identified a number of properties that a successful detection/quantitation procedure should have. These criteria were identified in the document, "What We Need A Procedure To Do." The modified ACIL procedure (DQ FAC Single Laboratory Procedure v2.4) addresses all of these criteria, except for determination of inter-laboratory Detection Limits, which is, of course, not required for a single laboratory procedure. (The modified ACIL procedure can be applied on a multi-laboratory basis.) In particular, the modified ACIL procedure (DQ FAC Single Laboratory Procedure v2.4) addresses those criteria that are not met by the current MDL procedure. These weaknesses of the current MDL, which were the primary reason for the formation of the Committee, include failure to provide explicit estimates for precision and bias at the Quantitation Limit; lack of verification of false positive and false negative rates; lack of requirement to meet qualitative identification criteria defined in the

analytical method; failure to incorporate routine variability; and failure to address situations where blanks have a non-zero response.

Ease of Adoption

The modified ACIL procedure (DQ FAC Single Laboratory Procedure v2.4) has some similarities to the current MDL that should result in easy adoption. In particular, the startup determination involves method blanks which laboratories will already have for most method/analyte combinations as well as spikes at or below the proposed Quantitation Limit which laboratories will also have from their existing MDL studies. It is important to recognize that for uncensored methods, laboratories will be able to define and calculate Detection and Quantitation Limits using the modified ACIL procedure without any need for additional analytical work. For censored methods, laboratories' existing MDL data can normally be used for the initial estimate of the Detection Limit.

The modified ACIL procedure is also similar in key respects to the drinking water MRL procedure. Analytical work that has been performed to determine a MRL will also suffice to define the ACIL procedure Quantitation Limit. Conversely, work done for a startup ACIL procedure will suffice for a MRL, if the performance of the method is adequate.

Measurement Quality Objectives for relevant Data Quality Indicators, such as precision, bias, false positive and false negative error rates must be established to achieve the objectives of the Committee. The modified ACIL procedure (DQ FAC Single Laboratory Procedure v2.4) is designed to achieve these and provides flexibility in that different specifications for precision and accuracy are easily accommodated for methods and/or analytes with differing performance.

Additional Considerations

- The procedure is written in such a way as to allow an adequate Quantitation Limit to be derived which meets laboratory, user and regulatory needs without excessive costs. If a lowest possible Quantitation Limit needs to be developed for a particular need (at an additional expense), a provision has been included in section 1.2.2.1 to allow for this.
- Adequate space is maintained between the Detection Limit and Quantitation Limit to protect against false negative errors (i.e., saying that an analyte is absent when it is actually present). The more precise and accurate the method, the narrower the gap between the Detection Limit and Quantitation Limit. This provision allows a wide range of Measurement Quality Objectives for precision and bias (Measurement Quality Objective flexibility), while still protecting against false negative errors. False negative protection at about the 5% level is targeted, which is akin to the IUPAC L_D.
- Recommendations regarding reduction of laboratory contamination are incorporated into the procedure. Laboratories with lower levels of laboratory contamination will be able to achieve lower Detection Limits and Quantitation

Limits, thus allowing market forces to drive them to reduce the level of cross contamination in the laboratory.

- The procedure was also designed to generate realistic Detection Limit and Quantitation Limit estimates based on routine laboratory performance. This had been one of the major criticisms of the MDL and was the primary reason why the USGS developed the Long- Term MDL (LT-MDL). The single laboratory procedure has been designed specifically to produce long- term estimates with periodic verification of those estimates, to assure that the Detection Limit and Quantitation Limit estimates represent the routine performance of the laboratory.
- Initial estimates for Detection Limit for uncensored methods are based on a "*K*" factor (tolerance interval) as opposed to a student *t* factor (confidence interval) to provide a better estimate of long -term variability using short- term data. Once long -term data are collected, the *K* factor is no longer needed. The use of *K* over *t* was decided because the Pilot Study data and long -term data sets indicated that it provided a better estimate of long -term variability and did a superior job in achieving the Committee objective of ≤ 1% false positive error rate at the Detection Limit.
  - The Committee Pilot Study report concluded that the modified ACIL procedure using a *K* factor to derive the Detection Limit for uncensored methods did the best job of achieving the targeted false negative error rate of 1% or less.
  - Historical blank data from method 200.7 for 27 metals yielded a long -term false positive error rate of approximately 2% when using a student *t* factor to determine the short -term estimate (n = 7) vs. a 1.2% false positive error rate when using a *K* factor.

Minority Report in Opposition to the Proposal

EPA supports most of the elements of the new single laboratory procedure for detection and quantitation, however, EPA has two principal concerns:

1. Student *t* vs. *K* Factor

   The student *t* factor is used throughout the procedure for detection and quantitation calculations except when uncensored methods are at issue, such as trace metals analyses. If a *K* factor is used, values can be as much as 94% larger than if a student *t* factor is used for seven samples. This higher multiplier would result in higher Detection Limits, which would decrease the ability to detect the analyte of interest and therefore increase the rate of false negatives. For uncensored methods, the majority believes a *K* factor is needed to keep false positive rates at ≤ 1%. EPA disagrees. Using *K* does not ensure that false positive rates will be consistently less than 1%. When the distribution is not normal, the false positive rate based on *K* may also exceed 1%; in these cases the Detection Limit would be adjusted based on ongoing verification regardless of which multiplier was originally used, and therefore there is no benefit to using *K* instead of student *t*. The student *t* factor

provides adequate protection against correction for high false positives by targeting an average false positive rate of 1% and allows for a consistent scaling factor for both censored and uncensored methods. At the same time use of the student *t* factor does not increase Detection and Quantitation Limits unnecessarily.

2. False Negative Correction

The use of a false negative correction factor is used in the procedure to satisfy the concern that there be "adequate space" between the Detection and Quantitation Limits. Because the Detection and Quantitation Limits are separately derived, there may be circumstances when the DQ FAC Single Laboratory Procedure v2.4 results in a Detection Limit equal to or greater than the initially and separately derived Quantitation Limit. In these cases, the procedure requires increasing the Quantitation Limit until it exceeds the Detection Limit by a certain amount.

EPA disagrees that this is the only, or best, solution to this circumstance. While there may be some desire that there be "adequate space" between the Detection and Quantitation Limits, this is not required, and there are circumstances where Detection and Quantitation Limits are equivalent. Moreover, use of the false negative correction factor to provide "adequate space" unnecessarily inflates the Quantitation Limit, resulting in inadequate protection of the environment.

Having both a false negative and false positive requirement in the same procedure requires added separation of the Detection and Quantitation Limits, inflating the Quantitation Limit beyond the true quantitation value. Furthermore, raising the Quantitation Limit to meet the false negative rate Measurement Quality Objective does not mean that there is greater protection against false negatives. Instead, it means that a more conservative statement is being made (i.e., you become more near-sighted) about where you can detect the analyte with high confidence. To better protect against false negatives, either the Detection Limit would need to be lowered (by calculating the Detection Limit using the student *t* instead of *K*, for example) or a more sensitive method would need to be used.

In addition to the two concerns identified by EPA above, one member of the Environmental Community Caucus had the following concern:

There is potential bias in identification of the Quantitation Level. For example, early discussion of a Quantitation Limit establishment (section 1.2.2) indicates that "the spiking level must be at or below the level that the laboratory intends to use as their Quantitation Limit for reporting." This could be read to imply that a good idea for the location of a Quantitation Limit exists even before a Quantitation Limit determination is carried out, and that only verification that a particular Quantitation Limit can be attained is needed. In addition, steps 1.2.3 –

1.2.6 (involving testing a particular spike level) imply that the main concern is that a level too low may have been chosen as the Quantitation Limit, rather than a level too high – i.e., all remedies for failure to meet criteria involve increasing the spike level (and thus the Quantitation Limit). While the procedure in section 1.2.2.1 outlines an approach to identifying the lowest possible Quantitation Limit when needed, it appears the rest of the procedure could produce a Quantitation Limit that is in at least some measure arbitrary (rather than more consistent with standard definitions of a Quantitation Limit). The overall effect is that the final Quantitation Limit in the general procedure may not reflect the true potential for analysis at lower levels, even absent an effort to determine the lowest possible Quantitation Limit.

There is a lack of clear rationale for use of some statistical or analytical approaches in the procedure (including via any experience in the literature). For example, in addition to questions on use of the *K*-statistic (as discussed by EPA), it is not clear if "Lowest Expected Result" in section 1.2.9 is an existing concept in the detection/quantitation literature.

There is an effective overall potential for over-protection against false positives at the expense of false negatives. In general, the remedies for failures to meet established criteria in the draft procedure involve raising either the Detection Limit or Quantitation Limit. (Section 2.7 of the DQ FAC Single Laboratory Procedure v2.4 does allow for "optional" lowering of Quantitation Limit if established criteria can be met.) In some cases these remedies may make sense from a statistical perspective, but they do not sufficiently consider the underlying measurement process. Laboratory contamination problems (for example) could lead to both high Detection Limit and Quantitation Limit values. A systematic reduction in contamination would lower the Detection Limit and potentially the Quantitation Limit (and/or help ensure that the Detection Limit was lower than the Quantitation Limit). The current MDL procedure addresses contamination, in part, in noting that the analyst should "prepare reagent (blank) water that is as free of analyte as possible." (40 CFR Part 136, Appendix B.) In addition to being consistent with good laboratory practices, a more formal recognition in the procedure of the importance of minimizing contamination would be consistent with the goal noted in the Great Lakes Initiative guidance for establishing a permit Quantitation Limit (or minimum level) when a nationally promulgated limit is not available, whereby "the permitting authorities must demonstrate that any minimum quantification level specified is as close to the WQBEL as practicable." (See Section VIII.H.2 in U.S. EPA, Water Quality Guidance for the Great Lakes System: Supplementary Information Document (SID), EPA-820-B-95-001, March 1995.)

One member of the Public Utilities Caucus and one member of the Environmental Laboratory Caucus raised the following concerns:

The MDL should be conducted over three to five days and then repeated at a minimum of once a year. The Laboratory Detection Limit and Quantitation Limit

cannot be any higher than the promulgated Detection Limit and Quantitation Limit for that method/analyte. Where a promulgated method/analyte is not available, the annual laboratory MDL cannot be any higher than the initial Laboratory Detection Limit and Quantitation Limit.

With each batch of samples, one should prepare and analyze a laboratory control spike at three to five times the Quantitation Limit. If precision is desired, then prepare and analyze the laboratory control spike in duplicate. Ideally the laboratory control spike should be at the Quantitation Limit. However, as everyone knows, some analytes have poor recoveries which would then put the quantitation below Quantitation Limit. This is not perfect but it is the best that can be done under the circumstances. What are really needed are better methods for the low recovery compounds, but that is not likely to happen anytime soon.

One Public Utility Caucus member expressed the following concern:

The proposed procedure is basically the same as the existing 40 CFR Part 136, Appendix B MDL procedure. The procedure at best predicts a 1% false positive rate when results from unspiked blanks are normally distributed. However, this condition is not met in the majority of situations where either the method produces no numeric results at all or, if numeric results are produced, they are non-normally distributed. As such, the proposed procedure does not actually produce a concentration at which a false positive rate would be 1%.

3.9 Determining a National Quantitation Limit

In order to fully implement the package of recommendations in Chapter 4, the Committee recognized that recommendations are needed on how to determine a National Quantitation Limit. The discussions in this section focus on how this would be accomplished.

Because of the regulatory significance of the proposed use of the National Quantitation Limit being considered by the Committee, it was extremely important to some caucuses that the procedure for determining a National Quantitation Limit be defined. Unfortunately, the Technical Work Group did not have adequate time to develop a detailed procedure. However, it did consider and bring forward some general recommendations for consideration by the Committee. These recommendations are intended to provide a framework to guide EPA in developing a detailed procedure.

The Technical Work Group and, subsequently, the Committee considered two alternative approaches to setting a National Quantitation Limit. One was an inter-laboratory procedure like the ASTM D6512-07. The other was a multi-laboratory procedure; however, there were no published multi-laboratory procedures for the Committee to consider. In discussing the merits of these two approaches, the Industry and Public Utility Caucuses expressed a desire that any procedure used for setting a National Quantitation Limit would assure that results on samples split between labs would be

comparable. While these caucuses felt that one viable approach to assuring comparability between laboratories was to base a National Quantitation Limit on an inter-laboratory procedure, they felt that this could be accomplished through other means. One example was by giving the issue of comparability special attention in the method validation process.

Future Method Promulgation – Validation Studies

> The Committee recommends that during the Data Quality Objective process, EPA give special attention to assuring the analytical method produces comparable results, at or near the National Quantitation Limit, on split samples, analyzed in different laboratories with the same method, and that EPA specifically describe the steps taken in the proposed rule.
>
> *Vote*: 14 Agree, 3 Not Opposed, 0 Disagree, 1 Absent
>
> Approved By Consensus
>
> Meeting #7, Decision 4.B

This consensus recommendation was left general to allow EPA flexibility to address the comparability issue differently for different situations and/or methods. During the discussion, it was observed that one means of assuring comparability might be in how Quality Assurance and Quality Control criteria are set, but there may also be other ways. In implementing the consensus recommendation, EPA should consider method validation studies that would specifically target comparability of results on split samples and then publish those studies when the methods are published for public comment. The adequacy of how it addressed the comparability issue would then be open for public review and comment.

At one point in its discussion of uses of detection and quantitation, the Committee entertained a process for collecting data through the Integrated Compliance Information System for the purpose of providing information to inform potential future updates of National Quantitation Limits. However, the Technical Work Group did not have time to develop general recommendations on how these data should be used to calculate future National Quantitation Limits. Because of concerns over the lack of a procedure for future updates of a National Quantitation Limit, the language pertaining to future updates was removed from further consideration.

Decision to Promulgate How National Quantitation Limits are Derived

Given the importance of the National Quantitation Limit for reporting, compliance and enforcement, the Committee recommended by consensus that EPA promulgate how the National Quantitation Limit would be derived and suggested a number of criteria that could be considered when EPA proposes such a procedure.

The Committee recommends that EPA promulgate how a National Quantitation Limit is derived.

Vote: 7 Agree, 10 Not Opposed, 0 Disagree, 1 Absent

Approved By Consensus

Meeting #7, Decision 5.B

Because a specific procedure for how a National Quantitation Limit would be determined was not recommended by consensus, the Committee felt that it is extremely important that EPA develop and promulgate an appropriate procedure.

Decision on Recommendation of Criteria to be Considered When EPA Promulgates Quantitation Limits

The Committee recommends:

a. EPA use the Data Quality Objective process to set target Measurement Quality Objectives for setting National Quantitation Limits for use in NPDES permit compliance testing.
b. A minimum of 6-7 labs be used to set National Quantitation Limits.
c. Data be collected, at a minimum, over 3- 6 months.
d. A minimum of 20 spikes be used in the calculation of each Laboratory Quantitation Limit.
e. The data and lab be evaluated for validity prior to acceptance.
f. An appropriate outlier test then is applied to the dataset.
g. The data are evaluated for normality, using standard statistical tests.
h. If the data are normally distributed then calculate the upper 95% confidence limit, which becomes the Quantitation Limit.
i. If the data are non-normally distributed then the 95th percentile of the Laboratory Quantitation Limit data becomes the Quantitation Limit.

Vote: 9 Agree, 8 Not Opposed, 1 Disagree, 2 Absent

Not Approved

States: 1 Agree, 2 Not Opposed, 1 Absent
Labs: 4 Not Opposed
Industry: 4 Agree
Public Utilities: 4 Agree
EPA: 1 Disagree
Environmental Community: 2 Not Opposed, 1 Absent
Meeting #10, Decision 4.H

Majority Report in Support of the Proposal

This majority opinion is consistent with others in that it refers to the Data Quality Objectives process to establish target Measurement Quality Objectives for NPDES compliance testing. The specification of between six and seven laboratories is consistent with well established inter-laboratory validation protocols (e.g., ASTM's D2777) and with the number of laboratories EPA has used previously in validating methods for 40 CFR Part 136. There are several reasons behind proposing that data be collected over

three to six months. First, most caucuses agreed that single laboratory Quantitation Limits should be based on routine operations implemented by collecting data over a period of time. The procedure considered by the Committee (but not approved by consensus) includes validation steps designed to assure that initial short-term estimates are valid. It takes time to let these validation procedures work effectively. In addition, to assure that any intermittent blank contamination is properly accounted for, the data must be collected over a suitable period of time. Most EPA methods take years to validate and promulgate, so three to six months of data gathering will not significantly delay promulgation of new methods and will assure that the checks and validations in the single laboratory procedure have time to work properly. The specification of 20 Quantitation Limit spikes is also intended to assure that a reliable estimate of the Quantitation Limit is obtained. The references to data validation and outlier testing are appropriate checks on quality control and protection against outliers, which are self evident. The final three points deal with concerns raised by some Technical Work Group members regarding the ability to determine whether the single Laboratory Quantitation Limits (from the small population represented if the minimum number of laboratories is used to derive a National Quantitation Limit) are normally distributed and to assure that appropriate statistics are applied. If the minimum number of laboratories is used, it will be impossible to determine whether the results are normally distributed, and the proposal defaults to use of the 95th percentile. However, if data from a large number of laboratories are available, it may be possible to determine if the data are normally distributed and, if so, to apply more powerful parametric statistics (e.g., the 95% confidence limit).

Concerns over the cost implications of this approach were raised. Clearly, as with most situations, a balance between cost and benefit must be determined.

Minority Report in Opposition to the Proposal

In a consensus recommendation at the June, 2007 meeting, EPA agreed to develop, propose and take public comment on a procedure to develop National Quantitation Limits from individual laboratory limits. At that meeting, the Technical Work Group was charged with developing a more specific recommendation but was unable to do so. Some of the specifics of this recommendation were part of the Technical Work Group's discussions; others were sent to members of the Committee less than a week before the Committee's September 19-21, 2007 meeting. EPA has not had sufficient time to consider the specifics of this proposal, has concerns that they were not thoroughly vetted, specifically, if they are the right criteria in all circumstances, and has concerns about EPA resource implications.

CHAPTER 4 – USES OF DETECTION AND QUANTITATION IN CLEAN WATER ACT PROGRAMS

4.1 Introduction

Any time water samples are analyzed, method Detection and Quantitation Limits are used as convenient benchmarks to conclude if an analyte is present and/or quantifiable. The Committee adopted consensus recommendations and developed majority/minority opinions for the determination and use of Detection and Quantitation Limits. These limits will serve to define the minimum required performance of a laboratory, may assist in comparing performance of one method to another (facilitating selection of a method most suitable for a given use), and may define important thresholds for use in evaluating compliance.

4.2 Uses of Detection and Quantitation in NPDES Permitting Where WQBELs Are Less Than Quantitation Limits

The Policy Work Group and the entire Committee spent a considerable amount of time discussing the many issues associated with uses of Detection and Quantitation Limits in NPDES permitting where WQBELs are less than Quantitation Limits. Since Committee caucuses had widely divergent positions on individual uses issues, the Committee decided early on that a recommendation on uses would need to be a "package deal," requiring caucuses to make trade-offs between individual aspects of the entire set of uses issues. The most current version of the working document representing this "package deal" is contained in *Appendix E: Uses Package.* The entire set of recommendations contained in this section represents the culmination of the Committee's discussions on uses in NPDES permitting programs. These recommendations are interlinked and were intended to represent the balanced "package" discussed by the Committee over the course of its deliberations. It was the Committee's intent that the recommendations of this section be implemented as a whole and not in a piecemeal fashion. It was originally intended that one vote on all NPDES Uses recommendations would be taken but instead, four votes were tallied at the September 2007 meeting. In those votes, the Committee did not reach consensus on all of the recommendations.

Votes of many Committee members on individual recommendations in this section assumed that all other components in this section would be approved. The Committee acknowledges that the outcome of the recommendations in this section might have been different if the voting process had been conducted with the original premise that each vote was representing the acceptance of the NPDES Uses recommendations in this section as an entire "package deal."

The Uses Package

Situations where Water Quality Based Effluent Limits (WQBELs) are less than Quantitation Limits present a challenge in setting permit limits and conditions as well as in making compliance determinations. In the absence of a regulatory requirement promulgated by EPA, state and other permitting authorities have been implementing different approaches for situations where the WQBEL is less than the identified Quantitation Limit. These include approaches for:

- Considering data reported at greater than the Detection Limit but less than the Quantitation Limit;
- Calculating monthly averages;
- Determining compliance with daily maximum limits and monthly average limits;
- Reporting data; and
- Appropriate compliance response in light of data uncertainty and the need for the protection of public health and the environment.

The Committee determined that it is appropriate to use the Quantitation Limit as the threshold for determining compliance with WQBELs as this is the lowest level where the accuracy demonstrated by the laboratory is appropriate for this purpose.

A) Need for a National Quantitation Limit

The Committee created the concept of a National Quantitation Limit as a key component of the "package of uses recommendations." The National Quantitation Limit concept recognizes the benefits to regulators and dischargers of a fair and uniform way to judge compliance with numeric NPDES effluent limitations where measurements are less certain. It is also intended to define the minimum level of acceptable performance for quantitation by a laboratory analyzing wastewater for compliance determinations. If implemented in federal regulation, the Committee proposals would set certain minimum requirements for permitting authorities implementing NPDES permit programs.

Where such a National Quantitation Limit is required, Section 3.9 discusses how it would be derived.

B) National Quantitation Limits for Existing and Future Methods

The Committee recommends that:

a. National Quantitation Limits be promulgated in a 40 CFR Part 122 table by analyte.
b. EPA generate National Quantitation Limits as rapidly as possible so that the Committee recommendation on permitting conditions and compliance determinations can be fully implemented.
c. Quantitation Limits be promulgated only using the nationally promulgated approach.
d. Methods may be promulgated without promulgating a Quantitation Limit for that method. As new methods are proposed without a promulgated Quantitation Limit, data (e.g., Single Laboratory Detection Limits, Single Laboratory Quantitation Limits, etc.) showing demonstrated method performance should be included in the method. The methods should include a statement that these performance levels are guidance and may not always be achievable.

Vote: 16 Agree, 4 Not Opposed, 0 Disagree
Approved By Consensus

States: 4 Agree
Labs: 2 Agree, 2 Not Opposed
Industry: 4 Agree
Public Utilities: 2 Agree, 2 Not Opposed
EPA: 1 Agree
Environmental Community: 3 Agree
Meeting #10, Decision 4.G

Many of the proposed recommendations in this Chapter are dependent on a national benchmark for quantitation and the development of Detection and Quantitation Limits are closely tied with promulgation and/or revision of analytical methods. Currently, the vast majority of method/analyte combinations promulgated in 40 CFR Part 136 do not have associated Quantitation Limits. The Committee made a consensus recommendation that EPA adopt National Quantitation Limits, using only the nationally promulgated approach, for situations where WQBELs are below the Quantitation Levels of existing Part 136 methods. The Committee agreed to list National Quantitation Limits by analyte in a table in Part 122. If EPA were to proceed on this path, it would need to create new National Quantitation Limits for most analytes before the benefits of the proposed recommendations of this Chapter can be fully realized. Therefore, the Committee recommended by consensus that EPA promulgate National Quantitation Limits as rapidly as possible. The Committee also recommended by consensus that EPA may promulgate new methods without promulgating a National Quantitation Limit for analytes under that method.

C) Addressing the Need for a National Detection Limit

The Committee debated the need for a National Detection Limit and the outcome of the discussion is shown below.

The Committee approves the removal of National Detection Limits from the Revised Uses document. *Vote*: 16 Agree, 2 Not Opposed, 1 Disagree Not Approved Meeting #8, Decision 1

Majority Report in Support of the Proposal

It was the majority opinion of the Committee to remove references to a National Detection Limit from the "Revised Uses Document" in *Appendix E*. In a separate vote, the majority opinion of the Committee was to include a provision under which the permitting authority would require a permittee to take action where a pollutant in a discharge is detected below the Permit Quantitation Limit by the permittee's laboratory a "significant number" of times. This opinion was based in large part on the recognition that many Laboratory Detection Limits would be below a National Detection Limit that might have been promulgated. The Laboratory Detection Limit would be used as a lower bound for reporting "detected less than Permit Quantitation Limit" in Part 2 of the vote associated with this section. EPA may still want to promulgate a Detection Limit associated with 40 CFR Part 136 methods as a valuable reference point.

Minority Report in Opposition to the Proposal

The Committee is proposing that a fixed National Quantitation Limit be established for each regulated analyte where generally available Quantitation Limits are above permit limits (e.g., a WQBEL), that a Permit Quantitation Limit

be established at the National Quantitation Limit and that individual laboratories need to have a laboratory specific Quantitation Limit less than or equal to the Permit Quantitation Limit when the National Quantitation Limit is greater than a permit limit (e.g., WQBELs). The Committee considered but could not reach consensus on the following reporting conventions:

- Results below the Detection Limit be reported as "not detected,"
- Results between the Permit Quantitation Limit and Detection Limit be reported as "detected but not quantified at or above the Permit Quantitation Limit," and
- That "not detected" and "detected but not quantified at or above the Permit Quantitation Limit" results be treated for averaging purposes as zero.

For this strategy to work, the values of Quantitation Limit and Detection Limit have to be sufficiently different to allow for "detected but not quantified" to be detected. A National Detection Limit would be a ceiling on the Detection Limit that individual laboratories could report. The National Detection Limit is needed to ensure that there is adequate "distance" between the Detection Limit determined by an individual laboratory and the National Quantitation Limit. It would be counterproductive to have a Detection Limit that was equal to the National Quantitation Limit, or nearly so.

The National Detection Limit is also needed to ensure equal protection to all receiving bodies with a given WQBEL and equity for all permittees discharging to receiving bodies with a given WQBEL. As the Pilot Study showed, laboratories can produce a Detection Limit with concentrations that differ over orders of magnitude. Without a National Detection Limit, it would be possible for two permittees to discharge water to a receiving body with the same concentration of an analyte. One would have to do a pollutant minimization program and the other would not, simply because of differences in the laboratory capability. In fact, with the range of differences in Detection Limits seen in the Pilot Study, it would be possible for a discharger with a higher concentration to have no pollutant minimization program whereas a discharger with a lower concentration would have to conduct a pollutant minimization program. This does not provide equal protection to all waters nor equity to permittees.

D) Establishing NPDES Permit Conditions and Determining Compliance

As indicated above, the Committee took a single vote on the four-part proposal that follows. These four parts of the proposal are presented separately as Parts 1, 2, 3, and 4 although the Committee took a single vote on the proposal as a whole. The majority report begins on page 49 and the minority report on page 50.

Recommendation on Reporting Data and Determining Compliance Where the WQBEL is Less Than the National Quantitation Limit

Except in cases where the permitting authority requires use of a method more sensitive than the method for which a National Quantitation Limit exists, the Committee proposed recommendations that EPA promulgate a rule to modify 40 CFR Part 122, as follows:

Part 1

The Committee recommends the following be required where EPA has promulgated a National Quantitation Limit in 40 CFR Part 122:

a. The default Quantitation Limit to be included in the permit or in rule as appropriate (Permit Quantitation Limit) is the 40 CFR Part 122 promulgated National Quantitation Limit unless the regulator determines that the Permit Quantitation Limit should be adjusted to account for sensitivity, selectivity, and/or matrix effects;
b. The permit shall contain a condition that the Quantitation Limit determined by the permittee's laboratory (Laboratory Quantitation Limit) shall be at or below the Permit Quantitation Limit. The permittee's laboratory may use any 40 CFR Part 136 method for which they can demonstrate a Laboratory Quantitation Limit at or below the Permit Quantitation Limit. If matrix effects have been given special attention in the permit then they would also have to be considered in compliance and enforcement;
c. The permit shall require the permittee to report the Laboratory Detection Limit and the Laboratory Quantitation Limit and maintain such information for a period of at least five years;
d. The permit shall require the permittee to maintain individual numeric results for a period of at least five years. The regulator may require the individual numeric result for any value that is greater than or equal to the Laboratory Detection Limit and less than the Permit Quantitation Limit be reported in a supplemental report;
e. The permit shall require that the Laboratory Detection Limit and the Laboratory Quantitation Limit be determined using the steps of the 40 CFR Part 136 procedure to establish the lowest possible value by the laboratory; and
f. That EPA require the Laboratory Detection Limit, the Laboratory Quantitation Limit, and the Permit Quantitation Limit be reported by the regulator to the Integrated Compliance Information System (ICIS).

Meeting #10, Decision 4.I

Part 2

The Committee recommends the following be required where EPA has promulgated a National Quantitation Limit in 40 CFR Part 136:

a. The permitting authority will set average and daily maximum permit limits at the WQBEL.
b. Permittees must report to the permitting authority all information in the following manner on the Discharge Monitoring Report (DMR):
    i. To report daily maximum sample results:
        a. For values not detected at the Laboratory Detection Limit, report "not detected."
        b. For values detected at the Laboratory Detection Limit but less than the Permit Quantitation Limit, report "detected less than the Permit Quantitation Limit."
        c. For values greater than or equal to the Permit Quantitation Limit, report the actual numeric values.
    ii. To report average sample results:
        a. When all values used to calculate an average are not detected at the Laboratory Detection Limit, report "not detected."
        b. When all values used to calculate an average are "detected less than Permit Quantitation Limit," report "detected less than the Permit Quantitation Limit."
        c. When values used to calculate an average are a combination of "not detected" and "detected less than the Permit Quantitation Limit," report "detected less than the Permit Quantitation Limit."
        d. When one or more value used to calculate an average is greater than or equal to the Permit Quantitation Limit, report the calculated numeric average after assigning zero to any individual sample result reported either as "not detected" or "detected less than the Permit Quantitation Limit."
c. To determine NPDES permit compliance with results reported on the DMR, the permitting authority will:
    i. Determine that any results reported as either "not detected" or "detected less than the Permit Quantitation Limit" are in compliance with the effluent limitation.
    ii. Compare any numeric result directly to the WQBELs

Meeting #10, Decision 4.I

Part 3

The Committee recommends the following be required where EPA has promulgated a National Quantitation Limit in 40 CFR Part 136:

Permits shall include language that triggers additional steps when a "significant number" (to be determined in permitting process) of values detected at the Laboratory Detection Limit but less than the Permit Quantitation Limit are reported. These steps may include additional or accelerated monitoring, analytical studies such as matrix studies, pollutant minimization programs, or other permit conditions outside of the determination of compliance with effluent limitations. Reports under such provisions will be done outside of the DMR process, except that any additional effluent testing performed using approved analytical methods as part of the special studies must be reported on the DMR.

Meeting #10, Decision 4.I

Part 4

The Committee recommends the following be required where EPA has not promulgated a National Quantitation Limit in 40 CFR Part 136

a. In the absence of a National Quantitation Limit, the permitting authority is free to establish it's method for determining compliance for analytes that have limits/water quality standards at a level lower than that which can be detected and/or quantified.
b. For a list of analytes as defined by EPA, the permit shall require that the Laboratory Detection Limit and the Laboratory Quantitation Limit be determined using the steps of the 40 CFR Part 136 procedure to establish the lowest possible value by the laboratory; and
c. That EPA require the Laboratory Detection Limit, the Laboratory Quantitation Limit, and the Permit Quantitation Limit be reported by the regulator to the Integrated Compliance Information System.

Meeting #10, Decision 4.I

Result of Vote on Parts One Through Four as a Package

Vote: 12 Agree, 4 Not Opposed, 4 Disagree
Not Approved

States: 3 Agree, 1 Not Opposed
Labs: 3 Not Opposed 1 Disagree
Industry: 4 Agree
Public Utilities: 4 Agree
EPA: 1 Agree
Environmental Community: 3 Disagree
Meeting #10, Decision 4.I

Majority Report in Support of the Proposal

This four-part majority opinion contains the specifics of how NPDES data reporting and compliance determinations would be made in situations where the WQBEL is less than the National Quantitation Limit. The goal of the proposal is to promote more uniformity and equity in reporting and in compliance determinations across the NPDES permitting program, resulting in efficiencies for permitting authorities and regulated parties alike. Besides the WQBEL, two benchmarks, the Permit Quantitation Limit and the Laboratory Detection Limit, are critical to implementing these proposals. The Permit Quantitation Limit in the NPDES permit would be the National Quantitation Limit promulgated in 40 CFR Part 122 unless the permitting authority determined that the National Quantitation Limit did not adequately account for differences in selectivity and sensitivity that are characteristic of the discharge matrix of the permittee. In that case, the permitting authority would adjust the Permit Quantitation Limit to account for these matrix effects, and reporting and compliance determinations would adjust accordingly. As indicated earlier in this Chapter, the Laboratory Detection Limit was chosen as the threshold for reporting detected below the Permit Quantitation Limit instead of a National Detection Limit because it was thought that laboratories would have Detection Limits below those that might be nationally promulgated. Laboratories would establish Detection (and Quantitation) Limits using the steps of the 40 CFR Part 136 procedure to establish the lowest possible value.

As previously stated, in the absence of a federal regulation regarding requirements for Detection and Quantitation Limits and their uses, states have implemented different approaches to address the situation where a WQBEL is less that the achievable Quantitation Limit. In deference to these existing state approaches, the Committee recognizes that, where authorized or not prohibited by law, any state or other permitting authority could adopt provisions that would go beyond the requirements proposed by the Committee. This is done with the understanding that entities that have been delegated the NPDES program from EPA have the authority under the Clean Water Act to adopt regulatory provisions that are different, but no less stringent than, those required under federal regulations. Such provisions would operate in lieu of the above four-part proposal and could include a Quantitation Limit value adopted by the state (State Quantitation Limit) lower than the nationally promulgated National Quantitation Limit.

In that case, the State Quantitation Limit adopted by a delegated state would be used for determining compliance, reporting, and other applicable requirements.

In deciding how to approach the calculation of the monthly average, the Committee needed to decide how to treat values between detection and quantitation. The Committee recognized that analytical results have a higher level of uncertainty where an analyte is detected at or above a Laboratory Detection Limit but below the Permit Quantitation Limit (detected less than the Permit Quantitation Limit) but that the science suggests they are unlikely to be zero. Given this uncertainty, assigning a non-zero value where an analyte is detected less than the Permit Quantitation Limit (DLPQL) would have significant compliance and enforcement implications. The Committee developed a coupled approach for determining compliance and responding to DLPQL values as described in the above proposal.

The Committee considered the recommendation that EPA promulgate a rule to modify 40 CFR Part 122 to incorporate the above proposal. Should the permitting authority require use of a method more sensitive than the method for which a National Quantitation Limit exists, the above proposal would not apply.

It may take many years for EPA to promulgate National Quantitation Limits for analytes with WQBELs less than currently achievable Quantitation Limits. Therefore, the situation where there is no promulgated National Quantitation Limit must be addressed. In this case, the Committee did not find it practical to establish requirements for determining compliance and suggests that the permitting authority be free to use its own process in this situation. However, the Committee believes that it is imperative that any new 40 CFR Part 136 procedure for determining the Laboratory Detection Limit and Quantitation Limit be implemented for all methods/analytes based on its determination that the new procedure will provide results at a higher level of confidence than those using the current MDL approach. In addition, reporting of data generated using the new procedure is important to provide EPA with information it can use to set priorities for modifying existing methods or developing new methods to improve Laboratory Detection and Quantitation Limits.

Minority Report in Opposition to the Proposal

Reporting of Detected but Not Quantified Values - The proposal would entail narrative reporting (e.g., "detected less than the Permit Quantitation Limit") in lieu of actual values for detected concentrations below the Quantitation Limit on the Discharge Monitoring Report. Such values (i.e., detected but not quantified, or DNQ) have a high probability of truly being non-zero results, and yet, in the proposal, would be reported only at the discretion of the permitting authority, on a supplemental report. This proposal would likely have the overall effect of providing less information to permitting authorities in general (including to EPA), information which could otherwise be potentially useful in several ways. For example, such data could be useful in assessing progress in reducing pollutants to non-detectable levels via implementation of pollutant minimization plans. (For

example, see discussion in Section VIII.H.4 in U.S. EPA, Water Quality Guidance for the Great Lakes System: Supplementary Information Document, EPA-820-B-95-001, March 1995.)

Calculating and Reporting Average Sample Results and Use of Zero - The proposal included a provision to report "detected less than the Permit Quantification Limit" in cases where samples show a mix of not-detected and detected not quantified values, as well as a provision to obtain numeric averages only in cases where at least one value was quantified, and with all non-quantified results assigned zero. This approach is different from more commonly used practices in the scientific literature, where it has long been recognized that substitution of zero in cases of not detected or not quantified values will bias an average low. For example, for an analyte whose measured value is occasionally above the Quantitation Limit but where zero is reported for more numerous instances of hits below Quantitation Limit, the average will be artificially lowered, resulting in lower apparent loads and less protection of a water body. The general practice of assigning zero to non-detects can lead to the "virtual absence" of the analyte from a data set. (See Currie, L.A., 2004, *Applied Radiation and Isotopes*, 61:145-149.)

Reporting of Low-Level Data and Uncertainties - There is recognition in the scientific community of the value in reporting low-level data and associated uncertainty. (See, for example, discussion in Currie, L.A., 1999, *Anal. Chim. Acta*. 391:105-126 and Currie, L.A., 1999, *Anal. Chim. Acta*. 391:127-134.) Currie (2004) further states, "There is near universal agreement that results of measurements and their uncertainties should be reported for *all* experimental data, including data in the region of the Detection Limit and below (ASTM, 1997, 2000; ISO, 1993; IUPAC, 1998)." (emphasis in original)

The opinion to use zero in averaging is not consistent with EPA guidance in the Great Lakes. In the compliance provision of the Great Lakes Initiative, EPA allowed permitting authorities the discretion to use their own averaging procedures (which may include, for example, assigning zero or one-half the quantitation level for values below Quantitation Limit). Furthermore, the total maximum daily load provision of the Great Lakes Initiative indicates it is acceptable (to EPA) to assign zero values to sample data only in cases where all values are below the Detection Limit (40 CFR Part 132, Appendix F, Proc. 3). In other cases, EPA guidance indicates that "States and Tribes are required to use commonly accepted statistical techniques…" that can include the use of default values such as one-half the Detection Limit or the mid-point between Detection Limit and Quantitation Limit, as appropriate (Section VIII.C. in U.S. EPA, 1995, *Op. Cit.*).

Additional Permit Requirements - The draft proposal included language stipulating that additional steps would be required when "a significant number" (to be determined in permitting process) of values detected at the Laboratory

Detection Limit but "less than the Permit Quantitation Limit are reported." These additional steps could – but would not necessarily – involve incorporation of a pollutant minimization plan provision in the permit. In contrast, the Great Lakes Initiative requires inclusion of a pollutant minimization plan in initial issuance of a permit in cases where the WQBEL for an analyte is less than the Quantitation Limit. In addition, in these situations the Great Lakes Initiative also requires a re-opener clause which authorizes modification or revocation and reissuance of a permit if new information indicates the presence of a pollutant above the WQBEL (40 CFR Part 132, Appendix F, Proc. 8); this is slightly more stringent than the proposed Committee permitting strategy.

Potential for Non-Compliance - The potential for increased non-compliance in a situation where values less than Quantitation Limit are reported should be addressed through alternative compliance and enforcement strategies rather than simply minimized through an inappropriate data censoring process. Measurement uncertainty should be considered in these situations, drawing on accepted protocols. (See, for example, the International Standards Organization Guide to the Expression of Uncertainty in Measurement.) Alternative compliance and enforcement strategies (which could include provisions so that single samples, for example, do not trigger enforcement actions) could include, for example, additional and/or more targeted monitoring of effluents or internal streams, fish tissue or other biota if appropriate, or re-examination of the pollutant minimization plans and proposal of additional research measures or practices to further reduce the pollutant load.

E) Great Lakes Initiative Compliance

The Committee recommends that its recommendations should not supersede the current Great Lakes Initiative provisions. The Committee believes that there is not a significant conflict between the Committee recommendations and the Great Lakes Initiative.

Vote: 20 Agree, 0 Not Opposed, 0 Disagree
Approved By Consensus

Meeting #10, Decision 4.A

In l995, EPA and the Great Lakes States agreed to a comprehensive plan to restore the health of the Great Lakes. The Final Water Quality Guidance for the Great Lakes System, also known as the Great Lakes Initiative, includes criteria for states to use when setting water quality standards for 29 pollutants. The Great Lakes Initiative, like this Final Report, recognizes and addresses the scenario where WQBELs are below the Quantitation Limit of the most sensitive method. In these situations, the Great Lakes Initiative provides for compliance determinations below the Quantitation Limit and for pollutant minimization plans similar to the Committee's proposal in this Final Report.

4.3 Other Uses

The Committee considered other potential Uses of detection and quantitation in Clean Water Act programs and made the following consensus recommendations.

A) Other Uses of Detection and Quantitation Detection and Quantitation

The Committee tabled discussion on considering whether to make recommendations regarding the use of detection and quantitation for other uses including, but not limited to, the following:

- ambient monitoring 305(b)
- pretreatment
- non-regulatory operational monitoring
- stormwater monitoring
- other studies, such as fish tissues or biosolids characterization
- reasonable potential analysis
- effluent guidelines development
- limit derivation
- development of water quality criteria
- 303(d) listing for Total Maximum Daily Loads

Vote: 20 Agree, 0 Not Opposed, 0 Disagree
Approved By Consensus

Meeting #10, Decision 4.B

Initially, the Committee did a preliminary review of most of the Clean Water Act programs and found potential differences in how these programs make use of method Detection and Quantitation Limits. Time did not permit the Committee to fully understand these differences so a decision was made early on to focus, instead, on the use of Detection and Quantitation Limits in the National Pollutant Discharge and Elimination System (NPDES) program. As a result, the Committee affirmatively decided to table discussion and recommendations on uses of method detection and quantitation in other Clean Water Act programs.

In the end, the Committee focused on NPDES permit and compliance uses and developed a proposal that EPA promulgate procedures for obtaining individual laboratory Detection Limit and Quantitation Limit values as well as a National Quantitation Limit value/s for specific methods.

B) Data Reporting Convention

During early discussions concerning Measurement Quality Objectives and the pilot test program design, an issue arose as to how values below the Quantitation Limit should be reported given the uncertainty associated with data below quantitation. The DQ FAC Single Laboratory Procedure v2.4, proposed by a majority of the Committee, would require reporting of all data values, regardless of the uncertainty associated with the value, and as indicated earlier, the laboratory would need to retain these values for five years. However, this protocol does not address what to do with these values when they

must be reported for Clean Water Act purposes. Earlier in the Committee's deliberations, several suggestions were made as to how to report data below quantitation, including, all values should be reported, that "0" should be reported, and that values should be "flagged." For various reasons, none of these suggestions met all stakeholder needs. The Committee agreed early on to the following reporting convention:

> Agreed, by consensus, that if or when data are reported below L_Q, then the data points that fall between L_C and L_Q would be reported, for example, as detected but not quantified (e.g., DNQ).
>
> *Vote*: 19 Agree, 0 Not Opposed, 0 Opposed, 2 Absent
>
> Approved By Consensus
>
> Meeting #4, Decision 4.B

For purposes of the Pilot Study, the Committee agreed to deviate from this reporting convention in order to facilitate the data analysis outlined in the Pilot Study design.

C) Alternative Test Procedures

> The Committee did not develop specific recommendations to EPA on updating the Alternative Test Procedures Program. The Committee, however, does recommend that the Alternative Test Procedures Program be updated to be consistent with recommendations from this document.
>
> *Vote*: 20 Agree, 0 Not Opposed, 0 Disagree
>
> Approved By Consensus
>
> Meeting #10, Decision 4.C

Under the Alternative Test Procedures Program, an organization may submit an application for approval of a modified version of a Part 136 method or for approval of a new method to be used as an alternate to a Part 136 method.[6] The submitting organization is responsible for validating the new or modified method. EPA reviews the Alternative Test Procedure Program validation package and, if approved, subsequently promulgates the approved Alternative Test Procedure Programs in Part 136. The Alternative Test Procedure Program and rulemaking processes make demands on limited EPA methods-related resources, and, as such, approval of Alternative Test Procedure Programs can take many months and two years or more to promulgate the approved method in Part 136. Initially, the Committee intended to address some of the shortcomings of the Alternative Test Procedure Program but did not have time to do so. However, because Alternative Test Procedure Program methods and EPA-validated methods are accorded equal status once they are promulgated in Part 136, the Committee believes recommendations in this report should apply equally to Alternative Test Procedure Program methods promulgated in Part 136.

[6] Requirements for approval of alternate analytical techniques (methods) are specified at 40 CFR 136.4 and 136.5 for wastewater methods

CHAPTER 5 – MATRIX EFFECTS

5.1 Introduction

Several stakeholder caucuses expressed concern over how matrix effects can adversely impact the performance of some analytical methods, including the possibility that Detection and Quantitation Limits based on reagent water could not be achieved in real world samples. Questions with respect to how matrix effects should be addressed included how they should be accounted for in method development, how a matrix effect should be demonstrated, and how, or if, a matrix-specific Detection or Quantitation Limit would be determined. In the absence of federal guidance that addresses the four majority opinions below, some states issuing permits that are confronted by matrix effects have developed guidance. However, this approach leads to inconsistencies and makes it harder for permittees and laboratories to address the issue.

Although there was interest in addressing matrix effects, there was insufficient time for the Committee to develop specific proposals. Rather than leave the issue unaddressed, several general proposals were formulated and considered. They generally involved having EPA develop guidance in specified areas and, to the extent time allowed, identify some specific issues that should be addressed. The formulation of these proposals in the form of guidance instead of regulations was a conscious choice, given the difficulty of writing regulatory language for a topic that really needs to allow for some flexibility and professional judgment, and a more basic question about whether such a regulation would be appropriate. Four proposals considered by the Committee and the outcome of the voting follow.

5.2 Matrix Effects: Discussion and Decisions

Matrix Effect Decision #1

The Committee recommends that EPA publish new guidance on matrix effects. At a minimum, the guidance should outline the appropriate level of matrix effects validation necessary for method promulgation for analytical methods to be considered for 40 CFR Part 136. The Committee recommends that EPA adhere to this guidance in methods it develops and validates for promulgation in 40 CFR Part 136. This guidance should also address the following:

- Determining the appropriate number of matrices to take into account.
- The level of validation required verses the proposed scope of use for the analytical method.
- Matrix effects validation in the Alternative Test Procedures Program.
- Impacts for consensus standards methods considered for Part 136.

Vote: 10 Agree, 7 Not Opposed, 3 Disagree

Not Approved

States: 4 Not Opposed
Labs: 1 Agree, 1 Not Opposed, 2 Disagree
Industry: 4 Agree
Public Utilities: 4 Agree
EPA: 1 Disagree
Environmental Community: 1 Agree, 2 Not Opposed
Meeting #10, Decision 8.A

Majority Report in Support of the Proposal

Some methods currently promulgated in 40 CFR Part 136 demonstrate matrix effects when applied to real world samples for some analytes, thus creating the problems that permit writers and permittees face when permit compliance testing is required. If greater attention to testing the ruggedness of a proposed Part 136 method were given during method development and validation, better methods would be promulgated, thus beginning to mitigate these issues in the future. However, it is impractical to validate a method for all possible matrices, so a trade-off between thorough ruggedness testing and cost benefit is warranted. Implementation of this approach would provide guidance and a framework for both EPA and third party method developers. It would also provide EPA a great deal of flexibility in determining the correct balance between characterizing method performance and cost. The overall reasoning behind the majority opinion is to generally improve the quality of methods that are promulgated, thereby reducing future difficulties in permitting.

Minority Report in Opposition to Matrix Effects Decisions 1-4 is on page 57.

Matrix Effect Decision #2

The Committee recommends that EPA develop a consistent protocol on how to demonstrate matrix effects. The Committee believes such a protocol should be sensitive to cost and required level of effort to ensure that it is applied consistently.

Questions to be addressed by the protocol:

- What level of effort is necessary to determine if the matrix effects can be resolved by modifications of the analytical method that are within the flexibility allowed within the method?
- What set of experiments and data interpretation framework would suffice to demonstrate a matrix effect if performed properly?
- Who should be responsible for implementing a procedure to determine a matrix specific Quantitation Limit?
- How broadly applicable shall a matrix effect be considered? What level of demonstration should be considered adequate for a single facility? What level of demonstration should be undertaken to extend the matrix specific Quantitation Limit to other like wastewaters?

Vote: 13 Agree, 6 Not Opposed, 1 Disagree

Not Approved

States: 3 Agree, 1 Not Opposed
Labs: 2 Agree, 2 Not Opposed
Industry: 4 Agree
Public Utilities: 3 Agree, 1 Not Opposed
EPA: 1 Disagree
Environmental Community: 1 Agree, 2 Not Opposed
Meeting #10, Decision 8.B

Majority Report in Support of the Proposal

Such a protocol could be used by EPA during method validation to evaluate ruggedness of the performance of an analytical method on different types of sample matrices. Similarly, the protocol could be a useful guidance document for third party method developers (e.g., consensus organizations or anyone submitting an Alternate Test Procedure application). If a standardized protocol were available, interested stakeholders would know what needed to be done and could elect to undertake the required testing to submit to EPA. The standardized protocol would assure that, if the protocol were followed, EPA would consider the data, thus leveraging EPA resources with stakeholder resources. The protocol could also be used by permittees, petitioning for consideration of matrix effects during the permitting process. Having one set of guidance apply across the nation would facilitate comparability and consistency and could result in cost savings and efficiency. Furthermore, it would help ease the burden on states and/or permit writers.

Minority Report in Opposition to Matrix Effects Decisions 1-4 is on page 57.

Matrix Effect Decision #3

The FACDQ recommends that EPA develop a procedure for determining matrix-specific Detection or Quantitation Limits for use where appropriate. Again, such a protocol should be sensitive to cost and required level of effort.

Questions that should be addressed include:

- Who should be responsible for implementing a procedure to determine a matrix specific Quantitation Limit?
- How broadly applicable shall a matrix effect be considered?
- What level of demonstration should be considered adequate for a single facility?
- What level of demonstration should be undertaken to extend the matrix specific Quantitation Limit to other like wastewaters?

Vote: 11 Agree, 8 Not Opposed, 1 Disagree

Not Approved

States: 2 Agree, 2 Not Opposed
Labs: 2 Agree, 2 Not Opposed
Industry: 4 Agree
Public Utilities: 3 Agree, 1 Not Opposed
EPA: 1 Disagree
Environmental Community: 3 Not Opposed
Meeting #10, Decision 8.C

Majority Report in Support of the Proposal

Regulations such as the Great Lakes Initiative provide for the possibility of a matrix-specific Quantitation Level in a permit but fail to provide instruction or guidance on how such a limit would be determined. Federal guidance on this topic would facilitate comparability and consistency. Comparability across the country would allow permittees and permit writers to consider data on a similar source developed in another jurisdiction, thus potentially saving costs. Consistency would make it easier and more cost effective for permittees to generate required data.

Minority Report in Opposition to Matrix Effects Decisions 1-4 is on page 57.

Matrix Effect Decision #4

When considering future updates of a National Quantitation Limit, the Committee recommends that EPA take into consideration any experience with the performance in different matrices.

Vote: 11 Agree, 4 Not Opposed, 5 Disagree

Not Approved

States: 2 Agree, 2 Disagree
Labs: 2 Agree, 2 Disagree
Industry: 4 Agree
Public Utilities: 3 Agree, 1 Not Opposed
EPA: 1 Disagree
Environmental Community: 3 Not Opposed
Meeting #10, Decision 8.D

Majority Report in Support of the Proposal

At various times during deliberations, Committee members expressed concern over the fact that EPA has not updated any analytical procedures promulgated in 40 CFR Part 136 and a concern that similar problems will exist for any promulgated Quantitation Limits. EPA expressed interest in approaches to updating National Quantitation Limits in the future, although the Committee offered no specific recommendation on how this should be accomplished. However, given EPA's expressed interest in procedures for possible future updates, this majority opinion expresses the common sense notion that what is learned about a method performance and/or limitations (e.g., with respect to matrix effects) through the benefit of using the procedure over time, should not be ignored when considering future updates of National Quantitation Limits.

The proposed recommendation leaves it to EPA to determine how it should consider such information and how, or if, it should affect the update of a National Quantitation Limit. It does not state that the National Quantitation Limit must be set at the highest Quantitation Limit observed in any given matrix. However, if experience shows that many industries or municipalities cannot achieve the National Quantitation Limit in their matrices, EPA may want to reconsider whether it would be appropriate to update the National Quantitation Limit based on reagent water if doing so would only exacerbate the already evident problems.

Minority Report in Opposition to Matrix Effect Decisions 1-4

Two members of the Laboratory Caucus are concerned about Matrix Effects Decision #1 in that additional demonstrations on different matrices would have a negative impact on the ability of EPA to quickly incorporate new and improved methods in 40 CFR Part 136.

Two State Caucus members and one Environmental Laboratory Caucus member are concerned about Matrix Effects Decision #4. If promulgated, National Quantitation Limits are presented as a single benchmark that laboratories across

the nation must achieve when analyzing samples for compliance determinations. In that context, a wide spectrum of matrices (and potential matrix effects) is conceivable. Some effluent matrices may have no adverse effect on the ability of laboratories to quantify contaminants at the National Quantitation Limit, whereas other matrices may contribute to analytical interference or "noise." It appears impractical that EPA could consider all possible matrix effects in various discharges when promulgating a National Quantitation Limit for nationwide applicability. The Committee's uses proposals gave latitude to the permitting authority to consider matrix effects when setting permit monitoring conditions, including required Quantitation Limits for reporting. It seems more practical to consider matrix effects when setting permit conditions where the matrix is demonstrated to be problematic in achieving required Quantitation Limits.

The EPA is concerned about all four matrix-related decisions based on concerns about resources and the difficulty of developing the recommended guidance. The core recommendations of the Committee – pilot test the new single laboratory procedure, promulgate and implement new rules incorporating the single laboratory procedure, propose an algorithm for determining the National Quantitation Limit, and define the uses of detection and quantitation in compliance and enforcement of NPDES permits – will require significant EPA resources over the next several years. At this time, EPA cannot commit additional resources to several of the other recommendations of this report, including those on matrix effects, until these core recommendations are implemented.

Additionally, EPA is concerned about the need to account for individual industry matrix effects when developing National Quantitation Limits and about the difficulty of developing matrix guidance[7] for individual NPDES permits that would work well in almost all situations. Currently, there are about 55 large categories of industrial facilities composed of 450 industrial subcategories, representing about 70,000 permitted facilities. This does not account for the over 16,000 publicly-owned treatment works that must be permitted and may also have matrix effects issues.

The complexity inherent in having many matrices in the NPDES program would affect permittees who would consider matrix effects in reporting compliance results whenever EPA used matrix effects to develop National Quantitation Limits.

[7] EPA has a guidance document on matrix effects that is more general than that proposed by the matrix effects recommendations. This guidance document, known as the "Pumpkin Book," allows a user to demonstrate a mitigation against matrix effects.

CHAPTER 6 – RECOMMENDATIONS ON OTHER ISSUES

6.1 Introduction

During its latter meetings, the Committee began to consider additional issues that needed to be addressed to maximize the success of any EPA-adopted Committee recommendations. This resulted in additional recommendations that, if implemented, would:

- Ensure consistency of procedures for detection and quantitation across EPA programs;
- Engender confidence in the procedures through post-promulgation performance confirmation;
- Have EPA continue its leadership role in the development of analytical methods and provide necessary resources to develop new high quality methods;
- Have EPA establish Data Quality and Measurement Quality Objectives for the use of detection and quantitation in Clean Water Act programs and consider addressing other Clean Water Act programs such as 303(d) listings and NPDES effluent limit determinations; and
- Have EPA develop guidance for implementing the new procedures and computer applications to assist in calculation of Detection and Quantitation Limits.

6.2 Implementation of a Committee Procedure in all EPA Programs Referencing 40 CFR Part 136

To maintain consistency and minimize effects on the environmental laboratory community, the Committee recommends that EPA programs that reference the present Part 136 Appendix B procedure consider adopting (the new procedure) that would replace it.

Vote: 20 Agree, 0 Not Opposed, 0 Disagree
Approved By Consensus

Meeting #10, Decision 5.D

A given analytical technique may be used for detecting and quantifying a given analyte or set of analytes for several different EPA programs. Thus, this consensus recommendation was proposed to emphasize the importance of having a consistent Detection Limit and Quantitation Limit procedure across as many EPA programs as possible.

Maintaining more than one Detection Limit procedure would be complex, costly and confusing for data users and the laboratory community. The Committee recommends by consensus that additional EPA programs/offices consider adopting the procedure which is finally promulgated by the Office of Water as a replacement for 40 CFR Part 136, Appendix B.

6.3 EPA Leadership Role in Developing New Analytical Methods

The Committee recommends that EPA continue to act as the national lead for Clean Water Act programs in developing analytical methods and setting the performance standards for those methods.

Vote: 20 Agree, 0 Not Opposed, 0 Disagree
Approved By Consensus

Meeting #10, Decision 5.A

6.4 Targeting EPA Resources for Analytical Methods Where Most Needed

The Committee recommends that EPA evaluate the federal resources dedicated to developing analytical methods with Detection/Quantitation Limits of sufficient quality (i.e., meet Data Quality Objectives) and capable of meeting the needs of Clean Water Act programs (e.g., quantitation at or below current water quality standards) and adjust those resources, where necessary, to meet data quality and program needs.

Vote: 19 Agree, 0 Not Opposed, 0 Disagree, 1 Abstain (EPA)
Approved By Consensus

Meeting #10, Decision 5.B

6.5 Evaluating and Defining Uses of Detection and Quantitation in Other Clean Water Act Programs

The Committee recommends that EPA evaluate and modify the uses of data in Clean Water Act programs (beyond those uses discussed in the Committee recommendations) based on data uncertainty and decision error rate requirements relative to corresponding Detection and Quantitation Limits. This could be accomplished through establishment of and adherence to data quality objectives for all Clean Water Act programs. How data relative to detection and quantitation limits are to be used in 303(d) listings, reasonable potential determinations, NPDES effluent limit derivation, the development of water quality criteria, and other uses should be documented.

Vote: 13 Agree, 6 Not Opposed, 1 Disagree
Not Approved

States: 1 Agree, 3 Not Opposed
Labs: 1 Agree, 3 Not Opposed
Industry: 4 Agree
Public Utilities: 4 Agree
EPA: 1 Disagree
Environmental Community: 3 Agree
Meeting #10, Decision 5.F

Majority Report in Support of the Proposal

This majority opinion emphasizes that, regardless of which Measurement Quality Objectives are adopted for Clean Water Act programs, data will have uncertainty based on the reliability of samples collected and analyses performed. As data uncertainty increases and all other variables remain constant, the error rate of regulatory decisions will increase. Uses of data in Clean Water Act programs will be limited by decision error, but EPA has not formally adopted decision error rate requirements for various Clean Water Act data uses. A majority of the Committee agrees that EPA should adopt decision error rates for Clean Water Act data uses relative to Detection and Quantitation Limits and that these error rates consider data uncertainty. Data uncertainty can be defined, in part, by Data Quality Indicators and Measurement Quality Objectives, but EPA also has not adopted Data Quality Indicators and Measurement Quality Objectives for data at relevant Detection and Quantitation Limits. Another approach to address data uncertainty is through the use of confidence intervals for each data point. It is also recommended that requirements for data uncertainty and the corresponding decision error rates be documented for states and EPA regional offices using data to make regulatory decisions pertaining to such activities as 303(d) listings; reasonable potential determinations; NPDES effluent limit derivation, compliance, and enforcement; development of water quality criteria, and any other uses in Clean Water Act programs.

Minority Report in Opposition to the Proposal

The EPA voted to disagree with the recommendation that EPA *Evaluate and Define Uses of Detection and Quantitation in Other Clean Water Act Programs* based on concerns about resources. The core recommendations of the Committee – pilot test the new single laboratory procedure, promulgate and implement new rules incorporating the single laboratory procedure, propose an algorithm for determining the National Quantitation Limit, and define uses of detection and quantitation in compliance and enforcement of NPDES permits – will require significant EPA resources over the next several years. At this time, EPA cannot commit additional resources to several of the other recommendations of this report, including the recommendation that EPA *Evaluate and Define Uses of Detection and Quantitation in Other Clean Water Act Programs*, until these core recommendations are implemented.

CHAPTER 7 – IMPLEMENTATION

7.1 Introduction

The Committee expects that EPA will proceed to develop proposed rules amending 40 CFR Parts 122 and 136 that implement the recommendations of the Committee. While the Committee did not reach consensus on all issues, the record of the Committee's extensive work and discussion of the issues will provide EPA with useful information as EPA considers the specifics of the proposed rules.

7.2 Further Development of the Single Laboratory Procedure

Recommendation that EPA Develop an Alternative to the Current 40 CFR Part 136, Appendix B Procedure

Although the Committee did not reach consensus on a procedure, we recommend that EPA act to develop an alternative to the current 40 CFR Part 136 Appendix B procedure. The results of the Pilot Study, and our evaluation of the DQ FAC Single Laboratory Procedure v2.4, indicate that there are deficiencies in the current 40 CFR Part 136 Appendix B procedure that can and should be corrected. The DQ FAC Single Laboratory Procedure v2.4 submitted contains elements that would be valuable to the agency in developing a new procedure.

Vote: 20 Agree, 0 Not Opposed, 0 Disagree
Approved By Consensus

Meeting #10, Decision 10.A

7.3 Additional Testing and Peer Review of the Single Laboratory Procedure

Post Committee Pilot of Proposed Procedure/s

The Committee recommends that EPA's Office of Water complete a follow up pilot study to confirm the performance of the procedure/s proposed for promulgation.

Vote: 17 Agree, 3 Not Opposed, 0 Disagree
Approved By Consensus

States: 3 Agree, 1 Not Opposed
Labs: 3 Agree, 1 Not Opposed
Industry: 4 Agree
Public Utilities: 3 Agree, 1 Not Opposed
EPA: 1 Agree
Environmental Community: 3 Agree
Meeting #10, Decision 5.E

Very early in the discussion of procedures it was agreed that the optimal detection or quantitation procedure might be a modification of one or more of the candidate procedures. Given this possibility, the Committee wanted to make it clear, if such an approach were recommended, that any procedure proposed for promulgation by EPA in the future should first be pilot tested to verify that it performed as desired.

The scope of the future pilot testing should be guided by the criteria delineated in the document, "What We Need A Procedure To Do," adopted by the Committee. Because of the extremely tight time constraints of the previous pilot testing performed under the guidance of the Committee, it was not possible to test some of the long-term and verification aspects of certain procedures. Although the Committee encourages EPA to implement its recommendations as soon as practicable, this should not result in haste that would preclude careful testing of proposed procedures to assure they perform as required because it is anticipated that these procedures will be in use for decades to come.

Peer Review of the Proposed Procedure/s

The Committee recommends that a formal peer review of the Committee recommended procedure take place.

Vote: 16 Agree, 4 Not Opposed, 0 Disagree
Approved By Consensus

States: 3 Agree, 1 Not Opposed
Labs: 3 Agree, 1 Not Opposed
Industry: 4 Agree
Public Utilities: 3 Agree, 1 Not Opposed
EPA: 1 Agree
Environmental Community: 3 Agree
Meeting #10, Decision 5.H

Consensus on this recommendation was obtained before the Committee voted on the proposed procedure/s and was thus formulated based on the assumption that the

Committee would recommend specific procedure/s. Although consensus on a procedure was not subsequently achieved, it was clearly the intent of the Committee that any procedure to be proposed should be submitted to a formal peer review.

7.4 Implementation of the New Regulations

Recommendation for EPA Development of Guidance and Outreach Materials for Stakeholders

> The Committee recommends that EPA develop guidance and outreach materials for stakeholders as EPA implements the Committee recommendations.
>
> *Vote*: 20 Agree, 0 Not Opposed, 0 Disagree
>
> Approved By Consensus
>
> Meeting #10, Decision 10.B

Recommendation for EPA Development of Guidance/Computer Applications for Determination of Detection and Quantitation Limits

> The Committee recommends that EPA develop and implement guidance on the new procedures as well as a computer-based program to assist in calculating detection and quantitation limits.
>
> Vote: 20 Agree, 0 Not Opposed, 0 Disagree
>
> Approved By Consensus
>
> Meeting #10, Decision 5.C

Implementation of the Committee recommendations represents a significant implementation challenge to EPA. A few of the many implementation issues EPA will need to consider include:

1. What should be the effective date of the new rules after promulgation? Laboratories will need time to familiarize themselves and become proficient with the new procedures and states may need time to make corresponding changes to their own regulations or guidance documents.

2. EPA will need to prioritize the creation of National Quantitation Limits, focusing on those analytes of most concern.

3. EPA will need to reach out to all parties, including its Regional offices, with guidance so that the new procedures and permitting schemes are well understood and can be implemented fairly. This will be especially challenging in the first years of the new program when EPA is essentially operating a dual system, one for analytes that do not have associated National Quantitation Limits, another for analytes with national Quantitation Limits. EPA needs to consider the most appropriate time for such guidance and some may need to be issued in parallel with the final rule.

APPENDIX A

Committee Charter

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY CHARTER

FEDERAL ADVISORY COMMITTEE ON DETECTION AND QUANTITATION APPROACHES AND USES IN CLEAN WATER ACT (CWA) PROGRAMS

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1. Committee's Official Designation (Title):

Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act (CWA) Programs

2. Authority:

This charter establishes the Federal Advisory Committee on Detection and Quantitation Approaches and Uses in CWA Programs (FACDQ) in accordance with the provisions of the Federal Advisory Committee Act (FACA), 5 U.S.C. App. 2 § 9 (c). FACDQ supports the Environmental Protection Agency (EPA) in monitoring and reporting chemical pollutants under the Clean Water Act (CWA).

The FACDQ is in the public interest and supports EPA in performing its duties and responsibilities.

3. Objectives and Scope of Activities:

EPA approves analytical methods (i.e., test procedures) used for monitoring and reporting chemical pollutants under the CWA. EPA's analytical methods specify detection limits to determine if a pollutant is present. Quantitation limits describe the concentration of a pollutant that can be measured with a known level of confidence. States, Tribes and EPA Regions that administer and enforce permit limits on direct discharges into water often use these values as reporting and compliance limits. Additionally, States and localities in administering and enforcing pretreatment programs for indirect discharges use these values. The major objectives of the FACDQ will be to provide advice and recommendations on approaches for the development of detection and quantitation procedures and uses of these procedures in CWA programs.

4. Description of Committee's Duties:

The duties of FACDQ are solely advisory in nature.

5. Official(s) to Whom the Committee Reports:

FACDQ will submit advice and recommendations and report to the EPA Administrator, through the Director, Office of Science and Technology, Office of Water.

6. Agency Responsible for Providing the Necessary Support:

EPA will be responsible for financial and administrative support. Within EPA, this support will be provided by the Office of Water.

7. Estimated Annual Operating Costs and Work Years:

The estimated annual operating cost of the FACDQ is $700K in FY05 and $350K in FY06 which includes 2.5 person-years of support in FY05 and 2.0 person-years of support in FY06.

8. Estimated Number and Frequency of Meetings:

FACDQ expects to meet approximately four (4) times a year. Meetings may occur approximately every three (3) months or as needed and approved by the Designated Federal Officer (DFO). EPA may pay travel and per diem expenses when determined necessary and appropriate. The DFO will be a full-time, or permanent part-time, employee of EPA. The DFO or a designee will be present at all meetings, and each meeting will be conducted in accordance with an agenda approved in advance by the DFO. The DFO is authorized to adjourn any meeting when he or she determines it is in the public interest to do so.

As required by FACA, FACDQ will hold open meetings unless the EPA Administrator determines that a meeting or a portion of a meeting may be closed to the public in accordance with subsection c of section 552b of Title 5, United States Code. Interested persons may attend meetings, appear before the Committee as time permits, and file comments with the FACDQ.

9. Duration and Termination:

FACDQ will be examined annually and will exist until the EPA Deputy Administrator determines the Committee is no longer needed. This charter will be in effect for two years from the date it is filed with Congress. After the initial two-year period, the charter may be renewed as authorized in accordance with Section 14 of FACA (5 U.S.C. App. 2 § 14)

APPENDIX B

History of Committee's Decisions

Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act (CWA) Programs

Hilton Alexandria Old Town, Salons A & B
1767 King Street
Alexandria, VA
Tuesday – Wednesday, June 21-22, 2005

Decisions at Meeting #1

The Committee: Committee members approved by consensus the revised ground rules for the Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act (CWA) Programs (20 Agree; 1 Absent).

Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act (CWA) Programs

FDIC Seidman Center, Rooms 203 & 205
3501 Fairfax Drive, Arlington, VA
Thursday – Friday, September 29-30, 2005

Decisions at Meeting #2

The Committee:

1. Approved, by consensus, the summary of the June 21-22 Committee meeting.
2. Adopted, by consensus, working draft definitions of terms for use in the Committee process with the understanding that the definitions would be refined as work progresses and decisions are made.
3. Developed and approved, by consensus, draft criteria to evaluate a final package of recommendations; the draft criteria will be finalized at a future Committee meeting.
4. Created a Policy Work Group to: 1) identify and define uses of detection and quantitation; 2) identify the existing situation for each use category and data quality objectives for each type of use and user; and 3) pose policy issues that emerged in carrying out their assignments.
5. Tasked the Technical Work Group with: 1) proposing an approach or approaches for conducting a pilot test, including possible purposes and objectives of the pilot test; and 2) identifying existing data sources and their possible uses in a pilot test. The group was asked to expand the definitions of the characteristics in the evaluation matrix and to add to the glossary of terms, as necessary.

Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act (CWA) Programs

FDIC Seidman Center, Rooms 203 & 205
3501 Fairfax Drive, Arlington, VA
Thursday – Friday, December 8-9, 2005

Decisions at Meeting #3

The Committee:

1. Approved, by consensus, the summary of Meeting #2, as drafted.
2. Approved changes to the description of the characteristics in the matrix, by consensus.
3. Approved, by consensus, revised goals for a final package of detection and quantitation recommendations.
4. Approved, by consensus, the draft pilot study purpose and objectives.
5. Approved, by consensus, to drop L_D for use in the single-lab pilot study.
6. Provided direction to the Technical Work Group in its further development of pilot studies requesting that the multi/inter-lab subgroup move forward with developing a pilot study design that incorporates a multi-lab study design and an inter-lab study design for the LCMRL procedure and present a draft design to the Committee at the March 2006 meeting. The Committee agreed to a stepwise pilot approach within the advisory process decision-making provisions. The term "multi-laboratory" will also be added to the glossary of terms.
7. Recommended, by consensus, further narrowed procedures for consideration in pilot testing by removing the Office of Solid Waste (OSW), ISO/IUPAC Quantitation Limit and Water Research Centre (WRC) procedures from pilot testing.
8. Agreed to the following responses to the Technical Work Group's questions related to a single-lab pilot study design:
    a. The Committee agreed that the single-lab pilot study should include both descriptive and prescriptive approaches.
    b. The Committee agreed that modification of procedures could be looked at, but that it should not be a high priority for the Technical Work Group. Most felt that changing procedures might happen after the pilot.
9. Approved, with amendments and by consensus, a framework for an interim report. The Policy Work Group was tasked with drafting the report that will be made available in time for Committee members to check with their constituencies before the March 2006 Committee meeting.

Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act (CWA) Programs (FACDQ)

FDIC Seidman Center, Rooms 203 & 205
3501 Fairfax Drive, Arlington, VA
Wednesday – Thursday, March 29-30, 2006

Decisions at Meeting #4

1. Meeting #3 Summary

The FACDQ approved by consensus the final summary of meeting #3 with amendments.
Vote: 20 Agree, 0 Not Opposed, 0 Opposed, 1 Absent

2. What We Need Procedures to Do

A. Approved, by consensus, the following list of priority characteristics (not in priority order) for evaluating procedures*:

1. Is bias explicitly derived by the procedure?
2. Is precision explicitly derived by the procedure?
3. Does the procedure provide for selection of a Type I error tolerance limit (false positive)?
4. Does the procedure provide for selection of a Type II error tolerance limit (false negative)?
5. Does the procedure require that qualitative identification take place at the determined detection and quantitation limit?
6. Does the procedure adequately represent variability in lab performance?
7. Does the procedure describe how to modify a detection or quantitation limit for applicability to real world samples?
8. Does the procedure evaluate the entire test method, including sample preparation and clean-up steps?
9. Does the procedure explicitly adjust or account for situations where method blanks always return a non-zero result/response (e.g., defects in calibration or consistent or chronic blank contamination of laboratory blanks)?
10. Does the procedure explicitly adjust or account for situations where method blanks are intermittently contaminated?
11. Is the procedure clearly written with enough detail so most users can understand and implement it?
12. Is the procedure cost-effective?
13. Is the procedure applicable to all users and test methods?
14. Does the procedure consider the differences between multi- and inter-lab approaches?

With respect to these characteristics, the Committee also agreed to the following stipulations:

1. The characteristics depend on the uses the Committee agrees to.
2. It is important to understand the specifics of the characteristics.

* For a more thorough understanding of these characteristics, please refer to the following documents: "What Does the FACDQ Need a Procedure to Do?" (document #4 from the March 29-30, 2006 advisory Committee meeting) and "Interpretation of Detection and Quantitation Procedure Evaluation Characteristics," from the December 8-9, 2005 FACDQ meeting.

3. The characteristics for the procedures need on-going verification.

Vote: 20 Agree, 0 Not Opposed, 0 Opposed, 1 Absent

B. Tasked a subgroup consisting of Richard Burrows, Tim Fitzpatrick, Michael Murray, John Phillips and Jim Pletl with incorporating comments from the five caucus groups into the narrative of what the Committee needs procedures to do. The revised narrative will be presented to the Committee in July.
Vote: 20 Agree, 0 Not Opposed, 0 Opposed, 1 Absent

3. Uses of Detection and Quantitation

Tasked a subgroup consisting of Chris Hornback, Larry LaFleur, Tom Mugan, Michael Murray and Mary Smith to develop a straw proposal on the uses of detection and quantitation approaches in Clean Water Act programs, including permit limits, compliance enforcement, data reporting, and data reporting for reasonable potential determinations. In particular, the group will develop options to address the "delta" between L_C and L_Q and other uses taking into consideration the Committee's discussion of these topics.
Vote: 20 Agree, 0 Not Opposed, 0 Opposed; 1 Absent

4. Measurement Quality Objectives

A. Agreed, for purposes of pilot testing, and by consensus, to set the false positive rate equal to or less than 1%.
Vote: 18 Agree, 1 Not Opposed, 0 Opposed, 2 Absent

B. Agreed, by consensus, that if or when data is reported below L_Q, then the data points that fall between L_C and L_Q would be reported, for example, as detected but not quantified (e.g., DNQ).
Vote: 19 Agree, 0 Not Opposed, 0 Opposed, 2 Absent

C. Agreed, by consensus, that determination of L_D is not a requirement for purposes of pilot testing, so long as data between L_C and L_Q is reported, for example, as detected but not quantified.
Vote: 19 Agree, 0 Not Opposed, 0 Opposed, 1 Absent

D. Agreed, by consensus, to set, for purposes of pilot testing, the false negative rate equal to or less than 1% measured at L_C for the true value at L_Q or L_D.
Straw vote: 12 Agree, 8 Not Opposed, 0 Opposed, 1 Absent

E. Agreed, by consensus, that the goal for the pilot test of 20% relative standard deviation (RSD) is based on the mean recovery, understanding that there will be instances where this % RSD may show conflicts with accuracy (that is, set precision targets may inherently define accuracy targets). This may not be applied universally after the pilot study is complete. The study design team will consider higher precision targets (higher % RSD) if the goal cannot be met.
Vote: 18 Agree, 1 Not Opposed, 0 Opposed, 2 Absent

F. Agreed, by consensus, that, for the pilot, the study design team will ask participating laboratories to use accuracy based on mean accuracy and that the Technical Work Group study design team should make decisions on specific goals for accuracy based on an evaluation of existing data. The study design team will ensure that the batch-by-batch data is available for the FACDQ to have

analyzed.
Vote: 16 Agree, 3 Not Opposed, 0 Opposed, 2 Absent

5. Pilot Study Design

A. Agreed, by consensus, to task the Technical Work Group and a "Study Design Team" consisting of one person from each caucus on the Technical Work Group with scoping the details of the pilot study.
Vote: 19 Agree, 1 Not Opposed, 0 Opposed, 1 Absent

B. Agreed, by consensus, to proceed with pilot testing the following five analytical methods:

- 200.7 (metals),
- 300.0 (ions),
- 625 (SOCs),
- 608 (PCBs, pesticides)
- 335.3 (cyanide)

Vote: 18 Agree, 1 Not Opposed, 0 Opposed, 2 Absent

Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act (CWA) Programs (FACDQ)

FDIC Seidman Center, Rooms 203 & 205
3501 Fairfax Drive, Arlington, VA
Thursday – Friday, July 13-14, 2006

Decisions at Meeting #5

The Committee:

1. Agreed to further refine the document describing characteristics the Committee wants in a final procedure by:

- Adding language in the introduction to read: "…the Committee generally agreed that the list of characteristics should be built with the final recommendations in mind and that those characteristics should drive the pilot study to test whether procedures met those characteristics. Committee members also generally agreed that the pilot test was an opportunity to inform the Committee's final recommendations and that some of the characteristics might be refined as a result of the pilot study data."
- Revising characteristic 5b to read: "Requiring revision of L_Q or L_D if all spikes at L_Q or L_D are not detected."
- Adding a new number 7 that would read: "Perform on-going verification of estimates. To be evaluated by:
  a) Continuously analyzing periodic blanks to assess the estimate of L_C;
  b) Continuously analyzing periodic low-level spike samples near L_Q to assess the estimate of L_Q; and
  c) Recalculating limits at a frequency that captures variability in performance relative to MQOs."
- Removing the appendix.

Vote: Agree = 19; Not Opposed = 1; Opposed = 0; Absent = 1

2. Accepted the pilot study design, excluding Attachment B, and recommended moving forward with the pilot study.
Vote: Approve = 18; Not Opposed = 1; Opposed = 0; Absent = 2

3. Agreed to send the "Features" document back to the Technical Work Group to provide more detail about what the pilot study would not do.

4. Agreed to a revised title for proposal #6 in the straw uses proposal. The new title and proposal were as follows:

- <u>Uses for 303(d) Listing</u>: Do not develop recommendations for how to use data for 303(d) listings for the following reasons:
  - 303(d) listing is a complex process that does not depend totally upon Part 136 analytical methods; it would require an effort to fully educate the Committee on this process.
  - However, if an opportunity arises to link the 303(d) listing process to uses and approaches for detection and quantitation, and if the FACDQ becomes educated

about the 303(d) listing process, then the FACDQ could revisit this issue prior to the final recommendations.

Vote: Approve = 20; Not Opposed = 0; Opposed = 0; Absent = 1

5. Agreed to postpone approving the draft summary of Meeting #4 until the next FACDQ meeting. In the meantime, another draft of the discussion surrounding the decisions on MQOs will be prepared using transcripts from the meeting. Both the transcription and redraft will be shared with a small group of representatives from the caucuses to ensure accuracy of the discussion for purposes of approving the summary at the December meeting. The Committee also agreed to include a statement about revisiting the setting of numerical MQOs after completion of the pilot study.

6. Agreed to add another meeting to the existing schedule. The new meeting will be Wednesday, December 6 – Friday, December 8, 2006, at the FDIC Seidman Center in Arlington, VA. The Committee also agreed to discuss extending the charter with Michael Shapiro and Ephraim King during their afternoon visit with the Committee on day 2.

Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act (CWA) Programs (FACDQ)

FDIC Seidman Center, Rooms 203 & 205
3501 Fairfax Drive, Arlington, VA
Wednesday – Friday, December 6-8, 2006

Decisions at Meeting #6

1. Ground Rules

Environmental Community Caucus member Rob Moore resigned; as a result, the Committee now consists of 20 members. The Committee agreed to amend the ground rules to reduce the number required for a quorum by one, from 17 to 16. The language now reads as follows: "The Committee will take no official action, such as offering advice or recommendations, with fewer than 16 participating Advisory Committee members."
Vote: 20 Agree, 0 Not Opposed, 0 Disagree

2. Meeting Summary #4

The Committee agreed to approve the summary from Meeting #4 with the revisions suggested by a subgroup convened to recommend final language.
Vote: 20 Agree, 0 Not Opposed, 0 Disagree

3. Meeting Summary #5

The Committee agreed to approve the summary from Meeting #5, with the following revisions:

- Move action box above section titled "Discussion of Data Analysis for the Pilot Study"
- Same section, third sentence, delete "…least helpful or…"
- Section titled "Discussion of Uses" under the state alternative proposal, the note for items 4 and 5 should read "…estimated value for data greater (~~less~~) than…"

Vote: 20 Agree, 0 Not Opposed, 0 Disagree

4. FACDQ Recommendations on Policy Issues (See full text on pages 13 – 16)

The Committee agreed to the general concepts outlined in the revised Recommendations on Policy Issues document and tasked the Policy Work Group with further refinements of the document. The Committee:

- Supports the intent of the policy recommendations, as revised;
- Recommends that the Policy Work Group refine the language in the recommendations per the FACDQ discussion in December, and also those items highlighted [in gray scale] in the document; and
- Recommends that the Policy Work Group bring back to the FACDQ their refinements for final decision-making.

Vote: 19 Agree, 1 Not Opposed, 0 Disagree

5. Final Report Work Group

The Committee agreed to task the Final Report Work Group with beginning work on the final report. The Committee asked the work group to begin assembling a draft of the final document, leaving placeholders where necessary, for the Committee to discuss at a future meeting.

Vote: 18 Agree, 0 Not Opposed, 0 Disagree, 2 Absent

6. Matrix Effects

The FACDQ recommends the Policy Work Group develop some guidance on the topic for the FACDQ to consider at a future meeting.
Vote: 18 Agree, 2 Not Opposed, 0 Disagree

7. Technical Work Group Assignments

The Committee agreed to assign the following tasks, in priority order, to the Technical Work Group:

- Complete the pilot results, report and recommendations for presentation to the Committee at its next meeting.
- Develop recommendations around a procedure or procedures for the Committee to consider at its next meeting.
- Develop recommendations and other details for initial and on-going verification (time permitting).
- Develop a list of existing methods and associated priorities for detection and quantitation limits (time permitting).

Vote: 20 Agree, 0 Not Opposed, 0 Disagree

8. Policy Work Group Assignments

The Committee agreed to assign the following tasks, in priority order, to the Policy Work Group:

- Complete refinements to the revised policy issues document, particularly highlighted sections.
- Develop recommendations on data quality objectives for the Committee to consider at its next meeting.
- Develop recommendations on implementation issues, using earlier one-pager (from Mary Smith) and ideas from FACDQ6 meeting.
- Develop guidance on matrix effects for the Committee to consider at a future meeting.
- Develop recommendations and other details for initial and on-going verification.
- Develop a list of existing methods and associated priorities for detection and quantitation limits.

Vote: 20 Agree, 0 Not Opposed, 0 Disagree

9. Working Definitions

The Committee agreed to table the discussion of its working definitions for a future meeting.

FACDQ Recommendations on Policy Issues
The FACDQ worked diligently at its sixth meeting in December 2006 to reconcile and reach agreement on the policy recommendations below.

The FACDQ voted on December 8, 2006 on the language that follows. EPA's votes reflect the views of the Office of Water for Clean Water Act Programs.

The FACDQ:
- supports the intent of the following policy recommendations, as revised;
- recommends that the Policy Work Group refine the language in the recommendations per the FACDQ discussion in December and also those items highlighted [in gray scale] below; and
- recommends that the Policy Work Group bring back to the FACDQ their refinements for final decision-making.

Vote: 19 Agree, 1 Not Opposed, 0 Disagree

[Note: must clarify lab-specific vs. national/state DL/QL vs. permit QL throughout the document.]

1. Lab-Determined Detection Limits (DLs) and Quantitation Limits (QLs)[1]
Recommendation: The FACDQ recommends that EPA promulgate the descriptive single-laboratory procedure recommended by the FACDQ for individual laboratories to determine their actual detection and quantitation limits. The FACDQ further recommends that this descriptive procedure replace the one currently in 40 CFR Part 136 Appendix B.

2. Method Promulgation
Recommendation: The FACDQ recommends that when the EPA promulgates future analytical methods in 40 CFR Part 136, detection limits (DLs) and quantitation limits (QLs) shall be included with the methods using the procedure recommended by the FACDQ. These limits will serve to define the minimum required performance of a laboratory, and may assist in comparing performance of one method to another (facilitating selection of a method most suitable for a given use), and may define important thresholds for use in evaluating compliance. (See the section titled "NPDES Permits and Compliance Uses.") The limits will be published in a table in a promulgated rule in 40 CFR Part 136. [2]

3. Demonstration of Laboratory Proficiency of Detection and Quantitation Limits
Recommendation: The FACDQ recommends developing a process for initial and on-going verification of DLs and QLs by laboratories. This recommendation includes the following guidance:
- The FACDQ recommended procedure (e.g., what goes into 40 CFR Part 136 Appendix B) should include the on-going demonstration (either explicitly within the procedure or as an "attachment" if the FACDQ chooses to recommend a consensus procedure).
- Separate initial vs. on-going demonstrations.
- Strive for feasibility, practicality, representativeness and cost-effectiveness.

[1] The Policy Work Group agreed to use the terms DL for detection limit and QL for quantitation limit.
[2] The Policy Work Group has agreed to incorporate a new table of promulgated detection and quantitation limits in a rule, but the Group has not had a full discussion of what would be included in the table.

4. Future Updates of Promulgated Analytical Method DLs and QLs

Recommendation: The FACDQ recommends that EPA periodically review current capabilities of promulgated analytical methods. The focus of this review should be on methods where there have been significant improvements in detection or quantitation limits or on methods that do not contain DLs or QLs. This review would be particularly important for cases where detection and quantitation limits are critical to the permit program (e.g., those required for very low WQBELs). EPA should focus on analytes for which current methods provide poor performance or do not meet program needs. Using best judgment and where resources are available, EPA shall update DL and QL limits on an on-going basis. EPA should also consider information submitted by states and/or other qualified third parties. EPA shall publish an annual Advanced Notice of Proposed Rulemaking (ANPR) announcing the DLs and QLs they propose to update.

5. Recommendations for NPDES Permits and Compliance Uses for WQBELs below QL:

Recommendation A:

The FACDQ recognizes that the existence of WQBELs at concentrations less than method QLs presents a number of NPDES-related issues. These include appropriate approaches for:

- Calculating monthly averages,
- Determining compliance with daily maximum limits and monthly average limits,
- Reporting data, and
- Appropriate compliance response in light of data uncertainty and the need for the protection of public health and the environment.

To deal with these various issues, the FACDQ recommends a balanced response as outlined below.

States that have been delegated the NPDES program from EPA have the authority under the Clean Water Act to adopt regulatory provisions that are different, but no less stringent than, those required under federal regulations. Such state-adopted provisions that would operate in lieu of the following recommendations could include a QL value lower than the nationally promulgated QL. In that case, the QL applicable under the state program would be used for determining compliance, reporting, and other applicable requirements.

i. The FACDQ recommends that a Part 136 DL and QL determined by the procedure recommended by the FACDQ be promulgated for each method/analyte combination which shall be the upper bound for lab performance. The default QL is the Part 136 promulgated value, unless states adopt an alternative but no less stringent approach. The permit must include the applicable QL. The NPDES permit must contain language that requires the use of a Part 136 method with a QL at or below the WQBEL. If no such method exists, the permit must provide that the appropriate method with the lowest QL be used. The facilities must require the lab to report lab-specific DLs and QLs as determined by the procedure recommended by the FACDQ and maintain such information for a period of at least five years. The FACDQ further recommends, for purposes of updating the Part 136 DLs and QLs, that EPA require the lab-specific information be reported in the Integrated Compliance Information System (ICIS).

[Note: This needs work in terms of implementation, particularly with respect to Part 122 but not Part 123. For example, the FACDQ needs to consider what happens when the national QL changes during the life of the permit, and whether there were suggestions from the FACDQ to address that.]

ii. Set average and daily maximum permit limits at the WQBEL.

iii. While the FACDQ recognizes that values between a given laboratory's DL and QL have a higher level of uncertainty, the science suggests they are unlikely zero. However, assigning a non-zero value where an analyte is detected but not quantified (DNQ) would have significant compliance and enforcement implications. Therefore, assign zero for values less than the permit QL when determining average and daily maximum discharge levels.

iv. To determine NPDES permit compliance, compare average and daily maximum discharge levels, calculated in accordance with item (iii.) above, to the respective WQBEL.

v. A permittee must report to the permitting authority all information in the following manner:

When reporting daily maximum sample results:

a. For values less than the DL, report "ND" (not detected) on the DMR.
b. For values greater or equal to the DL and less than the QL, report "DNQ" (detected not quantified) on the DMR.
c. For values greater than or equal to the QL, report the actual values on the DMR.

When reporting averages:

d. Where all values used to calculate an average are less than DL, report "ND" on the DMR.
e. Where all values used to calculate an average are greater than or equal to DL but less than QL, report "DNQ" on the DMR.
f. When values used to calculate an average are a combination of ND and DNQ values, report "DNQ" on the DMR.
g. When any value used to calculate an average is greater than or equal to QL, report on the DMR the average as calculated in item (iii.) above.

Additional reporting requirements:

h. Report the lab-specific DL and QL and the individual numeric result for any value that is greater than or equal to the lab-specific DL and less than the permit QL in a supplemental report.
i. The permitting authority shall report the lab-specific DL and permit QL for each analyte to EPA in ICIS.

vi. Permits shall include language that triggers additional steps when a "significant number of" (to be determined in permitting process) DNQ values are reported. These steps may include additional or accelerated monitoring, analytical studies such as matrix studies, pollutant minimization programs, or other permit conditions outside of the determination of compliance with effluent limitations. Reports under such provisions will be done outside of

the DMR reporting process, except that any additional effluent testing performed using approved analytical methods as part of the special studies must be reported according to the protocol in (v.).

Recommendation B: Current EPA guidance for implementing permit limits for WQBELs that challenge current analytical capabilities stipulates that the permit should specifically reference the most sensitive method approved in 40 CFR Part 136 and require its use to demonstrate compliance. The FACDQ recommends that EPA modify this reference to "the most appropriate method, taking into account sensitivity, selectivity and matrix effects" (i.e., "best method") and that EPA then incorporate this revised guidance into the regulation that it issues to implement the FACDQ recommendations.

6. Matrix Effects

Recommendation: The FACDQ recommends the Policy Work Group develop some guidance on the topic for the FACDQ to consider at a future meeting.

7. Other Uses to Consider

Recommendation: The FACDQ tabled the following list of additional uses:

- ambient monitoring 305(b)
- pretreatment
- non-regulatory operational monitoring
- stormwater monitoring
- other studies, such as fish tissues or biosolids characterization
- reasonable potential analysis

8. Another Issue to Consider: Alternative Test Procedures

Recommendation: The FACDQ tabled the option of developing recommendations to EPA on updating the Alternative Test Procedures (ATP) program.

9. Implementation of the FACDQ Recommendation

Recommendation: Initially, EPA would propose a new regulation that would essentially establish the recommendations of the FACDQ as regulations. This would include removing any current procedure (if that is the recommendation of the FACDQ), incorporating any recommended procedures, and making any other changes recommended by the FACDQ (e.g., new permitting regulations per our current discussion of uses).

Once those regulations are in place, the procedures would be utilized in all future EPA method development/validation work and DLs and QLs would be promulgated with all new methods. As deemed appropriate by EPA, additional Federal Register notices and rulemaking would be used to update the detection and quantitation limits.

Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act (CWA) Programs (FACDQ)

Virginian Suites
1500 Arlington Blvd.
Arlington, VA 22209
S.S. Virginian Conference Center
Wednesday – Friday, June 6-8, 2007

Decisions at Meeting #7

*Note: Highlighted votes are straw polls and not official votes taken by the Committee. All votes reflect the order they were considered and voted on during the meeting.

1. Meeting Summary #6

The FACDQ agrees to approve the summary from Meeting #6, with the following revisions: Correction of name spellings for Tim Fitzpatrick and David Piller and removal of "(except California)" from locations within the document.
***Vote*: 18 Agree, 0 Not Opposed, 0 Disagree** *(6/6/07 AM)*

2. Pilot Study Results & Draft Pilot Study Report

The FACDQ agree to use the Pilot Study results and the May 24, 2007 Draft Pilot Study Report to inform decision-making on choosing a procedure(s).
***Vote*: 15 Agree, 1 Not Opposed, 2 Disagree** *(6/6/07 AM)*

NOT APPROVED

3. DQOs Decision

The FACDQ recommends that EPA Office of Water use the *EPA Guidance on Systematic Planning Using the Data Quality Objectives Process* in all Clean Water Act (CWA) programs.
Straw Vote: 17 Agree, 1 Not Opposed, 0 Disagree *(6/6/07 PM)*
***Vote*: 17 Agree, 0 Not Opposed, 0 Disagree, 1 Absent** *(6/8/07 PM)*

4. Measurement Quality Objective (MQO) Decisions

A. False Positive Rate MQO

The FACDQ recommends that a ≤ 1% False Positive rate be used for Detection.
Straw Vote: 18 Agree, 0 Not Opposed, 0 Disagree *(6/6/07 PM)*
***Vote*: 17 Agree, 0 Not Opposes, 0 Disagree, 1 Absent** *(6/8/07 PM)*

B. Proposed Additional Language for MQOs – Future Methods

The FACDQ recommends that during the DQO process, EPA will give special attention to assuring the analytical method produces comparable results, at or near the QL_{nat}, on split samples, analyzed in different labs with the same method, and will specifically describe the steps taken in the proposed rule.
Straw Vote: 16 Agree, 1 Not Opposed, 1 Absent *(6/8/07 PM)*
***Vote*: 14 Agree, 3 Not Opposed, 0 Disagree, 1 Absent** *(6/8/07 PM)*

C. MQOs for Quantitation for Promulgated Methods

The FACDQ recommends that for promulgated methods in 40 CFR Part 136 without established MQOs, the initial MQO for Quantitation upon implementation of the new quantitation procedure is a specific False Negative rate (≤ 5%) to be implemented through a multiplier of the Detection Limit (determined by the FACDQ recommended Single Lab Procedure for Detection). The Precision and Accuracy MQOs for individual analytes/methods would be generated and promulgated, as the data to support those MQOs becomes available.

The FACDQ requests that the Technical Work Group establish or recommend a procedure to add MQOs to existing methods.
Straw Vote: 17 Agree, 0 Not Opposed, 0 Disagree, 1 Absent *(6/7/07 PM)*
***Vote*: 17 Agree, 0 Not Opposed, 0 Disagree, 1 Absent** *(6/8/07 PM)*

D. Limits for QL MQOs for Future Promulgation of New or Updated Methods

The FACDQ recommends the Technical Work Group develop recommendations for target MQO bounds for compliance and enforcement that define Quantitation. The TWG will bring these recommendations back to the FACDQ.
For example:

A. Precision ≤ 30% RSD
B. Accuracy (measured as recovery for single determination) = 20-180%
C. False Negative rate ≤ 10%
D. Ratio of Accuracy to Precision must be no less than 1.0
   Example: 40% Recovery / 20% RSD = 2 O.K.,
   Example: 20% Recovery / 30% RSD = .66 Not Acceptable

Straw Vote: 13 Agree, 5 Not Opposed, 0 Disagree *(6/8/07 PM)*
***Vote*: 12 Agree, 5 Not Opposed, 0 Disagree, 1 Absent** *(6/8/07 PM)*

E. MQO Bounds

The FACDQ recommends that EPA establish quantitative MQO bounds for relevant Data Quality Indicators (DQIs) that define Quantitation for intended CWA uses. These bounds would be offered for public comment by EPA.
Straw Vote: 13 Agree, 4 Not Opposed, 1 Disagree *(6/8/07 PM)*
***Vote*: 9 Agree, 7 Not Opposed, 1 Disagree, 1 Absent** *(6/8/07 PM)*

NOT APPROVED

F. MQOs for Future Promulgation of Methods

The FACDQ recommends, for future method promulgation, that target MQOs for DQIs, such as Precision, Accuracy, Method Specified Qualitative Identification, and False Negative error rates derived from the DQO process, be established for Quantitation Limits in Part 136. If the target MQOs cannot be met, EPA may promulgate with rationale.
Straw Vote: 9 Agree, 9 Not Opposed, 0 Disagree *(6/8/07 AM)*

The FACDQ recommends, for future method promulgation, that target MQOs for Precision and Accuracy derived from the DQO process be established for QLs in Part 136. In addition, DQIs such as method specified quality identification and False Negative error rate would be considered. If the target MQOs cannot be met, EPA may promulgate with rationale.

Straw Vote: 9 Agree, 5 Not Opposed, 4 Disagree *(6/8/07 AM)*

5. Multi/Inter Lab Approaches

A. The FACDQ asks the Technical Work Group to develop a recommended process for determining a QL_{nat}.
Straw Vote: 18 Agree, 0 Not Opposed, 0 Disagree *(6/8/07 AM)*
***Vote*: 17 Agree, 0 Not Opposed, 0 Disagree, 1 Absent** *(6/8/07 PM)*

B. The FACDQ recommends that EPA promulgate how QL_{nat} is derived.
Straw Vote: 10 Agree, 6 Not Opposed, 2 Disagree *(6/8/07 AM)*
Straw Vote: 10 Agree, 7 Not Opposed, 1 Absent *(6/8/07 AM)*
***Vote*: 7 Agree, 10 Not Opposed, 0 Disagree, 1 Absent** *(6/8/07 PM)*

C. The FACDQ recommends that EPA develop a procedure for establishing a QL_{nat} using the framework identified by the FACDQ. The Technical Work Group will develop this framework for FACDQ consideration.
Straw Vote: 6 Agree, 10 Not Opposed, 1 Disagree, 1 Abstained *(6/8/07 AM)*

D. The FACDQ asks the Technical Work Group to develop a recommended procedure(s) for determining QL_{nat}.
Straw Vote: 16 Agree, 1 Not Opposed, 1 Disagree *(6/8/07 AM)*

E. The FACDQ recommends that EPA establish after public comment how QL_{nat} is derived.
Straw Vote: 9 Agree, 4 Not Opposed, 5 Disagree *(6/8/07 AM)*

F. The FACDQ recommends that EPA develop a Multi Lab Procedure for establishing a QL_{nat} using the framework identified by the FACDQ. The Technical Work Group will develop this framework for FACDQ consideration.
Straw Vote: 0 Agree, 1 Not Opposed, 13 Disagree, 4 Abstained *(6/8/07 AM)*

G. The FACDQ asks the Technical Work Group to develop a recommendation for a process that considers both Multi and/or Inter Lab Procedures in developing a QLnat.
Straw Vote: 13 Agree, 3 Not Opposed, 1 Disagree, 1 Absent *(6/8/07 AM)*

6. Recommendations on Procedures

A. The FACDQ recommends the Technical Work Group continue to develop the specifics for the following:

Single Laboratory Detection Limit Procedure
The ACIL Procedure, with modifications indicated by the Pilot Study results and informed by concepts from the Consensus Group and LabQC Procedures, is recommended for a Single Laboratory Detection Limit Procedure.
***Vote*: 17 Agree, 1 Not Opposed, 0 Disagree** *(6/8/07 AM)*

B. The FACDQ recommends the Technical Work Group continue to develop the specifics for the following:

Single Laboratory Quantitation Limit Procedure
The ACIL Procedure, with modifications indicated by the Pilot Study results and informed by concepts from the Consensus Group and Lab QC procedures, as well as decisions by the FACDQ at its June 2007 meeting.
***Vote*: 16 Agree, 2 Not Opposed, 0 Disagree** *(6/8/07 AM)*

7. Uses Decisions

A. DL_{nat}

The FACDQ recommends the Policy Work Group explore the deletion of DL_{nat}, the possible policy changes to the document, and their implications for bringing back to the FACDQ. The Policy Work Group will also explore other policy issues not completed at the June 2007 meeting.
Straw Vote: 15 Agree, 3 Not Opposed, 0 Disagree *(6/807 PM)*
***Vote*: 16 Agree, 1 Not Opposed, 0 Disagree, 1 Absent** *(6/8/07 PM)*

B. Uses Document

The FACDQ directs the FACDQ Work Groups to use the straw vote decisions as a starting point for writing the Uses portion of the Final Report and other activities subject to revisions based on a final vote to occur later.
***Vote*: 16 Agree, 0 Not Opposed, 0 Disagree, 2 Absent** *(6/8/07 PM)*

- ***A subscript "nat" is used to designate the nationally-promulgated DL or QL – DL_{nat} or QL_{nat}***
- ***A subscript "lab" is used to designate the laboratory-specific DL or QL – DL_{lab} or QL_{lab}***
- ***A subscript "per" is used to designate the permit-specified QL – QL_{per}***
- ***A subscript "st" is used to designate the state-optional DL or QL – DL_{st} or QL***

> The FACDQ agreed to allow EPA come up with a new acronym for a situation where an analyte is detected below the QL_{per}. The acronym will replace "DNQ" and must fit into the conditions of the ICIS system. The facilitator used the acronym "DBQp" for purposes of completing this document. *(6/7/07 PM)*

1. Lab-Determined Detection Limits (DL_{lab}s) and Quantitation Limits (QL_{lab}s)

Recommendation: The FACDQ recommends that EPA promulgate the descriptive single-laboratory procedure(s) recommended by the FACDQ for individual laboratories to determine their Detection and Quantitation Limits. The procedure(s) should have the following two capabilities:

a. Demonstrate the lab's performance at a specified level.

b. Determine the lowest possible value achievable by the lab.

The FACDQ further recommends that the descriptive procedure(s) replace the one currently in 40 CFR Part 136 Appendix B.

2. Method Promulgation

Recommendation: The FACDQ recommends that when the EPA promulgates future analytical methods in 40 CFR Part 136, Detection Limits (DL_{nat}s) and Quantitation Limits (QL_{nat}s) shall be included with the methods using the procedure(s) recommended by the FACDQ.

> The FACDQ agreed to remove all language referring to a published table of limits in a promulgated rule in 40 CFR Part 136 as well as the pre-existing footnote. *(6/7/07*

> The FACDQ also agreed to remove the following language though it was agreed that the Final Report Work Group would keep it under consideration when drafting an introductory paragraph: "These limits will serve to define the minimum required performance of a laboratory and may assist in comparing performance of one method to another (facilitating selection of a method most suitable for a given use), and may define important thresholds for use in evaluating compliance. (See the section titled "NPDES Permits and Compliance Uses, Recommendation 5.A & B")." *(6/7/07 AM)*

3. Verification of Laboratory Proficiency of Detection and Quantitation Limits

Recommendation: The FACDQ recommends developing a process for initial and on-going verification of DL_{lab}s and QL_{lab}s by laboratories. This recommendation includes the following guidance:

- The FACDQ recommended procedure (e.g., what goes into 40 CFR Part 136 Appendix B) should include on-going verification of DL_{lab} and QL_{lab} (either explicitly within the procedure or as an "attachment" if the FACDQ chooses to recommend a consensus procedure)
- Meeting MQOs for use
- Separate initial vs. on-going verifications
- Strive for feasibility, practicality, representativeness, and cost-effectiveness

> The FACDQ agreed to replace "demonstration" from this section with the word "verification" and to strike the pre-existing footnote and to add the bullet: "Meeting MQOs for use." *(6/7/07 AM)*

4. Future Updates of Promulgated Analytical Method *DL_{nat}s* and *QL_{nat}s*

Recommendation: The FACDQ recommends that EPA periodically review current capabilities of promulgated analytical methods. The focus of this review should be on methods where there have been significant improvements in Detection or Quantitation Limits or on methods that do not contain DL_{nat}s or QL_{nat}s. This review would be particularly important for cases where Detection and Quantitation Limits are critical to the permit program (e.g., those required for very low WQBELs). EPA should focus on analytes for which current methods provide poor performance or do not meet program needs. Using best judgment and where resources are available, EPA shall update DL_{nat} and QL_{nat} limits on an on-going basis. EPA should also consider information submitted by states and/or other qualified third parties. EPA shall publish a Federal Register Notice announcing the DL_{nat}s and QL_{nat}s it proposes to

update. Provisions later in this document are for the purpose of providing EPA with robust data sets for updating and or creating DL_{nat}s and QL_{nat}s.

The FACDQ agreed to leave "4." as it is with the understanding that "shall" (…EPA shall update DL_{nat} and QL_{nat} limits on an on-going basis.) will remain. *(6/7/07 AM)*

5. **The FACDQ recognizes that the existence of WQBELs at concentrations less than quantitation limits presents a number of NPDES-related issues.** These include appropriate approaches for:
   - Calculating monthly averages
   - Determining compliance with daily maximum limits and monthly average limits
   - Reporting data, and
   - Appropriate compliance response in light of data uncertainty and the need for the protection of public health and the environment.

   To deal with these various issues, the FACDQ recommends a balanced response as outlined below.

   States that have been delegated the NPDES program from EPA have the authority under the Clean Water Act to adopt regulatory provisions that are different, but no less stringent than, those required under federal regulations. Such provisions, if authorized or not prohibited by state law, would operate in lieu of the following recommendations and could include a QL_{st} value lower than the nationally promulgated QL_{nat}. In that case, the QL_{st} applicable under the state program would be used for determining compliance, reporting, and other applicable requirements.

   A. Recommendations for NPDES Permits and Compliance Uses where a QL_{nat} exists and for WQBELs at concentrations less than QL_{nat}. If the permitting authority requires use of a method more sensitive than the method for which a QL_{nat} exists, go to section B:

The FACDQ agreed to include the following language: "If the permitting authority requires use of a method more sensitive than the method for which a QL_{nat} exists, go to section B."
Straw Poll: 14 Agree, 4 Not Opposed, 0 Disagree *(6/7/07 PM)*

1) The FACDQ recommends that a Part 136 DL_{nat} and QL_{nat} determined by the procedure recommended by the FACDQ be promulgated for each method/analyte combination which shall be the upper bound for lab performance. The regulator shall insert QL_{per}s in permit or in rule as appropriate. The default QL_{per} is the lowest Part 136 promulgated QL_{nat}. The regulator would then consider whether the method associated with this QL_{nat} is the most appropriate method considering sensitivity, selectivity, and/or matrix effects and adjust the QL_{per} accordingly.

The FACDQ agreed not to include the following language: "All the following does not apply if the QL_{nat} is not the most sensitive method $QL_{nat.}$"
Straw Poll: 8 Agree, 8 Not Opposed, 2 Disagree

The FACDQ agreed to the following language: "…the method associated with this QL_{nat} is the most appropriate method considering sensitivity…"
Straw Vote: 18 Agree, 0 Not Opposed, 0 Disagree *(6/7/07 PM)*

The FACDQ agreed to the following language: The regulator shall insert QL_{per}s in permit or in rule as appropriate.
Straw Vote: 15 Agree, 3 Not Opposed, 0 Disagree *(6/7/07 PM)*

2) The permit shall also contain a condition that the permittee's QL_{lab} shall be at or below the $QL_{per.}$ The permit shall require permittees to report DL_{lab}s and QL_{lab}s as determined by the procedure recommended by the FACDQ and maintain such information for a period of at least five years.

The FACDQ agreed to remove the following language: "The QL_{per} shall be applicable for the term of the permit unless the regulator reopens and modifies the permit" as well as #3 with the two options regarding the life of the permit.
Straw Vote: 9 Agree, 9 Not Opposed, 0 Disagree *(6/7/07 PM)*

3) For a list of analytes as defined by EPA, the permittee shall ensure that the DL_{lab}s and QL_{lab}s are determined using the steps of the procedure to determine the lowest possible value by the lab for setting QL_{lab}s and DL_{lab}s.

The FACDQ agreed on the following language:
3) For a list of analytes as defined by EPA, the permittee shall ensure that the DL_{lab}s and QL_{lab}s are determined using the steps of the procedure to determine the lowest possible value by the lab for setting QL_{lab}s and DL_{lab}s.
Straw Vote: 10 Agree, 8 Not Opposed, 0 Disagree *(6/7/07 PM)*

4) The FACDQ further recommends, for purposes of updating Part 136 DL_{nat}s and QL_{nat}s, that EPA require the lab-specific information be reported in the Integrated Compliance Information System (ICIS).

The FACDQ agreed to return to the option of deleting the new **4)** if it is found to be duplicative in later sections of the document. *(6/7/07 PM)*

5) Implementation in NPDES Permits:
- **a)** Set average and daily maximum permit limits at the WQBEL.
- **b)** Assign zero for values less than the permit QL_{per} when determining average and daily maximum discharge levels.

The FACDQ agreed to rename the title of the new section 5 from: ***"Recommendation for NPDES Permits and Compliance Uses for WQBELs when QL_{nat}s do exist" to*** "Implementation in NPDES Permits." *(6/7/07 PM)*

Rationale: While the FACDQ recognizes that values between a given laboratory's DL_{lab} and QL_{lab} have a higher level of uncertainty, the science suggests they are unlikely to be zero. However, assigning a non-zero value where an analyte is detected below the QL_{per} (DBQp) would have significant compliance and enforcement implications. Therefore, the Committee recommends assigning a zero in these cases.

The FACDQ agrees on the following language:
Note: The FACDQ agrees that this rationale concept is important and will be included in the Final Report.
Straw Vote: 18 Agree, 0 Not Opposed, 0 Disagree *(6/7/07 PM)*

c) To determine NPDES permit compliance, compare average and daily maximum discharge levels, calculated in accordance with item (d.ii.) below, to the respective WQBEL.

The FACDQ agreed to change "above" to "below." *(6/7/07 PM)*

d) A permittee must report to the permitting authority all information in the following manner:

i) When reporting daily maximum sample results:
   a. For values less than the DL_{lab}, report "ND" (not detected) on the DMR.
   b. For values greater or equal to the DL_{lab} and less than the QL_{per}, report "DBQp" (detected below QL_{per}) on the DMR.
   c. For values greater than or equal to the QL_{per}, report the actual values on the DMR.

ii) When reporting averages:
   a. Where all values used to calculate an average are less than DL_{lab}, report "ND" on the DMR.
   b. Where all values used to calculate an average are greater than or equal to DL_{lab} but less than QL_{per}, report "DBQp" on the DMR.
   c. When values used to calculate an average are a combination of ND and DBQp values, report "DBQp" on the DMR.
   d. When any value used to calculate an average is greater than or equal to QL_{per}, report on the DMR the average as calculated in item (5.A.5.b) above.

The FACDQ agrees that DL_{lab} will remain in **i.** and **ii.** With the proviso that there will be consideration of this post the MQO discussion.
Straw Vote: 15 Agree, 3 Not Opposed, 0 Disagree *(6/7/07 PM)*

iii) Additional reporting requirements:

a. The regulator shall require that the permittee report the DL_{lab} and QL_{lab} (for purposes of updating methods and to determine compliance with the conditions of the permit.) The permitting authority shall report the DL_{lab}, $QL_{lab,}$ and QL_{per} for each analyte to EPA in ICIS.

b. The regulator may require the individual numeric result for any value that is greater than or equal to the DL_{lab} and less than the QL_{per} be reported in a supplemental report.

The FACDQ agreed to the remove the second sentence in **iii.b**: "Potential uses would be to determine reasonable potential and for public knowledge."
Straw Vote: 17 Agree, 0 Not Opposed, 0 Disagree, 1 Absent *(6/7/07 PM)*

c. The permittees shall maintain individual numeric results for a period of at least five years.

6) Permits shall include language that triggers additional steps when a "significant number of" (to be determined in the permitting process) DBQp values are reported. These steps may include additional or accelerated monitoring, analytical studies such as matrix studies, pollutant minimization programs, or other permit conditions outside of the determination of compliance with effluent limitations. Reports under such provisions will be done outside of the DMR reporting process, except that any additional effluent testing performed using approved analytical methods as part of the special studies must be reported according to the protocol in (5.A.5.d.iii).

B. Recommendations for NPDES Permits and Compliance Uses for WQBELs when no QL_{nat} exists:

1) In the absence of QL_{nat}, the permitting authority is free to establish it's method for determining compliance for analytes that have limits/water quality standards at a level lower than that which can be detected and/or quantified.

2) For a list of analytes as defined by EPA, the permittee shall ensure that the DL_{lab}s and QL_{lab}s are determined using the steps of the procedure to determine the lowest possible value by the lab for setting QL_{lab}s and DL_{lab}s.

The FACDQ agreed to **1)** and **2)**
Straw Vote: 17 Agree, 1 Not Opposed, 0 Disagree *(6/7/07 PM)*

3) The FACDQ further recommends, for purposes of developing Part 136 DL_{nat}s and QL_{nat}s, that EPA require the lab-specific information be reported in the Integrated Compliance Information System (ICIS).
Note: The FACDQ recommends that EPA reconsider the usefulness of this requirement after time.

The FACDQ agreed to the following language:
3) The FACDQ further recommends, for purposes of developing Part 136 DL_{nat}s and QL_{nat}s, that EPA require the lab-specific information be reported in the Integrated Compliance Information System (ICIS).
Note: The FACDQ recommends that EPA reconsider the usefulness of this requirement after time.
Straw Vote: 18 Agree, 0 Not Opposed, 0 Disagree *(6/7/07 PM)*

7. Other Uses to Consider

Recommendation: The FACDQ tabled the discussion on recommendations regarding the use of Detection and Quantitation for other uses including but not limited to the following:

- ambient monitoring 305(b)
- pretreatment
- non-regulatory operational monitoring
- stormwater monitoring
- other studies, such as fish tissues or biosolids characterization
- reasonable potential analysis
- effluent guidelines development
- limit derivation
- development of water quality criteria

The FACDQ agreed to the language in the section "Other Uses to Consider."
Straw Vote: 17 Agree, 0 Not Opposed, 0 Disagree, 1 Absent *(6/7/07 PM)*

8. Alternative Test Procedures

Recommendation: The FACDQ tabled the option of developing recommendations to EPA on updating the Alternative Test Procedures (ATP) program. The FACDQ recommends that the ATP program be updated to be consistent with recommendations in this document.

The FACDQ agreed to the language in the section "Alternative Test Procedures."
Straw Vote: 18 Agree, 0 Not Opposed, 0 Disagree *(6/7/07 PM)*

9. Great Lakes Initiative (GLI)

Recommendation: ***The FACDQ recommends that FACDQ recommendations should not supersede the current GLI provisions. There is no significant conflict between the anticipated FACDQ recommendations and the GLI.***

The FACDQ agreed to the language in the section "GLI."
Straw Vote*:* 18 Agree, 0 Not Opposed, 0 Disagree (6/8/07 PM)

8. Matrix Effects (Use 6.)

The FACDQ recommends that EPA consider how Matrix Effects impact Detection and Quantitation. The FACDQ requests that the Policy Work Group bring back a conceptual recommendation including details to be considered.

***Vote*: 17 Agree, 1 Not Opposed, 0 Disagree** *(6/8/07 PM)*

9. Implementation of the FACDQ Recommendation

The FACDQ recommends "9. Implementation of the FACDQ Recommendation" be removed from the Uses Document for consideration by a work group. However, the importance of these issues related to Uses should not be separated. A work group of the FACDQ is tasked with bringing recommendations on the implementation issues back to the FACDQ.

***Vote*: 18 Agree, 0 Not Opposed, 0 Disagree** *(6/8/07 PM)*

Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act (CWA) Programs (FACDQ)

Teleconference Meeting
1-866-299-3188
202-566-1045#

July 25, 2007, 1 PM to 4 PM EDT

Decisions at Meeting #8

1. Removal of DL_{nat}

The FACDQ approves the removal of DL_{nat} from the Revised Uses document.

***Vote*: 16 Agree, 2 Not Opposed, 1 Disagree**

NOT APPROVED

2. Uses Recommendation on MQOs for Future Promulgation of Methods

The FACDQ recommends, for future method promulgation, that target MQOs for Data Quality Indicators (DQIs), such as Precision, Accuracy, Method Specified Qualitative Identification, and False Negative error rates derived from the Data Quality Objectives (DQO) process, be established for Quantitation Limits in Part 136. If the target MQOs cannot be met, EPA may promulgate with rationale.

Straw Vote: 9 Agree, 9 Not Opposed, 0 Disagree *(6/8/07 AM)*

***Vote*: 16 Agree, 2 Not Opposed, 1 Disagree**

NOT APPROVED

Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act (CWA) Programs (FACDQ)

Teleconference Meeting
1-866-299-3188
202-566-1045#

August 28, 2007, 1 PM to 4 PM EDT

Decisions at Meeting #9

NONE

Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act Programs
Meeting #10

FDIC Seidman Center, Rooms 203 & 205
3501 Fairfax Drive, Arlington, VA
Wednesday – Friday, September 19-21, 2007

Decisions at Meeting #10

*Note: Shaded votes are straw polls and not official votes taken by the Committee. The presentation reflects the order the recommendations were considered and voted on during the meeting. In Committee decision-making at this meeting, EPA voted as the Office of Water.

1. Ground Rules Amendment

The FACDQ agrees to amend the ground rules to include the following new and modified language: In the absence of consensus, the Committee will report its results as follows:

If the Committee is evenly split, the Committee will report different perspectives held on the issue, the rationale behind the perspectives, and the number of votes cast for each perspective.

If the voting tally shows a clear majority/minority split, the Committee will report the majority position with perspectives and rationale and the number of votes cast and the minority position with perspectives and rationale and the number of votes cast.

***Vote*: 20 Agree, 0 Not Opposed, 0 Disagree** *(9-19-07)*

APPROVED

2. Meeting Summary #8

The FACDQ agrees to approve the meeting summary of Meeting #8 with the added language regarding the following notes:

- That no transcript was prepared from this meeting
- That all perspectives offered at the meeting are not reflected in the meeting summary.

***Vote*: 20 Agree, 0 Not Opposed, 0 Disagree** *(9-19-07)*

APPROVED

3. Meeting Summary #9

The FACDQ agrees to approve the meeting summary of Meeting #9 with the added language regarding the following notes:

- That no transcript was prepared from this meeting
- That all perspectives offered at the meeting are not reflected in the meeting summary.

***Vote*: 20 Agree, 0 Not Opposed, 0 Disagree** *(9-19-07)*

APPROVED

4. Uses Recommendations

A. Use #6 - Great Lakes Initiative (GLI)

The FACDQ agrees to approve Use #6 - Great Lakes Initiative (GLI) of the Uses Document

as follows:
Recommendation: The FACDQ recommends that the FACDQ recommendations should not supersede the current Great Lakes Initiative provisions. The FACDQ believes that there is not a significant conflict between the FACDQ recommendations and the Great Lakes Initiative.
***Vote*: 20 Agree, 0 Not Opposed, 0 Disagree** *(9-19-07)*
APPROVED

B. Use #7 - Other Uses to Consider
The FACDQ agrees to approve Use #7 - Other Uses to Consider of the Uses Document as follows:
Decision: The FACDQ tabled the discussion on specific recommendations regarding the use of detection and quantitation for other uses including, but not limited to, the following:

- ambient monitoring 305(b)
- pretreatment
- non-regulatory operational monitoring
- stormwater monitoring
- other studies, such as fish tissues or biosolids characterization
- reasonable potential analysis
- effluent guidelines development
- limit derivation
- development of water quality criteria

***Vote*: 20 Agree, 0 Not Opposed, 0 Disagree** *(9-19-07)*
APPROVED

C. Use #8 - Alternative Test Procedures
The FACDQ agrees to approve Use #8 - Alternative Test Procedures of the Uses Document as follows:
Recommendation: The FACDQ did not develop specific recommendations to EPA on updating the Alternative Test Procedures (ATP) Program. The FACDQ, however, does recommend that the ATP Program be updated to be consistent with recommendations from this document.
***Vote*: 20 Agree, 0 Not Opposed, 0 Disagree** *(9-19-07)*
APPROVED

D. Moving Use #1-#3 from the Uses Document
The FACDQ agrees to remove Uses #1-#3 from the Uses Document.
***Vote*: 20 Agree, 0 Not Opposed, 0 Disagree** *(9-20-07)*
APPROVED

E. ICIS Language
The FACDQ agrees to remove the following language from two places in Use #5 in the Uses Document:
"for purposes of updating 40 CFR Part 136 National Quantitation Limits."
Vote*: 16 Agree (Dave A., Bob A., Tim F., Tom M., Steve B., Richard B., Nan T., Roger C., Larry L., John P., Dave P., David K., Michael M., Rick R., Barry S., Mary S.***), 3 Not Opposed (***Cary J., Chris H., Jim P.***), 0 Disagree, 1 Absent (***Zonetta E.***)** *(9-20-07)*

APPROVED

F. Promulgation of QL_{nat}

The FACDQ recommends that EPA promulgate a QL_{nat} with the following minimum requirements:

a. EPA will use the DQO process to set MQO target MQOs for NPDES permit compliance testing.
b. A minimum of 6-7 labs.
c. Data collected at a minimum over 3- 6 months.
d. A minimum of 20 QL spikes used in the calculation of each single lab limit.
e. The data and lab be evaluated for validity prior to acceptance.
f. An appropriate outlier test is then applied to the dataset.
g. Evaluate the data for normality, using standard statistical tests.
h. If the data is normally distributed then calculate the upper 95% confidence limit, which becomes the $QL_{nat.}$
i. If the data is non-normally distributed then the 95th percentile QL_{lab} becomes the $QL_{nat.}$
j. EPA should then promulgate the newly calculated QL_{nat}.

***Straw Vote*: 8 Agree, 10 Not Opposed, 1 Disagree, 1 Abstain** *(9-20-07)*

G. Promulgation of QL_{nat}s for Existing and Future Methods (Formerly Use #4)

The FACDQ recommends that:

a. QL_{nat}'s be promulgated in a Part 122 table by analyte
b. EPA generate QL_{nat}s as rapidly as possible so that recommendation #TBD (current section 5 of the Uses Document) can be fully implemented.
c. QL's be promulgated only using the nationally promulgated approach.
d. Methods may be promulgated without promulgating a QL for that method. As new methods are proposed without a promulgated QL, data (eg: Single Lab Detection, Single Lab Quantitation, etc.) showing demonstrated method performance should be included in the method. The methods should include a statement that these performance levels are guidance and may not always be achievable.

Vote*: 16 Agree, 4 Not Opposed (Cary J., Nan T., Zonetta E., Chris H.***), 0 Disagree** *(9-20-07)*

APPROVED

H. Promulgation of QLs

The FACDQ recommends the following criteria be considered when EPA proposes the procedure for determining a QL:

a. EPA will use the DQO process to set target MQOs for NPDES permit compliance testing.
b. A minimum of 6-7 labs.
c. Data collected at a minimum over 3- 6 months.
d. A minimum of 20 QL spikes used in the calculation of each QL_{lab}.
e. The data and lab be evaluated for validity prior to acceptance.
f. An appropriate outlier test is then applied to the dataset.
g. Evaluate the data for normality, using standard statistical tests.
h. If the data is normally distributed then calculate the upper 95% confidence limit, which becomes the QL.
i. If the data are non-normally distributed then the 95th percentile QL_{lab} becomes the QL.

Vote*: 9 Agree (Tom M., Roger C., Larry L., John P., Dave P., Zonetta E., Chris H., David K., Jim P.***), 8 Not Opposed (***Dave A., Bob A., Steve B., Richard B., Cary J., Nan T., Michael M., Rick R.***), 1 Disagree (***Mary S.***), 2 Absent (***Tim F., Barry S.***) (9-20-07)***

NOT APPROVED

I. Use #5 Setting Permit Conditions, Reporting and Using Data, and Determining Compliance When the Water Quality Based Effluent Limit (WQBEL) is Less Then Detection and Quantitation Capabilities of Existing Methods

The FACDQ recommends that EPA implement Section #5 of the Uses Document as follows:

Recommendation: The FACDQ recommends that the following recommendations be incorporated into 40 CFR Part 122, as appropriate.

A. Recommendations for NPDES Permit and Compliance Uses When a National Quantitation Limit Exists

If the permitting authority requires use of a method more sensitive than the method for which a QL_{nat} exists, go to section B.

1) **Permit Requirements Related to Detection and Quantitation**

Recommendation: The FACDQ recommends the following be required where EPA has promulgated a National Quantitation Limit in 40 CFR Part 122:

a) The default quantitation limit to be included in the permit or in rule as appropriate (Permit Quantitation Limit) is the Part 122 promulgated National Quantitation Limit unless the regulator determines that the Permit Quantitation Limit should be adjusted to account for sensitivity, selectivity, and/or matrix effects;

b) The permit shall contain a condition that the quantitation limit determined by the permittee's laboratory (Laboratory Quantitation Limit) shall be at or below the Permit Quantitation Limit. The permittee's laboratory may use any Part 136 method for which they can demonstrate a Laboratory Quantitation Limit at or below the Permit Quantitation Limit. If matrix effects have been given special attention in the permit then they would also have to be considered in compliance and enforcement.

c) The permit shall require the permittee to report the detection limit (Laboratory Detection Limit) and the Laboratory Quantitation Limit and maintain such information for a period of at least five years;

d) The permit shall require the permittee to maintain individual numeric results for a period of at least five years. The regulator may require the individual numeric result for any value that is greater than or equal to the Laboratory Detection Limit and less than the Permit Quantitation Limit be reported in a supplemental report.

e) The permit shall require that the Laboratory Detection Limit and the Laboratory Quantitation Limit be determined using the steps of the 40 CFR Part 136 procedure to establish the lowest possible value by the laboratory;

f) That EPA require the Laboratory Detection Limit, the Laboratory Quantitation Limit, and the Permit Quantitation Limit be reported by the regulator to the Integrated Compliance Information System (ICIS).

2) **Establishing Compliance Thresholds and Determining Compliance**

Recommendation: The FACDQ recommends the following be required where EPA has promulgated a National Quantitation Limit in 40 CFR Part 122:

a) Regulators will set average and daily maximum permit limits at the WQBEL.

b) Permittees must report to the regulator all information in the following manner on the Discharge Monitoring Report (DMR):

- **i)** To report daily maximum sample results:
  - a. For values not detected at the Laboratory Detection Limit, report "not detected."
  - b. For values detected at the Laboratory Detection Limit but less than the Permit Quantitation Limit, report "detected less than the Permit Quantitation Limit."
  - c. For values greater than or equal to the Permit Quantitation Limit, report the actual numeric values.
- **ii)** To report average sample results:
  - a. When all values used to calculate an average are not detected at the Laboratory Detection Limit, report "not detected."
  - b. When all values used to calculate an average are "detected less than Permit Quantitation Limit," report "detected less than the Permit Quantitation Limit."
  - c. When values used to calculate an average are a combination of "not detected" and "detected less than the Permit Quantitation Limit," report "detected less than the Permit Quantitation Limit."
  - d. When any value used to calculate an average is greater than or equal to the Permit Quantitation Limit, report the calculated numeric average after assigning zero to any individual value reported either as "not detected" or "detected less than the Permit Quantitation Limit."

c) To determine NPDES permit compliance with results reported on the DMR, regulators will:

- **i)** Determine that any daily maximum or monthly average results reported as either "not detected" or "detected less than the Permit Quantitation Limit" are in compliance with the effluent limitation.
- **ii)** Compare any numeric results directly to the WQBEL

3) **Additional Permit Requirements**

Recommendation: The FACDQ recommends the following be required where EPA has promulgated a National Quantitation Limit in 40 CFR Part 122: Permits shall include language that triggers additional steps when a "significant number" (to be determined in permitting process) of values detected at the Laboratory Detection Limit but less than the Permit Quantitation Limit are reported. These steps may include additional or accelerated monitoring, analytical studies such as matrix studies, pollutant minimization programs, or other permit conditions outside of the determination of compliance with effluent limitations. Reports under such provisions will be done outside of the DMR process, except that any additional effluent testing

performed using approved analytical methods as part of the special studies must be reported on the DMR.

B. Recommendations for NPDES Permits and Compliance Uses When No National Quantitation Limit Exists, or if the Permitting Authority Requires a Permit Quantitation Limit lower than the National Quantitation Limit.
Recommendations:

1) In the absence of a National Quantitation Limit, the permitting authority is free to establish its process for determining compliance for analytes that have limits/water quality standards at a level lower than that which can be detected and/or quantified.
2) For a list of analytes as defined by EPA, the permit shall require that the Laboratory Detection Limit and the Laboratory Quantitation Limit be determined using the steps of the 40 CFR Part 136 procedure to establish the lowest possible value by the laboratory;
3) That EPA require the Laboratory Detection Limit and the Laboratory Quantitation Limit and the Permit Quantitation Limit be reported by the regulator to the Integrated Compliance Information System (ICIS).

Vote*: 12 Agree (Dave A., Bob A., Tom M., Roger C., Larry L., John P., Dave P., Zonetta E., Chris H., David K., Jim P., Mary S.***), 4 Not Opposed (***Tim F., Richard B., Nan. T., Cary J.***), 4 Disagree (***Steve B., Michael M., Rick R., Barry S.***)** *(9-21-07)*

NOT APPROVED

5. Additional Recommendations

A. Additional Recommendation #3

The FACDQ agrees to approve the following Additional Recommendation:
"EPA continue to act as the national lead for Clean Water Act (CWA) programs in developing analytical methods and setting the performance standards for those methods."
***Vote*: 20 Agree, 0 Not Opposed, 0 Disagree** *(9-19-07)*

APPROVED

B. Additional Recommendation #4

The FACDQ agrees to approve the following Additional Recommendation:
"EPA evaluate the federal resources dedicated to developing analytical methods with detection/quantitation limits of sufficient quality (i.e., meet data quality objectives) and capable of meeting the needs of CWA programs (e.g., quantitation at or below current water quality standards) and adjust those resources, where necessary, to meet data quality and program needs."
Vote*: 19 Agree, 0 Not Opposed, 0 Disagree, 1 Abstain (Mary S.***)** *(9-19-07)*

APPROVED

C. Additional Recommendation #7

The FACDQ agrees to approve the following Additional Recommendation:
"EPA develop and implement guidance on the new procedures as well as a computer-based program to assist in calculating detection and quantitation limits."
***Vote*: 20 Agree, 0 Not Opposed, 0 Disagree** *(9-19-07)*

APPROVED

D. Additional Recommendation #1

The FACDQ agrees to approve the following Additional Recommendation:
"To maintain consistency and minimize effects on the environmental laboratory community, the FACDQ recommends that EPA programs that reference the present Part 136 Appendix B procedure consider adopting (the new procedure) that would replace it."
***Vote*: 20 Agree, 0 Not Opposed, 0 Disagree** *(9-19-07)*

APPROVED

E. Additional Recommendation #2

The FACDQ agrees to approve the following Additional Recommendation:
"The FACDQ recommends that EPA's Office of Water complete a follow up pilot study to confirm the performance of the procedure(s) proposed for promulgation."
Vote*: 17 Agree, 3 Not Opposed (Tom M., Steve B., David K.***), 0 Disagree** *(9-19-07)*

APPROVED

F. Additional Recommendation #5

The FACDQ agrees to approve the following Additional Recommendation:
"EPA evaluate and modify the uses of data in CWA programs (beyond those uses discussed in the FACDQ recommendations) based on data uncertainty and decision error rate requirements relative to corresponding detection and quantitation limits. This could be accomplished through establishment of and adherence to data quality objectives for all CWA programs. How data relative to detection and quantitation limits are to be used in 303(d) listings, reasonable potential determinations, NPDES effluent limit derivation, the development of water quality criteria, and other uses should be documented."
Vote*: 13 Agree (Dave A., Nan T., Roger C., Larry L., John P., Dave P., Zonetta E., Chris H., David K., Jim P., Michael M., Rick R., Barry S.***), 6 Not Opposed (***Bob A., Tim F., Tom M., Steve B., Richard B., Cary J.***), 1 Disagree (***Mary S.***)** *(9-20-07)*

NOT APPROVED

G. Additional Recommendation #6

The FACDQ agrees to approve the following Additional Recommendation:
"EPA establish data quality objectives (with indicators and measurement quality objectives) for CWA programs where detection/quantitation limits are used in decision making."
Vote*: 15 Agree (Dave A., Bob A., Tim F., Nan T., Roger C., Larry L., John P., Dave P., Zonetta E., Chris H., David K., Jim P., Michael M., Rick R.., Barry S.***), 4 Not Opposed (***Tom M., Steve B., Richard B., Cary J.***), 1 Disagree (***Mary S.***)** *(9-20-07)*

NOT APPROVED

H. Peer Review of the Procedure

The FACDQ recommends that a formal peer review take place for the FACDQ recommended procedure.
Vote*: 16 Agree, 4 Not Opposed (Bob A., Nan T., Zonetta E., Jim P.***), 0 Disagree** *(9-20-07)*

APPROVED

6. Single Lab Procedure Recommendations

A. Lab-Determined Detection Limits and Quantitation Limits (*As Is*)

The FACDQ recommends that EPA promulgate[3] the DQFAC Single Laboratory Procedure v2.4[4] recommended by the FACDQ for individual laboratories to determine their detection and quantitation limits. The DQFAC Single Laboratory Procedure v2.4 shall be used instead of the current MDL procedure in 40 CFR Part 136, Appendix B, for calculating all future Laboratory Detection and Quantitation Limits. The DQFAC Single Laboratory Procedure v2.4 has the following two capabilities:

- Demonstrates the lab's performance at a specified level.
- Determines the lowest possible value achievable by the lab while meeting the measurement quality objectives (MQOs).

***Straw Vote*: 9 Agree, 8 Not Opposed, 3 Disagree,** *(9/20/07)*

B. Lab-Determined Detection Limits and Quantitation Limits (*With Quick Resolution on Modifications*)

*Note: This vote reflects the Committee's desire to explore potential modifications and spend time on the language below:

The FACDQ recommends that EPA promulgate[1] the DQFAC Single Laboratory Procedure v2.4[2] recommended by the FACDQ for individual laboratories to determine their detection and quantitation limits. The DQFAC Single Laboratory Procedure v2.4 shall be used instead of the current MDL procedure in 40 CFR Part 136, Appendix B, for calculating all future Laboratory Detection and Quantitation Limits. The DQFAC Single Laboratory Procedure v2.4 has the following two capabilities:

- Demonstrates the lab's performance at a specified level.
- Determines the lowest possible value achievable by the lab while meeting the measurement quality objectives (MQOs).

***Straw Vote*: 10 Agree, 8 Not Opposed, 2 Disagree** *(9/20/07)*

C. Optional Batch Specific Verification

The FACDQ recommends that the following language be moved into the DQFAC Single Lab Procedure v2.4:

Blanks and QL spikes in each batch

a. If the method blank exceeds the DL and a cause cannot be identified, raise the DL to the blank result for future analysis
b. If the QL spike result (or QL spike times QL/spike level, if not spiking exactly at the QL) is less than the DL, elevate the QL by a factor of two and repeat the QL spike at the new QL. Repeat this until the QL spike is at or above the DL.

[3] The FACDQ recognizes that EPA cannot commit to promulgate the recommendations of the FACDQ without the benefit of public notice and comment. Wherever "promulgate" appears in the FACDQ recommendations, the FACDQ expects that EPA will propose a rule consistent with the FACDQ recommendations and then finalize a rule that fully considers those public comments.

[4] This procedure was created via modifications to the ACIL.

c. If the QL spike result is outside the average specified accuracy, elevate the QL by a factor of two and repeat the QL spike at the new QL. Repeat this until the QL spike meets the specified accuracy criteria.

Vote*: 4 Agree (Zonetta E., Chris H., Jim P., David K.***), 9 Not Opposed (***Richard B., Cary J., Nan T., Roger C., Larry L., John P., Dave P., Rick R., Barry S.***), 7 Disagree (***Dave A., Bob A., Tim F., Tom M., Steve B., Michael M., Mary S.***)** *(9-20-07)*

NOT APPROVED

D. Batch Verification

The FACDQ recommends that during promulgation, EPA include and/or develop language to incorporate batch specific verification as an option in the procedure.

Vote*: 16 Agree, 4 Not Opposed (Tom M., Richard B., Cary J., Mary S.***), 0 Disagree** *(9-20-07)*

APPROVED

E. QL Verification Frequency

The FACDQ recommends that the following be adopted into the DQFAC Single Lab Procedure v2.4:

Section 2.10 of the ACIL procedure specifies monthly QL verification spikes, evaluated on a quarterly basis. Section 2.2 of revised ACIL procedure specifies a minimum of quarterly QL verification spikes, evaluated on an annual basis. If we went to monthly QL verification spikes, evaluated annually this would provide a minimum of 24 QL spikes over a two year period to generate the long term estimate:

2.2 Continue to collect method blanks with each batch from which data were reported and QL spikes for every analyte[5] analyzed at least monthly (or four per 12 month period in separate batches spread across the time period during which analysis is conducted) which ever is greater. If multiple instruments are to be used for reporting data with the same DL and QL, analyze two to six QL spikes per instrument per 12 month period, so that a minimum of 12 QL spikes are generated each year.

2.2.1. Evaluate your DLs and QLs at least every year using all of the spikes available in a 24 month period using the procedures described in the Sections below. All method blanks and QL spikes collected within a 24 month period should be used for reassessing DLs and QLs, unless there is reason to believe that the DL or QL changed substantially at some point during that 24 month period. In that case the most recent data may be used for the reassessment, but not less than 20 method blanks and seven QL spikes per instrument.

Vote*: 4 Agree (Roger C., Larry L., John P., Dave P.***), 5 Not Opposed (***Zonetta E., Chris H., David K., Jim P., Rick R.***), 11 Disagree (***Dave A., Bob A., Tim F., Tom M., Steve B., Richard B., Cary J., Nan T., Michael M., Barry S., Mary S.***)** *(9-20-07)*

NOT APPROVED

[5] For multi component analytes a lab may use representative analytes to collect data for classes of compounds. When a representative analyte is monitored, the other analytes that compound represents must have similar sensitivity and method performance characteristics as demonstrated in initial DL/QL studies. If DLs or QLs for a monitored analyte are adjusted, as a consequence of on-going verification, the same adjustment must be applied to all analytes represented. An example is method 608 which includes several Aroclors, Toxaphene, and technical Chlordane. In this case, a mixture of Aroclors 1016 and 1260 might be used to represent all Aroclos. Toxaphene may be used to represent both Toxaphene and technical Chlordane.

F. QL Verification Frequency

The FACDQ recommends that EPA give additional consideration to increasing the frequency of QL verification and report its findings in the preamble of the Federal Register Notice and request specific comments on the final proposed frequency.

Vote*: 11 Agree, 9 Not Opposed (Bob A., Tim F., Tom M., Steve B., Richard B., Cary J., Nan T., Michael M., Mary S.***) 0 Disagree** *(9-20-07)*

APPROVED

G. DL Verification and Recalculation

The FACDQ recommends that the following be adopted into the DQFAC Single Lab Procedure v2.4:

Section 1.9 of the ACIL procedure specifies annual recalculation of DL and then uses an F test to determine if the DL should be revised. Section 2.2.2 (now 2.4) allows optional recalculation of the DL, with no decision criteria provided. By making the recalculation of the DL optional it is possible that the false positive error rate using the parametric statistical test could be greater than 1%.

2.2.2 Recalculate the DL using the formulas in 1.1.7. or 1.2.7.

Vote*: 8 Agree (Dave A., Roger C., Larry L., John P., Dave P., Zonetta E., David K., Jim P.***), 10 Not Opposed (***Bob A., Tom M., Steve B., Richard B., Cary J., Nan T., Chris H., Michael M., Rick R., Barry S.***), 2 Disagree (***Tim F., Mary S.***)** *(9-20-07)*

NOT APPROVED

H. Lab-Determined Detection Limits and Quantitation Limits

The FACDQ recommends that EPA promulgate[1] the DQFAC Single Laboratory Procedure v2.4[2] recommended by the FACDQ for individual laboratories to determine their detection and quantitation limits. The DQFAC Single Laboratory Procedure v2.4 shall be used instead of the current MDL procedure in 40 CFR Part 136, Appendix B, for calculating all future Laboratory Detection and Quantitation Limits. The DQFAC Single Laboratory Procedure v2.4 has the following two capabilities:

- Demonstrates the lab's performance at a specified level.
- Determines the lowest possible value achievable by the lab while meeting the measurement quality objectives (MQOs).

Vote*: 14 Agree (Dave A., Bob A., Tim F., Tom M., Steve B., Richard B., Nan T., Roger C., Larry L., John P., Dave P., Zonetta E., Jim P., Rick R.***), 1 Not Opposed (***Chris H.***), 5 Disagree (***Cary J., David K., Michael M., Barry S., Mary S.***)** *(9-20-07)*

NOT APPROVED

7. Target MQO Bounds Recommendation

The FACDQ recommends that a single set of MQO bounds be established for promulgated Part 136 methods that define Quantitation for CWA compliance and enforcement uses.

Vote*: 7 Agree (Larry L., John P., Dave P., Zonetta E., Chris H., David K., Jim P.***), 3 Not Opposed (***Dave A., Bob A., Tim F.***), 8 Disagree (***Tom M., Steve B., Cary J., Nan T., Michael M., Rick R., Barry S., Mary S.***), 2 Absent (***Roger C., Richard B.***)** *(9-21-07)*

NOT APPROVED

8. Matrix Effects Recommendations

A. Recommendation #1

The FACDQ recommends that EPA publish new guidance on matrix effects. At a minimum, the guidance should outline the appropriate level of matrix effects validation necessary for method promulgation for analytical methods to be considered for 40 CFR Part 136. The FACDQ recommends that EPA adhere to this guidance in methods it develops and validates for promulgation in 40 CFR Part 136. This guidance should also address the following:

- Determining the appropriate number of matrices to take into account.
- The level of validation required verses the proposed scope of use for the analytical method.
- Matrix effects validation in the ATP program.
- Impacts for consensus standards methods considered for part 136.

Vote*: 10 Agree (Nan T., Roger C., Larry L., John P., Dave P., Zonetta E., Chris H., David K., Jim P., Barry S.***), 7 Not Opposed (***Dave A., Bob A., Tim F., Tom M., Cary J., Michael M., Rick R.***.), 3 Disagree (***Steve B., Richard B., Mary S.***)** *(9-21-07)*

NOT APPROVED

B. Recommendation #2

The FACDQ recommends that EPA develop a consistent protocol on how to demonstrate matrix effects. The FACDQ believes such a protocol should be sensitive to cost and required level of effort to ensure that it is applied consistently.

Questions to be addressed by the protocol:

- What level of effort is necessary to determine if the matrix effects can be resolved by modifications of the analytical method that are within the flexibility allowed within the method?
- What set of experiments and data interpretation framework would suffice to demonstrate a matrix effect if performed properly?
- Who should be responsible for implementing a procedure to determine a matrix specific QL?
- How broadly applicable shall a matrix effect be considered? What level of demonstration should be considered adequate for a single facility? What level of demonstration should be undertaken to extend the matrix specific QL to other like wastewaters?

Vote*: 13 Agree (Dave A., Bob A., Tom M., Richard B., Nan T., Roger C., Larry L., Dave P., John P., Zonetta E., Chris H., Jim P., Rick R.***), 6 Not Opposed (***Tim F., Steve B., Cary J., David K., Michael M., Barry S.***), 1 Disagree (***Mary S.***)** *(9-21-07)*

NOT APPROVED

C. Recommendation #3

The FACDQ recommends that EPA develop a procedure for determining matrix-specific detection or quantitation limits for use where appropriate. Again, such a protocol should be sensitive to cost and required level of effort.

Questions that should be addressed include:

- Who should be responsible for implementing a procedure to determine a matrix specific QL?
- How broadly applicable shall a matrix effect be considered?
  What level of demonstration should be considered adequate for a single facility?
  What level of demonstration should be undertaken to extend the matrix specific QL to other like wastewaters?

Vote*: 11 Agree (Dave A., Tom M., Richard B., Nan T., Roger C., Larry L., John P., Dave P., Zonetta E., Chris H., Jim P.***), 8 Not Opposed (***Bob A., Tim F., Steve B., Cary J., David K., Michael M., Rick R., Barry S.***), 1 Disagree (***Mary S.***) (9-21-07)***

NOT APPROVED

D. Recommendation #4

When considering future updates of QL_{nat}, the FACDQ recommends that EPA take into consideration any experience with the performance in different matrices when considering a revision of the QL_{nat}.

Vote*: 11 Agree (Dave A., Tom M., Richard B., Nan T., Roger C., Larry L., John P., Dave P., Zonetta E., Chris H., Jim P.***), 4 Not Opposed (***David K., Michael M., Rick R., Barry S.***), 5 Disagree (***Bob A., Tim F., Steve B., Cary J., Mary S.***) (9-21-07)***

NOT APPROVED

9. Verification Recommendation

The FACDQ recommends that the Verification Document be used as a resource document for the Single Lab DL QL Procedure v2.4 majority/minority report.

Vote*: 18 Agree, 2 Not Opposed (Zonetta E., Chris H.***), 0 Disagree** *(9-21-07)*

APPROVED

10. Implementation Recommendations

A. Recommendation #1

Although the FACDQ did not reach consensus on a procedure, we recommend that EPA act to develop an alternative to the current 40 CFR Part 136 Appendix B procedure. The results of the pilot study, and our evaluation of the ACIL modified procedure, indicate that there are deficiencies in the current 40 CFR Part 136 Appendix B procedure that can and should be corrected. The Single Lab DL QL Procedure v2.4 submitted contains elements that would be valuable to the agency in developing a new procedure.

***Vote*: 20 Agree, 0 Not Opposed, 0 Disagree** *(9-21-07)*

APPROVED

B. Recommendation #2

The FACDQ recommends that EPA develop guidance and outreach materials for stakeholders as EPA implements the FACDQ recommendations.

***Vote*: 20 Agree, 0 Not Opposed, 0 Disagree** *(9-21-07)*

APPROVED

11. Definitions Recommendations

A. Recommendation #1

The FACDQ recommends adding the IUPAC L_C, L_D, and L_Q definitions into the glossary.
Vote*: 13 Agree, 6 Not Opposed (Bob A., Tim F., Tom M., Richard B., Cary J., David K.***), 0 Disagree, 1 Absent (***Dave A.***)** *(9-21-07)*

APPROVED

B. Definitions: Detection Limits

The FACDQ recommends that the definitions for Detection Limits below be adopted for use in the Final Report:

DETECTION LIMIT (DL) – LAYPERSON'S DEFINITIONS

1. **Detection Limit (DL) -** The minimum result which can be reliably discriminated from a blank (for example, with a 99% confidence level).
2. **Detection Limit (DL)** – The lowest result that can be distinguished from the blank at a chosen level, α, of statistical confidence.

DETECTION LIMIT (DL) - STATISTICAL DEFINITIONS

1. **Detection Limit (DL) -** Smallest measured amount or concentration of analyte in a sample that gives rise to a Type I error tolerance of alpha under the null hypothesis that the true amount or concentration of analyte in the sample is equal to that of a blank. (The alternative hypothesis is that the true amount or concentration of analyte is greater than that of a blank.)
2. **Detection Limit (DL) -** The minimum observed result such that the lower 100 (1-α)% confidence limit on the result is greater than the mean of the method blanks.

Vote*: 12 Agree, 7 Not Opposed (Steve B., Cary J., Zonetta E., Chris H., David K., Jim P., Mary S.***), 0 Disagree, 1 Absent (***Barry S.***)** *(9-21-07)*

APPROVED

C. Definitions: Quantitation Limits

The FACDQ recommends that the definitions for Quantitation Limits below be adopted for use in the Final Report:

QUANTITATION LIMIT (QL) - DEFINITIONS

1. **Quantitation Limit (QL)**: The smallest detectable concentration of analyte greater than the detection limit (DL) where the accuracy (precision & bias) achieves the objectives of the intended purpose.
2. **Lab Quantitation Limit (QL_{lab})**: The smallest detectable concentration of analyte greater than the detection limit (DL) where the accuracy (precision & bias) demonstrated by the laboratory achieves the objectives of the intended purpose.

Vote*: 3 Agree (John P., Rick R., Mary S.***), 16 Not Opposed, 0 Disagree, 1 Absent (***Barry S.***)** *(9-21-07)*

APPROVED

12. Final Report Recommendation

The FACDQ approves the proposed process and schedule below for the Final Report of the Committee's work.

- The lead for each section will work with the designated back-ups to draft that section.
- The Final Report Work Group has some discretion over what goes into the appendices.
- As soon as a section is drafted, the lead will circulate it electronically to the caucuses for review and comment on a quick turn-around basis.
- Reviewers will be asked to send their comments on the initial draft via "tracked changes."
- The drafting team for each section will address those comments to the extent possible, accepting or rejecting the comments or making appropriate revisions, eliminating the "tracked changes."
- Before sending the draft to the Final Report Work Group, the lead will highlight any unresolved issues for Final Report Work Group discussion in **bold** type.
- The Uses Document was not a consensus document and it should be indicated as such in the main report with majority/minority perspectives.
- The Uses Document will be modified and included in the Appendix and will reflect the decisions made at the 10th FACDQ Meeting prior to being presented for a vote:
  - Moving Uses #1-#3 outside of the document.
  - The edits made on #4 prior to being voted on.
  - The edits to #5 prior to being voted on.

Proposed Schedule

- October 5: Majority/Minority Reports due to leads for the relevant section in the report
- November 9: Final Report Work Group sends first draft to the Committee
- November 19: Submit comments back to Final Report Group.
- November 30: Final Report Work Group sends revised draft to the Committee.
-

Details

- Use Microsoft Word, Times New Roman, font size 12
- Put section number and name in footer with the date of the draft (not autodates)
- Be precise about references; credit those that are used.

Vote*: 17 Agree, 0 Not Opposed, 0 Disagree, 3 Absent (Barry S., Jim P., Steve B.***)** *(9-21-07)*

APPROVED

Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act Programs
Meeting #11

FDIC Seidman Center, Rooms 203 & 205
3501 Fairfax Drive, Arlington, VA
Wednesday – Thursday, December 5-6, 2007

Decisions at Meeting #11

*Note: The presentation reflects the order the recommendations were considered and voted on during the meeting. In Committee decision-making at this meeting, EPA voted as the Office of Water.

1. Meeting Summary #7

The Committee agrees to approve Meeting Summary #7.
***Vote*: 15 Agree** (*Rick R., Mike M., Barry S., Dave A., Tom M., Tim F., Roger C., John P., Dave P., Cary J., Nan T., Steve B., Richard B., David K., Zonetta E., Mary S.),* **1 Not Opposed** (*Larry L.),* **0 Disagree** *(12-5-07),* **1 Absent** *(Jim P.)*

APPROVED

2. Use of Terms in Final Report

The Committee agrees to use the following terms in the Final Report:
Consensus Recommendation/Consensus Decision **or** Majority Opinion **or** Majority of the Committee voted not to recommend.
***Vote*: 9 Agree** (*Rick R., Mike M., Barry S., Dave A., Tom M., Richard B., David K., Chris H., Mary S.),* **9 Not Opposed** (*Bob A., Roger C., John P., Dave P., Larry L., Cary J., Nan T., Steve B., Zonetta E.),* **0 Disagree, 2 Absent** *(Tim F., Jim P.)*

APPROVED

3. Approval of Final Report

"The Committee approves the Final Report (document—Final Report Revised Document afternoon 12/6/07), excluding the Executive Summary, To The Reader, Appendices, and the 2.2 Majority Report as the best summary of the decisions made over the life of the Committee, given the time available."
***Vote*: 11 Agree** (*Rick R., Michael M., Dave A., Tom M., Bob A., Richard B., Steve B., David K., Chris H., Jim P., Mary S*), **7 Not Opposed** (*Roger C., John P., Larry L., Dave P., Zonetta E., Cary J., Nan T.),* **0 Disagree, 2 Absent** (*Barry S., Tim F.)*

APPROVED

Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act Programs
Meeting #12

Teleconference Meeting
1-866-299-3188
202-566-1045#

December 21, 2007, 12:00 PM to 4 PM EST

Decisions at Meeting #12

*Note: The presentation reflects the order the recommendations were considered and voted on during the meeting. In Committee decision-making at this meeting, EPA voted as the Office of Water.

1. Approval of Final Report

The Committee approves the following Final Report sections: Executive Summary; To The Reader; Appendices; Section 1.5; and the 2.2 Majority Report to be added to the remainder of the Final Report approved by the Committee at its meeting, December 6, 2007 with the statement that these sections are the best summary of the decisions made over the life of the Committee, given the time available.

Vote: **16 Agree** (*Tim F., Nan T., Roger C., Larry L., John P., Dave P., Zonetta E., Barry S., Rick R., Michael M., Dave A., Tom M., Richard B., Chris H., Jim P., Mary S*), 1 **Not Opposed** (*Cary J.),* **0 Disagree, 3 Absent** (*Bob A., Steve B., David K.)*

APPROVED

2. Meeting Summary #10, 12-13-2007

The Committee agrees to approve Meeting Summary #10, 12-13-2007.

***Vote*:** **13 Agree** (*Rick R., Mike M., Barry S., Dave A., Tom M., Tim F., Roger C., John P., Dave P., Chris H., Jim P., Richard B., Mary S.),* **4 Not Opposed** (*Cary J., Nan T., Larry L., Zonetta E.),* **0 Disagree, 3 Absent** (*Bob A., Steve B., David K.)*

APPROVED

APPENDIX C:

What We Need A Procedure To Do

Adopted by Consensus on July 13, 2006
By the Federal Advisory Committee on Detection and Quantitation Approached and Uses in Clean Water Act Programs

Introduction

At its December 8-9, 2005 meeting, the Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act Programs (FACDQ, Committee) agreed by consensus that its recommendations concerning analytical procedures for detection and quantitation in Clean Water Act programs should be based on what members of the advisory Committee need procedures to do.

Members of the Committee discussed what they needed procedures to do in the ensuing months in Policy Work Group meetings, with additional input from the Technical Work Group. At its March 29-30, 2006 meeting, the Committee reviewed a draft document, "What do we need a procedure to do," which identified 13 objectives. After discussion, the Committee agreed to the 13 objectives and added a 14th. The Committee also agreed that the objectives would apply to long-term Committee recommendations, but that the setting of any numeric objectives (i.e., false positive, false negative, precision and accuracy) would apply only to the pilot study.

Individual caucuses then reviewed the draft document, including how each objective would be evaluated, and provided comments. The facilitators consolidated the comments into a revised document. The Committee created a subgroup, consisting of Bob Avery, Richard Burrows, Michael Murray, John Phillips and Jim Pletl, and asked it to consider the caucus comments and to refine the 14 objectives and ways to measure them as input into the pilot study design.

The subgroup held a two-hour call on Monday, April 24 to review the objectives and to revise the document in light of the comments. In carrying out this assignment, the subgroup noted the following:

- The objectives defined in the document are intended to be used to evaluate procedures tested in the pilot study. The Committee does not expect that procedures will meet all of these objectives. After receiving the pilot study results, the FACDQ may decide to revisit the objectives or it may seek to revise the procedures so they better meet the objectives.
- The Committee acknowledged that cost and contracting restraints are factors that will affect the pilot study. To the maximum extent possible, the pilot will be conducted using a wide range of labs and methods.
- The Committee agreed to specific measurement quality objectives (MQOs) for false positives, false negatives, and precision to be used in the pilot study. For accuracy (bias), the Committee assigned the Technical Work Group and Pilot Design Team to establish values based on the specific analytical methods accuracy levels and existing data.

Committee approval and intent

The Committee again reviewed the document at its July 13-14, 2006 meeting, added an objective, and adopted the document by consensus. The Committee generally agreed that the list of characteristics should be built with the final recommendations in mind and that those objectives should drive the pilot study to test whether procedures met those objectives. Committee members also generally agreed that the pilot test was an opportunity to inform the Committee's final recommendation and that some of the objectives might be refined as a result of the pilot study data.

The fifteen objectives

The remainder of this document identifies the 15 objectives for testing procedures and suggests how each objective could be evaluated as part of the pilot test. The term "limit" is used generally to refer to detection and quantitation limits since the FACDQ has not yet defined them. Examples of how to measure specific objectives are sometimes written broadly and may not apply in every case (L_C, L_D, L_Q, other).

The procedure(s) will:

1. **provide an explicit estimate of bias at L_Q for limits that must be verifiable by labs at those limits.**
   To be evaluated by:

   a. reviewing procedure(s) and specifically identifying the quantitative limit for bias at L_Q that is tested in the pilot study.
   b. requiring labs to analyze samples (spikes, blind or otherwise as appropriate) and comparing observed bias to that cited by the procedure(s).

   See Appendix for specific MQOs adopted by the Committee for the pilot study

2. **provide an explicit estimate of precision at L_Q for limits that must be verifiable by labs at those limits.**
   To be evaluated by:

   a. reviewing procedure(s) and specifically identifying the quantitative limit for precision at L_Q that is tested in the pilot study.
   b. requiring labs to analyze samples (spikes, blind or otherwise as appropriate) and comparing observed precision to that cited by the procedure(s).

   See Appendix for specific MQOs adopted by the Committee for the pilot study

3. **provide an explicit false positive rate for L_C.**
   To be evaluated by:

   a. reviewing procedure(s) and specifically identifying the false positive error rate predicted for each limit that is tested in the pilot study.

b. comparing the false positive rate of lab blanks at the estimated levels of L_C to those predicted by the procedure(s).

Note: *The intent is to look at long term performance, however for the pilot study the number of samples may be relatively small.*

See Appendix for specific MQOs adopted by the Committee for the pilot study

4. **provide an explicit false negative rate at L_C for the true value at L_D or L_Q that must be observed in labs at L_C for the estimated values of L_D or L_Q.**
   To be evaluated by:

   a. reviewing procedure(s) and specifically identifying the false negative error rate predicted for L_D/L_Q that is tested in the pilot study.
   b. comparing the false negative rate of results obtained by analyzing samples spiked at the L_D/L_Q concentration to those predicted by the procedure(s).

   Note: *The intent is to look at long term performance, however for the pilot study the number of samples may be relatively small.*

   See Appendix for specific MQOs adopted by the Committee for the pilot study

5. **provide that qualitative identification criteria defined in the analytical method are met at the determined detection and quantitation limits.**
   To be evaluated by:

   a. requiring that all method qualitative identification criteria be satisfied in order for detection to occur.
   b. requiring revision of L_Q or L_D if all spikes at L_Q or L_D are not detected.

6. **adequately represent routine variability in lab performance**.
   To be evaluated by determining whether the procedure(s):

   a. use data to calculate limits that are collected over enough time to capture variability in performance relative to MQOs.
   b. incorporate variability due to the use of multiple instruments per lab.
   c. incorporate variability due to the use of multiple analysts per lab.
   d. incorporate variability occurring across laboratories (not for single lab procedure).
   e. adjust or account for recovery.
   f. provide recommendations or limit choices for outlier tests.
   g. address varying numbers of different concentrations (spikes) that can be used among laboratories (may only apply to multi/inter lab procedures).
   h. address varying numbers of replicates per concentration (spike) that can be used among laboratories (may only apply to multi/inter lab procedures).
   i. address varying combinations of concentrations (spikes) that can be used among laboratories (may only apply to multi/inter lab procedures).

j. adequately accommodate different models of instruments used per analyte and corresponding technology used to calculate limits.

7. **perform on-going verification of estimates.**
   To be evaluated by:

   a. continuously analyzing periodic blanks to assess the estimate of L_C.
   b. continuously analyzing periodic low-level spike samples near L_Q to assess the estimate of L_Q.
   c. recalculating limits at a frequency that captures variability in performance relative to MQOs.

8. **be capable of calculating limits using matrices other than lab reagent grade water.**
   To be evaluated by:

   a. reviewing procedure(s) and determining that there is nothing precluding the use of matrices other than reagent grade water to calculate limits.
   b. reviewing procedure(s) to determine if they incorporate steps to verify when limits adopted for an analytical method can or cannot be met in a matrix other than lab reagent grade water.
   c. reviewing procedure(s) to determine if they provide instructions on preparing an analyte-free matrix that approximates the matrix in question.

9. **use only data that results from test methods conducted in their entirety.**
   To be evaluated by determining whether the procedure(s):

   a. require that samples used to calculate detection and quantitation limits undergo all routine steps outlined in an analytical method as specified in the laboratory's SOP (prep method, extraction, etc.).
   b. reviewing procedure(s) to determine if they incorporate steps to verify when limits adopted for an analytical method can or cannot be met when a sequence of non-routine steps are used.

10. **explicitly adjust or account for situations where method blanks always return a non-zero result/response.**
    To be evaluated by:

    a. reviewing procedure(s) and determining if they include a process to address occasions when method blanks always return a non-zero result.
    b. reviewing procedure(s) and determining if they require calculation of statistics regarding non-zero results/responses.
    c. reviewing procedure(s) and determining if they mathematically adjust limits for non-zero results/responses.

11. **explicitly adjust or account for situations where method blanks are intermittently contaminated.**
    To be evaluated by:

    a. reviewing the procedure(s) and determining if they define intermittent contamination and provide explicit instructions to deal with this situation.
    b. reviewing the procedure(s) and determining if they mathematically adjust limits for non-zero results/responses.

12. **be clearly written with enough detail so that most users can understand and implement them.**
    To be evaluated by:

    a. asking users to interpret data prior to the after-procedure calculations are carried out. Examples include: What is the resulting detection limit? What is the resulting quantitation limit? What is the blank bias?
    b. asking users questions about the procedure characteristics and the use of the matrix as a point of reference. Examples include: Do the procedures address recovery? How often is a limit calculated by the user? How often is data generated to calculate limits for a given procedure?
    c. asking users to perform calculations or run software and interpret results.
    d. asking users to select spikes for given circumstances.
    e. reviewing procedure(s) and determining which ones minimize the amount of data required to calculate analytical limits beyond that normally generated by analytical methods.
    f. determining that the procedure(s) do not require skills of users in addition to those that are normally required by laboratories.

13. **be cost effective.**
    To be evaluated by:

    a. reviewing procedure(s) and determining which ones minimize the amount of data required to calculate analytical limits beyond that normally generated by analytical methods.
    b. determining whether the procedure(s) require the purchase of software or equipment in addition to that which is normally required by laboratories.
    c. determining that the procedure(s) do not require skills of users in addition to those that are normally required by laboratories.

14. **assess multi- and inter-laboratory variability when data from more than one lab is used.**
    To be evaluated by:

a. comparing results from multi-, inter-, and single lab studies.
b. Calculating intra-lab, inter-lab, and pooled or multi-lab variability and the associated variance error components.

15. **be applicable to all users and test methods.**
To be evaluated by:

a. testing procedure(s) against objectives 1-13 among a representative sample of labs (states, EPA, commercial, municipal, small, medium and large, etc.).
b. testing procedure(s) against objectives 1-13 among a representative sample of analytical test methods (different technologies and analytes).

APPENDIX D:

DQ FAC Single Laboratory Procedure v2.4
8/30/2007

SCOPE
Procedures are provided by which an individual laboratory may derive accurate estimates of routine method sensitivity for most analytical methods.
These procedures set the Detection Limit (DL) at the lowest result that can be reliably distinguished from a blank (specifically a false positive rate of ≤ 1% is targeted). This is conceptually equivalent to the IUPAC term Critical Value, L_C. The DL is the normal censoring limit for analytical result reporting.

The Quantitation Limit (QL) is set at the level that meets specific criteria that are defined within this procedure.
The procedure requires that the specification of the precision and accuracy (measured as recovery of spikes) required for the intended use of the method be identified. The limits required may come from the analytical method, regulatory documents, or be set by the laboratory based on method performance if not available from these sources. The procedure requires that these criteria must be satisfied from samples spiked at or close to the QL
The lowest calibration standard (or low level calibration verification standard for tests with a single point initial calibration) must be at or below the QL. A false negative rate of ≤ 5% for a true concentration at the QL is targeted.
The QL is based on elements of the both the detection limit (Ld) and the quantitation limit (Lq) using international terminology.

This procedure is not applicable to analytical methods for which it is not feasible to create spiked samples at increasing levels of concentration. For example, it does not apply to measurements of temperature or pH.

In some cases it is not necessary to report results below the quantitation limit. In these cases the determination of the DL may be omitted and only those steps necessary to define the QL need to be followed. If the DL and the QL are both required then all steps in the procedure should be followed.

GENERAL REQUIREMENTS
This procedure should be followed for each method where a DL and QL need to be determined. In order to form reliable estimates of detection and quantitation limits, all steps in a method must be followed during the collection of blank and low level spiked sample data. A method is defined as the combination of steps that are performed on a sample. For example, preparation steps such as liquid/liquid extraction must be performed as well as analytical steps such as gas chromatography. The use of method blank data to determine detection limits is generally preferred. However, if the instrument system returns results of "Not detected" for an analyte/method combination rather than numerical results for most blanks, then low level spikes must be used as a substitute for the method blanks.

1. INITIAL STARTUP

1.1. If no historical data are available proceed to Section 1.1.1. If historical data demonstrate that 50% or more of method blanks for an analyte give a numerical result, then estimate a DL based on blanks as described in and beginning with section 1.1.3. If less than 50% of the historical method blank results give a numeric result then skip to Section 1.2. A numeric result includes positive, negative, and zero values.

1.1.1. Collect results for method blanks generated during routine operation of the method. The method blanks must go through all preparation and analysis steps of the method. A minimum of seven numerical method blank results, each from a different preparation batch, is required in order to calculate an initial estimate of the method DL. The minimum number of blanks needs to be analyzed on each instrument used to report data. If more than seven blank results are available then they should be used. In general, the greater the number of results used to create the estimate, the more accurate it will be.

1.1.2. If less than 50% of the method blank results give a numeric result then skip to Section 1.2.

1.1.3. If it is necessary to initiate analysis immediately, an estimate of the DL may be made by analyzing seven blanks in less than seven batches. This short term DL must be replaced by a DL determined from method blanks, in a minimum of seven different batches as soon as data are available in order to capture sufficient temporal variability.

1.1.4. If multiple instruments are to be used for the same test, and will have the same reporting limit or QL, a minimum of seven method blank results must be used for each instrument and a DL calculated for each instrument. If the same DL or QL is reported for multiple instruments, the laboratory shall use the highest DL for the purposes of reporting data,

1.1.5. Results associated with known errors that occurred during analysis should be discarded, or where appropriate, corrected. It is also acceptable to apply a statistically accepted outlier test, such as the removal of results more than two or three standard deviations from the mean. Results two standard deviations or less from the mean should not be removed. With the exception of known errors, this data rejection must be performed with caution, and no more than 5% of data may be rejected. Excessive rejection of data will result in a calculated DL lower than can be supported.

1.1.6. If not all of the blanks have numerical results, but over 50% do, set the value for those blanks that do not have numerical results to zero. Calculate the sample standard deviation of the method blank results.

$$s = \sqrt{\frac{\sum_{i=1}^{n}(X_i - \overline{X})^2}{n-1}}$$

Where:
n = the number of results used in the calculation
X_i = a result obtained from the analysis of a sample
$\overline{X}$ = the mean of the results

1.1.7 Calculate the DL: $DL = \overline{X} + s\,K_{(n-1,0.99,0.01)}$

Where:

- $\overline{X}$ is the mean result from the method blanks
- $K_{(n-1,0.99,0.01)}$ is a multiplier for a tolerance limit based on 99% coverage probability of 99% of the population of routine blanks and n-1 degrees of freedom. Values for K are listed in Table 1.

Note: In the case that a negative value for $\overline{X}$ is obtained, substitute zero for $\overline{X}$ in the equation for calculation of the DL.

1.1.8. If 5% or more blank results (after outlier removal) are greater than the DL, raise the DL as follows:

- to the highest result if less than 30 method blanks are available.
- to the next to the highest result if 30-100 method blanks are available.
- to the level exceeded by 1% of the method blanks if there are more than 100.

Only a blank that meets method specified qualitative identification criteria (where applicable) should be given a numerical result.

1.2. This section determines the DL for methods with less than 50% of blanks giving numerical results and also determines the QL for all methods.

1.2.1. If less than 50% of method blanks give numerical results then the DL is estimated using low level spiked samples. These spiked samples are also used to define the QL for all analytical methods.

1.2.2. Select the spiking level. The spiking level must be at or below the level that the laboratory intends to use as their QL for reporting. If an estimate of the DL has been made using method blanks, then the spiking level must be at least two times that DL. The laboratory may use prior experience or consideration of the signal to noise to form this estimate. All qualitative identification criteria in the analytical method must be met for spikes at the QL; (for example, identification of qualifier ions, ion ratios, etc). Where it is necessary to achieve the lowest QL possible, follow the optional procedure described in Section 1.2.2.1.

1.2.2.1 Using the laboratory's knowledge of the method, analyze spikes of the analyte(s) in blanks. Start at a measurable concentration and reduce the spike concentrations successively in steps of approximately 3 (e.g., 100, 30, 10, 3, 1 etc) until:

- signal to noise ratio is less than 3, or
- qualitative identification criteria are lost, or
- signal is lost, or
- the value is less than twice the detection limit determined in Section 1.1

Use the lowest concentration at which all the applicable criteria are met.

1.2.3. Test the selected spiking level.

1.2.3.1. Analyze at least a single spiked blank at the intended quantitation limit and carried through the entire analytical procedure

1.2.3.2. If the analyte is not detected, either because it does not yield a signal, or the result falls below a detection limit determined in Section 1.1., or qualitative identification criteria defined in the method are not achieved, repeat the test at twice the concentration used in Section 1.2.3.1.

1.2.3.3. If multiple instruments are to be used to perform the same test and the same reporting limit or quantitation limit will be used, then the test of the QL estimate must be performed on each instrument, and the highest value from all the instruments is used as the estimate.

1.2.4. Once the appropriate spiking level (which will become the QL) is selected, analyze a minimum of seven replicates, divided among at least three different preparation batches, each spiked at this level. If it is necessary to initiate analysis immediately, an estimate of the DL and QL may be made by analyzing seven QL spikes in less than three batches. The short term DL and QL must be replaced by a DL and QL determined from QL spikes in a minimum of three different batches as soon as possible.

1.2.5. If the analyte is not detected in any one of the replicates, analyze a minimum of seven replicates divided between three different preparation batches at twice the concentration. This new concentration is the QL estimate. If multiple instruments are used to report the same QL, at least two replicates in separate batches must be analyzed on each instrument.

1.2.6. Determine the mean recovery and relative standard deviation of the QL spike results. If precision and accuracy requirements are not met, then repeat the spike at a higher concentration (resulting in a higher QL).

Relative Standard Deviation = RSD = Standard Deviation / Mean Result

1.2.6.1. Precision and accuracy limits for the QL may be found in the analytical method or in regulatory documents. If not defined in these sources the laboratory specifies their own requirements. Precision and accuracy at the QL will be expected to be somewhat worse than at the mid level, so it is not appropriate to use criteria established for mid level spikes at the QL. In the absence of other guidance the laboratory may establish precision and accuracy limits based on the performance of the initial QL spikes.

1.2.7. Estimate the DL. If the DL has been estimated using method blanks according to Section 1, skip this section and continue to Section 1.2.8. If the DL has not been estimated using method blanks (i.e., less than 50% of method blanks had numerical results) then the DL is determined according to the following equation:

$$DL = s \times t_{(n-1,\,1-\alpha=0.99)}$$

- Where s is the standard deviation of the measured QL spike results.
- $t_{(n-1,1-\alpha=0.99)}$ is the 99th percentile of a t distribution with n-1 degrees of freedom. Values for t are listed in Table 2.

Note: The lowest achievable DL may be obtained by following the optional steps in Section 1.2.2.1.

1.2.8. If 5% or more blank results (after outlier removal) are greater than the DL, raise the DL as follows:

- to the highest result if less than 20 method blanks are available.
- to the next to the highest result if 20-100 method blanks are available.
- to the level exceeded by 1% of the method blanks if there are more than 100.

Only a blank that meets method specified qualitative identification criteria (where applicable) should be given a numerical result.

1.2.9. Estimate the Lowest Expected Result (LER) from spikes at the QL.

$$\text{LER} = \frac{\overline{X}_s * QL}{SL} - \left(s \times t_{(n-1,\,1-\alpha=0.95)}\right)$$

- Where s is defined in Section 1.2.7.
- Where $\overline{X}_s$ is the mean concentration result from the QL spikes.
- $t_{(n-1,1-\alpha=0.95)}$ is the 95th percentile of a t distribution with n-1 degrees of freedom. Values for t are listed in Table 1.
- SL is the spike level used for the QL spike sample.

1.2.10. Compare the LER to the DL. If the LER is less than the DL then the QL is raised according to the equation:

$$\text{QL}_{\text{new}} = \frac{[DL + s * t_{(1-\alpha=0.95;n-1)}] * QLold}{\overline{X}_s}$$

1.2.11. Do NOT adjust the spiking level for ongoing QL verification (see Section 2) unless the spiking level is outside the range of half to twice the new QL. If qualitative identification criteria are not met at the spiking level, increase the spiking by a factor of two.

2. ONGOING VERIFICATION

2.1. At least once every 12 months, or more frequently at the discretion of the QA manager, re-evaluate the DLs and QLs.

2.2. Continue to collect method blanks with each batch from which data were reported and QL spikes for every analyte[6] at a rate of at least four per 12 month period (in separate batches) spread across the time period during which analysis is conducted. If multiple instruments are to be used for reporting data with the same DL and QL, use at least two spikes per instrument per 12 month period.

2.2.1. Evaluate your DLs and QLs at least every year using all of the spikes available in a 24 month period using the procedures described in the Sections below. All method blanks and QL spikes collected within a 12 month period should be used for reassessing DLs and QLs, unless there is reason to believe that the DL or QL changed substantially at some point during that 12 month period. In that case the most recent data may be used for the reassessment, but not less than 20 method blanks and seven QL spikes per instrument. More than 12 months worth of data may be used if there is no reason to believe that the DLs and QLs have changed.

2.2.2. Optionally, recalculate the DL using the formulas in 1.1.7. or 1.2.7.

2.3. Blank Check: For all methods, check the blank results against the DL. If 5% or more blank results (after outlier removal) are greater than the DL, raise the DL as follows:

- to the highest result if less than 20 method blanks are available.
- to the next to the highest result if 20-100 method blanks are available.
- to the level exceeded by 1% of the method blanks if there are more than 100.

Only a blank that meets method specified qualitative identification criteria (where applicable) should be given a numerical result.

2.4. **Qualitative Identification Check**: At least 95% of the QL spiked data for each analyte must meet the qualitative identification criteria in the method. If 5% or more do not meet the qualitative criteria, then raise the QL and the spiking level to a level at which the qualitative identification criteria can be reliably met.

2.5. **Lowest Expected Result (LER) Check**: Estimate the lowest expected result (LER) from spikes at the QL. See Section 1.2.9.

2.5.1. Compare the LER to the DL. If the LER is less than the DL then the QL is raised according to the equation in Section 1.2.10.

[6] For multi component analytes a lab may use representative analytes to collect data for classes of compounds. When a representative analyte is monitored, the other analytes that compound represents must have similar sensitivity and method performance characteristics as demonstrated in initial DL/QL studies. If DLs or QLs for a monitored analyte are adjusted, as a consequence of on-going verification, the same adjustment must be applied to all analytes represented. An example is method 608 which includes several Aroclors, Toxaphene, and technical Chlordane. In this case, a mixture of Aroclors 1016 and 1260 might be used to represent all Aroclos. Toxaphene may be used to represent both Toxaphene and technical Chlordane.

2.5.2. Do NOT adjust the spiking level for ongoing QL verification (see Section 2) unless the spiking level is outside the range of half to twice the new QL. It is also necessary to adjust the spiking level if the spike results are not meeting the qualitative identification criteria in the method.

2.6. **Precision and Accuracy Check**: Determine the mean recovery and relative standard deviation of the QL spike results. If precision and accuracy requirements are not met, then the QL and spiking level must be raised

2.7. If the QL can be lowered by a factor of two or more, without causing the LER to be below the DL, qualitative identification can still be reliably maintained, and precision and accuracy requirements are met, then the QL, optionally, may be lowered. If the spiking level is then outside the range of half to twice the new QL, then the spiking concentration must be adjusted accordingly.

2.8. After verification, if the assessment process indicates that the DL or QL have increased by a factor of two or more, labs should investigate causes and take appropriate corrective action when necessary.

3. REPORTING DATA

3.1. The QL as described above is the lowest level for reporting quantitative results, but data may be reported down to the DL. If the requirements for quantitation cannot be met at any level, report all data as estimated.
For example, if the QL is 2.0 and DL is 0.6 then results are reported as follows:

Instrument result	Reported Result
2.1	2.1
1.9	1.9J or DNQ
0.91	0.9J or 0.91J or DNQ
0.54	<0.6 or 0.6U or ND
ND	<0.6 or 0.6U or ND

"DNQ:" Detected, Not Quantified
"U": A flag indicating non-detect
"J": A flag indicating increased uncertainty in the results

4. MATRIX EFFECTS

4.1. Optionally, to demonstrate whether or not you can achieve your estimated DL and QL in a specific matrix:

1) analyze the unspiked matrix to demonstrate that the analyte is below the DL and,
2) analyze a QL spiked matrix to demonstrate that the QL criteria can be achieved.

This procedure as outlined below could be applied to various matrices providing an analyte free matrix could be obtained. The procedure outlined in 4.1 will not allow False Positives caused by a Matrix Effect to be distinguished from true positive results.

Table 1.
K values for n replicates

n	K	n	K
7	6.101	54	2.977
8	5.529	55	2.97
9	5.127	56	2.963
10	4.829	57	2.956
11	4.599	58	2.949
12	4.415	59	2.943
13	4.264	60	2.936
14	4.138	61	2.93
15	4.031	62	2.924
16	3.939	63	2.919
17	3.859	64	2.913
18	3.789	65	2.907
19	3.726	66	2.902
20	3.67	67	2.897
21	3.619	68	2.892
22	3.573	69	2.887
23	3.532	70	2.882
24	3.494	71	2.877
25	3.458	72	2.873
26	3.426	73	2.868
27	3.396	74	2.864
28	3.368	75	2.86
29	3.342	76	2.855
30	3.317	77	2.851
31	3.295	78	2.847
32	3.273	79	2.843
33	3.253	80	2.839
34	3.234	81	2.836
35	3.216	82	2.832
36	3.199	83	2.828
37	3.182	84	2.825
38	3.167	85	2.821
39	3.152	86	2.818
40	3.138	87	2.815
41	3.125	88	2.811
42	3.112	89	2.808
43	3.100	90	2.805
44	3.088	91	2.802
45	3.066	92	2.799
46	3.055	93	2.796
47	3.045	94	2.793
48	3.036	95	2.79
49	3.027	96	2.787
50	3.018	97	2.784
51	3.009	98	2.782
52	3.001	99	
53	2.993	100	

If n >100 use values for n=100.

Table 2.
99th and 95th percentile *t* values for *n* replicates

n	$t_{(1-\alpha)}$=0.99	$t_{(1-\alpha)}$=0.95	n	$t_{(1-\alpha)}$=0.99	$t_{(1-\alpha)}$=0.95
7	3.143	1.943	54	2.399	1.674
8	2.998	1.895	55	2.397	1.674
9	2.896	1.860	56	2.396	1.673
10	2.821	1.833	57	2.395	1.673
11	2.764	1.812	58	2.394	1.672
12	2.718	1.796	59	2.392	1.672
13	2.681	1.782	60	2.391	1.671
14	2.650	1.771	61	2.390	1.671
15	2.624	1.761	62	2.389	1.670
16	2.602	1.753	63	2.388	1.670
17	2.583	1.746	64	2.387	1.669
18	2.567	1.740	65	2.386	1.669
19	2.552	1.734	66	2.385	1.669
20	2.539	1.729	67	2.384	1.668
21	2.528	1.725	68	2.383	1.668
22	2.518	1.721	69	2.382	1.668
23	2.508	1.717	70	2.382	1.667
24	2.500	1.714	71	2.381	1.667
25	2.492	1.711	72	2.380	1.667
26	2.485	1.708	73	2.379	1.666
27	2.479	1.706	74	2.379	1.666
28	2.473	1.703	75	2.378	1.666
29	2.467	1.701	76	2.377	1.665
30	2.462	1.699	77	2.376	1.665
31	2.457	1.697	78	2.376	1.665
32	2.453	1.696	79	2.375	1.665
33	2.449	1.694	80	2.374	1.664
34	2.445	1.692	81	2.374	1.664
35	2.441	1.691	82	2.373	1.664
36	2.438	1.690	83	2.373	1.664
37	2.434	1.688	84	2.372	1.663
38	2.431	1.687	85	2.372	1.663
39	2.429	1.686	86	2.371	1.663
40	2.426	1.685	87	2.370	1.663
41	2.423	1.684	88	2.370	1.663
42	2.421	1.683	89	2.369	1.662
43	2.418	1.682	90	2.369	1.662
44	2.416	1.681	91	2.368	1.662
45	2.414	1.680	92	2.368	1.662
46	2.412	1.679	93	2.368	1.662
47	2.410	1.679	94	2.367	1.661
48	2.408	1.678	95	2.367	1.661
49	2.407	1.677	96	2.366	1.661
50	2.405	1.677	97	2.366	1.661
51	2.403	1.676	98	2.365	1.661
52	2.402	1.675	99	2.365	1.661
53	2.400	1.675	100	2.365	1.660

If *n* >100 use values for *n*=100.

FACDQ Recommendations on Uses of Detection and Quantitation in Clean Water Act Programs

This Draft Revised Uses document incorporates changes made by the Policy Work Group on August 20 and August 30, as well as Policy Work Group authorized assignment changes.

1. **Lab-Determined Detection Limits and Quantitation Limits**
   Recommendation: The FACDQ recommends that EPA promulgate[1] the DQFAC Single Laboratory Procedure[2] recommended by the FACDQ for individual laboratories to determine their detection and quantitation limits. The DQFAC Single Laboratory Procedure shall[3] be used instead of the current MDL procedure in 40 CFR Part 136, Appendix B, for calculating all future Laboratory Detection and Quantitation Limits. The DQFAC Single Laboratory Procedure has the following two capabilities:

   - Demonstrates the lab's performance at a specified level.
   - Determines the lowest possible value achievable by the lab while meeting the measurement quality objectives (MQOs).

Comment [CG1]: shall be used "in all CWA programs"..

Comment [CG2]: Will Appendix B be put into Part 141 for Drinking Water? Labs may oppose implications of two procedures to use.

2. **Matrix Effects**

   Recommendation: The FACDQ recommends that EPA consider how matrix effects impact detection and quantitation. The FACDQ requests that the Policy Work Group bring back a conceptual recommendation including details to be considered.

Comment [CG3]: This section will include more substantive issues pending discussion by the Matrix Effects Work Group. For example:
1. How to demonstrate a matrix effect.
2. The level of matrix effect validation during method development to be performed
3. A cost effect procedure for determining specific matrix effect identification.
4. How impacts occur and how to deal with them.
5.How are DL and QL determined when matrix effects occur

3. **Verification of Laboratory Proficiency of Detection and Quantitation Limits**
   Recommendation: The FACDQ recommends developing a process for verification of detection and quantitation limits by laboratories which will strive for feasibility, practicality, representativeness, and cost-effectiveness. This recommendation includes the following guidance:

   - The process should include separate initial and on-going verification of Laboratory Detection and Quantitation Limits.
   - The process should verify that the method meets the chosen MQOs.

Comment [CG4]: The Verification Work Group will have material for this section shortly. They are deciding between general recommendations to EPA and specific recommendations.

[1] The FACDQ recognizes that EPA cannot commit to promulgate the recommendations of the FACDQ without the benefit of public notice and comment. Wherever "promulgate" appears in the FACDQ recommendations, the FACDQ expects that EPA will propose a rule consistent with the FACDQ recommendations and then finalize a rule that fully considers those public comments.
[2] This procedure was created via modifications to the ACIL.
[3] The Policy Work Group proposes that a small subgroup of the Policy Work Group examine each "shall," "should," and "must" to determine if they are being appropriately used.

- The Laboratory Quantitation Limit must be equal to or lower than the National Quantitation Limit, if a National Quantitation Limit exists.

See Attachment A on pg. 8 for a minority opinion in favor of retaining the DL_{nat} in the Uses recommendations.

4. Promulgation of National Quantitation Limits Recommendation

See Attachment B on pg. 9 for background discussion on the following two alternatives:

Alternative 1

Initial Statement of Purpose
It is the intent of the FACDQ to recommend that EPA adopt National Quantitation Limits for method and analyte combinations, particularly where compliance with the CWA cannot be determined using currently approved analytical methods (e.g. if WQBELs are less than the analytical capability of the methods). National Quantitation Limits should be set at the lowest concentration possible using approved analytical methods. A National Quantitation Limit shall be published in each analytical method used to analyze an analyte that needs a National Quantitation Limit. National Quantitation Limits can be different for each method approved for a given analyte. National Quantitation Limits are costly to develop and are not needed for regulatory determination for most analytes currently regulated under the Clean Water Act.

New Method Promulgation
Recommendation A (Placeholder): The FACDQ recommends that EPA promulgate a [multi-laboratory or inter-laboratory] procedure recommended by the FACDQ for determining National Quantitation Limits.

Recommendation B: The FACDQ recommends that when the EPA promulgates future analytical methods in 40 CFR Part 136, National Quantitation Limits shall be created and included with the methods. A National Quantitation Limit shall be created for each analyte determined by a method using the procedure(s) in Recommendation A.

Currently, this recommendation would require method developers applying for ATP approval, and standard-setting organizations, to submit to EPA multi-laboratory quantitation limits consistent with the FACDQ's multi-laboratory recommendations. These multi-laboratory limits would serve as National Quantitation Limits should the applicant's method later be promulgated in 40 CFR Part 136. For some standard-setting organizations, this may be a significant departure from what they do now. Moreover, some FACDQ members are concerned that this requirement may stifle the development of new methods. Many of the methods recently promulgated by EPA in Part 136 are the product of these outside organizations, reflecting advances in technologies that result in methods with greater sensitivity. Therefore, the FACDQ requests that EPA discuss and request public comment on this issue in the EPA Notice of Proposed Rulemaking that incorporates the recommendations of the FACDQ. Should significant concerns surface during public comment, EPA should make appropriate changes in the final rulemaking to ensure that the development of new methods is not adversely affected.

Future Updates of Promulgated Analytical Methods

Recommendation: The FACDQ recommends that EPA periodically review current capabilities of promulgated analytical methods and undertake updates based on priorities. Method updates shall include creation and incorporation of first-time or updated National Quantitation Limits. A National Quantitation Limit shall be created for each analyte determined by a method using the same procedure(s) as for new method promulgation. In determining update priorities, EPA should consider:

- Methods where there have been significant improvements in detection or quantitation limits
- Methods that do not contain National Quantitation Limits
- Cases where quantitation limits are critical to the permit program (e.g., those required for very low WQBELs)
- Analytes for which current methods provide poor performance or otherwise do not meet program needs
- Cost and resource considerations
- Information submitted by states and/or other qualified third parties.

EPA will work with method developers to update priority methods. EPA shall publish a Federal Register Notice announcing the methods it proposes to update to incorporate National Quantitation Limits. Provisions later in this document are for the purpose of providing EPA with robust data sets for updating and or creating National Quantitation Limits.

Alternative 2

Initial Statement of Purpose

It is the intent of the FACDQ to recommend that EPA adopt National Quantitation Limits for analytes listed in 40 CFR 136 based on a list of priorities. National Quantitation Limits should be set at the lowest concentration possible using approved analytical methods when compliance with the CWA cannot be determined. However, for analytes when compliance with the CWA can be comfortably determined, EPA may set a QL-something else at a concentration that allows the maximum number of laboratories and approved methods to be used. National Quantitation Limits and QL something elses shall be published in a table in 40 CFR 136 by analyte. Labs may use any approved method for an analyte so long as the Laboratory Quantitation Limit is equal to or lower than the National Quantitation Limit or QL something else for the analyte. This will provide a level playing field for all laboratories and permittees and allows maximum analytical flexibility.

Creation and Update of National Quantitation Limits

Recommendation A (Placeholder): The FACDQ recommends that EPA promulgate a [multi-laboratory or inter-laboratory] procedure recommended by the FACDQ for determining National Quantitation Limits.

Recommendation B: The FACDQ recommends that EPA periodically review capabilities of analytical methods for the purpose of establishing and updating National Quantitation Limits. Quantitation limits shall be evaluated by analyte and method using

the procedure(s) in Recommendation A. For a given analyte, the method that EPA judges has the lowest quantitation limit shall be used as the basis for setting the National Quantitation Limit.

EPA shall prioritize its efforts to create National Quantitation Limits using these or other factors:

- Cases where method sensitivity issues are critical to Clean Water Act programs (e.g., analytes with very low WQBELs)
- Analytes for which available methods have seen significant improvements in detection or quantitation limits
- Analytes for which there are no current National Quantitation Limits
- Cost and resource considerations
- Information submitted by states and/or other qualified third parties

EPA will work with method developers and others to establish and update National Quantitation Limits. EPA shall publish a Federal Register Notice announcing the analytes for which it proposes to create or update National Quantitation Limits. Provisions later in this document are for the purpose of providing EPA with robust data sets for creating or updating National Quantitation Limits.

Alternative 3 Creation and Update of Method Quantitation Limits for Use in Setting National Quantitation Limits

New Method Promulgation

Recommendation A (Placeholder): The FACDQ recommends that EPA promulgate a [multi-laboratory or inter-laboratory] procedure recommended by the FACDQ for determining National Quantitation Limits.

Recommendation B: The FACDQ recommends that when the EPA promulgates future analytical methods in 40 CFR Part 136, Method Quantitation Limits shall be created and included with the methods. A Method Quantitation Limit shall be created for each analyte determined by a method using the procedure(s) in Recommendation A.

Currently, this recommendation would require method developers applying for ATP approval, and standard-setting organizations, to submit to EPA multi-laboratory quantitation limits consistent with the FACDQ's multi-laboratory recommendations. These multi-laboratory limits could serve as National Quantitation Limits should the applicant's method later be promulgated in 40 CFR Part 136. For some standard-setting organizations, this may be a significant departure from what they do now. Moreover, some FACDQ members are concerned that this requirement may stifle the development of new methods. Many of the methods recently promulgated by EPA in Part 136 are the product of these outside organizations, reflecting advances in technologies that result in methods with greater sensitivity. Therefore, the FACDQ requests that EPA discuss and request public comment on this issue in the EPA Notice of Proposed Rulemaking that incorporates the recommendations of the FACDQ. Should significant concerns surface

during public comment, EPA should make appropriate changes in the final rulemaking to ensure that the development of new methods is not adversely affected.

Future Updates of Promulgated Analytical Methods
Recommendation: The FACDQ recommends that EPA periodically review current capabilities of promulgated analytical methods and work with method developers to update priority methods. Method updates shall include creation and incorporation of first-time or updated Method Quantitation Limits determined using the procedure in Recommendation A. EPA should prioritize its efforts to update analytical methods using these or other factors:

- Cases where method sensitivity issues are critical to Clean Water Act programs (e.g., analytes with very low WQBELs)
- Analytes for which available methods have seen significant improvements in detection or quantitation limits
- Analytes for which there are no current QLnats
- Cost and resource considerations
- Information submitted by states and/or other qualified third parties

EPA shall publish a Federal Register Notice announcing the methods it proposes to update to incorporate Method Quantitation Limits. Provisions later in this document are for the purpose of providing EPA with robust data sets for updating and or creating National Quantitation Limits.

Creation of National Quantitation Limits
Recommendation: The FACDQ recommends that EPA Periodically review methods to identify those suitable for use in setting National Quantitation Limits (QLnats) needed to implement the FACDQ recommended WQBEL permitting strategy. EPA shall promulgate a Table of QLnats by analyte. For a given analyte, the method that EPA judges has the lowest quantitation limit shall be used as the basis for setting the QLnat.

5. **Setting Permit Conditions, Reporting and Using Data, and Determining Compliance When the Water Quality Based Effluent Limit (WQBEL) is Less Than Detection and Quantitation Capabilities of Existing Methods**[4]

Recommendation: The FACDQ recommends that the following recommendations be incorporated into 40 CFR Part 122, as appropriate.

A. Recommendations for NPDES Permit and Compliance Uses When a National Quantitation Limit Exists

[4] The language previously here, relating to WQBELs at concentrations less than quantitation limits, was recommended as more appropriate elsewhere within the Final Report text and has been removed from the Uses document.

If the permitting authority requires use of a method more sensitive than the method for which a QL_{nat} exists, go to section B.

1) **Permit Requirements Related to Detection and Quantitation**
Recommendation: The FACDQ recommends the following be required where EPA has promulgated a National Quantitation Limit in 40 CFR Part 136:
   a. The default quantitation limit to be included in the permit (Permit Quantitation Limit) is the lowest Part 136 promulgated National Quantitation Limit unless the regulator determines that the Permit Quantitation Limit should be adjusted to account for sensitivity, selectivity, and/or matrix effects;
   b. The permit shall contain a condition that the quantitation limit determined by the permittee's laboratory (Laboratory Quantitation Limit) shall be at or below the Permit Quantitation Limit. The permittee's laboratory may use any Part 136 method for which they can demonstrate a Laboratory Quantitation Limit at or below the Permit Quantitation Limit. If matrix effects have been given special attention in the permit then they would also have to be considered in compliance and enforcement.
   c. The permit shall require the permittee to report the detection limit (Laboratory Detection Limit) and the Laboratory Quantitation Limit and maintain such information for a period of at least five years;
   d. The permit shall require the permittee to maintain individual numeric results for a period of at least five years. The regulator may require the individual numeric result for any value that is greater than or equal to the Laboratory Detection Limit and less than the Permit Quantitation Limit be reported in a supplemental report.
   e. The permit shall require that the Laboratory Detection Limit and the Laboratory Quantitation Limit be determined using the steps of the 40 CFR Part 136 procedure to establish the lowest possible value by the laboratory;
   f. The Permit Quantitation Limit shall be applicable for the term of the permit unless the regulator reopens and modifies the permit; and
   g. That EPA requires the Laboratory Detection Limit, the Laboratory Quantitation Limit, and the Permit Quantitation Limit be reported by the regulator to the Integrated Compliance Information System (ICIS) for purposes of updating 40 CFR Part 136 National Quantitation Limits.

2) **Establishing Compliance Thresholds and Determining Compliance**
Recommendation: The FACDQ recommends the following be required where EPA has promulgated a National Quantitation Limit in 40 CFR Part 136:

   a) Regulators will set average and daily maximum permit limits at the WQBEL.
   b) Permittees must report to the regulator all information in the following manner on the Discharge Monitoring Report (DMR):
      i) To report daily maximum sample results:
         a. For values not detected at the Laboratory Detection Limit, report "not detected".

b. For values detected at the Laboratory Detection Limit but less than the Permit Quantitation Limit, report "detected less than the Permit Quantitation Limit".
c. For values greater than or equal to the Permit Quantitation Limit, report the actual numeric values.

ii) To report average sample results:
a. When all values used to calculate an average are not detected at the Laboratory Detection Limit, report "not detected".
b. When all values used to calculate an average are "detected less than Permit Quantitation Limit," report "detected less than the Permit Quantitation Limit."
c. When values used to calculate an average are a combination of "not detected" and "detected less than the Permit Quantitation Limit", report "detected less than the Permit Quantitation Limit".
d. When any value used to calculate an average is greater than or equal to the Permit Quantitation Limit, report the calculated numeric average after assigning zero to any individual value reported either as "not detected" or "detected less than the Permit Quantitation Limit."

c) To determine NPDES permit compliance with results reported on the DMR, regulators will:

i) Determine that any daily maximum or monthly average results reported as either "not detected" or "detected less than the Permit Quantitation Limit" are in compliance with the effluent limitation.
ii) Compare any numeric results directly to the WQBEL

3) **Additional Permit Requirements**
Recommendation: The FACDQ recommends the following be required where EPA has promulgated a National Quantitation Limit in 40 CFR Part 136: Permits shall include language that triggers additional steps when a "significant number" (to be determined in permitting process) of values detected at the Laboratory Detection Limit but less than the Permit Quantitation Limit are reported. These steps may include additional or accelerated monitoring, analytical studies such as matrix studies, pollutant minimization programs, or other permit conditions outside of the determination of compliance with effluent limitations. Reports under such provisions will be done outside of the DMR process, except that any additional effluent testing performed using approved analytical methods as part of the special studies must be reported on the DMR.

B. Recommendations for NPDES Permits and Compliance Uses When No National Quantitation Limit Exists, or if the Permitting Authority Requires Use of a Method More Sensitive than the Method for Which a National Quantitation Limit exists:
Recommendations:

1) In the absence of a National Quantitation Limit, the permitting authority is free to establish its method for determining compliance for analytes that have limits/water quality standards at a level lower than that which can be detected and/or quantified.

2) For a list of analytes as defined by EPA, the permit shall require that the Laboratory Detection Limit and the Laboratory Quantitation Limit be determined using the steps of the 40 CFR Part 136 procedure to establish the lowest possible value by the laboratory;
3) That EPA require the Laboratory Detection Limit and the Laboratory Quantitation Limit and the Permit Quantitation Limit be reported by the regulator to the Integrated Compliance Information System (ICIS) for purposes of updating 40 CFR Part 136 National Quantitation Limits.

6. **Great Lakes Initiative**

Recommendation: The FACDQ recommends that the FACDQ recommendations should not supersede the current Great Lakes Initiative provisions. The FACDQ believes that there is not a significant conflict between the FACDQ recommendations and the Great Lakes Initiative.

7. **Other Uses to Consider**

Recommendation: The FACDQ tabled the discussion on recommendations regarding the use of detection and quantitation for other uses including, but not limited to, the following:

- ambient monitoring 305(b)
- pretreatment
- non-regulatory operational monitoring
- stormwater monitoring
- other studies, such as fish tissues or biosolids characterization
- reasonable potential analysis
- effluent guidelines development
- limit derivation
- development of water quality criteria

8. **Alternative Test Procedures**

Recommendation: The FACDQ tabled the option of developing specific recommendations to EPA on updating the Alternative Test Procedures (ATP) Program. The FACDQ, however, does recommend that the ATP Program be updated to be consistent with recommendations from this document.

Attachment A

Written by: **David Kimbrough**

Minority Report on DL-nat

At the December 2006 FACDQ meeting, the Committee voted unanimously on a document that recommended that EPA should establish National Quantitation Limits (QL-nats) and National Detection Limits (DL-nats) and publish them in a table in 40 CFR 136. The language about a table of QL-nats and DL-nats was withdrawn by the FACDQ at the June 2007 meeting. The PWG has also recommended that the entire concept of DL-nat be removed from all documents. At the July 25 meeting of the FACDQ the Committee was unable to reach consensus on withdrawing the DL-nat. There were two "not opposed" votes and one "opposed." This paper attempts to explain the minority position on this vote.

1) The first reason for keeping the concept of a DL-nat is to ensure that there is adequate "distance" between the DL-lab and the QL-nat. The FACDQ recommendations are for a two tiered approach with both a QL and DL. Results below the DL are reported as ND, results between the QL and DL are reported as DNQ, and results above the QL are reported as numeric values. ND and DNQ results are treated for averaging purposes as zero (i.e. not out of compliance) but there are important regulatory implications to DNQ results. Permittees reporting DNQs may be required to engage additional management practices such as increased or additional monitoring, special studies, or Pollutant Minimization Programs (PMPs). For this strategy to work, the values of QL and DL have been sufficiently different to allow for DNQs to be detected. In particular, it is by far most important when the WQBEL (or other regulatory limits) have lower concentrations than the capability of currently approved 40 CFR 136 analytical methodology can achieve. The FACDQ is proposing that at least in these cases, if not all, that a fixed QL-nat needs to be established. In having a DL-nat can be used as a ceiling on the DL-lab, ensuring that the DL-lab is not too high as to preclude the determination of DNQ.

2) The second reason for keeping the DL-nat is ensure equal protection to all receiving bodies with a given WQBEL and equity for all permittees discharging to receiving bodies with a given WQBEL. As noted above, the FACDQ recommended permitting strategy includes required management practices when DNQs are reported. As the pilot study showed, laboratories can produce DL-labs with concentrations that differ over orders of magnitude. If only the DL-lab is used, two permittees could be discharging water to a receiving body with the same concentration of an analyte, one would have to do a PMP and the other would not simply because of differences in the laboratory capability. In fact, with the range of differences in DLs seen in the pilot study, it would be possible for the dischargers with a higher concentration to have no PMP than a discharger with a lower concentration. This does not provide equal to protection to all waters nor equity to permittees.

Attachment B

Discussion of Alternatives for EPA Promulgation of QLnat

Work of the small group to investigate possibilities for QLnat promulgation (the small group was Tom Mugan, Richard Burrows, David Kimbrough and Michael Murray)

Alternative 1 in the August 15, 2007 Uses Document is basically the concept that was originally proposed perhaps a year or more ago.

The Alternative 1 proposal has two components.

- The first component would require a method developer of a new method to do the QL_{nat} procedure as part of method development and validation as part of the EPA promulgation procedure. The idea was that the QL_{nat} would be included with the method.
- The second component is a process that recommends that EPA update previously promulgated methods to include QL_{nats} (or update them) along with any other method improvements warranted. A number of Committee members have expressed the desire for EPA to undertake method updates on a much more regular basis. Again, the QL_{nat} would be included with the method.

The only significant recent change is that we had at one point added a process whereby a method developer could petition EPA for an exemption to the requirement to do the QL_{nat} procedure (multi-lab or inter-lab procedure). This was added in response to a concern that the requirement for new methods would stifle the development of new methods because method developers would have difficulty generating the QL_{nat}. *(This added language was later struck for several reasons including that it created a new administratively complex exemption process that could be problematic. As a possible solution, Mary Smith suggested that, when EPA proposes the requirement for QL_{nats} for new methods, it could specifically request comment on whether this requirement, if promulgated in the final rule, would stifle new method development.)*

The other change is the insertion of what is called an Initial Statement of Purpose as an additional explanation on the intent of the recommendation.

Alternative 2 was submitted in response to a continued concern that method developers would have difficulty finding enough labs to generate the necessary data to run the QL_{nat} procedure due to the difficulty of finding enough labs to generate the necessary data to run the QL_{nat} procedure. Therefore, Alternative 2 only has the update component.

With only an update component, it seemed reasonable that, to save on costs, EPA would only undertake update for problem analytes and, for a given analyte, would invest effort only for the method it thought was the most sensitive. Therefore, Alternative 2 was drafted as an update by analyte, rather than an update by analyte and method.

Once that draft was on paper, David K. thought that we needed to have a QL_{nat} for every analyte (in a table). Alternative 2 was then modified to say that, for analytes where current methods exist that

are capable to measuring to environmentally significant levels (non-bad boys), EPA may promulgate $QL_{somthing\ elses}$ (for lack of a better name) that were reflective of a value that represents the lowest environmentally significant level. The different name is to distinguish it from a QL_{nat} that is considered to be the lowest reasonably achievable level a lab can reach.

Again, the Statement of Purpose was added.

Alternative 3 is largely the same as alternative 1 except that it satisfies a desire by some members of the Policy Workgroup that QLnats be in a table by analyte. So, this alternative creates what I have called Method Quantitation Limits that could be the basis for promulgation, as a separate step (although it could happen simultaneously), of QLnats in Part 122 (or Part 123, I forget which we decided). Presumably all new methods would get a Method Quantitation Limit determined by the FACDQ multi-lab procedure but EPA would only translate these to QLnats as the need and priorities and dictate.

The Statement of Purpose was not added. Instead we tried to be clear as to the intent as we wrote the recommendations.

Analysis of Alternatives

The original vision of Alternative 1 came from the Hybrid Document many months ago. The idea was to set the ship in the right direction by developing QL_{nats} as we go forward. Thus, anytime a method is promulgated, either a new method or when an existing method is updated, a QL_{nat} would be generated and available for states to use for regulatory purposes.

The idea that implementation of the FACDQ's undertaking would need to be phased in carefully has guided a number of proposals in the uses document. If we develop a new method and do not generate a QL_{nat}, we may lose the opportunity that comes with the new method promulgation. History shows that bureaucratic momentum has a way of preventing EPA or states from re-opening a provision in law. Thus, while we may have good intentions of updating a method within a reasonably short time frame, the likelihood is not good.

A number of caucus groups have advocated for EPA being more responsive in promulgating and updating methods. Both alternatives recommend that EPA update methods to insert and revisit QL_{nats}. Would the hue and cry (and the pressure on EPA to update a method) be greater if an initially set QL_{nat} was demonstrated to be either too high or low or if there were no QL_{nat} at all?

We are trying to assess the validity of the concern of stifling method development. During a recent Policy WG discussion, Cary indicated that those applying for ATPs are already doing the QL_{nat} procedure. Cary is going to ask representatives of ASTM and Standard Methods if it might pose a problem with future methods they develop.[*]

[*] ASTM and Standard Methods provided input on this issue during the August 28, 2007 FACDQ Teleconference Meeting.

One attractive aspect of providing QL_{nats} by analyte, as is the case in Alternative 2, is that this appears to avoid the perceived difficulty (discussed as part of the discussion on the Uses Document) of permit conditions in a situation where the only method that has a QL_{nat} is regarded to be not the most sensitive one. This difficulty has been identified on several occasions and fixes have been made to the Uses Document.

Having a single QL_{nat} for an analyte may cause difficulties when there may be one or more methods available and there are matrix effect issues for what would otherwise be the most sensitive method. Without each method having a QL_{nat}, there would be little basis for deciding which other method is most appropriate. If we go with this alternative, we may need to provide for solutions to those problems.

The Initial Statement of Purpose adds length. This might be needed in a regulation where the meaning of words could be used for legal argument. In this case, if we need additional words to clearly state our intent, I think they should appear in the recommendation itself.

APPENDIX F

Glossary of Terms

The intent of this glossary is to define terms, commonly used in association with detection and quantitation and in environmental laboratories, which may be unfamiliar to the lay person. The definitions are taken from various sources. Where available, citations are provided following the definition. A list of acronyms for the citations is included at the end of the document.

A-posteriori Detection – A binary detection decision based upon the observed (net) signal and a definite criterion of detection. It corresponds to the critical level, L_C. (Lloyd A. Currie, "Limits for Qualitative Detection and Quantitative Determination," Analytical Chemistry, 586-593, 1968)

A-priori Detection – An estimate, based on a knowledge of the probability distribution of a net signal, of the detection capabilities of a given measurement process. It corresponds to the detection limit, L_D. (Lloyd A. Currie, "Limits for Qualitative Detection and Quantitative Determination," Analytical Chemistry, 586-593, 1968)

Accuracy – The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components, which are due to sampling and analytical operations; a data quality indicator. (NELAC)

Alpha, (α) – The tolerated probability of a "false positive" (i.e. Type I error). See False Positive.

Analyst – The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

Analytical Response – A numerical observation whose magnitude is related to the amount or concentration of the analyte in a sample. One or more analytical responses (as specified by a method) are used, in conjunction with a calibration curve or factor), to produce an analytical result. (D.T.E. Hunt and A.L. Wilson. "The Chemical Analysis of Water")

Analytical Result - A numerical estimate of the concentration of an analyte in a sample, which is obtained by carrying out once the procedure specified in an analytical method. Note that a method may specify analysis of more than one portion of a sample in order to produce one analytical result. (D.T.E. Hunt and A.L. Wilson. "The Chemical Analysis of Water")

Audit – A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity. (EPA-QAD)

Batch – Environmental samples that are prepared and/or analyzed together with the same process and personnel and using the same lot(s) of reagents. (NELAC)

Beta, (β) – The tolerated probability of a "false negative" (i.e. Type II error). See False Negative.

Bias – The constant or systematic distortion of a measurement process, different from random error, which manifests itself as a persistent positive or negative deviation from the known or true value. This can result from improper data collection, poorly calibrated analytical or sampling equipment, or limitations or errors in analytical methods and techniques. (EPA-QAD)

Blank – A specimen that is intended to contain none of the analytes of interest and which is subjected to the usual analytical or measurement process to establish a zero baseline or background value. (NELAC) Blanks include:

- Equipment Blank: a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

- Field Blank: blank prepared in the field by filling a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)

- Instrument Blank: a clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

- Method Blank: a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)

- Reagent Blank: (method reagent blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Blind Sample – A sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process. (NELAC)

Calibration – Set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by material measure or a reference material, and the corresponding values realized by standards. (VIM)

Calibration Curve – The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

Calibration Method – A defined technical procedure for performing a calibration. (NELAC)

Calibration Standard – A substance or reference material used to calibrate an instrument. (QAMS)

Censored Data – Date reported only as below or above some threshold. (USGS)

Censored Method – See Method.

Data Quality Objectives – Qualitative and quantitative statements derived from the DQO Planning Process that clarify the purpose of the study, define the most appropriate type of information to collect, determine the most appropriate conditions from which to collect that information, and specify tolerable levels of potential decision errors. (EPA-QAD)

Degrees of freedom – A statistical parameter, based on the amount of data (number of samples) used in a calculation.

Detection – To have obtained experimental evidence that the analyte concentration is greater than zero. (D.T.E. Hunt and A.L. Wilson. "The Chemical Analysis of Water," 2nd edition, 1986, page 289. The Royal Society of Chemistry, Burlington House, London W1V OBN)

Effluent Limitation (EL) – Restrictions established by a state or EPA on quantities, rates, and concentrations in pollutant discharges. (EPA-TRS)

Environmental Laboratory Advisory Board (ELAB) – A Federal Advisory Committee, with members appointed by EPA and composed of a balance of non-state, non-federal representatives, from the environmental laboratory community, and chaired by an ELAB member. (NELAC)

False Negative Quality Control Sample – The false negative quality control sample (FNQS) is a method blank (e.g., reagent water) or "clean" sample that is spiked at (or near) L_D with the analyte of interest and processed through the entire analytical procedure to verify that such a spike will produce a detection. (Osborn, Kenneth and Thomas Georgian. "The Limits of Method Detection Limits," *Water Environment & Technology* (December, 2004)).

False Negative – Concluding that the analyte is absent when in fact it is present.

False Positive – Concluding that the analyte is present when in fact it is absent.

Holding Times (Maximum Allowable Holding Times) – The maximum times that samples may be held, after the sample is taken, prior to analysis and still be considered valid or not compromised. (40 CFR Part 136)

Hypothesis Test – A statistical procedure for determining if a sample provides sufficient evidence to reject or accept one statement regarding the population of interest in favor of an alternative

statement. (EPA-QAD)

Inter-laboratory Procedure Study – A study where a centralized study design coordinator sends identical[11] samples to multiple different laboratories for analysis. The resulting raw data are analyzed by the study design coordinator by a given procedure to provide estimates of L_C, L_D and/or L_Q. The laboratories would generate only data that would be submitted to the study design coordinator who would compile the data, evaluate it and generate an inter-laboratory L_C, L_D and/or L_Q.

Inter-laboratory Test Comparison – Organization, performance and evaluation of tests on the same or similar items or materials by two or more laboratories in accordance with predetermined conditions. (ASTM)

International Union of Pure and Applied Chemistry 1997 Definitions – IUPAC definitions for L_C, L_D, and L_Q have been reproduced in Appendix I. For a more complete description see chapter 18 in the IUPAC document Compendium of Analytical Nomenclature, Definitive Rules 1997, 3rd Edition.
IUPAC website: http://www.iupac.org/publications/analytical_compendium/

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample) – A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (NELAC)

Laboratory Duplicate – Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

L_C DETECTION – LAYPERSON'S DEFINITIONS -

1. **Critical Value (L_C) -** The minimum result which can be reliably discriminated from a blank (for example, with a 99% confidence level).
2. **Critical Value (L_C)** – The lowest result that can be distinguished from the blank at a chosen level, α, of statistical confidence.

L_D DETECTION – LAYPERSON'S DEFINITIONS -

1. **Detection Limit (L_D) -** The lowest true concentration that will almost always be detected. (The Committee wants the term "detected" to be modified.)
2. **Detection Limit (L_D) -** The minimum detectable value is smallest amount or concentration of a particular substance in a sample that can be reliably detected by a specific measurement process.
3. **Detection Limit (L_D) -** The minimum true concentration that will return a result above the critical value given a specific measurement process and confidence level.

[11] Identical in every way possible including, but not limited to analyte concentrations, matrices, etc.

L_C DETECTION - STATISTICAL DEFINITIONS -

1. **Critical Value (L_C)** - Smallest measured amount or concentration of analyte in a sample that gives rise to a Type I error tolerance of alpha under the null hypothesis that the true amount or concentration of analyte in the sample is equal to that of a blank. (The alternative hypothesis is that the true amount or concentration of analyte is greater than that of a blank.)
2. **Critical Value (L_C)** - The minimum observed result such that the lower 100 (1-α)% confidence limit on the result is greater than the mean of the method blanks.

L_D DETECTION - STATISTICAL DEFINITIONS -

1. **The Minimum Detectable Value (L_D)** - Once L_C is established, L_D is the smallest concentration or amount of analyte at which the tolerance for Type II error is equal to beta.
2. **The Minimum Detectable Value (Ld)** - The lowest true concentration such that the frequency that the result is greater than L_C will be 100% (1-β).

L_Q QUANTITATION DEFINITIONS -

1. **Quantification Limit (L_Q)**: The smallest detectable concentration of analyte greater than the detection limit where the required* accuracy (precision & bias) is achieved for the intended purpose.

*Note: EPA requested additional conversation around the use of the word required in the definition.

Matrix – The material of which the sample is composed or the substrate containing the analyte of interest, such as waste water, stormwater, and biosolids. Also called medium or media. (EPA-QAD)

Matrix Effects – Manifestations of non-target analytes or physical/ chemical characteristics of a sample that prevents the quantification of the target analyte (i.e., the compound or element of interest being quantified by the test method) as it is routinely performed, typically adversely impacting the reliability of the determination. For example, a matrix effect can give rise to a high or low bias. (EPA-QAD)

Matrix Spike (spiked sample or fortified sample) – A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (QAMS)

Maximum Contaminant Level (MCL) – This is a contaminant-specific standard for acceptable drinking water under SDWA. MCLs also may be used for purposes of RCRA (Resource Conservation and Recovery Act) ground water monitoring to reach contaminant-specific clean-up levels.

Measurement Quality Objectives – Qualitative and quantitative statements of the overall level of uncertainty that a decision maker is willing to accept in results or decisions derived from measurements. MQOs/DQOs provide the statistical framework for planning and managing measurement plans consistent with the data user's needs. (EPA-QAD)

Median – The middle number or center value of a set of data in which all the data are arranged in sequence. (www.asq.org/info/glossary/a.html)

Method – 1. See Test Method. 2. Logical sequence of operations, described generically, used in the performance of measurements. (EPA-QAD)

> **Censored Method –** Analytical methods that frequently produce non-numerical results for blanks (i.e. ND for "non-detect"). (EPA-QAD)
>
> **Uncensored Method –** Analytical methods that nearly always produce numerical values for method blanks. (EPA-QAD)

Method Blank – For aqueous analysis, an unspiked or non-fortified reagent water sample which proceeds through the entire testing method, including all preparatory and determinative steps. (EPA-QAD) NELAC states that this should be the same matrix as samples, already addressed under "Blank."

Multi-laboratory Procedure Study – A study where multiple laboratories individually perform a L_C, L_D and/or L_Q estimation procedure (usually using self-selected spiking concentrations) and those individual estimates are summarized in some fashion (e.g. averaging, upper or lower confidence intervals) to characterize some measure of how well the analytical method performs in qualified laboratories. The multi-lab procedure study would include two steps: First, each individual lab would conduct the analysis and generate their unique L_C, L_D and/or L_Q level. Second, those levels would then be compiled from all laboratories, evaluated, and based on criteria, used to propose multi-lab L_C, L_D and/or L_Q levels, where appropriate[12].

National Environmental Laboratory Accreditation Conference (NELAC) – A voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. (NELAC Appendix A, Glossary, July 2005)

National Environmental Laboratory Accreditation Program (NELAP) – The overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (NELAC Appendix A, Glossary, July 2005)

Numeric Target – A measurable value determined for the pollutant of concern which, if achieved, is expected to result in the attainment of water quality standards in the listed waterbody. (EPA-TRS)

Outlier – An observation that is shown to have a low probability of belonging to a specified data population; any item rejected by the sampler, analyst, or data reviewer, usually accompanied by an attendant explanation.

Performance Based Measurement System (PBMS) – A set of processes wherein the data quality needs, mandates, or limitations of a program or project are specified, and serve as criteria for

[12] If, for example, there was a determination that variations in instrument design or analytical technique resulted in sensitivity differences that could not realistically be pooled, they may be excluded based on criteria.

selecting appropriate methods to meet those needs in a cost-effective manner. (October 6, 1997 62 FR 52098)

Power – the probability of reporting an analyte as detected at a given true concentration when the analyte is actually present. Statistical power equals one minus the Type II error. The power is dependent on the true concentration of a sample. (Note: if L_C is defined in terms of the blank rather than a concentration of zero, this definition is inappropriate. The definition would be the probability of reporting the level of analyte in a sample is greater than that observed in a blank, given that the true concentration in the sample is greater than that of the blank.)

Practical Quantitation Level (PQL) – Means the lowest concentration that can be reliably measured within specified limits of precision and accuracy for a specific laboratory analytical method during routine laboratory operating conditions. (EPA-TRS)

Precision – The consistency of measurement values quantified by measures of dispersion such as the sample standard deviation. Precision must be defined in context – e.g., for a certain analyte, matrix, method, perhaps concentration, lab or group of labs. (NELAC)

Protocol – A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis), which must be strictly followed. (EPA-QAD)

Quality Assurance – An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

Quality Assurance [Project] Plan (QAPP) – A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EPA-QAD)

Quality Control – The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

Quality Control Sample – An uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA-QAD)

Quantification Limit – A performance characteristic that marks the ability of a Chemical Measurement Process to adequately "quantify" an analyte. (IUPAC)

Quantitation versus Quantification – These are considered equivalent and can be used interchangeably. Both are commonly used in the literature.

Range – The difference between the minimum and the maximum of a set of values. (EPA-QAD)

Recovery – The degree to which a methodology measures all of the analyte contained in a sample, often expressed in percent recovered.

Relative Standard Deviation (RSD) – The standard deviation expressed as a percentage of the mean (i.e., the coefficient of variation). Mathematically, it is the mean divided by the standard deviation times one hundred percent.

Replicate Analyses – The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

Reporting Limit – The minimum value below which data are documented as non-detects. (EPA-QAD)

Sample – A representative part or a single item from a larger whole or group especially when presented for inspection or shown as evidence of quality. (Webster's)

Sensitivity – Sensitivity generally refers to the capability of a method or instrument to discriminate between small differences in analyte concentration.

Spike – A known quantity of an analyte added to a sample for the purpose of determining recovery or efficiency (analyst spikes), or for quality control (blind spikes).

Standard Deviation – A computed measure of variability indicating the spread of the data set around the mean.

Standard Operating Procedures (SOPs) – A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

Standard Uncertainty – Uncertainty of the result of a measurement expressed as a standard deviation. (NIST)

Test Method – An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP or published by a recognized authority. (NELAC)

Type I Error – See Alpha and False Positive.

Type II Error – See Beta and False Negative.

Uncensored Method – See Method.

Uncertainty – The range of values that contains the true value of what is being evaluated at some level of confidence.

Uncertainty (of measurement) – A parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand. (NIST)

Variability During Routine Operations – Changes during the routine running of samples that might contribute to variability of results. This might include instrument drift through the course of the day due to changes in the ion source (such as contamination from running samples), differences in performance of instruments used for the same analysis, difference in technique for different analysts, etc.

Water Quality Based Effluent Limit (WQBEL) – Effluent limitations applied to dischargers when mere technology-based limitations would cause violations of water quality standards. Usually WQBELs are applied to discharges into small streams. (EPA-TRS)

List of Acronyms

ASTM - American Society for Testing and Materials; Dictionary of Engineering Science and Technology, 9th Edition
EPA OSWER - US EPA Office of Surface Water
EPA-QAD - US EPA Quality Assurance Division
EPA-TRS – EPA Terminology Reference System
G&C - Gibbons and Coleman textbook
IUPAC – International Union of Pure and Applied Chemistry
NELAC - National Environmental Laboratory Accreditation Conference
NIST - National Institute of Standards and Testing
QAMS - US EPA Quality Assurance Management Section
USGS - US Geological Survey
VIM - International Vocabulary of Basic and General Terms in Meteorology
Webster's - Webster's Dictionary

APPENDIX G:

Contractor Information

Triangle Associates, Inc.
Triangle Associates is a consulting firm of professionals committed to helping people understand and resolve public policy issues and environmental conflicts. Triangle provides public involvement, facilitation/mediation services and environmental education programs to public agencies, businesses, and communities.

Triangle designs processes and programs that are tailored to the unique needs of each client.

- We serve as a neutral third party, helping clients resolve politically charged and scientifically complex issues.
- We facilitate the work of multi-party, collaborative groups to reach agreements that meet the needs of all parties. Our many successes include decisions about the future of old growth forests on the Olympic Peninsula, watershed management plans, reducing airport noise, clean up of Hanford's hazardous and radioactive waste, revitalization of an urban center, and keeping transportation projects on-track.
- We specialize in designing and carrying out comprehensive public involvement programs for public agencies so that communities are informed and can shape successful outcomes.
- We design and present innovative and award-winning educational programs for clients who want to reach out to students of all ages and provide them with the knowledge and tools to make smart choices in the future.

Triangle's facilitation team for the Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act programs included Alice Shorett, Robert Wheeler, Vicki King, Cole Gainer and Derek Van Marter.

A woman-owned business, Triangle was founded by Alice Shorett in 1979 and is now an employee-owned company. Additional information about the firm is available at www.triangleassociates.com.

CSC
CSC is a global leader in the information technology arena with 87,000 employees and 48 years of delivering high quality business results to Federal and commercial clients worldwide. We support a broad range of industries, including Government; Chemical, Energy and Natural Resources; Health Services; Transportation; Banking and Financial Services; Aerospace and Defense; Manufacturing; and Communications. CSC Environmental Solutions, has partnered with EPA for the last 29 years providing scientific, statistical, engineering, policy, regulatory, training, and information technology support that exceeds customer expectations. CSC Environmental Solutions currently supports water, hazardous waste, air, research and development, and pesticide programs, and EPA's Office of Environmental Information. Our primary focus is on ensuring that EPA has access to high

quality data to support decision-making and that EPA's actions and the data that supports them are appropriately communicated to stakeholders. The majority of our work is in data assessment, analytical method development (chemistry, biochemistry, microbiological, molecular, and radiochemistry methods), statistical data applications, environmental study design and management, water security, laboratory program management, training/outreach, and information management for environmental programs.

CSC Staff who were involved supporting EPA's Office of Water during the FACDQ through the coordination and management of the FACDQ Pilot Study, evaluation of Pilot Study and other data, and supporting the FACDQ Technical Work Group with statistical and other analyses include Ken Miller, Kristin Leinberger, Harry McCarty, and Lynn Walters. Additional CSC staff that provided intense support in the processing and review of data during the FACDQ Pilot Study includes Barbara Beard, Neal Jannelle, Julie Rest, Cristopher Robinson, Erin Salo, and Maria Vargas.

APPENDIX H

References & Web Links

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US EPA, Water Quality Guidance for the Great Lakes System: Supplementary Information Document, EPA-820-B-95-001, March 1995, http://www.epa.gov/gli05u03/docs/usepa_sid.pdf.

US EPA, *Guidance on Environmental Data Verification and Data Validation (QA/G-8)***,** EPA/240/R-02/004, November 2002, http://www.epa.gov/quality/qs-docs/g8-final.pdf.

US EPA, *Guidance on Systematic Planning Using the Data Quality Objectives Process (QA/G-4)***,** EPA/240/B-06/001, February 2006, http://www.epa.gov/quality/qs-docs/g4-final.pdf.

WEB LINKS

http://www.epa.gov/edocket/
http://www.epa.gov/waterscience/methods/
http://www.epa.gov/waterscience/methods/det/
http://www.epa.gov/waterscience/methods/det/faca/techworkgroup/
http://www.epa.gov/waterscience/methods/det/faca/policyworkgroup/

APPENDIX I

International Union of Pure and Applied Chemistry, 1997 Definitions*

Detection Decisions (L_C). The decision "detected" or "not detected" is made by comparison of the estimated quantity ($\hat{L}$) with the *critical value* (L_C) of the respective distribution, such that the probability of exceeding L_C is no greater than α if analyte is absent ($L = 0$, null hypothesis). The Critical Value is thus the minimum significant value of an estimated net signal or concentration, applied as a discriminator against background noise. This corresponds to a 1-sided significance test.

The above definition of L_C can be expressed as follows,

$$\Pr(\hat{L} > L_C \mid L=0) \leq \alpha \qquad (18.4.9)$$

Generally the equation is stated as an equality, but the inequality is given to accommodate discrete distributions, such as the Poisson, where not all values of α are possible. If $\hat{L}$ is normally distributed with known variance, Eq. 9 reduces to the following simple expression,

$$L_C = z_{1-\alpha}\,\sigma_o \qquad (18.4.10)$$

where $z_{1-\alpha}$ (or z_P) represents the (1-α)th percentage point or critical value of the standard normal variable, and σ_o is the standard deviation of the estimated quantity (net signal or concentration) under the null hypothesis (true value = 0). Taking the default value for α (0.05), $L_C = 1.645\ \sigma_o$.

Note that Eq. 18.4.9, *not* Eq. 18.4.10, is the defining equation for L_C, and the result ($1.645\sigma_o$) applies *only* if the data are normal with known variance and α is set equal to its default value. If σ_o is estimated by s_o, based on ν degrees of freedom, $z_{1-\alpha}$ must be replaced by Student's-t. That is,

$$L_C = t_{1-\alpha,\nu}\, s_o \qquad (18.4.11)$$

For $\alpha = 0.05$ and 4 degrees of freedom, for example, L_C would be equal to 2.132 s_o.

Notes:

(1) Some measurement systems impose an artificial hardware or software *threshold* (*de facto* L_C) to discriminate against small signals. In such cases statistical significance is problematic -- α may be quite small and perhaps unknown, but equations 18.4.12 and 18.4.13 below can still be applied to compute L_D, given L_C and β. The impact of such a threshold can be profound, severely eroding the inherent detection capability of the system.

(2) A result falling below L_C, triggering the decision "not detected" should not be construed as demonstrating analyte absence. (See section 18.4.3.6.) Reporting such a result as "zero" or as "$<L_D$" is *not* recommended; the estimated value (net signal, concentration) and its uncertainty should *always* be reported.

Minimum Detectable Value; Detection Limit (L_D). The *Minimum Detectable Value* of the net signal (or concentration) is that value (L_D) for which the false negative error is β, given L_C (or α). It is the true net signal (or concentration) for which the probability that the estimated value $\hat{L}$ does not exceed L_C is β. The definition of L_D can thus be expressed as

$$\Pr(\hat{L} \leq L_C \mid L = L_D) = \beta \qquad (18.4.12)$$

For normal data having known variance structure, this yields,

$$L_D = L_C + z_{1-\beta}\,\sigma_D \qquad (18.4.13)$$

For the special situation where the variance is constant between $L = 0$ and $L = L_D$, the right side of Eq. 13 reduces to $(z_{1-\alpha}+z_{1-\beta})\sigma_o$; if in addition α and β are equal, this gives $2z_{1-\alpha}\sigma_o$ which equals $2L_C$. Taking the default values for α and β (0.05), this equals 3.29 σ_o. If L_C employs an estimate s_o based on ν degrees of freedom (Eq. 18.4.11), then $(z_{1-\alpha}+z_{1-\beta})$ must be replaced by $\delta_{\alpha,\beta,\nu}$, the non-centrality parameter of the non-central-t distribution. For $\alpha = \beta$, this parameter is approximately equal to $2t$ and the appropriate expression (for constant variance) is,

$$L_D = \delta_{\alpha,\beta,\nu}\,\sigma_o \approx 2t_{1-\alpha,\nu}\,\sigma_o \qquad (18.4.14)$$

For 4 degrees of freedom, for example, the use of $2t$ would give $L_D = 4.26\ \sigma_o$. (The actual value for δ in this case is 4.067.) Note that σ_o *must be used* in Eq. 18.4.14. If only an estimate s_o is available, that means that the minimum detectable value is uncertain by the ratio (σ/s). Using the techniques of section 18.4.3.8, confidence limits may then be calculated for L_D. (A 95% upper limit for L_D, based on an observed s_o with 4 degrees of freedom, would be $\{4.07/(\sqrt{0.178})\}\ s_o$ or $9.65\ s_o$.

Notes:

(1) When ν is large, $2t$ is an excellent approximation for δ. For $\nu \geq 25$, with $\alpha = \beta = 0.05$, the difference is no more than 1 %. For fewer degrees of freedom, a very simple correction factor for $2t$, $4\nu/(4\nu+1)$, which takes into account the bias in s, gives values that are within 1 % of δ for $\nu \geq 5$. For the above example where $\nu = 4$, δ would be approximated as 2(2.132)(16/17) which equals 4.013.

(2) L_D is *defined* by Eq. 18.4.12 in terms of the distribution of $\hat{L}$ when $L = L_D$, the probability of the type-II error β, and L_C, with L_C being defined (Eq. 18.4.9) in terms of the distribution of $\hat{L}$ when $L = 0$, and the probability of the type-I error α. When certain conditions are satisfied, L_D can be expressed as the product of a specific coefficient and the standard deviation of the blank, such as $3.29\ \sigma_B$, when the uncertainty in the mean (expected) value of the blank is negligible, α and β each equal 0.05, and $\hat{L}$ is normally distributed with known, constant variance. L_D is *not defined*, however, simply as a fixed coefficient (2, 3, 6, etc.) times the standard deviation of a pure solution background. To do so can be extremely misleading. The correct expression must be derived from the proper defining equations (Eq. 18.4.9 and 18.4.12), and it must take into account degrees of freedom, α and β, and the distribution of $\hat{L}$ as influenced by such factors as analyte concentration, matrix effects, and interference.

(3) The question of detection has been treated extensively by H. Kaiser for spectrochemical analysis. In the earlier editions of the "Orange Book" the use of $3s_B$ is recommended as the "limit of detection". Although originally intended to serve as a measure of the detection capability, this quantity was then used as the "decision criterion" to distinguish an estimated signal from the background noise. Such a definition, which in effect sets L_C *and* L_D each equal to $3s$, corresponds for a normal distribution (large ν) to a type-I error probability of *ca.* 0.15 % but a type-II error probability of 50 % ! (See the Source Document [Currie, 1995] in section 18.9 for further comments and references.)

Minimum Quantifiable Value; Quantification Limit (L_Q). *Quantification limits* are performance characteristics that mark the ability of a CMP to adequately "quantify" an analyte. Like detection limits, quantification limits are vital for the planning phase of chemical analysis; they serve as benchmarks that indicate whether the CMP can adequately meet the measurement needs. The ability to quantify is generally expressed in terms of the signal or analyte (true) value that will produce estimates having a specified relative standard deviation (RSD), commonly 10 %. That is,

$$L_Q = k_Q \sigma_Q \tag{18.4.21}$$

where L_Q is the Quantification Limit, σ_Q is the standard deviation at that point, and k_Q is the multiplier whose reciprocal equals the selected quantifying RSD. The IUPAC default value for k_Q is 10. As with detection limits, the net *signal quantification limit* (S_Q) and *analyte (amount or concentration) quantification limit* (x_Q) derive from the relations in equations 18.4.1-5, and the variance structure of the measurement process. If the sensitivity A is known, then $x_Q = S_Q/A$; if an estimate $\hat{A}$ is used computing $\hat{x}$, then its variance must be considered in deriving x_Q. (See Note-1, below.) Just as with the case of S_D and x_D, uncertainties in assumed values for σ and A are reflected in uncertainties in the corresponding Quantification limits.

If σ is known and constant, then σ_Q in Eq. 18.4.21 can be replaced by σ_o, since the standard deviation of the estimated quantity is independent of concentration. Using the default value for k_Q, we then have

$$L_Q = 10\, \sigma_Q = 10\, \sigma_o \tag{18.4.22}$$

In this case, the quantification limit is just 3.04 times the detection limit, given normality and $\alpha = \beta = 0.05$.

Notes:

(1) In analogy with x_D, the existence of x_Q is determined by the *RSD* of $\hat{A}$. In this case the limiting condition for finite x_Q is RSD($\hat{A}$) < $1/k$. If x is estimated with Eq. 18.4.5, and $\hat{B}$ and $\hat{A}$ are independent, and $\sigma(\hat{S})$ is constant with value σ_o,

then $x_Q = (k\sigma_o/A)/[1-(k\sigma_A/A)^2]^{½}$, where $(k\sigma_o/A)$ is the limiting result when the random error in $\hat{A}$ is negligible.]

(2) One frequently finds in the chemical literature the term "Determination Limit." Use of this term is *not* recommended, because of ambiguity.

*J. Inczédy, T. Lengyel, A.M. Ure, International Union of Pure and Applied Chemistry, Compendium of Analytical Nomenclature, Definitive Rules 1997, 3rd Edition, Blackwell Science (1998).