

## WORKING DRAFT FOR DISCUSSION

Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act Programs  
November 17, 2005

# Interpretations of Detection and Quantitation Procedures Evaluation Characteristics

### Introduction

The Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act Programs (FACDQ) Technical Work Group (TWG) has refined a matrix drafted by the FACDQ that documents detection and quantitation procedural characteristics important in evaluating procedures for various uses. The evaluation process is supported below with an interpretation of each characteristic in the matrix and specifics on how each procedure should be evaluated. This document will maximize consistency among various reviewers of different procedures, thereby providing a more reliable product for use by the FACDQ. However, it is important to note that the matrix responses provided for the September, 2005 FACDQ meeting reflect interpretations of individual TWG members and do not reflect consensus of the TWG.

The TWG has deleted several of the procedural entries originally introduced by the FACDQ. The LOD, CRV, MDV, IIAG and LOQ entries were removed because they represent definitions for detection and quantitation rather than actual procedures. The evaluation approach identified by the FACDQ requires, for the most part, that a procedure be available for comparison to the characteristics of the matrix. The Navy Uncertainty Estimation Document was found to fall short of a true description of a detection or quantification limit procedure. However many of the concepts of this document may be useful to the FACDQ in future efforts. The OSW quantitation approach, although not much more than a definition in its present form was retained in the matrix because of its current regulatory importance. These changes will help conserve resources without compromising information.

The FACDQ TWG added the Osborn Lab QC detection procedure to the matrix because its characteristics were not adequately represented by other procedures. Although both the ACIL and the Consensus Group Detection Limit procedures are implementations of the ISO/IUPAC approach to detection, it was brought to the TWG's attention that the Water Research Centre also had a procedure which implemented ISO/IUPAC. Thus, for completeness, this was added to the matrix.

The procedure evaluation characteristics follow.

### Measurement Quality Objectives

Bias - Is bias explicitly derived by the procedure? (Y/N)

Precision - Is precision explicitly derived by the procedure? (Y/N)

% False Positives - Does the procedure provide for selection of a Type I error tolerance limit?  
(NA/Y/N)

% False Negatives - Does the procedure provide for selection of a Type II error tolerance limit?  
(NA/Y/N)

## WORKING DRAFT FOR DISCUSSION

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Qualitative Criteria Considered - Does the procedure require that qualitative identification take place at the determined detection or quantitation limit? (NA/Y/N)

Uncertainty Calculated - Does this procedure include a protocol or requirement to calculate the confidence interval for the parameter in question (Lc, Ld, Lq)? (Y/N)

Concentration Estimate Uncertainty – Does the procedure provide estimates of bias and/or precision as a function of concentration? (Y/N)

### **Evaluation of Method Performance**

Limit Evaluation Frequency - At what frequency does the procedure specify that detection or quantitation limits are to be determined? (Response options are: not specified, once, monthly, annual, on-going, other (specify))

Data Generation Frequency – How frequently does the procedure require data to be generated to support development of detection and quantitation limits? (Response options are: not specified, once, monthly, annual, on-going, other (specify))

Reflects Routine Performance - Does the procedure yield detection or quantitation limits that are reflective of routine test method performance (when executed by qualified staff in the absence of matrix effect, which are addressed below)? (Y/N)

Addresses Matrices – Does the procedure describe how to modify a detection or quantitation limit for applicability to real world samples? (Y/N)

Evaluates Entire Test Method - Does the procedure evaluate the entire test method including sample preparation and clean-up steps? Respond with “NC” if the procedure does not evaluate the entire test method but could be modified to evaluate the entire test method and footnote effort required for this modification. (Y/N/NC)

Addresses Recovery - Does the procedure explicitly adjust or account for impacts of recovery on analytical results? Respond with “NC” if the procedure does not adjust or account for impacts on recovery but could be modified to do so and footnote effort required for this modification. (Y/N/NC)

Consistent or Chronic Blank Bias Addressed - 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 contamination of laboratory blanks). Respond with “NC” if the procedure does not adjust or account for situations where method blanks always return a non-zero result/response but could be modified to do so and footnote effort required for this modification. (Y/N/NC) Comment: need to clarify what is being asked and why.

Intermittent Blank Contamination - Does the procedure explicitly adjust or account for situations where method blanks are intermittently contaminated? Respond with “NC” if the procedure does not explicitly adjust or account for situations where method blanks are intermittently

## WORKING DRAFT FOR DISCUSSION

Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act Programs  
November 17, 2005

contaminated but could be modified to do so and footnote effort required for this modification.  
(Y/N/NC). Comment: Is this characteristic applicable to detection /quantitation procedures?

Non-Zero Results - Does the procedure adjust for results that are less than zero? Respond with “NC” if the procedure does not adjust for results that are less than zero but could be modified to adjust for results that are less than zero and footnote effort required for this modification.  
(Y/N/NC/NA) Comment: Based on IUPAC/ISO definitions where a result is equal to sample response minus the blank response, is this characteristic necessary?

### **Prescriptive\Descriptive**

Prescriptive - Are specific, numeric performance benchmarks specified in the procedure in order to demonstrate and maintain proficiency? (Y/N)

*or*

Descriptive - Is the procedure intended to measure the current performance of the laboratory with regard to detection or quantitation limits, regardless of performance benchmarks? (Y/N)

Bias - What is the limit for bias specified (either explicitly or by the user) in the detection procedure? (If a limit is not specified, enter ‘None’.)

Precision - What is the limit for precision specified (either explicitly or by the user) in the quantitation procedure? ( If a limit is not specified, enter ‘None’.)

% False Positives - What is the default value (in percent) of false positive results (Type I errors) defined in the procedure? (If the frequency is not addressed, enter either ‘NA’ or ‘None’ depending on the procedure.)

% False Negatives - What is the default value (in percent) of false negative results (Type II errors) defined in the procedure? (If the frequency is not addressed, enter either ‘NA’ or ‘None’ depending on the procedure.)

### **Degree of Procedural Complexity**

Data Processing - What is the degree of complexity associated with processing analytical test method data to compute or determine detection or quantitation limits? (Rate from 1 to 10; 1 representing high complexity, 10 representing low complexity.)

Laboratory Procedures - What is the degree of complexity (or work) associated with laboratory activities required to compute or determine detection or quantitation limits? (Rate from 1 to 10; 1 representing high complexity, 10 representing low complexity.)

Is it a Clearly Written Procedure - Is the procedure written in such a way that it will be uniformly interpreted and implemented by qualified laboratory staff? (Y/N)

Can the Procedure be Clearly Written - Can the procedure be revised or re-written to yield a version that would be uniformly interpreted and implemented by qualified laboratory staff? (If answered ‘Y’ to previous question, enter ‘NA’ or ‘N’)

## WORKING DRAFT FOR DISCUSSION

Federal Advisory Committee on Detection and Quantitation Approaches and Uses in Clean Water Act Programs  
November 17, 2005

Ability to Communicate Concepts - Can the concepts embodied by the procedure be communicated in an effective manner to all interested stakeholders? (Rate from 1 to 10; 1 being very difficult to communicate the concepts, 10 being easy to communicate the concepts.)

**Relative Cost** - What is the relative cost of the procedure? (Rate from 1 to 10; 1 representing highest relative cost, 10 representing lowest relative cost.)

### **Interlaboratory Procedure**

As Written - Does the procedure account for interlaboratory variability in the determination of detection or quantitation limits? (Y/N)

Could be Written as - Could the procedure be written to account for interlaboratory variability in the determination of detection or quantitation limits? (If answered 'Y' to previous question, enter 'NA' or 'N')

**Relative Implications** - What is the relative impact of implementing the procedure on existing CWA programs? (Rate from 1 to 10; N/A for no impact; 1 for high impact, 10 for low impact.)

### **Procedural Properties**

Detection - What measure is used to define detection? (L<sub>c</sub>, L<sub>d</sub>, Other, NA, Unknown)

Quantitation - What measure is used to define quantitation? (L<sub>Q</sub>, %RSD, NA, Other, Unknown)

Can be Evaluated with Existing Data - Can the detection or quantitation procedure be evaluated using available, existing data? (Y/N)

Has been Evaluated with Existing Data - Has existing data been used to evaluate the detection or quantitation procedure? (Y/N/unknown)

One Size Fits All - Is the procedure applicable equally to all laboratories and test methods? (Y/N)

Flexibility for Each - Does the procedure allow flexibility to accommodate various laboratory or test methodology considerations? (Y/N)

### **Data Types**

Censored Methods - Can the procedure explicitly handle data generated using censored methods (as defined in the Glossary document)? (Y/N)

Uncensored Methods - Can the procedure explicitly handle data generated using uncensored methods (as defined in the Glossary document)? (Y/N)

## WORKING DRAFT FOR DISCUSSION

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### **Defensibility**

Scientific - Do you judge the procedure to be scientifically defensible? (Y/N)\*

Legal - Do you judge the procedure to be legally defensible? (Y/N)\*

\* The FACDQ TWG does not plan on addressing these characteristics directly given that they are policy oriented; these characteristics should therefore be addressed by the FACDQ.