

WORKING DRAFT FOR DISCUSSION

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

Multi-lab Pilot Study Design Progress Report

Potential Use of Existing Data

The sub-group is still identifying and compiling potential sources of existing data that may be helpful in addressing the pilot study objectives or in designing the pilot study. Although the sub-group needs to give further consideration to potential use or uses of existing data, it was determined that some of the experiences gained in the Michigan PCB IQE study and the LCMRL validation study may begin to answer some of the pilot study objectives. In particular, it was felt that these experiences could address Objectives 1 and 2. To date, similar potentially useful studies/datasets for the IDE have not been identified. Evaluation of the utility of existing data to address the Hubaux-Vos approach cannot be undertaken until the drafting of the procedure which implements it has been completed. All existing data will need to go through a pre-qualification process determined by the Technical Work Group.

Multi-Laboratory Study Design

A. Description of Recommended Approach

It was recognized that most if not all (see discussion of LCMRL in following paragraphs) of the candidate inter-laboratory procedures rely upon very similar data; specifically, measures of method performance (either precision or recovery) over a wide concentration range.

Furthermore, the procedures for generating the data are nearly identical; each procedure requires that identical spiked samples be distributed to a number of different laboratories.

Because of these similarities in how the raw data are generated, it was decided that the most powerful experimental approach at this point would involve developing an extensive dataset of inter-laboratory method performance over an appropriate concentration range. This would involve sending identical spiked samples to multiple laboratories and compiling the results into a master database. The exact spike levels would not be selected to conform to any specifications in any of the procedures, but would attempt to span the concentrations that would be required by all of the different approaches. Ultimately, the dataset would be designed to include more spiking levels and more laboratories than any one procedure required.

Although it is recognized that there could be some synergy or overlap in the spiking ranges for a dataset that would evaluate the IDE (the only inter-laboratory procedure that addresses L_c and L_d) and the other three procedures, it will not be possible to fully appreciate any potential cost savings until specific analytical methods are selected and a more detailed discussion of the optimum spiking concentrations is undertaken. Therefore, it may require a separate (or at least an expanded) database if it is necessary to evaluate the IDE, but the data required to evaluate the other three procedures should be identical.

Different sub-sets of this master database could be run through the different inter-laboratory procedure calculation protocols. Because we would have more than the minimum dataset required to implement each inter-laboratory procedure, we could use different sub-sets of data to

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explore the importance or dependence on spike concentrations, number of spike levels, number of laboratories, etc. This would facilitate a thorough evaluation of Objective 4.

There are several advantages to this approach. First, it should allow a thorough testing of how each procedure's results varies with various experimental parameters. If one or more procedures are found to be highly dependent on experimental design parameters, the FACDQ may decide not to consider them further. It is also quite possible that this thorough ruggedness testing might reveal opportunities to improve the different approaches (more or fewer replicates, spike levels, outlier testing procedures, etc) and/or to provide insight into where better guidance on selecting experimental variables might improve the procedure.

Although this approach does not provide specific information regarding Objectives 1 and 2, we feel that the above-mentioned study experiences provide some answers to those questions and that those issues about clarity of instructions, etc. may not necessarily need extensive pilot testing. The FACDQ should also understand that interlab procedures only require labs test specific spikes; the procedures do not require the labs to actually calculate detection or quantitation limits. An assessment of the capability of labs to conduct the procedures is not necessary. However, it will be necessary to assess Objectives 1 and 2 for interlab procedures at the level where interlab data is assembled and processed (EPA, states, consultants, etc.). Thus, it was felt that the proposed design would provide the best value in terms of information helpful to the FACDQ for the funds available.

A specific approach or procedure to validate the estimates of L_c , L_d , or L_q has not yet been determined and the Technical Work Group will need to continue looking into this. Also, it would be desirable for the single-laboratory and multi-laboratory sub-groups to coordinate in selection of candidate analytical methods to the extent practical.

B. Special Issues Regarding the LCMRL

In its present form the LCMRL is a single-laboratory procedure. In the LCMRL validation study, EPA-OGWDW explored two alternative approaches for using the single-laboratory data to formulate something that might equate to its historical Practical Quantitation Limit. Both approaches start with a measure of the central tendency of the single-laboratory LCMRLs (e.g., the mean). From there, one approach is to also calculate the standard deviation, then compare the mean plus three sigma to PQL values. The other approach simply explores using the mean times various multipliers. While these approaches involve analyzing data from different laboratories, they are not, strictly speaking, inter-laboratory (per our glossary definition). For discussion purposes, we refer to these approaches as multi-laboratory LCMRLs. However, the sub-group members noted that if identical spiked samples were sent to a number of different laboratories, the LCMRL concept and computational procedures could be implemented in an inter-laboratory context analogous to the IQE and Hubaux-Vos procedures (again, for discussion purposes they would be termed inter-laboratory LCMRLs). Additional input from the FACDQ on which of these approaches is preferable will be critical to furthering the discussion on how the multi-laboratory pilot study should be designed.

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From the point of view of the multi-laboratory pilot study design, if the FACDQ prefers to pilot test the multi-laboratory LCMRL a dedicated LCMRL pilot would be required, as it gathers data in a manner different from all the other procedures. If the inter-laboratory LCMRL is preferred, it could be pilot tested within the same study design as the IQE and Hubaux-Vos procedures.

Issue/Question for FACDQ: In considering the DL/QL uses where it is determined that an inter-laboratory DL or QL would be most appropriate, should this be implemented through a multi-laboratory design (e.g. pooling or otherwise utilizing single laboratory data) or through a true inter-laboratory (see glossary) design?

Other Study Design Considerations/Recommendations

All inter-laboratory studies are, by their natures, costly and resource intensive. Therefore, the Technical Work Group recommends that the pilot study be designed as a staged or stepwise study. The specifics of how that might best be accomplished have not been worked out and may require better definition of the scope, etc. from further discussion of policy issues by the FACDQ. However, in making this recommendation, the Technical Work Group would prefer to develop a step-wise design that would not require FACDQ consultation and approval to proceed through the steps in order to provide continuity of the pilot study. While written pilot study updates could be made available to the committee, the practicalities of requiring an interactive FACDQ with the pilot study would create real difficulties in timing and the potential need for numerous FACDQ meetings. The TWG proposes that it continue working on the design of the pilot to include clarification of how such a step wise approach would be implemented and have that plan available for review and approval by the FACDQ prior to initiation of the pilot.

Question/Issue for the FACDQ: Should the TWG continue the design of the pilot study incorporating and detailing how a stepwise approach would be utilized with the understanding that the plan would be initially approved by the FACDQ but thereafter, implementation would not require FACDQ approval, but rather be tracked by the Technical Work Group?