

CHAPTER 6

Data Review Reports

6-1. Introduction.

The primary objective of this review is to ensure that analytical sensitivity is adequate for project-specific action levels and to ensure that data are reported in a manner that is consistent with the laboratory's detection and quantitation limits. The evaluation of sensitivity will be a function of how the detection, quantitation, and reporting limits are defined and whether or not action levels are specified. Since it is impractical to discuss sensitivity in the context of multiple definitions for these limits, they will be defined as the **method detection limits (MDLs)**, **method quantitation limits (MQLs)**, and **method reporting limits (MRLs)** presented in the glossary. In particular, it is assumed that the detection limit is the method detection limit of 40 CFR, Appendix B, Part 136. The method quantitation limit is defined (primarily) as the low level calibration standard adjusted for method specific factors. Lastly, the method reporting limit is defined as the threshold or censoring limit below which target analyte concentrations are reported as "ND" (i.e., not detected) or as "<" (i.e., "less than").

6-2. Method Reporting Limits.

6-2.1. Establishing Method Reporting Limits.

a. The definition of the MRLs must be declared in each data package or in project documents such as the Quality Assurance Project Plan (QAPP).

Note: Merely listing numerical values for the MRLs will not satisfy this reporting requirement; the MRLs must be defined in terms of the laboratory's actual quantitation and detection limits.

b. In general, any analyte concentration greater than the detection limit may potentially be reported either as a "detection" or as a "nondetection" with respect to some censoring (reporting) limit greater than the detection limit. For example, if an action level, AL, is very large relative to the MQL, then it may be desirable to report all analyte concentrations less than 5% of the AL action level as "< MRL" or "MRL U," where $MQL < MRL = 0.05 AL$. In this context, "< MRL" indicates that (i) the analyte is present below the detection limit, or (ii) was detected at some concentration greater than the detection limit but less than 0.05 AL. Conversely, if low-level reporting is desirable, then it may be appropriate to establish a censoring limit (MRL) at some concentration greater than the method detection limit (MDL) but less than the MQL. Under these circumstances ($MDL < MRL < MQL$), analyte concentrations between the MRL and MQL would be reported as estimated and concentrations less than the MRL would be reported as "< MRL."

Note: The term *reporting limit* is being defined in a more general manner than is conventionally used for environmental testing. For example, according to the CLP Statement of Work (SOW) for organic analyses, the reporting limit for nondetections is nec-

essarily the CRQL. If an analyte is “not detected,” the reporting limit is the CRQL and detections below the CRQL are reported as estimated. *However, there is no a priori reason for setting the reporting limit equal to the quantitation for all data uses.* For example, presence-absence issues can typically be resolved at concentrations that are significantly less than the quantitation limits.

c. In the absence of project-specific guidance, assume that all reliable detections greater than the MDL or MRL must be reported. (Note that detections should be reported based upon the laboratory’s detection limits as well as the analyst’s judgement.) In addition, assume that the MRL for a **nondetection** must be no less than the **limit of identification (LOI)** or the **reliable detection limit (RDL)**. The RDL and LOI are approximately two times the MDL.

Note: Establishing *any* reporting limit constitutes a form of “data censoring.” Censoring results (i.e., reporting nondetections to twice the MDL) will typically be appropriate when action levels have been established and the results (detections and nondetections) are being compared to the action levels on a point-by-point basis. *However, this approach will not be appropriate for all projects.* For statistical applications, it is usually desirable to report results *without any censoring* (e.g., to report results less than the MDL). The reviewer must refer to project-specific objectives prior to performing censoring or evaluating the data with respect to the reporting limits.

6-2.2. Qualification.

a. *If the reporting limit is less than the LOI or the RDL (i.e., two times the MDL), then qualify nondetections (at the reporting limit) with the UN flag and discuss the potential high false negative probability at the reporting limit in the data review report.* Alternatively, if the project action levels (ALs) are relatively high (e.g., at least 10 to 20 times greater than the MQL), increase the reporting limit to the quantitation limit (if the quantitation limit was established by the lowest calibration standard) and qualify nondetections with the U flag.

b. If an action level (AL) is available, compare the MRL to the AL. *If the MRL is greater than the AL, qualify nondetections with the X or XU flag (since false negatives have not been adequately addressed).*

c. It is recommended that the MRL be no higher than %5 to 10% of the AL. If the MRL is less than but near the AL, then use professional judgement to qualify nondetections, especially when the AL is less than the MQL or the LCS acceptance limits are wide. For example, if the MQL = 50 ppb, the MRL = 10 ppb, AL = 15 ppb, the LCS acceptance range is 50–150% (e.g., for a 100 ppb spike near the mid-calibration range), and the LCS recovery associated with a set of environmental samples is 55%, then nondetections reported as “< 10 ppb” do not demonstrate the 15-ppb action level was met. Under these circumstances, nondetections would be qualified with the X or XU flag

6-3. Method Quantitation Limits.

6-3.1. Establishing Method Quantitation Limits.

a. Project planning documents (e.g., the QAPP) must define what constitutes a quantitation limit. In general, project documents should specify tolerances for uncertainty at the quantitation limit and strategies for verifying the tolerances have been satisfied (e.g., a low-level LCS at the quantitation limit must be recovered to within 20% of its expected value). *Unfortunately, quantitation limits are often poorly defined.*

Note: The laboratory's reported MQLs must not be evaluated solely upon the basis of "Practical Quantitation Limits" ("PQLs") or "Contract Required Quantitation Limits" (CRQLs) specified in published analytical methods or project documents unless these quantities are adequately defined (e.g., tolerances for uncertainty at the quantitation limits are specified).

b. The guidance presented below will typically be applicable.

(1) A low-level LCS or CCV (spiked with the target analytes at or near the MQL) may have been analyzed to verify the quantitation limit. Low-level CCVs would be appropriate for methods that do involve significant sample preparation or for methods in which the calibration standards are prepared with the environmental samples. Low-level CCVs can often be used to verify the quantitation limits for inorganic methods (e.g., when the sample preparatory process does not introduce too much uncertainty). However, this approach will not be valid for methods that involve significant sample preparation and the CCVs are not processed with the environmental samples. Under these circumstances, a low-level LCS (spiked with target analytes at or near the MQL) is required to verify the quantitation limit.

(2) If a low-level CCV (e.g., the lowest calibration standard) was used to check the MQL, verify that the CCV was recovered to within the tolerance for instrumental uncertainty (the acceptance limits must be equal to or slightly greater than the acceptance limits for mid-level CCVs). For example, for trace metals by ICP, the low-level CCV should be recovered to within 10% to 15% of its expected value. If a low-level LCS was used to check the MQL, then verify that the low-level LCS was acceptably recovered.

(3) If a low-level CCV or LCS spiked at the MQL or near the MQL (e.g., less than two times the MQL) was not analyzed, then compare the reported MDL to each corresponding MQL as discussed below (i.e., verify that each MQL is at least five to ten times greater than the MDL and was established using the lowest initial calibration standard).

(4) Use the calibration data to verify that the laboratory's reported quantitation limit for each analyte is established from the lowest calibration standard (or corresponds to a higher concentration that is within the calibration range).

Note: *This is not a sufficient condition to verify the project-required method quantitation limits.* It is often erroneously concluded that if the initial calibration curve is acceptable (e.g., as indicated by a high correlation coefficient), then the lowest calibration standard will be acceptable for establishing the MQL. However, an acceptable fit for the entire calibration curve does not necessarily imply that the uncertainty will be acceptable at concentrations near the lowest calibration standard. Conventional measures of fit are not adequately sensitive to high variability at the low concentration ranges. For example, when regression analysis is used to fit initial calibration results, a high correlation coefficient is possible when the lowest standard radically deviates from a linear fit (e.g., when instrumental response is inherently nonlinear at low concentrations).

(5) Compare the MDLs (if available) to the corresponding MQLs to ensure that the quantitation limits are sufficiently greater than the detection limits. *If the MQL is established from the lowest calibration standard but is not otherwise defined, ensure that the MQL is at least five to ten times greater than the method detection limit.*

Note: The quantitation limit will be dependent upon the magnitude of the analytical noise (whether chemical or electronic in nature) that constitutes the “background” signal or response for the analysis method, and the project-required tolerance for uncertainty for quantitation. Since the detection limit is measure of “background” response, the quantitation limit must typically be greater than the detection limit *by some multiplicative factor* in order to meet the project-required error tolerance. In general, when a low error tolerance is required, the quantitation limit must be significantly greater than the detection limit.

If it is assumed that the magnitude of the analytical uncertainty is approximately \pm MDL, then the relative uncertainty will be about $\pm 20\%$ at five times the MDL and $\pm 10\%$ at ten times the MDL. (It is being assumed that the standard deviation determined from the MDL study is not strongly dependent upon concentration and there is no significant bias.) However, the actual relative uncertainty will often be higher than 10% to 20% at five to ten times the MDL (e.g., because the standard deviation is often an increasing function of concentration).

(6) If the laboratory’s reported quantitation limit is less than the method quantitation limit calculated from the lowest initial calibration standard and the standard is at least five times greater than the MDL, then increase the quantitation limit using the lowest calibration standard.

(7) If the lowest calibration standard is not at least five times greater than the MDL and an acceptable low-level CCV or LCS was not analyzed to verify the MQL, then the initial calibration results must be evaluated. If the low-level calibration standard is less than five times the MDL, it may be appropriate to use the next highest calibration standard to establish the MQL. If possible, use the equation for the initial calibration curve to calculate the concentration of the lowest calibration standard (i.e., calculate the concentration of the lowest standard from the measured response) and ensure that the calculated value of the lowest standard is within the uncertainty tolerance for the CCV. If it is not possible or practical to determine the MQL from

the calibration data, then set the MQL to five to ten times the MDL, but indicate that the MQL is an estimate in the data evaluation report. Multiply the MDL by at least a factor of ten for ICP analyses.

6-3.2. Qualification.

a. Once the MQLs have been verified or established, *qualify all detections less than the MQLs as estimated using the J-flag* (e.g., unless the X or R flag is more appropriate because significant QC problems are observed).

b. If action levels are available, compare the MQLs to the action levels and ensure that the MQLs are *less* than the action levels. Although the MQLs should have been compared to the project's action levels during the planning stages of the project, sensitivity problems may still occur (e.g., because of dilutions). As a "rule of thumb" the MQL should not be greater than about one half of the AL for inorganic analyses and about one third of the AL for organic analyses.

c. *If the MQL is greater than a corresponding AL, adequate sensitivity has not been demonstrated; qualify detections less than the AL with the X flag.* Under these circumstances (MQL < AL), depending upon project DQOs, it may be appropriate to also qualify detections greater than the AL with the X flag (e.g., when a conservative estimate of contamination is not desirable).

Table 6-1
Data Qualification for Sensitivity When Action Levels Are Available

Sample Result (y) LOI ≤ MRLs ¹	Flag	Remarks
$y < \text{MRL} < \text{AL}$	U	Nondetections
$y < \text{MRL}, \text{MRL} > \text{AL}$	X, XU	
$\text{MRL} \leq y < \text{AL} < \text{MQL}$	X	Detections
$\text{MRL} \leq \text{AL} < y < \text{MQL}$	J or X ²	
$\text{MRL} \leq y < \text{MQL} < \text{AL}$	J	
$\text{MRL} \leq \text{MQL} < y$	No flag	

Notes: 1. The action level, method reporting limit, and method quantitation limit are denoted as AL, MRL, and MQL, respectively. The concentration of the target analyte in a field sample is denoted as y. (It is assumed that the limit of identification is less than or equal to the MRL.) 2. A detection above the AL was obtained. However, because quantitative uncertainty is high, the target analyte may not actually be present in the sample at a concentration that exceeds the AL; the X flag may be appropriate. The use of the J flag constitutes a conservative interpretation of the data (namely, that the AL has been exceeded).