Analytical Chemistry Detection Limits and the Evaluation of Dredged Sediment

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PURPOSE: This technical note presents information clarifying the proper use of analytical chemistry detection limit terminology with respect to the evaluation of dredged material prior to disposal in ocean or inland waters of the United States. This document is intended to support guidance presented in the Inland Testing Manual (USEPA/USACE 1998) and the Ocean Testing Manual (USEPA/USACE 1991). Valuable guidance regarding detection limit terminology can be found in Appendix I of EM-200-1-3 (USACE 2001) and Appendix D of the Department of Defense's (DoD's) Quality Systems Manual (USDoD 2002).

BACKGROUND: One of the most important aspects of evaluating dredged sediment prior to disposal is determining the presence or absence of environmental contaminants. With modern analytical chemistry techniques, contaminants of concern (COCs), when present above minimum levels, may be confirmed with a high degree of certainty. For a given analytical method, when COC concentrations fall below these minimum levels, it is impossible to know for certain whether such compounds are present in the environmental matrix being tested. Therefore, analytical chemistry data for non-detects should always be viewed relative to a predefined numerical level (i.e., concentration value) generically termed the "detection limit" (DL). Unfortunately, a wide array of terms have been coined that relate to analytical detection limits, and it is critical that data users understand the meaning and proper use of these terms when interpreting analytical chemistry data.

BASIC TERMINOLOGY AND CONCEPTS: In order to successfully interpret analytical chemistry data, it is critical to understand the meaning of common terms used to describe analytical detection limits. With respect to dredged material evaluation, Project Action Level (PAL), Target Detection Limit (TDL), Method Detection Limit (MDL), Method Quantitation Limit (MQL), and Laboratory Reporting Limit (LRL) are particularly important terms. It is also important to understand the relationship of these terms to absolute instrument sensitivity (AIS). These terms are discussed below.

Project Action Level. Decisions regarding disposal of dredged material with respect to each COC should be based on some predetermined concentration level or project action level (PAL) for the COC. Background contaminant levels at the disposal site may serve as a useful point of reference for establishing PALs. Sediment Quality Guidelines (SQGs) that are derived numerical values representing concentrations of COCs thought to adversely affect benthic organisms could also be used in developing PALs. Sediment Quality Guidelines have been developed by EPA through its Contaminated Sediment Management Strategy (Southerland et al. 1992). Other entities have also developed SQGs as reference values. COCs in elutriate samples may be compared to Water Quality Standards issued by states under Section 404 of the Clean Water Act

(USEPA/USACE 1991, 1998). Regardless of the benchmark used, it is critical that contract labs report COC concentrations to detection levels well below PALs.

Target Detection Limit. TDL is a term used in the Inland Testing Manual (ITM, USEPA/USACE 1998). The TDL is a performance goal set greater than the lowest, technically feasible detection limit for routine analytical methods and less than the available regulatory criteria or guidelines (i.e., the PAL) for evaluating dredged material. TDLs are values defined as part of the project planning process, and should be selected such that detection limits reported by the analytical lab are low enough to ensure that the presence of COCs can be ruled in or ruled out at or below the predetermined PALs. Typically TDLs should be no lower than one-tenth prevailing regulatory guidelines (USEPA/USACE 1998).

Method Detection Limit. The MDL is a statistically derived expression of theoretical method detection capability. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. Detailed procedures for determining MDLs are described in 40 CFR Part 136 Appendix B (Federal Register 1995), Appendix I of EM-200-1-3 (USACE 2001), and in Appendix D of the DoD's Quality Systems Manual (2002). In general, MDL values are determined by performing the complete analytical procedure (extraction/digestion, cleanup, and instrumental analysis) on replicate spiked samples (7 or more) in an otherwise clean, interference-free matrix representative of the environmental matrix to be tested. For sediment MDLs, clean sand or clay is typically used as the interference-free matrix. MDLs are calculated by multiplying the standard deviation of these replicate (n) measures by the Student's t-value at the 99-percent confidence level (at $n - 1$ degrees of freedom). If decisions with respect to disposal of dredged material are to be based on MDLs, it is highly recommended that the data user require the laboratory to verify its calculated MDL values by extracting and analyzing a duplicate sample (spiked at about twice the calculated MDL for each method target). For a given target compound, the MDL check is acceptable if it produces a detectable signal at least three times that of the background noise (USDoD 2002). Since MDL check samples do not test for real world sample matrix effects, an acceptable MDL check does not guarantee similar detection capability for field samples. MDL studies are performed annually or when significant changes in method standard operating procedures (SOPs) occur. Additionally, a new MDL study is typically performed when an analytical instrument is replaced for a given method. MDLs are based on a discrete set of measurements, and in the data reporting process, MDLs are not typically adjusted for sample-specific parameters such as sample weight, percent solids, or dilution. MDLs are estimates of detection capability and are valuable as references. Careful consideration should be given to the inherent uncertainties (as discussed above) associated with MDLs before using these values in making decisions regarding the disposal of dredged material.

Method Quantitation Limit. The Method Quantitation Limit (MQL) is a term used in current Corps guidance (USACE 2001). The MQL is set at a factor five to ten times the MDL for most target analytes in a method, but no lower than three times the MDL for any single target analyte. The MQL represents the value at which the laboratory has demonstrated the ability to reliably measure targets within prescribed performance criteria, and it establishes the lowest concentration at which data may be reported without qualification. In the absence of project-specific requirements to the contrary, the MQL is set at the level of the lowest calibration standard for the method, and the lowest calibration standard for each target must be at least three times the MDL

or greater. All target analytes detected below the MQL would be flagged as estimates (i.e., J-flagged). The MQL is a fixed reference value based on some multiple of the MDL, and it is not adjusted for sample-specific parameters.

Laboratory Reporting Limit. LRLs are the minimum levels at which a lab will report analytical chemistry data with confidence in the quantitative accuracy of that data. LRLs are threshold values below which the laboratory reports a given result as non-detected (i.e., U-flagged) and are presented as the "less than concentration value" (i.e., < ###, where ### equals the value of the LRL). LRLs are laboratory-determined values that may be based on project-specific reporting limits, regulatory action levels, or multiples of the MDL, but they should be no lower than the lowest calibration level. Typically, LRLs are no lower than the low calibration standard for the method. In general, LRLs should be 3-5 times the MDL for a given target analyte. LRLs are typically quoted by laboratories based on a default set of method conditions. For example, with sediment or soil for pesticides analysis by gas chromatography using a soxhlet extraction procedure, LRLs might be expressed based on a 30-gram dry, undiluted sample. However, for field samples, LRLs are adjusted for sample-specific parameters such as sample weight, percent solids, or dilution. These can have a significant influence on final reported values for the LRL, especially if dilution of the sample is required due to high levels of target analytes or interferences.

Absolute Instrument Sensitivity. Each and every compound-specific method that employs the use of modern analytical instrumentation has some form of detector that produces a signal in response to the presence of target compounds. The instrument itself, as well as some sample matrix components, also produces measurable detector output called noise. In general, absolute instrument sensitivity (AIS) is based only on the system noise and represents a level at which a reference signal should be measurable. In the context of analytical detection limits, the lowest concentration of a target analyte that produces a signal that can be reliably distinguished from the background noise is the true detection limit of the instrumental system (Willard et al. 1988). This can be expressed quantitatively as the minimum analyte concentration that produces a signal 2-3 times the standard deviation of the blank signal (noise). The signal-to-noise ratio (S/N) is often used to gauge instrument sensitivity, and as a rule of thumb, S/N values in the range of 3-5 would be considered sufficient to distinguish signal from noise. Being strictly a function of instrument noise, AIS does not account for non-instrument related factors (such as extraction/cleanup techniques or sample amount) that also impact overall method detection capability. Thus, absolute instrument sensitivity is a critical factor influencing the ability of a method to detect target analytes, but since it is not the only factor, AIS should not be used as the sole determinant when establishing analytical detection or reporting limits. Additionally, the AIS may vary from day to day and may be altered by the input of dirty samples into the instrument. For this reason, most routine methods employed by commercial labs do not attempt to measure AIS on a daily basis. Instead, they estimate sensitivity through periodic performance of Method Detection Limit (MDL) studies described above.

The relationship between PALs, TDLs, MDLs, MQLs, LRLs, and AIS is critically important. This relationship is summarized schematically in Figure 1. TDLs are project-specific data quality objectives that should be defined before the contract lab is selected, and the ability of the contract lab to meet project TDLs should be considered as part of the selection process. As a general rule, MDLs should *not* be used as the final determinant for whether or not TDLs have been met.

MDL values should be used for reference purposes only. MDL levels may be useful in gauging the presence or absence of a given COC, but quantitative accuracy of values reported near the MDL is uncertain. MQLs can be viewed as reference values against which field sample LRLs are compared. The LRL for a given target may be greater than or equal to the MQL, but as a general rule, it should not be lower than the MQL. Because LRLs represent quantitatively reliable concentration levels, they are the most appropriate values to determine if TDLs have been met by the laboratory.
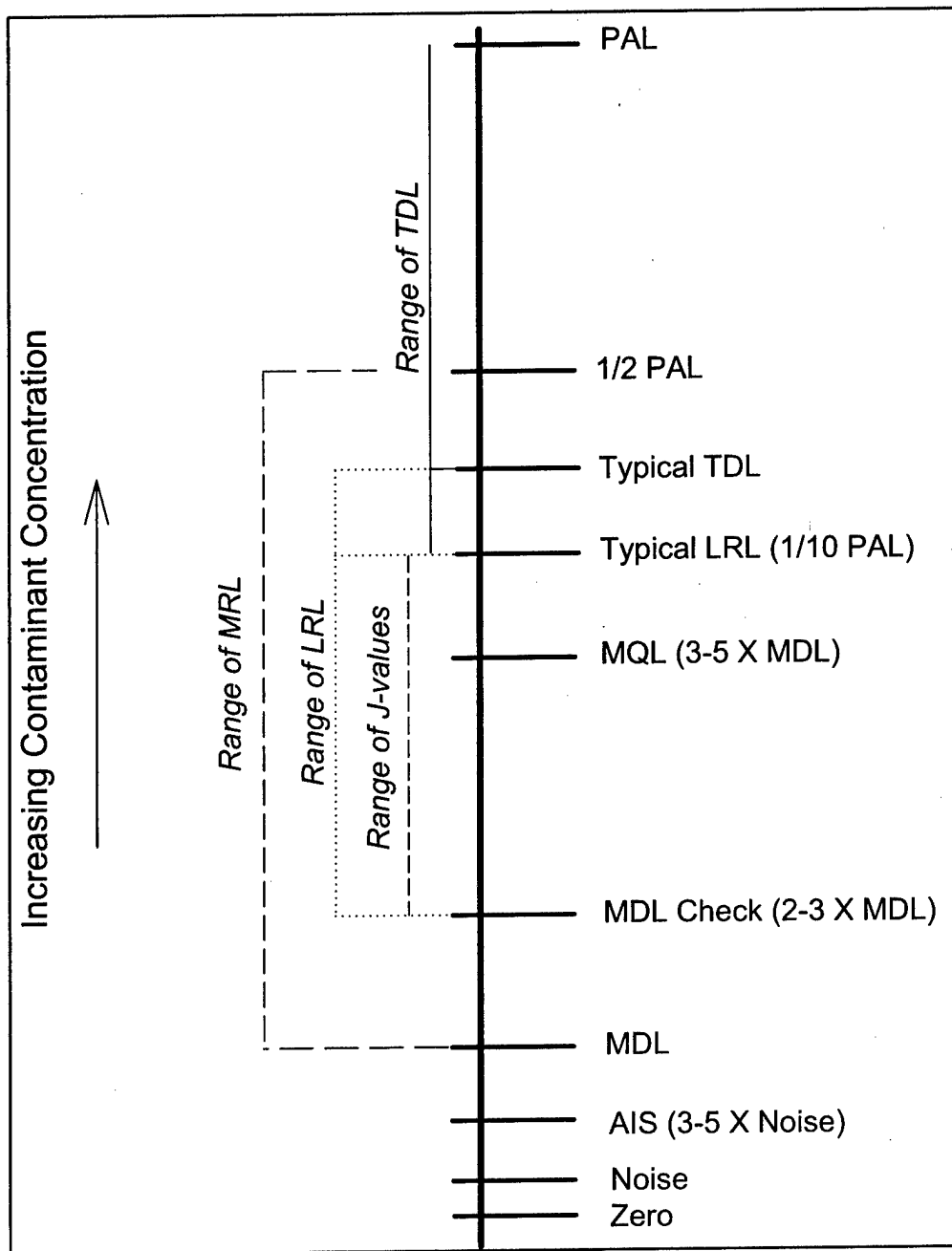

Figure 1. Schematic summary of detection limit terminology

OTHER COMMON DETECTION LIMIT TERMS: In addition to the terminology discussed above, a number of other related terms may be encountered by the data user. Some of these are similar in meaning to terms discussed above, but differences are apparent. It is critical to understand the similarity and differences to ensure proper interpretation of analytical data. Many of the definitions below are taken verbatim from cited guidance.

Method Reporting Limit. The Method Reporting Limit (MRL) is also used in current USACE (2001) guidance. It is defined as the threshold value below which the laboratory reports a given result as non-detected (i.e., U-flagged) and is presented as the "less than concentration value" (i.e., < ###, where ### equals the value of the MRL). MRLs are set based on project-specific factors that incorporate the needs of the data user, the sensitivity of the method/instrumentation, and the uncertainties associated with low-level data the user is willing to accept. As with LRLs, MRLs are adjusted based on sample matrix, weight/volume, percent solids, and dilutions. Depending on the planned use of the data, the MRL may be set as low as the sample adjusted level equivalent to the MDL or as high as one-half the PAL. In general, USACE (2001) recommends that MRLs be established at approximately one-half the PAL. While multiple factors are considered in establishing MRLs, to say they are set arbitrarily would be a vast oversimplification. Further details on the appropriate use and designation of MRL values are described in current guidance (USACE 2001). MRL and LRL are very similar terms, but unlike LRLs, MRLs can, for certain data uses, be equivalent to MDLs for individual compounds. The key difference is that MRLs are set with project-specific considerations in mind, but in contrast LRLs are defined by the contract lab and are generally based on method performance.

Instrument Detection Limit. The instrument detection limit (IDL) is considered the minimum detection concentration for the instrument only, and unlike the MDL, it ignores sample preparation effects (USACE 2001). EPA SW-846 Methods 6010B, 6020, and 7000 Series methods for metals analysis make reference to IDLs. For Method 6020, the IDL is estimated by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Because IDLs do not assess the impact of sample preparation procedures or sample matrix effects, they should not be achievable in environmental samples and should not be used as benchmarks when evaluating samples against project action levels.

Estimated Quantitation Limit. The estimated quantitation limit (EQL) is a term used in the EPA SW-846 compendium. It is defined as the lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5 to 10 times the MDL, but it may be nominally chosen within these guidelines to simplify data reporting. In SW-846, for many target analytes, the EQL is selected as the lowest non-zero calibration standard. EQLs in SW-846 are provided as guidance values and may not always be achievable.

Practical Quantitation Limit. The practical quantitation limit (PQL) is also used in the SW-846 compendium and is defined as the lowest level that can be reliably measured by routine laboratory operating conditions within specified limits of precision and accuracy (USACE 2001). PQLs are guidance values essentially synonymous with EQLs.

Sample Quantitation Limit. The sample quantitation (SQL) is a term established within the USEPA Risk Assessment Guidance for Superfund (RAGS) and is the limit of interest when reporting data for use in a risk assessment (USACE 2001). The SQL is defined as the MDL adjusted for sample-specific parameters such as dilution or sample aliquot sizes.

The terminology discussed above is summarized in Table 1. It should be noted that detection limit terms not presented in this article may be encountered by the data user. For this reason it is critical that the contract lab clearly define all detection limit terminology used in data reporting and that the data user understand the appropriate use of reported detection limits.

Table 1
Detection Limit Terms

Term	Abbreviation	Definition
Project Action Level	PAL	Concentration level of COCs that ultimately direct final decisions regarding disposal of dredged material.
Target Detection Limit	TDL	Performance goal set between the lowest, technically feasible analytical detection limit and available regulatory guidelines for evaluating dredged material.
Laboratory Reporting Limit	LRL	Lowest level lab reports with confidence in accuracy of quantitation and without qualification (i.e., J-flagged). May be set at low calibration standard for the method; Should be 3-5 times the MDL or greater. Adjusted for sample-specific parameters when reported.
Method Detection Limit	MDL	Statistically derived expression of theoretical detection capability; Based on standard deviation of replicate spiked samples taken through all steps of analytical procedure; Based on standard procedure from 40 CFR Part 136 Appendix B.
Absolute Instrument Sensitivity	AIS	Lowest level that reliably produces signal distinguishable from background noise.
Method Quantitation Limit	MQL	Level lab has shown ability to reliably measure targets within prescribed performance criteria; Lowest concentration reported without qualification (i.e., J-flagged); May be set at low calibration standard for the method; Typically 5 - 10 times the MDL, but must be at least three times the MDL.
Method Reporting Limit	MRL	Project-specific, sample adjusted threshold values below which lab reports data as non-detected and less than the threshold value (< ###). May or may not imply a high level of analytical certainty (e.g., when MRL = MDL).
Instrumental Detection Limit	IDL	Minimum detection concentration for instrument only.
Estimated Quantitation Limit	EQL	Lowest level that can be reliably measured under routine conditions.
Practical Quantitation Limit	PQL	Lowest level that can be reliably measured under routine conditions.
Sample Quantitation Limit	SQL	Used in risk assessment; MDL adjusted for sample-specific parameters.

FACTORS THAT INFLUENCE DETECTION LIMITS: A number of factors impact the final detection limits reported for environmental samples. Some of these are summarized below.

Sample Amount. Concentrations of target analytes in environmental samples are typically reported in units of mass of the target (mg) per mass (kg) or volume (L) of sample. For a given method, as the amount (weight or volume) of sample is reduced, the LRL is proportionally elevated. Sediment and soil methods typically report data on a dry weight basis, and all methods have physical limitations on the sample amount that can be handled by the sample prep apparatus. With sediment samples, the percent moisture can significantly impact the actual amount of dry sample analyzed. For example, if a typical method requires 30 g (dry weight) of sediment to

yield an LRL of 1.0 mg/kg, but 30 g of wet sediment with a moisture content of 33 percent is analyzed, the resulting LRL for the sample would be 1.5 mg/kg. For this particular sample, 45 g wet sediment would have been required to achieve the 1.0 mg/kg LRL, but physical constraints of the sample extraction equipment might make this impractical. Also, increasing the sample size to 45 g in this case could lead to increased interferences that might not be adequately removed through method cleanup procedures.

Sample Matrix. For dredged material evaluations, chemical analysis may be required on sediment, site water, and tissue samples. Each of these matrices will yield different detection limits even though the exact same instrumental technique may be used. For instance, a 30-g wet sediment sample as noted above might yield a 1.5 mg/kg (1.5 ppm) sample-specific LRL for the analysis of pesticides by gas chromatography (GC). The sample preparation process produces a 10-mL extract for analysis by GC. If a 1-L water sample were prepared for the same analysis with a resulting 10-mL extract, a sample-specific LRL of 0.03 mg/L (0.03 ppm) might be reported.

Dilution. Analytical methods typically work best over a range of concentrations for a given target compound. Just as there is a lower limit of detection, there is also an upper limit above which accurate analytical quantitation is unreliable. This upper limit is dictated primarily by the dynamic range of the instrument detector, and very high levels of any material that produces a signal could overload the detector. This results in a signal maximum that fails to increase with increasing target compound concentration. This situation can occur when high levels of target compounds or unwanted interferences are present in the sample. One remedy is simply to dilute the sample extract to a point that the instrument signal is within its dynamic range. With extract dilution, however, comes a proportional increase in LRL. If dilution of 1:100 was required of the sediment sample that otherwise would have produced an LRL of 1.5 mg/kg in the example above, then the LRL would increase to 150 mg/kg.

Extract Volume. After the initial sample extraction or digestion and cleanup steps have been completed, the resulting solution (solvent extract, acid digestate, or other solution for analysis) must typically be adjusted to a final volume prior to instrumental analysis. The concentration of this solution is then determined and used to calculate the final concentration of target analytes in the sample. The final volume of the sample extract can therefore impact analytical detection limits. In the example above referencing a 10-mL solvent extract, it is possible to reduce the extract volume through controlled evaporation techniques. In this manner the extract volume could be reduced to 1.0 mL, lowering the LRL for the sediment sample from 1.5 mg/kg to 0.15 mg/kg.

Analytical Technique. Different analytical techniques may show significant differences in detection capability. A prime example of this is seen with gas chromatography/mass spectrometry (GC/MS) as compared to gas chromatography with electron capture detection (GC/ECD). Both techniques may be used to detect the same compounds in some cases, but for ECD-active compounds, GC/ECD can give LRLs one or more orders of magnitude lower than GC/MS.

HANDLING NON-DETECTS: So what happens when non-detect data are reported as "less than concentration value"? Guidance for handling non-detected targets is described in the Inland Testing Manual (USEPA/USACE 1998), and detailed supporting information is given elsewhere (Clarke 1995, 1998). Briefly, several options are available including the following simple

approaches: (1) substitute the reported detection limit (DL) for all non-detects, (2) substitute one-half the reported detection limit (DL) for all non-detects, (3) substitute a value of zero for all non-detects. Other, more complex methods using techniques such as regression and maximum likelihood have been recommended in the statistical literature. In general, the simple substitution methods listed above work better than more complex techniques when sample sizes are very small, as is typically the case in dredged sediment evaluations (Clarke 1998). With small sample sizes, substitution of the DL when up to 40 percent of the data are censored (reported as less than a detection limit), or substitution of one-half the DL when greater than 40 percent are censored, are methods that work reasonably well (USEPA/USACE 1998). Alternatively, censored data sets may be statistically analyzed using non-parametric procedures such as Dunn's test (Hochberg and Tamhane 1987) for multiple comparisons. In general, when censoring exceeds 60 to 80 percent, any statistical analysis is likely to result in unacceptably high error rates.

While the above guidance is reasonably straightforward, the term DL has not been well-defined. Does DL in this guidance refer to MDL, SQL, LRL, MRL, or MQL? The answer to this question is simple and complex, and it really depends on the information available for a given data set as well as the level of uncertainty the data user is willing to accept. For instance, if an MDL value is reported, has the MDL been verified through the analysis of the appropriate MDL check samples, and was the verification performed recently? As noted above, the MDL check sample should be spiked at 2-3 times the stated MDL for each target. If MDL values have been satisfactorily verified (USACE 2001), then substituting values approaching the MDL may be acceptable when the data are being used to support a risk assessment, but such values should be adjusted for sample-specific parameters (including dilutions) that in effect transform MDL values into SQLs. For non-risk-related data uses, substitution values should go no lower than the sample-adjusted concentrations equivalent to the corresponding level of the MDL check sample for each respective target analyte. Half-value substitutions (i.e., one-half DL) for non-detects should not be employed when DL refers to either the SQL or the MDL check sample concentrations due to the inherent uncertainties associated with these values.

If MDL values are not available, or reported MDL values have not been appropriately verified, then substitution values for non-detects should be limited to detection threshold concentrations that are associated with a high degree of analytical certainty (i.e., LRL, MRL, or MQL). Since MQL is not a sample-adjusted quantity, it should not be used for field samples. By definition, MRL could be defined as equal to the MDL, so the use of MRL for substitution would in this case be related to the level at which it is defined. Thus the LRL would be the most appropriate substitution value to select, and according to the guidance described above, the substitution of one-half LRL would be appropriate for non-detects in dredged sediment evaluations. Guidance in the Inland Testing Manual (USEPA/USACE 1998) for substituting non-detects should be read carefully and should be fully understood before employing any of these techniques.

CONCLUSION: Analytical detection limits are an integral part of environmental chemistry data collected through dredged sediment evaluations. It is critical that data users understand the meaning and appropriate use of detection limits reported by the contract lab. Since many different terms are commonly used by commercial laboratories, the data user should insist that contract labs clearly define the exact meaning of reported detection limits. The data user should have this information prior to submission of samples to the laboratory, so that the ability of the lab to meet project-specific TDLs and PALs can be assessed. The data user must be aware that many

factors can influence final LRLs issued by the contract lab, and should set TDLs to allow for unexpected sample-specific elevation of LRLs. Finally, the assignment of numerical values to non-detect data should be approached cautiously, and careful consideration should be given to the uncertainties associated with this activity. Depending on the project, substitution of the MDL, the LRL, or one half the LRL may be acceptable. Ultimately, the project manager must make an informed decision regarding the appropriate use of analytical chemistry data with respect to the disposition of dredged material. A clear understanding of laboratory detection capability, and the language used to convey it, will greatly facilitate the decision-making process, and this in turn will increase the likelihood that resulting decisions will be firmly grounded on solid analytical data.

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