Introduction to Reporting Limits

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The Marine Pollution Studies Laboratory (MPSL) is a collaborative research consortium of scientists at Moss Landing Marine Laboratories (MLML).

**Services**
- Quality Assurance
- Data Management
- Field Sampling

**Scope**
- Monitoring
- Assessment
- Compliance
- Research
- Spill Response
- Emerging Contaminants
- Wastewater Treatment
- Litigation Support
Quality Assurance Services

Since 1998, partnering with the academic, government, and private sectors to build tools and processes that enhance the **transparency**, **accountability**, and **scientific defensibility** of environmental data collection, analysis, and reporting.

- California Department of Fish and Wildlife
- CALFED Science Program
- California State Water Resources Control Board
- National Oceanic and Atmospheric Administration
- United States Geological Survey
- United States Environmental Protection Agency
- Pesticide Industry
- Pharmaceutical Industry
- Mining Industry
- Timber Industry

**Build Large-Scale QA Programs**

**Provide Project QA Services**

Transparent • Accountable • Scientifically Defensible
Agenda

- Introduction
- Definitions
- Examples
- Intro to Determining Program/Project Reporting Limits
- Reporting Limits in Databases and Reports
- Working with a Laboratory and Reporting Limits
- Documents for Communicating Reporting Limits
- Conclusion
Desired Outcome

• A general understanding of common detection and quantitation terms

• An understanding that there are differences between detection and quantitation limits

• An introduction to determining RLs

• An appreciation for linking RLs to data use (e.g., decision making)
Text and References

• There are several slides with a significant amount of text and definitions.

• There are also several slides that show tools and list web site addresses.

• We will not be going over these verbatim; they are included so that you may use the slides later as a reference.
Introduction
Why Reporting Limits Matter
Data Use - Examples

Ability to make recommendations and/or decisions related to…

- Improved Water Supply
- Critical Species and Habitat
- Long-term Water Resources

Why Reporting Limits Matter
Desired Outcome

• A general understanding of common detection and quantitation terms

• An understanding that there are differences between detection and quantitation limits

• An introduction to determining RLs

• An appreciation for linking RLs to data use (e.g., decision making)
Concentration

1. **Action Limit**
   (or water quality standard)

2. **Reporting Limit**
   - Is Reporting Limit in the correct spot?
   - Practical Quantitation Limit

3. **Minimum Level**

4. **Method Detection Limit**

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Transparent • Accountable • Scientifically Defensible

Slide 11
EPA 1631
0.0002 ug/L

EPA 1631
0.0005 ug/L

CTR Water
0.05 ug/L

RL = PQL
0.0015 ug/L

0

Non-Detect
Detected but not Quantified
Quantified
Quantified with Statistical Rigor

Method Detection Limit
Minimum Level
Reporting Limit = PQL
Action Limit

Concentration
Definitions
Concentration

- **Method Detection Limit**
- **Minimum Level**
- **Reporting Limit**
- **Action Limit** (or water quality standard)

**Practical Quantitation Limit**
Method Detection Limit

Sample Prep + Analyses + Lab = MDL
Method Detection Limit

- 40 CFR Appendix B to Part 136 - Definition and Procedure for the Determination of the Method Detection Limit - Revision 2
  - Google: e-CFR title 40 part 136, go to App. B

- Definition: The method detection limit (MDL) is defined as the minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results. (EPA 821-R-16-006 December 2016)

- The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete, specific, and well-defined analytical method. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit.
Definition and Procedure for the Determination of the Method Detection Limit, Revision 2

Method Detection Limit

- Final rule update signed on December 15, 2016
- The new process takes background contamination into consideration in the determination of detection limits. This will reduce false positives due to blank bias.
- MDLs will be representative of lab performance over time, compared to capturing MDL data on a single day.
- Allows the lab to combine data from more than one instrument to calculate a lab-wide MDL, rather than individual instrument-specific MDLs.
Method Detection Limit Process Summary

- A lab determines its MDLs based on a minimum of **seven spiked samples** and **seven method blank samples** that go through all steps of the method.
- The spiking concentrations used to determine an MDL are between 1 and 10 times the estimated MDL and should be re-evaluated annually.
- The samples used for the MDL must be prepared in **at least three separate batches and analyzed on three separate days**. Existing data may be used for MDL calculation as long as it is collected on different days.
- Calculate the spiked sample MDL (MDL$_s$) by using standard deviation of the results and the appropriate student’s t-value and the blank sample MDL (MDL$_b$) by using the mean results and the appropriate Student t-value.
- Select the greater value between MDL$_s$ and MDL$_b$ as the initial MDL.
- During any quarter in which samples are being analyzed, prepare and analyze a minimum of two spiked samples on each instrument, in separate batches. Routine method blanks can be used to calculate MDL$_b$
- At least once every thirteen months, re-calculate MDL$_s$ and MDL$_b$
Method Detection Limit

Sample Prep + Analyses + Lab = MDL

The higher value of seven spike replicates or seven blank replicates

MDL = lowest level signal produced

A signal is detected
Concentration

Minimum Level

Method Detection Limit
Minimum Level

\[ \text{MDL} \times (3.18) \]

Method + MDL + Factor = ML

ML = lowest point on calibration curve
Vocabulary Catalog

Forum on Environmental Measurement (FEM) Glossary

Long Name: Forum on Environmental Measurements (FEM) Glossary

Description: Terms that are commonly used in association with detection, quantitation, and calibration in environmental laboratories

Publishing Organization: Office of Research and Development/Office of Science Advisor/Forum on Environmental Measurements

Program Site: https://www.epa.gov/measurements/forum-environmental-measurements-fem

Terms & Acronyms
A minimum level at which the analytical system shall give recognizable mass spectra (background corrected) and acceptable calibration points.
Example: EPA Method 1631 – Mercury in Water

“The method detection limit for Hg has been determined to be 0.2 ng/L when no interferences are present. The minimum level of quantitation (ML) has been established as 0.5 ng/L. An MDL as low as 0.05 ng/L can be achieved for low Hg samples by using a larger sample volume, a lower BrCl level (0.2%), and extra caution in sample handling.”
Minimum Level

- US EPA Method 1631 Revision E, 2002, Page 32, Office of Water

- The lowest level at which the entire analytical system must give a recognizable signal and acceptable calibration point for the analyte. It is equivalent to the concentration of the lowest calibration standard, assuming that all method-specified sample weights, volumes, and cleanup procedures have been employed.

- The ML is calculated by multiplying the MDL by 3.18 and rounding the result to the number nearest to (1, 2, or 5) x 10^n, where n is an integer.

- Minimum levels are used in some US EPA methods.
Minimum Level

\[ \text{Method} + \text{MDL} + \text{Factor} = \text{ML} \]

ML = lowest point on calibration curve
A signal is **quantified**
Method Detection Limit

Sample Prep + Analyses + Lab = MDL

The higher value of 7 spike replicates or 7 blank replicates

MDL = lowest level signal produced
A signal is detected

Minimum Level

Method + MDL + Factor = ML

ML = lowest point on calibration curve
A signal is quantified
Concentration

Minimum Level

Method Detection Limit

Practical Quantitation Limit
Practical Quantitation Limit

Instrument + Analyst + Factor = PQL

** or **

PQL = 3 times lowest point on calibration curve
Practical Quantitation Limit

- Subjective definitions?

- A quantity set at two to ten times above the method detection limit (MDL). By raising the MDL by a factor of two to ten, serving as a “safety factor,” commercial labs hope to quantify the environmental sample concentrations with a degree of certainty.

- The degree of the factor (2-10) is decided by the analytical lab depending upon the skill and experience of the analyst, the quality of the instrument, and the nature of the sample objectives.
Practical Quantitation Limit

The statewide PQL Robust and statewide PQL Minimum are derived by multiplying the detection limit by a factor of 10. This is consistent with the site-specific procedure. That value is then rounded based on the number of significant figures. Where there is one significant figure, the PQL is rounded up to the nearest 1, 2, 5, or 10 (or multiple of 10 of those values), in accordance with standard methodology. Where there are two significant figures (maximum), the second digit in the PQL is rounded up to the nearest 5 or 10. In very few cases, the work group rounded down slightly to establish the PQL (e.g., 5.1 to 5) where this would allow the PQL to be at or below the water quality standard. This was deemed appropriate given the use of the detection limit multiplier of 10.

Unnamed Western State Water Quality Control Division

The MDL is defined by the statistical window, the PQL is essentially arbitrary. There are recommendations, PQL = IDL x 10 or MDL x 6 and others... but no governmental regulation covers the PQL. It comes down to what the laboratory feels comfortable signing their name to, confidently, on a daily basis. The final arbiter of the PQL is the concentration of the lowest

US EPA Region III Fact Sheet 2006

Common Practice – 3 times the lowest level standard
Practical Quantitation Limit

Instrument + Analyst + Factor = PQL

A signal is quantified

** or **

PQL = 3 times lowest point on calibration curve

A signal is quantified with statistical rigor
Method Detection Limit

Sample Prep + Analyses + Lab = MDL

The higher value of seven spike replicates or seven blank replicates
MDL = lowest level signal is produced
A signal is detected

Minimum Level

EPA Std Method or SOP

MDL X (3.18)

Method + MDL + Factor = ML

ML = lowest point on calibration curve
A signal is quantified

Practical Quantitation Limit

Instrument + Analyst + Factor = PQL

A signal is quantified

** or **
PQL = 3x lowest point on calibration curve
A signal is quantified with statistical rigor
Concentration

- Method Detection Limit
- Minimum Level
- Reporting Limit

Transparent • Accountable • Scientifically Defensible
Reporting Limit

\[ \text{Program} + \text{Data Use} = RL \]

(Action Limit)
The minimum value below which data are documented as non-detects.

Question: Does this mean the RL = MDL
Question: What does “are documented” mean
Setting the Reporting Level

The USEPA MDL procedure does not address the issue of setting reporting levels. Both the USEPA MDL and the LT-MDL focus exclusively on minimizing the risk of reporting a false positive. At the MDL concentration, however, the risk of a false negative is not adequately limited. A sample with a true concentration equal to the USEPA MDL or LT-MDL has a 50-percent chance of not being detected (Keith, 1992). This is shown in figure 8, where the frequency distribution is centered on the calculated MDL. Assuming that the MDL concentration does, indeed, represent a detection “limit” (that is, the analyte cannot be detected reliably at less than this concentration), then up to 50 percent of the measurements made of a sample having a true concentration equal to the MDL would be less than the MDL (shaded region in fig. 8) and, thus, would result in a false negative. The NWQL views a 50-percent probability of a false negative as unacceptably high for use of the MDL as a reliable reporting level.

Recognizing the inadequacies of the MDL as a reporting level, laboratories often set quantification limits (operationally minimum reporting levels) at concentrations greater than the determined MDL’s and in a region that supports quantitative determination. For example, the reporting levels might be set at practical quantitation limits (PQL’s) that are 5 or 10 times the MDL (U.S. Environmental Protection Agency, 1985), or at the limit of quantitation (LOQ), which is a concentration 10 standard deviation units above the average blank response (Keith, 1992). More recently, the USEPA has suggested the use of a minimum level (ML), which is 3.18 times the MDL (for n = 7) (U.S. Environmental Protection Agency, 1993). Gibbons and others (1997a, b) recommend use of an alternative minimum level (AML) that is derived from a multiple-concentration calibration procedure that eliminates or minimizes many of the assumptions and limitations of the USEPA MDL and ML procedures.

In establishing the reporting level, the NWQL has set the acceptable rate of false negatives at no more than 1 percent. This requires the use of a different value from the LT-MDL as the reporting level. The laboratory reporting level (LRL) has been devised to meet this requirement and is comparable to the reliable detection level of Keith (1992) when the false positive and false negative rates are set at ≤1 percent. The LRL is calculated from the LT-MDL, as follows:

\[
LRL = 2 \times LT-MDL
\]

As shown in figure 9, the LRL can be lower or higher than the LT-MDL by a factor of 2. This allows for some flexibility in the choice of the reporting level. The LRL is a more realistic representation of the analytical capability of the laboratory than the LT-MDL.

Currie then defined measures of detectability, firmly based on the statistical theory of hypothesis testing.
Limits for Qualitative Detection and Quantitative Determination

A visiting professor at NIST once pointed out that our measurement professionals are given a difficult task by some of our customers. In a (macroscopically) continuum universe, we are asked to perform measurements with tools and techniques of finite precision and in the end to produce digital answers, preferably binary: yes or no, safe or unsafe, above or below the regulatory limit. A common triple question arises in the measurement of environmental radioactivity, atmospheric ozone, gold in rock, or the efficacy of a flu treatment: Is the signal there? What is the chance that we will detect it? How big is it?

Until Lloyd Currie's paper Limits for Qualitative Detection and Quantitative Determination: Application to Radiochemistry [1] was published, there was enough inconsistency in the definition of "detection limit" to conceal a great deal of disagreement. In just over seven pages, this tightly written communication established a high level of uniformity in answering these questions. The paper contains fundamental information that has made it influential far beyond its size, and it is rich enough to be discussed actively in e-mail newsgroups over 30 years later. This is surely one of the most often cited publications in analytical chemistry. The Science Citation Index lists 1280 published references to this paper—so far.

Currie asks and answers a disarming simple question: What do we mean by the detection limit of a measurement process? He found that the literature "revealed a plethora of mathematical expressions and widely-ranging terminology." The same terms have

Currie then defined measures of detectability, firmly based on the statistical theory of hypothesis testing. He began by defining the concepts of qualitative and quantitative analysis limits. Three limiting levels were defined:

- The critical level $L_c$, the signal level above which an observed instrument response may be reliably recognized as "detected."
- The detection limit $L_D$, the true net signal level that may be expected a priori to lead to detection.
- The determination limit $L_Q$, the signal level above which a quantitative measurement can be performed with a stated relative uncertainty.

Numerical values of these levels depend on four criteria, most importantly the standard deviation $\sigma_0$ of the blank, or background. By choosing a probability $\alpha$ (error of the first kind) for falsely deciding that the

Detection and quantification limits: origins and historical overview

Lloyd A. Currie

National Institute of Standards and Technology, Gaithersburg, MD 20899, USA

Received 17 February 1998; accepted 18 February 1998

Abstract

Detection and quantification can be traced back to the ancient world, yet there have been decades of coordinated documents prepared by international bodies. The International Union of Pure and Applied Chemistry (IUPAC) [L. A. Currie, IUPAC Methods including Detection and Quantification, Analytica Chimica Acta 391 (1999) 127–134] also addressed the issue of reporting. The recommendation is to always report both the estimated value of the measured quantity (\( \hat{L} \)) and its uncertainty, even when \( \hat{L} < L_C \) results in the decision “not detected”. Otherwise, there is needless information loss, and of course, the impossibility of averaging a series of results. The practice of quoting

The defining relations, with default parameter values in parentheses, are given as follows:

Detection decision (critical value) (\( L_C, \alpha=0.05 \)):

\[
\Pr(\hat{L} > L_C | L = 0) \leq \alpha.
\]

Detection limit (minimum detectable value) (\( L_D, \beta=0.05 \)):

\[
\Pr(\hat{L} \leq L_C | L = L_D) = \beta.
\]

Quantification limit (minimum quantifiable value) (\( L_Q, RSD_Q = 0.10 \)):

\[
L_Q = k_Q \sigma_Q, \text{ where } k_Q = 1/RSD_Q.
\]
Different Definitions - Examples

- An instrument-dependent quantity based on the lowest point on the calibration curve. ((Unnamed North Eastern State) Department of Environmental Protection)

- A limit imposed upon the reporting lab. The RL is usually demanded by the client or regulatory guidelines, and is basically associated with method detection limits (MDLs) or practical quantitation limits (PQLs). (Unnamed Western State Regional Water Quality Control Board)

- Reporting Limit (RL)—is the lowest concentration at which an analyte can be detected in a sample and its concentration can be reported with a reasonable degree of accuracy and precision. A criterion of ± 20% accuracy and 20% RSD for replicate determinations is often used to define “reasonable”. The acceptable ranges depend somewhat on the analytical methodology used. For samples that do not pose a particular matrix problem, the RL is typically about three to five times higher than the MDL. Similar to the MDL, the RL is a laboratory-specific number, which may change with time. When a sample has to be diluted before analysis, either because of matrix problems or to get the instrument response within the linear dynamic range, the RL is raised by a factor corresponding to the dilution factor. (Unnamed Federal Sanitation and Radiation Laboratory)
Term is Undefined

Reporting Limit as a term = pizza

Project or program must have a written definition
Reporting Limit

Program + Data Use = RL

(Action Limit)
Data Use - Examples

Ability to make recommendations and/or decisions related to...

- Improved Water Supply
- Critical Species and Habitat
- Long-term Water Resources

Why Reporting Limits Matter
Dependent on METHOD

Method Detection Limit
Minimum Level
Practical Quantitation Limit

Dependent on USE

Reporting Limit
Data Use in Decisions

Analytical Method

Transparent ● Accountable ● Scientifically Defensible

Slide 47
Reporting Limit

Program + Data Use = RL

(Action Limit)

A signal is quantified and is more robust if it incorporates statistical rigor
Method Detection Limit

Sample Prep + Analyses + Lab = MDL

The higher value of seven spike replicates or seven blank replicates.

MDL = lowest level signal is produced

A signal is detected

Practical Quantitation Limit

Instrument + Analyst + Factor = PQL

A signal is quantified

** or **

PQL = 3x lowest point on calibration curve

A signal is quantified with statistical rigor

Minimum Level

MDL × (3.18)

Method + MDL + Factor = ML

ML = lowest point on calibration curve

A signal is quantified

Reporting Limit

Program + Data Use = RL

A signal is quantified and is more robust if it incorporates statistical rigor

Transparent ● Accountable ● Scientifically Defensible

Slide 49
Concentration

Method Detection Limit

Minimum Level

Reporting Limit
• MDL = Method Detection Limit
  Detected

• ML = Minimum Level
  Quantified

• PQL = Practical Quantitation Limit
  Quantified

  If 3x the lowest point on calibration curve
  Quantified *with* Statistical Rigor

• RL = Reporting Limit
  Quantified

  May be defined as quantified with statistical rigor

LOD = Limit of Detection
  Detected

LOQ = Limit of Quantification
  Quantified
Concentration

- Method Detection Limit
- Minimum Level
- Reporting Limit
- Action Limit (or water quality standard)

Is Reporting Limit in the correct spot?

Practical Quantitation Limit

Transparent • Accountable • Scientifically Defensible
Reporting Limit

Program + Data Use = RL
(Action Limit)
Concentration

Method Detection Limit

Minimum Level

Reporting Limit

Action Limit (or water quality standard)

Protective of Standard

Practical Quantitation Limit
Marine Pollution Studies Laboratory at the Moss Landing Marine Laboratories

0

Method
Detection
Limit

Minimum
Level

Reporting
Limit

Action
Limit

Protection of Standard

Concentration

Transparent ● Accountable ● Scientifically Defensible
Examples
Example: *Unnamed Midwestern State Environmental Protection Agency*

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Units</th>
<th>Typical MDL</th>
<th>Practical Quantitation Limit</th>
<th>Maximum Contaminant Level</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon Tetrachloride</td>
<td>µg/L</td>
<td>0.02 (EPA 180.1)</td>
<td>0.5</td>
<td>25</td>
</tr>
<tr>
<td>Chlorobenzene</td>
<td>µg/L</td>
<td>0.03 (EPA 200.8)</td>
<td>2.0</td>
<td>14</td>
</tr>
<tr>
<td>Chloroform</td>
<td>µg/L</td>
<td>0.2 (EPA 200.8)</td>
<td>2.5</td>
<td>117</td>
</tr>
</tbody>
</table>

From Document’s Text:

“PQL variation may be due to such issues as ground water matrix interference, analytical method, laboratory, laboratory personnel or a change in analytical instruments. Such variability is not unexpected and reflects the nature of PQLs.”
Example: *Unnamed Western State Department of Ecology*

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Units</th>
<th>Typical MDL</th>
<th>Laboratory Quantitation Level</th>
<th>Benchmark</th>
</tr>
</thead>
<tbody>
<tr>
<td>Malathion</td>
<td>μg/L</td>
<td>0.02 (EPA 8141A)</td>
<td>0.5</td>
<td>25</td>
</tr>
<tr>
<td>Mirex</td>
<td>μg/L</td>
<td>0.03 (EPA 8081A)</td>
<td>2.0</td>
<td>14</td>
</tr>
<tr>
<td>Simazine</td>
<td>μg/L</td>
<td>0.2 (EPA 200.8)</td>
<td>2.5</td>
<td>117</td>
</tr>
</tbody>
</table>

“Minimum Level” and “Action Limit” are the same as “Benchmark.”

Concentration
Example: *Unnamed Southwestern State Commission on Environmental Quality*

- Surface Water Quality Standards are written by the ### under the authority of the Clean Water Act and the ### Water Code. The standards are effective for Clean Water Act purposes when approved by the EPA.

- **Limit of Quantification (LOQ)** Criteria are located in Appendix A

Example: *Unnamed North Eastern State Department of Environmental Protection*

- **Reporting Limit (RL)** is defined as the concentration of the **lowest standard** in the calibration curve for organics and the **lowest concentration standard** used in the calibration of the method and for inorganics, derived from the concentration of that analyte in the lowest level check standard (which could be the lowest calibration standard in a multi-point calibration curve).
Determining Reporting Limits
How do I determine program/project RLs?

• Program/project RLs should be based on the data use.
  o This may include, but is not limited to, water quality standards, assessment thresholds, TMDLs, regulatory contexts, and the use of results with other testing (such as toxicity testing).

• Labs are great resources for information!

• Other programs, states, projects, etc are great resources for information

• It is important to understand that you cannot set RLs below the lowest level of state-of-the-art analytical capabilities.

• Consider cost/benefit
  o Lower RL may mean fewer resources for field samples
Data Use – Examples

Ability to make recommendations and/or decisions related to…

- Improved Water Supply
- Critical Species and Habitat
- Long-term Water Resources

Why Reporting Limits Matter
How do I determine program/project RLs?

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  - Lower RL may mean fewer resources for field samples
Water Quality Standards: Establishment of Numeric Criteria for Priority Toxic Pollutants for the State of California (California Toxics Rule)

On May 18, 2000, the EPA promulgated numeric water quality criteria for priority toxic pollutants and other provisions for water quality standards to be applied to waters in the state of California. EPA promulgated this rule - also known as the California Toxics Rule (CTR) - based on the Administrator’s determination that the numeric criteria are necessary in California to protect human health and the environment.

The CTR fills a gap in California water quality standards that was created in 1994 when a state court overturned the state’s water quality control plans containing water quality criteria for priority toxic pollutants. Thus, the State of California has been without numeric water quality criteria for many priority
Example: California Toxics Rule

• On May 18, 2000, EPA promulgated numeric water quality criteria for priority toxic pollutants and other provisions for water quality standards to be applied to waters in the state of California. EPA promulgated this rule - also known as the California Toxics Rule (CTR) - based on the Administrator's determination that numeric criteria are necessary in California to protect human health and the environment. (EPA)

• CTR values can be used as guidance for setting action limits and RLs that are protective of human health and aquatic life.

Process for Determining Reporting Limits

1. Select Parameter
2. Determine Action Limit
3. Establish RL
4. Check Methods
5. Check Lab MDL & RL
<table>
<thead>
<tr>
<th># Compound</th>
<th>CAS Number</th>
<th>Criterion Maximum Conc.</th>
<th>Criterion Continuous Conc.</th>
<th>Water &amp; Organisms (µg/L) D1</th>
<th>Organisms Only (µg/L) D2</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Antimony</td>
<td>7440360</td>
<td></td>
<td></td>
<td></td>
<td>14 a,s</td>
</tr>
<tr>
<td>2. Arsenic</td>
<td>7440382</td>
<td>340 i,m,w</td>
<td>150 i,m,w</td>
<td></td>
<td>4300 a,t</td>
</tr>
<tr>
<td>3. Beryllium</td>
<td>7440417</td>
<td></td>
<td></td>
<td></td>
<td>n</td>
</tr>
<tr>
<td>4. Cadmium</td>
<td>7440439</td>
<td>4.3 e,i,m,w,x</td>
<td>2.2 e,i,m,w</td>
<td></td>
<td>n</td>
</tr>
<tr>
<td>5a. Chromium (III)</td>
<td>16065831</td>
<td>550 e,i,m,o</td>
<td>180 e,i,m,o</td>
<td></td>
<td>n</td>
</tr>
<tr>
<td>5b. Chromium (VI)</td>
<td>18540299</td>
<td>16 i,m,w</td>
<td>11 i,m,w</td>
<td></td>
<td>n</td>
</tr>
<tr>
<td>6. Copper</td>
<td>7440508</td>
<td>13 e,i,m,w,x</td>
<td>9.0 e,i,m,w</td>
<td></td>
<td>1300</td>
</tr>
<tr>
<td>7. Lead</td>
<td>7439921</td>
<td>65 e,i,m</td>
<td>2.5 e,i,m</td>
<td></td>
<td>n</td>
</tr>
<tr>
<td>8. Mercury</td>
<td>7439976</td>
<td>[Reserved]</td>
<td>[Reserved]</td>
<td>[Reserved]</td>
<td>0.050 a</td>
</tr>
<tr>
<td>9. Nickel</td>
<td>7440020</td>
<td>470 e,i,m,w</td>
<td>52 e,i,m,w</td>
<td></td>
<td>4600 a</td>
</tr>
</tbody>
</table>
Establish RL

10x < CTR = 0.005 ug/L

RL
0.005 ug/L

CTR Water
0.05 ug/L

Protection of Standard

0
Method Detection Limit

Minimum Level

Reporting Limit

Action Limit

Concentration

Transparent • Accountable • Scientifically Defensible
Marine Pollution Studies Laboratory at the Moss Landing Marine Laboratories

Select Parameter

Determine Action Limit

Establish RL

Check Methods

Check Lab MDL & RL

Mercury, total water

CTR water 0.05 ug/L

10x < CTR = 0.005 ug/L

Use National Environmental Methods Index to find methods: [www.nemi.gov](http://www.nemi.gov)
NEMI is a searchable database that allows scientists and managers to find and compare analytical and field methods for all phases of environmental monitoring.
<table>
<thead>
<tr>
<th>Method #</th>
<th>Source</th>
<th>Detection Level</th>
<th>Detection Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>200.8</td>
<td>EPA</td>
<td>0.2 ug/L</td>
<td>MDL</td>
</tr>
<tr>
<td>200.7</td>
<td>EPA</td>
<td>7 ug/L</td>
<td>MDL</td>
</tr>
<tr>
<td>245.1</td>
<td>EPA</td>
<td>0.2 ug/L</td>
<td>RNGE</td>
</tr>
<tr>
<td>I-1462</td>
<td>USGS</td>
<td>0.5 ug/L</td>
<td>RNGE</td>
</tr>
<tr>
<td>I-7462</td>
<td>USGS</td>
<td>0.5 ug/L</td>
<td>RL</td>
</tr>
<tr>
<td>D6502</td>
<td>ASTM</td>
<td>1 ug/L</td>
<td>ML</td>
</tr>
<tr>
<td>1631</td>
<td>EPA</td>
<td>0.0002 ug/L</td>
<td>MDL</td>
</tr>
<tr>
<td>245.7</td>
<td>EPA</td>
<td>0.0018 ug/L</td>
<td>MDL</td>
</tr>
</tbody>
</table>

\[ RL = 0.005 \text{ ug/L} \]
Select Parameter

Determine Action Limit

Establish RL

Check Methods

Check Lab MDL & RL

1631 Minimum Level = 0.0005 ug/L  Cost $51-200
245.7 Minimum Level = 0.005 ug/L  Cost $51-200
### Practical Quantitation Limit

3x lowest level point on calibration curve

0.0015 ug/L

- **Method Detection Limit**
  - EPA 1631
  - 0.0002 ug/L

- **Minimum Level**
  - EPA 1631
  - 0.0005 ug/L

- **Reporting Limit**
  - RL
  - 0.005 ug/L

- **Action Limit**
  - CTR Water
  - 0.05 ug/L
Method Detection Limit

EPA 1631 0.0002 µg/L

EPA 1631 0.0005 µg/L

RL 0.005 µg/L

CTR Water 0.05 µg/L

Quantified Minimum Level

Reporting Limit

Action Limit

Practical Quantitation Limit
3x lowest level point on calibration curve
0.0015 µg/L

Concentration
Select Parameter

Determine Action Limit

Establish RL

Check Methods

Check Lab MDL & RL

Back and forth process
Concentration

EPA 1631
- 0.0002 ug/L
- 0.0005 ug/L
RL
- 0.005 ug/L
CTR Water
- 0.05 ug/L

Protection of Standard

Method Detection Limit: 0

Quantified

Reporting Limit

Action Limit

Practical Quantitation Limit
3x lowest level point on calibration curve
0.0015 ug/L

Transparent ● Accountable ● Scientifically Defensible
**Method Detection Limit**
- EPA 1631: 0.0002 ug/L
- EPA 1631: 0.0005 ug/L

**Reporting Limit = PQL**
- RL: 0.0015 ug/L

**Action Limit**
- CTR Water: 0.05 ug/L

**Minimum Level**
- Concentration

**Protection of Standard**
- Transparent ● Accountable ● Scientifically Defensible
Reporting Limits in Databases and Reports
Reporting

When reporting data, other terms worth understanding include:

- **Not Detected (ND):** The sample result is less than the MDL. The analyte being tested cannot be detected by the equipment or method.

- **Detected Not Quantifiable (DNQ):** The sample result is between the MDL and the ML. These results may be reported as the measured value (not negative) with a flag that is carried all the way through data storage, handling, and reporting.
Marine Pollution Studies Laboratory at the Moss Landing Marine Laboratories

Method Detection Limit

EPA 1631
0.0002 ug/L

EPA 1631
0.0005 ug/L

RL = PQL
0.0015 ug/L

CTR Water
0.05 ug/L

Non-Detect
Detected but not Quantified
Quantified
Quantified w/ Stat. Rigor

Concentration

EPA
1631
0.0002 ug/L

EPA
1631
0.0005 ug/L

CTR Water
0.05 ug/L

0

Minimum Level

Reporting Limit = PQL

Protocol for Standard Concentration

EPA
1631
0.0005 ug/L

EPA
1631
0.0002 ug/L

Non Detect
Detected but not Quantified
Quantified
Quantified w/ Stat. Rigor

Concentration
NATIONAL FUNCTIONAL GUIDELINES
for Inorganic Superfund Data Review

Office of Superfund Remediation and Technology Innovation (OSRTI)
United States Environmental Protection Agency (EPA)
Washington, DC 20460

OSWER 9355.0-131
EPA-540-R-013-001
AUGUST 2014

Transparent ● Accountable ● Scientifically Defensible
II. Data Qualifier Definitions

The following definitions provide brief explanations of the national qualifiers assigned to results during the data review process. The reviewer should use these qualifiers as applicable. If the reviewer chooses to use additional qualifiers, a complete explanation of those qualifiers shall accompany the data review.

<table>
<thead>
<tr>
<th>Data Qualifier</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>U</td>
<td>The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.</td>
</tr>
<tr>
<td>J</td>
<td>The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.</td>
</tr>
<tr>
<td>J+</td>
<td>The result is an estimated quantity, but the result may be biased high.</td>
</tr>
<tr>
<td>J-</td>
<td>The result is an estimated quantity, but the result may be biased low.</td>
</tr>
<tr>
<td>UJ</td>
<td>The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.</td>
</tr>
<tr>
<td>R</td>
<td>The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.</td>
</tr>
</tbody>
</table>
0

Non-Detect

Detected but not Quantified

Quantified

Quantified w/ Stat. Rigor

Method Detection Limit

Minimum Level

Reporting Limit = PQL

Action Limit

EPA 1631
0.0002 ug/L

EPA 1631
0.0005 ug/L

CTR Water
0.05 ug/L

RL = PQL
0.0015 ug/L

National Functional Guidelines Flags – U, J, UJ

Other Laboratory Flags – ND, DNQ
Working with a Laboratory
Laboratory Reporting Limits

- Supply information to lab
  - Table with matrix/analyte combinations with MDL, ML, and RL
  - Written definitions for each term

- Generally, labs will establish RLs
  - Based on the lowest point in the calibration curve, or
  - As 3x the lowest point in the calibration curve, or
  - At 2-5x the MDL.

- Ask labs how
  - Ask how dilutions are handled
  - Ask how results are reported (esp. between 0-MDL, MDL-ML)
National Functional Guidelines Flags – U, J, UJ

Other Laboratory Flags – ND, DNQ
Laboratory Reporting Limits

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• Ask labs how
  o Ask how dilutions are handled
  o Ask how results are reported (esp. between 0-MDL, MDL-ML)

Tip – Put this info. in RFP!
Documents for Communicating Reporting Limits
Documents

- Request for Proposals (RFPs)
- QA Project Plans
- QA Manuals or other program documents
- Data Qualification/Flagging Manuals (or procedures)
- Permits
- Contracts

Tip – Have QA staff review

If it’s in the contract, people pay attention

- Measurement Quality Objectives
- Holding Times
- Methods
- MDLs, MLs, RLs
- How to handle NDs and DNQs
- Reporting Formats
- Timelines
- Subcontracting Work
Conclusion
Why Reporting Limits Matter
Concentration

- Method Detection Limit
- Minimum Level
- Reporting Limit
- Action Limit
  (or water quality standard)

Is Reporting Limit in the correct spot?

Practical Quantitation Limit
QUALITY ASSURANCE COMPONENTS
Measurable Metrics

Defensible
Usable
Comparable
Concentration

- Method Detection Limit
- Minimum Level
- Reporting Limit
- Action Limit (or water quality standard)

Is Reporting Limit in the correct spot?

Practical Quantitation Limit
Why Reporting Limits Matter
Why Reporting Limits Matter

Reporting limits must be protective of our water quality standards

0 Method Detection Limit
Minimum Level Reporting Limit
Action Limit

Concentration
Marine Pollution Studies Laboratory
Moss Landing Marine Laboratories

Beverly H. van Buuren, Principal Investigator
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206-297-1378

QUESTIONS?
QA Help Desk at
QAHelpDesk@mlml.calstate.edu