Introduction to Reporting Limits

National Water Quality Monitoring Council Webinar Series

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Marine Pollution Studies Laboratory
Moss Landing Marine Laboratories

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

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
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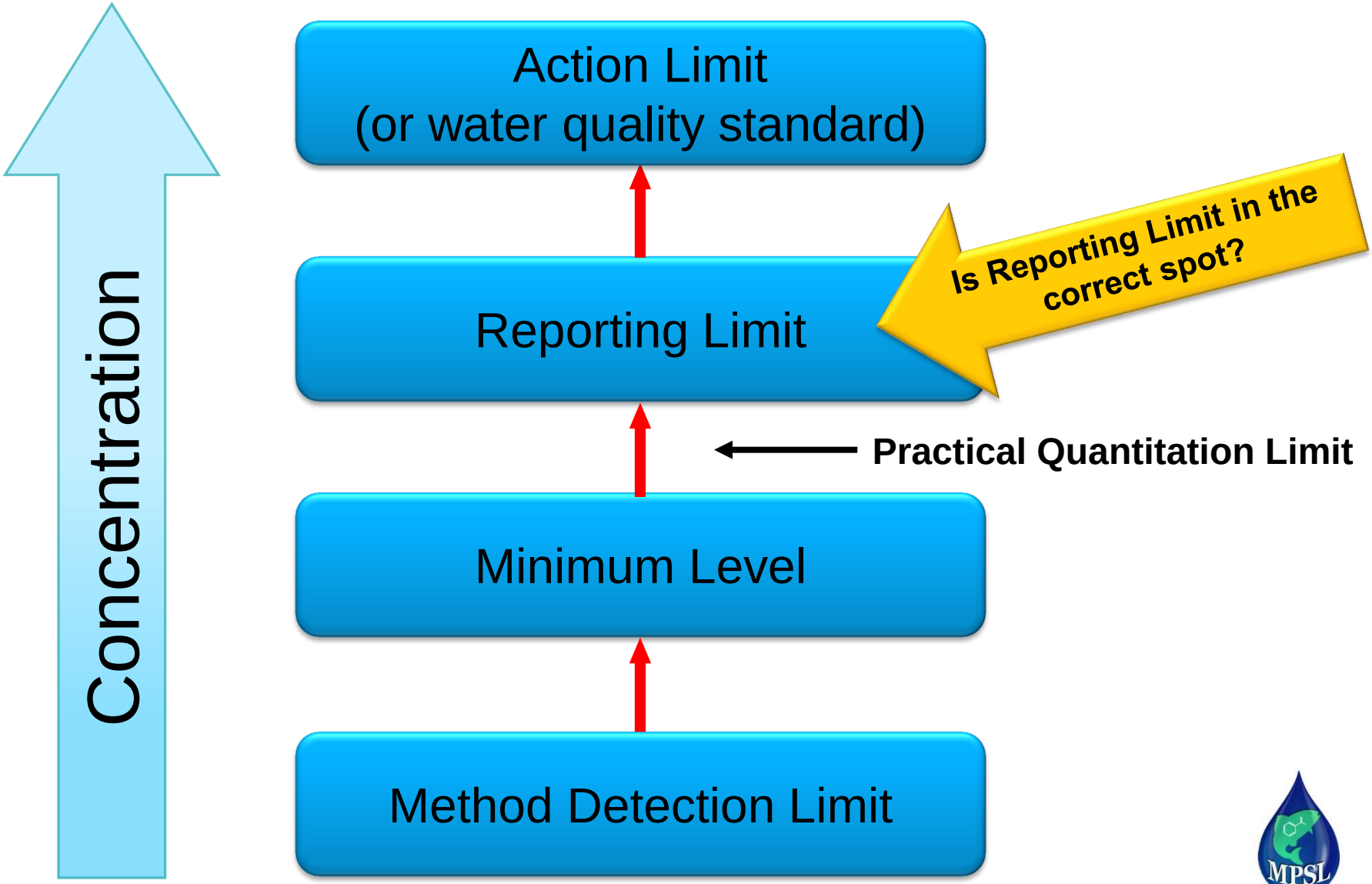
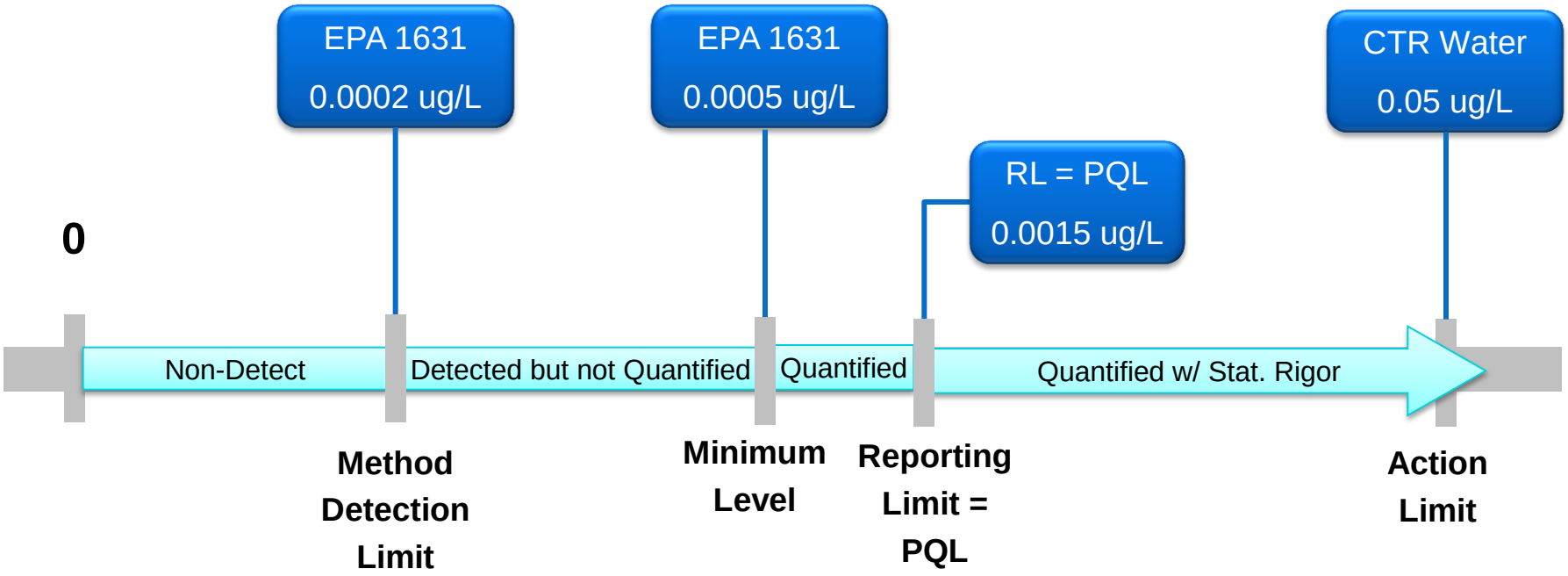
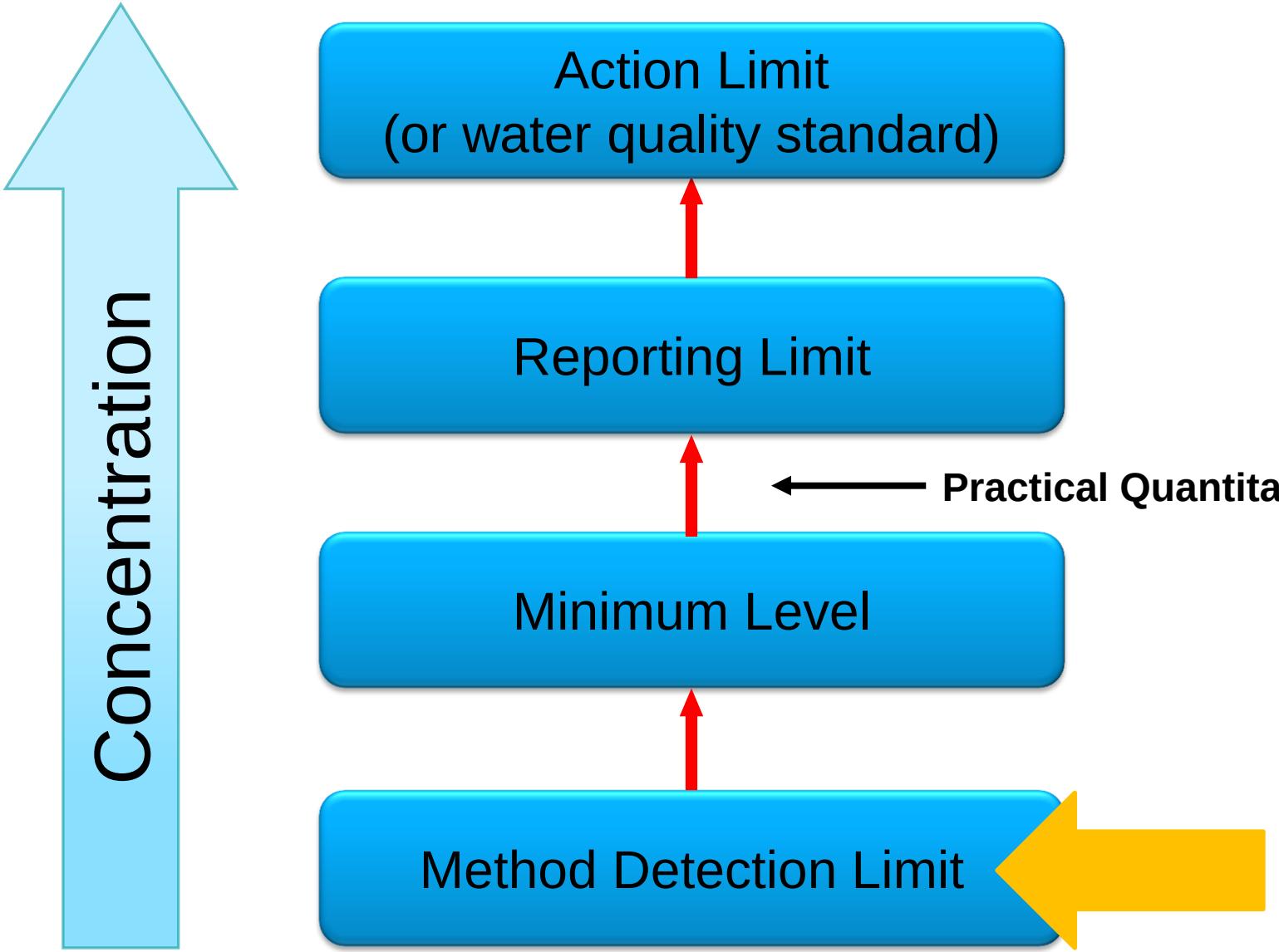
- An introduction to determining RLs
- An appreciation for linking RLs to data use (e.g., decision making)


Concentration
Action Limit
(or water quality standard)
Reporting Limit
Is Reporting Limit in the correct spot?
Practical Quantitation Limit
Minimum Level
Method Detection Limit



Concentration

Definitions


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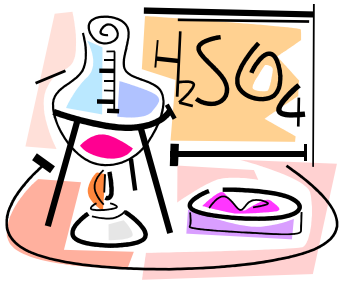
Minimum Level
Method Detection Limit

Method Detection Limit

Method



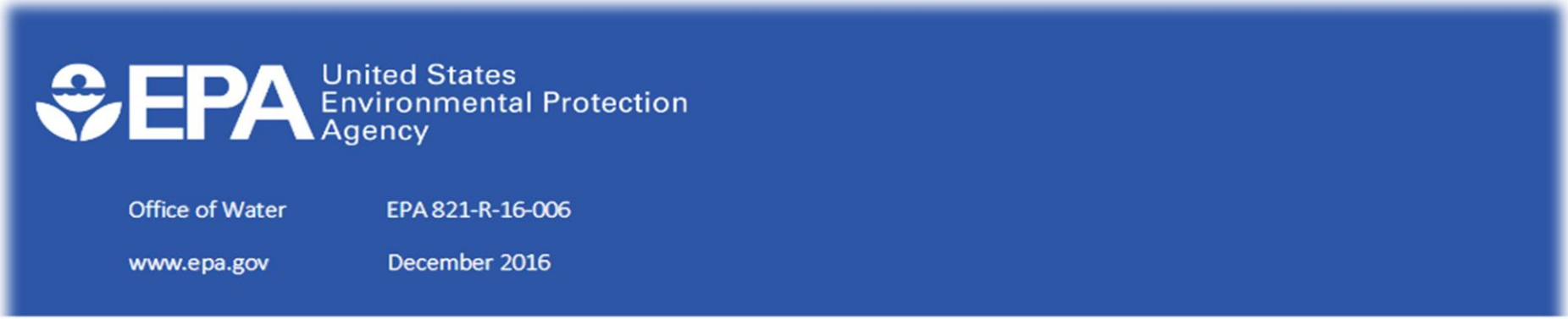
Sample Prep + Analyses + Lab = MDL

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

https://www.epa.gov/sites/production/files/2016-12/documents/mdl-procedure_rev2_12-13-2016.pdf

Method Detection Limit

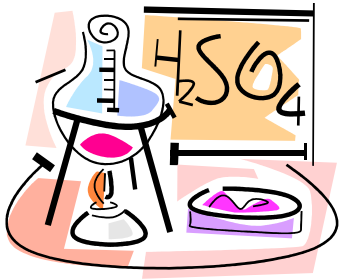
- Final rule update signed on December 15, 2016
- The new process takes background contamination into consideration in the determination of detection limits. This will reduce false positives due to blank bias.
- MDLs will be representative of lab performance over time, compared to capturing MDL data 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.
- Fact sheet available at: **www.epa.gov/cwa-methods/methods-update-rule-support-documents**
- FAQ available at: **www.epa.gov/cwa-methods/method-detection-limit-frequent-questions#questions-mur**

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

Method

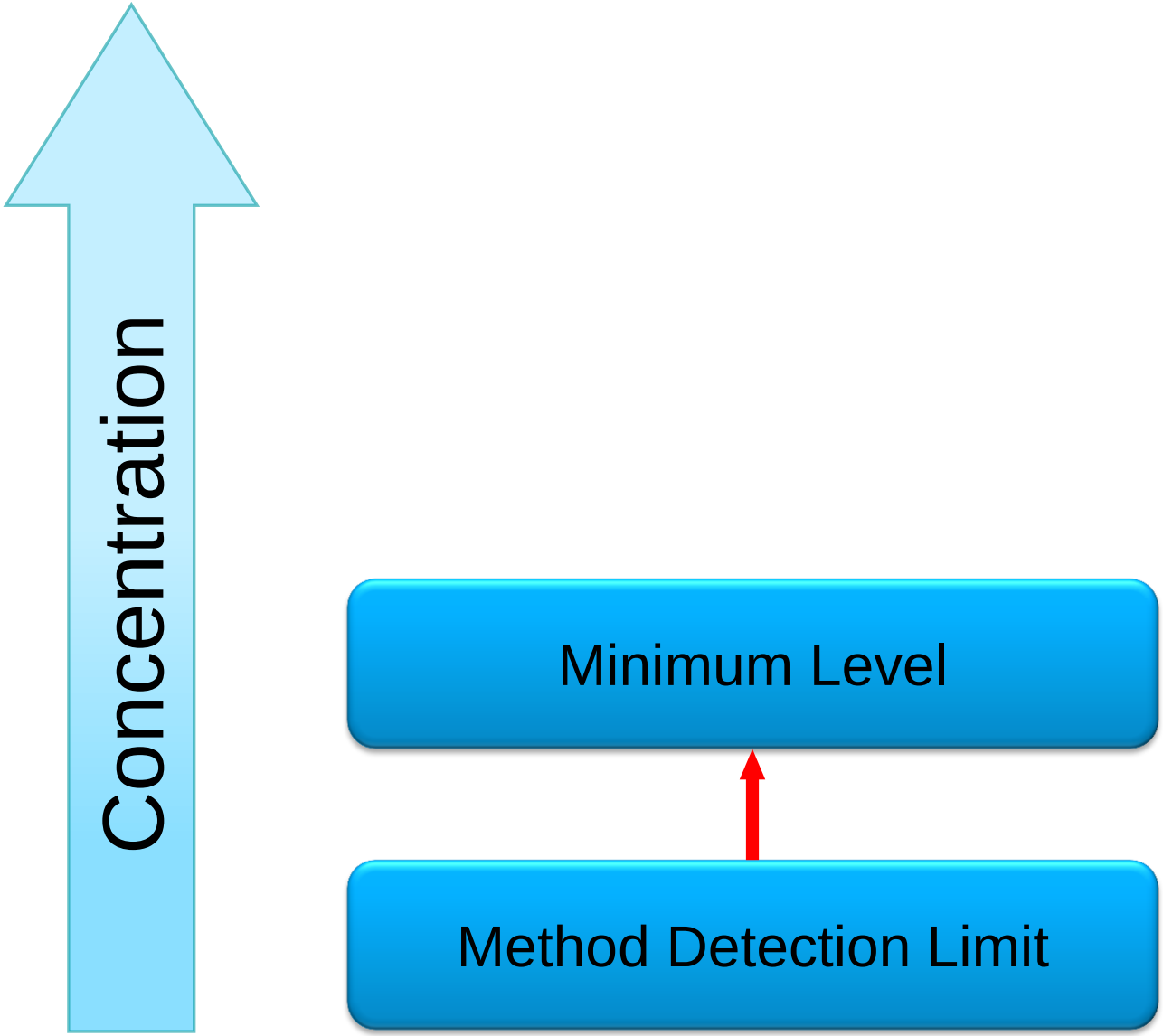


Sample Prep + Analyses + Lab = MDL

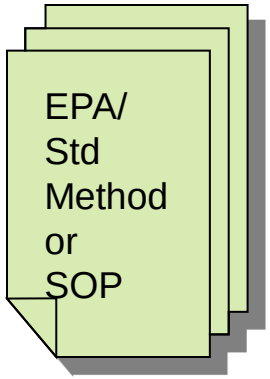
The higher value of seven spike replicates or seven blank replicates

MDL = lowest level signal produced

A signal is detected


Concentration
Minimum Level
Method Detection Limit

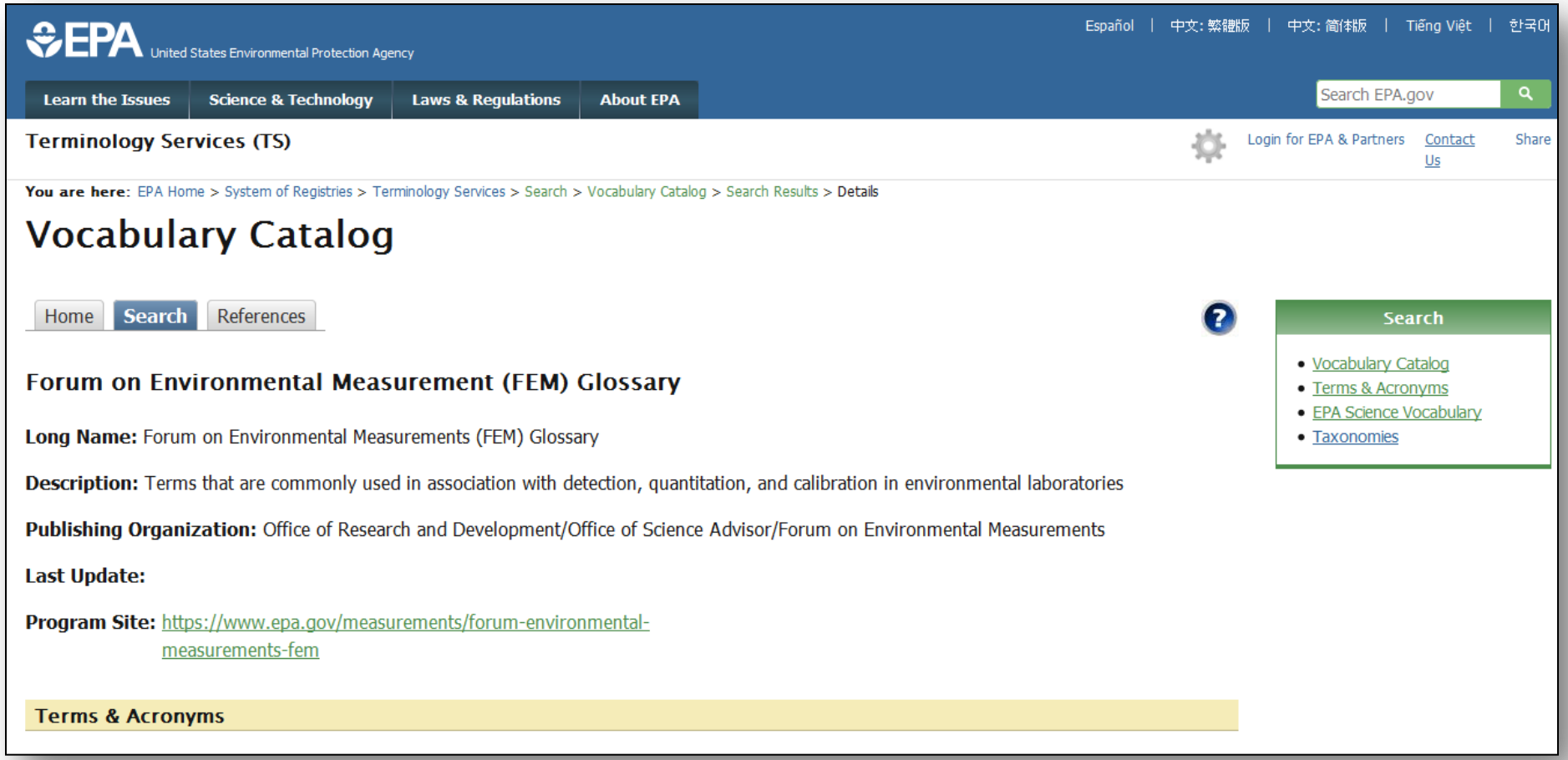
Minimum Level



MDL X (3.18)

Method + MDL + Factor = ML

ML = lowest point on calibration curve



EPA United States Environmental Protection Agency

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Vocabulary Catalog

Home | Search | References

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

Last Update:

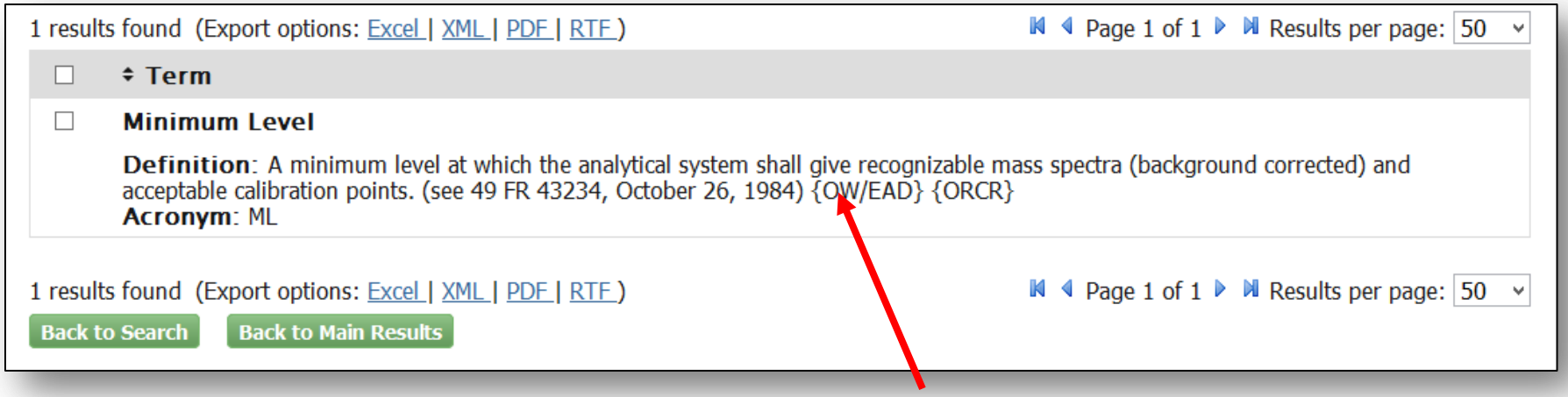
Program Site: https://www.epa.gov/measurements/forum-environmental-measurements-fem

Terms & Acronyms

Search

- Vocabulary Catalog
- Terms & Acronyms
- EPA Science Vocabulary
- Taxonomies

Minimum Level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Page 1 of 1 Results per page: 50

Term

Minimum Level

Definition: A minimum level at which the analytical system shall give recognizable mass spectra (background corrected) and acceptable calibration points. (see 49 FR 43234, October 26, 1984) {OW/EAD} {ORCR}
Acronym: ML

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Page 1 of 1 Results per page: 50

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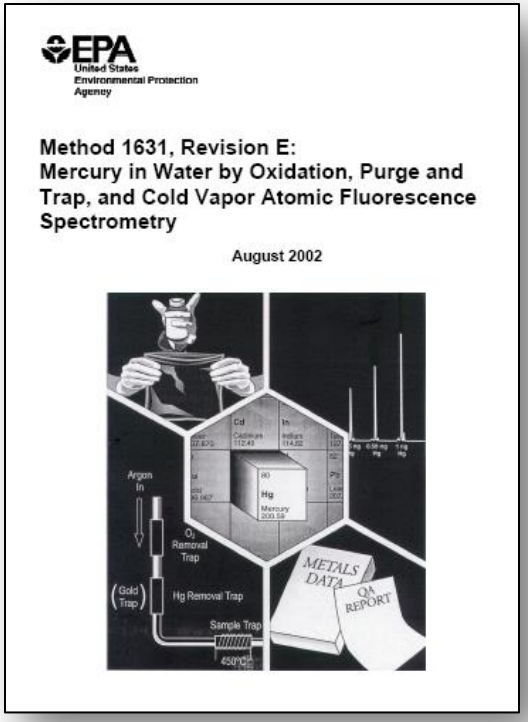
A minimum level at which the analytical system shall give recognizable mass spectra (background corrected) and acceptable calibration points.

Example: EPA Method 1631 – Mercury in Water


EPA
United States
Environmental Protection
Agency

Method 1631, Revision E:
Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry

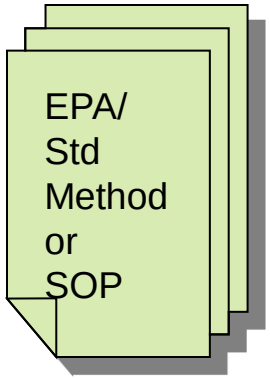
August 2002

*"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 10n, where n is an integer.
- Minimum levels are used in some US EPA methods.

Minimum Level



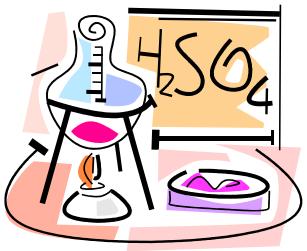
MDL X (3.18)

Method + MDL + Factor = ML

ML = lowest point on calibration curve

A signal is quantified

Method Detection Limit

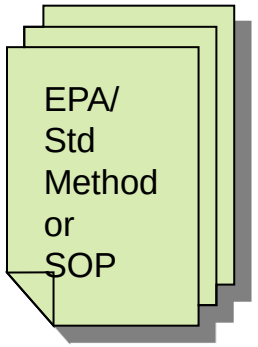


Sample Prep + Analyses + Lab = MDL

The higher value of 7 spike replicates or
7 blank replicates

MDL = lowest level signal produced
A signal is detected

Minimum Level



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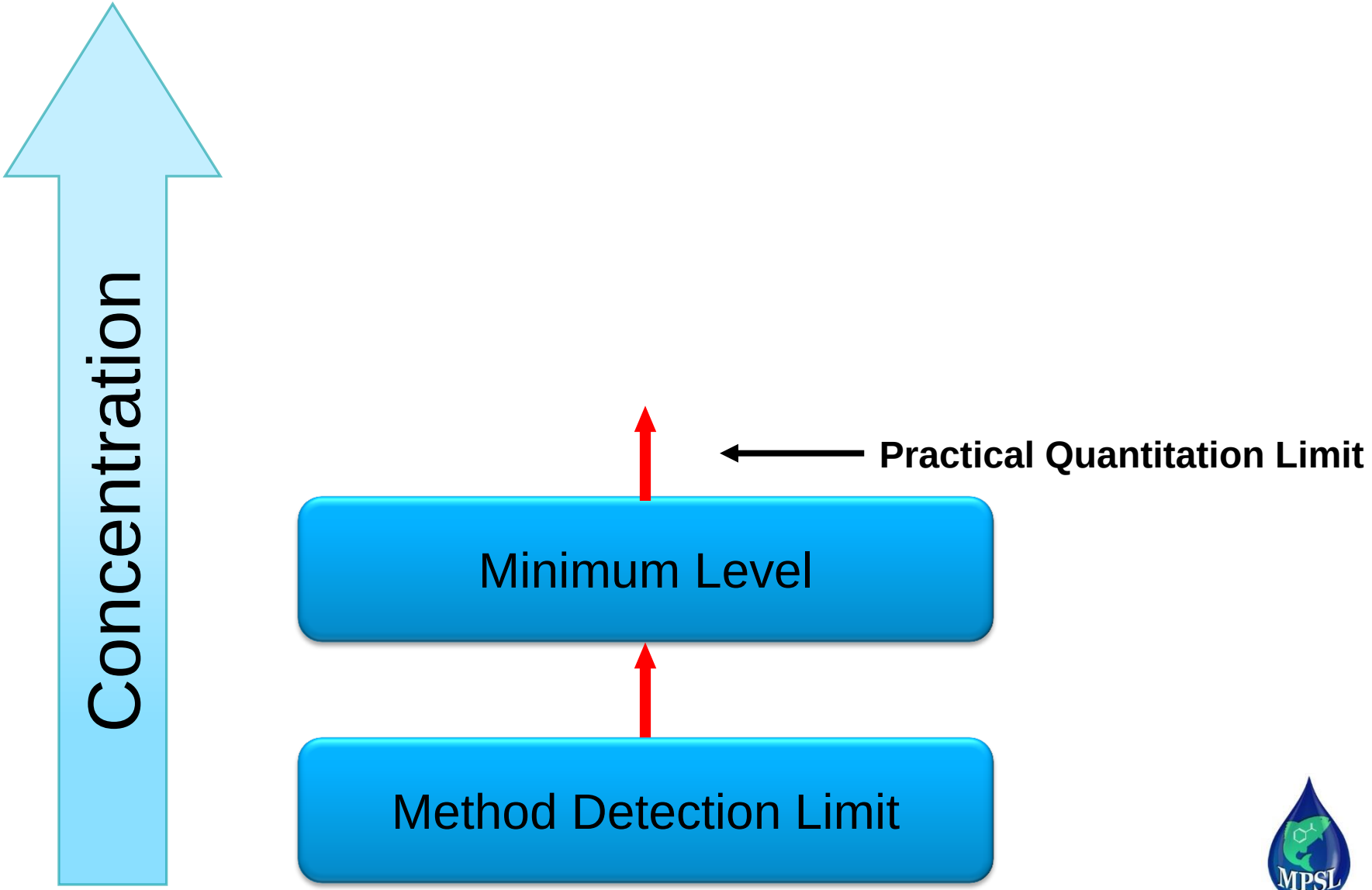
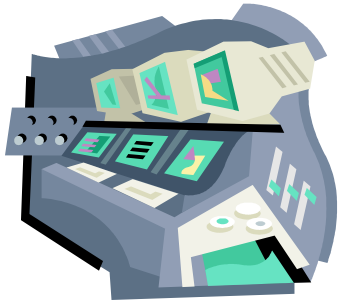
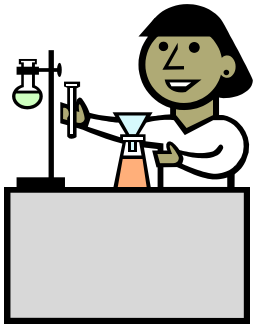

Concentration
Practical Quantitation Limit
Minimum Level
Method Detection Limit

Practical Quantitation Limit





X (2-10)

Instrument + Analyst + Factor = PQL

***** or *****

PQL = 3 times lowest point on calibration curve

Terms & Acronyms

Search Terms

practical quantitation limit ◉ Contains ○ Begins ○ Exact Match Search

Back to Search Back to Main Results

Terms & Acronyms

Search Terms (Contains): practical quantitation limit

Back to Search Back to Main Results

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Nothing found to display.

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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 a 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 our below the water quality standard. This was deemed appropriate given the use of the detection limit multiplier of 10.

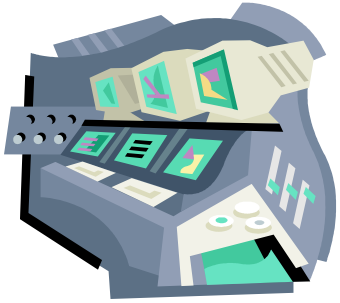
Unnamed Western State Water Quality Control Division

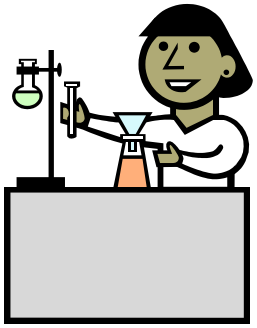
the MDL is defined by the statistical window, the PQL is essentially arbitrary. There are recommendations, PQL = IDL x 10 or MDL x 6 and others... but no governmental regulation covers the PQL. It comes down to what the laboratory feels comfortable signing their name to, confidently, on a daily basis.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





X (2-10)

Instrument + Analyst + Factor = PQL

A signal is quantified

***** or *****

PQL = 3 times lowest point on calibration curve

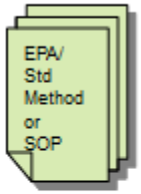
A signal is quantified *with* statistical rigor

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



MDL X (3.18)

Method + MDL + Factor = ML

ML = lowest point on calibration curve
A signal is quantified

Practical Quantitation Limit

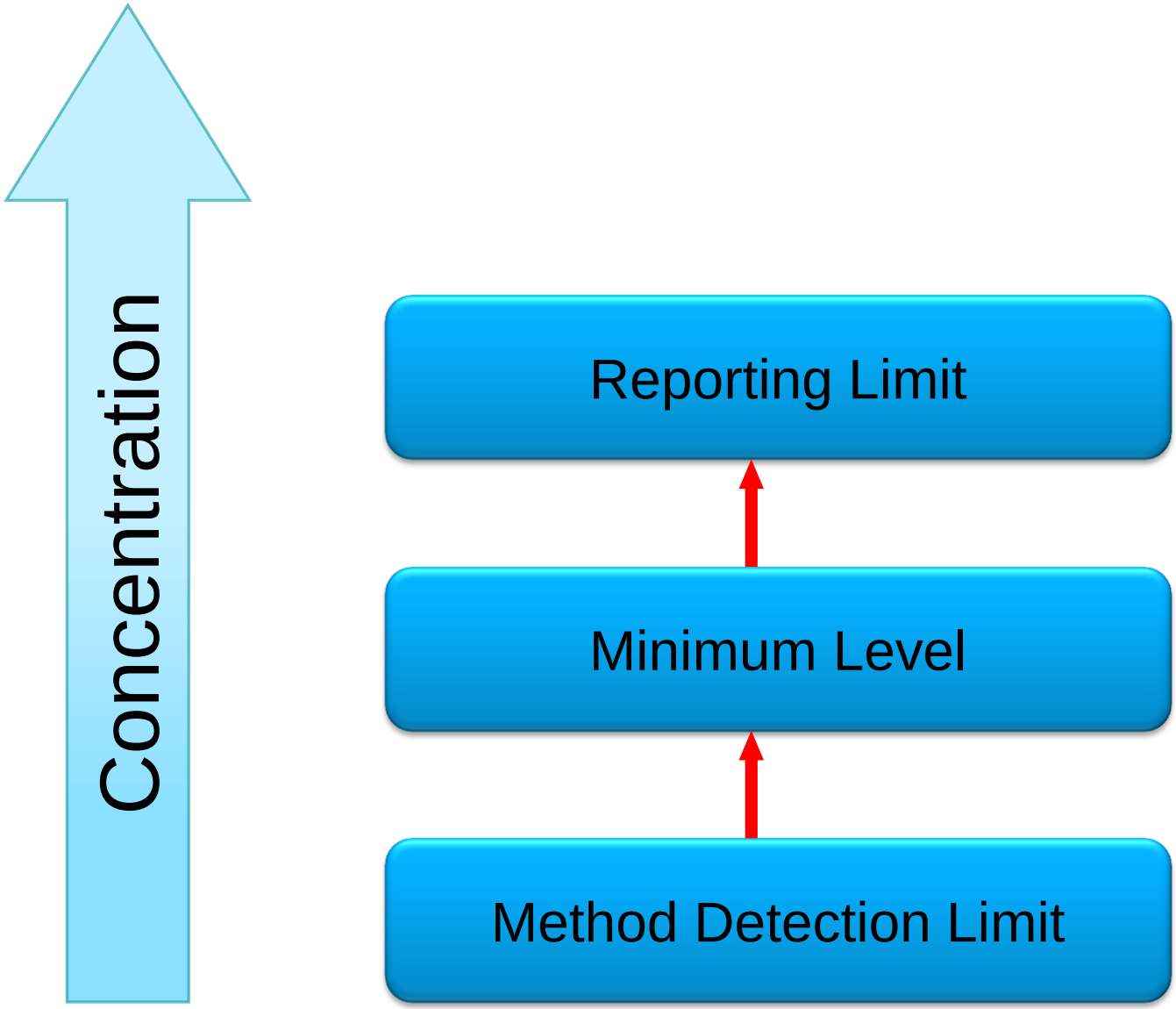

X (2-10)

Instrument + Analyst + Factor = PQL

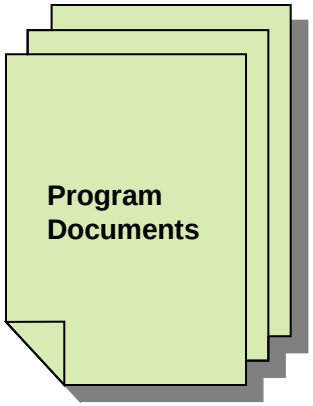
A signal is quantified

***** or *****

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

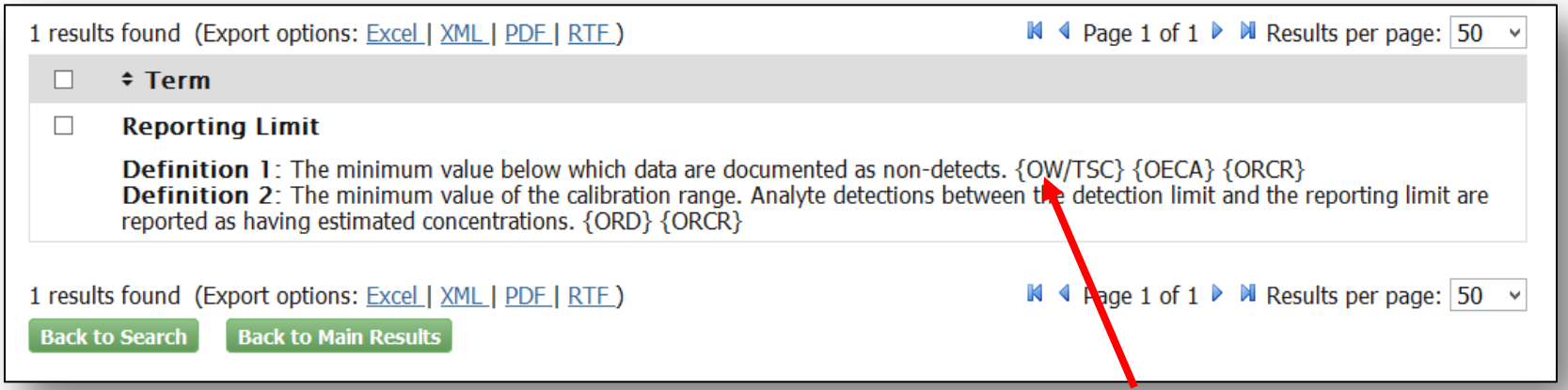

Concentration
Reporting Limit
Minimum Level
Method Detection Limit

Reporting Limit



Program + 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

USGS
science for a changing world

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.

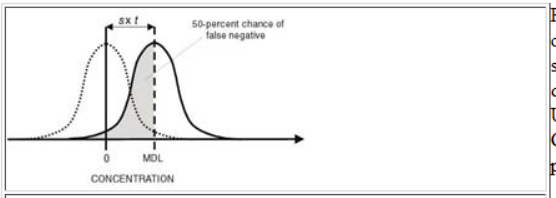


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Figure 8. False negative probability when a sample contains the analyte at the method detection limit (MDL) concentration.

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 *x* LT–MDL.

As shown in figure 9, mu
can be lower or higher th

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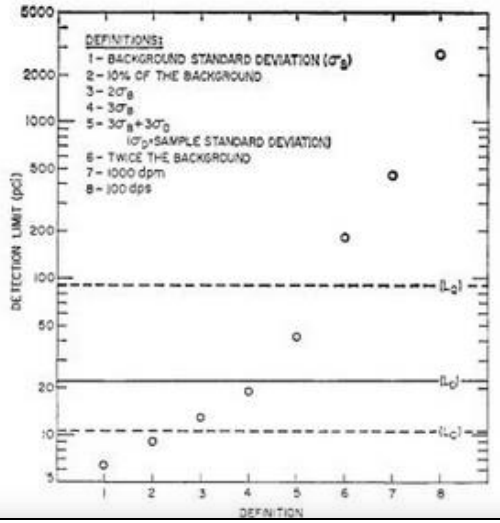
https://water.usgs.gov/owq/OFR_99-193/level.html

Limits for Qualitative Detection and Quantitative Determination

Application to Radiochemistry

Lloyd A. Currie
Analytical Chemistry Division, National Bureau of Standards, Washington, D. C. 20234

The occurrence in the literature of numerous, inconsistent and limited definitions of a detection limit has led to a re-examination of the questions of signal detection and signal extraction in analytical chemistry and nuclear chemistry. Three limiting levels have been defined: L_C–the net signal level (instrument response) above which an *observed* signal may be reliably recognized as "detected"; L_D–the "true" net signal level which may be *a priori* expected to lead to detection; and L_Q–the level at which the measurement precision will be satisfactory for quantitative determination. Exact defining equations as well as series of working formulae are presented both for the general analytical case and for radioactivity. The latter, assumed to be governed by the Poisson distribution, is treated in such a manner that accurate limits may be derived for both short- and long-lived radionuclides either in the presence or absence of interference. The principles are illustrated by simple examples of spectrophotometry and radioactivity, and by a more complicated example of activation analysis in which a choice must be made between alternative nuclear reactions.



IN THE COURSE of research dealing with photonuclear reactions and activation analysis, it became necessary to determine limits of detection of radiochemical procedures, to select among alternative procedures, and to op respect to certain experimental the analytical and radiochemical definition of the limit of detec mathematical expressions and One encounters, for example, ter tection (*1*), detection sensitivity detectable activity (or mass) (*4*, purity (*6*)—all used with approx The nomenclature problem is compounded, however, because other authors make use of the same, or very similar, terms to refer not to the minimum amount that may be *detected*, but rather to the minimum amount which may be *determined* with

detection limit is equated to the background, 10% of the background, 100 dps (γ-radioactivity), or 1000 dpm (α-, β-,γ-radioactivity). In order to compare some of the more commonly-used definitions, "detection limits" have been calculated for a

Currie then defined measures of detectability, firmly based on the statistical theory of hypothesis testing.

Richard M. Lindstrom, NIST – See Slide 41

Lloyd A. Currie, Limits for Qualitative Detection and Quantitative Determination: Application to Radiochemistry, *Anal. Chem.* 40, 586-593 (1968).

http://pubs.acs.org/doi/pdf/10.1021/ac60259a007

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 disarmingly 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

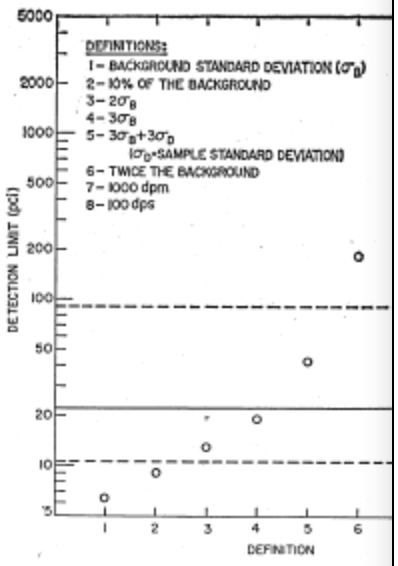


Fig.1. "Ordered" detection limits—literature definit
tion limit for a specific radioactivity measurement p
in increasing order, according to commonly-used al
tions. L_C, L_D, and L_Q are the critical level, detection
mination limit as derived in the text.

Currie then defined measures of detectability, firmly based on the statistical theory of hypothesis testing. He began by defining the concepts of qualitative and

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 σ_0 of the blank, or background. By choosing a probability α (error of the first kind) for falsely deciding that the

http://nvlpubs.nist.gov/nistpubs/sp958-lide/164-166.pdf

ELSEVIER

Analytica Chimica Acta 391 (1999) 127–134

ANALYTICA CHIMICA ACTA

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 ca… of me… there have been decades of co… ncept… coordinated documents prepare… (IUPAC) [L.A. Currie, IUPAC Commission on Analytical Nor… ethods including Detection and

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

Detection decision (critical value) (L_C, α=0.05):

$$\Pr(\hat{L} > L_C | L = 0) \leq \alpha. \quad (1)$$

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

$$\Pr(\hat{L} \leq L_C | L = L_D) = \beta. \quad (2)$$

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

$$L_Q = k_Q \sigma_Q, \quad \text{where } k_Q = 1/RSD_Q. \quad (3)$$

Also addressed in the IUPAC document is 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

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

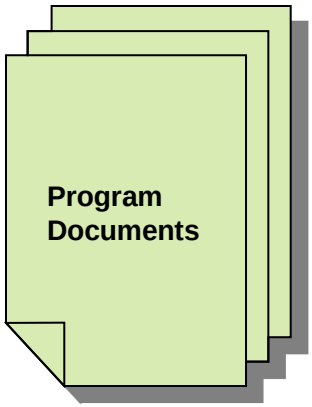
=

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

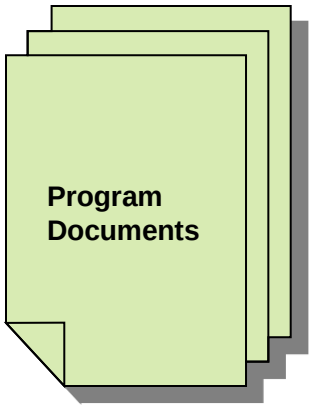
Analytical Method

Dependent on USE

Reporting Limit

Data Use in Decisions

Reporting Limit



Program + Data Use = RL

(Action Limit)

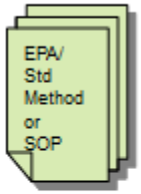
A signal is quantified and is more robust if it incorporates **statistical rigor**

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

Minimum Level



MDL X (3.18)

Method + MDL + Factor = ML

ML = lowest point on calibration curve
A signal is quantified

Practical Quantitation Limit


X (2-10)

Instrument + Analyst + Factor = PQL

A signal is quantified

**** or ****

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

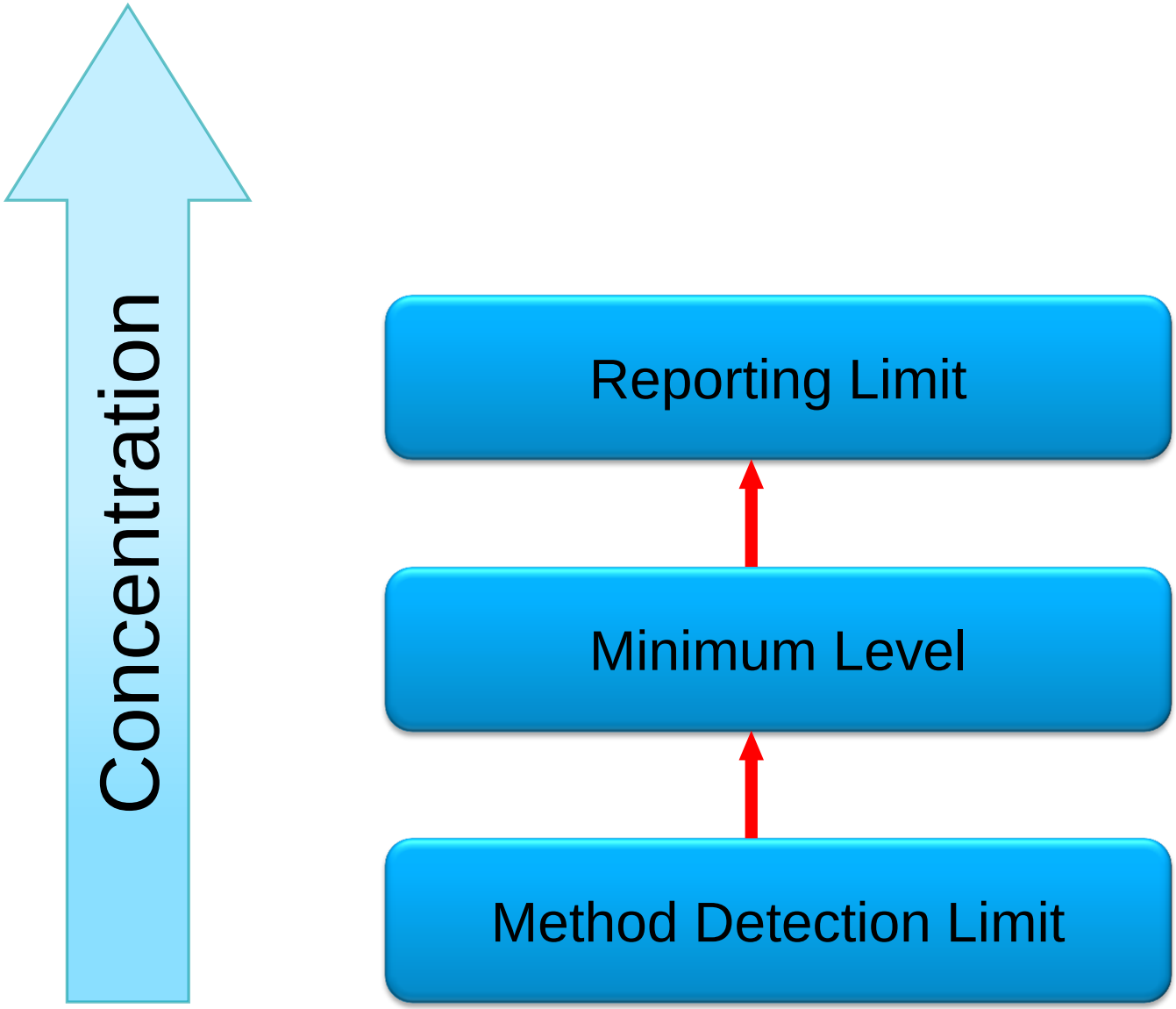
Reporting Limit

Program + Data Use = RL

(Action Limit)

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

MPSL

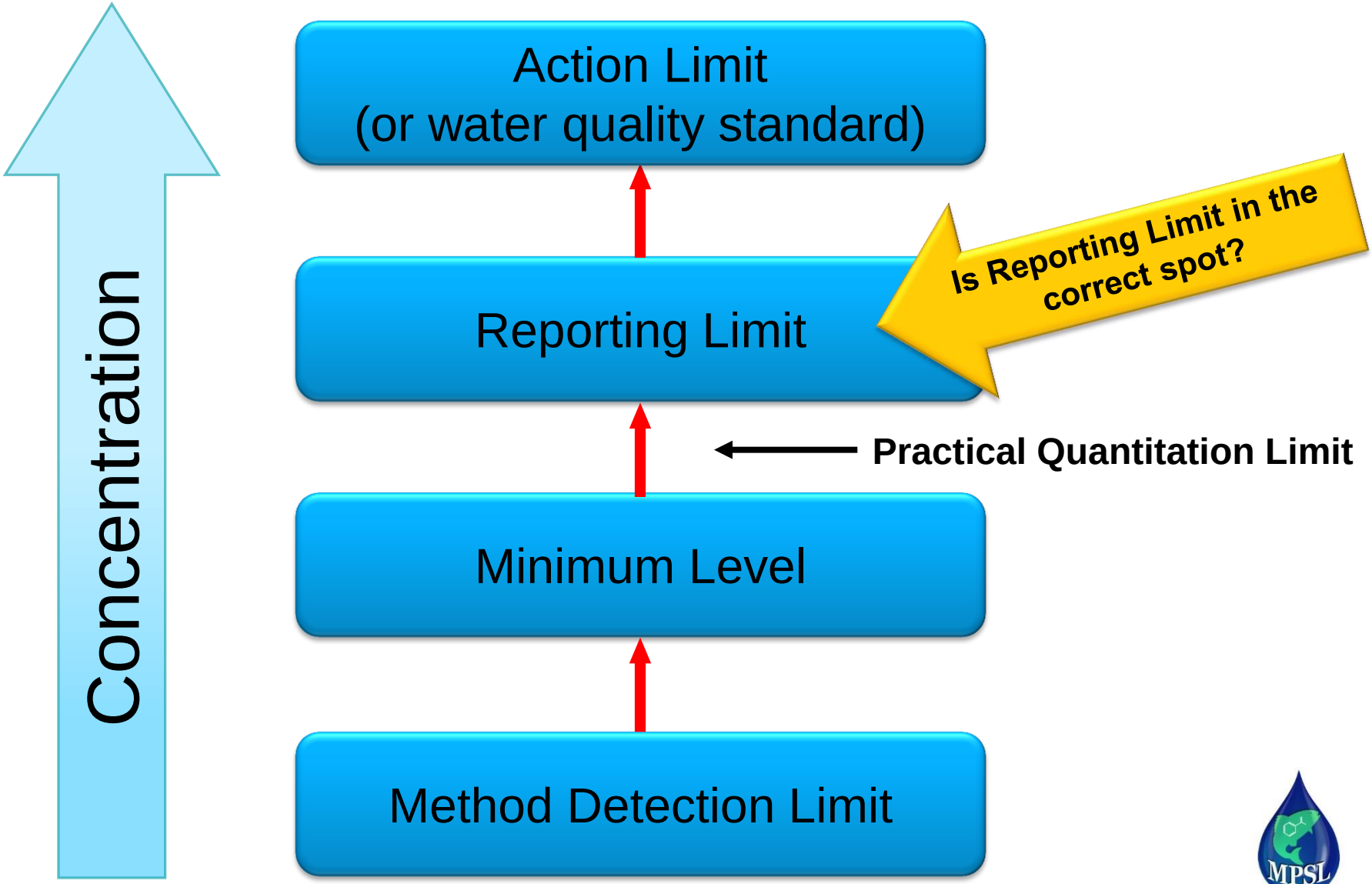

Concentration
Reporting Limit
Minimum Level
Method Detection Limit

- MDL = Method Detection Limit
  Detected
- ML = Minimum Level
  Quantified
- PQL = Practical Quantitation Limit
  Quantified

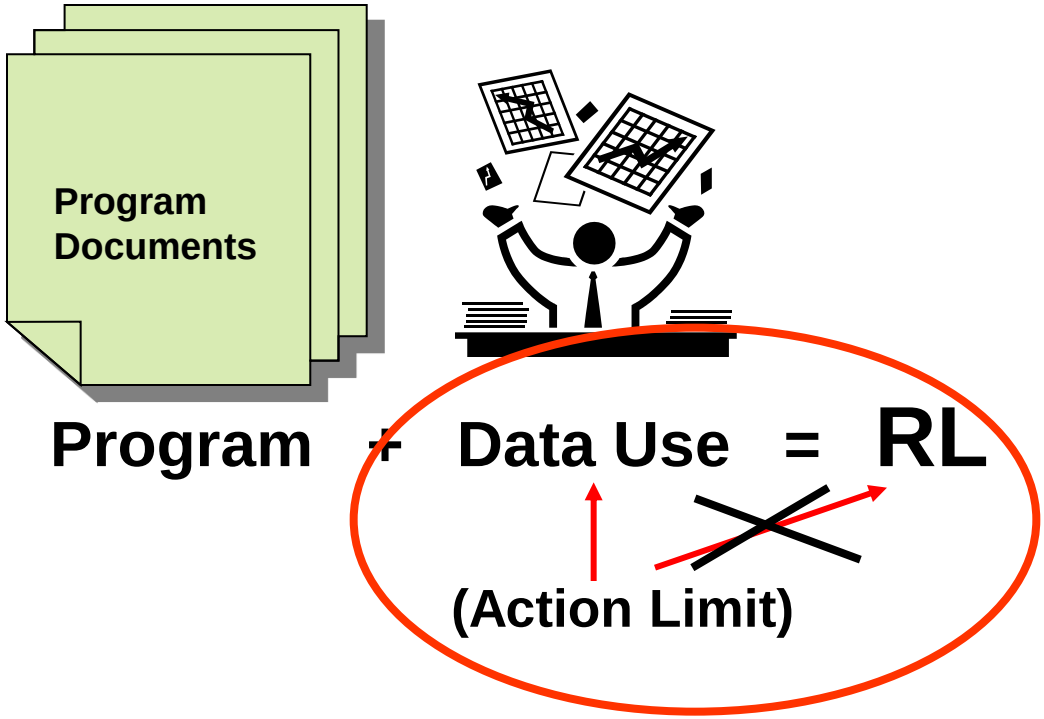
  If 3x the lowest point on calibration curve
  Quantified *with* Statistical Rigor
- RL = Reporting Limit
  Quantified
  May be defined as quantified with statistical rigor

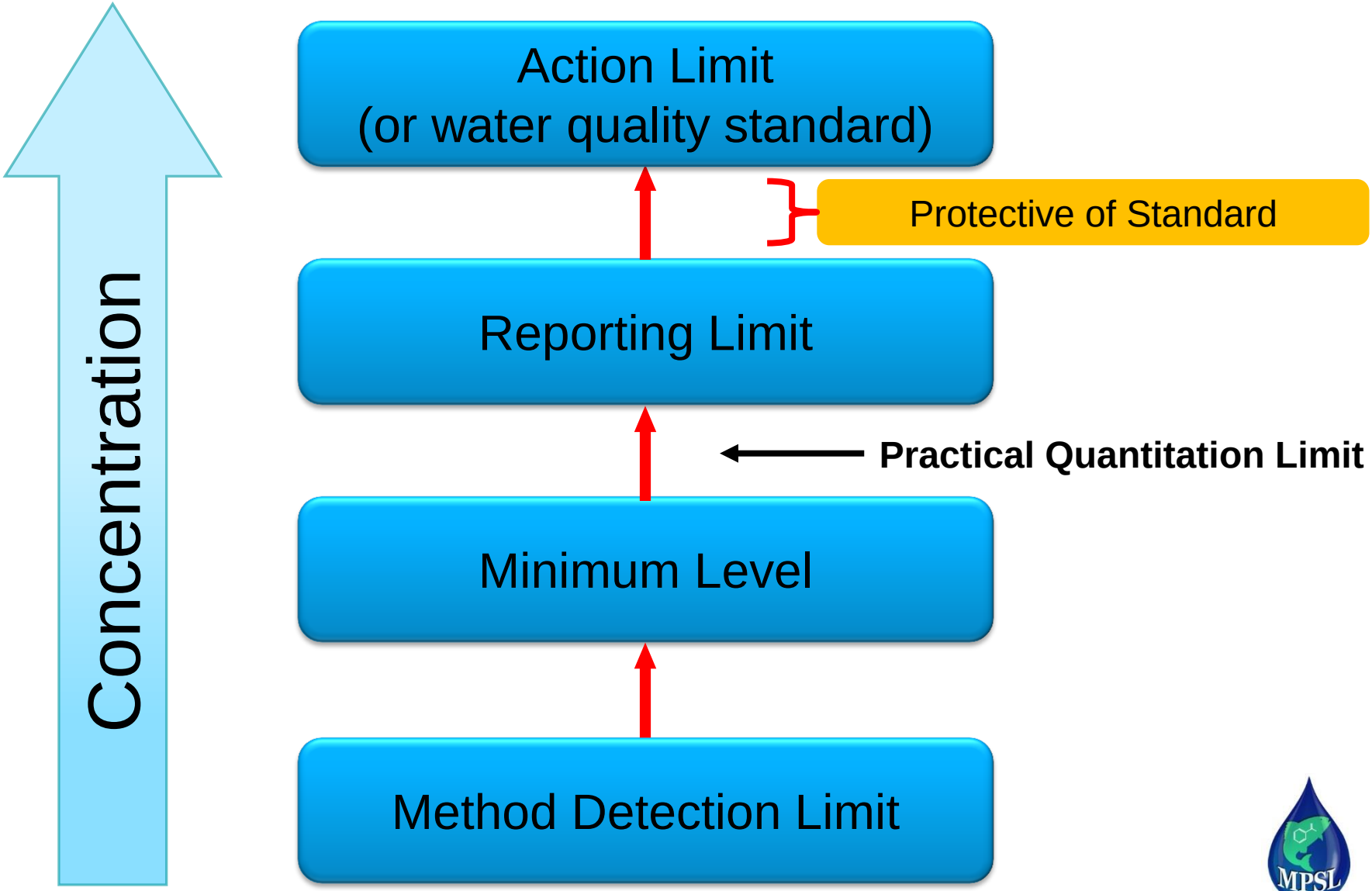
LOD = Limit of Detection
Detected

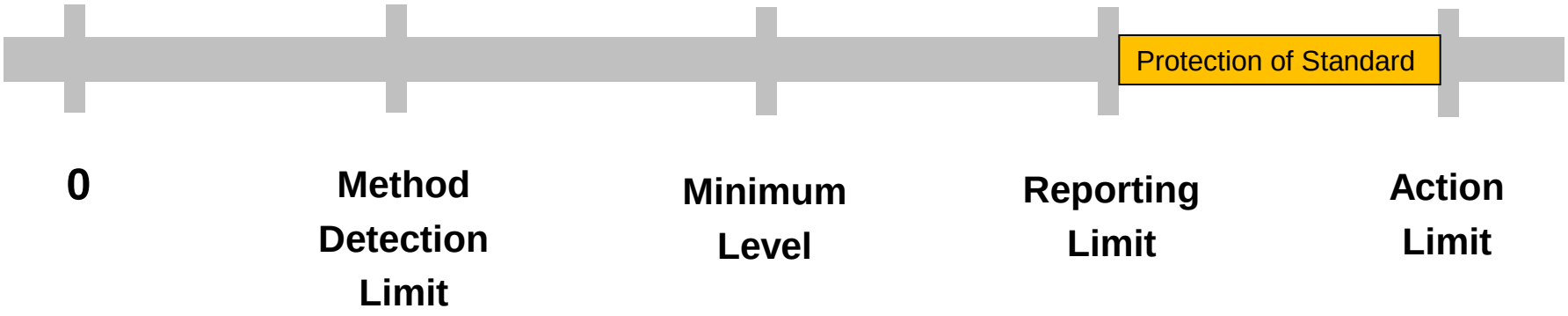
LOQ = Limit of Quantification
Quantified


Concentration
Action Limit
(or water quality standard)
Reporting Limit
Is Reporting Limit in the correct spot?
Practical Quantitation Limit
Minimum Level
Method Detection Limit

Reporting Limit




Concentration
Action Limit
(or water quality standard)
Protective of Standard
Reporting Limit
Practical Quantitation Limit
Minimum Level
Method Detection Limit


Protection of Standard
0
Method Detection Limit
Minimum Level
Reporting Limit
Action Limit
Concentration

Examples

Example: *Unnamed Midwestern State* Environmental Protection Agency

Same as "Action Limit"

Parameter	Units	Typical MDL	Practical Quantitation Limit	Maximum Contaminant Level
Carbon Tetrachloride	µg/L	0.02 (EPA 180.1)	0.5	25
Chlorobenzene	µg/L	0.03 (EPA 200.8)	2.0	14
Chloroform	µg/L	0.2 (EPA 200.8)	2.5	117

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

Same as "Minimum Level"

Same as "Action Limit"

Parameter	Units	Typical MDL	Laboratory Quantitation Level	Benchmark
Malathion	µg/L	0.02 (EPA 8141A)	0.5	25
Mirex	µg/L	0.03 (EPA 8081A)	2.0	14
Simazine	µg/L	0.2 (EPA 200.8)	2.5	117

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.
  - 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
  - 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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Numeric Water Quality Criteria

EPA United States Environmental Protection Agency

Environmental Topics | Laws & Regulations | About EPA | Search EPA.gov

Related Topics: Water Quality Standards: Regulations and Resources

Contact Us | Share

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.

Related Information

- Water Quality Standards Regulations: California

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

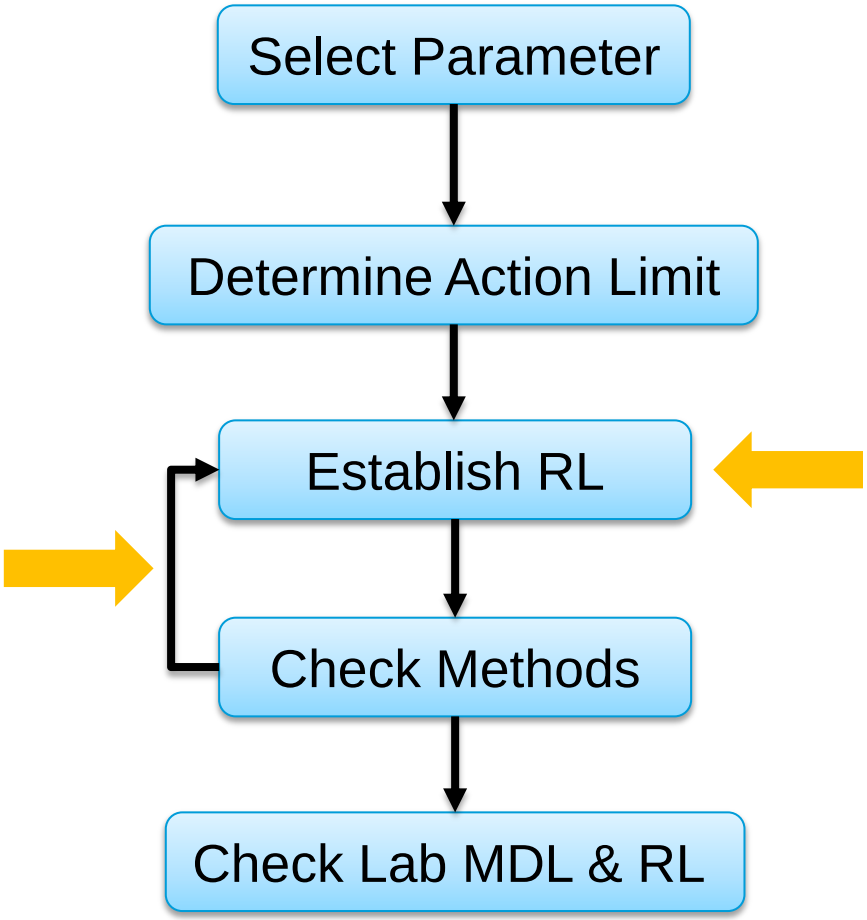
Example: California Toxics Rule

- 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.

- Specific CTR standards can be found in 40 CFR 131.38 www.gpo.gov/fdsys/pkg/CFR-2012-title40-vol23/pdf/CFR-2012-title40-vol23-sec131-38.pdf

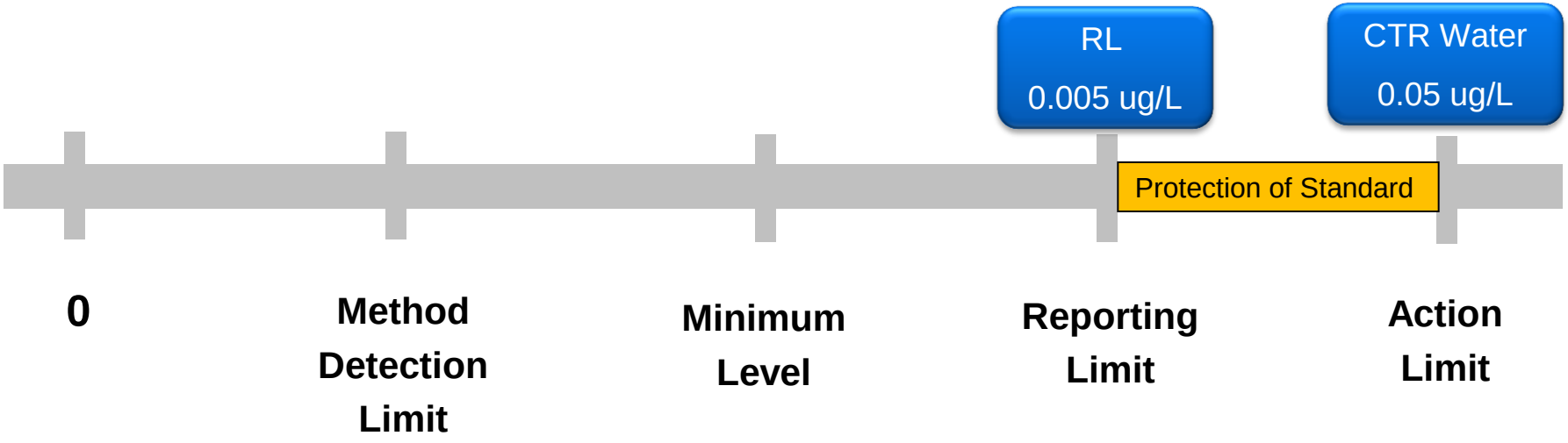
Process for Determining Reporting Limits



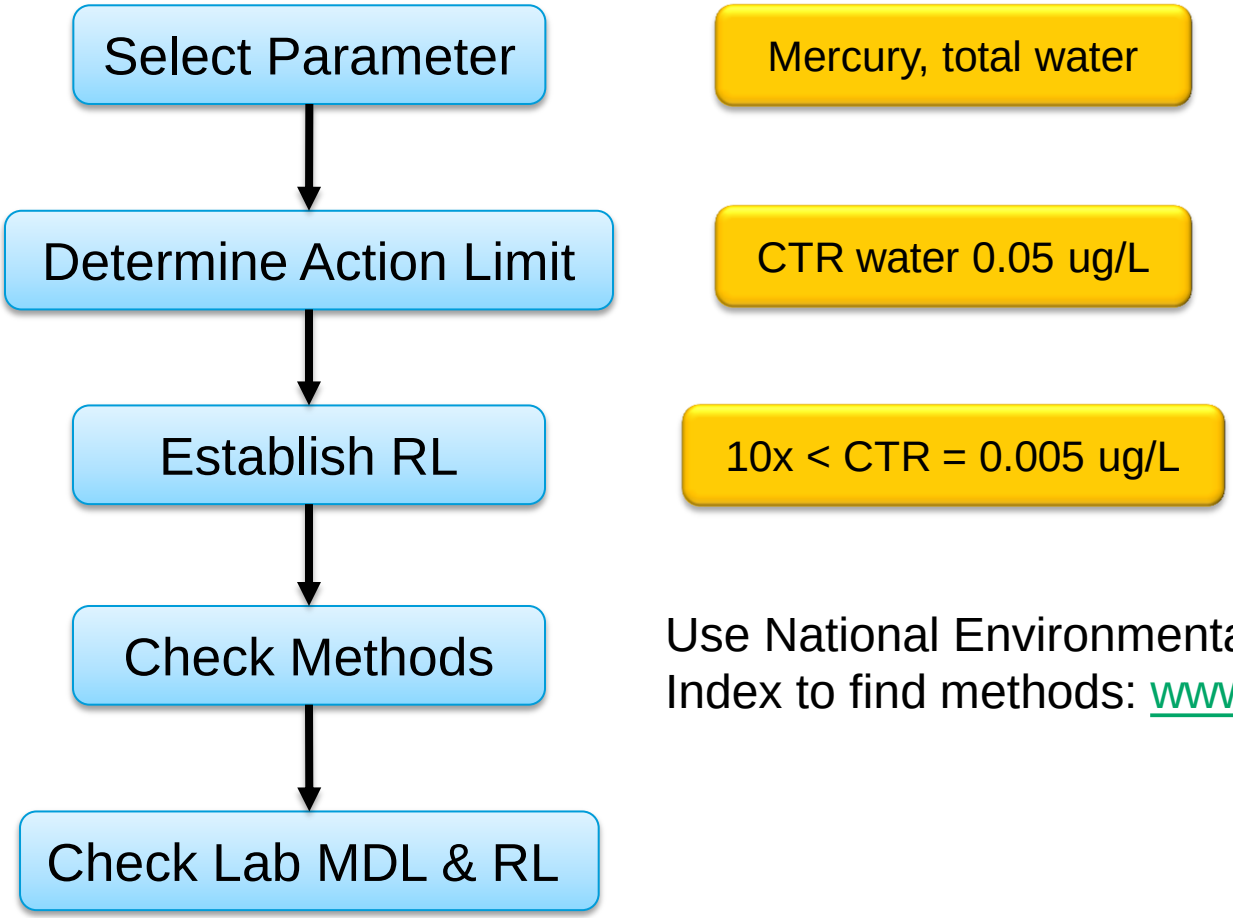
A		B Freshwater		C Saltwater		D Human Health (10^{-6} risk for carcinogens) For consumption of:	
# Compound	CAS Number	Criterion Maximum Conc. [d] B1	Criterion Continuous Conc. [d] B2	Criterion Maximum Conc. [d] C1	Criterion Continuous Conc. [d] C2	Water & Organisms (µg/L) D1	Organisms Only (µg/L) D2
1. Antimony	7440360					14 a,s	4300 a,t
2. Arsenic [b]	7440382	340 i,m,w	150 i,m,w	69 i,m	36 i,m		
3. Beryllium	7440417					n	n
4. Cadmium [b]	7440439	4.3 e,i,m,w,x	2.2 e,i,m,w	42 i,m	9.3 i,m	n	n
5a. Chromium (III)	16065831	550 e,i,m,o	180 e,i,m,o			n	n
5b. Chromium (VI) [b]	18540299	16 i,m,w	11 i,m,w	1100 i,m	50 i,m	n	n
6. Copper [b]	7440508	13 e,i,m,w,x	9.0 e,i,m,w	4.8 i,m	3.1 i,m	1300	
7. Lead [b]	7439921	65 e,i,m	2.5 e,i,m	210 i,m	8.1 i,m	n	n
8. Mercury [b]	7439976	[Reserved]	[Reserved]	[Reserved]	[Reserved]	0.050 a	0.051 a
9. Nickel [b]	7440020	470 e,i,m,w	52 e,i,m,w	74 i,m	8.2 i,m	610 a	4600 a

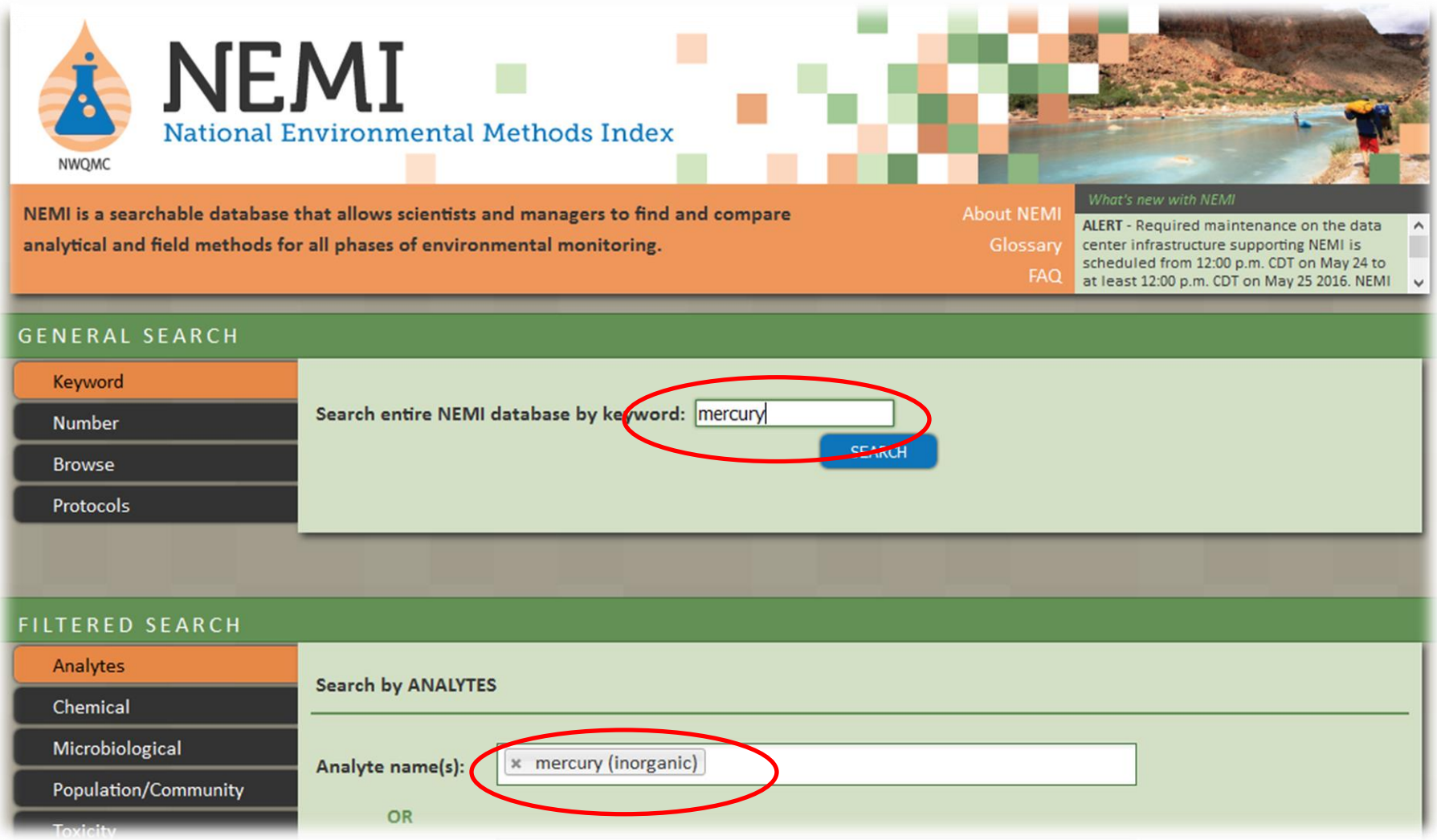
Establish RL

10x < CTR = 0.005 ug/L



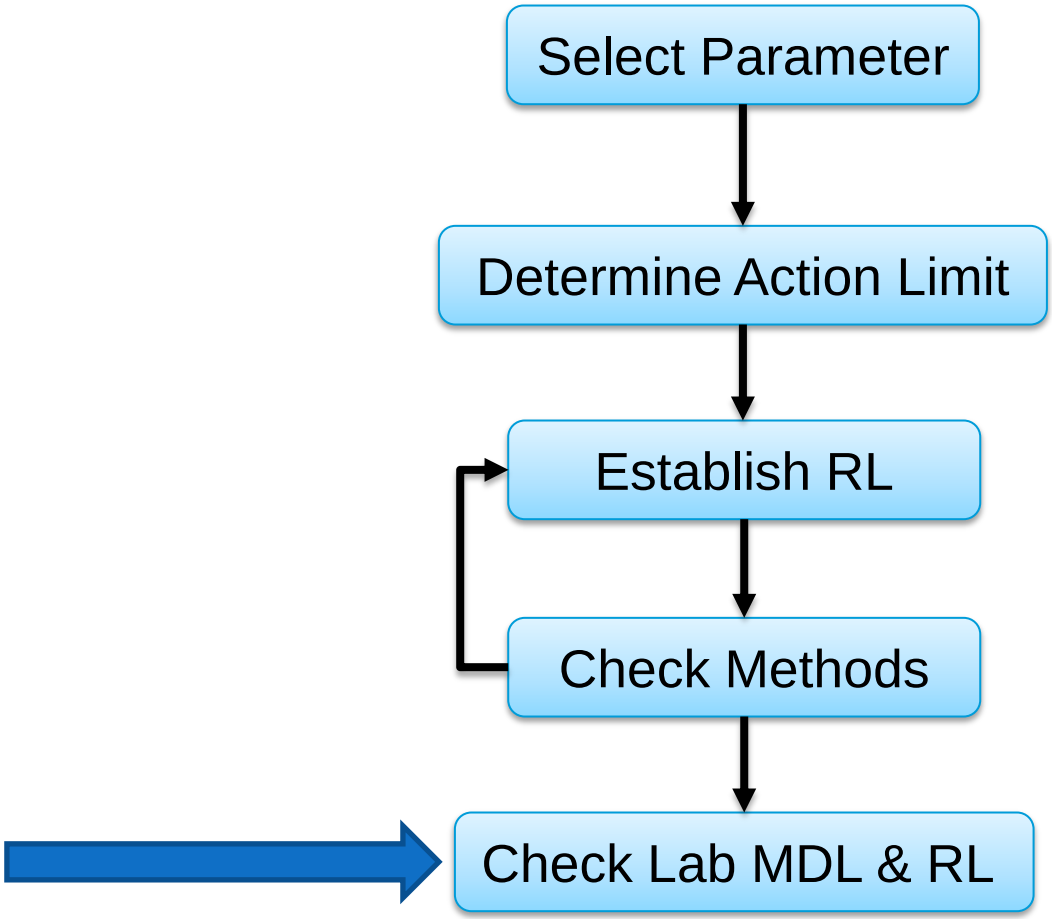
Concentration


Select Parameter
Mercury, total water
Determine Action Limit
CTR water 0.05 ug/L
Establish RL
10x < CTR = 0.005 ug/L
Check Methods
Use National Environmental Methods Index to find methods: www.nemi.gov
Check Lab MDL & RL

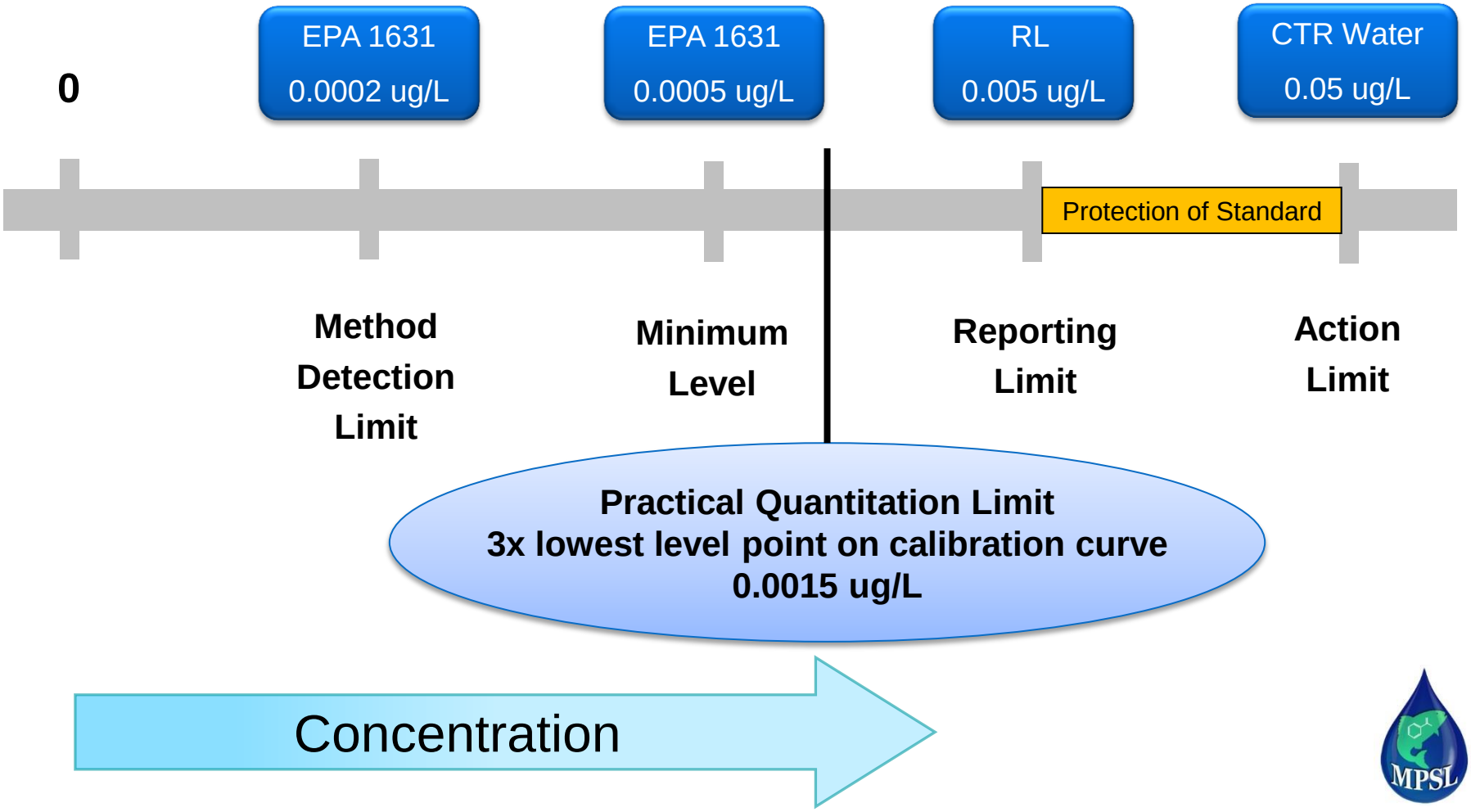

NWQMC
NEMI
National Environmental Methods Index
NEMI is a searchable database that allows scientists and managers to find and compare analytical and field methods for all phases of environmental monitoring.
About NEMI
Glossary
FAQ
What's new with NEMI
ALERT - Required maintenance on the data center infrastructure supporting NEMI is scheduled from 12:00 p.m. CDT on May 24 to at least 12:00 p.m. CDT on May 25 2016. NEMI
GENERAL SEARCH
Keyword
Number
Browse
Protocols
Search entire NEMI database by keyword:
mercury
SEARCH
FILTERED SEARCH
Analytes
Chemical
Microbiological
Population/Community
Toxicity
Search by ANALYTES
Analyte name(s):
mercury (inorganic)
OR

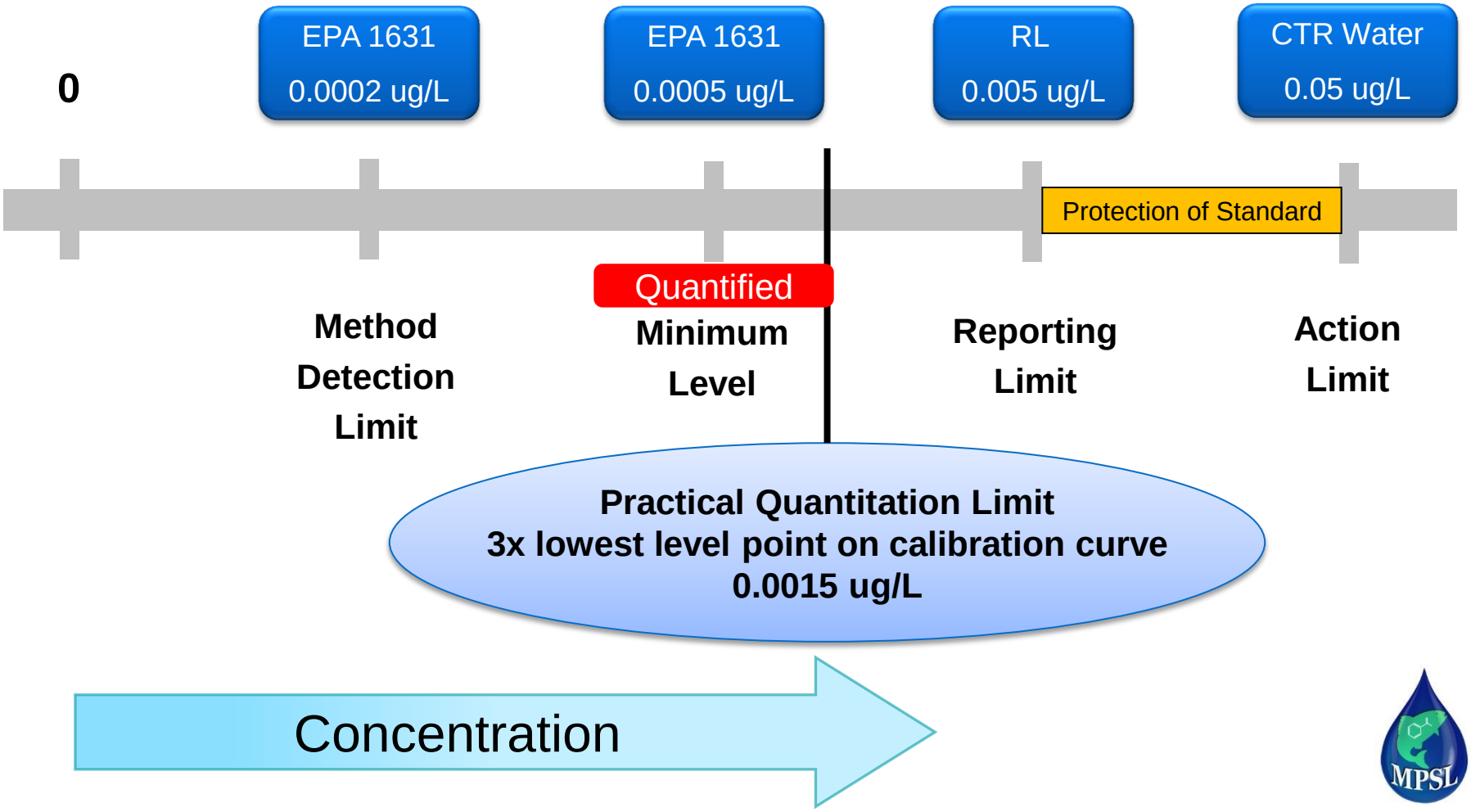
RL = 0.005 ug/L

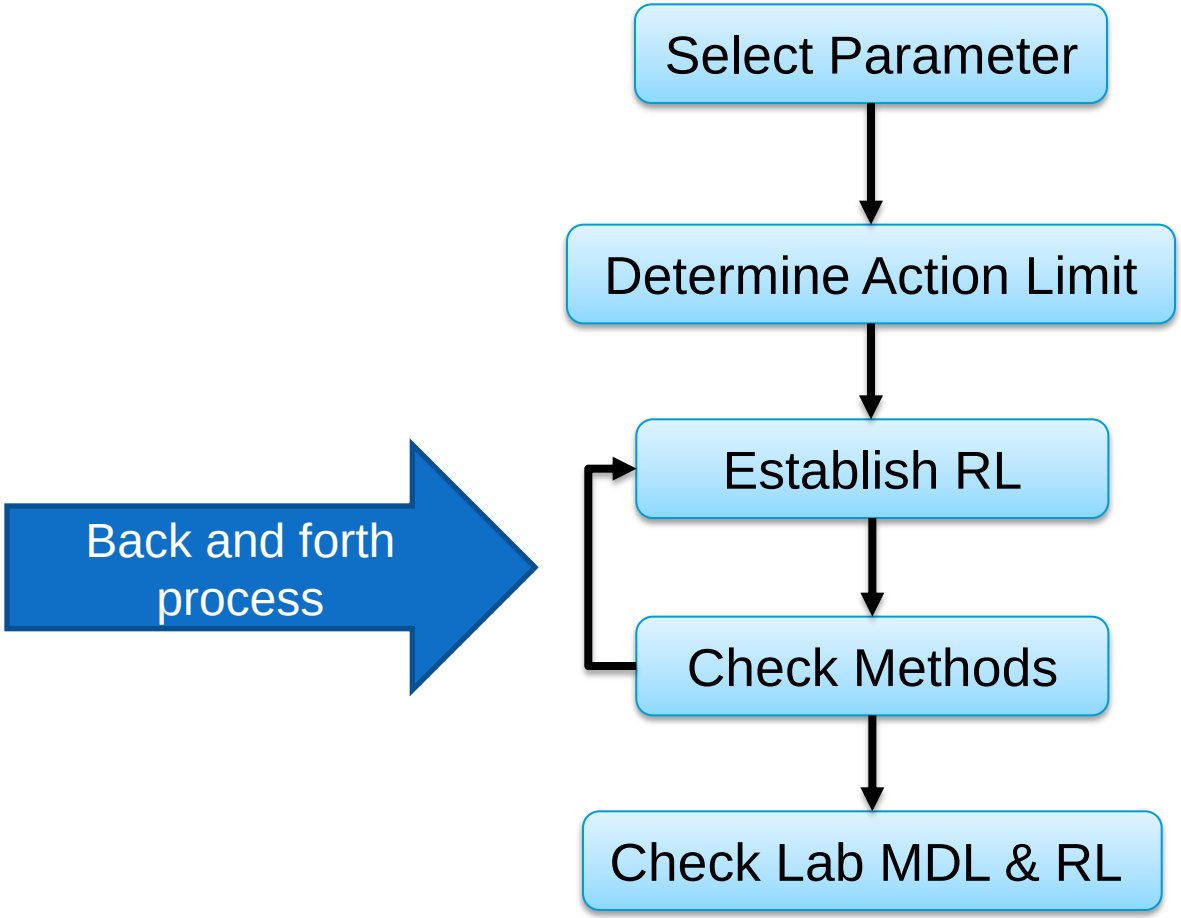
Method #	Source	Detection Level	Detection Type
200.8	EPA	0.2 ug/L	MDL
200.7	EPA	7 ug/L	MDL
245.1	EPA	0.2 ug/L	RNGE
I-1462	USGS	0.5 ug/L	RNGE
I-7462	USGS	0.5 ug/L	RL
D6502	ASTM	1 ug/L	ML
1631	EPA	0.0002 ug/L	MDL
245.7	EPA	0.0018 ug/L	MDL

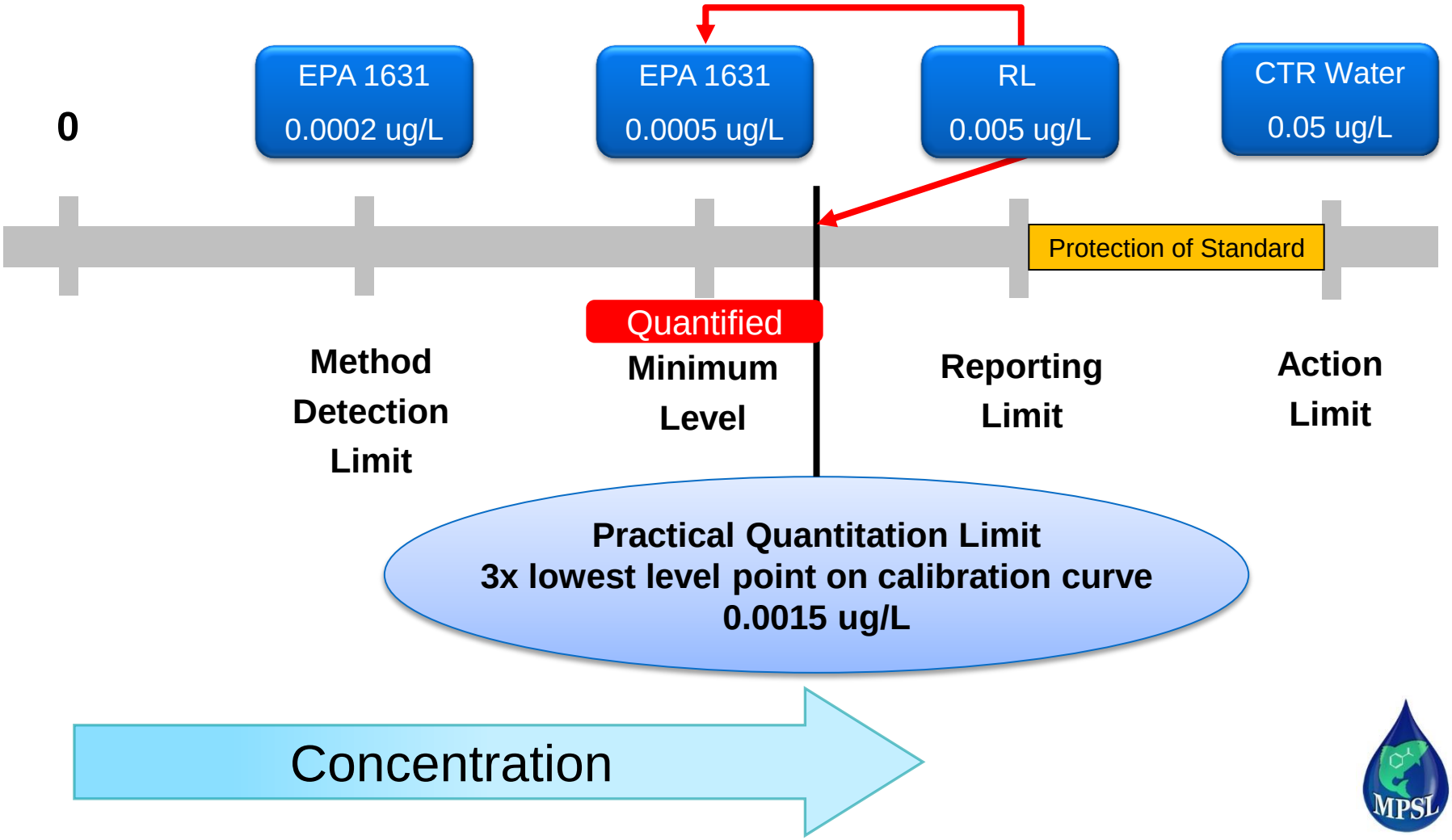


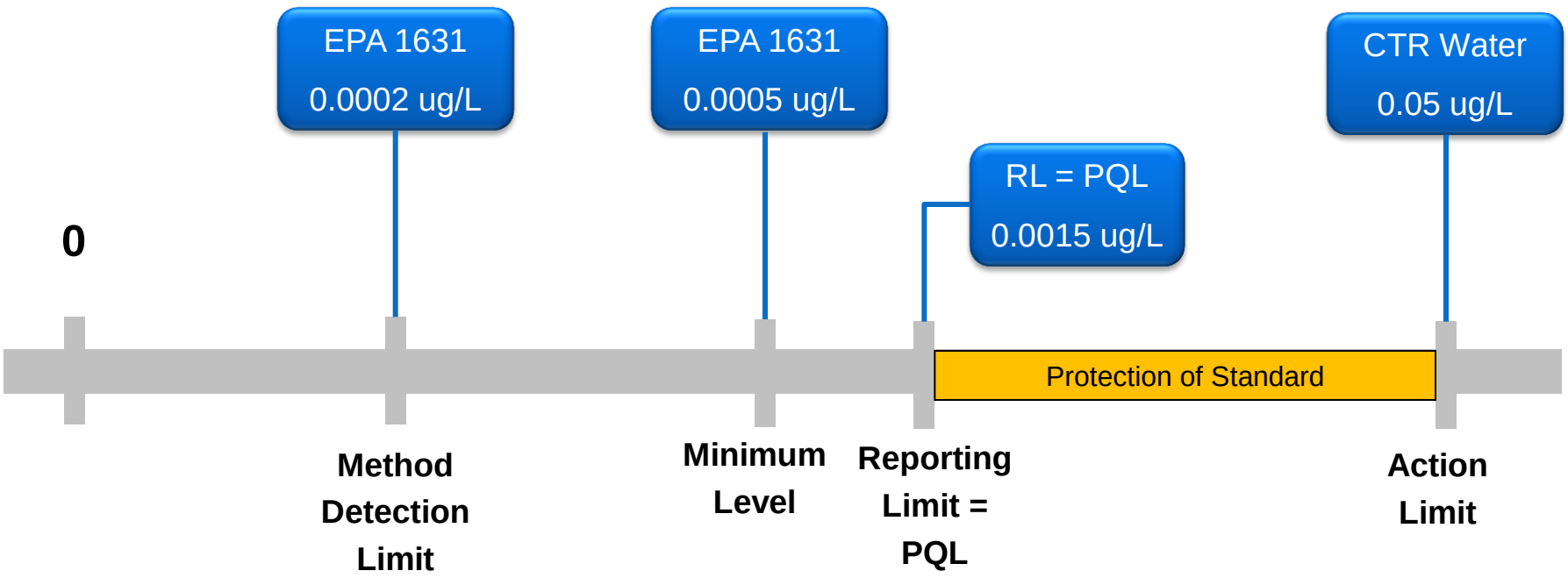
1631 Minimum Level = 0.0005 ug/L Cost $51-200
245.7 Minimum Level = 0.005 ug/L Cost $51-200


0
EPA 1631
0.0002 ug/L
EPA 1631
0.0005 ug/L
RL
0.005 ug/L
CTR Water
0.05 ug/L
Protection of Standard
Method Detection Limit
Minimum Level
Reporting Limit
Action Limit
Practical Quantitation Limit
3x lowest level point on calibration curve
0.0015 ug/L
Concentration


0
EPA 1631
0.0002 ug/L
EPA 1631
0.0005 ug/L
RL
0.005 ug/L
CTR Water
0.05 ug/L
Protection of Standard
Quantified
Method Detection Limit
Minimum Level
Reporting Limit
Action Limit
Practical Quantitation Limit
3x lowest level point on calibration curve
0.0015 ug/L
Concentration


Select Parameter
Determine Action Limit
Establish RL
Check Methods
Check Lab MDL & RL
Back and forth process


0
EPA 1631
0.0002 ug/L
EPA 1631
0.0005 ug/L
RL
0.005 ug/L
CTR Water
0.05 ug/L
Protection of Standard
Quantified
Method Detection Limit
Minimum Level
Reporting Limit
Action Limit
Practical Quantitation Limit
3x lowest level point on calibration curve
0.0015 ug/L
Concentration



Concentration

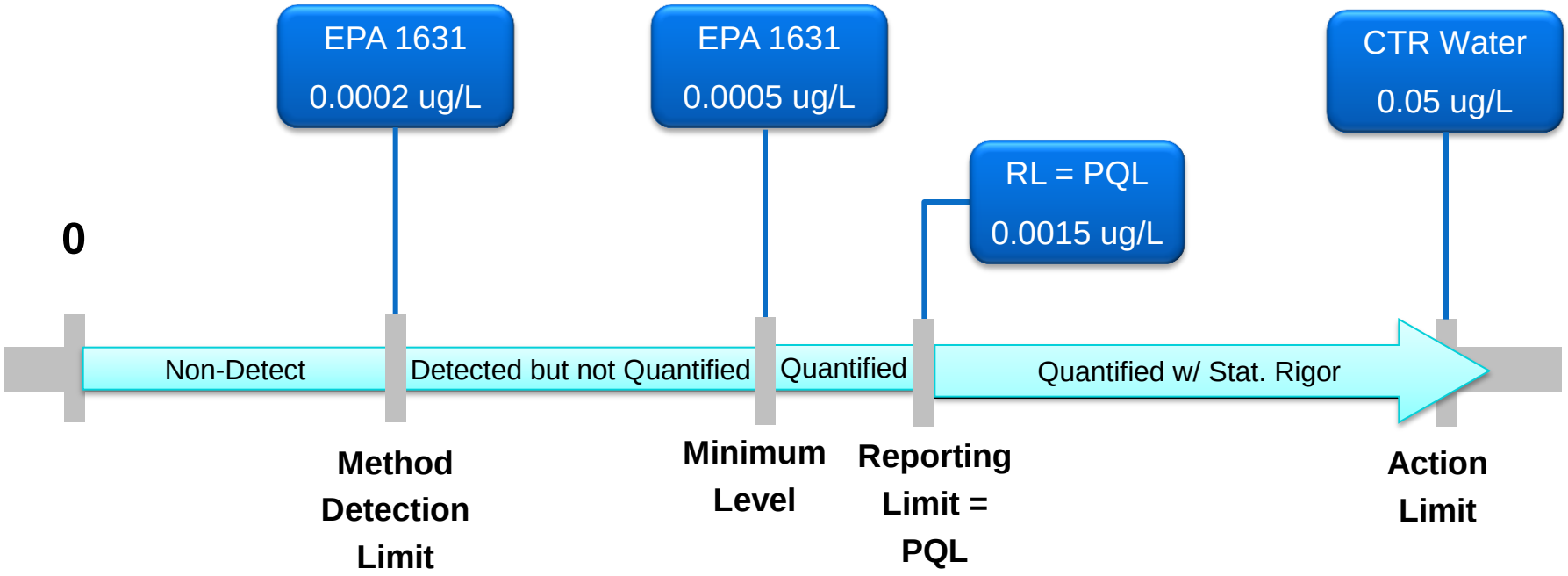
Reporting Limits in Databases and Reports

Reporting

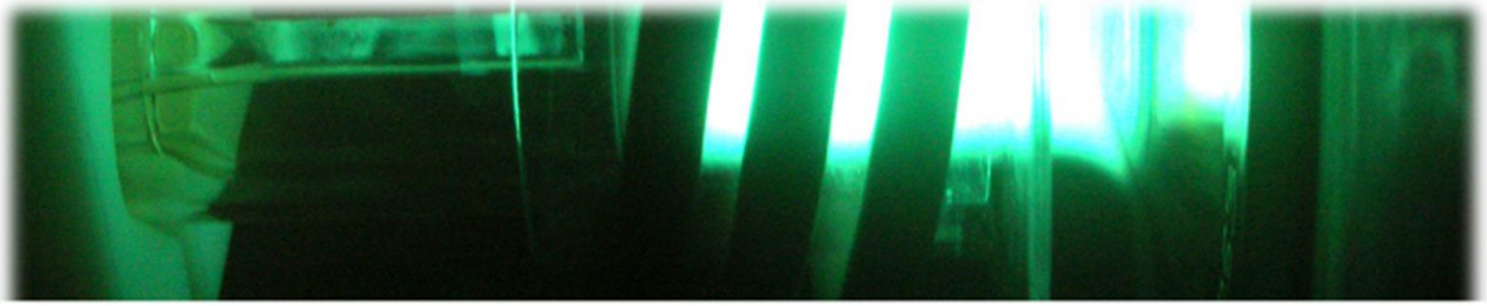
When reporting data, other terms worth understanding include:

- **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.



Concentration



NATIONAL FUNCTIONAL GUIDELINES

for Inorganic Superfund Data Review

EPA

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

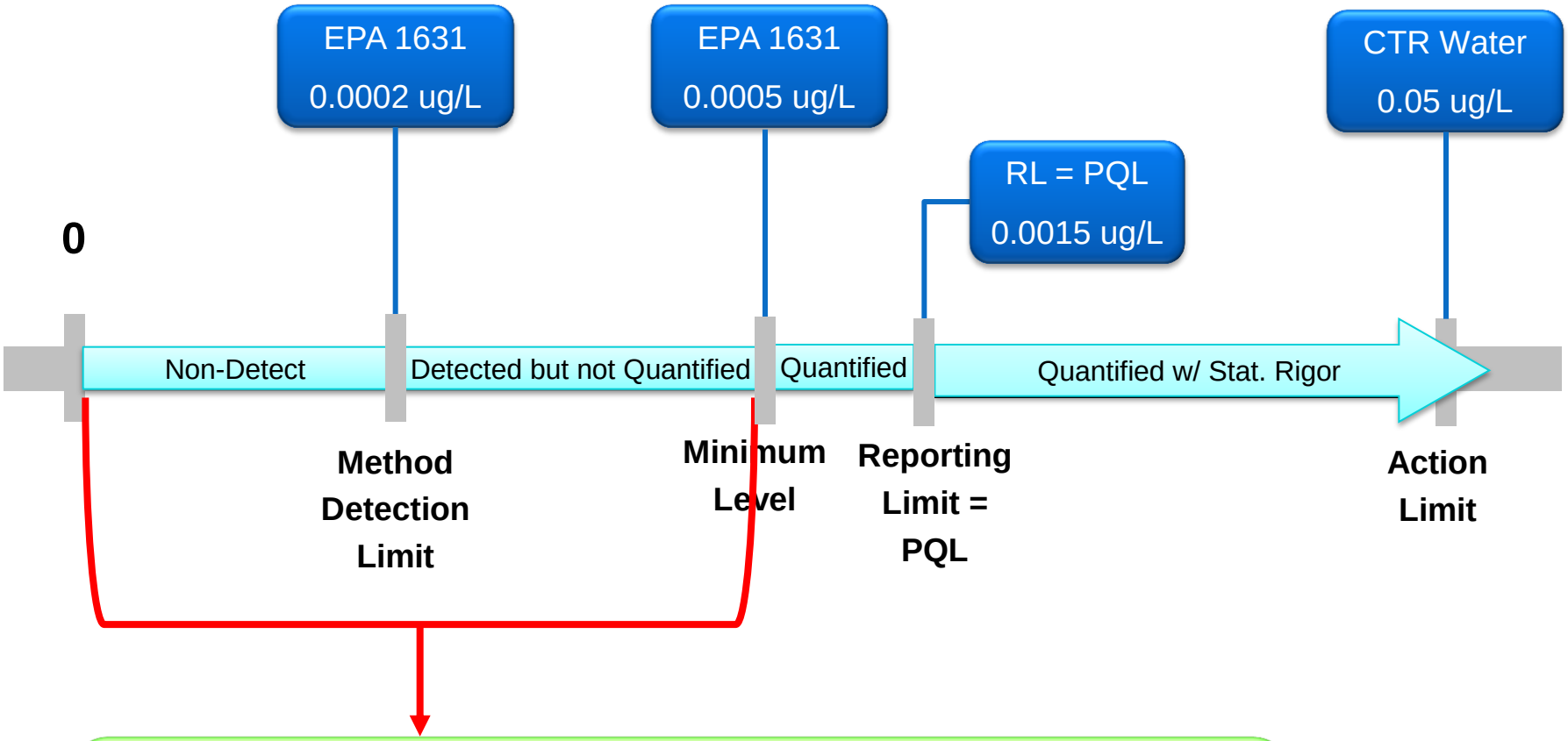
MPSL

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 1. Data Qualifiers and Definitions

Data Qualifier	Definition
U	The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.
R	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.



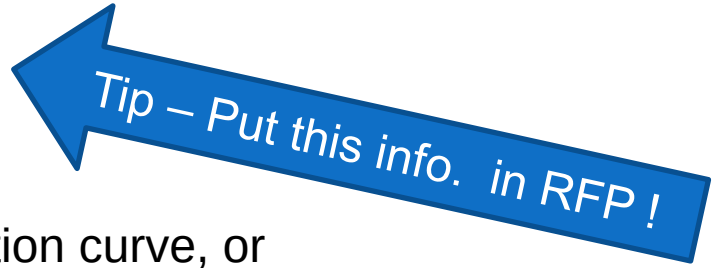
National Functional Guidelines Flags – U, J, UJ

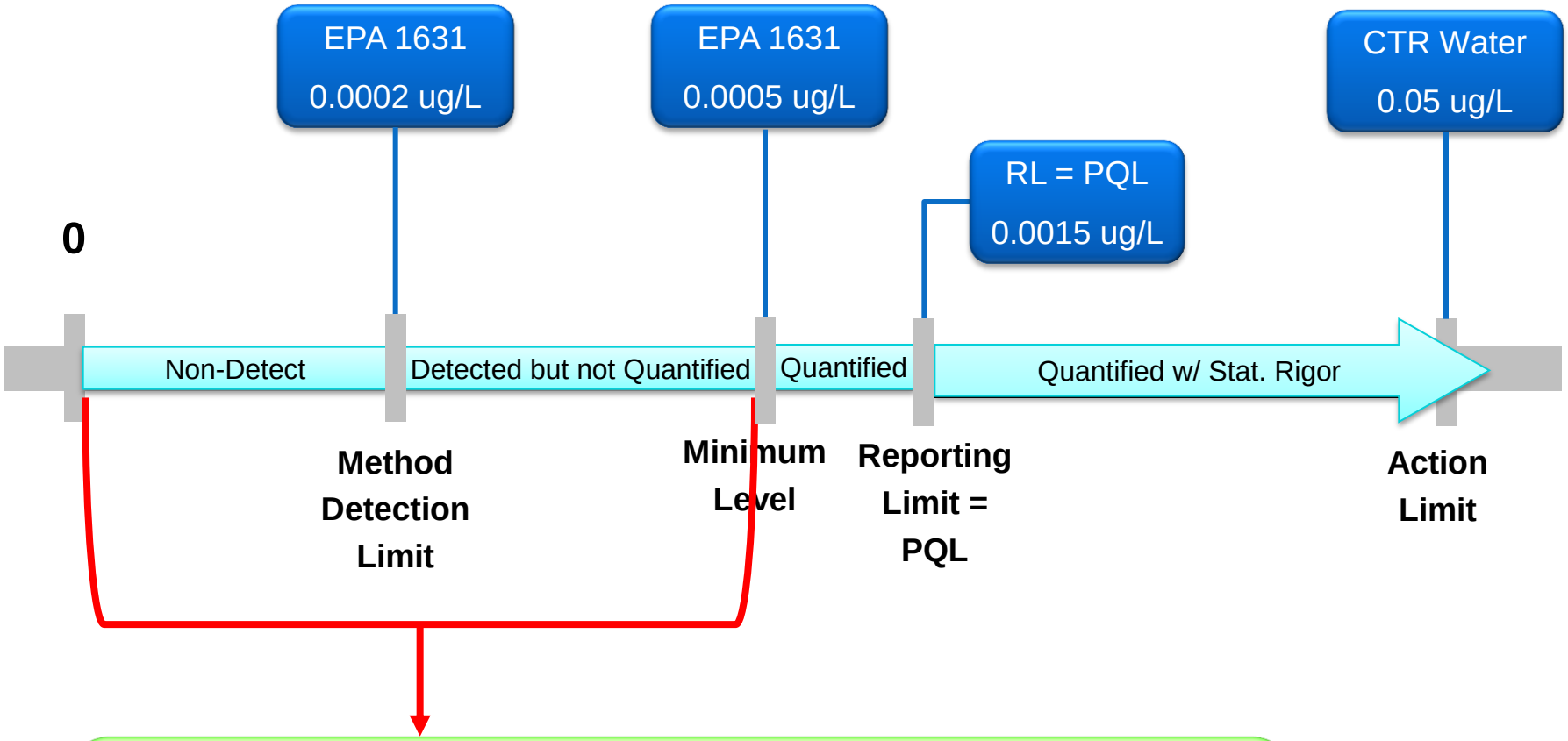
Other Laboratory Flags – ND, DNQ

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)





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Tip – Put this info. in RFP !

Reporting Limit as a term

Documents for Communicating Reporting Limits

Documents

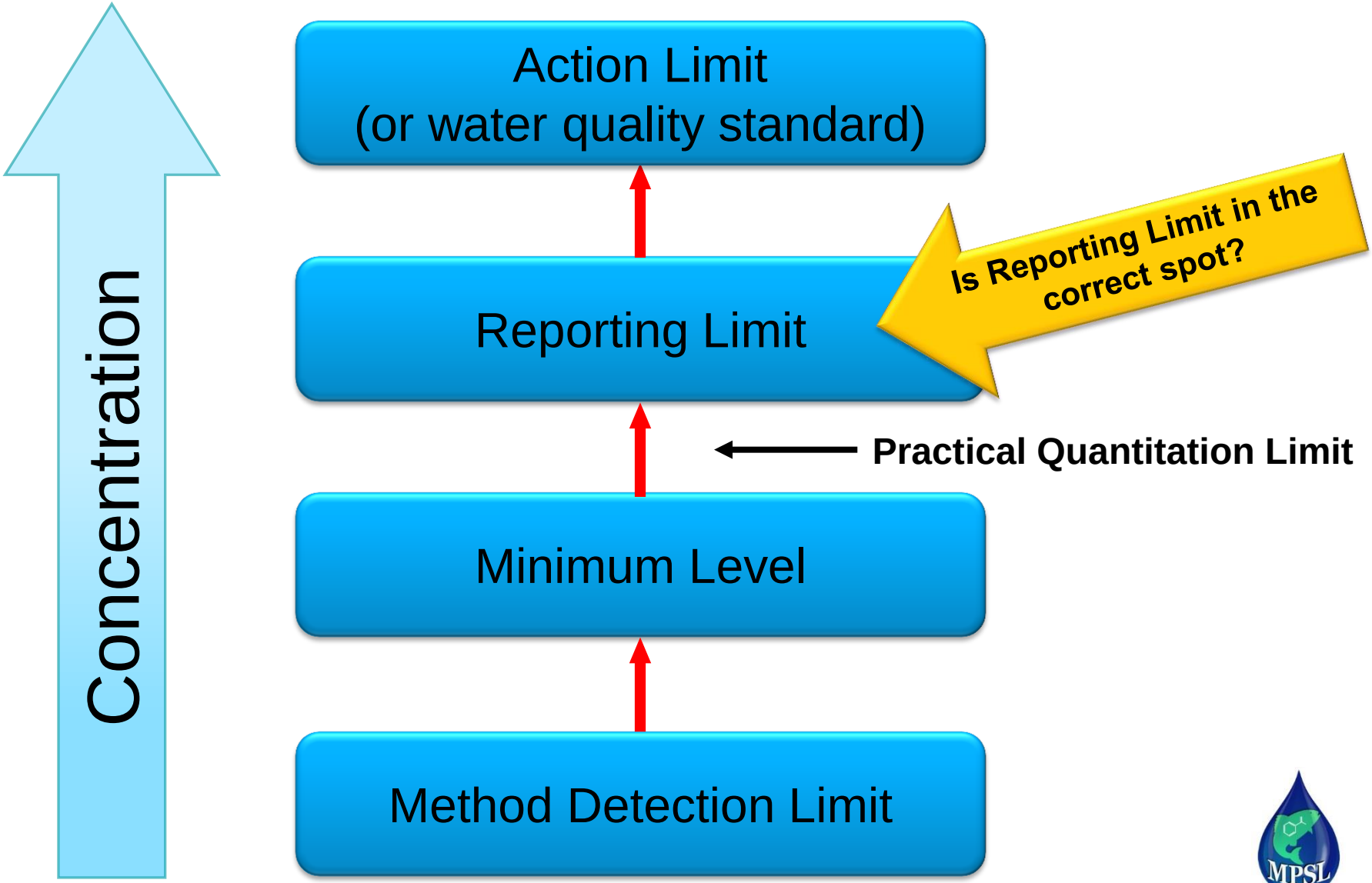
- Request for Proposals (RFPs) ← Tip – Have QA staff review
- QA Project Plans
- QA Manuals or other program documents
- Data Qualification/Flagging Manuals (or procedures)
- Permits
- Contracts ← Tip – Have QA staff review

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
Action Limit
(or water quality standard)
Reporting Limit
Is Reporting Limit in the correct spot?
Practical Quantitation Limit
Minimum Level
Method Detection Limit

Transparent

Accountable

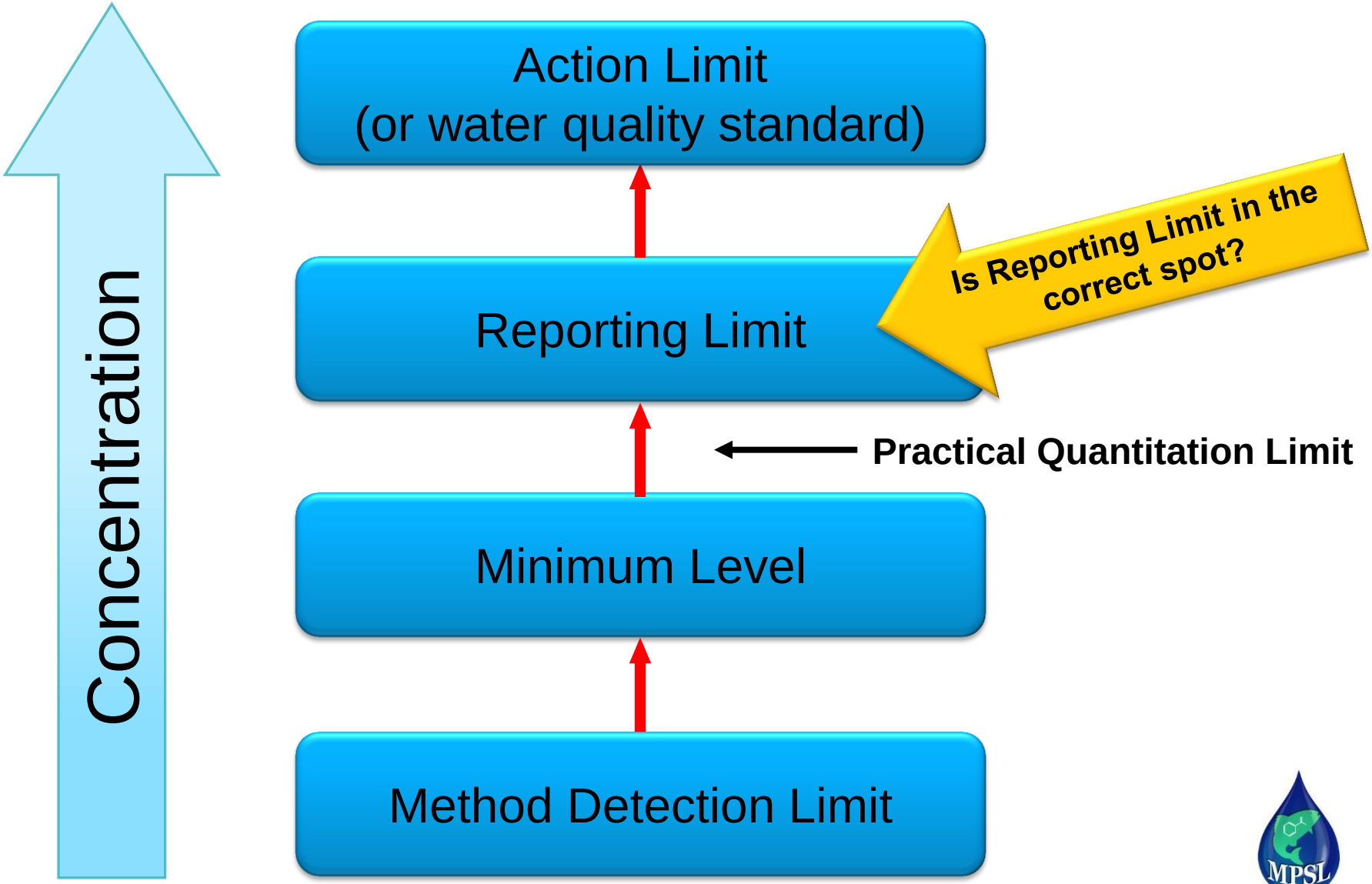
Scientifically Defensible

QUALITY ASSURANCE COMPONENTS
Measurable Metrics

Defensible

Comparable

Usable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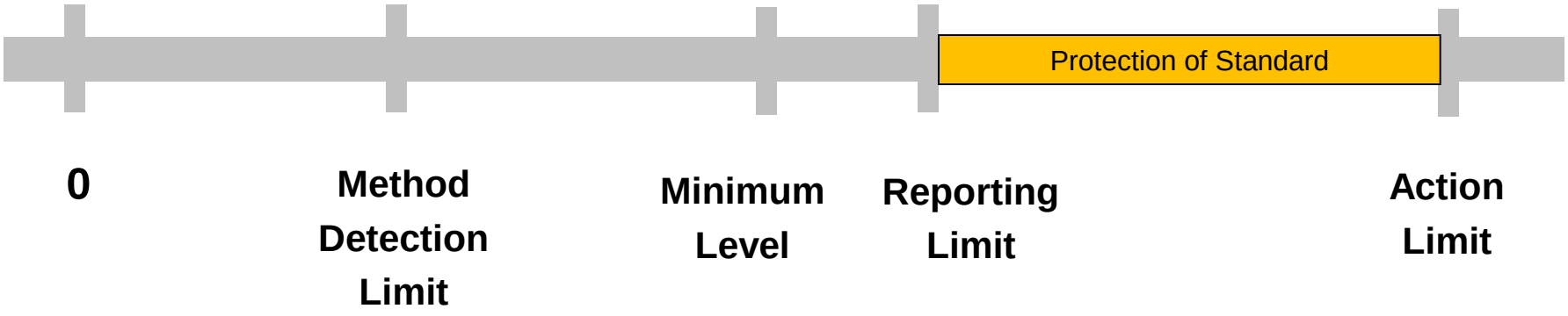
Why Reporting Limits Matter

Why Reporting Limits Matter

Reporting limits must be protective of our water quality standards



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