




The New EPA MDL Procedure


An Update to the 40 CFR Part 136
Method Detection Limit
Procedure






AGENDA


- Background
- Initial MDL Study
 - Practical Exercises
- BREAK
- On-going Verification
 - Practical Exercises
- FAQ
- Minimum Level and LOQ
- Drinking Water and other matrices






Why Do We Care?

- Comply with Part 136
 - Wastewater only
- Comply with NELAP (2016 Standard)
- Be able to defend your laboratory's results to regulators and customers both
 - Legally
 - Scientifically
- Have confidence and pride in what you do




The New EPA MDL Procedure


 **Lloyd Currie's Fundamental Work**

Analytical Chemistry, 1968


Limits for Qualitative Detection and Quantitative Determination
Application to Radiochemistry
Lloyd A. Currie
Analytical Chemistry, Volume 1, Number 1, February, 1968


- **Critical Level (L_C)**
 - Result indicates detection (*I'm sure it's there*)
- **Detection Limit (L_D)**
 - Procedure may be relied upon to lead to detection (*I'm sure I could have found it if it was there*)
- **Determination Level (L_Q)**
 - Procedure sufficiently precise to yield quantitative result (*I know how much is there*)
- Procedure: Standard deviation from blank measurements (i.e., precision-only)




 **Changes from 1968 to 1984**

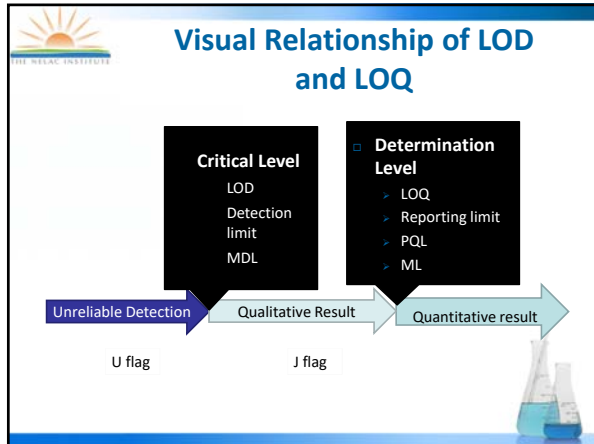
- **Critical Level** became internationally known as **Limit of Detection (LOD)**
 - Calculation revised to include mean of blank results
 - Method Detection Limit (MDL) established as one type of LOD
- **Determination Level** became internationally known as **Limit of Quantitation (LOQ)**
 - PQL, Reporting Limit are sometimes considered LOQs
- Currie's **Detection Limit (L_D)** seldom used

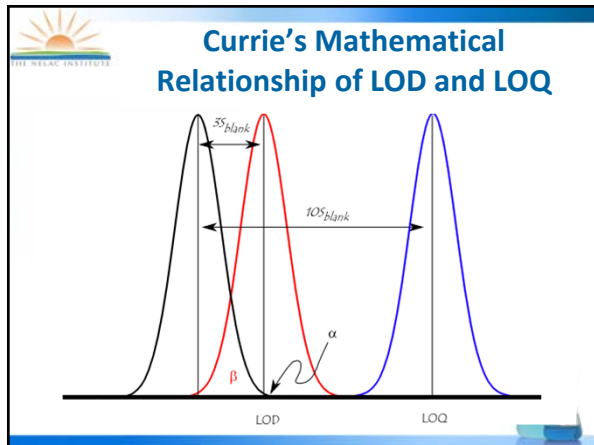


 **What Happened After 1984?**

- 1984 to 2000
 - Many articles, comments and lawsuits about problems with the MDL
 - MDL is widely used (US)
 - Most US labs run MDL studies
 - Most labs have little confidence in their calculated MDL
- 2000
 - Settlement Agreement
- 2003
 - EPA proposes MDL revision
- 2004
 - EPA withdraws MDL proposal
 - Federal Advisory Committee working on consensus procedure
- 2011
 - TNI forms Expert Committee



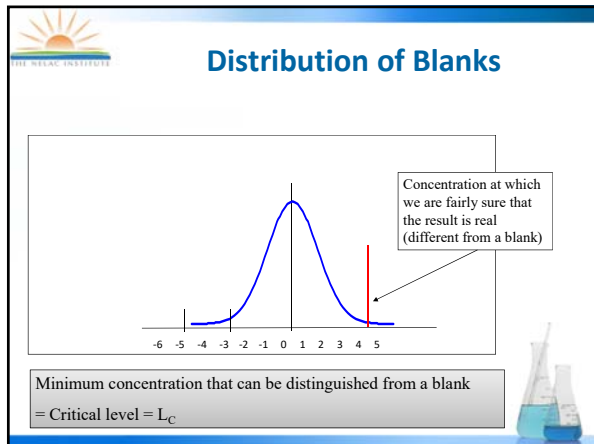




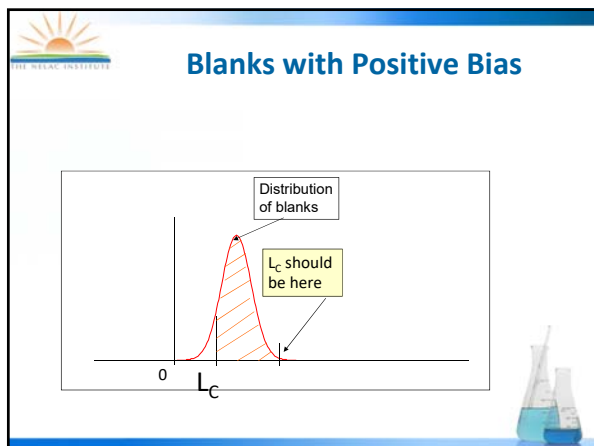
Critical Level = LOD = MDL

- If we detect it, it is in the sample
- Or: We know that the result is different from a blank
- This is L_C , the standard deviation of the blank times a constant directly related to sample size and the confidence level desired (normally 99%).
- Mathematically: $L_C = s * t$
- Assumes blanks normally distributed around 0


Note: Six degrees of freedom is the minimum number for any statistical confidence; requires 7 observations, student t value is 3.14 at 99%.



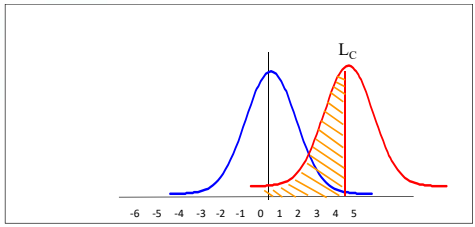
-
- Blanks with Positive Bias**
- L_C assumes normal distribution around 0
 - Values are biased low if the blank mean concentration (X_B) is greater than 0
 - IUPAC revised Currie's equation to account for this:
 - $LOD = (s * t) + X_B$
- The slide features a blue header with the NELAC Institute logo and a small image of laboratory glassware in the bottom right corner.




The New EPA MDL Procedure



False Negatives at Critical Level





True concentration at L_c results in false negatives 50% of the time!



The Old EPA MDL


- Changed “blanks” to “0”
- Changed “blank results” to “low-level spike results”
- $MDL = (s_{spikes} * t)$
- Published 1981 (ES&T)






The New EPA MDL


- Published August 28, 2017
 - 82 FR 40836
- Effective September 27, 2017
 - Effective in Virginia on 1/1/2019
- Based on work by TNI’s Chemistry Committee






A New Approach


- Go back to Lloyd Currie's original concepts
- Respect the old Part 136 procedure, but address problems
 - Blank contamination
 - Long-term precision
 - Actual detectability
- Ensure the procedure is cost-effective and implementable for all types of laboratories






Fundamentals Stay the Same


- Definition has the same intent
 - What is the lowest result that is qualitatively reliable, i.e., the lowest result that reliably indicates the analyte is in the sample?
- Calculation is unchanged
 - Calculate the MDL as Student's t times the standard deviation of results
- Incorporate entire analytical process, including sample preparation






New Elements in the Procedure


- Include data from both low-level spikes and method blanks
- Analyze spikes and blanks over multiple days
- Procedure for multiple instruments
- Include criteria for evaluating false positives in blanks
- Include criteria for evaluating qualitative identification
- Include Mean and SD of blanks in calculation
- Continually generate data and evaluate






The New Procedure


- Definition
- Scope and Application
 - Exceptions
- Procedure
 1. Estimate the initial MDL
 2. Determine the initial MDL
 3. Ongoing Data Collection
 4. Ongoing Annual Verification
- Addendum: MDL for a Specific Matrix
- Documentation






Slight Change in Definition


- 1984: The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is **greater than zero** and is determined from analysis of a sample in a given matrix containing the analyte.
- 2016: The minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is **distinguishable from method blank** results.







Exceptions


- Not applicable to methods that do not produce results with a continuous distribution such as:
 - Whole effluent toxicity
 - Presence/Absence methods
 - Micro methods that count colonies
- Not applicable to tests that cannot be spiked, or do not yield blank results, or are impractical
 - e.g., pH, color, odor, conductance, dissolved oxygen, BOD, many titration methods
- MDL determinations using spiked samples may not be appropriate for all gravimetric methods (e.g., residue or total suspended solids), but an MDL based on blanks can be determined in such instances.




 **Four Step Procedure**


1. Estimate an initial MDL
2. Determine the initial MDL Initial
3. Ongoing data collection On-going
4. Ongoing annual verification

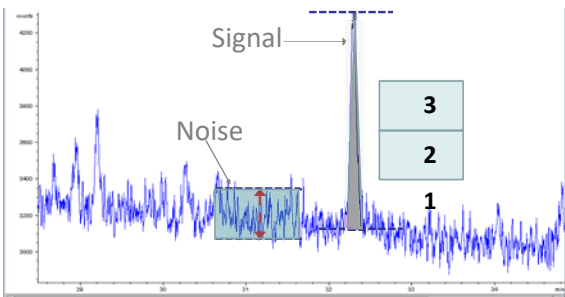


 **1. Estimate an Initial MDL**


- a. The mean plus three times the standard deviation of a set of method blanks, **OR**
- b. The concentration value that corresponds to an instrument signal/noise in the range of 3 to 5, **OR**
- c. Three times the standard deviation of spiked blanks, **OR**
- d. That region of the standard curve where there is a significant change in sensitivity, **OR**
- e. Instrumental limitations, **OR**
- f. Previously determined MDL.



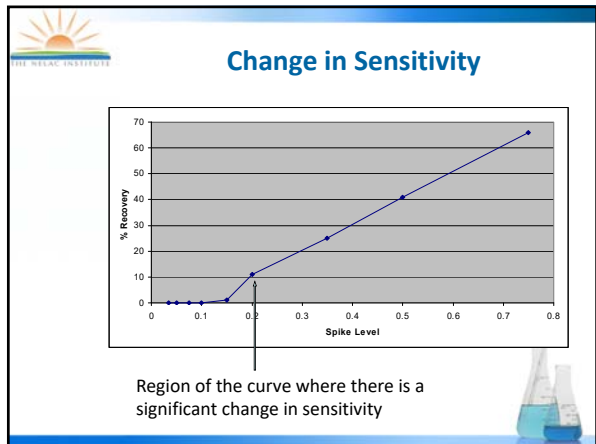
 **Defining Signal to Noise**

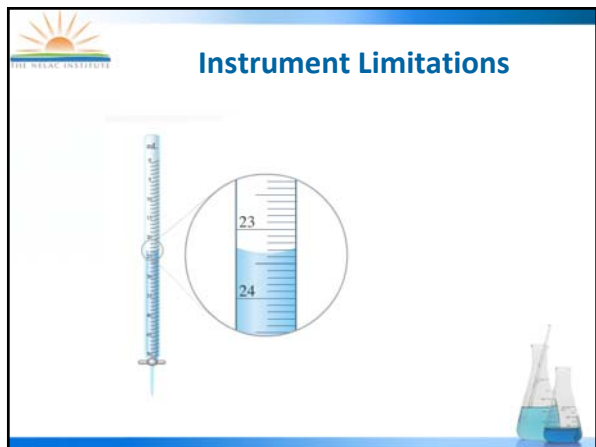



<https://www.agilent.com/cs/library/technicaloverviews/Public/5990-7651EN.pdf>




The New EPA MDL Procedure






- 
- ## 2. Determine the Initial MDL
- Select a spiking level, typically 2 – 10 times the estimated MDL. *
 - Analyze a minimum of 7 spikes and 7 blanks.
 - Include at least three batches on three separate days.
 - Existing data may be used if generated within the last 2 years.
 - Samples must be distributed across all of the instruments.
 - A minimum of two spikes and two blanks on different days for each instrument.
 - The same extract may be analyzed on different instruments
- * Spiking levels in excess of 10 times the estimated detection limit may be required for analytes with poor recovery




Determine the Initial DL (TNI)

- Select a spiking level, typically 2 – 10 times the estimated MDL.
- Analyze a minimum of 7 spikes and 7 blanks.
- Include **multiple** batches analyzed on **multiple** days.
- Existing data may be used if generated within the last 2 years.
- Samples must be distributed across all of the instruments.
- A minimum of **one** spike and **one** blank on different days for each instrument.
- The same extract may be analyzed on different instruments

TNI: The [spike] must be at or above the lowest corresponding calibration standard concentration.





Example – Single Instrument

Monday Tuesday Wednesday


Extraction Batch


Analysis Batch

Tuesday Wednesday Thursday

OR

Run a method blank with each spike, unless you have the data!







Multiple (4) Instruments

Tuesday Tuesday Wednesday Wednesday

Wednesday Wednesday Thursday Thursday


Run a method blank with each spike, unless you have the data!




 **2c. Evaluate the Spike Results and Calculate MDL_s**


- Statistical outlier removal not allowed, but “documented instances of gross failures” may be excluded as long as 7 spike and 7 blank results are available
- If any result from the spiked samples does not meet the qualitative identification criteria* or does not provide a result greater than zero then repeat the spikes at a higher concentration.
- $MDL_s = tS_s$ of spike results
 - $=STDEV(A1:Ax)*t$


* A set of rules or guidelines for establishing the identification or presence of an analyte. Qualitative identification does not ensure that quantitative results can be obtained.




 **EPA FAQ: What happens if the laboratory has less than 7 sample spikes when calculating the MDL?**


- The minimum number of samples is 7. If the analysis is performed regularly, then there will likely be 16 spiked samples per instrument (2 per quarter over 2 years) and many more blanks.
- If the analysis is performed very rarely, then there may be less than 7. In this case, the laboratory needs to perform a new initial MDL procedure, but can use the samples that are available over the last 2 years to contribute to calculating the new initial MDL.



 **EPA FAQ: Will laboratories have to analyze more samples for methods that are rarely used?**


- No, the MDL procedure could potentially require fewer samples for rarely used methods. For example, if a laboratory analyzed 7 batches of samples spread out over a 2-year period then the laboratory would have enough sample spikes and blanks to recalculate the MDL. This would be half of what was normally done year.






EPA FAQ: Why are acceptable calibrations and batch QC not mentioned?

- If the laboratory is performing an initial MDL without client samples, most batch QC is not required.
- The spiked samples are essentially laboratory fortified blanks, and the MS/MSD are not required if there are no client samples.
- Ongoing MDL samples should be analyzed with client samples, so all normal batch QC should be present.
- The methods already specify that calibrations must be completed before performing any analyses, so there is no need to add this requirement to the MDL procedure itself.







Evaluate Blank Results and Calculate MDL_b

- $MDL_b = X + tS_0$ if all blanks have numerical results*
 - $= (STDEV(A1:Ax) * t) + AVERAGE(A1:Ax)$
- $MDL_b =$ Not applicable if all results are ND
- $MDL_b =$ Highest blank result if some but not all blanks have numerical results*
- $MDL_b =$ 99th Percentile if >100 results are available
 - $= PERCENTILE(A1:Ax, 0.99)$

* A numerical result includes both positive and negative results, but not results of "ND" (not detected) commonly observed when a peak is not present in chromatographic analysis.







99th Percentile

- For "n" blank results where n > 100, sort the blanks in rank order
- Calculate $n * 0.99$ and select that value, or
 - $= PERCENTILE(A1:A164, 0.99)$


Example □ 159 blank results < 1.5 □ 160 = 1.5 □ 161 = 1.7 □ 162 = 1.9 □ 163 = 5.0 □ 164 = 10.0	$164 * 0.99 = 162.36$ 99 th percentile = 1.9
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EPA FAQ: Could a high blank drastically elevate the MDL?


- It depends – a method blank can be ignored if it is associated with an instance of gross failure.
- A lab might have over a hundred blanks over a two year period and then can use the 99th percentile option.
- There is also an option to use the most recent 50 blanks or last six months of data, whichever yields the greater number of blanks.






The Initial MDL


- MDL = Greater of MDL_b or MDL_s

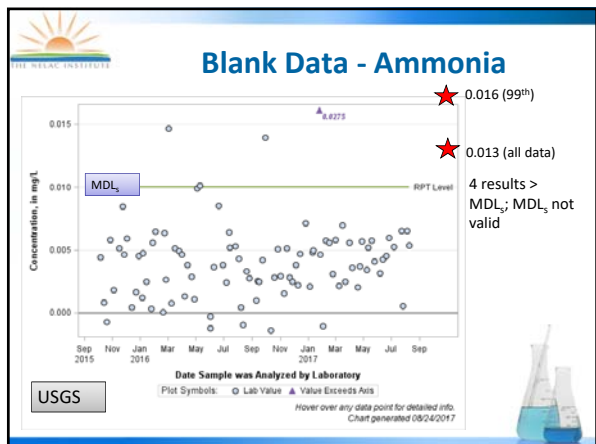


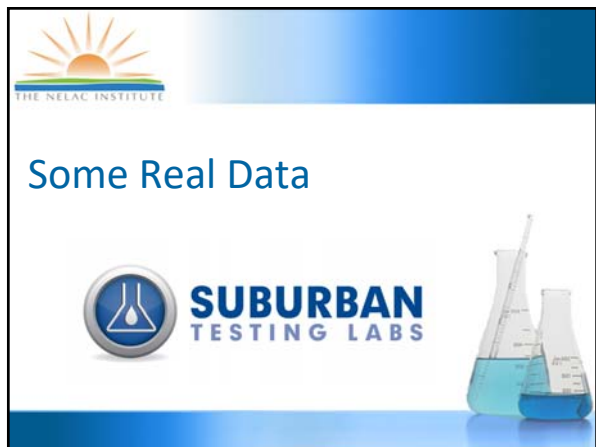


Let's Calculate Some MDLs

- Ammonia
 - > MDL_b ($X + tS_b$)
 - > MDL_b (99th percentile)
- Phosphorous
 - > MDL_s
 - > MDL_b
- Benzene
 - > MDL_s
- Your Data








MDL₅ - Phosphorous

Batch	Extracted	Analyzed	SampleID	Instrument	By	Analyst	Spike	Result	%R
B7H1623	8/22/2017	8/24/2017	B7H1623	FIA-02	UR	RCE	0.02	0.021	105%
B7H1624	8/22/2017	8/24/2017	B7H1624	FIA-02	UR	RCE	0.02	0.023	115%
B7H1687	8/23/2017	8/24/2017	B7H1687	FIA-02	TML	RCE	0.02	0.02	100%
B7H1827	8/24/2017	8/30/2017	B7H1827	FIA-02	UR	RCE	0.02	0.021	105%
B7H1881	8/24/2017	8/30/2017	B7H1881	FIA-02	UR	RCE	0.02	0.021	105%
B7H2076	8/28/2017	9/1/2017	B7H2076	FIA-02	UR	RCE	0.02	0.021	105%
B7H2086	8/28/2017	9/1/2017	B7H2086	FIA-02	UR	RCE	0.02	0.016	80%


Note: The procedure has no criteria for percent recovery, only that the recovery be documented. QC criteria for LFBs may not be appropriate since LFB spikes are generally at a mid-point concentration. The laboratory should however review the recovery and investigate any recoveries that appear to be unusually low, as this may indicate a problem with the method.


The New EPA MDL Procedure



MDL_b - Phosphorous

Batch	Extracted	Analyzed	SampleID	Instrument	Prep By	Analyst	Result
B7H1623	8/22/2017	8/24/2017	B7H1623-BLK1	FIA-02	UR	RCE	-0.003
B7H1624	8/22/2017	8/24/2017	B7H1624-BLK1	FIA-02	UR	RCE	-0.007
B7H1687	8/23/2017	8/24/2017	B7H1687-BLK1	FIA-02	TML	RCE	-0.002
B7H1827	8/24/2017	8/30/2017	B7H1827-BLK1	FIA-02	UR	RCE	0.005
B7H1881	8/24/2017	8/30/2017	B7H1881-BLK1	FIA-02	UR	RCE	0.006
B7H2076	8/28/2017	9/1/2017	B7H2076-BLK1	FIA-02	UR	RCE	-0.018
B7H2086	8/28/2017	9/1/2017	B7H2086-BLK1	FIA-02	UR	RCE	-0.019







Phosphorous MDL

MDL _s		MDL _b	
Average Result	0.020	Mean	-0.00543
StdDev	0.002	StdDev	0.0100
Avg Recovery	102%	MDL _b	0.031
MDL _s	0.007		

Since the mean is less than 0, substitute 0 for the mean
 $MDL_b = 0 + (.01 * 3.143)$


99th Percentile of 149 blanks = 0.020






Organics – Benzene by 624

Batch	Analyzed	SampleID	Instrument	Analyst	Spike	Result	%R
B7G1395	21-Jul-17	B7G1395	GCMS-08	KED	0.50	0.57	114%
B7G1745	26-Jul-17	B7G1745	GCMS-08	MWS	0.50	0.53	106%
B7F1327	20-Jun-17	B7F1327	GCMS-04	CEM	0.50	0.51	102%
B7F1514	22-Jun-17	B7F1514	GCMS-04	CEM	0.50	0.53	106%
B7F1610	23-Jun-17	B7F1610	GCMS-04	CEM	0.50	0.54	108%
B7E1368	20-May-17	B7E1368	GCMS-06	CEM	0.50	0.48	96%
B7F0843	13-Jun-17	B7F0843	GCMS-06	CEM	0.50	0.54	108%






BTEX By 624


MDL_s

	Benzene	Toluene	Ethyl	
			Benzene	Xylenes
Average Result	0.529	0.514	0.449	1.264
StdDev	0.028	0.045	0.067	0.280
Avg Recovery	106%	102%	90%	84%
MDLs	0.088	0.142	0.210	0.880

MDL_b


	Benzene	Toluene	Ethyl	
			Benzene	Xylenes
Numeric Results	0.00	0.00	0.00	0.00
Mean	0.00	0.00	0.00	0.00
StdDev	0.00	0.00	0.00	0.00
MDLb	NA	NA	NA	NA







EPA FAQ: If the MDLs change, permit limits may need to be reviewed.


- The new MDL procedure may cause some additional contaminants to have MLs above the permit requirements for a specific analysis. The "Sufficiently Sensitive Method" rule is very clear about what to do in this case; see [40 CFR 122.21\(e\)\(3\)](#).
- Additionally, supporting documents are available in the docket: EPA-HQ-OW-2014-0797 (www.regulations.gov)






Take a Break









3. On-going Data Collection

- At least two spikes and two blanks on each instrument per quarter in separate batches (unless no samples are analyzed)
- At least 7 spikes per year*
- At least 7 blanks per year

* If only one instrument is used, the 7 spikes can be spread over 2 years


If more than 5% of the spikes do not return positive numerical results that meet all identification criteria, then the spiking level must be increased and the initial MDL re-determined






The 5 % Rule (One Instrument)


- Must evaluate once per year
- Can use 2 years of data
- 2 years of data
 - Year 1 – 7 spikes from initial + 6 spikes from 3 quarters = 13
+ 5% = 0.65 = 0 failures
 - Year 2 – 7 spikes from initial + 6 spikes from 3 quarters +
8 spikes from 4 quarters = 21
+ 5% = 1.05 = 1 failure allowed
 - Year 3 – 8 spikes from 4 quarters +
8 spikes from 4 quarters = 16
+ 5% = 0.8 = 0 failures allowed






Example – Single Instrument

Extraction Batch



Extraction Batch




Analysis Batch

Any day

Analysis Batch

Any other day

Run a method blank with each spike, unless you have the data!



Multiple (4) Instruments

Any 2 Days Any 2 Days Any 2 Days Any 2 Days


Run a method blank with each spike, unless you have the data!

Additional Requirement 1

- If the method is altered in a way that can be *reasonably expected* to change its sensitivity, then re-determine the initial MDL, and the restart the ongoing data collection.


Additional Requirement 2


- If a new instrument is added to a group of instruments, analyze a minimum of two spiked replicates and two method blank replicates on the new instrument.
 1. If both method blank results are below the existing MDL, then the existing MDL_g is validated.
- Combine the new spiked sample results with the existing spiked sample results and recalculate the MDL_s.
- 2. If the recalculated MDL_s is within 0.5 to 2.0 times the existing MDL_g, then the existing MDL_g is validated. If either of these two conditions is not met, then calculate a new MDL.



EPA FAQ: If the laboratory does not use a method during a quarter, will the laboratory still need to analyze low-level spiked samples?


- No, the laboratory needs to analyze at least seven low-level spiked samples and seven method blanks for one instrument in a two-year period (spread over 3 batches), but is also supposed to analyze two spiked samples per quarter in separate batches any quarter samples are analyzed.






A Simple Way to Do This


- Analyze a low-level spiked sample with the first two analytical batches every quarter.
- If no samples are analyzed, then there is no need to analyze spiked samples or method blanks.
- If one batch of samples is analyzed during a quarter, then the laboratory should include one low-level spiked sample in that batch.
- If two or more batches of samples are analyzed, the laboratory should include one low-level spiked sample in at least two of those batches.






EPA FAQ: What if a laboratory buys a new instrument and wants to include it in a multi-instrument MDL?

- The laboratory needs to analyze a minimum of two spiked samples and two method blanks on the new instrument.







4. Annual Verification

- Every 13 months, recalculate MDL₀ and MDL₃ from collected blank and spike results.
- Include all data over a two-year period, but exclude any data with failed batch QC or other gross failures.
- Ideally, use all blanks, but as an option, you may use 6 months of blanks or the 50 most recent, whichever is greater.

If the verified MDL is within a factor of 0.5 to 2.0 of the existing MDL, and fewer than 3% of the method blank results have numerical results above the existing MDL, then the existing MDL may be left unchanged. Otherwise, adjust the MDL to the new verification MDL.







EPA FAQ: Is the lab required to recalculate the MDL every quarter?

- No, the MDL is only calculated once a year. MDL spiked samples are now analyzed every quarter in which the method is used, but the calculation is only required to be performed once a year.


59






EPA FAQ: Why is so much ongoing data collection necessary, and what additional quality is this providing?


- Ongoing data collection captures instrument drift and the variation in equipment conditions throughout the year.
- Many laboratories currently analyze the MDL aliquots immediately after the instrument is serviced and all consumable instrument parts are new, thus yielding a best-case MDL value.
- Ongoing data collection leads to an MDL that represents what is actually practiced throughout the year.






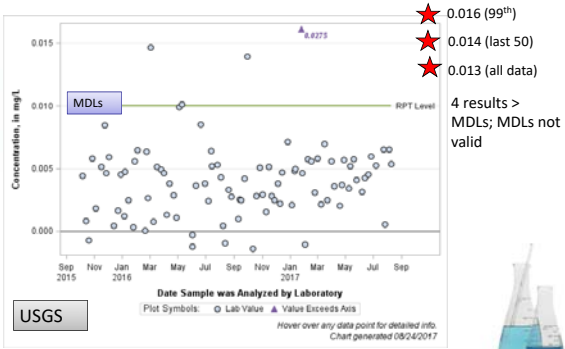
Let's Verify Some MDLs

- Ammonia
 - $MDL_b (X + tS_b)$
 - MDL_b (99th percentile)
 - MDL_b (last 50) or MDL_b (last six months)
- Acrolein
 - MDL_s
- Your Data





Blank Data - Ammonia





★ 0.016 (99th)
★ 0.014 (last 50)
★ 0.013 (all data)

4 results > MDLs; MDLs not valid

USGS

Plot Symbols: ○ Lab Value ▲ Value Exceeds Axis
 Hover over any data point for detailed info.
 Chart generated 08/24/2017







Acrolein Initial MDL_s

Analyte	Spike Level	R1	R2	R3	R4	R5	R6	R7	R6	SD	MDLs
Date analyzed		9/1/17	9/2/17	9/1/17	9/2/17	9/3/17	9/4/17	9/3/17	9/4/17		
Instrument ID		A	A	B	B	C	C	D	D		
Acrolein	10	8.1	8.2	11	12	9.3	9.5	9.1	9.3	1.3	4.0

$MDL = SD * 2.998 (n=8, df=7)$







Initial and Ongoing Spikes

Analyte	Spike	R1	R2	R1	R2	R1	R2	R1	R2	SD	MDLs
Instrument ID		A	A	B	B	C	C	D	D		
Date analyzed		9/1/2017	9/2/2017	9/1/2017	9/2/2017	9/3/2017	9/3/2017	9/4/2017	9/3/2017	9/4/2017	
Acrolein	10	8.1	8.2	11	12	9.3	9.5	12	11.9		
		12/2/2017	12/2/2017	12/3/2017	12/3/2017	12/4/2017	12/4/2017	12/4/2017	12/4/2017		
Acrolein	10	8	8.3	10.5	10.7	8.4	8.7	8.2	8.3		
		3/4/2018	3/5/2018	3/4/2018	3/5/2018	3/5/2018	3/6/2018	3/5/2018	3/6/2018		
Acrolein	10	8.5	8.7	11.2	11.5	9.5	9.7	9	9.4		
		6/4/2018	6/5/2018	6/4/2018	6/5/2018	6/5/2018	6/6/2018	6/5/2018	6/6/2018		
Acrolein	10	11	10.8	9	8.8	8.5	8.7	10.6	10.2	1.290	3.2

MDL = SD * 2.453 (n=32, df=31)
 3.2/4.0 = 0.76
 MDL is verified and may be changed or not







Documentation

- Data and calculations used
- Sample matrix
- Spike value and recovery
- Rationale for removal of outliers


Not stated in procedure, but also need date of analysis and instrument ID.
 Analyst name not required.






NEW Determine MDL in a Specific Matrix

Analyze the sample. If the response for the native concentration is at a signal-to-noise ratio of approximately 5-20, determine the matrix-specific MDL according to Section 2 but without spiking additional analyte.






10 X Rule is Gone

OLD

If the level of analyte exceeds 10 times the MDL of the analyte in reagent water, do not report a value for the MDL.

Intended to be used to establish MDLs in wastewater matrices where the analyte was present, but incorrectly used to evaluate low-level spikes.








Table 1: Student t Values

- As published, missing many values
- <http://www.itl.nist.gov/div898/handbook/eda/section3/eda3672.htm>
- Use the values in the 99% column. This particular table shows degrees of freedom, which is the number of replicates minus one. An easy way to check is to look at the student t for 7 (6 degrees of freedom) which is 3.143.


	1.00	0.50	0.20	0.10	0.05	0.025	0.01	0.005	0.001	0.0005
1	0.000	0.758	0.906	1.134	1.440	1.943	2.447	3.143	3.707	5.208
2	0.000	0.711	0.856	1.119	1.415	1.855	2.355	2.999	3.499	4.765
3	0.000	0.706	0.849	1.108	1.397	1.860	2.305	2.896	3.355	4.501
4	0.000	0.703	0.843	1.103	1.383	1.853	2.292	2.871	3.320	4.471
5	0.000	0.700	0.839	1.093	1.372	1.812	2.278	2.784	3.169	4.144
6	0.000	0.698	0.836	1.088	1.367	1.808	2.272	2.778	3.162	4.138
7	0.000	0.697	0.835	1.088	1.367	1.808	2.272	2.778	3.162	4.138
8	0.000	0.696	0.834	1.087	1.366	1.807	2.271	2.777	3.161	4.137
9	0.000	0.696	0.834	1.087	1.366	1.807	2.271	2.777	3.161	4.137
10	0.000	0.695	0.833	1.086	1.365	1.806	2.270	2.776	3.160	4.136






The MDL and ML in EPA


- Minimum level (ML) – The term “minimum level” refers to either the sample concentration equivalent to the lowest calibration point in a method or a multiple of the method detection limit (MDL), whichever is higher.
- Minimum levels may be obtained in several ways:
 - they may be published in a method;
 - they may be based on the lowest acceptable calibration point used by a laboratory; or
 - they may be calculated by multiplying the MDL in a method, or the MDL determined by a laboratory, by a factor of 3.
- For the purposes of NPDES compliance monitoring, EPA considers the following terms to be synonymous: “quantitation limit,” “reporting limit,” and “minimum level.”






DL and LOQ in TNI


- Designed to work in harmony
- One study, spike at LOQ
 - Used to verify LOQ
 - Used to calculate DL
- New Guidance document provides many examples






Tip


- Run your low-level spikes at your reporting limit
- This should result in an MDL about 1/3 your reporting limit
- Spikes will be at a level they should be detected 100% of time
- This approach also in compliance with the 2016 TNI standard






MDLs and Other EPA Programs


- Drinking Water
 - OGWDW Memo (See next few slides)
- RCRA
 - Uses LOQ concept; MDL not required but allowed
- Air
 - Many air methods cite Part 136






OGWDW Memo

- If a laboratory practices good hygiene by keeping their laboratory clean (i.e. sample prep areas, glassware, instrumentation, etc.), the method blanks should never indicate a recurring background as nearly all blank failures would invalidate analytical results.
- MDL specifically cited in Part 141 for some regulated analytes
- Part 136 specifically referenced in some EPA methods, but others reference the original publication
 - It becomes a judgement call. Just be consistent in applying such judgement across the region.
- **From the standpoint of conducting drinking water analyses, the MDL_s should not be the higher value.** If it is, that's a sure sign the lab needs to take corrective action.







OGWDW Memo (Cont)

- For some drinking water contaminants, qualification for reduced monitoring is based on specified low threshold levels. In order for a laboratory to meet those levels, they will need to optimize *lower* detection levels. Pooling data from multiple instruments will have the net effect of increasing variability, resulting in *higher* calculated MDL values.
- This specification of determining the MDL per method and per instrument precludes the option of determining a multi-instrument MDL for instruments that will be used to analyze drinking water.


An initial demonstration of capability (IDC) must be performed for each method. The IDC includes a determination of MDL. An IDC should be performed for each instrument. It is also recommended that an IDC be performed by each analyst. In addition, it is recommended that the IDC also address the variability introduced if more than one sample preparation technician is used. Precision, accuracy and MDL should be similar for each technician. (DW Cert Manual)






OGWDW Memo Impact


- Written as guidance (“should”)
- Subject to interpretation by states and EPA regions
- Virginia DCLS
 - When the new procedure is required for drinking water (i.e., when an approved DW method refers to 40CFR136 App B) we will assess to that requirement after January 1, 2019.
 - We have welcomed and encouraged laboratories to use the new procedure for all MDLs.
 - Silent on MDL_s > MDL_s issue
 - Silent on single instrument issue






Implementation


- Start by evaluating existing blank data
 - Establish MDL_b where justified
- You can use existing spike data, if you meet the new requirements
- If not, start collecting spike and blank data (at least 2 per instrument) to supplement your existing dataset






Frequently asked Questions - EPA


- <https://www.epa.gov/cwa-methods/method-detection-limit-frequent-questions>






Frequently Asked Questions – Jerry Parr

- > 100 questions organized into 36 subjects, e.g.
 - **3.0 Data Evaluation and Calculation of the Initial MDL**
 - + 3.1 Negative Values for Blanks
 - + 3.2 Requirement for an MDL Study and Reporting to MDL
 - + 3.3 Qualitative Identification Criteria
 - + 3.5 Protection from False Negatives
 - + 3.6 Validity of the MDL
 - + 3.7 MDL for Sample Preparation
 - + 3.8 Data Analysis Software
 - + 3.9 Student t Numbers
 - + 3.10 QC Failures and Outlier Tests
- Included in TNI Guidance Document



The New EPA MDL Procedure



Thank You
TNI Chemistry Committee
2010-2015

Richard Burrows, Chair TestAmerica Laboratories	Dan Dickinson New York, DOH
Francoise Chauvin New York City DEP	Brooke Connor USGS
Anand Mudambi US EPA OSA	Tim Fitzpatrick Florida DEP
Nancy Grams Advanced Earth Technologists	Lee Wolf Columbia Analytical Services
Scott Siders Illinois EPA	John Phillips Ford Motor Company
Gary Ward Oregon DOH	Valerie Slaven
Colin Wright Florida DEP	Gale Warren New York, DOH

91 meetings

