TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Working with the New MDL

Richard Burrows, Ph.D. – Corporate Technical Director

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

March 17, 2015

A Revision to the Method Detection Limit

EPA published a revision to the 40 CFR Part 136 MDL procedure in the Federal Register on Thursday February 19th

This is a proposed rule with public comments due by April 20th

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

Major Differences Between the Old and New MDL

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

First, what stays the same?

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

- Fundamental concept is unchanged
  - What is the lowest result that is qualitatively reliable, i.e., the lowest result that reliably indicates the analyte is in the sample?
- Fundamental approach is unchanged
  - Describe the distribution as Student's t times the standard deviation of results

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

Blanks

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

OLD

- No consideration of blanks

NEW

- Analyze a minimum of 7 blanks
  - Method blanks
  - Existing data is OK and preferred
  - $MDL_B = X + Ts_B$
- MDL is the greater of MDL_B and MDL_S

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

Variance

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

OLD

- Not stated, usually one batch

NEW

- Spread over at least 3 batches (prep and analysis)
  - Spikes and blanks
  - Existing data is OK

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

On-going data collection

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

OLD

- None – redo once per year

NEW

- Collect spike data quarterly
- Collect ongoing routine method blanks
- Recalculate to verify once per year, update only if necessary

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

10x Rule

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

OLD

- MDL is not valid if more than 10X less than spiking level

NEW

- No 10 X rule

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

Does the 10X rule protect against MDLs that are too low?

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

True	1	2	3	4	5	6	7
100	100	103	99	102	97	98	102

Results within =/- 3%

Mean	RSD	Std Dev	MDL
100	2.3%	2.3	7.1

True	1	2	3	4	5	6	7
1.0	1.0	1.4	0.8	1.3	0.7	0.8	1.3

Results within =/- 40%

Mean	RSD	Std Dev	MDL
1.0	29%	0.29	0.90

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

Does the 10X rule protect against MDLs that are too high?

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Example, Iron

ICPMS detection limit (ultra clean semiconductor industry lab)

< 1 part per trillion

ICPMS detection limit (“clean” environmental lab)

1-10 part per billion

Typical level of environmental interest

Part per million range

6-8 orders of magnitude difference between absolute detection limit and level of interest

Length of a virus 7 orders of magnitude less than length of a person
Radius of the earth 7 orders of magnitude greater than length of a person

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

Multiple instruments

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

OLD

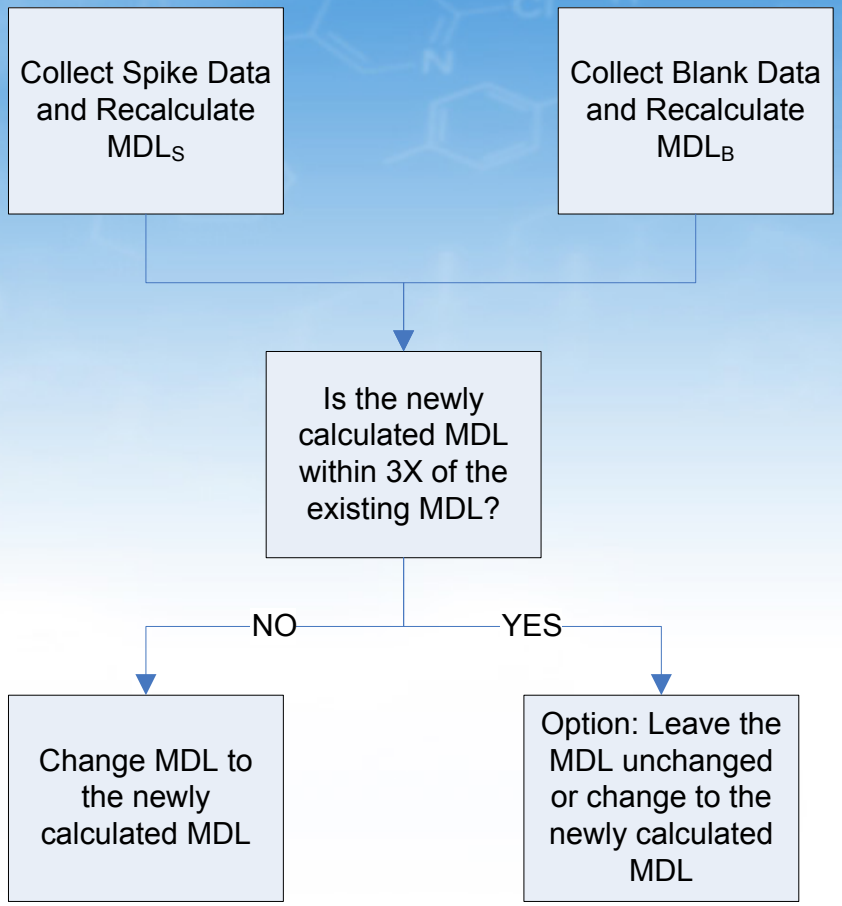
- No instructions

NEW

- Initial determination
- At least 2 samples per instrument
- Ongoing
- Quarterly spike on each instrument (can be same prep)

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

Annual recalculation



Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

MDL, TNI LOD and TNI LOQ

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

MDL and LOQ

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

MDL

- Low level spike, 2-10X estimated MDL
  - Calculate MDL_S = ts

LOQ

- Low level spike at or below LOQ
  - LOQ is spiking level or above
  - Use the collected data to calculate precision and bias

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

Good precision, good recovery

Spike	1	2	3	4	5	6	7
10	9.5	9.8	10.2	10.6	9.4	9.7	9.9

MEAN	STD. DEV	MDL S
9.9	0.4	1.3

Blanks	ND	ND	ND	ND	ND	ND	ND

MEAN	STD. DEV	MDL B
0.0	0.0	0.0

MDL	3X MDL	LOQ
1.3	3.9	10.0

LOQ = Spiking level

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

Good precision, moderate recovery

Spike	1	2	3	4	5	6	7
10	7	7.3	6.9	8.1	7.7	7.3	7.9

MEAN	STD. DEV	MDL S
7.5	**0.5**	**1.4**

Blanks	ND	ND	ND	ND	ND	ND	ND

MEAN	STD. DEV	MDL B
0.0	0.0	0.0

MDL	3X MDL	LOQ
1.4	**4.3**	**10.0**

LOQ = Spiking level

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

Poor/Moderate precision and recovery

Spike	1	2	3	4	5	6	7
10	6	7.3	7.6	5.7	7.2	7.9	5.3

MEAN	STD. DEV	MDL S
6.7	1.0	3.2

Blanks	ND	ND	ND	ND	ND	ND	ND

MEAN	STD. DEV	MDL B
0.0	0.0	0.0

MDL	3X MDL	LOQ
3.2	9.7	10.0

LOQ = Spiking level

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

Poor precision, poor recovery

Spike	1	2	3	4	5	6	7
10	5	7.1	3.2	6.5	7.4	3	3.3

MEAN	STD. DEV	MDL S
5.1	1.9	6.1

Blanks	ND	ND	ND	ND	ND	ND	ND

MEAN	STD. DEV	MDL B
0.0	0.0	0.0

MDL	3X MDL	LOQ
6.1	18.2	18.0

LOQ = 3X MDL
(greater than spiking level)

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

How the modifications improve the procedure

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

- Sensible MDLs when there is blank bias
  - 1980 Lead in tuna results overstated by 1000X due to blank contamination
  - 2004 EPA Episode 6000 data Chromium by ICPMS, 1400% recovery at the MDL and 600% recovery at the ML due to blank bias
  - 2013 Multi-lab blank detection rates
    - ~ 8270 SIM 6.4%
    - ~ 8921B 16%
    - ~ ICPMS 8%
  - 2014 Lead in particulate matter
    - ~ All blanks in the validation study exceeded the MDL

This problem is getting worse because of the need for low level data and increasing sensitivity of instrumentation

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

How the modifications improve the procedures

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

- Long term vs. short term bias
  - The difference varies from method to method and lab to lab, but can be large
  - Long term bias is what matters when it comes to the detection and quantitation performance
- Ongoing verification
- Very consistent with EPA office of Water MRL, EPA ORCR LLOQ

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

What does this mean to labs?

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

- Clear requirements
- Sensible MDLs
- Level playing field
- Low transition costs since existing data can be used
  - Note – labs should start complying with 3 batch rule right now
- Some additional organizational requirements

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

What does this mean to data users?

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

- MDLs that make sense
- Much lower rate of false positives, especially for ICP, ICPMS and some general chemistry tests
- Easier to compare labs
- In general, more reliable data = better decision making

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

How much will MDLs change?

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

- Analytes with minimal or no detects in blanks, eg most GC/MS analytes at normal levels:

Not Much

- Analytes with frequent detects in blanks, eg, metals, very low level PAH, some general chemistry tests:

Depends

  - If the lab is currently adjusting MDLs to avoid excessive false positives, not much
  - If the lab has been pushing MDLs below levels justified by the blanks, potentially quite a bit

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Questions

Copyright © 2015, TestAmerica Laboratories, Inc. All rights reserved.