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Really, A Revised MDL Procedure

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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 2015

EPA expects that the rule will be finalized in late 2016

TNI Chemistry Standard

Revisions to the Detection, Quantitation and Calibration language in TNI Volume 1 Module 4 (Quality Systems for Chemical Testing) are complete and will be incorporated into the TNI 2015/2016 Standard.

The 2015/2016 TNI Standards should be adopted by the States in 2017/2018

MDL and TNI revisions!!??

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- What if they have different requirements?
- What if they are implemented at different times?
- What if different states adopt the new requirements at different times?
- Will I have to have different detection limits for different states?


ORGANISE!

Good News

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- **The revised MDL and TNI Chemistry standards are fully compatible**
- If you are compliant with the revised EPA MDL then you are also compliant with the current MDL
  - One exception, discussed later
- If you are compliant with the revised TNI Chemistry standard then you are compliant with the current TNI Chemistry standard

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How to do a LOQ / MDL study

Select LOQ

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- **Choose your LOQ**
  - Must be at or above low calibration standard
  - If blanks are non zero, then calculate mean + 3X standard deviation. This will approximate the MDL and spiking level should be at least 2-3 times higher
  - Spike ideally in the range 0.5-1 x LOQ
-

Run the spiked and blank samples

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- Spikes
  - Minimum 7 spikes
  - Minimum 3 separate batches and three separate days
    - ~ Prep and analytical
  - Minimum 2 on each instrument
- Blanks
  - Use existing
  - If new method, minimum 7, at least 3 batches, at least 3 days, at least 2 per instrument

Initial evaluation

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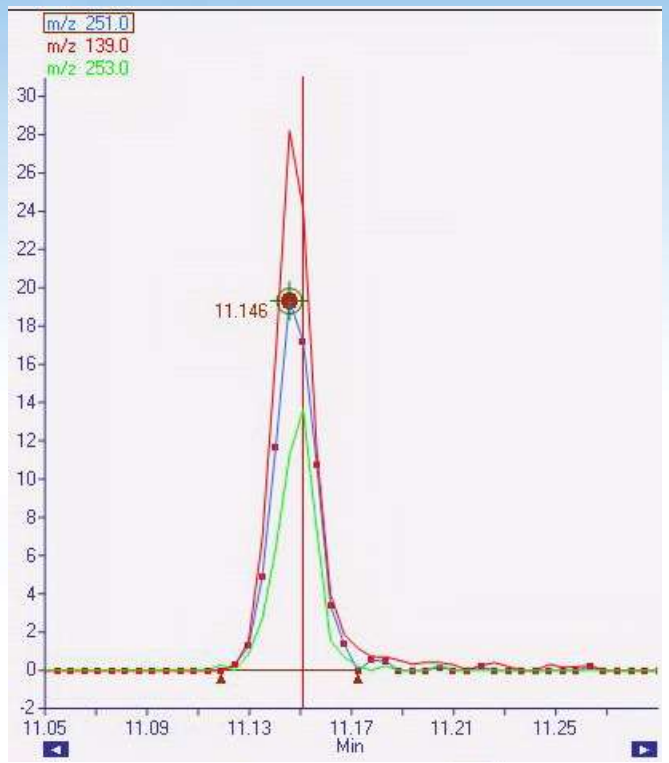
- Do the spike results meet qualitative identification criteria in the method?
- Calculate precision and accuracy, and the MDL
  - Adjust the LOQ if necessary



Slide on calculation of MDL b and MDLs

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$$MDL_{spike} = ts_{spike}$$

$$MDL_{blank} = X + ts_{blank}$$

MDL = the greater of MDL_{spike} and MDL_{blank}

Slide on evaluation of LOQ recovery

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Lab sets recovery criteria for the LOQ

Based on what?

Options

- Method criteria
- QAPP criteria
- LCS historical with some extra leeway (Method 8000)

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**

Success!
Spikes work for calculation of the MDL
And
Verification of the LOQ

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**

Success!
Spikes work for calculation of the MDL
And
Verification of the LOQ

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

Marginal!
Spikes work for calculation of the MDL
<u>And</u>
Verification of the LOQ
<u>But</u>
LOQ must be elevated above the spiking level

Poor precision, poor recovery

Spike at ½ desired LOQ

Spike	1	2	3	4	5	6	7
5	2.5	3.55	1.6	3.25	3.7	1.5	1.65

MEAN	STD. DEV	MDL S
2.54	0.97	3.04

Blanks							
	ND	ND	ND	ND	ND	ND	ND

MEAN	STD. DEV	MDL B
0.0	0.0	0.0

MDL	3X MDL	LOQ
3.04	9.12	10

Success!
Spikes work for calculation of the MDL
And
Verification of the LOQ

Good precision, elevated blanks

Spike	1	2	3	4	5	6	7
10	13.5	12.8	14.2	14.6	13.4	13.7	13.9

MEAN	STD. DEV	MDL S
13.7	**0.6**	**1.8**

Blanks	3.1	4.2	3.9	4.4	3.8	3.2	4

MEAN	STD. DEV	MDL B
3.8	**0.5**	**5.3**

MDL	3X MDL	LOQ
5.3	**16.0**	**16.0**

Marginal!
Spikes work for calculation of the MDL
<u>And</u>
Verification of the LOQ
<u>But</u>
LOQ must be elevated above the spiking level

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Ongoing MDL LOQ

Quarterly spikes

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- Run at least one spike on each instrument once per quarter, at the same concentration as the initial spikes
- MDL
- Must be detected, above zero, using qualitative identification criteria in the method
- LOQ
- Must meet the recovery criteria in the lab SOP

Annual assessment

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- MDL
  - Recalculate the MDLs and MDLb
  - MDLb uses the routine method blanks
- Compare with the existing MDL
  - If within a factor of 2 – Lab option to update or not
  - If outside factor of 2 – Must update

- LOQ
- Update the precision and bias statement

What do we expect if the true value is at the LOQ?

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Known precision? 

Known accuracy? 

Ability to detect and report? 

- Freedom from false negatives? 

What do we expect from a true value at the MDL?

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Known accuracy? 

Ability to detect and report? 

Freedom from false negatives? 

We do expect freedom from false positives (99%) when the analyte is not present

What does this mean to labs?

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- 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

What does this mean to data users?

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- 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

How much will MDLs change?

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- 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

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Questions?