DRAFT

The NELAC Institute Presents

VERIFICATION OPTIONS FOR LOD

THE NELAC INSTITUTE

TNI Limit of Detection

A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility.

LOD

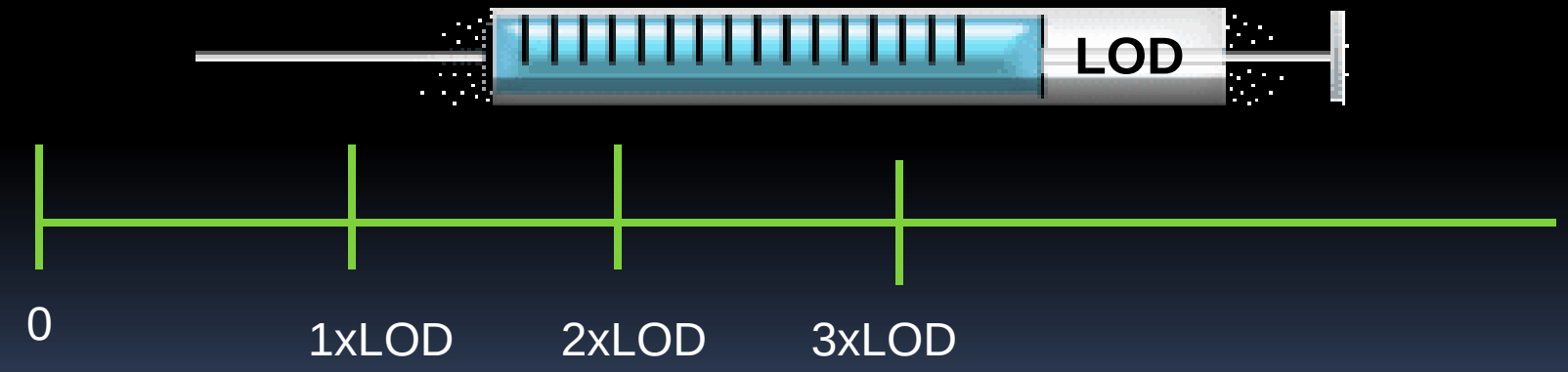
TNI LOD Verification

The standard says you verify the LOD by detecting a spike near the LOD.

“Detecting” according to TNI means it returns a result greater than zero.

TNI LOD Verification Rules…

1. You must spike no more than 3x LOD for single analytes and no more than 4 x LOD for multiple analytes in a mix.



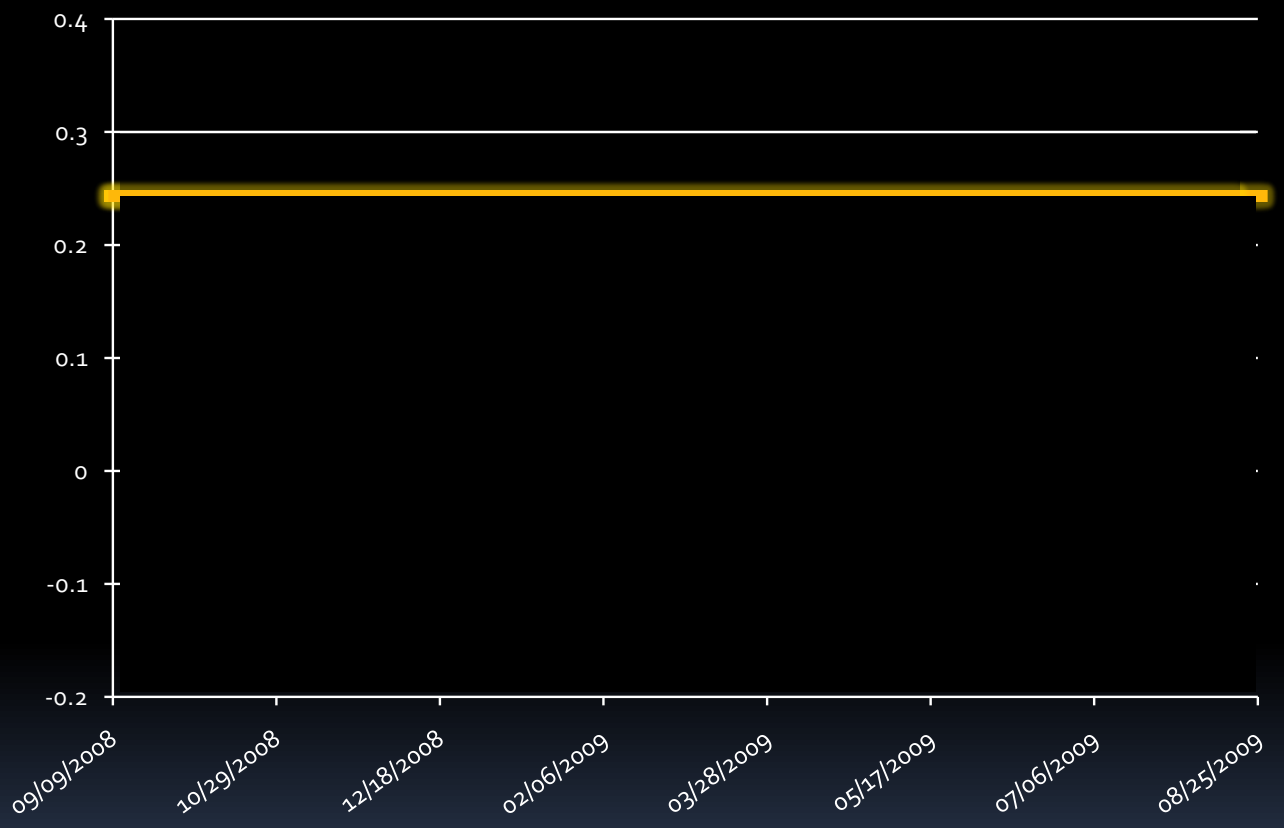
The 2003 NELAC standard says 2-3 x (1-4 x) for multiple analytes.

TNI LOD Verification Rules…

3. Your spike result must be detected (a value above zero) in each quality system matrix.

TNI LOD Verification Rules…

4. All sample-processing and analysis steps of the analytical method shall be included in the validation of the LOD.



BACKGROUND SIGNAL

TNI LOD Verification Rules…

5. The verification shall be performed on every instrument that is to be used for analysis of samples and reporting of data.

TNI LOD Verification Rules…

6. You perform the validity test as part of the LOD determination process.

TNI LOD Verification Rules…

7. Verification shall be done prior to the use of the LOD for sample analysis.

TNI LOD Verification Rules…

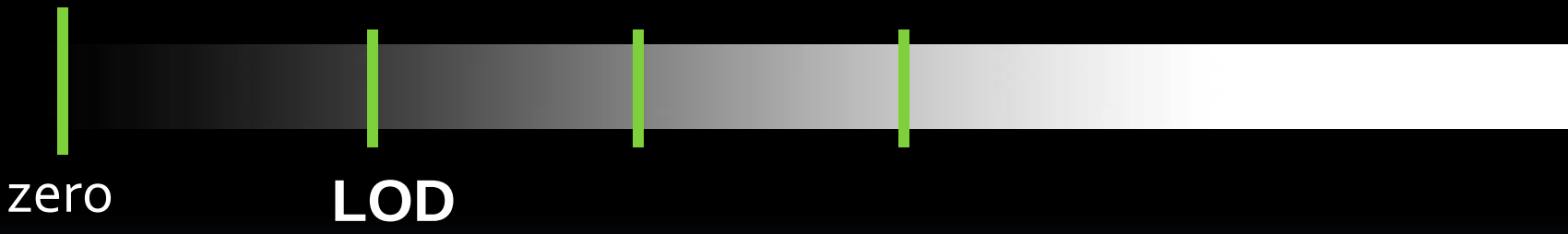
8. The LOD, if required, shall be verified annually for each quality system matrix, technology, and analyte.

The Bottom Line:

- Don't settle for an LOD that is too low - you'll have trouble verifying it.
- Pick a spike concentration that you can see. If it needs to be greater than the TNI allowable concentration to be reliably seen, your LOD is too low. Change it.

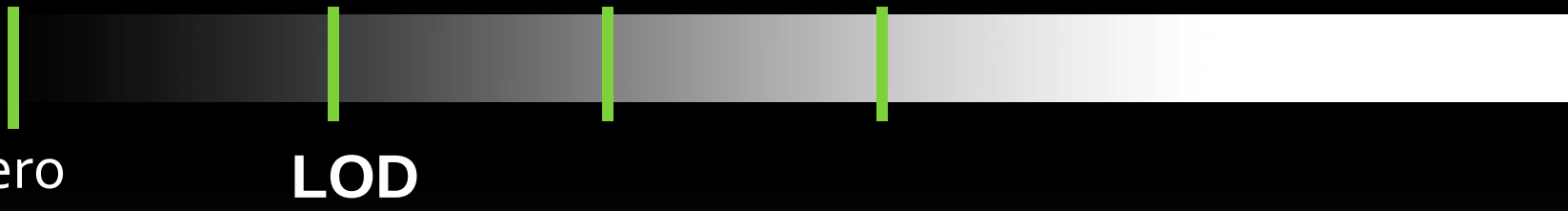
Tips for Successful Verification - Pick the right spike concentration

Not Detected



If you spike close to the LOD, you increase the risk of Not Detecting your analyte.

Tips for Successful Verification - Pick the right spike concentration



If you spike at the maximum allowable concentration, you should always detect the analyte.


What can go wrong?
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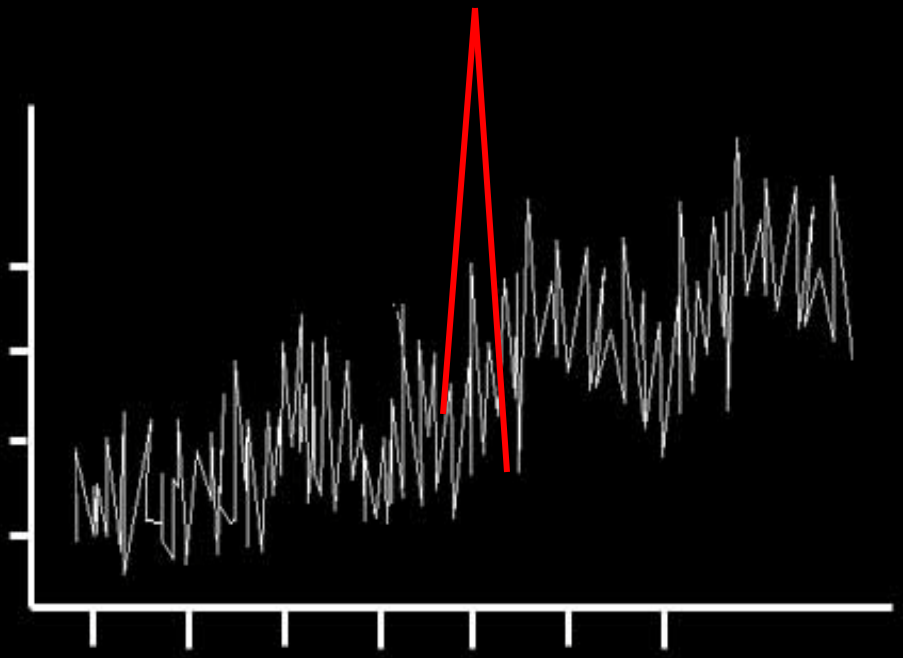
HOW TO KNOW WHEN LOD IS TOO LOW

If your LOD won't verify (you can't detect the spiked analyte even after corrective action), your LOD is too low.

REMEDY

Increase your LOD.

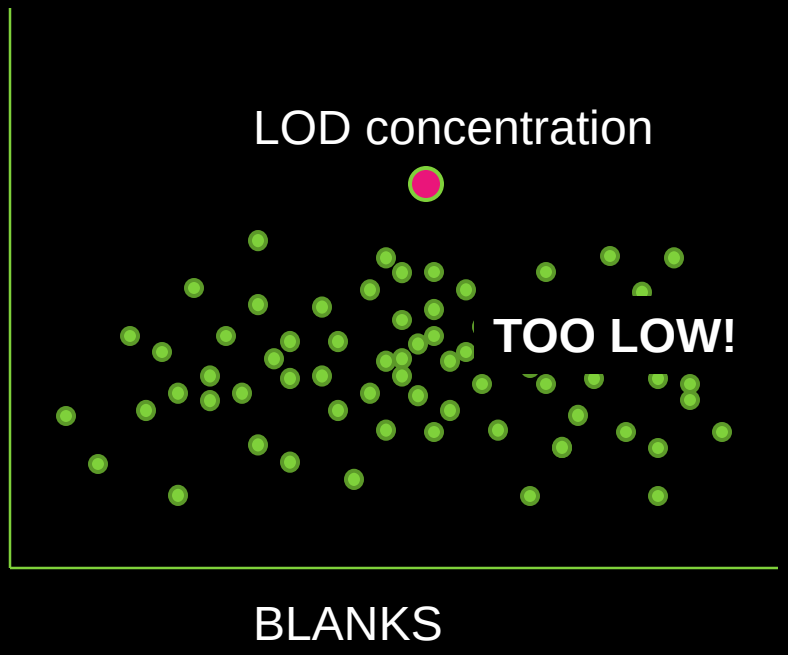
Background…



3x signal to noise is one choice. You could choose something else if not otherwise mandated.

The LOD is best when it's out of the noise or background. Otherwise you may not be verifying your spike, you may be verifying noise.

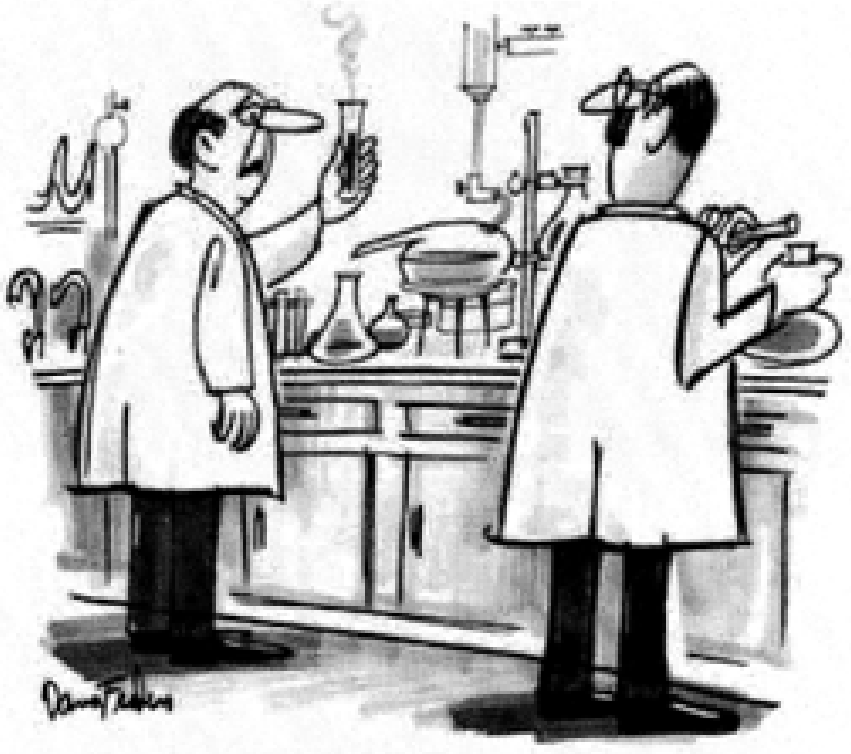
Background…



The LOD is best when it's out of the noise or background. Otherwise you may not be verifying your spike, you may be verifying noise.

HOW TO KNOW WHEN LOD IS TOO HIGH

If your LOD always verifies, it probably could be lower. It's up to you and your requirements to lower it or not.



What is the opposite of 'Eureka!'?

REMEDY

Nothing required.

To verify GC/MS LOD's

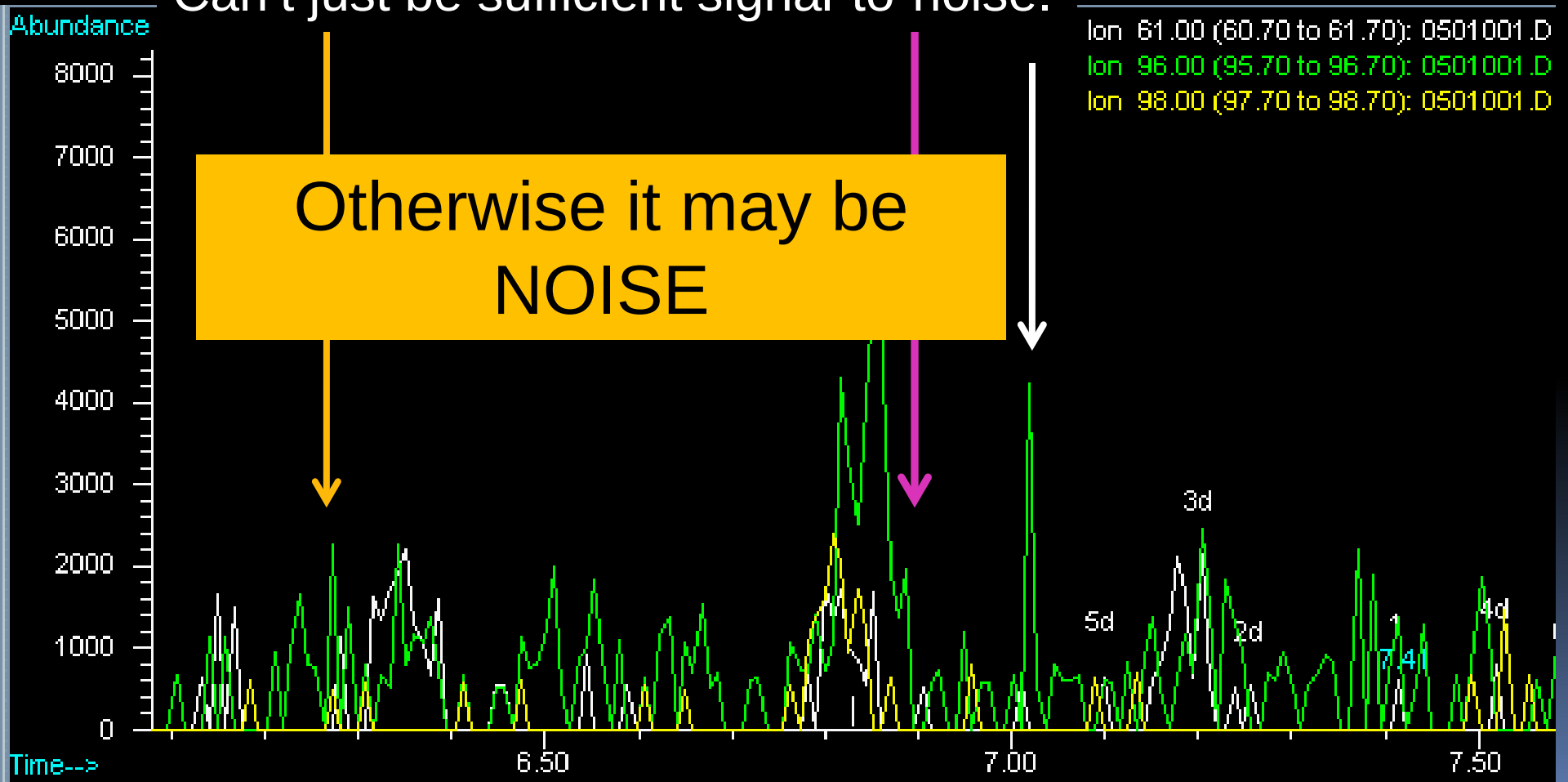
- "Background" for organic GCMS analyses is compound specific. It may a non-issue for many compounds.
- False positives are less common and false negatives become the concern at very low concentrations for organic GC/MS.
- Spike at **1-4** times the LOD (if multi-analyte, otherwise 1-3) and take through the whole process.
- If detected according to your identification criteria, the LOD is verified.

Detected on a MS

Can’t just be correct retention time.

Can’t just be presence of correct ions.

Can’t just be sufficient signal-to-noise.

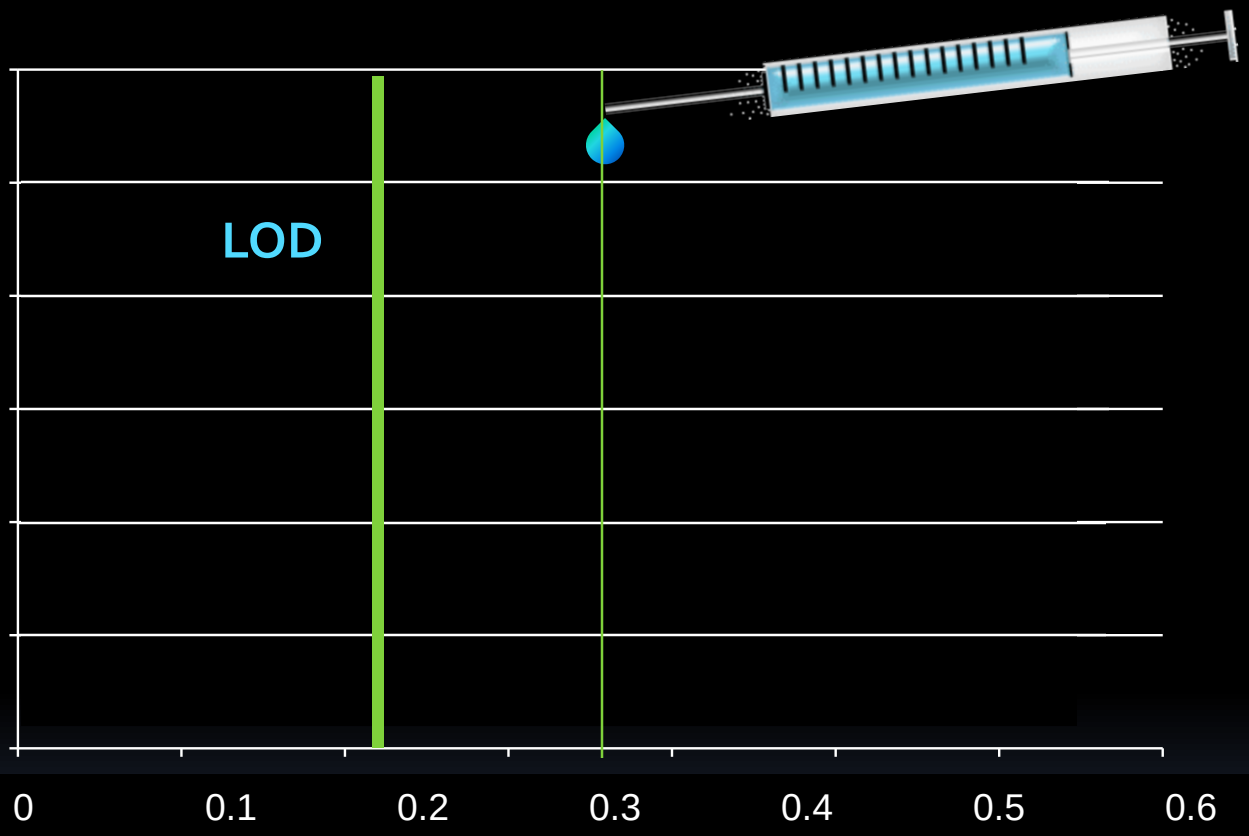


WARNING:

If you spike at 0.03….

But recovery is only 50%…

You'll only get 0.015 to the detector



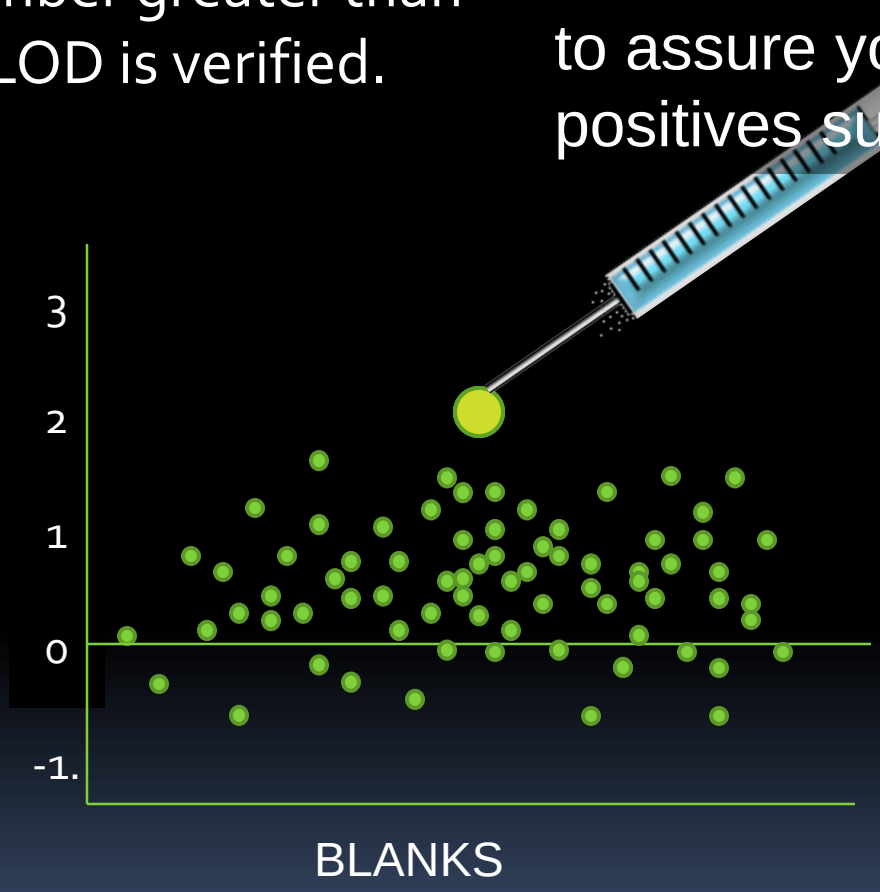
Choose a spike concentration that you will detect AFTER it has gone through the whole method, not a concentration you can see in a calibration standard. For an analyte that typically has 50% recovery or less, spiking near LOD will end up undetected.

TNI REQUIREMENT

If you detect the verification spike (a number greater than zero), your LOD is verified.

OPTIONAL CRITERIA

You may wish to identify certain detection criteria in your SOPs to assure you minimize false positives such as...



…out of the noise to minimize false positives…

…all criteria used for environmental samples be required for LOD verification spikes…

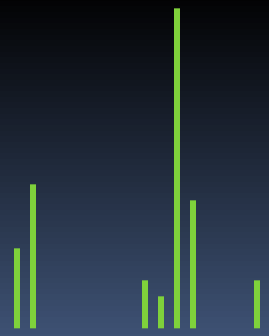
TNI REQUIREMENT

If you detect the verification spike (a number greater than zero), your LOD is verified.

OPTIONAL CRITERIA

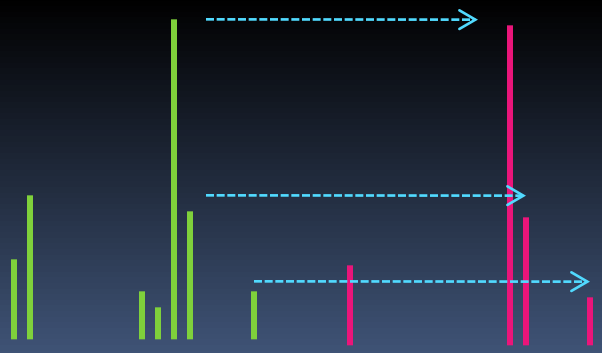
You may wish to identify certain detection criteria in your SOPs to assure you minimize false positives such as...

…sufficient signal to produce a gaussian-shaped curve



BLANKS

…with the correct IONS



…in the correct ratio's


QUESTIONS