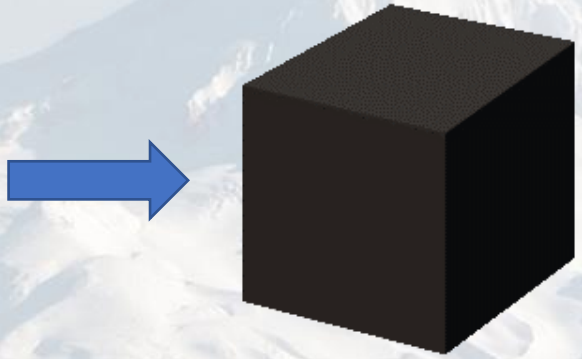
The Laboratory:
A Peek Inside the Black Box

Sheri Heldstab
CHESTER LabNet

Cleaner Air Oregon Workshop
5 November 2020

“Magic Numbers”

Samples

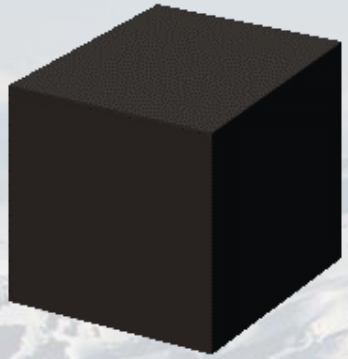


Laboratory

1.79 mg/Sample

Results

Laboratory Accreditation – Is It Important?



Accreditation by ORELAP requires the Laboratory to:

1. Document every stage of data generation.
2. Have policies and procedures in place for data that may be compromised.
3. Maintain all equipment, both analytical and support equipment.
4. Demonstrate that equipment is working properly.
5. Provide an “audit trail” capable of tracing all data back to National Standards.
6. Be audited a minimum of every other year by an outside auditor to ensure compliance with all requirements.

Laboratory Accreditation = No Surprises

Opening the Black Box

- **Receipt**
  - Chain of Custody
  - Sample Condition
- **Log In**
  - Unique ID
  - Info from Chain of Custody
- **Analyze**
  - Goes to Analysts
- **Report**
  - Detection Limits

Inside the Black Box – Sample Receipt

1. Samples checked for condition:
   - Broken?
   - Leaking?
   - Correct Temperature?

2. Samples checked against Chain of Custody:
   - All samples present?
   - All sample ID's match?
   - Analysis clearly defined on Chain of Custody?
   - Sample information included?

3. Corrective actions, if necessary (contact client).

4. Samples logged in:
   - Given unique Laboratory ID in database
   - Passed along to analysts

Inside the Black Box – Sample Preparation

Can be as easy as loading into an autosampler.

- Method 26/26A
- CTM027/ST-1B
- Method 316
- Method 6 & 8 (ALT133)

~OR~

Can take up to a month.

- Method 29 – evaporation time
- Method 13B – 3 days of hands-on preparation
- Method 202 – convoluted preparation
- Method 5 – evaporation, especially when water present

Inside the Black Box – Sample Analysis

Analysis time is “per sample, per analyte”

One metal in a sample may only take 3 minutes, while 18 metals may result in over a week of analytical time.

Some methods require all samples be run in duplicate – if one analysis requires 15 minutes (M26/26A), then one sample requires 30 minutes.
(Longest run = 52 hours)

Gravimetric methods require desiccation times of 24 hours followed by 6 hours: tare weighed twice, gross weighed twice, for 60 hours of total desiccation time, not including actual time to weigh the vessels.

If results do not pass internal and method-specific Quality Controls, repeat until resolved.

Inside the Black Box – Reporting Results

- All data is checked for Quality Control, both numerical and typographical.
- Data is reported to clients.
- CLN: data is reported both “at the instrument” and “per sample” with *detection limits* for each.

Inside the Black Box – Detection Limits

Per NELAC, a Detection Limit is “The minimum result which can be reliably discriminated from a blank with a predetermined confidence level.” (99% chance not a false positive – does not account for false negatives)

ORELAP/NELAC accredited labs must follow “Definition and Procedure for the Determination of the Method Detection Limit, Revision 2” (aka “MUR method”)

- Standard deviation of low concentration standards.
- Standard deviation of, and concentration of, blank samples.
- Taken through entire process, including all preparatory steps.
- Verified annually.

Does not take into account sampling or mathematical activities.

Inside the Black Box – Detection Limits, How Low Can You Go?

- Detection Limit is function of method and instrumentation. Cannot be lowered by laboratory due to [math].
- Some methods, samples can be “concentrated” (M26), some methods require concentration (M29). Interferents will also be concentrated.
- Can sometimes be lowered by pulling more gas volume, but risk increasing interferents as well as target analyte.
- Detection Limit *only* takes into account uncertainty from laboratory operations. Actual Detection Limit would be higher if all uncertainty accounted for.

Inside the Black Box – Reported Detection Limits

- Laboratory Method Detection Limit reported as “at the instrument” and “per sample”.
- “At the instrument” DL determined by EPA MUR Method, in keeping with ORELAP accreditation.
- “Per sample” DL = “At the instrument” DL × sample volume (liquid).
- (Sample volume may be a digestate volume if the entire sample is consumed during preparation).

Detection Limits – Points to Ponder

Do we really need to see that low?

Mercury DL = 7 ppt (part per trillion)

Population of Earth = ~8 billion
(7 ppt = ~1/125 ppb; 125B / 8B = ~16)

Could identify 1 individual from ~16 earths' worth of people with 99% confidence that that individual existed. Would need 5 people in ~16 earths to accurately quantify the number of individuals found (LoQ = 5x DL).

Detection Limits – Points to Ponder

But…

Our lungs are incredibly efficient at exchanging gases and even particulates as the blood–gas barrier has a very large area and is extremely thin.

Some pollutants are persistent, and accumulate in the environment over years.

Some pollutants ($PM_{2.5}$) are small enough to enter the blood stream as a particle, and can bio-accumulate.

Some chemicals are just plain nasty pieces of work.

Recap of the Laboratory “Black Box”

Receipt
- Chain of Custody
- Sample Condition

Log In
- Unique ID
- Info from Chain of Custody

Analyze
- Goes to analyst

Report
- Detection Limits

Recap of Detection Limits

1. Accredited laboratories must follow the EPA's method for the determination of detection limits (this is a good thing).

2. Detection Limits represent the point at which there is a 99% certainty that the result reported is not a false positive.

3. Detection Limits are a function of sample preparation, sample analysis, and the instrumentation used during analysis.

Questions?

Sheri Heldstab
CHESTER LabNet

sheldstab@chesterlab.net