WWTP Lab Inspections

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Performance Audit Inspections (PAIs)

- DES conducts ~ 12 per year
- 6 per each Quality Assurance Officer

Performance Audit Inspections

- How are labs selected?
- What do we look for when we do audit inspections?

How are labs selected?

- You're a major discharger if:
  - > 1 MGD
  - Industrial facility scoring >80 points on the permit assessment
  - The Ohio EPA director says so

How are labs selected?

- Division of Surface Water requests
  - Plant inspectors can notice deficiencies
  - Permit writers see inconsistencies in data?
  - Permit holders can request assistance

DMR-QA

- Discharge Monitoring Report - Quality Assurance
- Done annually to “test” analytical capabilities of labs
- An indicator for unsatisfactory lab work, but does not tell the whole story
- For all major dischargers, the DMR-QA study is an indicator of performance.
- Some facilities ‘farm out’ tests that are too costly to perform in-house.
- DES also conducts inspections on contract laboratories.

What to do they do if they fail a test?

- Don’t ignore it!
- Investigate why the test was missed.
- Send a corrective action letter to us describing the problem and the game plan.

AND

- Participate in a performance test (PT)

…… *OR*……..

- Order a quality control (QC) sample and demonstrate correct analysis by meeting the range specified.

What happens during an audit inspection?

For a full-blown inspection we look at:

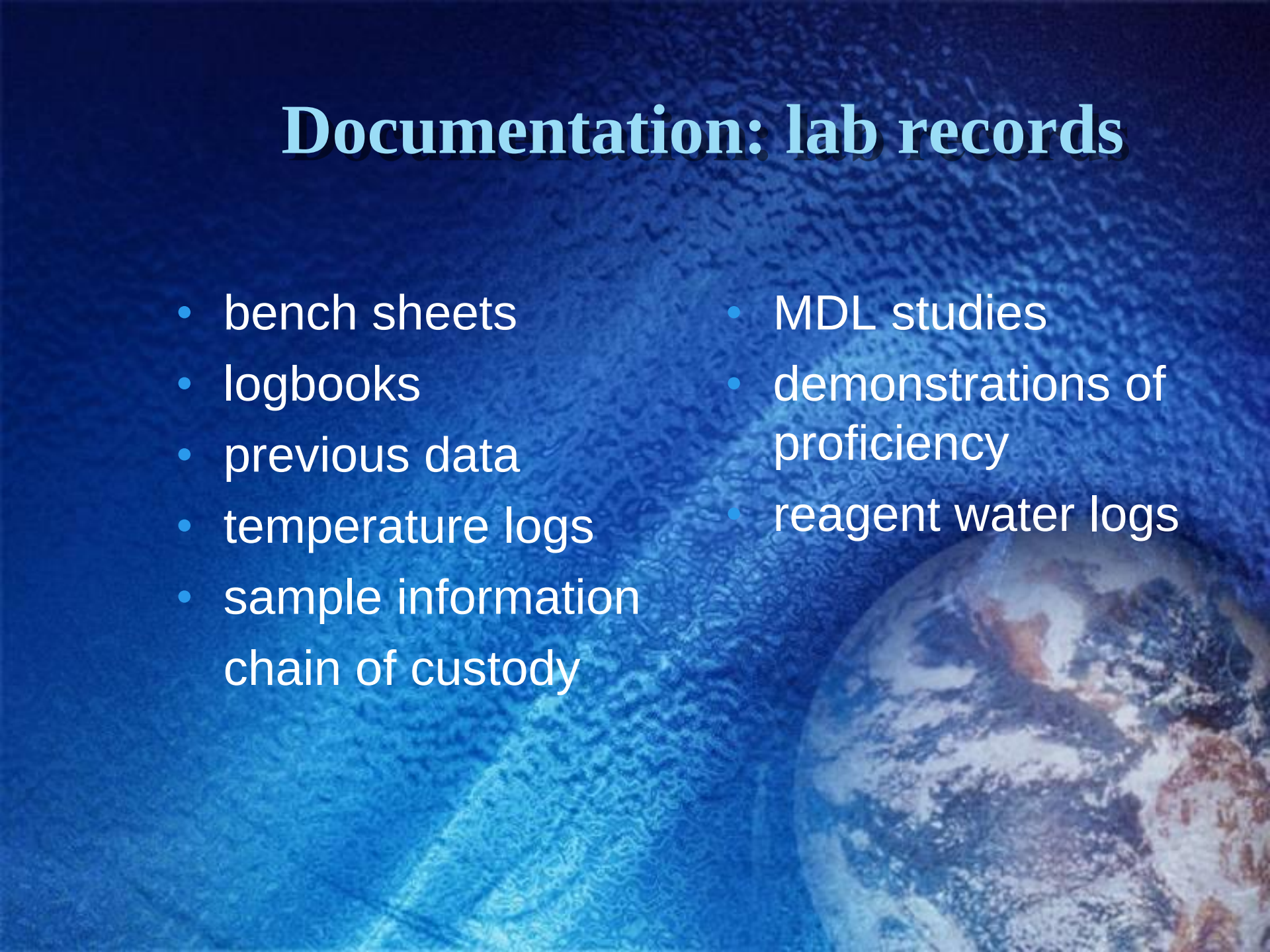
- records
- lab condition
- chemicals
- documentation
- and talk through some analysis

Documentation: lab records

- traceability:
  - Can all the processes and solutions be traced back to a standardized chemical, weight, volume, or temperature?

Documentation: lab records

- bench sheets
- logbooks
- previous data
- temperature logs
- sample information
- chain of custody
- MDL studies
- demonstrations of proficiency
- reagent water logs



documentation: lab records dos and don'ts

- Don't…
  - Use pencil…
  - Use correction fluid/tape…
  - Completely scratch out entries…
- Do…
  - Use blue or black ink…
  - Make corrections by drawing a single line through a mistake. Initial and write the correct entry nearby…

Logbooks…

- … should be hardbound with sequentially numbered pages.
- … should be maintained for each analysis performed.
- … must be labeled with title and dates of use.

Inside the Logbook…

- All information must be legible and in English.
- Entries must be made the day the work is performed.
- When making entries, ask yourself if you can trace chemicals back to when they were received. If not, chances are no one else can either.

What info needs to be entered in the logbook?

- Date
- Initials
- Sample identifier(s)
- Chemical information
- Calibration details
- Volume/mass of sample analyzed (if not routine)
- Serial number(s) for instrument(s)
- Unusual sample characteristics
- Dilutions

- Do not skip pages or leave blank spots to fill in later.
- Logbooks are chronological.

Chemicals

- Expired chemicals on shelves?
- Are they labeled with dates of receipt and opening?
- Are flasks and other containers labeled?

Chemical information:

- Lot number and manufacturer of all chemicals used
- Dates: receipt, open, and expiration
  - Manufacturer expiration date trumps all.
  - Dry chemicals have a shelf life of 6 years.
  - pH buffers are good for 6 months, once opened.
  - Prepared stock standards are good for 6 months.
  - Working standards have a shelf-life of the holding time for that analysis.
- Amounts of chemicals used
- Concentration of prepared solution
- Calculations

Chemical information

- Chemical Solutions
  - Are prepared solutions labeled correctly?
    - Chemicals used
    - Date of preparation and expiration
    - Concentration
    - Initials of preparer
    - Is there a matching entry in a logbook?

Logbooks - Often Used Info:

- For solutions prepared the same way each time, a recipe can be entered in the logbook and referred to.
- Referring to a page where a solution was made or opened is totally “OK.”
- Label units at the tops of columns of measurements.

Temperature Logs

- Every day that a chemist is in the lab, temperature info must be recorded.
- Use the thermometer, not the readout - they are rarely accurate.
- Adjustments or maintenance must be recorded.
  - Incubators
  - Sample storage coolers
  - Standard storage coolers
  - Ovens used for solids determinations

Thermometers:

- Maintain a logbook for thermometers.
- Tag all thermometers uniquely (tape around the top) and assign a correction factor.
- Certify all thermometers annually against a NIST-certified thermometer.
  - Compare them at the temperature of their intended use, in an ice bath, or in boiling water.
- Thermometers should be placed in liquid, not touching the sides of the container. A rubber stopper with a hole is perfect for this.

Sample cooler

- Is there food in the sample cooler?
- Are chemical standards stored with the samples?
- Is the temperature kept within limits (< 6°C)?
- Is the tip of the thermometer immersed in liquid and not touching anything (this applies to all thermometers not in ovens)?

Laboratory Observations

- Glassware washing
  - Lab Detergent (Alconox or similar)?
  - Tap water rinse?
  - Acid rinsed?
  - Distilled water rinse?
  - Is there a film or residue on the glassware?

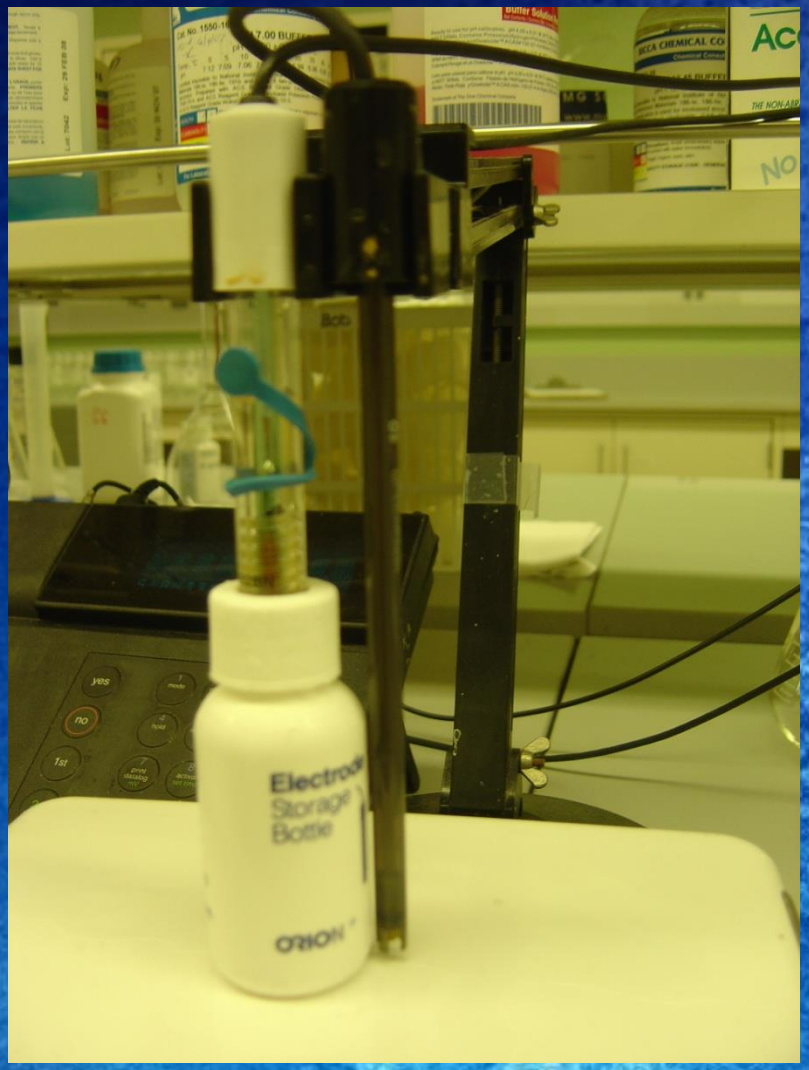
All labs should have:

- analytical balance and weight set
- BOD incubator with thermometer
- BOD carboy or bottle for holding dilution water
- pH meter and buffers
- volumetric glassware
- desiccator
- chemicals
- lab reagent water
- sample cooler
- standard cooler
- SOP documents

Analytical balance

- Level?
- Dirty?
- Weight set dirty?
- Calibration records?
- Non-vibrating slab

pH meter



- Probe crusty?
- Probe filled?
- Calibration slope recorded?
- Buffers expired (open more than 6 months - for bottles)

Volumetric glassware

- must be class A
- standards must be made in these, not graduated cylinders or beakers
- Volumetric pipettes

Desiccator and desiccant

- Is there desiccant?
- Is it old (pink)?
- Does the seal work?

BOD: A History of Problems

- No other subject will cause a better argument than BOD. Why? Because this determination is apparently subject to more variations and discrepancies due to as yet, unknown causes than any other test used in the sewage works laboratory (Eldridge, 1933).

BOD a little extra information

- The BOD test is a bioassay rather than a chemical analysis
- The test can produce results that are reproducible: however the reproducibility is directly related to the analyst's level of attention to detail in exactly following the standard procedure

BOD incubator

- Records? (19-21°C)
- Bubbles in bottles?
- Thermometer?

BOD dilution water carboy

- Crud growing inside?
- Tubing black or discolored?

Lab reagent water

- What is the reagent water source?
  - Delivered in a jug by a local water company?
  - Filtered onsite using filters or a still?
- Checked for pH or conductivity?
  - pH should be between 5-9
  - conductivity should be <2 µmhos/cm
    - Municipal lab found 100 µmhos/cm: contaminated BOD
    - Will affect all analyses
    - Is there a film or residue on the glassware?

Laboratory observations

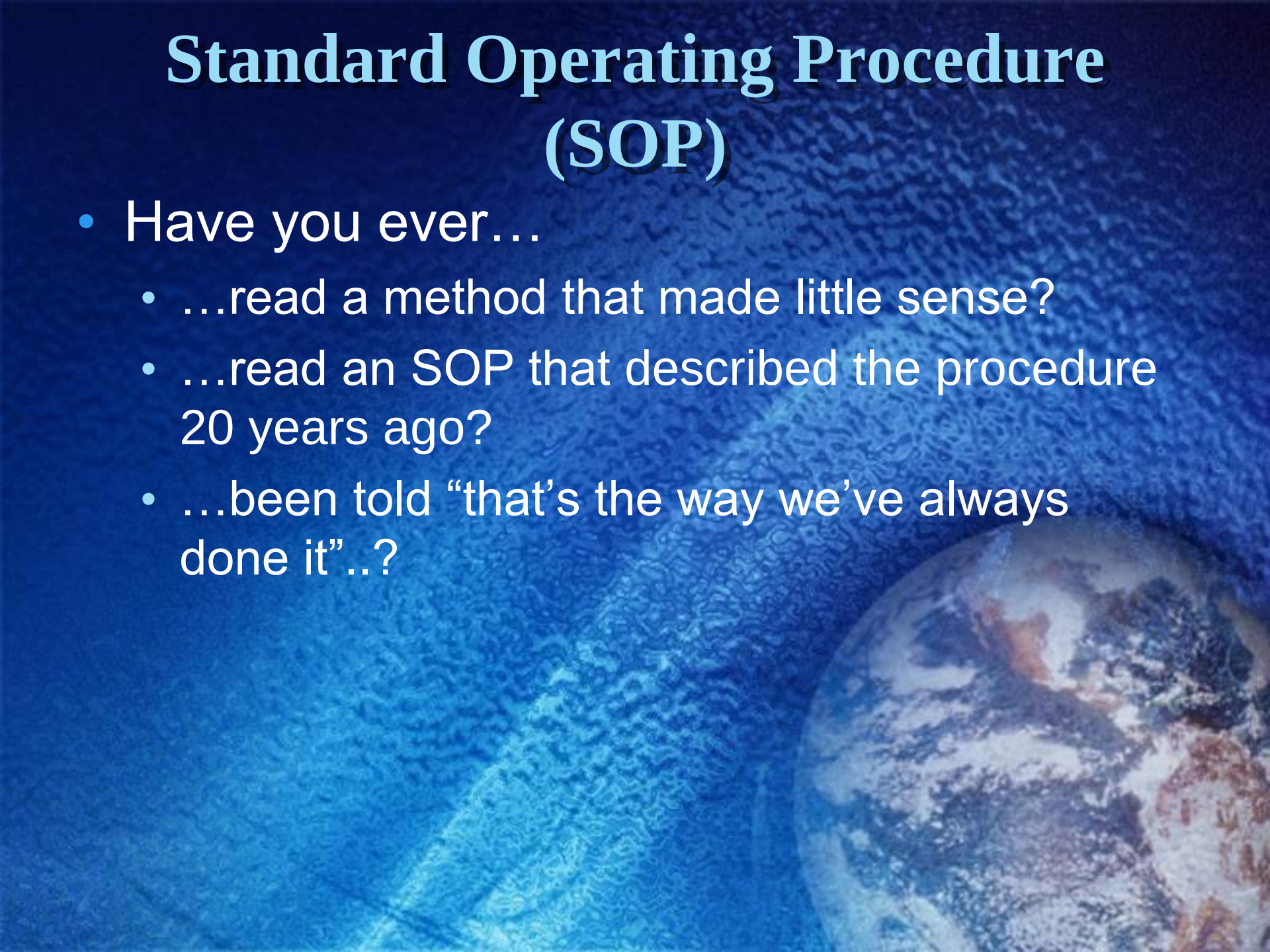
- coolers
  - Thermometer?
  - Temperature log filled out?
- incubators
  - Thermometer?
  - Temperature log filled out?
- ovens
  - Are there thermometers in each?
  - Temperature log filled out?
  - Is there rust or other crud that could fall into weighing pans?
- Is the lab filthy and cluttered?

Documents on hand:

- SOPs
- MSDS
- instrument manuals
- MDL studies

Standard Operating Procedure (SOP)

- Have you ever…
  - …read a method that made little sense?
  - …read an SOP that described the procedure 20 years ago?
  - …been told “that’s the way we’ve always done it”..?



SOPs should…..

- …contain information pertaining to your particular lab
- …describe the procedure followed in your particular lab
- …be easy to follow and understand
- …be updated annually or anytime changes are made to the method
- …not be so detailed that they're confusing

They should have an SOP for…

- …all methods of analysis
- …sample receiving protocol
- …glassware washing
- …MDL procedure
- …records retention
- …calibrating thermometers
- …calibrating pipettes

SOPs - individual ‘methods’

- Title
- Scope and Application
- Summary
- Sample Handling & Preservation
- Interferences
- Apparatus & Materials
- Reagents
- Procedure
- Calculations
- Quality Control
- Maintenance
- Corrective Action
- Reference (parent method)

MSDS

- Material Safety Data Sheets
- Come with each chemical
- Should be filed and in an easy to find location
- OSHA requires them to be on-site and easy to find
- Should be organized alphabetically by chemical name

Instrument manuals

- Contain valuable information for troubleshooting, ordering replacement parts, and calibration
- Should be kept near the instrument or a known location
- Can often be ordered from the manufacturer if misplaced
- Electrode meters often have slope requirements in percentages or millivolts.

Method Detection Limits (MDLs)

- Probably the most confusing aspect of laboratory analysis
- Are not applicable to all analyses (like pH and BOD)
- Studies do not have to be performed all in one day
- MDL ‘samples’ must go through all steps of preparation and analysis
- Must be re-evaluated whenever changes are made to a method or instrument
- Variation is the key

MDLs

- Typically 7 or more aliquots are analyzed
- The standard deviation is calculated
- The standard deviation is multiplied by the corresponding student's T value, the result is the MDL
- If the level of the spike is greater than the MDL and less than ten times the MDL it is considered acceptable

MDLs

- TSS calculation
- Spike concentration was 7.0 mg/L
- Results were 7.0, 6.0, 7.6, 5.1, 5.0, 5.6, and 6.1 mg/L
- For seven aliquots the 99% confidence interval student's T value is 3.143
- 3.143 * 0.959 (the standard deviation) gives and MDL of 3.01 mg/L

Method Update Rules

- The 2007 MUR removed a lot of US EPA methods from the CFR
- The 2012 MUR updated many things
  - New methods (Luminescence DO)
  - New sections (136.6, 136.7)
  - Updated how Standard Methods is referenced
- The 2014 MUR has opened, though the deadline for method submittal has passed.

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