

WISCONSIN DEPARTMENT OF NATURAL RESOURCES

ASK THE AUDITOR

BRING US YOUR LAB & AUDIT QUESTIONS

October 2024 DNR.WI.GOV



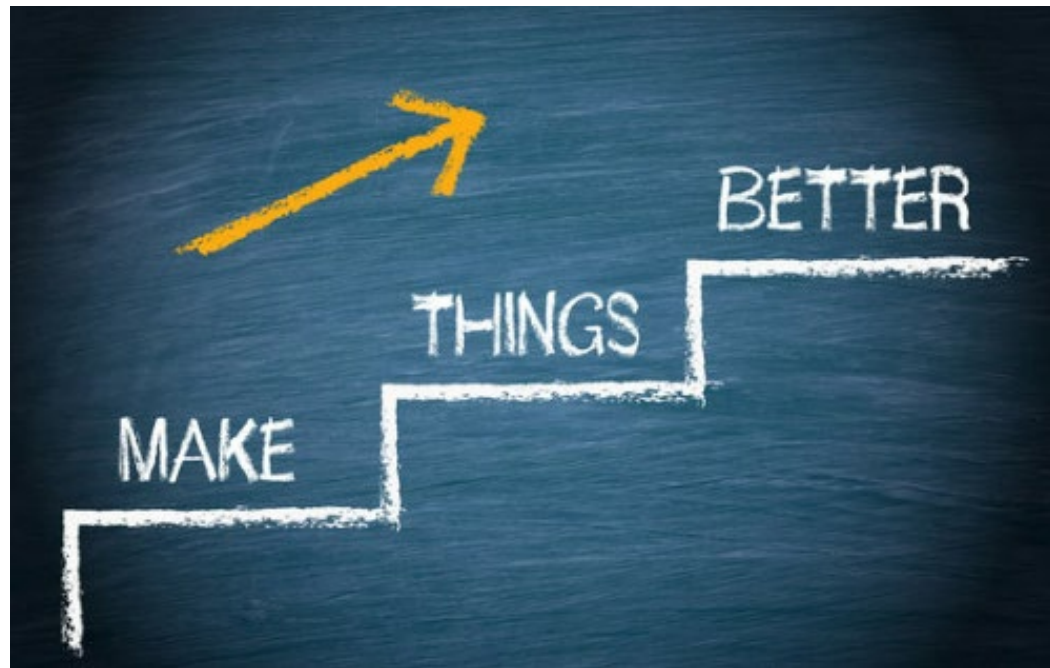
Deficiencies

How “BAD” is it to have deficiencies after an audit?

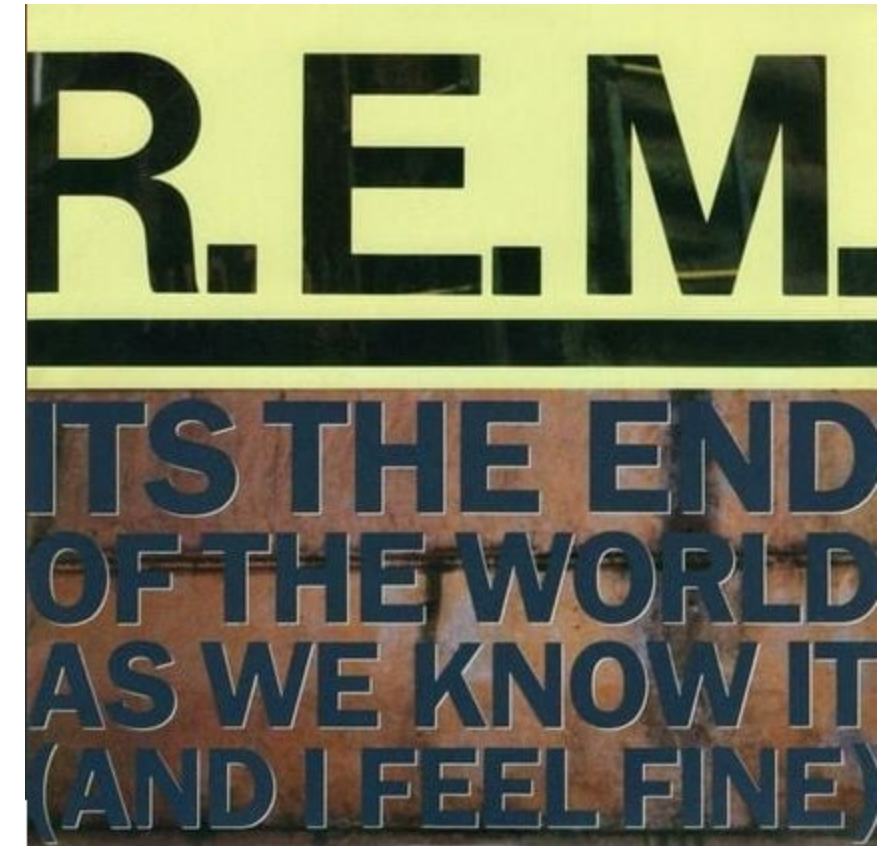


Deficiencies

How “BAD” is it to have deficiencies from an audit?



- It's **NOT** the “*end of the world as we know it.*”
- The audit is a continuous improvement effort.



- Unless warranted for serious violations (e.g., falsification), no one should be afraid of being “reprimanded” or “scolded” by anyone because an auditor found some things that need improvement.

Deficiencies

Why did we get cited for something this time but not before?



- It's possible that the last auditor(s) missed it.
- Method and regulations often change.
- It's possible that the program was informed by the EPA or other authoritative source to look at the issue differently.

Traceability & Records

How do I track standards and reagents I purchased?

Newly Received Chemicals Logbook = open - discarded version [10.4.13]

Chemical Name	Lot #	Date Received	Date Expires	Date Opened	Date Discarded	Vendor	Analyst who filled out form	Date form filled out

- *We have example forms on our website.*
- This is the “Open – Discarded” version.

Traceability & Records

How do I track standards and reagents I purchased?

Purchased Chemicals Log – Lab ID Tracking [Version 6/30/23]

Lab assigned ID #	Chemical Name	Date Received	Initials	Vendor	Lot #	Concentration [include units]	Date Expires	Date Discarded

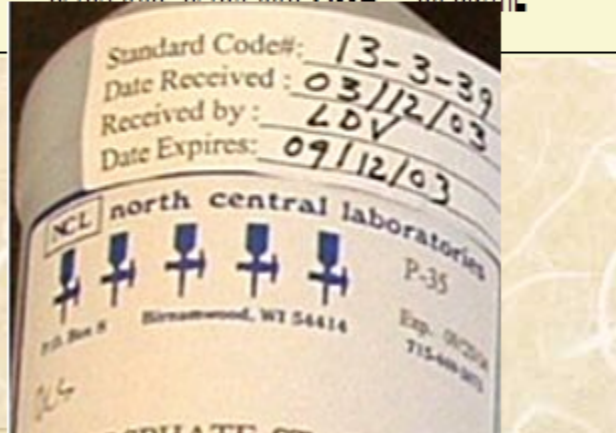
- *We have example forms on our website.*
- This is the “Lab Assigned ID #” version.

Traceability & Records

How do I track standards and reagents I purchased?

Page	Standard	Parameter/	Date	Exp.					
line	Code	Name	analyte(s)	Manufacturer	Lot#	Rcv'd	Date	Initials	Concentration
39	13-3-39	TP 2nd source	PO ₄ ⁻³	NCL	P350020923	3/12/2003	9/24/2004	RGM	50 ug/mL
40	13-3-40	TP stock std	PO ₄ ⁻³	VWR	3027	3/15/2003	6/15/2003	GWA	50 ug/mL

Standard Labeled: 13-3-39
13 = Log # - 13th log book
3 = Page Number
39 = line on the page – 39th line



- *We have example forms on our website.*
- This is the “Book – Page – Line number” version.

Traceability & Records

How do I track standards and reagents I prepare in the lab?

Chemical Preparation Logbook [2.26.13]

Preparation Details										
Prepared Chemical Lab ID #	Prepared Chemical Name	Prepared Chemical Conc <i>(include units)</i>	Source Chemical Lab ID #	Source Volume used <i>(include units)</i>	or Source Weight used <i>(include units)</i>	Prepared Chemical Final Volume <i>(include units)</i>	Prep Notes <i>consider recording (dilution solvent) or (source chemical conc)</i>	Date Expires	Prep By	Prep Date

- *We have example forms on our website.*
- This is the “Prep Notes” version.

Traceability & Records

How do I track standards and reagents I dilute in the lab?

Prepared Chemicals Log – Lab ID Tracking [Version 6/30/23]

Prepared Chemical Lab Assigned ID #	Chemical Name	Prep Date	Prep By	Source (Parent) Lab ID #	Source Volume or Weight [include units]	New (Child) Final Volume [include units]	New (Child) Conc. [include units]	Date Expires	Date Discarded

- *We have example forms on our website.*
- This is the website version.

Traceability & Records

How can I make my lab digital only - no paperwork?

- Benchsheets and records (QM, SOPs, corrective actions, maintenance, etc.) can be stored electronically.
- A process would be needed to save everything uniquely, so it's not written over.
- Handwritten data can be scanned and saved electronically.
- *Raw data may be stored electronically.*
- *Records and documents that are stored only on electronic media shall be supported by the hardware and software necessary for retrieval and reproduction into hard copy.*
- *Analytical and technical records may be electronic media.*

Accreditation

Why are certain tests exempted from needing accreditation?

These tests are exempt from accreditation per NR 219:

- ❑ Dissolved oxygen, pH, bacteria tests, residual chlorine, temperature, turbidity, conductance, flow measurements.
- ❑ Physical properties (color, texture, bulk density, viscosity, ...)
- ❑ Nutrient tests (TKN, NH_4 , TP, K, WEP) of soils and sludges.

Accreditation

Why are certain tests exempted from needing accreditation?

- It is believed that these parameters were exempted after discussing the feasibility of small municipalities and industries being required to obtain accreditation, including the workload on DNR staff and the burden on those facilities.
- Approved test methods still must be used.
- Basin engineers may review these procedures with facilities.

QM & SOPs

Why are so many items required in the SOPs?

A laboratory shall maintain written standard operating procedures that document or reference activities needed to maintain its quality systems and that enable performing or reproducing an analysis in its entirety as performed at the laboratory.



QM & SOPs

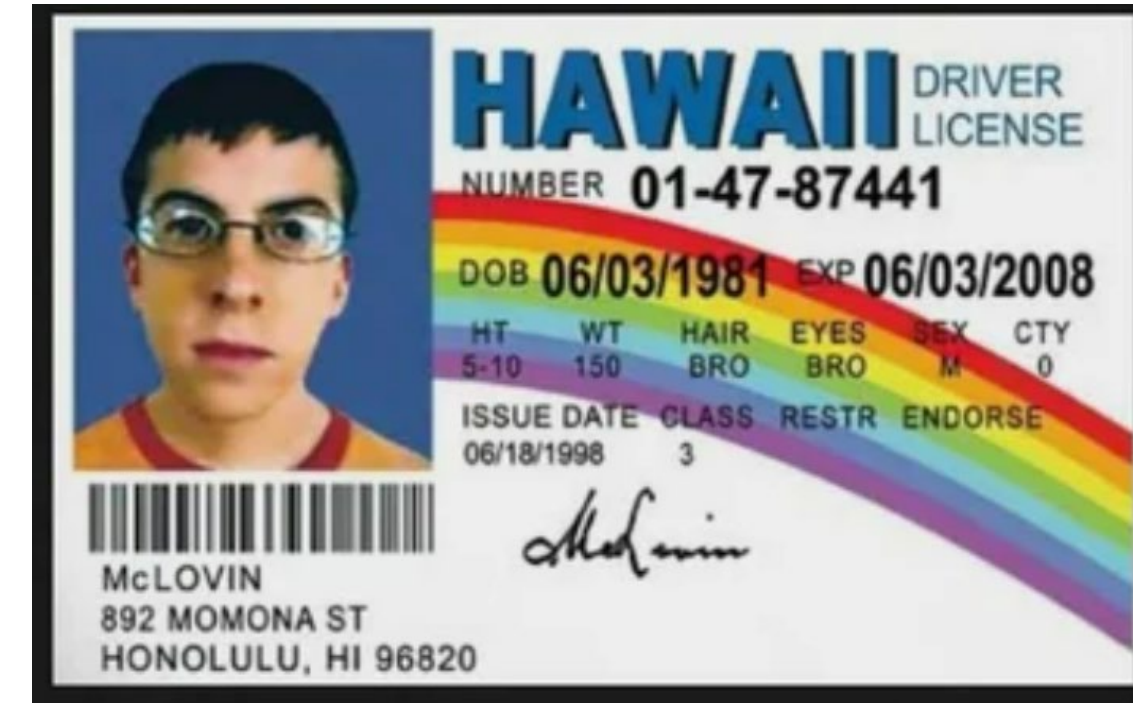
Writing SOPs is too hard.

- Template SOPs for BOD, TSS, TP, and NH₃-N tests are on the website.
- NR 149.40 provides a checklist of all items that need to be addressed in the SOP.
- Review the template SOP provided and update it specific for your laboratory. Do a good job the first time and then updates are only needed when things change.
- Be detailed enough that a trained, new analyst could easily follow the SOP with no help and perform it the same way you would.

Initial Demonstrations of Capability (IDCs)

What is an IDC, and what do I have to do for it?

- The IDC demonstrates that the analyst has the skills required to generate quality results.
- The laboratory must establish the criteria they will use for the IDC for each test.
- The laboratory shall retain documentation of each person's IDC.



Initial Demonstrations of Capability (IDCs)

What is an IDC, and what do I have to do for it?

- Document IDC requirements in any of these:
 - Each test's SOP
 - In the QM
 - Separate IDC only document

- This is a one-time requirement for each analyst per test.

Initial Demonstrations of Capability (IDCs)

What is an IDC, and what do I have to do for it?

- Example parts of the IDCs
 - Read the reference method
 - Read the lab SOP
 - Shadow an analyst that has passed an IDC
 - Perform the test being shadowed by an analyst that passed an IDC
 - Pass the quality control samples in the method

NH₃ and TP

When and how do I have to do pH checks?

- Some required pH checks
 - BOD – pH of sample in the sample bottle
 - TP – pH of acid preserved sample in the sample bottle
 - TP – pH of neutralized sample
 - NH₃ – pH of acid preserved sample in the sample bottle
 - NH₃ – pH of neutralized sample (Hach TNT+)

NH₃ and TP

When and how do I have to do pH checks?

- Don't use a pH meter due to the risk of contamination.
- It is best to use narrow range pH paper.
- Frequency of checking pH:
 - BOD – each analysis day
 - TP – quarterly if effluent doesn't vary
 - NH₃ – quarterly if effluent doesn't vary

NH₃ and TP

When and how do I have to do pH checks?



NH₃ and TP

How and what do I have to document for pH checks?

- Measuring the pH is just half the job.
- Need to make sure that the measured pH is the pH needed.
- Need to make sure that the pH was documented.
- Should be documenting the ID of any acid or base used to adjust pH.
- Should be documenting the analyst who performed the pH checks.

NH₃ and TP

How often is a calibration curve required?

- At the beginning of implementing a new test.
- When an ICV (second source) fails.
- When a CCV fails (unless a passing second CCV is analyzed right after the failed CCV).
- When the instrument leaves the lab and comes back.
- When something occurs to the instrument that would significantly affect the response (such as placing a new lamp in the spectrophotometer).

NH₃ and TP

Is a CCV required before the method blank?

- A CCV is required at the beginning of each analysis for TP and NH₃.
- The CCV is analyzed after the zero blank (instrument blank) is analyzed on the spectrophotometer.
- Performing the CCV first demonstrates the instrument is in control before any other measurement is made (i.e. method blank).

NH₃ and TP

Is a CCV required before the method blank?

Sample	pH adjust 6-8 (Y/N)?	Sample Vol. (mLs)	Sample + DI Vol. (mLs)	Dilution Factor	Absorbance	Total Phosphorus (mg/L)	Final Total Phosphorus (mg/L)	True Value (mg/L)	Quality Control
Calibration Blank				1					
Standard 1				1					
Standard 2				1					
Standard 3				1					
Standard 4				1					
Standard 5				1					
Standard 6				1					
Standard 7				1					
Standard 8				1					
ICV								Criteria: 90-110%	
LCS/CCV								Criteria: 90-110%	
Method Blank								Criteria: <LOD	

How do I know if my desiccant is still good?

- Use a color changing desiccant that is in view of the analyst so that visual changes can be observed.
- The analyst needs to know what color the “good” dry desiccant is.
- When there is about <25% of “good” dry desiccant left is a good time to either re-dry the desiccant or add new desiccant.



TSS

How do I figure out my TSS reporting limit?

The reporting limit is based on the volume of sample filtered.

1000 (mg)

Volume Filtered (mL)

TSS

How do I figure out my TSS reporting limit?

<u>Filtered volume (mL)</u>	<u>Reporting Limit (mg/L)</u>
1000	1.0
900	1.1
800	1.3
700	1.4
600	1.7
500	2.0
400	2.5
300	3.3
250	4.0
200	5.0
100	10
50	20

TSS

Can I use pre-weighed filters?

- Yes! Pre-weighed filters can be used under one condition:
 - The pre-weighed filters must be verified per the TSS method.
- For each LOT of pre-weighed filters, one method blank using a pre-weighed filter must be analyzed.
- The weight of the pre-weighed filter must be within ± 0.5 mg of the weight determined from the method blank.



TSS

What do I do if my TSS oven is reading below 103 °C?

- Adjust the oven to read a temperature between 103-105 °C.
 - If it is reading low, it might be safer to be at 104-105 °C.
- Document that you adjusted the oven temperature upwards.
- Let the TSS samples dry for at least 8 hours again.
- Samples do not need to be qualified if the temperature is in control the next day.

TSS

What do I do if my TSS oven is reading above 105 °C?

- In this case, there is no way to meet the method requirement.
- Samples must be rerun if enough sample is left, or the results must be qualified accordingly.
- Adjust the oven temperature to read 103-105 °C.
 - If it is reading high, it might be safer to be at 103-104 °C.
- Document that you adjusted the oven temperature downwards.

Do I adjust LODs and LOQs for dilution?

- YES!
- Example:
 - I had to dilute my effluent 2x (half sample, half H₂O) to get it into my curve range.
 - Normal LOD = 0.04 and LOQ = 0.1
 - With the 2x dilution, multiple the LOD and LOQ by 2.
 - Report: LOD = 0.08 and LOQ = 0.2

eDMR

How do I report sample results less than the LOD or RL?

- Ex: Result = 0.5, LOD = 1, Report: < 1
- Ex: Result = 0.056, LOD = 0.3, Report: < 0.3
- Don't report lower than your detection limit (LOD or RL).
- It is recommended to include multiple examples on how to report the results correctly in the SOP or Quality Manual and train staff.
- Another recommendation is to put a sticky note of current LODs and RLs on the computer used to enter eDMR data.

Weights

What are the weight expiration dates?

- Every 5 years, weights need to be verified, or new weights purchased.



Thermometers

What thermometer records are required?



THERMCO
PRODUCTS, INC.

ISO 9001:2015
NIST
FILA
Calibration

NIST TRACEABLE CALIBRATION CERTIFICATE

INSTRUMENT	
Catalog No:	ACC350DIG
Serial No:	92308
Calibration Date:	Oct 31, 2019
Calibration Due Date:	Oct 31, 2021
Expanded Uncertainty ±:	0.05
In Tolerance:	Yes

TEST REFERENCE:
ASTM E844-09 Standard Test Method 8.2.1 Volume 14.03.2011
Platinum Resistance Thermometer Calibration.

PHYSICAL EXAMINATION:
The physical integrity of the thermometer was verified and inspected for any malfunctions of the digital readout and any defects. Upon completion, it was determined the thermometer is suitable for calibration.

NIST TRACEABLE CALIBRATION EQUIPMENT:

Instrument	Serial Number(s)
Fuke 1594A	070365
Fuke 5628	4167, 4164, 4165, 4169, 4161, 4162

The instrument listed above is part of an unbroken chain of calibration reference standards.

CALIBRATION APPROVED BY: R. CASARIO

This certificate may not be reproduced without the express written approval of THERMCO PRODUCTS, INC.

For recertification information go to: www.thermcoproducts.com/Recertification.html

www.ThermcoProducts.com
10 Millpond Drive Unit #10 Lafayette, NJ 07848 • Phone: 973.303.5100
CL-102 Rev. 08 boxed 8-2018

Thermometers

What thermometer records are required?

Revision 0

7/11/1905

THERMOMETER CALIBRATION RECORD*

FREQUENCY: 12 MONTHS

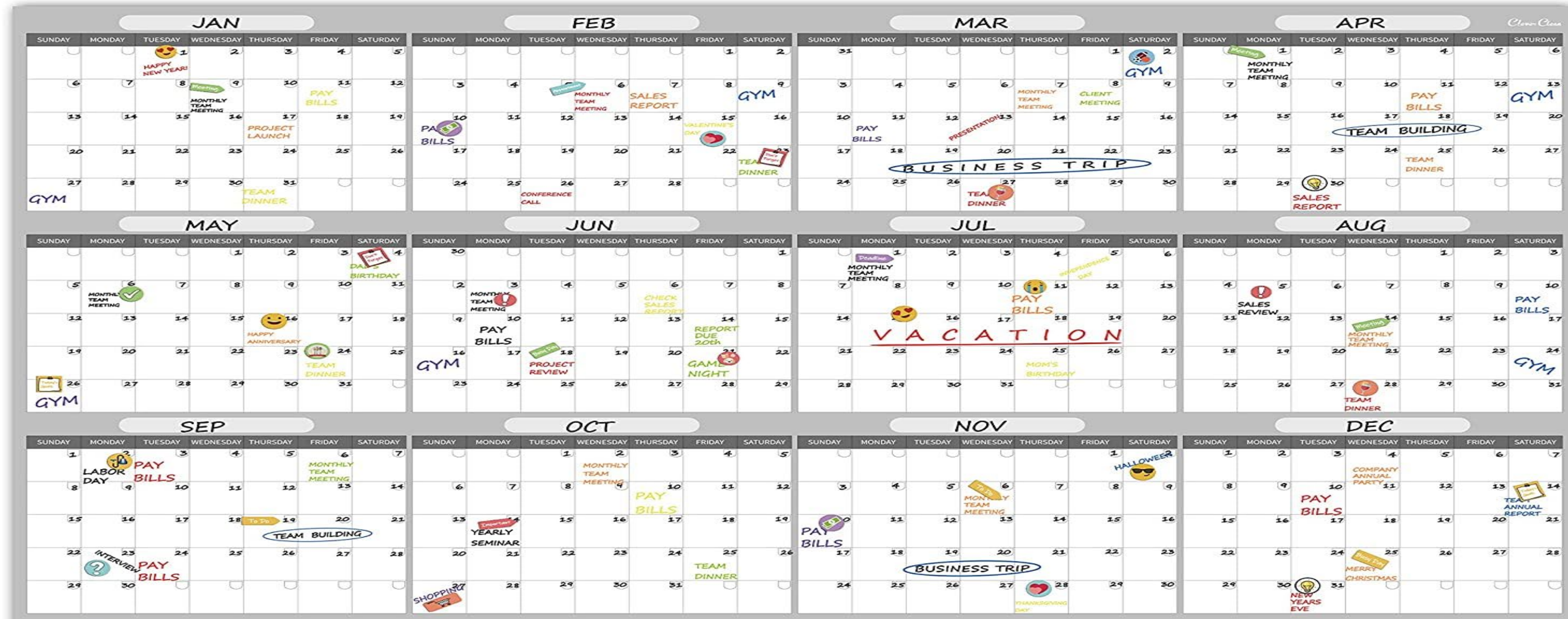
Master thermometer: Certified or NIST Traceable Thermometer Serial #: _____ Expiration date: _____

Date	Location of reference Thermometer	Serial # of Thermometer	Master Certified or Traceable Temp Reading (°C)	Master Certified Thermometer Correction Factor (°C)	Master Certified Thermometer Corrected Reading (°C)	Lab Reference Thermometer Reading (°C)	Correction Factor (°C)**	Initials
example 5/10/18	BOD incubator	15708	20.0	N/A no CF on Master	N/A no CF on Master	20.1	N/A if < 0.5	BAM

Periodic Tasks Tricks

How can I remember to perform infrequent tasks?

Create an annual to-do list that is in an area that will be frequently viewed, and have a system to mark them done when completed.



Periodic Tasks Tricks

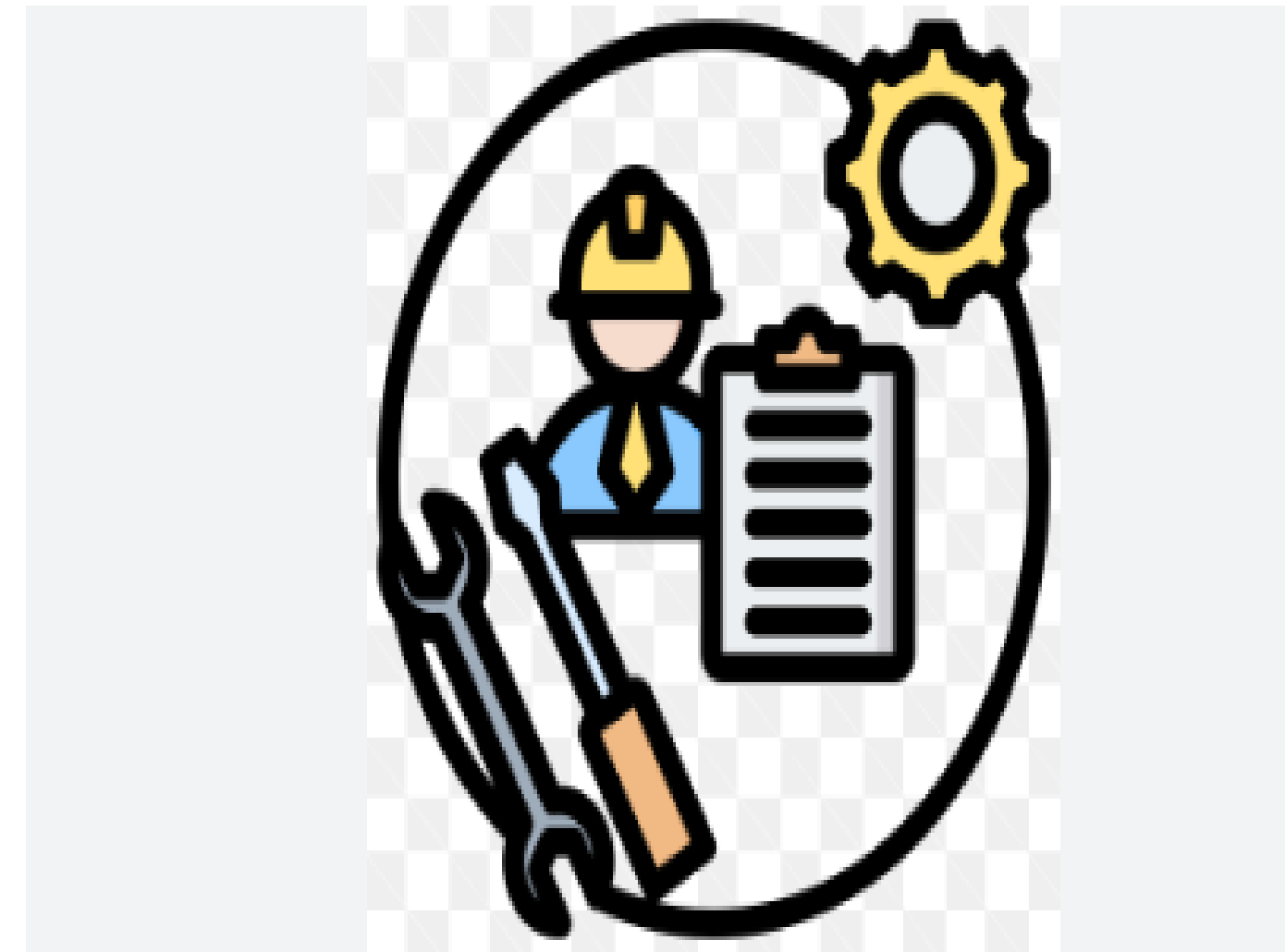
How can I remember to perform infrequent tasks?

- Use your Outlook calendar to set reminder pop-ups.
- Use your SCADA software or other maintenance software to record these items so that they pop up as a reminder.
- Create a monthly checklist with the items to do and a place to mark them complete. Place the checklist in an area that is frequently viewed.

Maintenance

What maintenance items are required?

- Balance weights – every 5 years
- Thermometers – every year
- Barometer – every year
- Mechanical pipettors – every quarter
- Balances checked – every month



Maintenance

What maintenance items are required?

- Change cap or membrane on BOD probe – as occurs
- Change membrane on ISE probe – as occurs
- Cleaning (probes, filter disks, water system) – as occurs
- Replacing tubing or equipment – as occurs

Maintenance

Where should I document all my maintenance items?

Lab Maintenance Log

Instructions: Complete this form and save it. Whenever maintenance is performed on any piece of laboratory equipment record that information below. If you need more than one line you can just continue on the next line. Include as much detail as you can. You do not have to limit yourself to one line per entry.

Common lab equipment which require maintenance (there are others):

pH meter / electrode	Incubator	DO meter barometer	Analytical Balance	Composite Sampler Tubing
TSS oven	DO meter / probe	NH3-N meter / electrode	Desiccators	Thermometers

Date	What equipment was worked on?	What maintenance or corrective action did you perform on this equipment?	Did Fix work (Y/N)	Other Comments	Initials

Miscellaneous

Ways the Lab Cert program informs its customers:

- Emailed information bulletins
- Lab Cert Council minutes
- Website

