

WISCONSIN DEPARTMENT OF NATURAL RESOURCES

WDNR LABORATORY AUDITS

FREQUENT AUDIT FINDINGS & HOW TO AVOID THEM

October 2022 DNR.WI.GOV



HAVE YOU EVER WONDERED WHAT AUDIT
FINDINGS OTHER LABORATORIES ARE BEING
CITED FOR?

**Is everyone enforcing the rules the
same way?**

Fair Play

- ✓ **All auditors use the same checklists.**
- ✓ **Potential findings are reviewed by all staff.**
- ✓ **The section chief reviews all final reports.**
- ✓ **Auditors meet weekly to go over questions.**
- ✓ **Each lab is provided a survey after the audit.**

- ❑ **Total labs audited = 77**
- ❑ **Time period = 1 year**
- ❑ **9.1.21 to present (new code)**
- ❑ **Average findings = 3 (range of 0 – 9)**
- ❑ **Labs with no findings = 13 (17%)**
- ❑ **Municipal & industrial WWTF (TSS, BOD, TP, NH₃-N)**
- ❑ **5 auditors**

TIME FOR ANSWERS



HERE ARE YOUR TOP 15 MOST FREQUENT FINDINGS...

... AND EXAMPLES FOR HOW TO AVOID THEM.

Finding 15

(6% of audits)

Quality control failures occur but they are not documented on the eDMR.



HOW TO AVOID – use 1 or more of these options

Quality control failures occur but are not documented on the eDMR.

- Highlight the QC failures on the benchsheet using color highlighters.
- Have special places on the benchsheets to record QC failures.
- Review all benchsheets and QC failures at the time of eDMR entry.
- At the end of each day, document on a piece of paper all QC failures encountered and review this paper when you fill out the eDMR.
- Have a 2nd person review eDMR entry for missing QC failures.
- Make sure the person entering the eDMR data is trained and in agreement with the process used to identify QC failures.
- Have this reminder – Did I enter my eDMR quality control failures?
J, F, M, A, M, J, J, A, S, O, N, D – crossing off each month when done.

Finding 14

(6% of audits)

TSS analysis information is not documented.

- Analyst in and out of oven
- Date in and out of oven
- Time in and out of oven
- Temperature in and out of oven

**Incomplete or missing
documentation**

TSS analysis information is not documented. (real life example)

TSS benchsheet

Method Number: SM 2540 D-1997

Read In Weights		Read Out Weights	
Analyst		Analyst	
Sample Date	10-27-21	KS	
Date Samples In	10-28-21	Date Samples Out	
Time Samples In	8:15	Time Samples Out	
Temp. °C	104.3	Temp. °C	

Control Limits	
Minimum Sample Wt	0.1
Maximum Sample Wt	0.5

Sample ID (be unique id)	Filter, Crucible or Pan Number	Sample Volume Used (mL)	Sample plus container weight after 1st dry (g)	Sample container weight (g)	Sample Weight (g)	TSS (mg/L)	Sample plus container weight after re-dry (g)	Wt b
Effluent	1	500	1.4447	1.4423	.0024	4.8		
Effluent	2	30	1.4461	1.4432	.0029	96.6		
Upoto	3	30	1.4411	1.4381	.0003	100.0		
AT#3	4	10	1.5060	1.4426	.0634	634.0		

HOW TO AVOID – use 1 or more of these options

TSS analysis information is not documented.

- Have specific fields on your benchsheets to enter this information.
- Put a note on your TSS oven front door to check that your benchsheet has been completed.
- Before you leave for the day, double check your benchsheets to make sure all fields are completed.
- Have a 2nd person review your work at the end of the day.
- Complete temperature in and out on benchsheet instead of temperature log.

Finding 13

(8% of audits)

An expired weight is used.

- Every 5 years, the weight needs to be re-verified or a new weight purchased.
- Records missing

**Incomplete or missing
documentation**



HOW TO AVOID – use 1 or more of these options

An expired weight is used.

- If you receive weight certificates, which you should, include them in your quality manual or TSS SOP so you can always find them.
- Use your outlook calendar to set a reminder to have them verified before the 5-year expiration date.
- Use a paper calendar to set a reminder to have them verified before the 5-year expiration date (keep at end of year).
- Put a reminder on the box that holds the weights, so you see it every time you use them.

Finding 12

(8% of audits)

The analytical balance is not verified monthly.

- Not performed
- Not documented

Incomplete or missing documentation



HOW TO AVOID – use 1 or more of these options

The analytical balance is not verified monthly.

- Create a monthly to-do list for the lab and place it in an area that will be frequently viewed.
- Provide fields for “checking off” work when it is completed.
- Have a 2nd person verify work has been completed.
- Have this reminder – Did I verify my balance this month and record it?
J, F, M, A, M, J, J, A, S, O, N, D – crossing off each month when done.



Finding 11

(8% of audits)

TP and NH₃-N standards and samples are not stored separately.



HOW TO AVOID – use 1 or more of these options

TP and NH₃-N standards and samples are not stored separately.

- Do your best to separate the standards from the samples by storing the standards in a separate, sealed container or bag.
- Any Tupperware or plastic container will do.
- Zip-loc bags are ok too.



Finding 10

(8% of audits)

**A CCV is not analyzed
before the method blank
(TP, NH₃-N).**

Finding 9

(9% of audits)

**Corrective actions were not taken
or
they were taken, but not documented.**



CORRECTING A PROBLEM

MEANS

**CORRECTING THE
PROCESS**

**Incomplete or missing
documentation**

Corrective action is not documented. (real life example)

Log Sheet for Corrective Lab Procedures

Date	Analyst	Lab Test Performed Failed	Corrective Action Taken	Did Corrective Action Work
1-15-20	B.S.	GGA	Bleach Bath Pipets and Bottles	
7-15-20	Z.S.	GGA	Cleaned Pippets	
7-22-20	B.S.	GGA	Changed Bleach Water.	

HOW TO AVOID – use 1 or more of these options

Corrective actions were not taken/documented.

- On your benchsheet, include a checkbox or use color highlighters to indicate that corrective action was completed and documented.
- Have a 2nd person review the benchsheet to make sure corrective actions and documentation have been completed.
- When you complete the eDMR, make sure you check that corrective actions were documented for each one of the QC failures.
- Train everyone about corrective action and documentation.
- Have this reminder – Did I complete my corrective action records?
J, F, M, A, M, J, J, A, S, O, N, D – crossing off each month when done.

Finding 8

(10% of audits)

The barometer is not verified annually or is not documented.

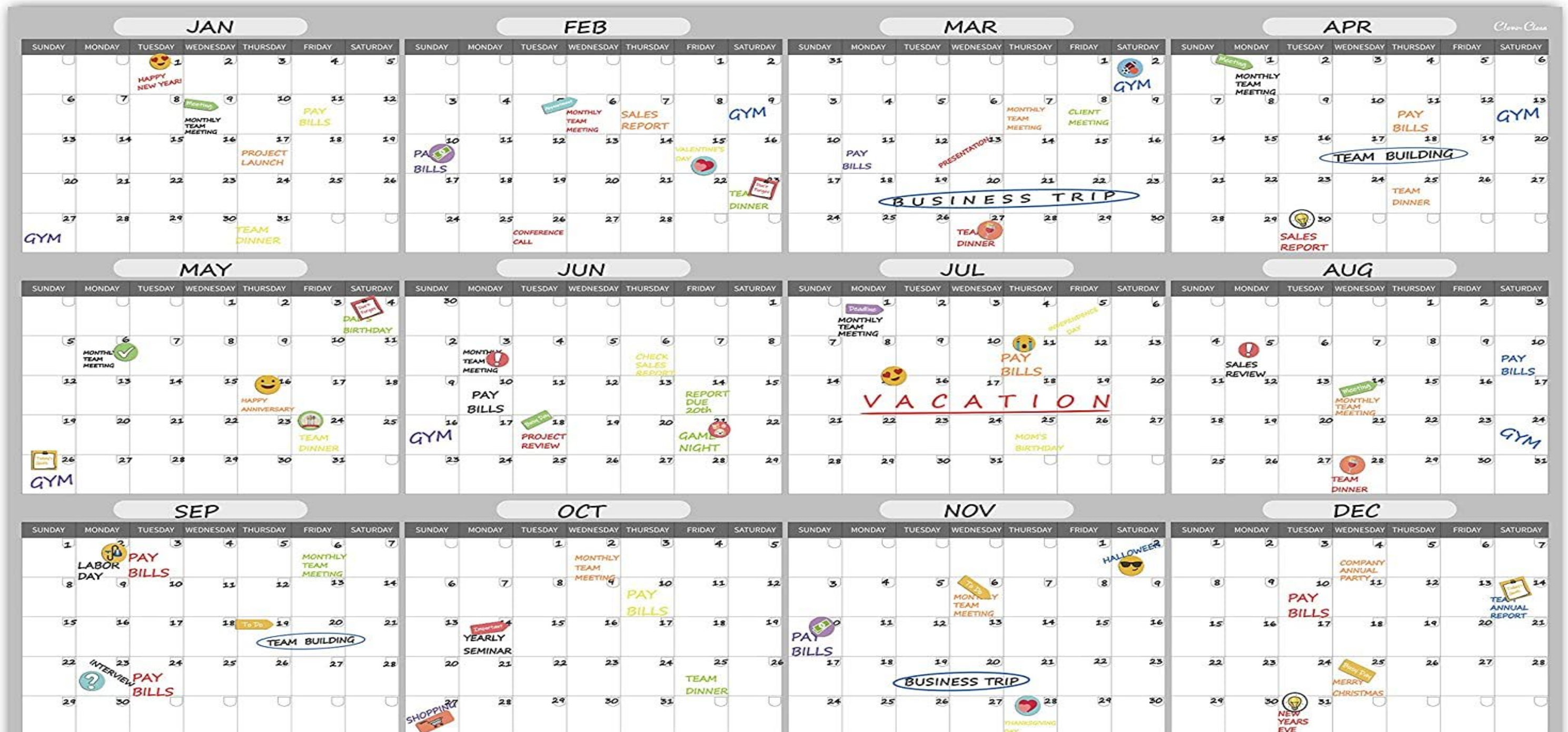


**Incomplete or missing
documentation**

HOW TO AVOID - use 1 or more of these options

The barometer is not verified annually or is not documented.

Create an annual to-do list for the lab that is in an area that will be frequently viewed and provide fields for checking work when it is completed.



HOW TO AVOID – use 1 or more of these options

The barometer is not verified annually or is not documented. (cont.)

- Have a 2nd person verify it has been completed.
- Have this reminder – Did I verify my barometer this month and record it?
J, F, M, A, M, J, J, A, S, O, N, D – crossing off each month when done.

Finding 7

(12% of audits)

Results that are less than the LOD are not reported as “<” on the eDMR.

- 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.

HOW TO AVOID – use 1 or more of these options

Results that are less than the LOD are not reported as “<” on the eDMR.

- In your Quality Manual, include multiple examples of raw data and how to report the results correctly.
- Train staff using the information in the Quality Manual.
- Put a sticky note of current LODs on the computer used to enter eDMR data.
- Have a 2nd person verify results are reported correctly.

Finding 6

(13% of audits)

BOD analysis information is not documented.

- Nutrients added
- DO meter calibration value
- Temperature and pressure
(theoretical saturation point recommended)

**Incomplete or missing
documentation**

BOD analysis information is not documented.

(real life example)

Nutrient Pillow ID (for 300ml)		A9289		Sample Dates/Times <u>3-11-21</u> Collected By <u>CS</u>				DO Depletion ≥ 2.0 mg/L	
Seed ID or Sample Date of Influent								Room/Sample Temp 20 \pm	
GGA ID		B12D200306B2							

Read in Calibration		Read out Calibration	
Analyst	TIC	Analyst	CS
DO Std Temp ($^{\circ}$ C)	20.4	DO Std Temp ($^{\circ}$ C)	20.6
Local Pressure (mm Hg)	745	Local Pressure (mm Hg)	734
DO Satr from T/P (mg/L)	8.43	DO Satr from T/P (mg/L)	8.66
Theo. DO Satr/chart (mg/L)	8.81	Theo. DO Satr/chart (mg/L)	8.66
Room Temp ($^{\circ}$ C)	21	Room Temp ($^{\circ}$ C)	21.0
Date bottles in	3-12	Date bottles out	3-17-21
Time Bottles in	1:00	Time Bottles out	1:00

Composite Sample Checks		
Sample ID	pH	Temp prior to dilution ($^{\circ}$ C)
Dilution Water	-----	
Final effluent		
Raw Influent		
Saputo		
Seed		

Code Definitions											
N = Extra Nutrients added to											
I = Inhibitor added to sample											
P = Sample pre-diluted per											

Sample ID (must be unique)	Bottle ID	Sample Volume Used (mL)	Pre-dilution Factor (if other than 1)	Seed Volume added (mL)	Initial DO (mg/L)	Final DO (mg/L) ≥ 1.0	DO Depletion (mg/L) ≥ 2.0	Seed Control CF (mg/L)	DO Depletion - Seed Contribution (mg/L)	Dilution Factor	BOD (mg/L)
Blank	1	-			8.59	8.54	0.15				5.89
Final	2	200			8.72	4.79	3.93				4.77
Final	3	250			8.76	4.78	3.98				4.06
Final	4	300			8.81	4.75	4.06				119.4
Final					8.53	4.54	1.99				21.3

BOD analysis information is not documented.

(real life example)

Read in Calibration		Read out Calibration		Composite Sample Checks					
Analyst	CS	Analyst	CS	Sample ID	pH	Temp prior to dilution (°C)			
DO Sat Temp (°C)	20.3	DO Sat Temp (°C)	20.3	Dilution Water					
Local Pressure (mm Hg)	739	Local Pressure (mm Hg)	778	Final effluent					
DO Sat from T/P (mg/L)	8.46	DO Sat from T/P (mg/L)		Raw Influent					
Theo. DO Sat/chart (mg/L)	8.64	Theo. DO Sat/chart (mg/L)		Septa					
Room Temp (°C)	23.0	Room Temp (°C)	21.5	Seed					
Date bottles in	4-30-21	Date bottles out	5-5-21						
Time bottles in	10:30	Time bottles out	4:00						

Sample ID (must be unique)	Bottle ID	Sample Volume Used (mL)	Pre-dilution Factor (if other than 1)	Seed Volume added (mL)	Initial DO (mg/L)	Final DO (mg/L) ≥ 1.0	DO Depletion (mg/L) ≥ 2.0	Seed Control CF (mg/L)	DO Depletion - Seed Contribution (mg/L)
Blank	1	0			8.53	8.46	.07		
Final 150	2	150			8.68	4.77	3.91		
Final	3	250			8.79	3.45	5.34		
Final	4	300			8.89	1.21	7.67		
		0			8.16	5.93	2.23		

BOD analysis information is not documented.

(real life example)

Date on: 11-18-21 Date off: _____ Time on: _____ Time off: _____
 Analyst on: B.S. Analyst off: _____
 Sample Temp Inf: 20^o
 Sample Temp Eff: 20^o

	Bottle No.	mls sample	Add Nutr.	Dissolved Initial	oxy. Final	Diff ence	Dilu tion	BOD	Valid Test
Blanks	12	 	 	8.45	8.42				
	18	 	 	8.46	8.41				
Raw Inf.	11	3	 	8.41	4.67	3.74	100	374.0	
	13	5	 	8.37	2.04	6.33	60	379.80	
			 						

HOW TO AVOID – use 1 or more of these options

BOD analysis information is not documented.

- Have specific fields on your benchsheets to enter this information.
- Put a note on your DO meter to check that your benchsheet has been completed.
- Before you leave for the day, double check your benchsheets to make sure all fields are completed.
- Have a 2nd person review your work at the end of the day.

Finding 5

(14% of audits)

Thermometers are not verified annually, or certificates are missing.

Calibration complies with ISO/IEC 17025, ANSI/NCSL Z540-1, and BIPM

Instrument Identification:
Model: 6500 S/N: 170782841 Manufacturer: Control Company

Standards/Equipment:

Description	Serial Number	Due Date	NIST Traceable Reference
Calibration Bath TC-329	85A267		
Platinum Resistance Thermometer	4327	7/07/18	87710051
Digital Thermometer	ASA393	5/18/18	1000409448
Temperature Calibration Bath TC-191	A42238		
Thermistor Module	A27129	12/01/17	1000401760
Temperature Probe	5394	9/25/18	87919117
Temperature Calibration Bath TC-231	A79341	12/01/17	1000401760
Thermistor Module	A27129	12/01/17	1000401760
Temperature Probe	5267	12/06/17	86830059

Certificate Information:
Technician: 104 Procedure: CAL-6500 Cal Date: 11/08/21 Due Date: 11/08/23
Test Conditions: 23.1°C 57.0 %RH 1020 mBar

Calibration Data: (New Instrument)

Unit(s)	Nominal	As Found	In Tol	Nominal	As Left	In Tol	Min	Max	±U	TUR
P1 °C	N.A.	-40.002	-39.99	Y	-40.25	-39.75	0.074	3.4:1		
P2 °C	N.A.	-40.002	-39.98	Y	-40.25	-39.75	0.074	3.4:1		
P1 °C	N.A.	0.002	0.00	Y	-0.25	0.25	0.074	3.4:1		
P2 °C	N.A.	0.002	0.00	Y	-0.25	0.25	0.074	3.4:1		
P1 °C	N.A.	49.999	49.99	Y	49.75	50.25	0.074	3.4:1		
P2 °C	N.A.	49.999	49.99	Y	49.75	50.25	0.074	3.4:1		

This instrument was calibrated using instruments traceable to National Institute of Standards and Technology.

Maintaining Accuracy:
In our opinion, once calibrated, your 8071 Digital Thermometer should maintain its accuracy. There is no exact way to determine how long calibration will be maintained. 8071 Digital Thermometers change 0.1°C, if any at all, but can be affected by aging, temperature, shock, and contamination.

Recalibration:
For factory calibration and re-certification traceable to National Institute of Standards and Technology contact Control Company.

Incomplete or missing documentation



Thermometers expired. (real life example)

THERMCO PRODUCTS, INC.  

Accreditation No. 49474
to 84937, 1783, 1861

NIST TRACEABLE CALIBRATION CERTIFICATE

INSTRUMENT		TEST REFERENCE:	
Catalog No	ACC02018LS	NIST Publication 250-22 Liquid in Glass Thermometer Calibration	
Serial No	34948	PHYSICAL EXAMINATION: The physical integrity of the thermometer was verified for any defects in structure. Upon completion, it was determined the thermometer is suitable for calibration.	
Calibration Date	Jan/04/2016	CALIBRATION EQUIPMENT:	
Calibration Due Date	Jan/04/2017	NIST Traceable Instrument	NIST Calibration Report Number
Expanded Uncertainty \pm	0.11	Hot Block/Block 2542/13	46 50001 VT00000
In Tolerance	Yes	Block PIT	46 50001 VT99000
		46 2542, 1046, 1090, 600006, 603277	

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

Calibration Approved By: R. CAGGIO

www.ThermcoProducts.com
10 Millpond Drive, Unit #10, Lafayette, NJ 07848 - Phone: 873.380.9100

RECERTIFICATION PROGRAM

When should a Thermometer be Recertified?
To determine recertification of thermometers, such as usage, temperatures measured, in-house procedures, and agency guidelines and policies.
Agency guidelines for good laboratory and manufacturing practices, thermometers should be recertified on an annual basis.

Recertification Program Consists of...
Visual inspection for any physical damage.
Thermometers will be repaired if possible.
Thermometers are compared against NIST Standards in high precision baths using the comparison method at determined test intervals.

Order Recertification:
Copy of the current calibration certificate, complete the information on the bottom of the copy and carefully pack in a sturdy container (do not send liquid filled bottles), along with the certificate and payment method, we accept purchase order - check - Visa & MasterCard. to: Thermco Products Inc, 10 Millpond Drive, Unit #10, Lafayette, NJ 07848

NCL - (2) R-020
(2) TB-1C

SHIP TO (if different than Bill To)

Name _____
Dept. _____
Address _____
City _____
State _____ Zip _____
Phone _____

HOW TO AVOID – use 1 or more of these options

Thermometers are not verified annually/documentated.

- If you receive thermometer certificates, which you should, include them in your quality manual so you can always find them.
- Use your outlook calendar to set a reminder to have them verified before they expire.
- Use a paper calendar to set a reminder to have them verified before they expire.
- Put an actual expiration tag on each thermometer so you see it each time you use it as a reminder.
- Put a reminder on your annual to-do list to complete this.

Finding 4

(17% of audits)

SOPs are incomplete.

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.

Incomplete or missing documentation



S T A N D A R D
O P E R A T I N G
P R O C E D U R E

SOPs incomplete. (real life example)

BOD (SOP) Procedure

BOD testing is performed on Wednesday, Thursday, and Friday. Samplers are turned on the day before the testing, to get a 24-hr sample. The day of the test, collect samples, and warm them to 17-23°C. Warm up DO meter (see QC manual for procedure). Next prepare dilution water, using BOD buffer pillows for 3 liters of dilution water. Determine dilutions required (usually we use between 100ml to 250ml) When using 200ml or more, we add a BOD nutrient buffer. Measure out samples, fill bottles about ¼ full with dilution water, and your seed, then top off with dilution water. Measure initial DO (DO_i), incubate for 5 days at 20°C, then measure final DO (DO_f). Determine BOD.

Calculating BOD

$$\text{BOD (mg/L)} = (\text{DO}_i - \text{DO}_f) \times \text{DF}$$

DO_i = Initial DO (mg/L)

DO_f = Final DO

DF (dilution factor) =

$$300 \text{ mL} \div \text{sample volume (mL)}$$

TSS (SOP) Procedure

TSS testing is performed on Wednesday, Thursday, and Friday. On Tuesday we prepare all our glass-fiber filters (47mm) by inserting disk wrinkled side up in filtration apparatus. Apply vacuum and wash disk with three 20-mL of distilled water. Remove filter from vacuum, and transfer to an aluminum weighing dish. Dry in the oven at 103 to 105°C for 1 hour, we leave them in the oven until test day. On day of test, we take 3 filters out of the oven (1) for effluent, (1) for influent, and (1) for mlss, and place them in a desiccator to balance temperature and weight. Begin by weighing for initial weight, then put disk in the filtration apparatus, next wet filter with a small volume of distilled water, then add sample volume of 1000mL for effluent, (500mL is effluent is cloudy) 25mL for influent, and 10mL for mlss. Wash filter (3) times with 10mL volumes of distilled water, remove the disk, place in the aluminum dish and place in the oven, we keep them in over night and weigh them the next day. On the next day, take them out of the oven and place in the desiccator to balance temperature and weight. Weight them to get your final weight.

$$\text{Mg total suspended solids/L} = \frac{(\text{initial weight} - \text{final weight}) \times 1000}{\text{Sample volume, mL}}$$

Blanks - No MORE than .20 Drop from Initial Read.

INF. Bottle - At Least a 2.00 Drop from Initial Read
MORE than 1.00 on final Read

EFF. Bottle - Same as INF

Dilution - $300 \text{ mL} \div \text{SAMPLE SIZE}$

BOD - Dilution \times Difference of Initial and Final Read.

Mondays - read bottles + test ammonia

Tuesdays - read ~~and make~~ bottles + rinse disc + PH Sample EFF
Make disc + Make ammonia 2.5ml acid

Wed. read bottles + make bottles, read disc, + make disc + ammonia
prep 900ml raw for

thur - make bottles, read disc + make disc + ammonia
Final has to be 20°
Make GGA

Fri - make bottles, read disc + make disc

Sat - read disc

press F2, Channel 1 press F2

ISA buffer 2ml in Standards + 2ml in Samples + add until blue

press F3 to start

* Need to make 7 bottles - .2 bottle - 100 ml .2 Standard, 2.0 bottle - 100 ml 20 Standard, 2.0 bottle - 100 ml 20 Standard.

• 3 Sample bottles - 100 ml

• 10 bottle - 50 ml 20 Standard + 50 ml DI water

Heat 20°
Cal done
log print

SOPs incomplete. (real life example)

Ammonia ISE Procedure

Make sure you are turned out!

1. Pour out 50 or 100 mL of sample.
2. Place the electrode in the beaker.
3. Turn on the magnetic stirrer.
4. Set the meter to begin reading.
5. Add 1 mL of buffer solution (typically 10 M NaOH).
(SM Buffer solution is based on 1 mL per 100 mL of sample)
(Orion buffer is based on 2 mL per 100 mL of sample)
(Orion buffer stays blue if the sample pH is > 11)
6. Read millivolts and concentration (if using software).

Ammonia ISE Procedure

- Keep the electrode at an angle to minimize air bubbles
- Stir at the same speed for standards and samples.
- Prevent heating the solution; insulate between beaker & stirrer
- Do not add NaOH before immersing electrode
—ammonia is in gaseous form at this pH and will be lost!

Ammonia ISE Procedure

- Electrodes work slightly differently than other analyses
- Response follows a logarithmic pattern (Nernst equation)
- Nernst's law: slope **MUST** be -54 to -60 mV (target = -59)

NOTE: If your full calibration range is TWO *decades, (e.g., 0.1 to 20), the difference in mV between the upper and lower standard should be $2 \times 59.2 = 117\text{mV}$

- Procedure identical to regular linear regression except:
....**MUST** first convert concentration to logarithm
.... Then, a second conversion is required to convert to mg/L

HOW TO AVOID – use 1 or more of these options

SOPs are incomplete.

- We provide template SOPs for BOD, TSS, TP, and NH₃-N tests.
- Ask any of the lab cert staff for a copy of the template.
- NR 149.40 provides a checklist of all items that need to be addressed in the SOP.
- Review the SOP and update it (for your laboratory) well once, and then updates are only needed when things change.
- Try to be detailed enough that a backup operator can easily follow the SOP and would do the test the same way as the primary operator.
- Can use as a training tool and will be a great reference for testing.

Finding 3 (17% of audits)

Incomplete or missing
documentation

TP and NH₃-N sample pH (< 2) is not verified or documented.



HOW TO AVOID – use 1 or more of these options

TP and NH₃-N sample pH is not verified/documented.

- Record the pH measurement checks on your yearly to-do list.
- Have a 2nd person verify it has been completed.
- Use your outlook calendar to set a reminder to have them verified.
- Have this reminder – Did I verify TP and NH₃-N sample pH < 2 each quarter and record it?
Q1, Q2, Q3, Q4 – crossing off each quarter when done.
- Just do it each analysis day.

Finding 2

(22% of audits)

Benchsheets are prepopulated with raw data.

- Sample volumes
- Names/initials
- Dates

Raw data shall be recorded at the time it occurs.

HOW TO AVOID – use 1 or more of these options

Benchsheets are prepopulated with raw data.

- Best option: Just don't do it.
- Look at your template benchsheet and remove any prefilled-out information in the following fields:
 - Sample volumes
 - Names/initials
 - Dates/times
- On your benchsheets, include a statement such as, “any prefilled data will be verified prior to analysis and, if necessary, will be crossed out and the correct data written in and initialed.” Also indicate which data will be prefilled in each SOP along with the notation above.

Finding 1 – by a landslide

(42% of audits)

LOD studies are not performed as required.

- Not performing 2 spikes per quarter.
- Not performing the 2 spikes in different batches.
- Not using 2 years of spike data (using more or less).
- Not using 2 years of method blank data – or the greater of the last 50 or last 6 months.
- Method blanks recorded as 0 instead of negative.
- Using the initial LOD instead of the ongoing LOD.

HOW TO AVOID – use 1 or more of these options

LOD studies not performed as required.

- This being the winner for most frequent finding is not a surprise.
- There are a lot of moving pieces to it.
- Use the LOD benchsheet from lab certification.
- Use and review the LOD summary form from lab certification so that you understand what is required.
- Ask someone from lab certification for help.
- Have this reminder – Did run my quarterly LOD samples Q1, Q2, Q3, Q4 – crossing off each quarter when done.
- Put a reminder on the benchsheet to calculate my LOD by _____

**Incomplete or missing
documentation**

**If it wasn't documented, it
wasn't done.**