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Subject: Washing of Glassware and Plasticware

Approval

Title	Name	Signature	Date
Laboratory Supervisor	Kelley E. Keenan		06-01-25
Quality Assurance Officer	Jim Sumner		06-01-25

Document Revision History

Effective Date	Revision number	Review Type	Evaluators	Revisions
12-01-00	0	Internal	Jim Sumner (ETS)	Original document
12-01-11	1	Internal	Jim Sumner (ETS)	• Added quality control requirements for disposable plasticware used in toxicity tests.
09-28-16	2	External	Rick Sherrard (TVA) Don Snodgrass (TVA) Jim Sumner (ETS)	• Updated references to include Standard Methods 22nd Edition and NELAP standard. • Clarified SOP to indicate that term glassware includes any new or used item in the laboratory, which may include glassware, plasiticware, nitex mesh, field equipment, etc.
03-01-20	3	Internal	Jim Sumner (ETS)	• Updated procedure to include NELAP requirements.
06-01-25	4	Internal	Jim Sumner (ETS)	• Updated procedure during document review.

Subject: Washing of Glassware and Plasticware

Scope and Application

To clean dirty glassware, plasticware, nitex mesh, and field equipment (The term glassware in this SOP refers to any new or used items in the laboratory, which may include afore mentioned items).

Quality Control

Each batch of items washed must be checked for soap residue using 0.04% bromothymol blue indicator.

Equipment and Materials

Alconox or Sparkleen™ 1 and 2 detergent powder
Nitric acid
Sodium bicarbonate
Tap water
Deionized water
Bristle brushes
Scouring pads
30-gallon plastic tub
Rubber gloves
Safety glasses
Sink
Dishwasher
0.04% Bromothymol blue indicator
Storage area for clean glassware
Acetone, pesticide grade
Rinse bottles
Fume hood
Detergent Residue Check Log

Procedure

Note: Gloves and safety glasses must be worn while using nitric acid and acetone.

A. Preparation of the Nitric Acid Bath (10% nitric acid solution).

1. Add 22.5 L of deionized water to a 30-gal plastic tub.

2. Carefully add 2.5 L of nitric acid to the deionized water and mix.

Subject: Washing of Glassware and Plasticware

B. Washing of Glassware and Plasticware.

1. New disposable glassware/plasticware do not require thorough cleaning before use. It is sufficient to rinse new sample containers with sample or dilution water before use. New non-disposable glassware or plasticware must be soaked overnight in 10% nitric acid (see procedure below) and rinsed well with deionized water.
   - New disposable sample containers (i.e. cubitainers), medicine cups, and Solo® cups must be checked before use in toxicity tests. Dependent on the use of the consumable, either the survival/reproduction of *Ceriodaphnia* or survival/growth of *Pimephales* are evaluated using the new consumable. Side-by-side comparisons of the organisms with the new and old consumables are used (SOP-AT11 or AT20). Organism survival and reproduction/growth are compared between the old and new consumables. If detrimental effects are noted with the new consumables, they must not be used in toxicity tests.

2. All non-disposable glassware/plasticware that comes in contact with samples must be washed after use to remove contaminants in the manner described below.

   If equipment is washed by hand:

   a. Add a small amount of Alconox or Sparkleen™ 1 detergent (~ 1 Tablespoon) to the sink and fill with hot tap water. Place the dirty glassware in the sink and allow it to soak for at least 15 minutes.

   b. Using scouring pads and bristle brushes, scrub the dirty glassware until it is clean.

   c. Immediately rinse the soapy glassware well with hot tap water.

   d. Test for soap residues using the 0.04% bromothymol blue indicator. Choose a random piece of glassware and place a few drops inside the vessel. If the indicator turns blue, repeat steps a-c. Document the positive/negative result in the Detergent Residue Check Log along with date, analyst initials, and indicator chemical number (Exhibit G1.1).

   If equipment is machine-washed:

   a. Add a small amount of Alconox or Sparkleen™ 2 detergent to the soap tray in the dish washer.

b. Start the dish washer.

c. After the dish washer's cycle is complete, test for soap residues using the 0.04% bromothymol blue indicator. Choose a random piece of glassware and place a few drops inside the vessel. If the indicator turns blue, repeat steps a-b. Document the positive/negative result in the Detergent Residue Check Log along with date, analyst initials, and indicator chemical number (Exhibit G1.1).

To remove scale and metals:

a. Soak clean glassware in the 10% nitric acid bath. Remove the glassware from the nitric acid bath and rinse well with deionized water.

To remove organic compounds (oily residues):

a. Rinse clean glassware with full-strength, acetone under the fume hood and then triple rinse with deionized water.

Place the clean glassware upside down in the storage area to dry.

Glassware/plasticware for toxicity tests should be thoroughly rinsed with dilution water immediately before use in cultures or toxicity tests.

C. Disposal of the Nitric Acid Bath.

1. Check the nitric acid bath before each use. If the nitric acid becomes dirty or turns yellow, the nitric acid bath must be renewed.

   a. Sodium bicarbonate is first used to neutralize the acid. Once neutralized, the acid bath may be disposed down the drain with tap water.

   b. Once completed, see step A.1 for the preparation of the 10% nitric acid bath.

Safety and Hazardous Waste Management

Safety glasses, gloves and lab coats should always be worn.

Review Policy-P6: General Safety Policy and Policy-P9: Radiation Protection Policy for additional safety requirements.

Subject: Washing of Glassware and Plasticware

References

Standard Methods for the Examination of Water and Wastewater, 24th Edition, 2023. American Public Health Association, 800 I Street, NW, Washington DC 20001-3710.

USEPA. October 2002. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, 5th ed. EPA-821-R-02-012. US Environmental Protection Agency, Cincinnati, OH.

USEPA. October 2002. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms, 4th ed. EPA-821-R-02-013. US Environmental Protection Agency, Cincinnati, OH.

USEPA. October 2002. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms, 3rd ed. EPA-821-R-02-014. US Environmental Protection Agency, Cincinnati, OH.

TNI Standard. Management and Technical Requirements for Laboratories Performing Environmental Analysis. EL-V1-ISO-2016-Rev2.0. The NELAC Institute, PO Box 2439, Weatherford, TX 76086.

Exhibits

Exhibit G1.1: Detergent Residue Check Log

Subject: Washing of Glassware and Plasticware

Exhibit G1.1: Detergent Residue Check Log

Page ______

Detergent Residue Check Log
(0.04% Bromothymol Blue Indicator)

Indicator CHM #_________

Date	Analyst	Detergent Residue Check	
		Positive (BLUE) Rewash Glassware	Negative (YELLOW/GREEN)

Note: B = back dishwasher, H = hand washed, No mark = front dishwasher in dish room.

SOP G7-Revision 4–Exhibit G1.1

Subject: Bottle Preparation and Quality Assurance

Approval

Title	Name	Signature	Date
Laboratory Supervisor	Kelley E. Keenan		06-01-25
Quality Assurance Officer	Jim Sumner		06-01-25

Document Revision History

Effective Date	Revision number	Review Type	Evaluators	Revisions
06-01-11	0	Internal	Jim Sumner (ETS)	Original document
06-01-25	1	Internal	Jim Sumner (ETS)	• Updated procedure during document review.

Scope and Application

To prepare bottles for clients to use for collecting samples.

Equipment and Materials

250-mL plastic bottles
500-mL plastic bottles
1000-mL plastic bottles
Trace Metals Grade Nitric Acid
A.C.S. Certified Sulfuric Acid
Glass Pipettes, pipette bulbs
Permanent red marker
Permanent black marker
Bottle labels

Subject: Bottle Preparation and Quality Assurance

Procedure

A. Ordering bottles.

1. Order unpreserved bottles from an approved vendor.

B. Preparation of bottles for chemical analyses

1. Preparation of bottles for analyses that require Nitric Acid.

   a. Clients that are collecting samples for lead and copper must be provided unpreserved 1000-mL bottles. Samples must be received by the laboratory and preserved within 14 days of collection. Upon receipt, each bottle must receive 2.0 mL of nitric acid (to be preserved for analysis). The date that the sample bottle is preserved is recorded on the top of the bottle lid with a permanent marker.

   b. The amount of nitric acid used for each bottle size is provided in the table below. Use a glass pipette to deliver the required amount of acid to each bottle.

Bottle size (mL)	Amount of Nitric acid (mL)
250	0.5
500	1.0
1000	2.0

   c. All bottles that are prepared with Nitric Acid are marked with HNO_3 on the top of the bottle lid with a permanent red marker.

2. Preparation of bottles for analyses that require Sulfuric Acid.

   a. The amount of sulfuric acid used for each bottle size is provided in the table below. Use a glass pipette to deliver the required amount of acid to each bottle.

Bottle size (mL)	Amount of Sulfuric acid (mL)
250	0.5
500	1.0
1000	2.0

   b. All bottles that are prepared with Sulfuric Acid are marked with a ~ on the top of the bottle lid with a permanent red marker.

C. Preparation of sample kits.

1. Analysis performed in house.

   a. Prepare all sample kits in accordance with the client's needs and/or permit requirements. Sample containers are prepared according to test requirements. The volume and preservation requirements for each test performed by the laboratory are provided in QAP Q5: Table Q5.1.

   b. Place a label on each bottle with the correct preservative listed. This should be written with a permanent black marker.

   a. Provide chain-of-custody forms with each kit ordered.

   b. If kits must be shipped, pack coolers to preserve the integrity of the bottles.

2. Analysis performed in a sub-contract laboratory.

   a. Sub-contract laboratories will provide all bottles to Environmental Testing Solutions, Inc. for testing that will be performed by that laboratory.

   b. Prepare all sample kits in accordance with the client's needs.

   c. Place a label on each bottle with the correct preservative listed. This should be written with a permanent black marker.

d. Provide chain-of-custody forms with each kit ordered.

e. If kits must be shipped, pack coolers to preserve the integrity of the bottles.

Safety and Hazardous Waste Management

Safety glasses, gloves and lab coats should always be worn.

Review Policy-P6: General Safety Policy and Policy-P9: Radiation Protection Policy for additional safety requirements.

References

Standard Methods for the Examination of Water and Wastewater, 24th Edition, 2023. American Public Health Association, 800 I Street, NW, Washington DC 20001-3710.

Subject: Receipt, Handling, and Storage of Samples

Approval

Title	Name	Signature	Date
Laboratory Supervisor	Kelley E. Keenan		06-01-25
Quality Assurance Officer	Jim Sumner		06-01-25

Document Revision History

Effective Date	Revision number	Review Type	Evaluators	Revisions
12-01-00	0	Internal	Jim Sumer (ETS)	Original document
09-01-09	1	External (TVA, Environmental Standard, Inc.) Internal	William Rogers (TVA) Cynthia Russell (TVA) Rick Sherrard (TVA) Rock Vitale (Environmental Standards, Inc.) Jim Sumner (ETS)	• The sample receipt log, including the assignment of project and sample numbers, is completed as samples are received in the laboratory.
08-01-11	2.	External (NC DENR) Internal	Lance Ferrell (NC DENR) Jim Sumner (ETS)	• NC 72-hour hold time exception removed from SOP.
01-03-12	3	Internal	Jim Sumner (ETS)	• Updated exhibits during document review.
07-01-20	4	Internal	Jim Sumner (ETS)	• Procedure for screening toxicity samples for chlorine was moved to SOP-C8 and removed from this SOP.
06-01-25	5	Internal	Jim Sumner (ETS)	• Updated exhibits during document review.

Subject: Receipt, Handling, and Storage of Samples

Scope and Application

To provide instructions to ensure the proper receipt, handling, and storage of water samples used for toxicity and analytical tests. This procedure also provides instructions for documenting sample custody.

Equipment and Materials

Mercury-filled or hand-held thermometers
Sample containers
Shipping container (cooler)
Indelible ink pen
Refrigerator
Chain-of-Custody Form
pH Strips
DPD Total Chlorine powder
1-oz Medicine cups
Sample Receipt Log
Sample Condition Form

Procedure for Receipt, Handling, and Storage of Toxicity Samples

A. Transfer of Sample Custody for Samples Delivered by Facility Personnel.

1. Upon receipt at the laboratory, a member of the laboratory inspects the shipping container (cooler) to ensure that the container was not tampered with and that the custody seals are intact (if used).

2. The cooler is opened and the sample container (Cubitainer®) is removed and inspected to ensure that the lid remained secure during transport.

3. The chain-of-custody form (Exhibit G4.1) is examined for completeness and correctness of information. At a minimum, the review should ensure that:
   - All pertinent blanks on the form are completed.
   - The sample collection date and times are completed.
   - The sample collector signed and dated the form.
   - The client/facility name, county, permit number, state, and outfall are completed.
   - Test requirements (including test dilutions, test species and test type) and sample information are complete.

4. If the sample container is not labeled, write the name of the sample and the sample date on the Cubitainer® with an indelible ink marker.

5. Have the person delivering the sample complete or correct the chain-of-custody form, if necessary, before accepting custody of the sample.

6. Measure and record the temperature of the sample (SOP-C1).

7. Have the person delivering the sample relinquish custody by printing their name, signing, and recording the date and time in the "Relinquished to ETS by:" line on the form.

8. A member of the laboratory then prints their name, signs, and records the date and time in the "Received at ETS by:" line on the form.

9. As samples are received, complete the Sample Receipt Log (Exhibit G4.2) by entering the following information into the Excel spreadsheet:
   - Date and time received.
   - Name of laboratory analyst that received the sample.
   - Name of the person that the sample was received by.
   - Sample temperature.
   - Sample name and description.

10. As samples are received, assign the sample a unique project and sample number.

    a. Project numbers are assigned by the next sequential project number for the laboratory. Multiple samples for the same project are given the same project number.

    b. Sample numbers are assigned with an 8-digit number consisting of the Year, Month, Date, and the sequential number for the sample received by the laboratory on that day (YYMMDD.NN). For example, the second sample received by the laboratory on February 24, 2025 would be assigned the following sample number: 250224.02.

    c. Enter the project and sample numbers into the sample receipt log and record on chain-of-custody form.

11. If requested, make a photocopy of the chain-of-custody form for the person delivering the sample to the laboratory.

12. A copy of all chain-of-custody forms is maintained in the project folder in the laboratory files.

B. Samples Received by Overnight Carrier.

1. Upon receipt at the laboratory, inspect custody seals (if used) to ensure that the shipping container (cooler) and samples were not tampered with. If the custody seals are broken, contact the Laboratory Supervisor to determine appropriate action and document that the custody seals were not intact upon receipt on the chain-of-custody form (Exhibit G4.1).

2. After inspection, the cooler is opened and the sample container (Cubitainer®) is removed and inspected to ensure that the lid remained secure during transport.

3. Review the chain-of-custody form for completeness and correctness of information. At a minimum, the review should ensure that:
   - All pertinent blanks on the form are completed.
   - The sample collection date and times are completed.
   - The sample collector signed and dated the form.
   - The client/facility name, county, permit number, state, and outfall are completed.
   - Test requirements (including test dilutions, test species and test type) and sample information are complete.

4. If the sample container is not labeled, write the name of the sample and the sample date on the Cubitainer® with an indelible ink marker.

5. Measure and record the temperature of the sample (SOP-C1).

6. Check the appropriate boxes if the custody seals were intact and samples were received in good condition.

8. Record the carrier's name and tracking number (if provided) that the sample was delivered by in the "Relinquished to ETS by:" line on the chain-of-custody form.

9. A member of the laboratory then prints their name, signs, and records the date and time in the "Received at ETS by:" line on the form.

10. As samples are received, complete the Sample Receipt Log (Exhibit G4.2) by entering the following information into the Excel spreadsheet:
    - Date and time received.
    - Name of laboratory analyst that received the sample.
    - Name of the delivery company that the sample was received by.
    - Sample temperature.
    - Sample name and description.

11. As samples are received, assign the sample a unique project and sample number according to A.10.

12. If the form is not complete, contact the facility and obtain the required information. Have the facility complete the chain-of-custody form and email a copy to the laboratory. Facility personnel are required by regulatory authorities to correct any errors or omissions they have made to the COC forms. Corrections of facility errors or omissions must not be made by laboratory personnel. Both the incomplete and complete COC forms are included in the test report submitted to the facility.

13. A copy of all chain-of-custody forms is maintained in the project folder in the laboratory files.

C. Acceptable Temperature upon Receipt

1. Samples must be received at the laboratory at 0.0 to 6.0°C. Only grab samples collected relatively short distances from the laboratory may be received above 6.0°C. This exception is only provided if a legitimate effort is made to cool the sample. A legitimate effort is considered if the sample is buried in crushed ice in its shipping container immediately after collection and if the sample arrives at the laboratory within 3 hours of collection. This must be documented on the chain-of-custody form.

2. Samples received at the laboratory above 6.0°C apart from the above circumstance will be considered invalid.

D. Sample Storage.

1. Samples collected and shipped or delivered to the laboratory must be chilled to 0.0 to 6.0°C and received on ice. Sufficient ice should be placed in the shipping container to ensure that ice will still be present when the sample arrives at the laboratory.

2. If samples are not to be analyzed upon receipt by the laboratory, they must be stored in a darkened refrigerator at 0.0 to 6.0°C.

3. Any unused portions of samples not to be disposed of must be stored in a darkened refrigerator at 0.0 to 6.0°C.

E. Sample Holding Times.

1. Each sample must first be used within 36-hours of completion of the sampling period.

F. Sample Disposal.

1. All excess sample and test water are disposed of by established laboratory protocols (SOP-G7).

Procedure for Receipt, Handling, and Storage of Analytical Samples

Note: All samples received by the laboratory are collected by facility personnel and are hand delivered or shipped through overnight delivery.

A. Transfer of Sample Custody for Samples Delivered by Facility Personnel.

1. Upon receipt at the laboratory, a member of the laboratory reviews the chain-of-custody form (Exhibit G4.3) for completeness and correctness of information. At a minimum, the review should ensure that:
   - All pertinent blanks on the form are completed.
   - The sample collection date and times are completed.
   - The sample collector signed and dated the form.
   - Test requirements and sample information is complete.

2. If the sample container is not labeled, write the name of the sample and the sample date on the bottle with an indelible ink pen or use a bottle label.

3. Have the person delivering the sample complete or correct the chain-of-custody form, if necessary, before accepting custody of the sample.

4. Measure and record the temperature of the sample (SOP-C1). If applicable, verify that the correct preservations were used (i.e. verify pH requirements using pH strips and verify dichlorination using DPD Total Chlorine powder).

5. Have the person delivering the sample relinquish custody by printing their name, signing, and recording the date and time in the "Relinquished by:" line on the form.

6. A member of the laboratory then signs their name and records the date and time in the "Accepted by:" line on the form.

7. Assign the sample a unique project and sample number and record in the Sample Receipt Log (Exhibit G4.4).

   a. Project numbers are assigned by the year, date and a sequential number beginning with 500 (YYMMDD.500).
   b. Sample numbers are assigned with a sequential number.

8. Give a copy of the chain-of-custody form (pink copy) to the person delivering the sample to the laboratory.

9. A copy of all chain-of-custody forms is maintained in the project folder in the laboratory files.

B. Samples Received by Overnight Carrier.

1. Upon receipt at the laboratory, open the shipping container. Review the chain-of-custody form (Exhibit G4.3) for completeness and correctness of information. At a minimum, the review should ensure that:
   - All pertinent blanks on the form are completed.
   - The sample collection date and times are completed.
   - The sample collector signed and dated the form.
   - Test requirements and sample information are complete.

2. If the sample container is not labeled, write the name of the sample and the sample date on the bottle with an indelible ink pen or bottle label.

3. Measure and record the temperature of the sample (SOP-C1). If applicable, verify that the correct preservations were used (i.e. verify pH requirements using pH strips and verify dichlorination using DPD Total Chlorine powder).

4. A member of the laboratory then prints their name, signs, and records the date and time in the "Accepted By:" line on the form.

5. Assign the sample a unique project and sample number according to A.7.

6. If the form is not complete, contact the facility and obtain the required information.

7. A copy of all chain-of-custody forms is maintained in the project folder in the laboratory files.

C. Acceptable Temperature upon Receipt

1. Samples must be received at the laboratory at 0.0 to 6.0°C. Only grab samples, collected relatively short distances from the laboratory, may be received above 6.0°C. This exception is only provided if a legitimate effort is made to cool the sample. A legitimate effort is considered if the sample is buried in ice in its shipping container immediately after collection and if the sample arrives at the laboratory and demonstrates a downward trend. The initial field temperature must be documented on the chain-of-custody form.

2. If the temperature of the sample is above 6.0°C, and no initial field temperature is provided on the chain-of-custody the client must be notified, and a determination is to be made about recollecting the sample. If another sample cannot be secured a Sample Condition Form (Exhibit G4.5) must be completed and emailed to the NC DEQ regional office.

D. Sample Preservation and Dechlorination.

1. If any sample that requires preservative or dechlorination (as indicated in QAP-Q5 or Chemistry/Bacteria SOP) is not properly preserved or dechlorinated, the client must be notified, and a determination is made about recollecting the sample. If a sample cannot be secured a Sample Condition Form must be completed and emailed to the NC DEQ regional office (Exhibit G4.5).

E. Sample Storage.

1. Samples collected and shipped or delivered to the laboratory must be chilled to 0.0 to 6.0°C and received on ice. Sufficient ice should be placed in the shipping container to ensure that ice will still be present when the sample arrives at the laboratory.

2. If samples are not to be analyzed upon receipt by the laboratory, they must be stored in a darkened refrigerator at 0.0 to 6.0°C.

3. Any unused portions of samples not to be disposed of must be stored in a darkened refrigerator at 0.0 to 6.0°C.

F. Sample Holding Times.

1. Please refer to each individual SOP for sample holding times.

2. If a sample is out of hold time, the client must be notified, and a determination is made about recollecting the sample. If a sample cannot be secured, then a Sample Condition Form must be completed and emailed to the NC DEQ regional office (Exhibit G4.5).

G. Sample Disposal.

1. All excess sample and test water are disposed of by established laboratory protocols (SOP-G7).

Safety and Hazardous Waste Management

Safety glasses, gloves and lab coats should always be worn.

Review Policy-P6: General Safety Policy and Policy-P9: Radiation Protection Policy for additional safety requirements.

References

Standard Methods for the Examination of Water and Wastewater, 24th Edition, 2023. American Public Health Association, 800 I Street, NW, Washington DC 20001-3710.

USEPA. October 2002. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, 5th ed. EPA-821-R-02-012. US Environmental Protection Agency, Cincinnati, OH.

USEPA. October 2002. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms, 4th ed. EPA-821-R-02-013. US Environmental Protection Agency, Cincinnati, OH.

USEPA. October 2002. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms, 3rd ed. EPA-821-R-02-014. US Environmental Protection Agency, Cincinnati, OH.

TNI Standard. Management and Technical Requirements for Laboratories Performing Environmental Analysis. EL-V1-ISO-2016-Rev2.0. The NELAC Institute, PO Box 2439, Weatherford, TX 76086.

Exhibits

Exhibit G4.1: Whole Effluent Toxicity Chain-of-Custody Form.
Exhibit G4.2: Whole Effluent Toxicity Sample Receipt Log.
Exhibit G4.3: Analytical Chain-of-Custody Form.
Exhibit G4.4: Analytical Sample Receipt Log.
Exhibit G4.5: Sample Condition Form.

Subject: Receipt, Handling, and Storage of Samples

Exhibit G4.1: Whole Effluent Toxicity Chain-of-Custody Form.

Environmental Testing Solutions, Inc.

Whole Effluent Toxicity Chain-of-Custody Form

351 Depot Street
Asheville, NC 28801
Phone: (828) 350-9364
Fax: (828) 350-9368

Client: Cape Fear Public Utility — NPDES #: NC0002879 — Outfall: 001 — County: New Hanover
Facility: Sweeney WTP — Purchase order:
Species: *Americamysis bahia* — Effluent dilution: 22.7%
Test type: Pass/Fail Chronic — Parameter code: TGP3E

Sample information: (to be completed by sample collector)

Grab sample:
Start date: _____ Time: _____
Composite sample:
Start date: _____ Time: _____
End date: _____ Time: _____
Flow proportional (✓): ☐ or # samples/hour: ☐
Chilled during collection (yes/no)? _____
If chilled, specify temperature (°C): _____

Sample location: _____
Volume collected for testing: _____
Number of containers filled for testing: _____
Method of transport to laboratory: _____
Comments: _____

Triple rinse sample container with sample before filling. Completely fill the sample container with no air space. Pack the sample container completely in ice. The sample must be < 6.0°C upon receipt at the laboratory.

Sample custody: (to be completed by sample collector and facility personnel)

BY SIGNING BELOW, I CERTIFY THAT THE PERMIT AND TEST REQUIREMENTS IDENTIFIED ON THIS FORM ARE ACCURATE.

	Print	Signature	Date and time
Sample collected by:			
Relinquished by:			
Received by:			
Relinquished by:			
Received by:			
Relinquished by:			
Received by:			

Sample receipt information: (to be completed by ETS personnel)

	Print	Signature	Date and time
Relinquished to ETS by:			
Received at ETS by:			

Custody seals intact (✓)? ☐ Yes ☐ No ☐ Not used
Samples received in good condition (✓)? ☐ Yes ☐ No
Sample temperature upon receipt at ETS: ____ °C
Project number: _____ Sample number: _____
FedEx/UPS tracking number: _____
Comments: _____

Subject: Receipt, Handling, and Storage of Samples

Exhibit G4.2: Whole Effluent Toxicity Sample Receipt Log.

ETS

Whole Effluent Sample Receipt Log

Page ____

*Sample temperature performed using verified General Use Thermometer SN: _____

Date Received	Time Received	Received by	Received from	*Sample Temp. (°C)	Project number	Sample number	Sample name and description	State	Comments
05-06-25	1030	J. Sumner	FedEx	0.4	20557	250506 .01	Farmville WWTP - Pp	NC	
05-06-25	1030	J. Sumner	FedEx	2.4	20558	250506 .02	Hoke Co WWTP - Pp	NC	
05-06-25	1030	J. Sumner	FedEx	0.7	20559	250506 .03	Loughlin WWTP - Pp	NC	
05-06-25	1030	J. Sumner	FedEx	3.0	20560	250506 .04	Western Wake WRF - Pp	NC	
05-06-25	1030	J. Sumner	FedEx	1.8	20561	250506 .05	Bogue Banks WTP	NC	
05-06-25	1030	J. Sumner	FedEx	2.4	20562	250506 .06	Carolina Beach WWTP - Ab/Mb	NC	
05-06-25	1030	J. Sumner	FedEx	1.1	20563	250506 .07	Engelhard WTP	NC	
05-06-25	1030	J. Sumner	FedEx	1.4	20564	250506 .08	Pasquotank WTP	NC	
05-06-25	1030	J. Sumner	FedEx	1.5	20565	250506 .09	Manteo WWTP	NC	
05-06-25	1030	J. Sumner	FedEx	2.0	20566	250506 .10	Apex Oil Co	NC	
05-06-25	1102	J. Sumner	Sonic Delivery	1.2	20567	250506 .11	TVA /SQN Outfall 101	TN	
05-06-25	1102	J. Sumner	Sonic Delivery	0.9	20567	250506 .12	TVA /SQN Intake	TN	
05-06-25	1132	X. Lancaster	Dash Courier	0.4	20568	250506 .13	OWASA	NC	
05-06-25	1141	X. Lancaster	M. Ringgenberg	7.7	20569	250506 .14	COA, Mills River WTP	NC	Invalid sample.
05-06-25	1155	X. Lancaster	J. Brown	3.4	20570	250506 .15	Andrews WTP	NC	
05-06-25	1155	X. Lancaster	J. Brown	3.4	20571	250506 .16	Robbinsville WTP	NC	
05-06-25	1155	X. Lancaster	A. Kramer	1.1	20572	250506 .17	Riverstone Industrial Park WWTP - Cd	NC	
05-06-25	1300	J. Perez	ShipTeam	2.1	20573	250506 .18	McGuire NS, Outfall 001	NC	
05-06-25	1300	J. Perez	ShipTeam	2.0	20574	250506 .19	McGuire NS, Outfall 002	NC	
05-06-25	1300	J. Perez	ShipTeam	2.3	20575	250506 .20	McGuire NS, Outfall 005	NC	

Subject: Receipt, Handling, and Storage of Samples

Exhibit G4.3: Analytical Chain-of-Custody Form.

Environmental Testing Solutions, Inc.
PO Box 7565, Asheville, NC 28802
Phone: (828) 350-9364, Fax: (828) 350-9368

CHAIN-OF-CUSTODY / Analytical Request

Client Name: _____
Address: _____
_____
Report to: _____
Phone: _____ Fax: _____
PO Number: _____

Project Name: _____
Project Number: _____

Item Number	Sample Identification	G = Grab, C = Composite	Sample Number ETS Use Only	Matrix: Water, Solid, Liquid	Date Collected (MM/DD/YY)	Time Collected (HH:MM)	# Containers	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH + Dechlor.	$Na_2S_2O_3$	Requested Analyses	pH Check Upon Receipt	Cl_2 < 0.2 mg/l Upon Receipt	Field Temp. (°C)	Comments
1																		
2																		
3																		
4																		
5																		
6																		

Sample Condition Upon Receipt		Relinquished By	Company	Date	Time	Accepted By	Company	Date	Time
Temperature (°C)									
Received on Ice:	Y / N								
Sealed Cooler:	Y / N								
Samples Intact:	Y / N								

Additional Comments:

Sampler Name and Signature — Date
Printed Name of Sampler:
Signature of Sampler:

Subject: Receipt, Handling, and Storage of Samples

Exhibit G4.4: Analytical Sample Receipt Log.

Date received	Client name	Sample identification	Date collected	Time collected	Project number	Sample number	Analyses
27-May-25	Polk Central	Effluent - Grab	27-May-25	0704	250527.500	298145	BOD, 5 day Solids, Total Suspended Fecal Coliform
27-May-25	Adventure Village	Effluent - Composite	27-May-25	0'835	250527.501	298146	BOD, 5 day Solids, Total Suspended Ammonia Nitrogen Fecal Coliform
27-May-25	Saluda WWTP	Influent - Composite	27-May-25	0715	250527.502	298147	BOD, 5 day Solids, Total Suspended
		Effluent - Composite	27-May-25	0709		298148	BOD, 5 day Solids, Total Suspended Ammonia Nitrogen
		Effluent - Grab	27-May-25	0656		298149	Fecal Coliform
27-May-25	Royal Water Works	Toxaway Falls - Grab	27-May-25	0825	250527.503	298150	BOD, 5 day Solids, Total Suspended Fecal Coliform
27-May-25	Rosman WWTP	Influent - Grab	27-May-25	0830	250527.504	298151	BOD, 5 day Solids, Total Suspended
		Effluent - Composite	27-May-25	0830		298152	BOD, 5 day Solids, Total Suspended
		Effluent - Grab	27-May-25	0830		298153	Fecal Coliform
27-May-25	Sapphire Lakes	Effluent - Grab	27-May-25	0745	250527.505	298154	BOD, 5 day Solids, Total Suspended Fecal Coliform Mixed Liquor Suspended
27-May-25	Columbus WWTP	Influent - Composite	27-May-25	0800	250527.506	298155	BOD, 5 day Solids, Total Suspended
		Effluent - Composite	27-May-25	0805		298156	BOD, 5 day Solids, Total Suspended Ammonia Nitrogen
		Effluent - Grab	27-May-25	0805		298157	Fecal Coliform
27-May-25	Hot Springs	Influent - Grab	27-May-25	0834	250527.507	298158	BOD, 5 day Solids, Total Suspended
		Effluent - Grab	27-May-25	0832		298159	BOD, 5 day Solids, Total Suspended Ammonia Nitrogen Fecal Coliform
27-May-25	HSHA	Effluent - Grab	27-May-25	0803	250527.508	298160	BOD, 5 day Solids, Total Suspended Fecal Coliform
27-May-25	Baxter	Influent - Composite Effluent - Composite	26-May-25 26-May-25	0802 0810	250527.509	298161 298162	BOD, 5 day BOD, 5 day

Exhibit G4.5: Sample Condition Form.

ETS Environmental Testing Solutions, Inc.

SAMPLE CONDITION UPON RECEIPT (SCUR)
DEVIATION

15A NCAC 02H .0805 (a) (7) (M): Sample preservation shall be verified and documented. If a laboratory receives a sample subject to G.S. 143-215.1 and 143-215.63 that does not meet sample collection, holding time, or preservation requirements, the laboratory shall document the incident, notify the sample collector or client, and secure another sample that meets the regulatory requirements, if possible. If another viable sample cannot be secured, the original sample may be analyzed but the results reported shall be qualified with the nature of the sample collection, holding time, or preservation infractions and the laboratory shall notify the State Laboratory of the infractions. The notification shall include a statement indicating corrective action taken to prevent future infractions.

The sample(s) identified below deviated from required preservation, hold time, sampling protocol or sample documentation.

Attention: Mr. Jason Smith (NC DEQ, Laboratory Certification Branch)

Date: _____

Received from:

Name _____

Sample collector _____

Phone number _____

Deviation:

Sample date/time _____

Analysis _____

Deviation

☐ The sample(s) was not received within holding time.

☐ The sample(s) was not received on ice (0 to 6.0°C).

☐ The sample(s) was not received within holding time.

☐ The sample(s) was not collected and/or preserved correctly (e.g. head space in volatiles, improper container, or preservation/dechlorination) _____

_____

☐ The chain-of-custody did not have all the appropriate information (e.g. collector's name, date collected, time collected, sample identification, number of containers for each analysis).

☐ Other: _____

_____

Action taken:

☐ Sample(s) accepted and analyzed per client request.

☐ SCUR and COC e-mailed to NC DEQ, Asheville Regional Office – Laboratory Certification Branch.

NC Wastewater / Groundwater Certificate Number: Wastewater: 600 SOP Q3-Revision 5 – Exhibit Q3.2

Subject: Preparation of Toxicity Samples

Approval

Title	Name	Signature	Date
Laboratory Supervisor	Kelley E. Keenan		06-01-25
Quality Assurance Officer	Jim Sumner		06-01-25

Document Revision History

Effective Date	Revision number	Review Type	Evaluators	Revisions
12-01-00	0	Internal	Jim Sumner (ETS)	Original document
07-01-13	1	External (NC DENR) Internal	Lance Ferrell (NC DENR) Jim Sumner (ETS)	• Included clarification that the UV sterilization of samples is only performed for NPDES permits, which includes this testing modification. Testing in support of North Carolina NPDES permits are not treated prior to testing.
03-01-20	2	External (TVA) Internal	Rick Sherrard (TVA) Jim Sumner (ETS)	• The use of SSW for NC testing was removed. • Updated procedure to include NELAP requirements. • Section B.1 corrected to state: Use a 50-mL graduated cylinder to measure 10 to 50 mL of sample.
06-01-25	3	Internal	Jim Sumner (ETS)	• Updated procedure during document review.

Subject: Preparation of Toxicity Samples

Scope and Application

To prepare samples for use in toxicity tests.

Equipment and Materials

60-µm Nitex mesh
Serological, fixed, or adjustable-volume pipettes (Eppendorf®)
Volumetric flasks
Various sized graduated cylinders
Synthetic water
Mercury-filled or hand-held thermometers
Tap water
Temperature controlled incubator
Various sized beakers
Dissolved oxygen (DO) meter equipped with a DO probe
Aeration pumps and aquarium tubing
Serological pipettes
Marine Mix® - Forty Fathoms® sea salt
Stir bar
Stir plate
Salinity meter
Safety glasses
Gloves
40 watt UV Sterilizer
Various test benchsheets

Procedure

A. Presence of Indigenous Organisms.

1. Filter the water sample through a 60-µm Nitex mesh, if the sample contains indigenous organisms (e.g. copepods or Cladoceran species) that may be confused with the test species. Document on the appropriate toxicity test bench sheet that the sample was filtered through a 60-µm Nitex mesh.

B. Preparation of Dilutions.

1. If dilutions of the water sample need to be made, use the appropriate volumetric flasks, graduated cylinders and pipettes. The level of precision in the preparation of dilutions is increased when smaller volume graduated cylinders and/or serological pipettes are used to prepare the test concentrations. For example, to measure < 10 mL of sample, use a 10-mL serological pipette. Use a 50-ml graduated cylinder to measure 10 to 50 mL of sample.

2. The type of control/dilution water used will depend on the test species (salt or freshwater species).

   a. For freshwater samples using *Ceriodaphnia dubia, Daphnia* or *Pimephales promelas*, moderately hard synthetic water (MHSW) is used (SOP-AT1). MHSW must have a hardness of 80 – 100 mg $CaCO_3$/L, an alkalinity of 57 – 64 mg $CaCO_3$/L and an initial pH of 6.5 – 8.5 S.U. (recommended 7.4 – 7.8 S.U.).

   b. For marine and estuarine samples using *Americamysis bahia* or *Menidia beryllina*, salt synthetic water (SaltSW) is used (SOP-AT1). SaltSW must have a salinity of 5 – 32 ppt (typically 25.0 ± 2.0 ppt) and an initial pH of 6.5 – 8.5 SU.

C. Achieving Test Temperature, Equilibration of Dissolved Gases.

1. Heat or cool the water samples in a water bath or temperature-controlled incubator, as required, to achieve a final temperature of 25.0 ± 1.0°C (26.0 ± 1.0°C for *A. bahia* chronic tests)

2. Measure the dissolved oxygen (D.O.) concentration (SOP-C2) of each sample after it has been warmed to the required temperature. If the D.O. is > 9.0 mg/L or < 4.0 mg/L, then it may be necessary to aerate the sample. If the sample was warmed too quickly, then the D.O. concentration may be greater than 100% saturation (> 9.0 mg/L).

   a. Shake the sample to equilibrate the dissolved gases. In most cases, vigorously shaking the sample will lower the D.O. concentration.

   b. Re-measure the D.O. concentration.

   c. If the D.O. concentration is still outside the acceptable range, moderately aerate the sample for 1 minute and re-measure the D.O. concentration. Continue aerating for 1-minute intervals and measuring the D.O. concentration until the concentration is within the acceptable range. The sample may be aerated for a total of 15 minutes;

however, aeration during the preparation of samples should be minimized to prevent the loss of volatile compounds. Document the total aeration time on the appropriate toxicity test bench sheet for the sample.

d. If the D.O. concentration is not within the acceptable range after 15 minutes of aeration, each replicate test vessel must be gently aerated throughout the test period. This activity must be documented on the appropriate toxicity test bench sheet for the sample.

D. Saltwater Toxicity Tests, "Salting-Up" the Sample.

1. Measure the salinity of the water sample according to SOP-C5. If the salinity is below the required range (25.0 ± 2.0 ppt unless otherwise specified by the test species and permit requirements), then the sample must be "salted-up" through the addition of sea salts to achieve the required salinity. Consult with the Laboratory Supervisor prior to "salting-up" the sample.

   a. Pour an aliquot of the water sample into a large beaker with a stir bar.

   b. Place the beaker on a stir plate and stir the sample.

   c. Place the tip of the salinity probe in the sample. Measure and record the initial salinity (SOP-C5) on the toxicity test bench sheet for the sample.

   d. Slowly add Marine Mix® sea salt to the sample, allow the salinity to stabilize. Continue to add sea salt until the desired salinity is obtained (25.0 ± 2.0 ppt, or as required by the test species and NPDES permit). Record the final salinity on the toxicity test bench sheet for the sample.

E. UV Sterilization of Water Samples to Remove Pathogenic Interferences.

Note: UV sterilization of samples is only performed for NPDES permits, which include this testing modification.

1. To remove pathogenic interferences in minnow chronic toxicity tests, samples may be treated with UV light. A 80-watt UV Sterilizer is used for treating ambient, wastewater, and industrial samples.

   a. Clean the inside of the UV sterilizer according to procedures outlined in SOP-G1.

b. Set the UV sterilizer horizontally in the supports on the counter, with the inlet and outlet tubes facing up.

c. Rinse the inside chamber of the sterilizer with deionized water. Carefully pour approximately 2000 ml deionized water into the outlet tube of the sterilizer. Using a rubber stopper, plug the outlet tube.

d. Remove the UV sterilizer from the counter, making sure to keep the sterilizer horizontal. Gently rock the sterilizer back and forth to thoroughly rinse the inside chamber.

e. Remove the rubber stopper and drain the sterilizer.

f. Beginning with the lowest dilution or control sample, carefully pour the required volume to be treated into the outlet tube. Using a rubber stopper, plug the outlet tube.

g. Plug the ballast cord into the electrical outlet. Ensure the UV light is illuminated, by looking through the view port.

h. Set the timer for 2 minutes and turn the timer on.

i. Remove the UV sterilizer from the counter, making sure to keep the sterilizer horizontal. Gently rock the sterilizer back and forth to provide the maximum exposure of the sample to the UV light.

j. Return the UV sterilizer to the supports on the counter, horizontally with the inlet and outlet tubes facing up.

k. After 1 minute has elapsed, repeat steps i and j.

l. After 2 minutes have elapsed, unplug the ballast cord from the electrical outlet and turn off the timer.

m. Remove the stopper from the outlet tube and carefully pour the sample into a clean glass beaker or flask. The UV treated sample must never come in contact with glassware or equipment that has been exposed to non-treated sample.

n. Repeat steps c through m.

Safety and Hazardous Waste Management

Safety glasses, gloves and lab coats should always be worn.

Review Policy-P6: General Safety Policy and Policy-P9: Radiation Protection Policy for additional safety requirements.

References

Standard Methods for the Examination of Water and Wastewater, 24th Edition, 2023. American Public Health Association, 800 I Street, NW, Washington DC 20001-3710.

USEPA. October 2002. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, 5th ed. EPA-821-R-02-012. US Environmental Protection Agency, Cincinnati, OH.

USEPA. October 2002. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms, 4th ed. EPA-821-R-02-013. US Environmental Protection Agency, Cincinnati, OH.

USEPA. October 2002. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms, 3rd ed. EPA-821-R-02-013. US Environmental Protection Agency, Cincinnati, OH.

TNI Standard. Management and Technical Requirements for Laboratories Performing Environmental Analysis. EL-V1-ISO-2016-Rev2.0. The NELAC Institute, PO Box 2439, Weatherford, TX 76086.

Subject: Disposal of Samples and Containers

Approval

Title	Name	Signature	Date
Laboratory Supervisor	Kelley E. Keenan		06-01-25
Quality Assurance Officer	Jim Sumner		06-01-25

Document Revision History

Effective Date	Revision number	Review Type	Evaluators	Revisions
05-06-01	0	Internal	Jim Sumner (ETS)	Original document
03-01-20	1	External (TVA) Internal	Rick Sherrard (TVA) Jim Sumner (ETS)	• Updated procedure to include NELAP requirements. Corrected grammatical errors. • For TVA sample containers, obliterate the name "TVA" (if present) with a sharpie.
06-01-25	2	Internal	Jim Sumner (ETS)	• Updated procedure to include requirements for disposing sample containing tritium and colilert samples. • Removed requirement for TVA sample containers (TVA is not documented on container).

Scope and Application

To properly dispose of samples and containers.

Equipment and Materials

Sample containers (Glass or Plastic)
Samples
Recycling bin
Sharpie

Procedure

A. Disposal of Non-hazardous Samples

1. Only samples can be disposed of after all analyses have been performed.

2. All sample containers must be properly disposed.

a. To purge a sample, turn on the cold water tap and slowly pour the sample down the drain.

b. After the sample container is empty, rinse it with tap water.

c. Place the container in a recycling bin.

d. Recycled materials are taken to the recycling center every week.

B. Disposal of Hazardous Samples and Containers.

1. All containers that contain hazardous waste must be returned to the client.

2. The laboratory must not dispose of any samples that contain asbestos, PCB's or oil contaminated waste.

3. Colilert samples are disposed of according to procedures described in SOP-B6.

4. Samples containing tritium are disposed of according to procedures described in Policy-P6.

Safety and Hazardous Waste Management

Safety glasses, gloves and lab coats should always be worn.

Review Policy-P6: General Safety Policy and Policy-P9: Radiation Protection Policy for additional safety requirements. Additional requirements for the disposal of tritiated samples is contained within this policy.

Subject: Reagent Water System Maintenance

Approval

Title	Name	Signature	Date
Laboratory Supervisor	Kelley E. Keenan		06-01-25
Quality Assurance Officer	Jim Sumner		06-01-25

Document Revision History

Effective Date	Revision number	Review Type	Evaluators	Revisions
12-01-00	0	Internal	Jim Sumner (ETS)	Original document
06-29-09	1	Internal	Jim Sumner (ETS)	• Included a flow diagram for the deionized water system.
03-01-20	2	External (TVA) Internal	Rick Sherrard (TVA) Jim Sumner (ETS)	• The Milli-Q water system was removed. • Increased deionized water system tanks to 3.6 CF. • Updated procedure to include NELAP requirements.
06-01-25	3	**Internal**	Jim Sumner (ETS)	• Updated chlorine requirements. • Updated reference to 24th Edition of Standard Methods.

Scope and Application

To maintain the deionized water system for cultures and toxicity/analytical tests.

Quality Control

1. Total residual chlorine is monitored in deionized water through the testing of blanks (SOP-C8). Blanks are tested with each batch of toxicity samples analyzed for chlorine. Levels of chlorine should be less than ½ the detection limit (<25 µg/L). If detectable results are obtained, corrective action must be taken.

2. The activated carbon tank is replaced every 6 months.

3. The prefilters and post filters in the deionized water system are replaced every 6 months.

4. The working and polishing deionizers are replaced when the resistivity light goes out or every 6 months (whichever comes first).

5. All maintenance activities must be recorded in the Reagent Water System Log (Exhibit G8.1).

Subject: Reagent Water System Maintenance

Equipment and Materials

Potable water
Pressure regulators
Course filter
Prefilters and housings
Prefilter Cartidge, 1 micron
Prefilter housing assembly, 20''
Activated carbon tank, 3.6 cf
Mixed bed deionizers, 3.6 cf
Post filter Cartridges, 0.2 micron, 10"
Post filter housing assemblies, 10''
Interconnecting assembly (includes resistivity lights, pressure regulator, standard FDA grade tubing and fittings, and water meter)
Water valve
Reagent water system log

Procedure

A. Flow Chart.

1. City of Asheville, NC potable water is diverted from the city supply line into the laboratory. The main line passes through a pressure regulator and then through a course filter and prefilter to supply the laboratory with potable water (Figure G8.1).

2. Standard FDA grade tubing is diverted from the source potable water line to a pressure regulator.

3. From the pressure regulator, water is passed through a 20-inch, 1-micron prefilter and then a 3.6 cubic-foot activated carbon tank.

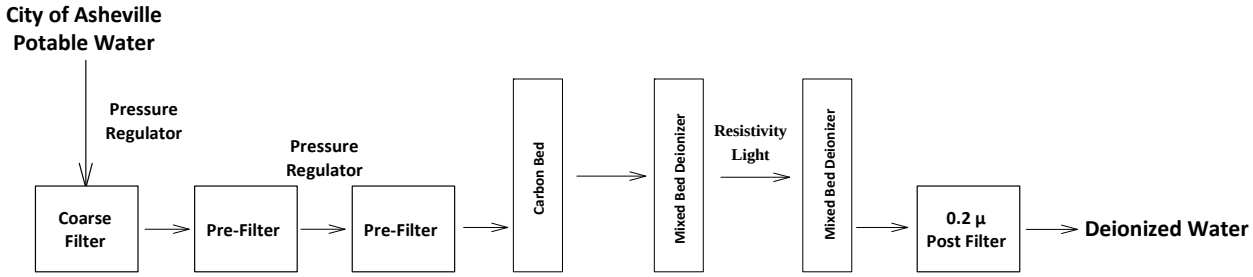
4. From the activated carbon tank, water is passed through a 3.6 cubic-foot working deionizer. After the working deionizer, a resistivity light indicates the quality of megaohm-cm water. The light is illuminated green with high quality water. When the working deionizer is exhausted, the light will turn red. At this time, the working deionizer and activated carbon tank must be replaced.

5. From the working deionizer, the water passes through a second 3.6 cubic-foot polishing deionizer.

6. After the polishing deionizer, the water passes through a 10-inch, 0.2-micron post filter.

7. From the post filter, deionized water is available.

Figure G8.1: Diagram of the deionized water system.



Safety and Hazardous Waste Management

Safety glasses, gloves and lab coats should always be worn.

Review Policy-P6: General Safety Policy and Policy-P9: Radiation Protection Policy for additional safety requirements.

References

Standard Methods for the Examination of Water and Wastewater, 24th Edition, 2023. American Public Health Association, 800 I Street, NW, Washington DC 20001-3710.

USEPA. October 2002. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, 5th ed. EPA-821-R-02-012. US Environmental Protection Agency, Cincinnati, OH.

USEPA. October 2002. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms, 4th ed. EPA-821-R-02-013. US Environmental Protection Agency, Cincinnati, OH.

USEPA. October 2002. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms, 3rd ed. EPA-821-R-02-014. US Environmental Protection Agency, Cincinnati, OH.

TNI Standard. Management and Technical Requirements for Laboratories Performing Environmental Analysis. EL-V1-ISO-2016-Rev2.0. The NELAC Institute, PO Box 2439, Weatherford, TX 76086.

Exhibits

Exhibit G8.1: Reagent Water System Log.

Subject: Reagent Water System Maintenance

Exhibit G8.1: Reagent Water System Log.

Page _____

Reagent Water System Log

Location: ☐ Main Laboratory ☐ Fish Culture Laboratory

Service Date	Analyst	Activity (type of service performed and comments)

SOP G8-Revision 3–Exhibit G8.1

Subject: Instrument Maintenance and Repair

Approval

Title	Name	Signature	Date
Laboratory Supervisor	Kelley E. Keenan		06-01-25
Quality Assurance Officer	Jim Sumner		06-01-25

Document Revision History

Effective Date	Revision number	Review Type	Evaluators	Revisions
09-01-09	0	Internal	Jim Sumner (ETS)	Original document
06-20-12	1	External (TVA) Internal	William Rogers (TVA) Donald Snodgrass (TVA) Rick Sherrard (TVA) Jim Sumner (ETS)	• Exhibit G9.1 amended to include the dates of routine maintenance, removed from service and returned to service.
03-01-20	2	Internal	Jim Sumner (ETS)	• Updated procedure to include NELAP requirements.
06-01-25	3	Internal	Jim Sumner (ETS)	• Updated Instrument Maintenance and Equipment Maintenance Logs. • Updated procedure regarding individual item repairs.

Scope and Application

To provide instruction and documentation on the maintenance and repair of laboratory instruments.

Procedure

A. Maintenance, Repair, and Nonconformances

1. Service contracts or in-house preventive maintenance and/or repair are documented in a central maintenance log, which may include documentation for each instrument (example provided in Exhibit G9.1). Larger equipment (e.g. incubators) repairs may also be recorded on the side of the equipment. Documentation may include annual maintenance and maintenance performed at other times due to malfunction (except for routine cleaning).

2. Nonconformances (e.g. exceeding quality control limits) for each instrument are also documented in the maintenance log. This includes what led to the nonconformance, probable cause, and how the non-conformance was resolved. In addition,

nonconformances are documented through QAP-Q3, Surveillance and Corrective Action Reports.

3. Manuals provided by the instrument manufacturers are maintained on file as reference tools to trouble shoot problems with instrumentation. If problems cannot be resolved internally, service technicians are hired to repair or calibrate the malfunctioning instruments.

4. Calibration documentation for each instrument is maintained in various logbooks for the tests performed on that instrument or in calibration logbooks maintained for that instrument. Calibration information is not documented in the maintenance log.

References

Instrument Manuals

Exhibits

Exhibit G9.1: Example Maintenance Log (attached)

Subject: Balance and Weight Calibration

Approval

Title	Name	Signature	Date
Laboratory Supervisor	Kelley E. Keenan		06-01-25
Quality Assurance Officer	Jim Sumner		06-01-25

Document Revision History

Effective Date	Revision number	Review Type	Evaluators	Revisions
12-01-00	0	Internal	Jim Sumner (ETS)	Original document
09-01-09	1	External (TVA, Environmental Standard, Inc.) Internal	William Rogers (TVA) Cynthia Russell (TVA) Rick Sherrard (TVA) Rock Vitale (Environmental Standards, Inc.) Jim Sumner (ETS)	• Balance calibration range changed to bracket all potential measurements performed in the laboratory. • Removed warning limits from acceptance criteria. • Corrective action included if certified weights exceed acceptance criteria. • Changed balance logs and exhibits to reflect these changes and to provide traceability to the last balance and NIST calibrations performed by external vendors. • NIST certified weights recalibrated, which changed the true values. Amended associated exhibit and values throughout document. • Ammended wall chart to provide the source (SOP G10 – Table G10.1, revision 09-01-09).
01-03-12	2	Internal	Jim Sumner (ETS)	• Included calibration procedure for the Cahn 28 Automatic Electrobalance. • Updated exhibits during document review.
07-01-20	3	Internal	Teresa Sleeper (ETS) Jim Sumner (ETS)	• Replaced calibration instructions for Fisher Scientific ACCU-224 with instructions for Mettler-Toledo ME204. • Updated procedure to include NELAP requirements. • Additional guidance included in SOP. • Updated log to reflect new control limits for certified weights.
06-01-25	4	Internal	Jim Sumner	• Updated exhibits during document review.

Subject: Balance and Weight Calibration

Scope and Application Purpose

To maintain balance and weights for toxicity and analytical tests.

Quality Control

1. Balances must be serviced yearly by an approved company (e.g. Laboratory Instruments, Inc.). Documentation is maintained in the laboratory's QC files.

2. Balances must be verified and/or calibrated before each use.
    - Mettler-Toledo ME204:
        - Internal calibration is performed at least weekly.
        - Balance level verified and corrected as needed before each use.
        - Balance tared before each use.
        - Balance verified with at least two Class "S" Weights each day prior to use. The weights must bracket the items to be weighed.
        - Balance verified with seven Class "S" Weights weekly.
    - Cahn 28 Automatic Electrobalance:
        - Balance tared and calibrated before each use.
        - Balance verified with two Class "S" Weights before and after use. The weights must bracket the items weighed.

2. Class "S" Weights must be verified every 5 years against NIST certified weights. Precision Weighing, Inc. in Cary, NC is used to perform this verification. Documentation is maintained in the laboratory's QC files.

3. All certified weight measurements must be within the control limits before any materials or samples can be analyzed. If the measurements are out of range, perform the balance internal calibration and document in the balance log. Re-analyze the certified weights. If the measurements still exceed the control limits, notify the Laboratory Supervisor to determine if the balance requires cleaning or adjustment. If the balance cannot be corrected to meet the control limits through cleaning or adjustment, the balance must be serviced by an approved instrument calibration company (e.g. Laboratory Instruments, Inc.). If final measurements using the Cahn Electrobalance exceed the control limits, all weight measurements performed that day must be re-analyzed. Refer to Table G10.1 for the lower and upper control limits for weights used to calibrate balances.

Table G10.1: Lower and upper control limits for Class "S" Weights used to calibrate balances.

True value (g)	Control Limit (g) (0.01005 to 0.1005 g weights ± 2.0%) (1.0000 to 149.9998 g weights ± 0.10%)	
	Lower	**Upper**
0.01005	0.0985 (9.85 mg)	0.01025 (10.25 mg)
0.05004	0.04904 (49.04 mg)	0.05104 (51.04 mg)
0.1005	0.0985	0.1025
1.0000	0.9990	1.0010
10.0001	9.9901	10.0101
49.9999	49.9499	50.0499
99.9999	99.8999	100.0999
149.9998	149.8498	150.1498

Equipment and Materials

Balance (Mettler-Toledo ME204, Cahn 28 Automatic Electrobalance)
Class "S" Weights
0.01005, 0.05004, 0.1005, 0.20001, 1.0000, 10.0001, 49.9999, 99.9999 and 149.9998 g
Anti-static brush
Forceps
Spatula
Weigh boat or medicine cup
Balance Log

Subject: Balance and Weight Calibration

Procedure

Note: Always use forceps to place weights on the balance.

A. Calibration of the Mettler-Toledo ME204.

1. The balance must be verified before each use. Record calibration information in the Balance Log (Exhibit G10.1).

2. Turn the balance on by pressing the **→0/T←** button.

3. Level the balance using the leveling feet so that the air bubble is centered within the circle of the level indicator and document in the Balance Log.

4. Wait until the balance reads 0.0000 g before starting. Use the **→0/T←** key to tare the reading.

5. For weighing chemicals and materials, use two certified weights that bracket the weight of chemicals or materials to be measured. Record the measurement of both weights in the Balance Log.

6. Weekly perform the internal calibration of the balance and record in the Balance Log. Press and hold the **Cal** button until **ADJUST** appears. Select **ADJ.INT** by pressing **Menu** and allow the balance to perform the internal calibration. Once the calibration is complete, the balance will read **ADJ.DONE** and **0.0000 g** will appear when the balance is ready.

7. Weekly (when the internal calibration is performed), verify the balance with all the Class "S" Weights identified on the log. Record the measurements in the Balance Log.

B. Weighing Chemicals or Materials using the Mettler Toledo ME204 Balance

1. Before weighing any materials or at a minimum daily, verify the balance according to B.4 – B.9.

2. Place a weigh boat on the balance pan and press **→0/T←** to tare to 0.0000 g.

3. Using a spatula, carefully spoon the chemical or place the material onto the center in a weigh boat. Make sure to close the glass doors to ensure a correct reading. Allow the reading to stabilize and record the measurement.

4. Once completed, remove the weigh boat and sweep out the interior with an anti-static brush.

5. Place the balance in standby by pressing and holding the **→0/T←** button until **STANDBY** appears. The balance will display **MT.GREEN** while in standby mode.

C. Calibration of the Cahn 28 Automatic Electrobalance.

1. The balance must be verified before each use. Record calibration information in the Balance Log (Exhibit G10.1).

2. Turn the balance on by pressing the **POWER** button (located in the back of the balance). The balance should warm for a minimum of 1-hour before use.

3. Set the balance to the **A** Range (**200 mg – 1 µg**).

4. Press the **TARE** button on the balance (the balance must read 0.00 mg before starting).

5. Place the 200.01 mg weight on balance Pan A and wait until ready. Record the answer in the Balance Log. Press the **CAL** button to correct the reading and record the corrected value in the Balance Log.

6. Remove the weight from Pan A and allow the balance to stabilize to zero. If the reading has drifted, recalibrate the balance.

7. Place the 10.05 mg weight on balance Pan A and wait until ready. Record the answer in the Balance Log.

8. Place the 50.04 mg weight on balance Pan A and wait until ready. Record the answer in the Balance Log.

D. Weighing Materials (weigh pans used in toxicity tests).

1. Before weighing any materials, calibrate the balance according to A.4 – A.9.

2. Using the forceps, carefully place the weigh pan onto the center of Pan A. Make sure to close the glass door to ensure a correct reading. Allow the reading to stabilize and record the measurement. Record the date that the measurements are being made and your initials on the bench sheet.

3. Once completed, remove the pan.

4. Once all measurements are completed, measure the 10.05 mg and 50.04 mg weights. Allow the readings to stabilize and record the answers in the Balance Log.

5. Turn the balance off by pressing the **POWER** button (located in the back of the balance).

References

Instrument Manual

Exhibits

Exhibit G10.1: Balance Log.

Exhibit G10.1: Balance Log (Mettler-Toledo ME204).

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Mettler-Toledo ME204 Balance Log

Instrument	Serial number	Calibration date	Calibration company
Mettler-Toledo ME204 Balance	C006982938	01-16-25	Laboratory Instrument Services
Certified Weights	20410	03-03-23	Precision Weighing

Date	Internal Cal. (√)	Level (√)	Certified weight (g) and control limits*							Analyst initials
			0.0102	0.1005	1.0000	10.0001	49.9999	99.9999	149.9998	
			0.0100–0.0104	0.0985–0.1025	0.9990–1.0010	9.9901–10.0101	49.9499–50.0499	99.8999–100.0999	149.8498–150.1498	

*Certified weights must be within established control limits before measurements are made.
If certified weights exceed control limits, perform balance internal calibration and re-measure certified weights.

SOP G10-Revision 4–Exhibit G10.1

Subject: Balance and Weight Calibration

Exhibit G10.1: Balance Log (Cahn 28 Automatic Electrobalance).

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Cahn 28 Automatic Electrobalance Log

Instrument	Serial number	Calibration date	Calibration company
Cahn 28 Automatic Electrobalance	41520	01-16-25	Laboratory Instrument Services
Certified Weights (10.05 and 50.04 mg)	20410	03-03-23	Precision Weighing
Certified Weight (200.01 mg)	1000055140	03-03-23	Precision Weighing

Date	Zero Tare (v)	Calibration weight (200.01 mg)		Initial verification weights (mg) (before measurements are taken)		Final verification weights (mg) (after measurements are taken)		Analyst initials
		Initial	Final (calibrated to 200 mg)	10.05 Control Limit* (9.85-10.25)	50.04 Control Limit* (49.04-51.04)	10.05 Control Limit* (9.85-10.25)	50.04 Control Limit* (49.04-51.04)	

*Initial and final verification weights must be within established control limits. If verification weights exceed control limits, re-zero and re-calibrate balance and re-analyze samples.

SOP G10-Revision 3– Exhibit G10.1

Subject: Mechanical Pipette Calibration and Polypropylene Cylinder Volume Verification

Approval

Title	Name	Signature	Date
Laboratory Supervisor	Kelley E. Keenan		06-01-25
Quality Assurance Officer	Jim Sumner		06-01-25

Document Revision History

Effective Date	Revision number	Review Type	Evaluators	Revisions
12-01-00	0	Internal	Jim Sumner (ETS)	Original document
01-03-12	1	Internal	Jim Sumner (ETS)	• Updated exhibits during document review. Added leak test to procedure.
06-20-12	2	External (TVA) Internal	William Rogers (TVA) Donald Snodgrass (TVA) Rick Sherrard (TVA) Jim Sumner (ETS)	• Graduated cylinder volume verification procedure added.
10-31-12	3	External (TVA) Internal	William Rogers (TVA) Donald Snodgrass (TVA) Rick Sherrard (TVA) Jim Sumner (ETS)	• Clarified graduated cylinder volume verifications for only polypropylene cylinders. • Updated exhibit to provide acceptable ranges for polypropylene cylinders and included a table with these limits.
06-01-25	4	Internal	Blake Lancaster (ETS)	• Updated Z factor table to be based on only current temperature and air pressure • Updated pipette and graduated cylinder verification log sheets to include serial numbers and verification start and end times. • Updated recommended limits.

Scope and Application

To maintain the accuracy of mechanical pipettes and graduated cylinders used for toxicity and analytical testing.

Subject: Mechanical Pipette Calibration and Polypropylene Cylinder Volume Verification

Quality Control

1. All mechanical pipettes must be calibrated once every 6 months. In addition, mechanical pipettes must pass a leak check. 100 mL and 250 mL polypropylene graduated cylinders must be verified annually.

2. All pipettes must be within the recommended limits established by the manufacturer. If the readings are out of range, notify the Laboratory Supervisor and take the pipette out of use. If the pipette cannot be corrected to meet the recommended limits through cleaning or adjustment, the pipette must be sent to an approved instrument calibration company (e.g. Precision Weighing) to be recalibrated. Refer to Table G11.1 for the recommended limits for pipette volumes used to calibrate mechanical pipettes.

Table G11.1: Recommended limits for mechanical pipette volumes

Repeater Pipettes:

True value (ml)	Recommended Limits (g)		Recommended CV (%)
	Lower	Upper	
0.0500	0.0491	0.0509	< 1.5%
0.1000	0.0982	0.1018	< 1.5%

Adjustable Finnpipettes:

True value (ml)	Recommended Limits (g)		Recommended CV (%)
	Lower	Upper	
0.0100	0.0092	0.0108	< 3.0%
0.0500	0.0492	0.0508	< 1.5%
0.1000	0.0992	0.1008	< 1.5%
0.2500	0.242	0.258	< 1.5%
0.5000	0.492	0.508	< 1.5%
1.0000	0.992	1.008	< 1.5%

3. Polypropylene graduated cylinders must be within ± 5% of the true value. If the readings are out of range, notify the Laboratory Supervisor and take the cylinder out of use. Refer to Table G11.2 for the recommended limits for cylinder volumes used to verify polypropylene graduated cylinders.

Table G11.2: Recommended limits for polypropylene graduated cylinder volumes

True value (mL)	Recommended Limits (g)	
	Lower	Upper
100	95	105

Equipment and Materials

Balance (Mettler-Toledo ME204, or equivalent)
Class "S" Weights
100-mLbeaker
Deionized water
Thermometer
Adjustable-Fixed Mechanical Pipettes (Eppendorf® and Fisherbrand HandyStep®)
Mechanical pipette tips
100 mL and 250 mL Graduated Cylinders
Pipette Volume Verification Logsheet
Graduated Cylinder Volume Verification Logsheet

Procedure

A. Mechanical Pipette Verification Procedure.

1. Calibrate the balance before pipette verification (SOP-G10).

2. Complete the following on the Pipette Volume Verification Logsheet (Exhibit G11.1):
   - Analyst's initials
   - Date checked
   - Pipette serial number
   - Pipette manufacturer

- Water and ambient temperature
- Volumes checked
- Time started

3. Fill a 100-ml beaker with deionized water. Allow the water to adjust to room temperature. Record the temperature on the Logsheet.

4. Perform a leak test by placing a tip on the pipette and filling with deionized water to the maximum nominal volume and placing the pipette on a vibration-free stand. Observe the meniscus in the tip for 1 minute. After 1 minute, there should be no visible droplet formation at the tip. If there are no signs of droplet formation, proceed with the calibration check. If droplet formation is observed, successful maintenance and corrective action must be taken or the pipette must not be used.

5. Place a weigh boat on the balance pan and **TARE** to 0.0000 mg

6. Draw up the required volume of deionized water.

7. Release the water into the weigh boat and wait until the balance is stable. Record the reading on the Pipette Volume Verification Logsheet (Exhibit G11.1). Repeat this process 10 times. Record verification end time when completed.

8. Continue to verify and record the different pipette volumes.

9. Enter the results into the Fixed-Volume Pipette Calibration excel spreadsheet (Exhibit G11.2). Results are corrected for temperature and air pressure, where a Z-factor is applied (Exhibit G11.3).

B. Polypropylene Verification Procedure.

1. Calibrate the balance before graduated cylinder verification (SOP-G10).

1. Complete the following on the Polypropylene Graduated Cylinder Volume Verification Logsheet (Exhibit G11.4):
   - Date
   - Analyst
   - Water and ambient temperature
   - Volume verified
   - Cylinder type (100 mL, 250 mL, etc.)

- Cylinder identification number

2. Fill an Erlenmeyer flask with deionized water. Allow the water to adjust to room temperature. Record the temperature on the logsheet.

3. Place the cylinder on the balance pan and **TARE** to 0.0000 mg

4. Fill the cylinder with deionized water to the appropriate volume to verify. Wait until the balance is stable. Record the reading on the Graduated Cylinder Volume Verification Logsheet (Exhibit G11.4).

5. Continue to verify and record for each cylinder to be checked.

6. Enter the results into the Fixed-Volume Pipette Calibration excel spreadsheet (Exhibit G11.5). Results are corrected for temperature and air pressure, where a Z-factor is applied (Exhibit G11.3).

References

Instrument Manual

Exhibits

Exhibit G11.1: Example Pipette Volume Verification Logsheet.
Exhibit G11.2: Fixed-Volume Pipette Calibration excel spreadsheet
Exhibit G11.3: Z-factor Correction Factor Table
Exhibit G11.4: Polypropylene Graduated Cylinder Volume Verification Logsheet.
Exhibit G11.5: Polypropylene Graduated Cylinder Volume Verification excel spreadsheet

Subject: Mechanical Pipette Calibration and Polypropylene Cylinder Volume Verification

Exhibit G11.1: Example Pipette Volume Verification Logsheet

Pipette Volume Verification

Analyst			
Date			
Verification start time		End time	

Pipette	**Fisherbrand Handy Step**	Serial number	**17E59354**
Water and ambient temperature (°C)		Digital thermometer SN	
Balance	Mettler-Toledo ME204	Serial number	C006982938

Pipette Volume (mL)	
0.0500	0.1000
1	
2	
3	
4	
5	
6	
7	
8	
9	
10	

1-minute Leak Check		Pass ☐	Fail ☐
Start time		End time	

Subject: Mechanical Pipette Calibration and Polypropylene Cylinder Volume Verification

Exhibit G11.2: Fixed-Volume Pipette Calibration excel spreadsheet

Fixed-Volume Pipette Calibration

Reference: ISO 8655-6:2002 (E)

Analyst:	JS
Date of Calibration:	12-31-24
Calibration due date:	06-25
Pipet manufacturer:	**Finnpipette**
Serial/ID number:	U98294
Nominal Volume:	0.01
Location of use:	Chemistry lab

Test Conditions	**Normal**
Barometric pressure, kPa or mbar	101.2
Relative Humidity, %	87
Ambient temp., degrees C	23.0
Test liquid temp., degrees C	23.0
Z correction factor	1.0035

Leak Test	
Time begin:	0704
Time end:	0705
Pass/Fail	Pass

Balance check	
Balance manufacturer	Mettler Toledo
Serial Number	C006982938
Location of use	Chemistry lab
Weight set serial #	20410

Pipet Calibration

Dial setting:	**0.01**	**units:**	mL
Time begin:	0655		
Replicate	**Mass**	**Units**	**Volume (V)**
m_1	0.0098	g	0.00983
m_2	0.0102	g	0.01024
m_3	0.0101	g	0.01014
m_4	0.0098	g	0.00983
m_5	0.0097	g	0.00973
m_6	0.0099	g	0.00993
m_7	0.0101	g	0.01014
m_8	0.0101	g	0.01014
m_9	0.0098	g	0.00983
m_{10}	0.0098	g	0.00983
Mean mass	0.00993		PASS
Time end:	0705		
Mean vol.	**0.0100**		
s	**0.0002**		
% CV	**1.7794**		
CV Tolerance	PASS		
PASS/FAIL	**PASS**		

COMMENTS:
Mean mass acceptable range: ± 8.0% or 0.0008 g
Mean mass acceptable range: 0.0092 to 0.0108 g
CV tolerance acceptable range: < 3.0%

A **leak test** may be performed by filling the pipette tip with distilled or deionized water to the maximum nominal volume and placing the pipette on a vibration-free stand. Observe the meniscus in the tip for 1 minute. After 1 minute, there should be no visible droplet formation at the tip. If there are no signs of droplet formation, proceed with the calibration check. If droplet formation is observed, successful maintenance and corrective action must be taken or the pipette must not be used.

Complete green-shaded cells only (obtain Z Corr. Factor from Table 1 on Sheet 2). The spreadsheet will populate all unshaded cells for you.

Obtain RE and CV tolerances from the pipettor manufacturer's specifications.

Use appropriate ASTM 1 or 2 tolerances for the balance checks (refer to sheet 3).

If dial setting (volume) is in mls, mass should be recorded in gm. If dial setting (volume) is in µl, mass should be recorded in mg.

Subject: Mechanical Pipette Calibration and Polypropylene Cylinder Volume Verification

Exhibit G11.3: Z-factor Correction Factor Table

Temp degrees C	Air pressure, kPa (mbar)						
	80 (800)	85 (850)	90 (900)	95 (950)	100 (1000)	101.3 (1013)	105 (1050)
15	1.0017	1.0018	1.0019	1.0019	1.002	1.002	1.002
15.5	1.0018	1.0019	1.0019	1.002	1.002	1.002	1.0021
16	1.0019	1.002	1.002	1.0021	1.0021	1.0021	1.0022
16.5	1.002	1.002	1.0021	1.0021	1.0022	1.0022	1.0022
17	1.0021	1.0021	1.0022	1.0022	1.0023	1.0023	1.0023
17.5	1.0022	1.0022	1.0023	1.0023	1.0024	1.0024	1.0024
18	1.0022	1.0023	1.0023	1.0024	1.0025	1.0025	1.0025
18.5	1.0023	1.0024	1.0024	1.0025	1.0025	1.0026	1.0026
19	1.0024	1.0025	1.0025	1.0026	1.0026	1.0027	1.0027
19.5	1.0025	1.0026	1.0026	1.0027	1.0027	1.0028	1.0028
20	1.0026	1.0027	1.0027	1.0028	1.0028	1.0029	1.0029
20.5	1.0027	1.0028	1.0028	1.0029	1.0029	1.003	1.003
21	1.0028	1.0029	1.0029	1.003	1.0031	1.0031	1.0031
21.5	1.003	1.003	1.0031	1.0031	1.0032	1.0032	1.0032
22	1.0031	1.0031	1.0032	1.0032	1.0033	1.0033	1.0033
22.5	1.0032	1.0032	1.0033	1.0033	1.0034	1.0034	1.0034
23	1.0033	1.0033	1.0034	1.0034	1.0035	1.0035	1.0036
23.5	1.0034	1.0035	1.0035	1.0036	1.0036	1.0036	1.0037
24	1.0035	1.0036	1.0036	1.0037	1.0037	1.0038	1.0038
24.5	1.0037	1.0037	1.0038	1.0038	1.0039	1.0039	1.0039
25	1.0038	1.0038	1.0039	1.0039	1.004	1.004	1.004
25.5	1.0039	1.004	1.004	1.0041	1.0041	1.0041	1.0042
26	1.004	1.0041	1.0041	1.0042	1.0042	1.0043	1.0043
26.5	1.0042	1.0042	1.0043	1.0043	1.0044	1.0044	1.0044
27	1.0043	1.0044	1.0044	1.0045	1.0045	1.0045	1.0046
27.5	1.0045	1.0045	1.0046	1.0046	1.0047	1.0047	1.0047
28	1.0046	1.0046	1.0047	1.0047	1.0048	1.0048	1.0048
28.5	1.0047	1.0048	1.0048	1.0049	1.0049	1.005	1.005
29	1.0049	1.0049	1.005	1.005	1.0051	1.0051	1.0051
29.5	1.005	1.0051	1.0051	1.0052	1.0052	1.0052	1.0053
30	1.0052	1.0052	1.0053	1.0053	1.0054	1.0054	1.0054

Z correction facotrs are for distilled water as a function of liquid test temperature and air pressure.
Z values are in microliters per milligram.

Reprinted from ISO 8655-6:2002(E).

If your laboratory does not have a mercury barometer, the true local barometric pressure for locations throughout North Carolina may be obtained from the North Carolina State University, State Climate Office of North Carolina website at http://www.nc-climate.ncsu.edu/.

Subject: Mechanical Pipette Calibration and Polypropylene Cylinder Volume Verification

Exhibit G11.4: Polypropylene Graduated Cylinder Volume Verification Logsheet.

Polypropylene Graduated Cylinder Volume Verification

Pan balance calibration:

Instrument	Serial number	Calibration company
Denver Instruments Company XL-3K	B039128	Laboratory Instrument Services
Certified Weights	20410	Precision Weighting

Date	Level verification (V)	Certified weight (g) (control limits*)			Analyst initials
		50.0001 (47.5001 - 52.50001)	100.0001 (95.0001 - 105.0001)	200.0002 (190.0002 - 210.0002)	

Date: _____

Analyst: _____

Water and ambient temperature: _____

Cylinder type	Cylinder identification number	Volume Verified (mL)	Weight of volume in cylinder (g)	Corrected Volume* (mL) (weight of volume in cylinder x Z-factor)
100 mL	1	100 mL		
100 mL	2	100 mL		
100 mL	3	100 mL		
100 mL	4	100 mL		
100 mL	5	100 mL		
100 mL	6	100 mL		
100 mL	7	100 mL		
100 mL	8	100 mL		
100 mL	9	100 mL		
100 mL	10	100 mL		
100 mL	11	100 mL		
100 mL	12	100 mL		
100 mL	13	100 mL		
100 mL	14	100 mL		
100 mL	15	100 mL		
100 mL	16	100 mL		
100 mL	17	100 mL		
100 mL	18	100 mL		
100 mL	19	100 mL		
100 mL	20	100 mL		
100 mL	21	100 mL		
100 mL	22	100 mL		
100 mL	23	100 mL		

*The corrected volume for each cylinder must be within ±5% of the true volume or volume verified (100 mL volume acceptance limit = 95 – 105 mL. If a cylinder exceeds these limits, it must be taken out of service.

Subject: Mechanical Pipette Calibration and Polypropylene Cylinder Volume Verification

Exhibit G11.5: Polypropylene Graduated Cylinder Volume Verification excel spreadsheet

Polypropylene Graduated Cylinder Volume Verification

Pan balance calibration:

Instrument	Serial number	Calibration company
Denver Instruments Company XL-3K	B039128	Laboratory Instrument Services
Certified Weights	20410	Precision Weighting

Date:	12-17-24
Analyst:	JP
Water and ambient temperature:	23.3°C
Z Factor:	1.0036

Cylinder type	Cylinder identification number	Volume Verified (mL)	Weight of volume in cylinder (g)	Corrected Volume* (mL) (weight of volume in cylinder x Z-factor)
100 mL	1	100 mL	98	98
100 mL	2	100 mL	97	97
100 mL	3	100 mL	97	97
100 mL	4	100 mL	99	99
100 mL	5	100 mL	.	.
100 mL	6	100 mL	99	99
100 mL	7	100 mL	98	98
100 mL	8	100 mL	98	98
100 mL	9	100 mL	99	99
100 mL	10	100 mL	99	99
100 mL	11	100 mL	98	98
100 mL	12	100 mL	98	98
100 mL	13	100 mL	98	98
100 mL	14	100 mL	99	99
100 mL	15	100 mL	97	97
100 mL	16	100 mL	98	98
100 mL	17	100 mL	99	99
100 mL	18	100 mL	97	97
100 mL	19	100 mL	99	99
100 mL	20	100 mL	99	99
100 mL	21	100 mL	99	99
100 mL	22	100 mL	100	100
100 mL	23	100 mL	97	97
100 mL	24	100 mL	97	97

Subject: Thermometer Calibration

Approval

Title	Name	Signature	Date
Laboratory Supervisor	Kelley E. Keenan		06-01-25
Quality Assurance Officer	Jim Sumner		06-01-25

Document Revision History

Effective Date	Revision number	Review Type	Evaluators	Revisions
12-01-00	0	Internal	Jim Sumner (ETS)	Original document
09-01-09	1	External (TVA, Environmental Standard, Inc.) Internal	William Rogers (TVA) Cynthia Russell (TVA) Rick Sherrard (TVA) Rock Vitale (Environmental Standards, Inc.) Jim Sumner (ETS)	• Incubator, refrigerator, and drying oven temperatures section and associated exhibits removed and added to SOP C1.
06-01-25	2	Internal	Jim Sumner (ETS)	• Updated reference to 24th Edition of Standard Methods.

Scope and Application

To calibrate thermometers used to measure the temperature of water samples used in toxicity tests, wastewater, receiving water, drinking water, and incubators/refrigerators.

Quality Control

Standardization: All mercury, red spirit-filled, hand-held thermometers, and meters, which measure temperature, must be verified at least **annually** (once every calendar year) with traceable NIST thermometers.

In addition, any thermometer used to monitor the temperature of incubators or water baths must be verified at least **quarterly** with traceable NIST thermometers.

All digital thermometers must be verified at least **quarterly** with traceable NIST thermometers.

Subject: Thermometer Calibration

Equipment and Materials

Mercury-filled thermometers
Hand-held thermometers
Digital thermometers
NIST traceable thermometers
Rinse bottle
Deionized water
Waste container
Sand
Thermometer Calibration Logsheet

Procedure

A. Calibration.

1. All mercury-filled and hand-held thermometers and meters, which measure temperature, must be calibrated with a traceable NIST thermometer (refer to Exhibit G12.1 for NIST certification records).

    a. Thermometers used to measure the temperature within temperature-controlled incubators are calibrated at 20.0 ± 1.0°C, 25.0 ± 1.0°C, or 35.0 ± 0.5°C.

    b. Thermometers used to measure the temperature within refrigerators are calibrated at 0.1 - 6.0°C.

    c. Thermometers used to measure the temperature within drying ovens are calibrated at 60.0 ± 2.0°C, 105 ± 2.0 °C, or 180 ± 2.0 °C.

    d. General use thermometers are calibrated at 25.0 ± 1.0°C and 0.1 - 4.0°C.

2. Place all thermometers to be calibrated at the desired temperature in a beaker of water (or sand for ovens) with the NIST thermometer. Place the beaker of water (or sand) in the respective temperature-controlled incubator for calibrating thermometers at 20.0 ± 1.0°C, 25.0 ± 1.0°C, or 35.0 ± 0.5°C, a refrigerator for calibrating thermometers at 0.1 ± 6.0°C, or the respective drying oven for calibrating thermometers at 60.0 ± 2.0°C, 105 ± 2.0°C, or 180 ± 2.0 °C. Allow the water (or sand) to reach the desired temperature. For each thermometer, compare the temperature measurement to the measurement on the NIST thermometer. If the thermometer has an immersion line, it must be utilized. If

it does not have an immersion line, both calibration and measurements must be made under the same water depth.

3. Record the date of calibration, serial number of the thermometer, calibration temperature, and correction factor (temperature adjustment of the thermometer to equal the measurement of the NIST thermometer) on the Thermometer Calibration Logsheet (Exhibit G12.2) and on a label affixed to the thermometer.

4. Thermometers having breaks in the mercury or having a correction factor > 1.0 are taken out of service.

References

Standard Methods for the Examination of Water and Wastewater, 24th Edition, 2023. American Public Health Association, 800 I Street, NW, Washington DC 20001-3710.

Exhibits

Exhibit G12.1: NIST Thermometer Certification Records.
Exhibit G12.2: Example Thermometer Calibration Logsheet.

Subject: Thermometer Calibration

Exhibit G12.1: NIST Thermometer Certification Records.

PRECISION pwi WEIGHING, INC

Precision Weighing
1949 Evans Road
Cary, North Carolina 27513
Phone: (919) 678-0077 * Fax: (919) 678-0078
Email: pweighing@aol.com

Client:	Contact:	Department:
ETS Inc.	Jim Sumner	Lab

Description:	Digital Thermometer	Calibration SOP:	2020 Rev 1.1
Instrument ID:	61786906	Serial Number:	61786906
Manufacturer:	Fisher Scientific	Operating Range:	0 to 180 °C
Model:	15-078-2	Instrument Range:	-350 to 2200 °C
Calibration Interval:	Annually	Calibration Tolerance:	+/- 1 deg C

Calibration Notes: *Nominal Targets are 0,20,25,35,44.5,60,105,150,180*

Calibration Data ("As left" data is identical to "As found" data, if "As left" is blank)

Test Points	Units	Standard	As Found	Error	As Left	Error	Pass/Fail
0	°C	0.0036	0.1	0.0964			Pass
20	°C	20.0022	19.9	-0.1022			Pass
25	°C	24.9429	24.9	-0.0429			Pass
35	°C	34.9700	34.8	-0.1700			Pass
44.5	°C	44.3375	44.4	0.0625			Pass
60	°C	59.8021	59.8	-0.0021			Pass
105	°C	104.690	105.0	0.310			Pass
150	°C	149.333	150.0	0.667			Pass
180	°C	179.713	180.2	0.487			Pass

Instrument Found in Tolerance? Yes
Instrument Left in Tolerance? Yes

Calibration Date:	20 Nov 24	Calibration Due Date:	Nov 25

Test Standards:	Standard ID:	Expiration Date:
Digital Thermometer	230670827	03 Oct 25

Technicians Remarks: None.

Calibration Performed By: 20 Nov 24

Approved Signature: Jim Sumner

Subject: Thermometer Calibration

Exhibit G12.2: Example Thermometer Calibration Logsheet.

Thermometer Calibration

Traceable Thermometer:	Fisher Scientific Digital Thermometer	Date:	
Probe Serial number:	61786906	Analyst:	
Meter Serial number:	61789314		
Calibration date:	Probe and Meter: 11-20-24	Ice Point of Traceable Thermometer: _____	
Re-calibration due:	Probe and Meter: 11-2025		

Thermometer Serial Number	Location	Location Serial Number	Thermometer Temperature (°C)	Traceable Thermometer Temperature (°C)	Correction Factor (°C)
----------	Refrigerator # 1	BA64722418	Not in use.		
96-01587	Refrigerator # 2	BA65013194			
96-01566	Refrigerator # 3	BA11411547			
6959	Refrigerator # 4 TOP	WA42501619			
4934	Refrigerator # 4 BOTTOM	WA42501619			
4565	Refrigerator # 5 TOP	WA42500397			
6315	Refrigerator # 5 BOTTOM	WA42500397			
95-02120	Refrigerator – Algae/YWT	E2001648367			
WB72043714	BOD Incubator # 1	WB72043714	Calibrated to 20.0°C		
7126	BOD Incubator # 1 Thermometer	WB72043714			
WB94157442	BOD Incubator # 2	WB94157442	Calibrated to 20.0°C		
7049	BOD Incubator # 2 Thermometer	WB94157442			
WB22114719	BOD Incubator # 3	WB94157442	Calibrated to 20.0°C		
6209	BOD Incubator # 3 Thermometer	WB94157442			
WB42676093	Reagent Incubator # 1	WB42676093	Calibrated to 25.0°C		
5030	Reagent Incubator # 1	WB42676093			
WB41340472	Toxicity Incubator # 1	WB41340472	Calibrated to 25.0°C		
160724968	Toxicity Incubator # 1 MIN/MAX	WB41340472			
6272	Toxicity Incubator # 1 TOP	WB41340472			
4673	Toxicity Incubator # 1 BOTTOM	WB41340472			
WB22114693	Toxicity Incubator # 2	WB22114693	Calibrated to 25.0°C		
130761061	Toxicity Incubator # 2 MIN/MAX	WB22114693			
4676	Toxicity Incubator # 2 TOP	WB22114693			
4755	Toxicity Incubator # 2 BOTTOM	WB22114693			
WB95219633	Toxicity Incubator # 4	WB95219633	Calibrated to 25.0°C		
160761060	Toxicity Incubator # 4 MIN/MAX	WB95219633			
4547	Toxicity Incubator # 4 TOP	WB95219633			
6216	Toxicity Incubator # 4 BOTTOM	WB95219633			

Note: If correction factor is ≥ 1.0°C, the thermometer must be taken out of service.

SOP G12–Revision 2-Exhibit G12.2

Subject: Desiccator Maintenance

Approval

Title	Name	Signature	Date
Laboratory Supervisor	Kelley E. Keenan		06-01-25
Quality Assurance Officer	Jim Sumner		06-01-25

Document Revision History

Effective Date	Revision number	Review Type	Evaluators	Revisions
12-01-00	0	Internal	Jim Sumner (ETS)	Original document
03-01-20	1	External (TVA) Internal	Rick Sherrard (TVA) Jim Sumner (ETS)	• Updated procedure and benchsheet to include the serial number of the meter used to perform chlorine.
06-01-25	2	Internal	Jim Sumner (ETS)	• Updated procedure during document review.

Scope and Application

To ensure proper desiccator maintenance by drying the desiccant.

Quality Control

Desiccator is to be dried at least monthly.

Equipment and Materials

Desiccator with plastic desiccant pans
Color Indicating Desiccant
Desiccator Maintenance Log
103 -105 °C forced air oven
Metal drying pan
High temperature gloves

Subject: Desiccator Maintenance

Procedure

A. Drying the Desiccant

1. Empty the desiccant into a metal drying pan. Desiccant should be blue if dry or purple if it has absorbed moisture.

2. Place the metal pan into the 103 – 105°C oven until desiccant turns blue.

3. After the desiccant turns blue, use the high temperature gloves and pour the desiccant into the plastic desiccant trays.

4. Place the plastic trays into the bottom of each desiccator.

5. Record the date and the desiccant is dried in the desiccator maintenance log.

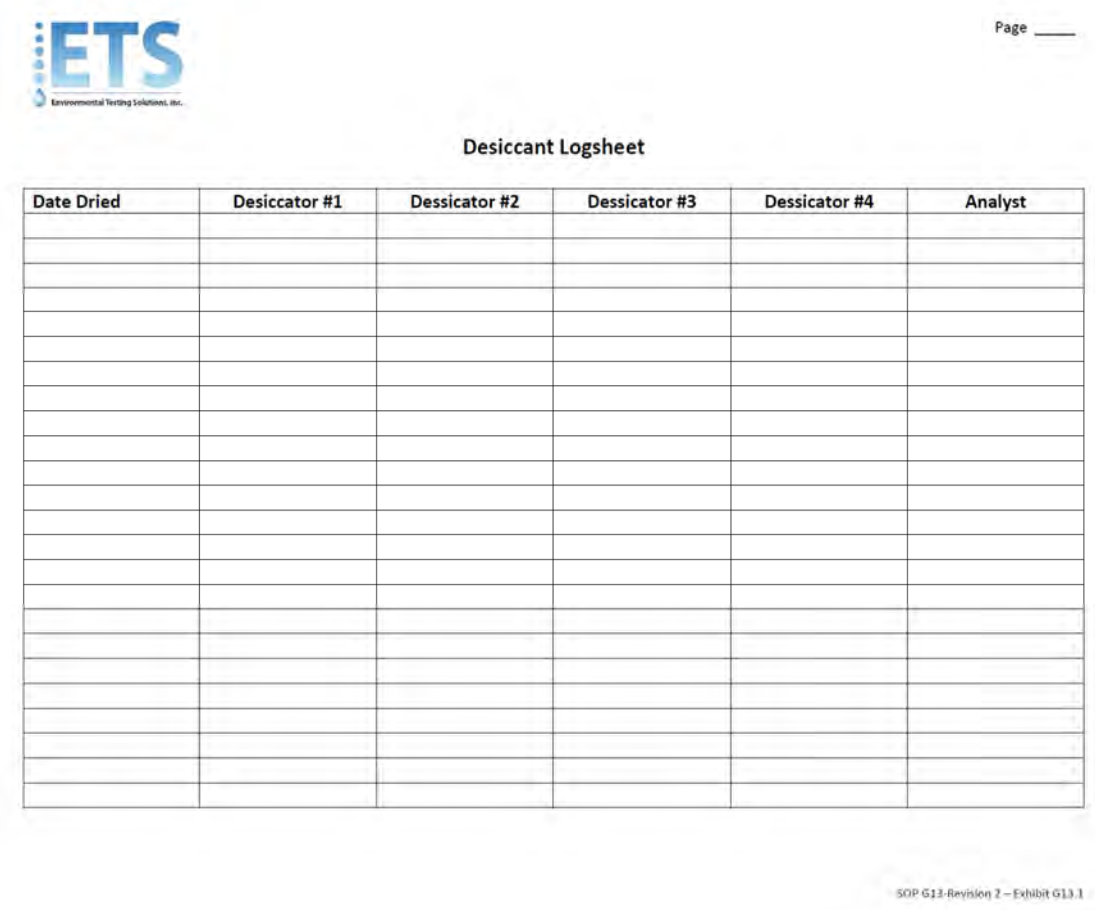
6. Desiccator is to be dried at least monthly.

Exhibits

Exhibit G13.1: Desiccator Maintenance Benchsheet.

Subject: Desiccator Maintenance

Exhibit G13.1: Desiccator Maintenance Benchsheet.



Page ____

Desiccant Logsheet

Date Dried	Desiccator #1	Dessicator #2	Dessicator #3	Dessicator #4	Analyst

SOP G13-Revision 2 – Exhibit G13.1

Subject: Illumination

Approval

Title	Name	Signature	Date
Laboratory Supervisor	Kelley E. Keenan		06-01-25
Quality Assurance Officer	Jim Sumner		06-01-25

Document Revision History

Effective Date	Revision number	Review Type	Evaluators	Revisions
12-01-00	0	Internal	Jim Sumner (ETS)	Original document
01-03-12	1	Internal	Jim Sumner (ETS)	• Updated exhibits during document review.
03-01-20	2	External (TVA) Internal	Rick Sherrard (TVA) Jim Sumner (ETS)	• Updated procedure and bench sheet to describe the current quality control and measurement of luminosity.
06-01-25	3	Internal	Jim Sumner (ETS)	• Updated exhibits during document review.

Scope and Application

To document the photoperiod and intensity of light necessary for maintaining cultures and toxicity tests.

Quality Control

1. The intensity of light (luminosity) in all incubators used for maintaining cultures and toxicity tests and the algae culture area must be measured at least **quarterly** or when light fixtures are replaced. The luminosity should be maintained between 50 – 100 foot-candles (ft-c) for maintaining cultures and toxicity tests and between 360 – 440 foot-candles for maintaining algae cultures. For toxicity tests in support of AL NPDES discharges, the luminosity of incubators should be measured each day that the tests are performed. This is documented on the test specific bench sheets.

2. A uniform photoperiod of 16-hours light and 8-hours dark must be maintained in all incubators used for maintaining cultures and toxicity tests as well in the fish culture laboratory. Automatic timers and internal timers within the incubators regulate this photoperiod. The correct time displayed on these timers is documented and adjusted according to SOP-G16. The correct

photoperiod is documented **quarterly** when luminosity is documented. Timers may need to be adjusted when incubators are serviced. Algae cultures are maintained under constant illumination.

Equipment and Materials

Light meters
Incubators
Light fixtures
Window screen or black electrical tape
Automatic timers
Luminosity Log Sheet

Procedure (Meter: TACKlife LM01 Light Meter, SN 201612033203)

A. Verification of Light Meter.

Prior to taking luminosity measurements, verify the luminosity of the meter. To verify the Tacklife LM01 Light Meter, compare the luminosity displayed to a separate meter (Leaton L860 LUX Meter, SN not provided). Place each probe face up on the surface of an area to be measured. Each display must be with 10% of one another prior to taking luminosity measurements. Measure and record the luminosity of each meter and percent difference on the Luminosity Log Sheet (Exhibit G14.1).

B. Measuring Luminosity.

1. Turn the light meter on and set the meter to read in foot-candles (ft-c).

2. Place the probe with the sensor face up on the surface of the area to be measured. Check the luminosity throughout the area where cultures or tests are maintained. In the fish culture laboratory to monitor the luminosity of each tank, hold the probe with the sensor face up close to the water surface.

3. If the luminosity is outside the 50 – 100 ft-c suggested range (360 – 440 ft-c for algae), adjust the light intensity by adding or removing light fixtures. Window screen or black electrical tape may also be placed over light fixtures to adjust the light intensity in specific areas.

4. Once the luminosity has been corrected in all areas, measure and record the luminosity at the four corners and middle of each shelf in each incubator, the location were algae is

cultured and at the water surface for each tank in the fish culture laboratory. Allow readings to stabilize before recording in the Luminosity Log. Measurements must be in foot-candles (ft-c) and reported to the nearest 1 ft-c.

5. Verify and document in the Luminosity Log that the time is correct, and the photoperiod program is set for 16-hour light and 8-hours dark for each incubator and in the fish culture laboratory.

Safety and Hazardous Waste Management

Safety glasses, gloves and lab coats should always be worn.

Review Policy-P6: General Safety Policy and Policy-P9: Radiation Protection Policy for additional safety requirements.

References

USEPA. October 2002. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, 5th ed. EPA-821-R-02-012. US Environmental Protection Agency, Cincinnati, OH.

USEPA. October 2002. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms, 4th ed. EPA-821-R-02-013. US Environmental Protection Agency, Cincinnati, OH.

USEPA. October 2002. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms, 3rd ed. EPA-821-R-02-014. US Environmental Protection Agency, Cincinnati, OH.

TNI Standard. Management and Technical Requirements for Laboratories Performing Environmental Analysis. EL-V1-ISO-2016-Rev2.0. The NELAC Institute, PO Box 2439, Weatherford, TX 76086.

Instrument Manuals

Exhibits

Exhibit G14.1: Luminosity Log Sheet.

Subject: Illumination

Exhibit G14.1: Luminosity Log Sheet.

Luminosity Log

Meter:	TACKlife Digital LUX Meter, LM01			Date:	
Serial number:	201612033203			Analyst:	
Purchase date:	06-23-17				
Light Meter Verification	Comparison to Leaton L830 Lux Meter SN not provided. (must be ± 10% prior to taking measurements)	TACKlife (ft-c)			
		Leaton (ft-c)			
		% Difference			

Incubator number	Shelf	Luminosity (ft-c) at location indicated on shelf Acceptable range = 50 – 100 ft-c	Incubator number	Shelf	Luminosity (ft-c) at location indicated on shelf Acceptable range = 50 – 100 ft-c
Incubator 1 SN: WB41340472	A	< 50 Do not use.	Incubator 2 SN: WB22114693	A	< 50 Do not use.
	B			B	
	C			C	
	D			D	
	E			E	
	F			F	
	Initials	Verified photoperiod program (16-hours light and 8-hours dark) and current time.		Initials	Verified photoperiod program (16-hours light and 8-hours dark) and current time.
Incubator 4 SN: WB95219633	A	< 50 Do not use.	Incubator 5 SN: WB60643874	A	< 50 Do not use.
	B			B	
	C			C	
	D			D	
	E			E	
	F			F	
	Initials	Verified photoperiod program (16-hours light and 8-hours dark) and current time.		Initials	Verified photoperiod program (16-hours light and 8-hours dark) and current time.

SOP G14-Revision 3– Exhibit G14.1

Luminosity Log

Meter:	TACKlife Digital LUX Meter, LM01	Date:	
Serial number:	201612033203	Analyst:	

Incubator number	Shelf	Luminosity (ft-c) at location indicated on shelf Acceptable range = 50 – 100 ft-c	Incubator number	Shelf	Luminosity (ft-c) at location indicated on shelf Acceptable range = 50 – 100 ft-c.
Incubator 6 SN: WB42667925	A	< 50 Do not use.	Incubator 7 SN: WB42668003	A	< 50 Do not use.
	B			B	
	C			C	
	D			D	
	E			E	
	F			F	
	Initials	Verified photoperiod program (16-hours light and 8-hours dark) and current time.		Initials	Verified photoperiod program (16-hours light and 8-hours dark) and current time.
	A	< 50 Do not use.	Algae Culture Area	Luminosity (ft-c) at location indicated Acceptable range = 360 – 440 ft-c	
	B				
	C				
	D				
	E				
	F				
	Initials	Verified photoperiod program (16-hours light and 8-hours dark) and current time.			

SOP G14-Revision 3– Exhibit G14.1

Subject: Illumination

Luminosity Log, Fish Culture Laboratory

Meter:	**TACKlife Digital LUX Meter, LM01**			**Date:**	
Serial number:	**201612033203**			**Analyst:**	
Purchase date:	**06-23-17**				
Light Meter Verification	Comparison to Leaton L830 Lux Meter SN not provided. (must be ± 10% prior to taking measurements)	TACKlife (ft-c)			
		Leaton (ft-c)			
		% Difference			

Verified photoperiod program (16-hours light and 8-hours dark) and current time.	**Analyst:**	

Breeding Tank Set	ID #	Luminosity (ft-c) at water surface Acceptable range = 50 – 100 ft-c	Breeding Tank Set	ID #	Luminosity (ft-c) at water surface Acceptable range = 50 – 100 ft-c
A	1		B	1	
	2			2	
	3			3	
	4			4	
	5			5	
	6			6	
C	1		D	1	
	2			2	
	3			3	
	4			4	
	5			5	
	6			6	
E	1		F	1	
	2			2	
	3			3	
	4			4	
	5			5	
	6			6	
G	1		H	1	
	2			2	
	3			3	
	4			4	
	5			5	
	6			6	

Stock tank 1: ____ 2: ____ 3: ____ 4: ____ 5: ____ 6: ____ 7: ____ 8: ____

SOP G14-Revision 3– Exhibit G14.1

Subject: Reagent, Stock Standard, and Chemical Preparation

Approval

Title	Name	Signature	Date
Laboratory Supervisor	Kelley E. Keenan		06-01-25
Quality Assurance Officer	Jim Sumner		06-01-25

Document Revision History

Effective Date	Revision number	Review Type	Evaluators	Revisions
12-01-00	0	Internal	Jim Sumner (ETS)	Original document
09-01-09	1	External (TVA, Environmental Standard, Inc.) Internal	William Rogers (TVA) Cynthia Russell (TVA) Rick Sherrard (TVA) Rock Vitale (Environmental Standards, Inc.) Jim Sumner (ETS)	• Updated exhibits during document review. • Exhibits modified to include the initials and date of the author making entries to the logbooks. Logbooks modified to provide additional traceability. • Provided further clarification on the use of expiration dates.
01-03-12	2	Internal	Jim Sumner (ETS)	• Updated exhibits during document review. • Provided further clarification on the use of expiration dates.
06-20-12	3	External (TVA) Internal	William Rogers (TVA) Donald Snodgrass (TVA) Rick Sherrard (TVA) Jim Sumner (ETS)	• Expiration dates changed from 1 year or 5 years from open to 1 year or 5 years from receipt.
03-01-20	4	External (TVA) Internal	Rick Sherrard (TVA) Jim Sumner (ETS)	• Provided additional guidance for chemicals and consumables used for toxicity testing, which require testing prior to use.
06-01-25	5	Internal	Jim Sumner (ETS)	• Updated reference to 24th Edition of Standard Methods.

Scope and Application

To provide traceability of all reagents, stock standards, and chemicals used in the laboratory.

Toxicity Testing Quality Control

In addition to being "checked in" (as described in this SOP), analytical testing or toxicity "checks" may be required of certain chemicals or consumables used for toxicity testing. These chemicals or consumables must be tagged as "do not use" prior to testing. After testing is performed and the chemicals or consumables meet quality control requirements, these items will be tagged with "Acceptable to Use" and the date that testing was performed. At this time, these chemicals or consumables are cleared for

use. Chemicals and consumables that fall under this requirement include: cubitainers, medicine cups, Solo cups, MarineMix and synthetic water chemicals used in toxicity tests.

Equipment and Materials

Reagent log
Stock standard log
Chemical log

Procedure

A. "Checking-In" Reagents, Stock Standards, Chemicals and Consumables.

1. All reagents, stock standards, and chemicals must be checked-in and assigned an identification number, date received, date opened sticker (or placed into use), and a safety label. Information is recorded in one of the following logs (Exhibit G15.1):
    - Chemical Log
    - Reagent Log
    - Stock Standard Log

2. Chemicals, stock standards, and reagents are used on or before their expiration dates. At the time of analysis, any unused, expired chemicals, stock standards, and reagents are disposed. Expiration dates for chemicals, stock standards, and reagents (that are not assigned as expiration date by the manufacturer) are assigned in a MONTH – DAY – YEAR format. These chemicals, stock standards, and reagents expire on the expiration date.

3. The chemical log is used to record information for all chemicals and consumables (e.g. Petri dishes, filters). Each chemical or consumable is assigned an identification number (CHM#).

    a. Each chemical receives a label with the CHM#, date received, expiration date, date opened (or placed into use), and initials. The expiration date for dry chemicals that are not assigned an expiration date by the manufacturer is five years from the date received. The expiration date for wet chemicals, that are not assigned an expiration date by the manufacturer, is one year from the date received. Consumables, such as containers, medicine cups, solo cups, acetone or acid used for cleaning purposes only, are not assigned an expiration date.

b. If a Safety Data Sheet (SDS) is not received with the chemical or is not contained in the SDS Log, contact the supplier to obtain the SDS. SDS's for each chemical must be stored in the SDS Log. As new chemicals are received, SDS's are replaced with the most current SDS received from the manufacturer.

c. If the chemical is not compatible with other types of chemicals, it must be stored in a separate location. This may include acids, oxidizers, ammonium hydroxide, flammables, etc.

d. If more than one container of the same chemical is received, assign each container the same chemical number (provided they are the same lot or batch). For example. If two containers are received, they should be labeled "1 of 2" and "2 of 2".

e. Record the following information for the chemical or consumable in the chemical log:

- Page number
- Chemical number
- Analyst
- Date
- Chemical name
- Test or application used for
- Material type
- Toxicity Testing Quality Control required?
- Manufacturer
- Lot number
- Chemical concentration
- Number or volume received
- Date received
- Expiration date
- Received by
- Comments
- Storage
- Hazards
- Personal hygiene (health, fire, reactivity, personal equipment)

4. The reagent log is used to record information for all reagents. Each reagent is assigned an identification number (INR#).

   a. Each reagent receives a label with the INR#, date prepared or received, expiration date (1 year from preparation or manufacturer expiration date), date opened (or placed into use) and initials. The expiration date for reagents, that are not assigned an expiration date by the manufacturer, is one year from the date received or preparation date.

   b. If an SDS is not received with the reagent or is not contained in the SDS Log, contact the supplier to obtain the SDS. SDS's for each reagent must be stored in the SDS Log. As new reagents are received, SDS's are replaced with the most current SDS received from the manufacturer.

   c. If the reagent is not compatible with other types of chemicals, it must be stored in a separate location. This may include acids, oxidizers, ammonium hydroxide, flammables, etc.

   d. If more than one container of the same reagent is prepared or received, assign each container the same reagent number (provided they are the same lot or batch). For example. If two containers are prepared or received, they should be labeled "1 of 2" and "2 of 2".

e. Record the following information for the reagent in the reagent log:
- Page number
- Reagent number
- Analyst
- Date
- Reagent name
- Test or application used for
- Type
- Manufacturer
- Lot number
- Reagent concentration
- Number and/or volume
- Date received/prepared
- Expiration date
- Received/prepared by
- Directions for preparing the reagent
- Reagent prepared from
- Storage
- Hazards
- Personal hygiene (health, fire, reactivity, personal equipment)

5. The stock standard log is used to record information for all stock standards. Each stock standard is assigned an identification number (INSS#).

a. Each stock standard receives a label with the INSS#, date prepared or received, expiration date (1 year from preparation/received or manufacturer expiration date), date opened (or placed into use) and initials. The expiration date for stock standards, that are not assigned an expiration date by the manufacturer, is one year from the date received or preparation date.

b. If the stock standard is not compatible with other types of chemicals, it must be stored in a separate location. This may include acids, oxidizers, ammonium hydroxide, flammables, etc.

c. If more than one container of the same stock standard is prepared or received, assign each container the same stock standard number (provided they are the same lot or batch). For example. If two containers are prepared or received, they should be labeled “1 of 2” and “2 of 2”.

f. Record the following information for the stock standard in the stock standard log:

- Page number
- Stock standard number
- Analyst
- Date
- Standard name
- Test or application used for
- Type
- Manufacturer
- Lot number
- Stock concentration
- Number and/or volume
- Date received/prepared
- Expiration date
- Received/prepared by
- Directions for preparing the stock
- Stock prepared from
- Storage
- Hazards
- Personal hygiene (health, fire, reactivity, personal equipment)

Subject: Reagent, Stock Standard, and Chemical Preparation

Safety and Hazardous Waste Management

Safety glasses, gloves and lab coats should always be worn.

Review Policy-P6: General Safety Policy and Policy-P9: Radiation Protection Policy for additional safety requirements.

References

Standard Methods for the Examination of Water and Wastewater, 24th Edition, 2023. American Public Health Association, 800 I Street, NW, Washington DC 20001-3710.

USEPA. October 2002. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, 5th ed. EPA-821-R-02-012. US Environmental Protection Agency, Cincinnati, OH.

USEPA. October 2002. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms, 4th ed. EPA-821-R-02-013. US Environmental Protection Agency, Cincinnati, OH.

USEPA. October 2002. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms, 3rd ed. EPA-821-R-02-014. US Environmental Protection Agency, Cincinnati, OH.

TNI Standard. Management and Technical Requirements for Laboratories Performing Environmental Analysis. EL-V1-ISO-2016-Rev2.0. The NELAC Institute, PO Box 2439, Weatherford, TX 76086.

Exhibits

G15.1: Example Chemical, Reagent and Stock Standard Logs

G15.1: Example Chemical, Reagent and Stock Standard Logs

Page _____

ETS Environmental Testing Solutions, Inc.

Chemical Log

Chemical number: CHM_____

Analyst: _____

Date: _____

Chemical name: _____

Test or application used for: _____

Material type: □ Consumable □ Chemical

Toxicity Testing:

Quality Control required? □ Yes □ No

Manufacturer: _____

Lot number: _____

Chemical concentration: _____

Number and/or volume: _____

Date received: _____

Expiration date: _____

Dry Chemicals = 5 years from receipt, if not assigned by manufacturer
Wet Chemicals = 1 year from receipt, if not assigned by manufacturer
Consumables = no expiration date, unless assigned by manufacturer

The Chemical identified on this form was discarded on or before the expiration date listed above.

Received by: _____

Comments:

_____

_____

_____

_____

Storage: _____

Hazards:

_____

_____

_____

Personal hygiene:

Health: _____
Fire: _____
Reactivity: _____
Personal equipment: _____

SOP G15-Revision 5–Exhibit G15.1

Page _____

Environmental Testing Solutions, Inc.

Reagent Log

Reagent number: INR_____

Analyst: _____

Date: _____

Reagent name: _____

Test or application used for: _____

Type: □ Purchased □ Laboratory prepared

Manufacturer: _____

Lot number: _____

Reagent concentration: _____

Number and/or volume: _____

Date received/prepared: _____

Expiration date: _____ (1 year from preparation/receipt, if not assigned by manufacturer)

The Reagent identified on this form was discarded on or before the expiration date listed above.

Received/prepared by: _____

Directions for preparing reagent:

_____

_____

_____

_____

Reagent prepared from: _____ (CHM # or INR #)

Storage: _____

Hazards:

_____

_____

_____

Personal hygiene:

Health: _____

Fire: _____

Reactivity: _____

Personal equipment: _____

SOP G15-Revision 5– Exhibit G15.1

Page _____

Environmental Testing Solutions, Inc.

Stock Standard Log

Stock standard number: INSS_____

Analyst: _____

Date: _____

Standard name: _____

Test or application used for: _____

Type: □ Purchased □ Laboratory prepared

Manufacturer: _____

Lot number: _____

Stock concentration: _____

Number and/or volume: _____

Date received/prepared: _____

Expiration date: _____ (1 year from preparation/receipt, if not assigned by manufacturer)

The Stock Standard identified on this form was discarded on or before the expiration date listed above.

Received/prepared by: _____

Directions for preparing stock:

_____

_____

_____

_____

Stock standard prepared from: _____ (CHM #)

Storage: _____

Hazards: _____

_____

_____

Personal hygiene:

Health: _____

Fire: _____

Reactivity: _____

Personal equipment: _____

SOP G15-Revision 5– Exhibit G15.1

Subject: Time Calibration

Approval

Title	Name	Signature	Date
Laboratory Supervisor	Kelley E. Keenan		06-01-25
Quality Assurance Officer	Jim Sumner		06-01-25

Document Revision History

Effective Date	Revision number	Review Type	Evaluators	Revisions
06-01-11	0	Internal	Jim Sumner (ETS)	Original document
06-01-11	1	External (TVA) Internal	Cynthia Russell (TVA) Rick Sherrard (TVA) Jim Sumner (ETS)	• Method for calibrating, recording and documenting time.
09-01-11	2	External (TVA) Internal	Rick Sherrard (TVA) Jim Sumner (ETS)	• Corrected typographical error.
03-01-20	3	Internal	Jim Sumner (ETS)	• Updated procedure to include NELAP requirements.
06-01-25	4	Internal	Jim Sumner (ETS)	• Updated procedure to calibrate clocks to NIST Official U.S. Time.

Scope and Application

To calibrate various clocks used to record and document time throughout the laboratory

Quality Control

All clocks, which are used to document or record time, must be calibrated quarterly.

Equipment and Materials

Various clocks
NIST Official U.S. Time
Cell phone
Time Calibration Log

Subject: Time Calibration

Procedure

1. All clocks, which are used to document or record time, must be calibrated quarterly.

2. Clocks are calibrated to the nearest minute, since the recording of time on individual log sheets is documented to the nearest minute in the laboratory.

3. The NIST Official U.S. Time is used as the standard to calibrate clocks within the laboratory.
   https://time.gov

4. A cell phone is calibrated to the NIST Official U.S. Time as indicated above and will be used to calibrate all individual clocks throughout the laboratory. This is documented in Time Calibration Log (Exhibit G16.1).

References

Time and Frequency Division, National Institute of Standards and Technology (NIST) Measurement Laboratory, US Department of Commerce.

Instrument Manual

Exhibits

Exhibit G16.1: Time Calibration Log.

Subject: Time Calibration

Exhibit G16.1: Time Calibration Log.

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Environmental Testing Solutions, Inc.

Time Calibration Log

Standard:

NIST Official U.S. Time
https://time.gov

Date	NIST U.S. Official Time	All Clocks Calibrated to NIST Official U.S. Time (Analyst Initials)

SOP G16-Revision 4–Exhibit G16.1