AGP Lab Blank Analysis and Blank Data Review

1. SCOPE AND APPLICATION

    1.1. This method was developed by laboratory management as part of the quality assurance protocol for Algal Growth Potential (AGP) testing. It is applicable to both freshwater and saltwater AGP tests.

2. SUMMARY OF METHOD

    2.1. Procedure for analyzing laboratory method blanks to monitor contamination of a typical sample. Types of contaminations include nutrients carried over in the inoculum, unwanted materials in DI rinse water, and foreign particles from improperly cleaned sample bottles. These blanks are not to be treated differently from other samples in terms of preparation, set-up, and counting. This method also describes the review and corrective action for data from lab blanks.

    2.2. Interferences:

        2.2.1. Contaminated glassware

        2.2.2. Airborne particulates including dust and microscopic organisms

3. EQUIPMENT AND SUPPLIES

    3.1. General

        3.1.1. Traceable Bench Conductivity Meter, SN 170814827. Calibrated per SOP TA-06.36

        3.1.2. Fisherbrand Accumet AB315 pH/mV Meter. Calibration per SOP TA-06.43

        3.1.3. Labeling tape (blue or red colored tape, for freshwater and saltwater blanks respectively, is generally used to indicate blank containers but any color that differentiates from an actual sample may be used).

        3.1.4. 30 mL disposable plastic cup, one per blank

        3.1.5. Calibrated thermometer (located in B246 thermometer drawer) or certified digital thermometer. See SOP TA-06.13 and LB-035.

        3.1.6. *AGP Sample Log-in Record* logbook

    3.2. For freshwater

        3.2.1. AGP sample container

    3.3. For saltwater

        3.3.1. Erlenmeyer flask

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3.3.2. Mettler Toledo MS 403TS/00 Balance, SN B815673069 (Calibrated per SOP TA-06.34-1.0)

3.3.3. Small weigh boat (plastic if available)

3.3.4. Metal spatula

3.3.5. Magnetic stir plate

3.3.6. Magnetic stir bar

3.3.7. Stir bar retriever

4. REAGENTS AND STANDARDS

4.1.1. For freshwater

4.1.1.1. Deionized (DI) water

4.1.1.1.1. Distilled water can be substituted for DI water at the analyst's discretion (ex. problems with DI water system). If distilled water is used, substitute distilled water for DI water throughout the SOP.

4.1.2. For saltwater

4.1.2.1. Autoclaved DI water (autoclaved per SOP BG-08)

4.1.2.1.1. **Program 3 for large media cycle (121.0, S=0:30).** Program 2 is for smaller media cycle (121.0, S=0:15) and Program 1 gravity cycle (121, S=0:15) is for empty glassware.

4.1.2.2. Crystal Sea® Marine Mix Bioassay Laboratory Grade artificial sea salts

5. METHODS

5.1. For freshwater blank

5.1.1. Label a clean AGP bottle using blue label tape. Write "Lab Blank", date collected, and analyst's initials on the label.

5.1.2. Fill the bottle with DI water from the sink.

5.1.3. Record "Lab Blank" in the *AGP Sample Log-in Record* under the LIMS # column.

5.1.4. Measure conductivity, salinity, and pH with the appropriate meters. Measure temperature with the certified/calibrated digital thermometer. Record the values in the appropriate columns of the *AGP Sample Log-in Record*.

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5.1.4.1. See SOP TA-06.43 for measuring pH and TA-06.36 for measuring conductivity and salinity.

5.1.5. Autoclave and filter the blank.

5.1.5.1. Autoclave and filter per SOP TA-08.01 for samples <1.0 ppth salinity.

5.1.6. If the blank is to be set up immediately, leave it out. Otherwise, place the blank in the walk-in cooler.

5.1.6.1. Set up blanks per SOP TA-08.02.

5.2. For saltwater blank

5.2.1. Label a clean 500-mL Erlenmeyer flask using red label tape. Write “Salt Blank”, date collected and analyst's initials on the label.

5.2.2. Fill the flask at least half full with autoclaved DI water.

5.2.2.1. Not filling the flask completely allows room to add DI water for diluting, if necessary.

5.2.3. Scoop an estimated 3-4 grams of artificial sea salts into a weigh boat using the metal spatula. Use balance for weighing if desired.

5.2.4. Place the flask on the magnetic stirrer. Place the stir bar in the flask and turn the stirrer on.

5.2.5. Add a small amount of artificial sea salts to the flask using the metal spatula and wait until the salts are dissolved.

5.2.6. Measure the salinity. (See SOP TA-06.25 for measuring salinity)

5.2.6.1. If the salinity is less than 6.8 ppth (parts per thousand), repeat STEP 5.2.5. above until the salinity measures 7 ± 0.2 ppth.

5.2.6.2. If the salinity of 7 ± 0.2 ppth is exceeded, dilute with autoclaved DI water. Repeat salinity measurement as needed.

5.2.6.2.1. If dilution is needed, gradually add DI water to the mixture. Swirl to mix, then repeat the salinity measurement.

5.2.7. Remove the flask from the stirrer and turn the stirrer off. Use the stir bar retriever to remove the stir bar from the flask.

5.2.8. Record “Salt Blank” in the *AGP Sample Log-in Record* under the LIMS # column.

5.2.9. Measure conductivity, salinity, and pH with the appropriate meters. Measure temperature with the certified/calibrated digital thermometer. Record the values in the appropriate columns of the *AGP Sample Log-in Record*.

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5.2.9.1. See SOP TA-06.43 for measuring pH and SOP TA-06.36 for measuring conductivity. Record the final salinity as measured in Step 5.2.6.

5.2.9.2. The comments section for the "Salt Blank" should mention the amount of marine mix salt added to 500 mL of Autoclaved DI water.

5.2.10. Filter the blank.

5.2.10.1. Filter per SOP TA-08.01 using a 0.45 µm filter.

5.2.11. If the blank is to be set up immediately, leave it out. Otherwise, place the blank in the walk-in cooler.

5.2.11.1. Set up blanks per SOP TA-08.02.

6. DATA REVIEW

6.1. Select the blank result that will be connected to the run based on SOP TA-08.07, Section 4.15. Also, determine if lab blank results passed or failed based on the MDLs listed in SOP TA-08.07, Section 2.2.

6.1.1. This should be done before each run's printed Excel file is verified in case the data must be qualified.

6.1.2. Blanks are not uploaded into LIMS. This is for quality control checks.

6.2. After the data for each run has been checked by two-person verification, open the folder X:\Labs\Lab5\Biology\BenchCommon\ToxCommon\AGP\Control Charts

6.2.1. Data-Entry Verification per SOP BG-01.

6.3. Open Excel spreadsheet named **Control charts-updated**.

6.4. Select either the **FW Lab Blanks** tab for freshwater blanks or **SW Lab Blanks** tab for saltwater blanks, scroll down to the first available line, and enter the lab blank's set-up date under **Analysis Date** header.

6.4.1. If folder is locked, please see supervisor.

6.5. Enter the lab blank values for days 10, 12, and 14 in the next three columns. When a value is not available for a day, enter NV (no value).

6.6. In the column titled Reported Value, enter in the result that was selected in Step 6.1.

6.7. Fill each remaining cell in the row with the formula from the cell above it.

6.7.1. See Microsoft Excel Help for help with table functions.

6.8. Click the **FW Blanks Last 20** tab or **SW Blanks Last 20** and update the source data of the chart to reflect the newly entered data. This chart should reflect the 20 most recent lab blank maximum values.

6.9. Repeat steps 6.4-6.8 for the duplicate samples.

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6.10. If the lab blank's maximum value exceeds the MDL (0.300 for freshwater and 3.00 for saltwater), then all samples from that batch should be qualified with a "V" qualifier, which indicates that the analyte was detected in both the blank and the sample. Qualify sample data where necessary and notify a supervisor of the problem.

6.11. If a blank exceeds the control limit or if 2 of 3 successive blanks exceed the warning limit, the samples should be set up again in the next run (where sufficient sample is available). Notify a supervisor of the problem.

6.11.1. See supervisor if the blanks exceed the MDL and the control limit or if 2 of 3 successive blanks exceed the warning limit.

6.11.2. The value of the current Mean + 1SD of the 20 most recent blanks is the warning limit. The value of the current Mean+2SD is the control limit.

6.11.3. If the subsequent lab blank also exceeds the limit in question, analyses should stop while possible causes of contamination and error in method are examined.

6.12. Save the file and exit but see supervisor first.

7. DATA ARCHIVAL

7.1. Use a black, waterproof pen for all bench sheets and logbooks.

7.2. Record information in *AGP Sample Log-in Record* logbook.

7.3. To correct raw data entries, place a single line through the incorrect or illegible entry, write the corrected entry near the error, date and initial the correction. Do not overwrite entries.

7.3.1. The corrected entry is written as near as possible to the error or in the comment box on the appropriate bench sheet.

7.3.2. If the correction requires an explanation, write a comment and reference the error. Initial and date the explanation. Do not attempt to put all information (correction and comments) into a small space.

7.4. Use sequential alphabetical annotation to identify errors and corresponding comments (use A for first error and use A for corresponding explanatory comment; use B for second error, etc.).

8. QUALITY CONTROL

8.1. Refer to QA manual for corrective action and handling out-of-control data.

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9. SAFETY/HAZARDOUS WASTE MANAGEMENT

9.1. Proper laboratory personal protection, including lab hood or ventilation system, lab coat, gloves and safety glasses, is required when working with chemicals and unprocessed samples.

9.2. Refer to Laboratory Quality Manual for sample disposal

9.3. Refer to the Laboratory Health and Safety Manual and Hazardous Waste Management

10. REFERENCES

10.1. DEP Laboratory Quality Manual

10.2. DEP Laboratory Health and Safety Plan

10.3. DEP SOP TA-08.01, Initial Algal Assay Sample Handling

10.4. DEP SOP TA-08.02, Algal Growth Potential (AGP) and Limiting Nutrient (LN) Algal Assay Set-Up

10.5. DEP SOP TA-08.07, Algal Growth Potential and Limiting Nutrient Data Verification and LIMS Upload

10.6. DEP SOP TA-08.09, Sample Custody, Logbook Labels and Prep Worksheet Manager for Algal Growth Potential (AGP) Samples

10.7. DEP SOP TA-06.34, Calibration and Use of the Mettler Toledo PB403-TS/00 Balance

10.8. DEP SOP TA-06.43, Calibration and Use of Fisherbrand Accumet AB315 pH/mV Meter

10.9. DEP SOP TA-06.36, Calibration and Use of the Fisherbrand Traceable™ Bench Conductivity Meter for Conductivity and Salinity

10.10. DEP SOP BG-01, Data-Entry Verification

10.11. DEP BG-08, STERIS AMSCO Series 250LS Autoclave Calibration and Use

10.12. DEP LB-035, Calibration of Lab Thermometers using a Constant Temperature Calibration Bath

APPENDIX OF CHANGES

12/20/2007 Added Scope and Application; Summary of Method, and Appendix of Changes.

10/08/2013 Reformatted SOP.

09/11/2014 Changed temperature measurement to digital thermometer.

01/28/2015 Formatted for 508 compliance.

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08/17/2015 Updated links. Added instrument Serial Numbers.

09/15/2016 Updated links and references.

09/14/2017 Updated the Control Chart file location in the Data Review section.

09/05/2018 Changed font to Calibri; minor formatting correction; included new meters in Equipment and Supplies section; removed reference to Tox telephone location in Sec 3.1.5; removed reference to Prep Worksheet label from Sec 4.1 and 4.2; clarified Sec 5.1; removed section about AGP results getting V-qualified.

08/02/2019 Updated links

09/03/2020 Split equipment and reagents sections. Updated data archival section with universal language.

07/08/2021 Updated links for current updates. Updated control chart directions based on updated version.

09/07/2022 Updated links, updated pathways, and checked for grammar.

09/07/2023 Updated links. Added general autoclave SOP. Updated tape colors. Added comment notes for “Salt Blank.” Added to SOP section.

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