OPERATIONS CHALLENGE LABORATORY PROCEDURE 2021

Version 02.17.2021

Goal

Analyzing and determining total suspended solids, conductivity / TDS and performing solids mass balance across a treatment system.

Introduction

Laboratory results are valuable as a record of plant operations. This data lets the operator know how efficiently the plant is running and help predict and prevent troubles that may be developing within the various processes. Laboratory results are required as a record of performance for regulatory agencies and are of value to the operations staff and design engineers for performance optimization, troubleshooting, determination of loadings, and for determining when plant expansions are necessary. For these reasons, laboratory tests should be conducted as carefully and consistently as possible and according to appropriate analytical methods.

The total suspended solids (TSS) test is one of the most important process control and regulatory tests the operator / analyst can run. The results of the test can be used to estimate process loadings to treatment plants as a whole and the efficiency of various processes throughout the plant; calculate the mean cell residence time (MCRT) or sludge age; and determine the sludge wasting rate, loadings to solids handling processes, and removal and capture efficiency of solids handling processes. The TSS test result is also needed to calculate the sludge volume index (SVI).

Samples can be collected in glass or plastic bottles. The samples may be refrigerated at <6 °C until they are ready for analysis with the samples being brought to room temperature just before analysis. Samples should not be held longer than 7 days. Prewashing of the filter papers is required for TSS analysis to remove loose filter material to obtain accurate and precise results. For the purpose of this event, the teams will assume that this prewashing step is already complete.

Team members will be required to analyze samples representing a normal treatment process including influent, effluent and intermediate process samples for total suspended solids (gravimetric method) and TDS after calibration of YSI Xylem MultiLab Meter based on conductivity as well as calculate a mass balance across the treatment system to evaluate the removal efficiency of various treatment trains and other data related to solids.

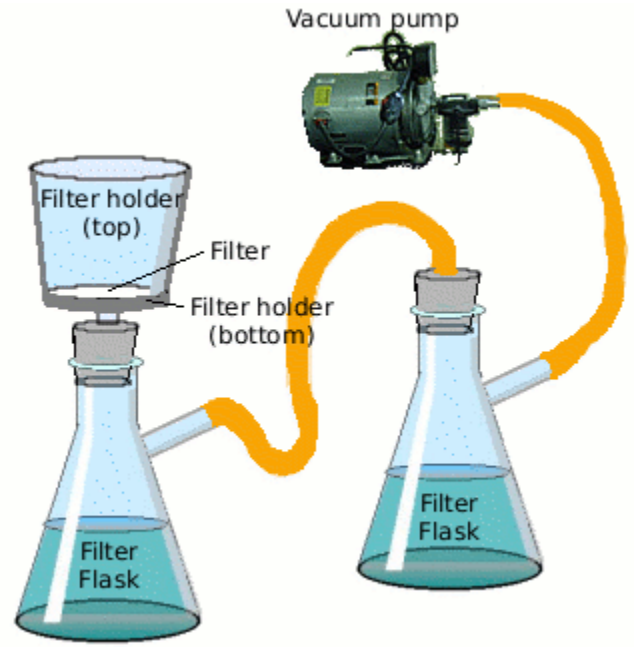
Finally, penalties referenced on the Lab Event Judges Sheets are incorporated by reference into this document. Any discrepancies in language will be resolved by the judging team.

Equipment Check List and Supplies

USA BLUE BOOK	
44565	Black Sharpie Marker, Fine Tip (3) and 3 pencils
40676	Safety Pipet Filler Bulbs (2)
205152	Corning Low Form Polypropylene 250 ml beakers (6)
37930	25 ml graduated cylinder, Nalgene Class B (2)
37940	50 ml graduated cylinder, Nalgene Class B (1)
37950	100 ml graduated cylinder, Nalgene Class B (4)
37960	250 ml graduated cylinder, Nalgene Class B (2)
37980	1000 ml graduated cylinder, Nalgene Class B (1)
202822	500 ml Wash Bottles (Actual set up may include 1 Quart and 1 500ml)
39828	2000 ml PP Beaker Container for Used Pipets (1)
41489	50 g Calibration Mass
37742	Kimble Serological Pipet, 10ml, Large Opening (3)
90148	OHAUS Pioneer PX Analytical Balance, 84 mg, 0.1mg
40947	Bottle Carrier (2) For samples
39829	5000 ml Waste Beakers (2)
31410	Filter Funnel (Magnetic)
63050	Multipurpose Trays (4) For Used Glassware and Carboy overflow
40853	500 ml Polypropylene Beakers (1) Wash Water for TDS Meter
35493	500 ml Wide Mouth Sample Bottles (8)
35450	DI Carboy with spigot, 10L
31072	Glass Filter Flask - 1000ml (2)
31320	Forceps (2)
72109	10 Quart plastic buckets for trash (1)
40792	Timer (2)
39170	USA Bluebook TSS Grade Glass Fiber Filters 47mm, 100/pk
39842	Vacuum Pump
24769	Vacuum Tubing
34660	Aluminum Weighing Pans
33265	Stopper, Size 8, One Hole, 1 lb pack (about 10 stoppers)
28980	Tongs
39919	1/8” NPT(M) x 3/8” Hose Barb Fitting
36989	Kim Wipes
	Calculators (2)
	Cookie Sheet (approximate size 14” x 20”)

	S, M, L, XL nitrile gloves
	Paper Towels
	YSI Xylem
1FD560Y	MultiLab Dual Channel
301710Y, 060906	4310 Conductivity Probe and 1413 uS conductivity solution

Total Suspended Solids Set-Up



General Notes

1. Team Captain tells the Head Judge they are ready to begin and the Head Judge says "START" to signal the beginning of the event. The Head judge and one other judge will be the timekeepers.
2. Event is complete when all tasks have been completed and Team Captain hands in the work sheets to the Head judge and says the team is finished.
3. To ensure a fair contest and to avoid challenges, judges will not speak to contestants while the event is being performed.
4. The Event Coordinator will settle disputes with input from the event judges.
5. All team members must participate in the event, but are not limited to performing only one task.

6. After the event, the Head Table Judge may explain to the Team Captain what was done incorrectly, but will NOT reveal penalty points.
7. Team members may ask judges questions before the beginning of the event, but the Judge(s) may choose not to answer the question, depending on the question asked. Questions related to specific steps in the procedure will not be answered.
8. 3 Pencils, 3 Sharpies and 2 Calculators will be provided for each set-up. Teams may bring their own to substitute if they wish.
9. All bench sheets must be completed in their entirety. Any blanks will counted as incorrect with an associated penalty.

NOTE: ALL STEPS OF THE PROCEDURE MUST BE PERFORMED FROM MEMORY. NO BOOKS OR PRINTED MATERIALS ARE ALLOWED IN THE LABORATORY COMPETITION AREA.

SETUP

Teams will have two minutes before beginning the event to organize items on the tables. Any item, with the exception of the vacuum pump, vacuum flasks, carboy, analytical balance and conductivity meter may be moved. The Head Judge will time the setup. At the end of the setup time, the judge will say "TIME", team members must remove their hands from the table. Judges will then place bench sheets face down on the tables. See "General Notes #1" above for instructions.

NOTE TO EVENT COORDINATORS

For the pre-prepared TSS filters, event coordinators may utilize media or samples of their choice to provide some weight to the papers. Remember that the team members are weighing for a final weight and then calculating the TSS in mg/l so it will be helpful to provide them with filters with an appropriate sample weight. Event Coordinators should use the TSS_Calculation_Sheet from the Team Resource Center to enter the initial filter weight and sample volume to be utilized.

For the TSS preparation please use the TSS_Preparation_Sheet. Clean water with no sample shall be utilized for the TSS preparation and teams shall be judged on procedure only. There will be no solids in the samples team members are filtering.

LABELING OF GLASSWARE AND TSS PANS

Labeling must be completed before any rinsing of bottles, measuring of samples and prior to placement of any sample into any container.

Please note that this does not mean the competitor must wait for all containers to be labeled before proceeding with the competition. As each individual bottle or container is labeled, the team member may begin the next steps of the procedure.

All graduated cylinders, beakers, pans and any other container must be properly and legibly labeled according to the bench sheet provided. There will be a total of 3 sharpies provided for labeling.

The following samples will be utilized for the purpose of this event: Influent (500 ml), Primary Effluent (500 ml), Aeration Basins (1 & 2 – 500 ml each), Secondary clarifier effluent (2 – 500 ml bottles), and Final effluent (2 – 500 ml bottles).

Label and proceed as follows:

1. Label the graduated cylinders for TSS analysis as follows (the information in parenthesis is not to be included on the label):
    1. 25 ml – INF (Influent)
    2. 25 ml – INF Dup (Influent - Duplicate)
    3. 50 ml – PRI (Primary Effluent)
    4. 250 ml – SEC (Secondary Effluent)
    5. 250 ml – EFF (Final Effluent)
    6. 1000 ml – BLK (Blank)

2. Label five (5) 250 ml beakers for the TDS test as follows:
    1. BLK (Blank)
    2. SEC (Secondary Effluent)
    3. EFF (Final Effluent)
    4. EFF Dup (Final Effluent - Duplicate)
    5. TDS STD (TDS Calibration Standard)

3. Label three (3) 100 ml graduated cylinders for the TDS test as follows:
    1. BLK (Blank)
    2. SEC (Secondary Effluent)
    3. EFF (Final Effluent)

    NOTE: EFF cylinder can be utilized to measure Effluent Duplicate sample.

4. Label TSS pans as follows (Labels shall be placed on the inside bottom of the pans):
    1. INF (Influent)
    2. INF Dup (Influent Duplicate)

3. PRI (Primary Effluent)
4. AER 1 (Aeration Basin 1)
5. AER 2 (Aeration Basin 2)
6. SEC (Secondary Effluent)
7. EFF (Final Effluent)
8. BLK (Blank)

5. Mix all samples and the calibration solution by inverting 5 times. NOTE: A single inversion consists of taking a bottle from the upright position (12 o'clock) and turning it 90° (3 o'clock), then back to the upright position again. The intent of mixing the samples it to produce a homogenous sample for TSS and TDS/conductivity analysis. The intent is that the sample be mixed immediately prior to rinsing of the beaker and measuring of the sample. It is NOT acceptable to mix samples and allow them to sit on the counter for any period of time. **For the purposes of this event immediately shall mean within 5 seconds.**

6. Rinse all beakers, graduated cylinders and pipettes with sample. For clarity purposes, the aluminum pans do not need rinsed either with sample or with DI water.

7. For TSS Analysis, using rinsed graduated cylinders, measure the following amounts for the corresponding samples and proceed with the TSS testing procedure:
    1. 15 ml – INF (Influent)
    2. 15 ml – INF Dup (Influent - Duplicate)
    3. 50 ml – PRI (Primary Effluent)
    4. 250 ml – SEC (Secondary Effluent)
    5. 250 ml – EFF (Final Effluent)
    6. 400 ml – BLK (Blank)

8. For TSS Analysis of Aeration Basin samples, use 10 ml Large Opening Pipets.

9. For TDS, Using 100 ml graduated cylinder place 200 ml of the following samples into rinsed 250 ml beakers: Blank (Distilled Water), Secondary Effluent, Final Effluent and Final Effluent Duplicate. Proceed with TDS / Conductivity Calibration and Analysis. This will require the team member to fill the 100 ml graduated cylinder twice and pour into the sample beaker. The cylinder does not need to be rinsed in between measured amounts (only rinse prior to measuring first 100 ml aliquot).

PREPARATION AND ANALYSIS OF TOTAL SUSPENDED SOLIDS SAMPLES

For this procedure, there will be a total of eight (8) samples analyzed for total suspended solids (TSS). They would be as follows:

1. Influent
2. Influent Duplicate
3. Primary Effluent

4. Aeration Basin 1
5. Aeration Basin 2
6. Secondary Effluent
7. Final Effluent
8. Blank

1. Obtain a prewashed filter from the box for each sample using the tweezers (forceps).
2. Weigh each of the filter papers and place them in your labeled pans. Place the filter paper onto the balance and close the balance door. Start the timer and wait the allotted 10 seconds. Record the initial weight to the nearest 0.0001 g (0.1 mg). Repeat this step for each filter paper.
3. Using tweezers, place the filter paper onto the funnel base and replace the magnetic top portion of the funnel. Turn on the vacuum pump.
4. Starting with the sample with the least amount of visible solids (blank should be analyzed first and then move on to effluent, secondary effluent, primary effluent, influent and aeration basins), add the sample to the filter funnel. When using a graduated cylinder, rinse the cylinder with water and add the rinsate to the filter funnel with the sample. When using wide opening pipet, rinse pipet with water by removing the bulb and using the distilled water bottle to rinse the inside of the pipette. All rinse water should be added to the filter funnel with the sample. Rinse each cylinder and pipette twice ensuring all sides are rinsed with water. When using a pipette, use a new pipette for each new sample (Aeration Basin 1 & 2).
5. Allow the sample to completely pass through the filter paper. **This means that no standing water should be visible on the pad and no sample would be lost if the funnel were to be removed from the base.**
6. Using the DI water squirt bottle, rinse the filter paper and inside of the funnel with three (3) portions of approximately 10 ml rinse water ensuring the entire circumference of the funnel is rinsed. Wait between each rinse until the water has completely drained. **(For the purpose of judging, this shall mean that there is no standing or ponded water on the filter paper that would spill off of the base if the top were to be removed.)** Ensure you are rinsing the sides of the funnel to move any solids onto the filter paper and off of the funnel.
7. Turn off the vacuum pump and remove the filter paper and place in the appropriate aluminum pan. Repeat steps for remaining samples.
8. **NOTE: DO NOT OVERFILL the filter flask**. Empty the flask into the liquid waste container as necessary to ensure that filtered water does not enter the bypass filter flask or vacuum pump.
9. Place all sample pans on the drying rack (cookie sheet) and place rack in the designated drying area.
10. Put all used glassware, trash and samples into appropriate containers and place back at their starting points of the event and dry any spills on the table. The entire table does not need to be dried. Only areas where there is visible water must be dried.
11. Complete all highlighted areas of the bench sheet as required with correct information. Must be legible.

WEIGHING OF DRIED / DESSICATED SAMPLES AND CALCULATIONS

1. Visually check that the balance is properly adjusted and the bubble is within the circle. NOTE: No actual adjustments are necessary if the bubble is found to be outside of the circle.
2. Visually ensure the weighing pan is free of debris and dust.
3. Using the tweezers (forceps), place the 50.0 gram verification weight on the balance pan and record the value on the bench sheet. **STEPS 1-3 must be complete prior to proceeding with any other measurements on the balance. These steps shall only be performed once during the entire event but must be completed prior to using the balance for any weighing of samples or filter papers.**
4. Using the analytical balance, weigh each pre-prepared, dried filter paper and record the final weight of the sample on the provided bench sheet. After each filter has been placed on the balance and the door to the balance has been closed, start the time for 10 seconds. Record the weight shown after time goes off.
5. Return each sample filter to the appropriate pan and place back on the cookie sheet. Do not throw filter paper / sample away.
6. Calculate the TSS in milligrams per liter. Please note: There is not a "correct" answer for the TSS value; however, the math on your bench sheet must be correct and will verified by a judge assigned to this or by the event coordinator.
7. Complete the bench sheet, filling in all necessary information. Must be legible.

CONDUCTIVITY AND TOTAL DISSOLVED SOLIDS PROCEDURE

TDS Measurement with the YSI MultiLab

Overview:

This procedure describes how to calibrate a YSI 4310 conductivity probe on the YSI MultiLab using 1,413 µS/cm standard, then take total dissolved solids (TDS) measurements.

Materials Needed:

- YSI MultiLab 4010-2W or YSI MultiLab 4010-3W multiparameter benchtop meter
- YSI 4310 conductivity probe for the MultiLab
- 1,413 µS/cm conductivity standard

Calibration Procedure:

1. Rinse the probe with DI water. Blot end and outside of probe with Kim Wipe.
2. The meter will begin the event in the conductivity mode. The units on the screen will be **µS/cm**.
3. Press the **<CAL>** key to enter the calibration screen. The probe's cell constant will be displayed; the unit is **1/cm**.
4. Invert standard 5 times to mix. Rinse 250 ml beaker with approximately 50 ml of standard and discard into the waste container. Ensure the inside surface of the beaker is rinsed completely.
5. Pour approximately 200 ml of fresh 1,413 µS/cm conductivity standard into the 250 ml beaker. Approximately shall mean plus or minus 25 ml.
6. Immerse the 4310 probe in the 1,413 µS/cm conductivity standard. The probe should not be placed directly against the bottom or sides of the calibration container. In addition, the slot in the conductivity probe where the electrodes and temperature sensor are located should be completely immersed in solution – the solution level should be no lower than the blue line in the image to the right.
7. Start the calibration by pressing the **<ENTER>** key. The AutoRead (AR) function will check for stability. While it is checking for stability, the parameter name (μ) and the AR function will flash.
8. Once stability has been reached, the MultiLab will end the calibration and the calibration record will be displayed. A successful calibration has occurred if **+++** appears next to **Sensor**.
9. Press the **F1** key to exit the calibration record and return to the main measurement display.

NOTE: Care should be taken to use fresh standard when calibrating the meter. Using old standard or diluted standard can result in a calibration error. If a calibration error occurs during the competition, move forward with the event steps as noted in the measurement procedure and record "error" in the result area of the benchsheet.

TDS Measurement Procedure:

1. Rinse the probe with DI water. Blot sides and bottom of probe with Kim Wipe.
2. Press the **<M>** key until the TDS value is displayed. The units on the screen will either be **mg/L** or **g/L**.
3. Immerse the 4310 probe in the solution to be measured. The probe should not be placed directly against the bottom or sides of the measurement container. In addition,

the slot in the conductivity probe where the electrodes and temperature sensor are located should be completely immersed in solution.

4. Press the **<AR>** key to use the AutoRead function of the MultiLab. This will ensure the measurement being recorded is stable.
5. Start the AutoRead function by pressing the **<ENTER>** key. While it is checking for stability, the parameter name (TDS) and AR function will flash.
6. Press the 20 second timer.
7. **<HOLD>** will be displayed once stability has been reached. Do not record the measurement until the 20 second timer has gone off even if **HOLD** is displayed.
8. Manually record the measurement value on the bench sheet.
9. Repeat steps 1-8 for any additional samples.
10. At the conclusion of the measurements, the probe must be rinsed and blotted clean.

MASS BALANCE CALCULATION SHEET

See calculation sheet that will need to be completed pulling information from a plant diagram. Examples are provided. Must be legible.

SAFETY PROCEDURES

Safety is of major concern during laboratory operations. As such, "good housekeeping" practices for laboratory operations have been incorporated into the procedure.

1. Safety glasses must be worn at all times. Prescription glasses may be worn in lieu of safety glasses.
2. Approved laboratory gloves must be worn at all times. You may bring your own gloves or wear gloves that we provide. The team member that is completing the mass balance calculations worksheet may remove their gloves during that time period, but if they are performing any other action on the tables, they will need to have gloves on during that time period including assisting with clean-up etc. Please note that if you have a latex allergy, we encourage you to provide your own gloves.
3. Good housekeeping will be required. Competitors will be required to wipe counters, instruments, and work areas ***dry*** with paper towels where they have spilled or splashed any liquids during the competition. The entire surface does not need to be wiped down but all visible water must be dried or penalties will be assessed.
4. Competitors must place used graduated cylinders and used beakers in trays provided (Note: beakers and graduated cylinders must be emptied in waste receptacle prior to going into tray).

5. Sample bottles must have lids in place (screwed on), and placed back in bottle carriers.
6. All trash must be disposed in waste receptacles along with used latex gloves. Latex gloves are considered “contaminated”, and must be removed and disposed in waste receptacles prior to turning in paperwork.
7. Penalties for Safety related issues can also be found on the Judges Penalty Sheets.