|  |  |
| --- | --- |
| **TITLE:** | **Total Suspended Solids Dried at 103 - 105ºC** |
| **ANALYTE:** | **Total Suspended Solids** |
| **FACILITY:** | **Acme WWTP** |
| **REFERENCE METHOD:** | **Standard Methods 2540 D – 1997** |
| **DATE OF ISSUE:** | **7/18/18** |

1. Applicable Matrices
	1. This method is applicable to drinking, surface, and saline waters as well as domestic and industrial waste matrices.
2. Summary of the Test Method
	1. A well-mixed sample is filtered through a weighed standard glass-fiber filter, which is then dried to constant weight at 103 - 105ºC. The increase in weight of the filter represents the total suspended solids (TSS).
	2. After completion of the TSS test, the suspended material may be used to test for Total Volatile Solids (TVS).
3. Safety
	1. Proper personal protection equipment, such as a lab coat, safety glasses, and nitrile gloves should be worn when performing any laboratory test.
4. Equipment and Supplies
	1. Analytical balance – Capable of weighing to 0.1 mg for samples having a mass up to 200 g
	2. Laboratory Oven – Themostatically controlled and capable of maintaining a uniform temperature of 103 to 105°C throughout the drying chamber
	3. Filter funnel (neither Buchner funnels or Gooch crucibles are allowed) – Clean or replace filter frit when dirty or clogged
	4. Vacuum Pump
	5. Glass fiber filters, 4.7 cm diameter
	6. Aluminum weighing dishes
	7. Desiccator with color indicating desiccant – Replace desiccant when turns from blue to purple/pink
	8. Class A graduated cylinders, various sizes
	9. Wide-tip pipettes
	10. Wash bottle filled with distilled water
	11. Magnetic stirrer
	12. Magnetic stir bars
	13. Gloves, tongs, or a suitable holder for moving and handling hot containers after drying
	14. Forceps
5. Reagents and Standards
	1. Distilled water
	2. Proficiency Test samples from an approved provider
6. Interferences
	1. If a large particle appears to be non-representative of the sample and appears that it may skew the results, it may be removed. This is a decision of the analyst, but it is to be noted on the bench sheet.
	2. Heavy loading of the filter surface (taking more than 30-60 seconds to pass through) will decrease the effective pore size which will in turn add a positive error.
	3. Sample size should be limited to no more than 200 mg residue. This prevents a water entrapping crust to form on the filter paper.
	4. Sample size should have a minimum residue of at least 2.5 mg. The WDNR allows a minimum residue of at least 1.0 mg.
	5. Negative errors may be produced from solids that volatilize in the 103 - 105°C range.
7. Sample Collection, Preservation, and Storage
	1. Resistant-glass or plastic bottles may be used for sample collection. Sample material should not adhere to the container walls.
	2. Refrigerate samples from 0 ≤ 6ºC.
	3. Hold times may not exceed 7 days from the time of collection.
8. Quality Control
	1. Initial Quality Control
		1. Method Detection Limit (MDL): The Laboratory is not required to complete a MDL study for TSS. The reporting limit used, as specified by the method, is 2 mg/L if 500 mL of sample volume is used.
		2. All analysts shall perform an IDC once for each lab technician. An IDC is required of all new lab technicians.
	2. On-going Quality Control
		1. Annually a Proficiency Testing (PT) sample must be obtained and analyzed from one of the Wisconsin DNR approved providers.
	3. Filters are dried for at least 8 hours (typically overnight) so re-weighs of the filters are not required.
9. Calibration and Standardization
	1. The balance is checked at least once per month with at least 2 weights, one in the g range and one in the mg range. The acceptance limits for the mg weight are 5% and for the g range is 0.5% or as specified on the Monthly Balance Verification log sheet. The checks are documented on the Monthly Balance Verification log sheet.
	2. The ASTM Class 1 or Class 2 weights used are certified or replaced every 5 years by an outside source and the documentation is kept on file in the laboratory.
	3. Although not required for certification, the analytical balance may be checked and certified annually by an outside source and the documentation kept on file in the laboratory.
	4. The temperature of the solids oven is checked daily when in use and recorded on the Temperature Log or on the TSS bench sheet. This thermometer is NIST certified.
10. Procedure
	1. Prepare the glass-fiber filter: pre-rinse glass-fiber filters the day prior to use by rinsing 3 consecutive times with at least 20 mL DI water. Put the filter into a labeled aluminum weighing dish, and dry in the oven at 103 - 105°C for at least one hour. Record times and lot numbers on the TSS bench sheet. Store cleaned filters in the desiccator or oven until needed.
	2. Record the weight of each filter and weighing dish just prior to use.
	3. Assemble the filtering apparatus. Place the filter paper on the filter support and wet with a small volume of distilled water. Apply the vacuum.
	4. Select the sample volume using the following guidelines:
		1. The sample volume must yield a residual weight of 1.0 to 200 mg.
		2. If the volume filtered fails to meet the requirement for minimum yield of 1.0 mg, at least 500 mL sample volume must be used.
		3. If the time of filtration exceeds 10 minutes, use smaller sample volumes.
		4. Experience with a particular sample will help determine the volume to filter.
			1. Normal Sample Size:

|  |  |
| --- | --- |
| Sample ID | Sample Size |
| Raw Influent | 1 – 300 mL |
| Final Effluent | 100 – 1000 mL |

* 1. Shake or stir the sample to disperse particles. If using a graduated cylinder, immediately pour sample into volumetric cylinder and transfer to the suction funnel. If using a pipette, pipette the sample immediately into the suction funnel. It’s important not to let the solids settle.
	2. After the sample has filtered through the disk to dryness, rinse 3 times with 10-mL portions of distilled water, vacuuming dry between each washing and making sure that particles are washed off the walls of the filtering apparatus. Samples with high dissolved solids may require additional washings.
	3. Continue suction for at least 3 minutes after filtration is complete.
	4. Carefully remove the filter from the filter support with the forceps and put into a labeled aluminum weigh pan. Dry in an oven overnight at 103 - 105ºC. Cool in a desiccator until the filter disk has achieved room temperature. Weigh the filter and pan, and record on the bench sheet.
	5. If drying less than 8 hours, then repeat the drying (at least one hour), desiccating, and weighing steps. The re-weigh result must be within 0.5 mg of the first weigh result or 4%, whichever is less. If it is not then repeat all three steps until the results agree. Document the re-dry dates, times, re-weigh weights, and analysts.
	6. The temperature of the solids oven is checked each day of use and recorded on the Temperature Log or on the TSS bench sheet. The thermometer is verified prior to expiration, or each year, and is NIST traceable.
	7. The filter disk from the total suspended solids test may now be used for the total volatile suspended solids test.
1. Calculations
	1. mg/L TSS = ( A – B ) x 1000
	 sample volume, L

Where:

A = weight of filter disk + dried residue, g

B = weight of filter disk, g

1. Method Detection Limits and Reporting
	1. The Laboratory is not required to analyze a Method Detection Limit for TSS analysis. A Reporting Limit is used and is based on a minimum residue of 1 mg/L. A 500-mL sample has a reporting limit of 2 mg/L. Clean effluents must have sufficient volume filtered to achieve the reporting limit of 2 mg/L, so at least 500 mL of sample must be used.
	2. If you use the following volumes and the resulting TSS value is less than the reporting limits listed, then report <RL on the eDMR, not the actual calculated concentration. The formula used to determine these reporting limits is as follows: 1000 x 1 mg / Volume filtered (mL).

|  |  |
| --- | --- |
| Filtered Volume (mL) | Reporting Limit (mg/L) |
| 1000 | 1.0 |
| 900 | 1.1 |
| 800 | 1.3 |
| 700 | 1.4 |
| 600 | 1.7 |
| 500 | 2.0 |
| 400 | 2.5 |
| 300 | 3.3 |
| 200 | 5.0 |
| 100 | 10 |
| 50 | 20 |

1. QC Data Assessment, Acceptance Criteria, and Corrective Actions and Contingencies for Out-of-Control QC Measures
	1. The Proficiency Testing (PT) sample must be within the criteria of the provider. If the criteria limits are not met, the technician must immediately order another sample to be analyzed.
	2. Qualify all TSS results on the bench sheet and the eDMR if the oven temperature is not within 103 - 105°C (if overnight, then check both when placed in the oven and when taken out).
		1. Samples that fail the Quality Control will have to be qualified back to the last date that the quality control met the above conditions. Include a lab comment on the DMR.
	3. If it is discovered during the drying period or at the end of the drying period that samples are being dried at a temperature below 103°C, the laboratory can adjust the oven temperature until it is back within 103 - 105°C, and let the samples dry again overnight. Document the adjustment (as an example, record an “arrow up”) next to your temperature recordings for the TSS oven. In this case, no data qualification is needed because the minimum drying period at the required temperatures will still be achieved.
	4. If it is discovered during the drying period or at the end of the drying period that samples are being dried at a temperature above 105°C then there is no way to achieve the method requirement. The laboratory can adjust the oven temperature until it is back within 103 - 105°C, and let the sample dry for the remaining required time. However, in this case, all the samples in the oven must be qualified on the eDMR as having been dried at a temperature greater than 105°C for some period. Also, a formal corrective action record needs to be documented because the resulting data needs to be qualified.
	5. For any of the above items, complete the Corrective Actions Log and resolve the problem. Notify the Supervisor.
	6. The residue of the sample must be between 1.0 and 200 mg. If not, the test should be repeated with more or less sample as appropriate.
	7. The balance weights used to calibrate the analytical balance must meet ASTM class 1 or 2 tolerances and be checked every 5 years by an outside source and certified by that source. If no longer within compliance, new weights must be purchased.
2. Pollution Prevention
	1. Consider environmental impact when purchasing materials, handling chemicals, and disposing of wastes.
	2. Prevent pollution at the source whenever possible.
3. Waste Management
	1. All laboratory waste, excess reagents, and samples must be disposed of in a manner that is consistent with applicable rules and regulations.
4. References
	1. Standard Methods for the Examination of Water and Wastewater, Method 2540 D, 1997.
5. Disclaimer
	1. The mentioning of company or product names does not constitute endorsement by the Wisconsin Department of Natural Resources or the authors.