

Total Suspended Solids, SM 2540 D – 2011 (SM 22nd edition)

Initial demonstration of capability

- 2020 B.1 - each analyst must run a known standard concentration at least four times and compare limits listed in the method (under Precision). Table 2020:II lists duplicates and MB for QC only.
- Recommend running replicates and compare results and calculate the standard deviation to compare with that reported in 2540 D.5.
- **Real people language - each operator running this test needs to analyze 4 samples of a TSS Standard**
 - **Keep a copy of the analyst's DOC in his personal folder.**
 - **Documentation (signed form) that the analyst has read and understands all appropriate SOPs and Methods of analyses.**
 - **Recommend backup analyst do this once per year.**

Method Detection Limit

- Annual (every 13 months) verification required using data collected within the past 24 months. Include all data from the On-going Data Collections and the initial MDL determination where appropriate (<24 months old). A minimum of 7 data points are required for *both spiked samples and method blanks*. **However, for TSS only the method blanks are required to determine the MDL. (MDL_b only.)**
- On-going Method Blank Data Collection - population should include all routine method blanks analyzed with each batch during the course of sample analysis
- Refer to the [MDL Examples and EPA Guidance](#) for complete requirements.
- Refer to document titled "Method Update Rule – Method Detection Limit Math 2019" on [Fleming Training Center website](#) for MDL Calculator.

Initial Calibration Verification

- 2020 B.2.a. - check instrument balance daily as stated below.
- 9020 B.4.b. - Service balances annually or more often as conditions change or problems occur...

Check balance routinely, preferably daily before use, with at least two working weights that bracket the normal usage range. (e.g., ANSI/ASTM Class 1 or NIST Class S accompanied by appropriate certificate) for accuracy, precision, and linearity. Record results along with date and technician's initials.

Recertify reference weights as specified in the certificate of calibration or at least every 5 years.

- 2540 B.2. - analytical balance, with a sensitivity of 0.1 mg

Real people language – check balance *daily* with at least 2 working weights that bracket the normal usage range and record results on bench sheet or separate log book.

Method Blank

- 2020 B.2.d. – include at least 1 method blank (MB) daily or with each batch of 20 or fewer samples, whichever is more frequent.
- **Real people language – filter 100 mL of distilled water with each batch of 20 or fewer samples.**

Laboratory Fortified Blank

- 1020 B.6. – A laboratory-fortified blank is a reagent water sample to which a known concentration of the analyte of interest has been added.
 - Sample batch = 5% basis = 1 every 20 samples
 - Use an added concentration of at least 10 times the MDL, less than or equal to the midpoint of the calibration curve.
- 2020 B.2.e. – Using stock solutions, prepare fortified concentrations so they are within the calibration curve.
- **Real people language – analyze TSS Standard sample that can be prepared from recipe below or bought premade.**
 - **Run on a 5% basis, see batch size for more information**

Procedure to Omit Re-drying/Re-cooling/Re-weighing Cycle

How to acquire acceptable results for the total suspended solids comparability data:

- The maximum holding time for a total suspended solids sample prior to analysis is 7 days if stored at temperatures of 6°C and below (not 0°C). (*40CFR part 136, Table II*)
- EPA recommends that 4-7 different samples, in duplicate, be collected and analyzed for this procedure in order to prove that the step for “reheating, recooling, and reweighing” is unnecessary. “Different” could mean samples collected 4-7 consecutive days or 4-7 samples run in one day. These 4-7 samples are dried **overnight** at 103-105°C.
- The next morning, the filters are removed from the oven, allowed to cool in the desiccator and weighed.
- The samples are then returned to the drying oven for one hour, re-cooled and reweighed.
- The resulting data should be examined to determine if the difference between the overnight values and the redried values are less than 4% or 0.5 mg, whichever is less. If so, the redrying step may be omitted for a normal set of samples.
- This procedure excludes atypical samples. (i.e. high fat, oil and grease samples).
- The operator may choose not to perform this study and continue to follow the procedure for redrying/recooling/reweighing cycle as stated the method (SM 2540 D.3.c.).

The study should be re-evaluated at least once per year or whenever a change in sample characteristics occurs and kept on file at the treatment plant.

Duplicate –

- 2540 D.3.c. - Analyze at least 10% of all samples in duplicate.
- Duplicates must agree within 5% of their average weight.
- 2540 A.2. - *“To aid in quality assurance, analyze samples in duplicate. Dry samples to constant weight if possible. This entails multiple drying-cooling-weighing cycles for each determination.”*
- 2020 B.2.f. - states to include at least one duplicate for each matrix type daily or with each batch of 20 or fewer samples.
- 1020 B.8. - states as a minimum to include one duplicate sample with each sample set or on a 5% basis whichever is more frequent.

- **Real people language – analyze 2 samples for TSS.**
 - For example, filter 100 mL of effluent through filter pad A then filter another 100 mL of effluent through filter pad B. Dry, cool and weigh. Figure RPD for both samples.
 - Target value is to get close to the first value and have a small RPD, less than 5%
 - Analyze a duplicate at a 10% rate or with each batch of 10 or fewer samples.

Laboratory Fortified Matrix (LFM)/Laboratory Fortified Matrix Duplicate (LFMD)

- NONE

Control Charts

- NONE

Corrective Action - 1020 B.5., B.8., & B.15.

Batch Size –

- For samples that need to be analyzed on a 10% basis or once for every 10 samples follow these criteria:
 - If a permit stated that 3 analyses per week, we would allow for a duplicate to be analyzed at least once per month.
 - Pick a date and be consistent, the 1st of every month or the 1st Thursday of every month. Mark your calendar!!
 - If a permit stated 5 analyses per week, we would suggest twice a month.
 - Pick a date and be consistent, the 1st and 15th of every month or the 1st and 3rd Thursday of every month. Mark your calendar!!

Calculations –

- % Recovery for LFB
 - = $\frac{\text{LFB concentration}}{\text{Expected concentration}} \times 100\%$
- RPD – relative percent differences for duplicates and LFM/LFMD
 - = $\frac{\text{Difference between sample and duplicate}}{\text{Average of the sample and duplicate}} \times 100\%$

Optional in-house standard recipe:

TSS Standard Samples

To prepare TSS check samples from dry reference material:

6. Dry the reference material* in the desiccator
7. On an analytical balance, weigh 0.1000 gram of the dry powder, put it in a 1000 mL volumetric flask, bring it to the mark with distilled or deionized water and shake well until well suspended.
8. Measure 100 mL and process as usual for environmental samples.
9. A difference of 10 mg should be obtained.
10. Calculation:

$$\frac{(A - B) (1000)}{\text{Vol. used}} = \frac{(10 \text{ mg}) (1000)}{100 \text{ mL}} = 100 \text{ mg/L}$$

*Example of material available from Fisher

- Celite 545 Filter Aid (Powder), Fisher Chemical, 500 gram bottle – Cat#C212-500

