Total Suspended Solids, SM 2540 D, 22nd edition (1997)

Initial Demonstration of Capability (DOC)

- 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 folder for each analyst, keep a copy here**
    - **Documentation (signed form) that analyst has read and understands all appropriate SOPs and Methods.**
    - **Recommend backup analyst do this once a year.**

Method Detection Limit (MDL)

- NONE

Initial Calibration Verification (ICV)

- 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* *(day of)* 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 – on a 5% basis (see batch size for more information) filter 100 mL of distilled water.**
    - **Should be less than 2.5 mg/L.**

Laboratory Fortified Blank (LFB)

- 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

- 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).**

TSS Standard Samples

To prepare TSS check samples from dry reference material:

1. Dry the reference material* in the desiccator
2. 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.
3. Measure 100 mL and process as usual for environmental samples.
4. A difference of 10 mg should be obtained.
5. 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 Fitler Aid (Powder), Fisher Chemical, 500 gram bottle – Cat#C212-500

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, recooled 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

- 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.
- 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.
- 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."*
- 2540 D.3.c. Analyze at least 10% of all samples in duplicate.
- **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 should be close to the first value and have a small RPD (less than 15%)**
- **Analyze a duplicate at a 10% rate (see batch size for more information).**

- **For reporting purposes, average sample and duplicate.**

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

- NONE

Control Charts

- NONE

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

QC Acceptance Criteria

- Blanks < 2.5 mg/L
- LFB ± 15%
- RPD± 15%

Batch Size

- *Influent and Effluent are 2 different samples*
- For samples that need to be analyzed on a 5% basis or once for every 20 samples follow these criteria:
  - If a permit stated that 3 analyses per week, that would be 6 samples per week, we would allow for a blank and LFB to be analyzed at least 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!!
  - If a permit stated 5 analyses per week, that would be 10 samples per week, we would allow once a week.
    - Pick a date and be consistent, every Monday. Mark your calendar!!
- 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, that would be 6 samples per week, we would allow for a duplicate to be analyzed at least 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!!
  - If a permit stated 5 analyses per week, that would be 10 samples per week, we would allow once a week.
    - Pick a date and be consistent, every Monday. Mark your calendar!!

Calculations

- % Recovery for LFB
  - = LFB concentration / Expected concentration X 100%
- RPD – relative percent differences for duplicates and LFM/LFMD
  - = Difference between sample and duplicate / Average of the sample and duplicate X 100%