

DWR-NPDES-SOP-G-10-TSS 2540 D-01012024
Total Suspended Solids (TSS), SM 2540 D-2015

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1) EFFECTIVE DATE: 01/01/2024

2) SIGNATURES:

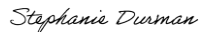


April Grippo (May 30, 2024 16:25 CDT)

April Grippo, DWR Interim Director



Brianne Begley, Environmental Consultant
Drafter / Preparer



Stephanie Durman, Deputy General Counsel
Reviewer

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- 2020 B.1. - each analyst must run a known standard concentration at least four times and compare limits listed in the method (under Precision)
- Recommend running replicates and compare results and calculate the standard deviation to compare with that reported in 2540 D.5
- **Summary: each operator running this test needs to analyze four samples of a TSS Standard**
 - **Keep a copy of each analyst's DOC**
 - **Keep documentation (signed form) that the analyst has read and understands all appropriate SOPs and Methods of analyses**
 - **A backup analyst should conduct a DOC once per year**

Method Detection Limit (MDL)

- 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 (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, 2, or 3 or NIST Class S (redefined as ASTM Class 1 weights and no longer available), accompanied by appropriate certificate] for accuracy, precision, and linearity. Record results along with date and technician's initials
 - Recertify reference [working] weights as specified in the certificate of calibration or at least every 5 years
- 2540 B.2. - analytical balance, capable of weighing to 0.1 mg
- **Summary: check balance *daily* with at least two working weights that bracket the normal usage range and record results on bench sheet or separate log book**

Method Blank (MB)

- 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

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- 2540 A.5. – Analyze one method blank (MB) per batch of 20 samples for each method except settleable solids (2540F). Blank analysis includes all container- and filter-preparation steps and procedures except sample addition
- **Summary: filter 100 mL of distilled water with each batch of 20 or fewer samples**

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 = 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
- 2540 A.5. – Include one laboratory-fortified blank (LFB) per batch of 20 samples. Plot the percent recoveries on a control chart for laboratory evaluation. Laboratories may purchase known standards or prepare in-house working controls for use
- **Summary: 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**
 - **Plot percent recoveries on a control chart**

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

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

- The maximum holding time for a total dissolved solids sample prior to analysis is 7 days if stored at temperatures of 6°C and below (not 0°C). (40 C.F.R. part 136, Table II)
- EPA recommends that 4-7 different samples, in duplicate, be collected and analyzed for this procedure 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 $180 \pm 2^\circ\text{C}$.
- The next morning, the evaporating dishes 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 0.5 mg. 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 C.3.d.).

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 A.5. - Analyze $\geq 5\%$ of all samples in duplicate or at least one duplicate sample with each batch of ≤ 20 samples
- Typically, the relative percent difference (RPD) of duplicates should not exceed 10%, but RPDs may vary considerably due to sample matrix and concentration
- 2540 A.2. & D.3.c. - Dry samples to constant weight if possible; this entails multiple cycles of drying, cooling, and weighing for each sample
- 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
- **Summary: analyze two 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 10%**

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- Analyze a duplicate at a 5% rate or with each batch of 20 or fewer samples
- A precision control chart is required for duplicates (see control chart section below for more information)

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

- NONE

Control Charts

- 2540 A.5. – Include one laboratory-fortified blank (LFB) per batch of 20 samples. Plot the percent recoveries on a control chart for laboratory evaluation
- 1020 B.13.a. – The accuracy chart for QC samples... is constructed from the average and standard deviation of a specified number of measurements of the analyte of interest. The accuracy chart includes upper and lower warning levels (WLs) and upper and lower control levels (CLs). Common practice is to use $\pm 2s$ and $\pm 3s$ limits for the WL and CL, respectively, where s represents standard deviation
- 1020 B.13.b. – The precision chart is also constructed from the average and standard deviation of a specified number of measurements [e.g., %RSD or relative percent difference (RPD)] for replicate or duplicate analyses of the analyte of interest. Perfect agreement between replicates or duplicates results in a difference of zero when the values are subtracted, so the baseline of the chart is zero. Therefore for precision charts, only upper WLs and upper CLs are meaningful
- **Summary: Create and maintain control charts once you have 20-30 data points**

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

Batch Size

- 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, 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 =

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$$\left(\frac{\text{LFB concentration}}{\text{expected concentration}} \right) \times 100$$

- RPD – relative percent differences for duplicates and LFM/LFMD =

$$\left(\frac{|\text{sample result} - \text{duplicate result}|}{(\text{sample result} + \text{duplicate result})/2} \right) \times 100$$

Optional in-house standard recipe (Also see SM 2540 D.1.a.):

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

Revision Number	Date	Brief Summary of Change
0	December 2013	Initial issuance of the Guidance
1	February 26, 2018	Method editorial revision date changed from 1997 to 2011. MDL procedure revised to incorporate Revision 2.
3	July 2019	Added links to additional guidance documents pertaining to MDL.
4	December 15, 2021	Method editorial revision date changed from 2011 to 2015, duplicate information added from SM 2540 (2015), added

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		information on control charts.
5	December 11, 2023	Changes to summary information, grammar, and effective date.