TOTAL SUSPENDED SOLIDS

TABLE OF CONTENTS:

Page

1. SCOPE AND APPLICATION 2
2. SUMMARY OF THE METHOD 2
3. EQUIPMENT AND SUPPLIES 2
4. REAGENTS AND STANDARDS 3
5. SAMPLE HANDLING AND PRESERVATION 5
6. SAMPLE ANALYSIS/QUANTITATION 5
7. DATA ARCHIVAL/VALIDATION 8
8. QUALITY CONTROL/DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES 9
9. SAFETY/HAZARDOUS WASTE MANAGEMENT 10
10. REFERENCES/DEFINITIONS 10

APPENDIX OF SIGNIFICANT CHANGES 11

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1. SCOPE AND APPLICATION

1.1. Total suspended solids (TSS) is defined as the portion of solids that is retained on a filter of 2.0 microns nominal pore size. This method is applicable to drinking, surface, and saline waters. The practical range of determination is from the method detection limit (MDL) 2 mg/L (3 mg/L for samples with conductivity ≥ 15,000 µmhos/cm) to 20,000 mg/L. The practical quantitation limit (PQL) is determined by the concentration at which 2.5 mg of residue as required by the method remains on the filter paper. For a sample volume of 250 mL, this is 10 mg/L.

1.2. The test covered by this SOP is W-TSS.

2. SUMMARY OF THE METHOD

2.1. This method is based on Standard Methods 2540 D-2015.

2.2. A well-mixed sample is filtered through a pre-weighed glass fiber filter. The filter and any residue are then dried to a constant weight at 103-105 °C. The filter is cooled in a desiccator, weighed and the result used to compute the TSS of the sample.

Note: The filtrate from this analysis may be used for a total dissolved solids (TDS) determination if the sample requires both tests. See DEP SOP NU-047 for details.

2.3. Interferences – samples high in filterable residue (dissolved solids), such as saline waters, brines and some wastes, may be subject to a positive interference. See section 6.3. for the procedure to analyze such samples in order to minimize this interference.

3. EQUIPMENT AND SUPPLIES

3.1. Instrumentation

Component	Manufacturer	Model
Analytical Balance	Mettler Toledo	XP204
Analytical Balance	Mettler Toledo	XPE204

3.2. Equipment

Equipment	Manufacturer/Vendor[1]	Model/Catalog Number
Oven	Fisher Scientific	IsoTemp Oven 13-247-650G
Oven	Thermo Scientific	Lindberg/Blue M LB305760M, and Heratherm Oven OMH180
Digital Thermometer	Fisher Scientific Traceable	Platinum High Accuracy, and 15-081-108
Desiccator	Fisher Scientific	
Assorted Graduated Cylinders, Class A	Fisher Scientific (SR)	08-564A, 08-548D, 08-553A, 08-548B
Volumetric Flasks (1 L and 2 L)	Fisher Scientific (SR)	10-212-5L, 10-212-5M
Dish Tongs		
1 L Filter Flask	Fisher Scientific (SR)	10-180F
Magnetic filter holder and funnel, 300 mL	Fisher Scientific	NC-0006472

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Vacuum Pump	Welch	25468
Forceps		
Handheld Conductivity Meter	Fisherbrand	15-077-977
Evaporation Dishes	CoorsTek(SR)	08-690L, 60200
Stir Bar	Fisher Scientific (SR)	14-511-65
Spatula	Fisher Scientific	14-357Q
Stirrer	Corning Stirrer	Scholar 171
Or Equivalent [1]**Note:** (SR) designates equipment is typically ordered through the in-house stockroom		

3.3. Supplies/Consumables

Supplies/Consumables	Vendor[1]	Catalog Number
Filter, Glass Fiber, Proweigh	Fisher Scientific (SR)	09-735-56
Beaker, Polystyrene, 50 mL	Fisher Scientific (SR)	08-732-122
Pipette, Transfer, Polyethylene, 5 mL	Fisher Scientific (SR)	13-711-5AM
Or Equivalent [1]**Note:** (SR) designates supply is typically ordered through the in-house stockroom		

4. REAGENTS AND STANDARDS (SEE SOPS NU-041 and NU-042)

Note: Before preparing reagents or standards, or running samples, please read the safety notes in the Safety/Hazardous Waste Management section of this SOP and the MSDS for the chemicals used in this analysis.

Note: Standard preparations must be documented in the Laboratory Information Management System (LIMS) 'Standard Preparation Tracker' module. Unique ID numbers for each standard are generated in the module and documented in the TDS excel worksheet submitted with each analysis folder. A Certificate of Analysis for each purchased standard is maintained in CertificatesofAnalysis folder for TDS, TSS, and TS on the Nutrients drive at \\floridadep\data\DEAR\Labs\Lab3\Chem_Data\Inorganic\NutrientsRunData\MetaData\Solids\. All containers holding standards must be labeled with their unique ID and expiration date.

4.1. Chemicals

	Chemical	Alias	Manufacturer[1]	Catalog Number	Purity	Expiration	Storage
4.1.1.	Deionized (DI) Water	H_2O	In house	N/A	N/A	Daily	N/A
4.1.2.	Drierite, 10-20 Mesh, White	$CaSO_4$, Desiccant	W. A. Hammond DRIERITE Co. LTD (SR)	14005/07-577-4B	>99.5% $CaSO_4$	N/A	Room Temp
4.1.3.	Drierite, 8 Mesh, Blue	$CaSO_4$ and $CoCl_2$, Desiccant	W. A. Hammond DRIERITE Co. LTD (SR)	23005/S692-212	≥98% $CaSO_4$, <2% $CoCl_2$	N/A	Room Temp
4.1.4.	WatR™ Pollution Solids Concentrate	ALTQC, ALTQC_SLT	Environmental Resource Associates	4032	See CoA	See CoA	Room Temp
4.1.5.	Sea Water, Synthetic	MBLK_SLT, Salt Blank	Ricca Chemical Company (SR)	8363-5	N/A	12 months	Room Temp
4.1.6.	Sodium Chloride	NaCl	Fisher Chemical	S271-500	≥99% NaCl	See CoA	Room Temp

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4.1.7.	Cellulose Type 20	Cellulose	Sigma-Aldrich Corp.	S3504	N/A	See CoA	Room Temp
Or Equivalent							
[1]**Note:** (SR) designates chemical is typically ordered through the in-house stockroom							

4.2. Desiccant –

4.2.1. A mixture of approximately 10 % blue desiccant (4.1.3.) and 90 % white desiccant (4.1.2.) should be placed in the desiccator(s) used to store filters and dishes.

4.2.2. The blue desiccant changes color to indicate water absorption. Once the desiccant has turned from blue to pink, heat the desiccant in a 180 °C oven for one hour, let cool and reuse. Desiccant may be regenerated 3 times before replacing with new desiccant. The old desiccant can be discarded in the trash.

4.3. Salt blank (MBLK_SLT) –

4.3.1. Ricca Synthetic Sea Water (4.1.5.) is used as a salt blank for high conductivity samples.

See certificate of analysis for expiration date.

4.3.2. If needed, a salt blank may be prepared using the following procedure:

Sodium Chloride (4.1.6.)	30 g
DI Water (4.1.1.)	to 2000 mL

In a 2L flask containing approx. 1000 mL of DI Water, dissolve 30 g of sodium chloride. Bring to volume and invert to mix.

Holding time, 30 days.

4.4. ALTQC –

WatR Pollution Solids Concentrate (4.1.4.)	See ERA's Instructions
DI Water (4.1.1.)	See ERA's Instructions

The ALTQC is prepared using a purchased certified quality control sample. See the certificate of analysis for the preparation instructions, concentration, and expiration date of the purchased standard.

Holding time, 180 days.

4.5. ALTQC_SLT –

WatR Pollution Solids Concentrate (4.1.4.)	See ERA's Instructions
Salt Blank (4.3.)	See ERA's Instructions

The LFB_SLT is prepared using a purchased certified quality control sample. Since the LFB_SLT is used for high conductivity samples, the salt blank is substituted for DI water. See the certificate of analysis for the preparation instructions, concentration, and expiration date of the purchased standard.

Holding time, 180 days.

4.6. LFB (50 mg/L TSS) –

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Cellulose (4.1.7.)	0.100 g
DI Water (4.1.1.)	to 2000 mL

To a 2 L flask containing approx. 1000 mL DI water, add 0.100 g of the cellulose. If making a combined TDS/TSS LFB, add 0.500 g NaCl (4.1.6., dried for at least 1 hour at 105 °C) before adding the cellulose. Promptly mix thoroughly using a magnetic stir plate (for approx. 15 minutes), as the cellulose can clump. Bring to volume and invert several times.

Holding time, 30 days.

4.7. LFB_SLT (50 mg/L TSS) –

Cellulose (4.1.7)	0.100 g
Salt Blank (4.3.)	to 2000 mL

To a 2 L flask containing approx. 1000 mL Salt Blank, add 0.100 g of the cellulose. Promptly mix thoroughly using a magnetic stir plate (for approx. 15 minutes). Bring to volume and invert several times.

Holding time, 30 days.

5. SAMPLE HANDLING AND PRESERVATION

5.1. Samples are stored on ice or in a refrigerator at ≤ 6 °C (not frozen) in order to minimize microbiological decomposition of solids.

5.2. Non-representative particulates such as leaves, sticks, fish and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result.

5.3. Samples are stored in the Nutrient refrigerator in the receiving room of the DEP laboratory. Samples must be brought to room temperature before processing.

5.4. Samples should be analyzed within seven days of collection. Samples analyzed after this holding time must be reported with a test comment describing the nature of the delay as well as the appropriate qualifying code, Q.

6. SAMPLE ANALYSIS/QUANTITATION (SEE SOP NU-033)

Note: Be sure to record all of the necessary information neatly and correctly on the TSS Excel worksheet (NU-TSS). Make sure to always use the latest version of the NU-TSS form The worksheet is located at \\floridadep\data\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\RunData\MetaData\Solids\ .

The TSS results are automatically calculated on the worksheet.

Important: The worksheet contains functional formatting to indicate unacceptable results – do not copy/paste or "click and drag" any information in the worksheet.

Note: Do not handle the filters with your hands. Always use forceps when handling the filters.

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Note: All ovens are kept at a constant temperature. The minimum and maximum (MIN/MAX) oven temperatures are recorded for each drying cycle of the analysis on the Oven Temperature Log Sheet (NU-OVENTEMPLOG). The log sheet is located at \\floridadep\data\\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\RunData\MetaData\Solids\.

To use the MIN/MAX function on the digital thermometer, see the manufacturer directions located on the door of each oven. Record the START time of the drying cycle as the time when the oven has reached its set temperature, and the MIN/MAX function on the thermometer has been activated. After the MIN/MAX settings have been checked and recorded to verify that the oven was in compliance, record the STOP time for the drying cycle as the time when samples are removed from the oven. If an oven temperature is found to be out of compliance, an explanation and corrective action is recorded on the log sheet, and another drying cycle is monitored. The oven temperature log sheet is submitted with each analytical folder. Make sure that whenever samples are in the oven at least one temperature reading is recorded every day or the Min/Max temperature is recorded over an extended period of time. If samples are in the oven over the weekend the MIN/MAX temperature is recorded as well.

6.1. ProWeigh filters are precleaned, preweighed and certified by the manufacturer. Each run, one ProWeigh filter must be placed in the oven with the samples, then desiccated and weighed and the result compared with the manufacturer's certified weight. Record these weights on page 2 of the TSS worksheet.

An example run sequence includes:

- ALTQC (4.4.) or ALTQC_SLT (4.5.)
- MBLK (DI Water, 4.1.1.) or MBLK_SLT (salt blank, 4.3.)
- LFB (4.6.) or LFB_SLT (4.7.)
- Batched samples, with one sample duplicate per batch of up to 20 samples
- MBLK or MBLK_SLT

6.2. Procedure for samples with conductivity < 15,000 µmhos/cm

Note: If the field conductivity for the sample is not available, the lab conductivity must be determined (hand-held conductivity meter or auto-titrator instrumentation can be used) and recorded on the TSS worksheet.

6.2.1. When ready to filter, assemble the filtering apparatus. Connect the vacuum hose to the filter flask.

6.2.2. Remove the ProWeigh filter from the aluminum dish, place it onto the filter apparatus and attach the membrane filter funnel over the filter. Thoroughly wet the filter with DI water and apply vacuum. When all water has passed through the filter, turn off the vacuum, remove the filter flask, and pour out the rinse water, re-attach the flask, and re-apply vacuum.

6.2.3. While the vacuum is still on, rapidly invert the sample and transfer 250 mL to the filtering device using a clean 250 mL graduated cylinder. Rinse the cylinder with enough DI water to remove any remains of the sample.

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Note: If TDS is also being determined, be sure to remove the filtrate and obtain the TDS aliquot before rinsing the cylinder. This will ensure the filtrate used for TDS is not diluted.

Note: If it is not possible to filter 250 ml of sample due to particulate matter, determine the sample volume you are able to filter and start over at step 6.2.1 through 6.2.3.

6.2.4. Once all the liquid has passed through the filter, rinse the filter with DI water (Approx. 3 x 30 mL) to remove any sample that remains on the filter and on the sides of the filtering apparatus. Allow the filter to completely dry in between washings.

6.2.5. Continue to apply the vacuum until no more liquid drips into the flask. This may take up to three minutes depending on the matrix of the sample.

6.3. Procedure for samples with conductivity > 15,000 µmhos/cm (batched separately)

6.3.1. When ready to filter, assemble the filtering apparatus. Connect the vacuum hose to the filter flask.

6.3.2. Remove the ProWeigh filter from the aluminum dish and place it onto the filter apparatus. Thoroughly wet the filter with DI water, apply vacuum, and attach the membrane filter funnel over the filter. When all water has passed through the filter, turn off the vacuum, remove the filter flask, pour out the rinse water, re-attach the flask, and re-apply vacuum.

6.3.3. While the vacuum is still on, rapidly invert the sample and transfer 250 mL to the filtering device using a clean 250 mL graduated cylinder. Rinse the cylinder with enough DI water to remove any remains of the sample.

6.3.4. When all the water has been drawn through the filter, turn off vacuum and rinse with approx. 30 mL DI water. Reapply vacuum, drawing DI water through the filter until dry.

6.3.5. Repeat step 6.3.4.

6.3.6. Rinse the filtration tower once more with minimal volume and flow of DI water to remove any sample that remains on the sides of the tower.

6.3.7. While the vacuum is still on, remove the filter tower and gently rinse the portion of the filter that was under the filter tower with DI water one time. Use minimal flow and direct the water toward the center of the filter to avoid washing any particulate material from the filter.

6.3.8. Continue to apply the vacuum after the sample has completely passed through and the filter has been rinsed until all traces of water have been removed.

6.4. Carefully remove the filter with forceps, return to the aluminum dish, and place in an oven set at 103-105 °C. Wash the graduated cylinder, the filter holder, membrane filter funnel, and the filter flask with DI water before filtering the next sample to avoid sample carryover. The sample filters should remain in the oven for at least 1 hour, but usually remains overnight to ensure thorough drying. See

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the **Note** above regarding documentation of drying cycles on the Oven Temperature Log Sheet.

6.5. Remove the dried filters and place in a desiccator to cool for at least 30 minutes. Check the calibration of the balance using certified weights and document the results in the balance maintenance logbook. Weigh the filters and record their weights accurately and legibly on the TSS worksheet under Filter and Residue Weight (g): 1st. Do not leave the desiccator door open too long. If the samples are exposed to the air for a long period of time, the sample results may be affected.

6.6. Repeat drying cycle in the 103-105 °C oven for at least one more hour, then cool in a desiccator and weigh again following the same procedures as described in step 6.5. Make sure to record the weights on the TSS worksheet under Filter and Residue Weight (g): 2nd.

6.7. If the weight change is less than 0.5 mg then the analysis is complete. If this is not the case, repeat step 6.6. Record the weights on the TSS worksheet under Filter and Residue Weight (g): 3rd. If after three weighing cycles the weight change between the last two readings is still greater than 0.5 mg, consult a supervisor.

6.8. The TSS is calculated using the following equation,

$$\text{TSS (mg/L)} = \frac{\{[\text{final dried wt. of residue + filter (g)}] - [\text{wt. of filter (g)}]\} \times 1000000}{\text{Volume of sample (mL)}}$$

6.9. Save the TSS Excel file as a text file (.txt) and use the GenChem: TSS-TDS Balance-XPE2042 driver or the GenChem: TSS-TDS Balance-XP204 driver to convert the text file into QC Manager. Check that the number of significant figures has been set correctly.

6.10. Place the completed TSS worksheets along with the standard sheet in a folder labeled YYMMDD**XE**S (for the Mettler Toledo XPE204-2 balance) or YYMMDD**X**S (for the Mettler Toledo XP204 balance), where YY is the year, MM is the month, DD is the day of the analyses, XE or X identifies the balance, and S indicates a TSS analysis. Initial the folder and hand to a supervisor for review.

7. DATA ARCHIVAL/VALIDATION

7.1. The raw data is archived in paper format. A report folder is created for each analytical run. Occasionally more than one run may be in one folder. The folder is labeled with the test (W-TSS), filename(s), and initials of the analyst.

7.2. Each report folder should contain the following items - the TSS excel worksheet, bench sheet(s), the oven temperature log sheet, the backlog containing the batching documentation, and a copy of the data and QC assessments uploaded from QC Manager to LIMS at the time of test authorization.

7.3. The report folders are on file with the nutrients and general chemistry supervisory personnel. Periodically, the files are moved to the State Records Center for long time storage according to DEP SOP LB-006.

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7.4. Excel transfer files for upload to QC manager are stored in electronic format in the current year's subfolder of the appropriate instrument located at \\floridadep\data\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\RawData\Solids\.

7.5. Method validation data is on file with the supervisor. Digital copy is also stored on the network, under an appropriate sub-folder in \\floridadep\data\DEAR\Labs\Lab5\Chemistry\Inorganic\Nutrient\Validations.

8. QUALITY CONTROL/DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES (SEE DEP SOPS NU-038 AND NU-043)

8.1. An MBLK (analyzed at the beginning of every batch of twenty samples or less) and final blank are analyzed with samples having a conductivity below 15,000 µmhos/cm and must be < 2.0 and > -2.0 mg/L. If the result is outside of this range, the blank and all associated samples may be rerun until the blank passes or the results may be qualified with an appropriate code and comment (refer to DEP SOP NU-043, Section 4.2.1.).

8.2. One set of sample duplicates is analyzed per batch of twenty samples or less. If the sample result is above the PQL (10 mg/L), the relative percent difference (RPD) must be less than 10% to report the result without qualification. If the replicate sample result is below the PQL, no precision data is uploaded and an appropriate test comment is added to all samples in the batch.

8.3. An ALTQC (4.4.) or ALTQC_SLT (4.5.) is run at the beginning of every analytical run. An ALTQC is run when the sample conductivities are below 15,000 µmhos/cm, whereas an ALTQC_SLT is run when the sample conductivities are above 15,000 µmhos/cm. ALTQC and ALTQC_SLT recovery must be within the manufacturer's certified limit but not exceed 85 to 115%.

8.4. An LFB (4.6.) or LFB_SLT (4.7.) is run at the beginning of every batch of twenty samples or less. An LFB is run when the sample conductivities are below 15,000 µmhos/cm, whereas an LFB_SLT is run when the sample conductivities are above 15,000 µmhos/cm. The acceptance limits for the LFB and LFB_SLT recoveries are 85 to 115%.

8.5. An MBLK_SLT (4.3.) (analyzed at the beginning of every salt batch of twenty samples or less) and final salt blank are run with samples having a conductivity above 15,000 µmhos/cm and must be < 3.0 and > -3.0 mg/L. If the result is outside of this range, the blank and all associated salt samples may be rerun until the blank passes or the results may be qualified with an appropriate code and comment (refer to DEP SOP NU-043, Section 4.2.1.).

8.6. One ProWeigh filter for every run must be dried, desiccated, and weighed. Record this weight and the certified weight of the filter on the TSS worksheet. The determined weight should be ± 0.4 mg of the certified weight. If the weight is outside this range, put the filter back in the desiccator and leave it for at least 30 mins. Reweigh. If the weight is still outside the acceptable range, inform a supervisor.

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8.7. Sample results obtained based on a residue exceeding 200 mg on the filter must be confirmed using a smaller sample volume or qualified appropriately. It is recommended to filter an aliquot for confirmation as soon as the first weight indicates that the residue may exceed the limit.

8.8. For general method performance and acceptance criteria for QC measures and corrective action for out-of-control data and contingencies for handling out-of-control or unacceptable data, refer to DEP SOP NU-038 and DEP SOP NU-043.

8.9. For general calculations, including RPD, see Section 5.9 of the FDEP Quality Manual.

9. SAFETY/HAZARDOUS WASTE MANAGEMENT

9.1. Safety glasses, gloves and lab coats should be worn at all times whilst handling samples. Excess samples may be flushed down the sink.

9.2. Review the Laboratory Health and Safety Plan (accessible at https://floridadep.sharepoint.com/dear/SitePages/Lab Health and Safety.aspx).

10. REFERENCES/DEFINITIONS

For definitions see section 3 of the FDEP Quality Manual. All abbreviations used throughout the document are defined at least once in the document usually at the first time of use.

10.1. Standard Methods Online, 2540 D-2015.

10.2. DEP SOP NU-033, Summary of Sample Analysis Procedures for the General Chemistry and Nutrients Workgroups.

10.3. DEP SOP NU-038, Nutrients and General Chemistry: Quality Assurance and Quality Control.

10.4. DEP SOP NU-041, Reagent Preparation Procedure.

10.5. DEP SOP NU-042, Standard Preparation Procedure.

10.6. DEP SOP NU-043, Nutrients and General Chemistry Quality Assurance and Data Authorization Guidelines.

10.7. DEP SOP NU-047, Total Dissolved Solids.

10.8. Oven manuals.

10.9. Quality Manual, Bureau of Laboratories, FDEP.

10.10. Laboratory Health and Safety Plan.

10.11. DEP SOP LB-006, Standard operating procedure for records maintenance and storage.

10.12. DEP SOP CM-011, Gravimetric Analysis: Analytical Weight and Balance Calibration.

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Appendix of Significant Changes

03/07/2008	Batches changed from 20 samples to ten. Acceptance limits for duplicates changed from 20% to 10%.
06/03/2008	Reference method changed from 160.2 to SM 2540 D.
05/19/2011	The Method Detection Limit (MDL) and Practical Quantitation Limit (PQL) were changed to 2 and 10, respectively. See Section 1 for details.
05/08/2012	Section 6 was edited to include the new analysis procedures for samples below and above 15000 umhos/cm put into practice on 05/21/2012. The MDL for samples above 15000 µmhos/cm was also added.
08/23/2012	MDL for samples with conductivity ≥ 15,000 µmhos/cm was set to 3.0 mg/L.
05/08/2013	Section 4.3 was edited to correct the holding time of the ALTQC. Section 6.2.3 was further clarified. Section 7.2 was edited to correct the RPD control limit.
04/16/2015	Data archival information was clarified in section 7. Oven temperature recording procedure was modified in section 6.
11/29/2018	Appendix A and B were removed. The relevant forms are referenced in section 6. Reference method version was updated to the 2011 revision.
11/23/2021	Added tables for instrumentation, equipment, supplies/consumables, and chemicals.
10/03/2022	Updated reference method from SM 2540 D 2011 to SM 2540 D 2015. Changed batch size from ten to 20 samples. Replaced ALTQC and ALTQC_SLT with LFB/ALTQC and LFB/ALTQC_SLT and added LFB/ALTQC acceptance limits. Added holding time to prepared salt blank.
10/03/2023	In section four, the LFB and LFB_SLT preparations were changed (Cellulose Type 20). Updated data drive file path links.

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