

SOP-C-109

**Determination of Total Dissolved
Solids (Filterable Residue)**

Revision 13

Working Copy

Approval:

Laboratory Manager

Date

3-14-22

Concurrence

Date

03-14-2022

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Initials: _____

SOP-C-109
Determination of Filterable Residue

- i. Identification of the method**
 - a. Standard Methods 2540C, approved 2017.
- ii. Applicable matrix or matrices**
 - a. Water
- iii. Limits of detection and quantitation**
 - a. LOQ of 10 mg/L up to 20,000 mg/L. Samples may be concentrated or reduced in volume to extend the ranges.
- iv. Scope and application, including parameters to be analyzed**
 - a. The filtrate from the total suspended solids determination may be used for determination of total dissolved solids.
- v. Summary of the method**
 - a. Gravimetric analysis of dissolved matter from filtered water samples: A well-mixed sample is filtered through a standard glass fiber filter, and the filtrate is evaporated to dryness in a weighed dish and dried to constant weight at 180°C.
 - b. This SOP may be used in conjunction with SOP-C-107, "Determination of Total Suspended Solids," SOP-C-108, "Determination of Nonfilterable Volatile and Fixed Solids," and SOP-C-130, "Determination of Total and Percent Solids," to determine other types of solids. This method has been enhanced by TIAER to include a check standard.
- vi. Definitions**
 - a. None; refer to QAM-Q-101, "Laboratory Quality Control," for standard QC definitions
- vii. Interferences**
 - a. Results for residues high in oil or grease may be questionable because of the difficulty of drying to constant weight in a reasonable time.
 - b. Highly mineralized waters with a considerable calcium, magnesium, chloride, and/or sulfate content may be hygroscopic and require prolonged drying, proper desiccation, and rapid weighing.
 - c. Samples high in bicarbonate require careful and possibly prolonged drying at 180°C to insure complete conversion of bicarbonate to carbonate. Because excessive residue in the dish may form a water-trapping crust, limit sample to no more than 200 mg residue.
- viii. Safety**
 - a. Routine laboratory safety precautions
 - b. Use the oven mitts to remove the sample trays from the oven.

SOP-C-109
Determination of Filterable Residue

ix. Equipment and supplies

- a. Glass fiber filter discs without organic binder (Millipore AP40, Reeves Angel 934-AH, Gelman type A/E or equivalent)
- b. Filtering apparatus with 40-60 micron fritted disc filter support
- c. Side-arm suction flask with vacuum attachment
- d. Class A graduated cylinder calibrated "to contain"
- e. Drying oven set at $180 \pm 2^{\circ}\text{C}$ (lower for initial evaporation)
- f. Desiccator
- g. Analytical balance capable of weighing increments of 0.1mg.
- h. ISCO™ plastic sample bottles, or equivalent (1liter) with cap
- i. Sample tray
- j. Aluminum weighing pans (also see SOP-C-107, "Determination of Total Suspended Solids")
- k. Forceps and tongs
- l. Evaporating dishes/crucibles
- m. Wide bore pipettes, beakers, stirrers, general lab equipment

x. Reagents and standards

- a. Sodium Chloride, 100 mg/L, LCS/LCSD. Weigh 0.1000 g of NaCl (dried at 180°C and cooled in a desiccator) and dissolve into DI water in a 1L volumetric flask. Shelf life is 7 days.

xi. Sample collection, preservation, shipment and storage

- a. Holding Time: 7 days, begin analysis as soon as possible
- b. Preservation: Refrigerate sample to $>0\text{-}\leq 6^{\circ}\text{C}$ until analysis
- c. Refer to field procedures for sampling

xii. Quality control

- a. Refer to QAM-Q-101, "Laboratory Quality Control."
- b. For every 10 environmental samples, analyze at least one sample duplicate. Some projects may also require a field split, which will not count as one of the group of 10 samples.
- c. Method blanks and LCS/LCSD pair (with no sample duplicate) are analyzed for every set of 20 samples or less.
- d. No CCV, CCB, AWRL or LOQ standard is required for this procedure.
- e. The analytical balance should be calibrated with the 0.1 g class 1 weight before the initial and final weights of the filters are taken. Calibrate in accordance with QAM-I-101, "Operation and Calibration of the Analytical Balance".

SOP-C-109
Determination of Filterable Residue

xiii. Calibration and standardization

a. None

xiv. Procedure

- a. Preparation of glass-fiber filter disk: If pre-prepared glass fiber filter disks are used, eliminate this step. Insert disk with wrinkled side up into filtration apparatus. Apply vacuum and wash disk with three successive 20-mL volumes of DI water. Continue suction to remove all traces of water. Discard washings. Dry filters at 103-105°C for at least one hour. Store in the desiccator.
- b. Preparation of evaporating dish: If volatile solids are to be measured, ignite cleaned evaporating dish at 540-555°C for at least 1 hour in a muffle furnace. If only total dissolved solids are to be measured, heat clean dish to $180 \pm 2^\circ\text{C}$ for at least 1 hour in an oven. Store in desiccator until needed. Weigh immediately before use.
- c. Selection of filter and sample sizes: Choose sample volume to yield between 2.5 and 200 mg dried residue. If more than 10 min are required to complete filtration, increase filter size or decrease sample volume. A 100-ml volume of sample is typically used for a filter with the diameter of 4.7 cm. More sample will give a more accurate representation of the sample if duplicates or other analyses permit. For cleaner samples, 1000-ml may be more appropriate.
- d. Increase the sample volume if the amount of residue that remains in the evaporating dish/crucible is less than 10.0 mg. This is accomplished by performing the analysis again after an initial determination of insufficient residue.
- e. Do not use more than 1 L of sample, unless the sample is DI or ultrapure water for quality control monitoring within the laboratory.
- f. Bring samples to room temperature before analysis. Save approximately 100-ml of sample for analysis when preparing aliquots. This may be adjusted due to volumes required for other analyses or for the presence of more suspended matter that precludes larger volumes passing through the filter. Moisten and seat the filter with a small amount of DI prior to introducing sample.
- g. Sample analysis: Shake the sample container at least 25 times and measure sample into a Class A graduated cylinder (calibrated "to contain"), then pour onto a glass-fiber filter. Alternatively for higher level samples, stir sample with a magnetic stirrer and pipette a measured volume onto the filter with applied vacuum.

SOP-C-109

Determination of Filterable Residue

While stirring, pipette from a point mid-depth and midway between the container wall and vortex. Wash container, pipette and/or apparatus with at least three successive 10-mL volumes of reagent-grade water, allowing complete drainage between washings, and continue suction for about 3 min after filtration is complete. Transfer total filtrate (with washings) to a weighed evaporating dish and evaporate to dryness in a drying oven at 103-105°C. If necessary, add successive portions to the same dish after evaporation.

- h. Dry evaporated sample for at least 1 hour more in an oven at 180 ± 2°C, cool in a desiccator to balance temperature, and weigh. Repeat drying cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained or until weight change is less than 4% of previous weight or 0.5 mg, whichever is less. Analyze at least 10% of all samples in duplicate. If volatile solids are to be determined, follow procedure in SOP-C-108, "Determination of Nonfilterable Volatile and Fixed Solids."

xv. **Data analysis and calculations;**

- a. Approved spreadsheets or Elog on laboratory computer to calculate TDS and QC may be used or required.
- b. Weigh the evaporating dish/crucible to the nearest 0.1 mg and record the weight in the personal logbook or Elog.
- c. Filterable residue mg/L = $\{(A-B)*1000\}/C$
A = weight of evaporating dish/crucible + residue in mg
B = weight of evaporating dish/crucible
C = ml of sample

xvi. **Method performance**

- a. Duplicate determinations should agree within 5% of their average weight.
- b. Analyze the LCS/LCSD standards in the same manner as any sample.

xvii. **Pollution prevention**

- a. Not applicable

xviii. **Data assessment and acceptance criteria for quality control measures**

- a. Refer to QAM-Q-101, "Laboratory Quality Control."
- b. If any QC samples do not pass the acceptance criteria as described in QAM-Q-101, "Laboratory Quality Control," reanalyze immediately if possible and complete a Corrective Action Report in accordance with QAM-Q-105, "Corrective Actions".

SOP-C-109

Determination of Filterable Residue

- c. Duplicate acceptance limits are $\pm 10\%$ RPD for sample duplicates, $\pm 20\%$ RPD for LCS/LCSD and $\pm 30\%$ RPD for field splits.

xix. Corrective actions for out-of-control data

- a. Refer to QAM-Q-105, "Corrective Actions."

xx. Contingencies for handling out-of-control or unacceptable data

- a. Refer to QAM-Q-105, "Corrective Actions," and QAM-Q-101, Laboratory Quality Control."

xxi. Waste management

- a. Not applicable; for general waste management and pollution prevention, refer to QAM-W-101, "Disposal of Laboratory Waste".

xxii. References

- a. Standard Methods for the Examination of Water and Wastewater, latest online edition approved 2017, ed. by Arnold E. Greenberg, et al., APHA, AWWA, Washington D.C., Method 2540 C.
- b. National Environmental Laboratory Accreditation Conference (The NELAC Institute) standard, 2016.
- c. Code of Federal Regulations, 40 CFR 136, July 2012

xxiii. Any tables, diagrams, flowcharts and validation data

- a. Example TDS Map
- b. Example TDS Map showing formulas

SOP-C-109 Determination of Filterable Residue

Attachment 1: Example TDS Map

Total Dissolved Solids													
Revised 10/1/21 jrh		QA: jrh 4/20/21				Filters from PAN		oven temp: 103-182C then 178-182C		oven: O-6, M-1			
ANALYST	Start Date/time					End Date/time		balance:		S-2			
Container	SAMPLE	Initial Wt.	Second Weight	VOLUME			Final wt. 1 Date/time	Final wt. 2 Date/time	cylinders:		mg/L		
ID	NUMBER			% difference	difference	% check	FILTERED (L)		% difference	difference	% check	TDS	
1	mb-			#DIV/0!	0.0000	#DIV/0!	0.100		#DIV/0!	0.0000	#DIV/0!	0.0	
2	LCS-			#DIV/0!	0.0000	#DIV/0!	0.100		#DIV/0!	0.0000	#DIV/0!	0.0	
3	LCSd-			#DIV/0!	0.0000	#DIV/0!	0.100		#DIV/0!	0.0000	#DIV/0!	0.0	
4	dup-			#DIV/0!	0.0000	#DIV/0!	0.100		#DIV/0!	0.0000	#DIV/0!	0.0	
5				#DIV/0!	0.0000	#DIV/0!	0.100		#DIV/0!	0.0000	#DIV/0!	0.0	
6				#DIV/0!	0.0000	#DIV/0!	0.100		#DIV/0!	0.0000	#DIV/0!	0.0	
7				#DIV/0!	0.0000	#DIV/0!	0.100		#DIV/0!	0.0000	#DIV/0!	0.0	
8				#DIV/0!	0.0000	#DIV/0!	0.100		#DIV/0!	0.0000	#DIV/0!	0.0	
9				#DIV/0!	0.0000	#DIV/0!	0.100		#DIV/0!	0.0000	#DIV/0!	0.0	
10				#DIV/0!	0.0000	#DIV/0!	0.100		#DIV/0!	0.0000	#DIV/0!	0.0	
11				#DIV/0!	0.0000	#DIV/0!	0.100		#DIV/0!	0.0000	#DIV/0!	0.0	
12				#DIV/0!	0.0000	#DIV/0!	0.100		#DIV/0!	0.0000	#DIV/0!	0.0	
13				#DIV/0!	0.0000	#DIV/0!	0.100		#DIV/0!	0.0000	#DIV/0!	0.0	
14				#DIV/0!	0.0000	#DIV/0!	0.100		#DIV/0!	0.0000	#DIV/0!	0.0	
15				#DIV/0!	0.0000	#DIV/0!	0.100		#DIV/0!	0.0000	#DIV/0!	0.0	
		In M-1:		In C-6			In M-1:	end:					

Attachment 2: Example TDS Map showing formulas

Total Dissolved Solids													
Revised 10/1/21 jrh		QA: jrh 4/20/21				Filters from PAN		oven temp: 103-182C then 178-182C		oven: O-6, M-1			
ANALYST	Start Date/time					End Date/time		balance:		S-2			
Container	SAMPLE	Initial Wt.	Second Weight	VOLUME			Final wt. 1 Date/time	Final wt. 2 Date/time	cylinders:		mg/L		
ID	NUMBER			% difference	difference	% check	FILTERED (L)		% difference	difference	% check	TDS	
1	mb-			=IF(I10*ABS(D7-C7)/C7) > 0.1, ABS(D7-C7), IF(I10*ABS(D7-C7)/C7) > 0.1, "FAIL", IF(ABS(D7-C7)/C7 > 0.1, "Over 4%", "ok")			0.1		=IF(L7*0.0005*FAL"/JFK7) > 4, "Over 4%", "ok")			=1000*(J7-D7)/H7	
2	LCS-			=IF(I10*ABS(D8-C8)/C8) > 0.1, ABS(D8-C8), IF(I10*ABS(D8-C8)/C8) > 0.1, "FAIL", IF(ABS(D8-C8)/C8 > 0.1, "Over 4%", "ok")			0.1		=IF(L8*0.0005*FAL"/JFK8) > 4, "Over 4%", "ok")			=1000*(J8-D8)/H8	
3	LCSd-			=IF(I10*ABS(D9-C9)/C9) > 0.1, ABS(D9-C9), IF(I10*ABS(D9-C9)/C9) > 0.1, "FAIL", IF(ABS(D9-C9)/C9 > 0.1, "Over 4%", "ok")			0.1		=IF(L9*0.0005*FAL"/JFK9) > 4, "Over 4%", "ok")			=1000*(J9-D9)/H9	
4	dup-			=IF(I10*ABS(D10-C10)/C10) > 0.1, ABS(D10-C10), IF(I10*ABS(D10-C10)/C10) > 0.1, "FAIL", IF(ABS(D10-C10)/C10 > 0.1, "Over 4%", "ok")			0.1		=IF(L10*0.0005*FAL"/JFK10) > 4, "Over 4%", "ok")			=1000*(J10-D10)/H10	
5				=IF(I10*ABS(D11-C11)/C11) > 0.1, ABS(D11-C11), IF(I10*ABS(D11-C11)/C11) > 0.1, "FAIL", IF(ABS(D11-C11)/C11 > 0.1, "Over 4%", "ok")			0.1		=IF(L11*0.0005*FAL"/JFK11) > 4, "Over 4%", "ok")			=1000*(J11-D11)/H11	
6				=IF(I10*ABS(D12-C12)/C12) > 0.1, ABS(D12-C12), IF(I10*ABS(D12-C12)/C12) > 0.1, "FAIL", IF(ABS(D12-C12)/C12 > 0.1, "Over 4%", "ok")			0.1		=IF(L12*0.0005*FAL"/JFK12) > 4, "Over 4%", "ok")			=1000*(J12-D12)/H12	
7				=IF(I10*ABS(D13-C13)/C13) > 0.1, ABS(D13-C13), IF(I10*ABS(D13-C13)/C13) > 0.1, "FAIL", IF(ABS(D13-C13)/C13 > 0.1, "Over 4%", "ok")			0.1		=IF(L13*0.0005*FAL"/JFK13) > 4, "Over 4%", "ok")			=1000*(J13-D13)/H13	
8				=IF(I10*ABS(D14-C14)/C14) > 0.1, ABS(D14-C14), IF(I10*ABS(D14-C14)/C14) > 0.1, "FAIL", IF(ABS(D14-C14)/C14 > 0.1, "Over 4%", "ok")			0.1		=IF(L14*0.0005*FAL"/JFK14) > 4, "Over 4%", "ok")			=1000*(J14-D14)/H14	
9				=IF(I10*ABS(D15-C15)/C15) > 0.1, ABS(D15-C15), IF(I10*ABS(D15-C15)/C15) > 0.1, "FAIL", IF(ABS(D15-C15)/C15 > 0.1, "Over 4%", "ok")			0.1		=IF(L15*0.0005*FAL"/JFK15) > 4, "Over 4%", "ok")			=1000*(J15-D15)/H15	
10				=IF(I10*ABS(D16-C16)/C16) > 0.1, ABS(D16-C16), IF(I10*ABS(D16-C16)/C16) > 0.1, "FAIL", IF(ABS(D16-C16)/C16 > 0.1, "Over 4%", "ok")			0.1		=IF(L16*0.0005*FAL"/JFK16) > 4, "Over 4%", "ok")			=1000*(J16-D16)/H16	
11				=IF(I10*ABS(D17-C17)/C17) > 0.1, ABS(D17-C17), IF(I10*ABS(D17-C17)/C17) > 0.1, "FAIL", IF(ABS(D17-C17)/C17 > 0.1, "Over 4%", "ok")			0.1		=IF(L17*0.0005*FAL"/JFK17) > 4, "Over 4%", "ok")			=1000*(J17-D17)/H17	
12				=IF(I10*ABS(D18-C18)/C18) > 0.1, ABS(D18-C18), IF(I10*ABS(D18-C18)/C18) > 0.1, "FAIL", IF(ABS(D18-C18)/C18 > 0.1, "Over 4%", "ok")			0.1		=IF(L18*0.0005*FAL"/JFK18) > 4, "Over 4%", "ok")			=1000*(J18-D18)/H18	
13				=IF(I10*ABS(D19-C19)/C19) > 0.1, ABS(D19-C19), IF(I10*ABS(D19-C19)/C19) > 0.1, "FAIL", IF(ABS(D19-C19)/C19 > 0.1, "Over 4%", "ok")			0.1		=IF(L19*0.0005*FAL"/JFK19) > 4, "Over 4%", "ok")			=1000*(J19-D19)/H19	
14				=IF(I10*ABS(D20-C20)/C20) > 0.1, ABS(D20-C20), IF(I10*ABS(D20-C20)/C20) > 0.1, "FAIL", IF(ABS(D20-C20)/C20 > 0.1, "Over 4%", "ok")			0.1		=IF(L20*0.0005*FAL"/JFK20) > 4, "Over 4%", "ok")			=1000*(J20-D20)/H20	
15				=IF(I10*ABS(D21-C21)/C21) > 0.1, ABS(D21-C21), IF(I10*ABS(D21-C21)/C21) > 0.1, "FAIL", IF(ABS(D21-C21)/C21 > 0.1, "Over 4%", "ok")			0.1		=IF(L21*0.0005*FAL"/JFK21) > 4, "Over 4%", "ok")			=1000*(J21-D21)/H21	
		In M-1:		In C-6			In M-1:	end:					