

SOP-C-130

**Determination of Total Solids
(Total Residue) and Percent Solids**

Revision 13

Approval:



Laboratory Manager

4-18-22

Date



Concurrence

04-18-22

Date

Effective date: 4-18-22

Renewal date: 4-18-23 Initials: Jmt

Texas Institute for Applied Environmental Research

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- i. **Identification of the method**
 - a. SM 2540 G & B (approved 2011)
- ii. **Applicable matrix or matrices**
 - a. Nonpotable water (Not TNI accredited)
 - b. Soils and other solids (not TNI accredited)
- iii. **Limits of detection and quantitation**
 - a. LOD determined annually; refer to Laboratory Manager's most recent memorandum
 - b. For upper limits, solids may have values up to 1,000,000 mg/Kg or 100%.
 - c. Practical range for water samples is 10 to 20,000 mg/L.
- iv. **Scope and application, including parameters to be analyzed**
 - a. The purpose of the procedure is to provide a method for the determination of total solids (TS) using the EPA and TNI approved protocols.
 - b. This procedure establishes routine guidelines for determining TS to obtain comparable results from one analyst to another.
- v. **Summary of the method**
 - a. Gravimetric (weighing) of sample before and after drying
- vi. **Definitions**
 - a. Refer to QAM-Q-101 for standard QC definitions.
 - b. Residue, total (total solids, TS) refers to the sum of the homogenous total suspended solids (TSS) and total dissolved solids (TDS) material in a water sample. By determining any two of these solids types, the third may be derived mathematically. In solid samples, the term "percent solids" is substituted for "total solids", but the primary concept of removing moisture, or water, to obtain the amount of actual solids is the same.
- vii. **Interferences**
 - a. Highly mineralized water with a significant concentration of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and require prolonged drying, proper desiccation, and rapid weighing.
 - b. Large, floating particles or submerged agglomerates of nonhomogeneous materials from the sample are excluded if

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it is determined that their inclusion is not desired in the final result.

- c. Visible floating oil and grease may be dispersed with a blender before withdrawing a sample portion for analysis. If oil and grease sticks to blender sides and blades, thus potentially affecting sample composition, note this in the lab report.
- d. Because excessive residue in the dish may form a water-trapping crust, sample is limited to no more than 200 mg residue.
- e. For solids samples, the container remains closed to prevent moisture loss and loss of ammonium carbonate or volatile organic matter.
- f. All weighings are made quickly because wet samples tend to lose weight by evaporation. After drying or ignition, residues often are very hygroscopic and rapidly absorb moisture from the air.
- g. Highly alkaline residues may react with silica in the samples or silica containing crucibles.
- h. Residues dried at 103-105°C may retain both water of crystallization and some mechanically occluded water. There will be CO₂ loss when bicarbonate converts to carbonate during drying. Usually, very little organic matter will volatilize. It may take a long time to attain constant weight because occluded-water removal is marginal at this temperature.
- i. Determination of total and volatile solids in solid and semisolid samples is subject to negative error due to loss of ammonium carbonate and volatile organic matter during drying. Take all weight measurements quickly because wet samples tend to lose weight via evaporation. After drying, residues are often hygroscopic, rapidly absorbing moisture from the air.
- j. Highly alkaline residues may react with silica in samples or in silica-containing crucibles.

viii. Safety

- a. Use the tongs to remove the evaporating dish/crucible from the oven, or oven mitts to remove a tray of dishes/crucibles.

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- b. All aspects of this procedure comply with QAM-S-101, "Laboratory Safety".
- ix. **Equipment and supplies**
 - a. Drying oven set at $104 \pm 1^{\circ}\text{C}$
 - b. Desiccator
 - c. Analytical balance capable of weighing increments of 0.1 mg.
 - d. Isco™ plastic sample bottles, or equivalent (liter), with cap
 - e. Tongs
 - f. Evaporating dishes/crucibles made of Vycor™, platinum, porcelain, or tempered high silica glass with sufficient capacity to hold 100 mL or the volume of filtered water or solid aliquot
 - g. 100-mL graduated Class A cylinder, glass (To Contain)
 - h. Various laboratory soil preparation devices may be required, including a pulverizer, blender, or grinder
 - i. Wide-bore pipettes, beakers, stirrers, other general lab equipment
- x. **Reagents and standards**
 - a. No reagents are used
 - b. Standard- TS/TVS/TFS standard, 300/134/166 mg/L (LCS/LCSD- may be used in conjunction with SOP-C-108, "Determination of Nonfilterable Volatile and Fixed Solids" also).
 - i. Ignite about 0.5 grams of NaCl at 550°C in a muffle furnace for 20 minutes, cool and store in a desiccator.
 - ii. Dry 1 gram of KHP at $104 \pm 1^{\circ}\text{C}$ in an oven for one hour, cool and store in a desiccator until needed.
 - iii. Weigh 0.1000 g of prepared NaCl and 0.2000 g of prepared KHP and dissolve in about 800 mL DI water in a 1L volumetric flask. Dilute to volume. Standard is ready for analysis. Prepare a duplicate in the same manner.
- xi. **Sample collection, preservation, shipment and storage**
 - a. Refrigerate sample to $>0\text{-}\leq 6^{\circ}\text{C}$
 - b. Samples are not normally collected or shipped by the lab.
 - c. Holding Time: 7 days for liquid samples, 6 months for solids

xii. Quality control

- i. All weights are measured quickly due to loss by evaporation or adsorption of moisture.
- ii. A method blank is analyzed for each group of 20 samples or less. Initiate corrective action if the method blank exceeds the project required reporting limit. If the blank exceeds 5 times the limit, the batch of samples is reanalyzed where possible.
- iii. For every 10 samples, analyze at least one sample duplicate. Analyze an LCS/LCSD pair and a method blank with every 20 samples or less. If any duplicate relative percent deviation (RPD) does not pass the project-specific acceptance criteria for precision and holding time has not been exceeded, reanalyze if possible.
- iv. Complete a Corrective Action Report in accordance with QAM-Q-105, "Corrective Actions" for all failures in quality control.
- v. Additional QC samples may be required, as specified in project quality assurance project plans.
- vi. Use a controlled spreadsheet on the Laboratory Manager's computer to calculate results, if desired and available.
- vii. The analytical balance is calibration checked with the 0.1g class S weight, before determining the initial and final weight of the evaporating dish/crucible, in accordance with QAM-I-101, "Operation and Calibration of the Analytical Balance".
- viii. All data are documented and maintained in accordance with QAM-A-102, "Documentation and Data Control".
- ix. Laboratory Control Standards using sodium chloride and KHP are to be used for acceptance criteria. The standard is dried to calculate percent recovery. Duplicate precision, recovery and bias are calculated from these standard recoveries depending on specific project requirements, which determine data acceptability. There is no LCS/LCSD for % solids analysis of solid samples.

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- x. All aspects of this procedure comply with QAM-Q-101, "Laboratory Quality Control".
- xi. Duplicate acceptance limits are $\pm 10\%$ RPD for sample duplicates, $\pm 20\%$ RPD for LCS/LCSD and $\pm 30\%$ RPD for field splits.
- xii. No AWRL/LOQ standard is applicable for this procedure.

xiii. Calibration and standardization

- a. not applicable other than LCS/LCSD described above

xiv. Procedure**a. Sample Handling**

- i. Non-representative particulates, such as leaves, sticks, fish and lumps of fecal matter, are excluded from the sample if it is determined that their inclusion is not desired in the final result. If floating oil or grease is present, it may be dispersed by a blender device before aliquoting.
- ii. Analysis begins as soon as possible. Samples are immediately be placed on ice or refrigerated to maintain a temperature of $0 \leq 6^{\circ}\text{C}$, and thereby minimize microbiological decomposition of solids. Bring samples to room temperature before analysis.
- iii. Save approximately 100 mL of the water sample for analysis. Mix the sample well before dividing it into aliquots.

b. Preparation of Evaporating Dish/crucible

- i. Heat a clean evaporating dish/crucible to $104 \pm 1^{\circ}\text{C}$ for at least one hour in drying oven. Cool in desiccator to ambient temperature.
- ii. Weigh the evaporating dish/crucible to the nearest 0.1 mg immediately before use after removal from desiccator and record. Store dish /crucible in desiccator until use.

c. Selection of Sample Volume

- i. Choose an aliquot of sample sufficient to contain a residue of at least 0.0025 g but no more than 0.20 g.

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Repeat where necessary to obtain this where volume and holding time allow. A 100 mL volume of sample aliquot is usually sufficient enough to produce an adequate amount of residue. Record the volume.

- ii. To obtain a weighable residue, successive aliquots of sample may be added to the same dish/crucible. The volume of each successive aliquot is measured and recorded in the same manner as the original measurement.
 - iii. For semisolid samples, stir or shake to homogenize, transfer approximately 25-50 g to a prepared evaporating dish and weigh (dish plus sample).
 - iv. For solid samples—If sample consists of discrete pieces of solid material, then take care to obtain a representative sample whose particle size will not impede drying. Sample may be pulverized on a clean surface by hand (covered in clean gloves) or using a clean mortar and pestle. Manually process samples as quickly as possible to prevent moisture loss. Processing via mechanical grinding is not recommended because moisture levels could drop during processing. Transfer approximately 25-50 g to a prepared evaporating dish and weigh.
 - v. Record aliquot weights in the personal logbook/Elog.
- d. Evaporating or drying the selected sample
- i. For liquid samples, visually inspect the sample. For apparently low suspended solid samples, shake the container vigorously at least 25 times to mix and pour a measured volume into a calibrated graduated cylinder (To Contain type). For higher apparent suspended solids, stir sample with a magnetic stirrer in a beaker. While stirring, pipet from a point mid-depth and midway between the container wall and vortex.
 - ii. Transfer the measured amount of aliquot to the pre-weighed dish/crucible and evaporate to dryness in a drying oven.
 - 1. For liquid or semisolid samples, the temperature is lowered to approximately 98°C. If no boiling

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and splattering occurs, samples may be dried at 103-105°C to dryness. Evaporate overnight at this temperature, if needed.

2. Dry the evaporated water sample for another 1 hour at 103-105°C, if not already done.
3. For solid samples, place sample in an oven at 103-105°C overnight.
4. Cool in a desiccator and weigh dish/crucible to the nearest 0.1 mg. Record the weight in the personal logbook/Elog.
5. Repeat the cycle of drying at 103-105°C, cooling, desiccating and weighing until a constant weight is obtained or until loss of weight is less than 4% of the previous weight, or 0.0005 g, whichever is less. Normally, the cycle repeated twice is sufficient. Record the weight to the nearest 10 mg. Depending on sample sizes, convert measurements to mg, Kg, L or mL as needed before using the calculations.
6. When weighing dried samples, be alert to changes in weight due to air exposure and/or sample degradation.

xv. Data analysis and calculations;

- a. Calculations for Total Solids or Percent Solids:

$$\text{Total Solids (water) mg/L} = \{(A-B) / C\}$$

A = weight of dried sample + evaporating dish/crucible in mg

B = weight of evaporating dish/crucible in mg

C = L of sample used

$$\% \text{ Solids} = \{100*(A-B) / C\}$$

A = weight of dried sample + evaporating dish/crucible in g

B = weight of evaporating dish/crucible in g

C = g of sample used

xvi. Method performance

- a. Method performance: refer to QAM-Q-101, "Laboratory Quality Control"

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xvii. Pollution prevention

- a. Pollution prevention: refer to QAM-W-101, "Disposal of Laboratory Waste"

xviii. Data assessment and acceptance criteria for quality control measures

- a. Data assessment and acceptance: refer to QAM-Q-101, "Laboratory Quality Control"

xix. Corrective actions for out-of-control data

- a. Corrective action: refer to QAM-Q-105, "Corrective Actions"

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

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

xxi. Waste management

- a. Waste management: refer to QAM-W-101, "Disposal of Laboratory Waste". No hazardous waste is expected to be generated from this procedure unless from samples.

xxii. References

- a. Standard Methods for the Examination of Water and Wastewater, ed. by Andrew D. Eaton, et al., APHA, AWWA, WEF, Washington, D.C., latest online edition, Methods 2540 G and B, Quality control method 2020 (approved 2017).
- b. The National Environmental Laboratory Accreditation Conference Institute (TNI) standard, 2016.

xxiii. Any tables, diagrams, flowcharts and validation data

- a. Example TS Map
- b. Example TS Map showing formulas
- c. Example Percent Solids Map
- d. Example Percent Solids Map showing formulas

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Attachment 1: Example TS Map

Total Solids / Total Volatile and Fixed Solids															
ANALYST		Start Date/Time		End Date/Time		PAN		Balance		M=1 540-555C		after 540-555C			
JTH		04/11/2013		04/11/2013		540-555C		540-555C		540-555C		Furnace ID: M-1			
Container		SAMPLE		INITIAL weight (g)		Second Weight		% difference		#DIV/0!		Furnace ID: M-1		Furnace ID: M-1	
ID	NUMBER					VOLUME (L)		mg/L		mg/L		mg/L		mg/L	
A1	mb-					0.100		0.0		0.0					
A2	LCS-					0.100		0.0		0.0					
A3	LCSd-					0.100		0.0		0.0					
A4						0.100		0.0		0.0					
A5	dup-					0.100		0.0		0.0					
A6						0.100		0.0		0.0					
A7						0.100		0.0		0.0					
A8						0.100		0.0		0.0					
A9						0.100		0.0		0.0					
A10						0.100		0.0		0.0					
A11						0.100		0.0		0.0					
A12						0.100		0.0		0.0					
A13						0.100		0.0		0.0					
A14						0.100		0.0		0.0					
A15						0.100		0.0		0.0					
A16						0.100		0.0		0.0					
In O-6 @ M-1		In O-6 @		back in O-6 @		In M-1 @		back in M-1 @		back in M-1 @		back in M-1 @		back in M-1 @	

Attachment 2: Example TS Map showing formulas

Total Solids / Total Volatile and Fixed Solids															
ANALYST		Start Date/Time		End Date/Time		PAN		Balance		Furnace ID: M-1		Furnace ID: M-1		Furnace ID: M-1	
JTH		04/11/2013		04/11/2013		540-555C		540-555C		540-555C		540-555C		540-555C	
Container		SAMPLE		VOLUME		mg/L		mg/L		mg/L		mg/L		mg/L	
ID	NUMBER	INITIAL weight g	Second Weigh g	% difference	#DIV/0!	% difference	#DIV/0!	% difference	#DIV/0!	% difference	#DIV/0!	% difference	#DIV/0!	% difference	#DIV/0!
A1	mb-														
A2	LCS-														
A3	LCSd-														
A4															
A5	dup-														
A6															
A7															
A8															
A9															
A10															
A11															
A12															
A13															
A14															
A15															
A16															
In O-6 @ M-1		In O-6 @		back in O-6 @		In M-1 @		back in M-1 @		back in M-1 @		back in M-1 @		back in M-1 @	

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Attachment 3: Example Percent Solids Map

Percent Solids										PAN		oven O-6 103-105C					
ANALYST		Start Date/time		QA: jrh 11/21/19		End Date/time		beaker prep 540-555C		balance: S-2		cylinders:					
Container	SAMPLE	INITIAL Weight (g)	Second Weight	% difference	difference	% check	(g)	Penultimate Weight (g)	Ultimate Weight (g)	Percent Solids	QC Check	% difference	difference	% check	Comments		
1	mb-			#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		
2				#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		
3	dup-			#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		
4				#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		
5				#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		
6				#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		
7				#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		
8				#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		
9				#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		
10				#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		
11				#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		
12				#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		
13				#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		
14	dup-			#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		
15				#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		
16				#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		
17				#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		
18				#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		
19				#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		
20				#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		
21				#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		
22				#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		
23				#DIV/0!	0.000	#DIV/0!				#DIV/0!	Too little residue	#DIV/0!	0.000	#DIV/0!	na		

Container ID	SAMPLE NUMBER	Final Weight 1 (g)	Final Weight 2 (g)	Final Weight 3 (g)	Final Weight 4 (g)	Final Weight 5 (g)	Final Weight 6 (g)	Final Weight 7 (g)	Final Weight 8 (g)	Final Weight 9 (g)	Final Weight 10 (g)	Final Weight 11 (g)	Final Weight 12 (g)	Final Weight 13 (g)	Final Weight 14 (g)
1	mb-														
2															
3	dup-														
4															
5															
6															
7															
8															
9															
10															
11															
12															
13															
14	dup-														
15															
16															
17															
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22															
23															

