



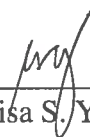

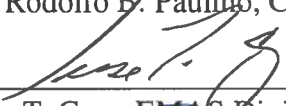

Guam EPA Laboratory
 B-15-6101 Mariner Ave.
 Tiyan, Barrigada
 Guam 96921

Title: Total Suspended Solids, SM 2540 D
 Number: CH-01-10
 Date: 9/25/2015
 Rev. no. 000

**GUAM ENVIRONMENTAL PROTECTION AGENCY
 EMAS ANALYTICAL PROGRAM**

STANDARD OPERATING PROCEDURE

**ANALYSIS OF TOTAL SUSPENDED SOLIDS
 DRIED at 103-105°C BY SM 2540 D**

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1 SCOPE AND APPLICATION

- 1.1 This method is applicable for drinking, ground, surface and saline waters, domestic and industrial wastes.
- 1.2 The practical range of determination is from 5 to 2000 mg/L. The method reporting limit is 5 mg/L total suspended solids (TSS).

2 METHOD SUMMARY

- 2.1 A well-mixed sample is filtered through a weighed standard glass-fiber filter (GF/F), and the residue retained on the filter is dried to constant weight at 103 to 105°C. The increase in weight of the filter represents the total suspended solids.
- 2.2 If total dissolved solids (TDS) is also being determined, the filtrate from this method may be used for the TDS analysis.

3 INTERFERENCES

- 3.1 Samples high in total dissolved solids, such as saline waters, brines and some wastes, maybe subject to a positive interference from residual TDS on the filter pad. Care must be taken during the rinse step to remove all traces of TDS from the filter pad.
- 3.2 Prolonged filtration times resulting from filter clogging may produce high results owing to increased colloidal materials captured on the clogged filter.

4 DEFINITIONS

- 4.1 Total Suspended Solids (TSS) – TSS is defined as the solids incapable of passing through a glass fiber filter and dried to constant weight at 103-105°C. TSS is often referred to as non-filterable residue.
- 4.2 Total Dissolved Solids (TDS) – TDS is defined as the residue from an aqueous sample which is passed through a glass fiber filter and dried to constant weight at 180°C. TDS is often referred to as filterable residue.
- 4.3 Laboratory Duplicate (LD) – an aliquot of sample prepared and analyzed separately, using identical procedures. Analyses of a sample and LD indicate precision associated with laboratory procedures, but do not indicate precision in sample collection, preservation or storage procedures.



- 4.4 Laboratory Reagent Blank (LRB) – An aliquot of reagent water that is treated exactly as a sample. The LRB is used to detect sample contamination resulting from the procedures used to prepare and analyze the samples in the laboratory environment.

5 SAMPLE HANDLING AND PRESERVATION

- 5.1 Samples must be stored at 4°C up to the time of analysis to minimize microbiological decomposition of solids.
- 5.2 Samples must be analyzed within 7 days from the date of collection.
- 5.3 Samples must be at room temperature for analysis.

6 EQUIPMENT AND SUPPLIES

- 6.1 Analytical balance capable of weighing to 0.1 mg
- 6.2 NBS Class “S” weights
- 6.3 Filter holder or membrane filter funnel, 47 mm
- 6.4 Glass fiber filter discs, 47 mm, without organic binder, Whatman type GF/F (0.45um)
- 6.5 Tweezers
- 6.6 Suction flask, 1000 mL, or manifold
- 6.7 Aluminum weighing dishes
- 6.8 Desiccator, containing dry desiccant
- 6.9 Vacuum pump
- 6.10 Plastic or Teflon spray bottle
- 6.11 Graduated cylinders, 100-mL, 250-mL and 500-mL
- 6.12 Drying oven, for operation at 103 to 105°C

7 REAGENTS AND STANDARDS

- 7.1 Nanopure reagent grade water (ASTM Type I water)

8 QUALITY CONTROL

- 8.1 The analytical balance and NBS Class “S” weights must be calibrated at least annually.
- 8.2 A monthly calibration check using NBS Class “S” weights must be performed on analytical balances. See Appendix E for details including tolerance range.



8.3 A balance calibration verification with NBS Class “S” weights of 100 mg, 1 g and 10 g must be performed on analytical balance each day prior to use for TSS measurements. This step enables the analyst to determine if the balance is working properly on a daily basis. See Appendix E.

8.4 Routine Analytical Quality Control

8.4.1 LRB – The laboratory must analyze at least one LRB with each 20 or fewer samples of the same matrix. LRB data are used to assess contamination from the laboratory environment. LRB values that exceed the reporting limit of 5 mg/L indicate potential laboratory contamination. If the potential contamination significantly impacts the analytical results, the LRB must be re-prepared along with the affected samples, and re-analyzed.

8.4.2 LD – Sample homogeneity and laboratory variability can affect the quality and interpretation of data. LD results can be used to assess both the sample homogeneity and laboratory variability.

One LD must be prepared for every 10 routine samples of the same matrix in a sample batch (e.g., 1 LD for a batch containing 1-10 routine samples) Shake the sample selected as the LD, obtain a representative aliquot, and proceed with the sample preparation and analysis, treating the LD sample as a routine sample.

Calculate the relative percent difference (RPD) using the following equation:

$$RPD = \frac{(C_{ld} - C)}{(C_{ld} + C) / 2} \times 100$$

Where

- RPD = relative percent difference
- C_{ld} = measured TSS in the LD
- C = measured TSS in the routine sample

The relative percent difference (RPD) must be ≤20% for samples with TSS greater than or equal to 50 mg/L. The absolute difference between duplicate results must be less than the reporting limit (5 mg/L) for samples containing less than 50 mg/L TSS. If the control limits are exceeded, re-analyze the sample and duplicate once. If the control limits are exceeded again, flag all associated sample results. Document actions in the **Notes** section of the LIMS analytical results report.



9.0 ANALYTICAL PROCEDURES

9.1 Preparation of the glass-fiber filter disk:

- 9.1.1 With tweezers, insert the filter disk on to the base and clamp on funnel.
- 9.1.2 While pressure is applied, wash the disk with three successive 20 mL volumes of reagent water.
- 9.1.3 Remove all traces of water by continuing to apply vacuum after water passage.
- 9.1.4 Remove the funnel from the base, and with tweezers place filter in the aluminum dish and dry in an oven at 103-105°C (for at least two hours for initial drying and 1 hour for subsequent drying). Place the dried filter and the dish in a desiccator to cool.
- 9.1.5 Weigh the filter and the dish to the nearest 0.1 mg and record the tare weight.
- 9.1.6 Repeat drying, desiccating and weighing until a constant weight is obtained (weight loss is less than 0.5 mg). Record the tare weights (filter + dish) in a logbook and store the pre-weighed filters in a desiccator.

9.2 Selection of sample sizes:

- 9.2.1 Choose sample volume (generally 100 to 200 mL) to yield no more than 200 mg dried residue. If the mass of dried residue is less than 0.5 mg, increase the sample volume up to 1 L.
- 9.2.2 If filtration time takes more than 5 to 10 minutes, begin the procedure again using a new pre-washed and pre-weighed filter and a small sample aliquot (i.e. pour sample onto the filter funnel in portions of 10 ml volume) so that filtration time approximates 2 to 4 minutes.

9.3 Sample Analysis:

- 9.3.1 Assemble filtering apparatus. With tweezers, place a pre-weighed filter on the base and clamp on funnel and apply vacuum. Wet the filter with a small volume of reagent grade water to seat it.
- 9.3.3 Shake the sample vigorously, and with a Class "A" graduated cylinder, rapidly transfer the appropriate sample volume onto the seated glass-fiber filter.



- 9.3.4 Rinse the graduated cylinder, filter funnel wall and the non-filterable residue onto the filter with three successive 10 mL portions of reagent water. Remove all traces of water by continuing to apply vacuum after the water has passed through.
- 9.3.5 Carefully remove the filter from the filter assembly with tweezers and place in the aluminum dish (the same one used to obtain tare weight). Dry at 103-105°C (at least two hours for initial drying and one hour for subsequent drying). Cool in desiccator and weigh to the nearest 0.1 mg. Record the weight in a logbook.
- 9.3.6 Repeat drying, desiccating and weighing cycle until the weights from two consecutive cycles differ by less than 0.5 mg. Use the smallest weight from the final two cycles for calculation purposes.

9.4 Data Reduction and Reporting:

- 9.4.1 Calculate TSS as follows:

$$\text{TSS} = \frac{(A-B)}{C} \times 1000$$

Where: TSS = Total Suspended Solids in mg/L

A = weight of suspended solids + filter + weighing dish, mg
B = tare weight of filter + weighing dish, mg
C = sample volume, mL

If the mass of suspended solids in a sample (A-B) is less than 0.50 mg, the sample must be re-analyzed using a larger volume.

- 9.4.2 Reporting – Results will be reported to 2 significant figures for TSS less than 100 mg/L and 3 significant figures for TSS greater than or equal to 100 mg/L. For rounding results, adhere to the following rules:
- If the number following those to be retained is less than 5, round down;
 - If the number following those to be retained is greater than 5, round up; or
 - If the number following the last digit to be retained is equal to 5, round down if the digit is even, round up if the digit is odd.

10 DOCUMENTATION

- 10.1 When samples are received in the laboratory, the laboratory personnel verify that the Chain of Custody Form (Appendix A) is properly filled out. The Chain of Custody Form should contain the following information:



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- a. Project name
- b. Sampler's name and signature
- c. Date and time of sample collection
- d. Field sample ID
- e. Source of sample (including name, location and sample type)
- f. Analyses required
- g. Preservative used if applicable

The person who delivers the samples may relinquish custody and laboratory personnel may then receive and sign the Chain of Custody Form.

- 10.2 Raw analytical data and results are recorded in the logbook, Total Suspended Solids – Dried at 103 – 105°C, SM 2540 D (GEPA Log: CH-02-04) (Appendix C), and then entered in the Laboratory Information Management System (LIMS). Analytical results are reported either in LIMS format or Microsoft Excel format as required by customer. (Appendix B).
- 10.3 Oven temperature readings are recorded in the Drying Oven Temperature Logbook (GEPA Log: QC-12-01) (Appendix D).
- 10.4 Balance calibration data are recorded in the Balance #2 (Sartorius) Calibration Logbook (GEPA Log: QC-10-02) (Appendix E).

11 REFERENCES

- 11.1 SM 2540 D, Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998.
- 11.2 USEPA Region 9 Lab, SOP #462, Analysis of Total Suspended Solids By EPA Method 160.2, Revision 1, June 16, 1998.



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Appendix A: Chain of Custody Form



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Appendix B: Analytical Results Report (LIMS or Spreadsheet)

GUAM ENVIRONMENTAL PROTECTION AGENCY
EMAS Analytical Program

ANALYTICAL REPORT

Report: TSS-02564
 Parameter: Total Suspended Solids
 Method: SM 2540 D
 Date of Report: 10/14/2013
 Matrix: water
 Analyst: E. Yanit *EY*

GEPA Lab ID	Field Sample ID	Date Collected	Date Analyzed	MDL	Result	Unit	Remarks
	Lab Blank	1/22/2013	1/23/2013	5	<5	mg/L	Lab Blank
02564-041	AGRD	1/22/2013	1/23/2013	5	<5	mg/L	
02564-042	AGRA-3	1/22/2013	1/23/2013	5	6.00	mg/L	
02564-043	AGRF-2	1/22/2013	1/23/2013	5	56.0	mg/L	
02564-044	ASRI-2	1/22/2013	1/23/2013	5	23.6	mg/L	
02564-045	ASRI-4	1/22/2013	1/23/2013	5	35.2	mg/L	
02564-046	ASRM	1/22/2013	1/23/2013	5	<5	mg/L	
02564-047	APRM-1B	1/22/2013	1/23/2013	5	<5	mg/L	
02564-048	ATRN-2	1/22/2013	1/23/2013	5	<5	mg/L	
02564-049	ATRO	1/22/2013	1/23/2013	5	<5	mg/L	
02564-050	ATRF-3	1/22/2013	1/23/2013	5	<5	mg/L	
02564-051	ATRT-2	1/22/2013	1/23/2013	5	<5	mg/L	
02564-052	ULRU-2	1/22/2013	1/23/2013	5	<5	mg/L	
02564-053	MZRT-2	1/22/2013	1/23/2013	5	<5	mg/L	
02564-054	MZRG-2	1/22/2013	1/23/2013	5	<5	mg/L	
02564-055	INRAGB-3	1/22/2013	1/23/2013	5	37.60	mg/L	
02564-056	INRI-2	1/22/2013	1/23/2013	5	<5	mg/L	
02564-057	TUETO	1/22/2013	1/23/2013	5	11.0	mg/L	

Peer Reviewed By: *EY*
 Date Reviewed: 10/28/13

Approved By: *EY*
 Date Approved: 10/28/13

GUAM ENVIRONMENTAL PROTECTION AGENCY
EMAS Analytical Program

ANALYTICAL REPORT

Report: TSS-02564_02567_02569
Parameter: Total Suspended Solids
Method: SM 2540 D

Date of Report: 10/14/2013

Matrix: water

Analyst: E. Yanit *EY*

GEPA Lab ID	Field Sample ID	Date Collected	Date Analyzed	MDL	Result	Unit	Remarks
02564-058	TURTG-1C	1/22/2013	1/23/2013	5	16.0	mg/L	
02564-059	PGMPW bridge	1/22/2013	1/23/2013	5	<5	mg/L	
02564-045	ASRI-4 (LD)	1/22/2013	1/23/2013	5	36.4	mg/L	Lab duplicate
	Lab Blank	1/28/2013	1/31/2013	5	<5	mg/L	Lab Blank
0267-015	AGMS	1/28/2013	1/31/2013	5	<5	mg/L	
0267-016	AGMD	1/28/2013	1/31/2013	5	<5	mg/L	
0267-017	AGMP	1/28/2013	1/31/2013	5	<5	mg/L	
0267-018	AGM11	1/28/2013	1/31/2013	5	<5	mg/L	
0267-019	AGMB	1/28/2013	1/31/2013	5	<5	mg/L	
0267-020	AGMFB	1/28/2013	1/31/2013	5	<5	mg/L	
0267-021	ASM22	1/28/2013	1/31/2013	5	<5	mg/L	
0267-022	ASM14	1/28/2013	1/31/2013	5	<5	mg/L	
0267-023	PBM15	1/28/2013	1/31/2013	5	<5	mg/L	
0267-024	APM18	1/28/2013	1/31/2013	5	<5	mg/L	
0267-025	APMOB	1/28/2013	1/31/2013	5	<5	mg/L	
02567-025	APMOB (LD)	1/28/2013	1/31/2013	5	<5	mg/L	Lab duplicate
	Lab Blank	1/29/2013	1/31/2013	5	<5	mg/L	Lab Blank
02569-014	APM20	1/29/2013	1/31/2013	5	6.0	mg/L	

Peer Reviewed By: *MAN*
Date Reviewed: 10/28/13

Approved By: *JJA*
Date Approved: 12/28/13



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Appendix C: TSS Analytical Results Logbook page

GUAM ENVIRONMENTAL PROTECTION AGENCY - EMAS Analytical Program
Total Suspended Solids Dried at 103-105°C, SM 2540 D.

Date: _____

Analyst: _____

GEPA LAB ID #	Sample ID/Station Location									Remarks
	A = Weight of filter + dried residue (g)									
	Trial # 1									
	Trial # 2									
	Trial # 3									
	B = Weight of filter (g)									
	Trial # 1									
	Trial # 2									
	Trial # 3									
	(A-B)x1000 = Weight of dried residue (mg)									
	Sample Volume (L)									
	TSS (mg/L) = A-B (mg)/sample volume (L)									



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Appendix D: Drying Oven Temperature Logbook page

GUAM EPA LABORATORY

DRYING OVEN

TEMPERATURE LOG

GEPA Log: QC-11-02

Date	Time	Temperature (°C)	Acceptable (Y/N)	Initials	Notes

CRITERIA: Temperature range: 103°C to 105°C (SM 2540D - Total Suspended Solids)
105°C (SM10300C - Dry and Ash-Free Weight)
180 ± 2°C (SM 2540C - Total Dissolved Solids)



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Appendix E: Balance Calibration Logbook page

GUAM EPA LABORATORY
ANALYTICAL BALANCE # 2 (Sartorius ED224S)
CALIBRATION LOG

Date	Class "S" Weights (g)				Initials	Notes
	0.1000	1.0000	10.0000	100.0000		

CRITERIA: Monthly calibration check with NBS Class "S" weights to $\pm 0.0005g$.
ACTION: If value is greater than criteria, inform supervisor.
NOTE: Class "S" weights equivalent to ASTM type 1 or 2.

