



Guam EPA Laboratory
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Title: Total Dissolved Solids, SM 2540 C
Number: CH-01-11
Date: 09/25/2015
Rev. no. 000

**GUAM ENVIRONMENTAL PROTECTION AGENCY
EMAS ANALYTICAL PROGRAM**

STANDARD OPERATING PROCEDURE

**ANALYSIS OF TOTAL DISSOLVED SOLIDS
DRIED at 180°C BY SM 2540 C**

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Periodic Review:		
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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 8,000 mg/L. The method reporting limit is 5 mg/L total dissolved solids (TDS).

2 METHOD SUMMARY

- 2.1 A well-mixed sample aliquot is filtered through standard glass-fiber filter (GF/F). The filtrate is evaporated to dryness in a weighed evaporating dish and dried to constant weight at 180°C. The increase in dish weight represents the total dissolved solids.
- 2.2 If total suspended solids (TSS) is also being determined, the residue retained on the filter from this method may be used for TSS analysis.

3 INTERFERENCES

- 3.1 Samples high in calcium, magnesium, chloride and/or sulfates content may be hygroscopic and require prolonged drying, proper desiccation and rapid weighing.
- 3.2 Samples high in bicarbonate require careful and possibly prolonged drying at 180°C to insure complete conversion of bicarbonate to carbonate.
- 3.3 Because excessive residue in the evaporating dish may form a water-trapping crust, limit sample to no more than 200 mg residue.

4 DEFINITIONS

- 4.1 Total Dissolved Solids (TDS) – (or Filterable Residue) are the solids from an aqueous sample capable of passing through a glass fiber filter and dried to constant weight at 180°C.
- 4.2 Total Suspended Solids (TSS) – (or Non-Filterable Residue) are the solids incapable of passing through a glass fiber filter and dried to constant weight at 103-105°C.
- 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 disks, 47 mm, without organic binder, Whatman type GF/F (0.45um)
- 6.5 Tweezers
- 6.6 Suction flask, 250 to 500 mL capacity
- 6.7 Evaporating dishes (porcelain) or beakers, 150- mL volume
- 6.8 Desiccator, containing dry desiccant
- 6.9 Vacuum pump
- 6.10 Plastic or Teflon spray bottle
- 6.11 Graduated cylinders, 100-mL
- 6.12 Safety tongs for evaporating dishes
- 6.13 Drying oven, for operation at $180 \pm 2^{\circ}\text{C}$ and $104 \pm 1^{\circ}\text{C}$

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 heavier or lighter than the weight of the evaporating dishes (i.e., 100 g, 10 g and 1 g) must be performed on analytical balance each day prior to use for TDS 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 TDS in the LD
- C = measured TDS in the routine sample

The relative percent difference (RPD) must be ≤20% for samples with TDS 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 TDS. 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 evaporation dishes:

9.1.1 Heat the clean evaporating dishes to 180°C for at least one hour. Cool in a desiccator and weigh.

9.1.2 Repeat heating, cooling, desiccating and weighing until a constant weight is obtained (weight loss is less than 0.5 mg). Store in desiccator until needed.

9.2 Preparation of glass fiber filter disk:

9.2.1 With tweezers, insert disk with wrinkled side up into filtration apparatus.

9.2.2 Apply vacuum and wash disk with three successive 20-mL volumes of reagent-grade water.

9.2.3 Remove all traces of water by continuing to apply vacuum after water has passed through.

9.2.4 Discard washings.

9.3 Determination of volume of sample to be filtered:

9.3.1 Determine the sample conductivity following the SOP for Conductivity by EPA 120.1 (SOP Number: CH-01-05) and record the results in the Analytical Results Logbook (GEPA Log: CH-02-02).

9.3.2 From the following table, determine the correct volume to filter for TDS determination. Failure to check the conductivity beforehand may cause technical holding times to be exceeded if samples are reanalyzed because of high TDS.

Sample Conductivity (μ mhos/cm)	Volume to filter (mL)
0 – 3000	100
3000 – 6000	50
6000 - 12000	25



9.4 **Sample Analysis:**

9.4.1 Assemble the filtering apparatus and begin suction.

9.4.2 Shake the sample vigorously and rapidly transfer the appropriate volume to the pre-washed filter apparatus using a 100-mL Class A graduated cylinder.

9.4.3 Wash with three successive 10-mL volumes of reagent-grade water, allowing complete drainage between washings, and continue suction for about 3 minutes after filtration is complete.

9.4.4 Transfer total filtrate (with washings) to a weighed evaporating dish and evaporate to dryness in a drying oven. Continue to dry the evaporated sample for at least 1 hour in an oven at $180 \pm 2^{\circ}\text{C}$.

9.4.5 Cool in desiccator to balance temperature, and weigh.

9.4.6 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.

9.5 **Data Reduction and Reporting:**

9.5.1 Calculate TDS as follows:

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

Where: TDS = Total Dissolved Solids in mg/L

A = weight of dried residue + dish in mg after drying

B = weight of dish in mg

C = sample volume in mL

9.5.2 Reporting – Results will be reported to 2 significant figures for TDS less than 100 mg/L, and 3 significant figures for TDS greater than or equal to 100 mg/L. For rounding results, adhere to the following rules:

- a) If the number following those to be retained is less than 5, round down;
- b) If the number following those to be retained is greater than 5, round up; or



c) 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:

- 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 Dissolved Solids – Dried at 180°C, SM 2540 C (GEPA Log: CH-02-05) (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 C, Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998.

11.2 USEPA Region 9 Lab SOP #461, Analysis of Total Dissolved Solids By EPA Method 160.1, Revision 1, June 16, 1998.



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Attachment A: Chain of Custody Form



Chain of Custody Record

PROJECT NAME: _____ **ASSIGNED LAB ID NUMBER** _____

SAMPLERS _____ **# OF CONTAINERS** _____
 (Print name and sign)

STATION LOCATION _____

COMPOSITE _____
 GRAB _____

FIELD SAMPLE ID	DATE	TIME	COMPOSITE	GRAB	STATION LOCATION	ASSIGNED LAB ID NUMBER	# OF CONTAINERS	REMARKS

RELINQUISHED BY (Print name and sign)	DATE	TIME	RECEIVED BY (Print name and sign)
RELINQUISHED BY (Print name and sign)	DATE	TIME	RECEIVED BY (Print name and sign)
RELINQUISHED BY (Print name and sign)			RECEIVED FOR LAB by (Print name and sign)



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Attachment B: Analytical Results Report
(LIMS or Spreadsheet)

GUAM ENVIRONMENTAL PROTECTION AGENCY
EMAS Analytical Program

ANALYTICAL REPORT

Report: TDS-02347_02348
Parameter: Total Dissolved Solids
Method: SM 2540 C

Date of Report: 02/14/2012

Matrix: water

Analyst: E. Yanit *EY*

GEPA Lab ID	Field Sample ID	Date Collected	Date Analyzed	MDL	Result	Unit	Remarks
	Lab Blank		**	**	**	**	Note: ** Not analyzed
02347-015	AGMS	2/1/2011	**	**	**	**	for the parameter
02347-016	AGMD	2/1/2011	**	**	**	**	
02347-017	AGMT	2/1/2011	**	**	**	**	
02347-018	AGMP	2/1/2011	**	**	**	**	
02347-019	AGM11	2/1/2011	**	**	**	**	
02347-020	AGMB	2/1/2011	**	**	**	**	
02347-021	AGMFB	2/1/2011	**	**	**	**	
02347-022	ASM22	2/1/2011	**	**	**	**	
02347-023	ASM14	2/1/2011	**	**	**	**	
02347-024	PBM15	2/1/2011	**	**	**	**	
02347-025	PBM16	2/1/2011	**	**	**	**	
	Lab Blank		2/7/2011	10	<10	mg/L	Lab blank
02348-018	AGRD	2/2/2011	2/7/2011	10	1014	mg/L	
02348-019	AGRA-3	2/2/2011	2/7/2011	10	638	mg/L	
02348-020	AGRF-2	2/2/2011	2/7/2011	10	208	mg/L	
02348-021	ASRI-2	2/2/2011	2/7/2011	10	296	mg/L	
02348-022	ASRI-4	2/2/2011	2/7/2011	10	221	mg/L	

Peer Reviewed By: *EY*
Date Reviewed: 2/15/12

Approved By: *EY*
Date Approved: 2/22/12

GUAM ENVIRONMENTAL PROTECTION AGENCY
EMAS Analytical Program

ANALYTICAL REPORT

Report: TDS-02348_02350_02351
Parameter: Total Dissolved Solids
Method: SM 2540 C

Date of Report: 02/14/2012

Matrix: water

Analyst: E. Yanit *EY*

GEPA Lab ID	Field Sample ID	Date Collected	Date Analyzed	MDL	Result	Unit	Remarks
02348-023	ASRM	2/2/2011	2/7/2011	10	314	mg/L	
02348-024	APRM-1B	2/2/2011	2/7/2011	10	172	mg/L	
02348-024	APRM-1B - LD	2/2/2011	2/7/2011	10	163	mg/L	Lab duplicate
	Lab Blank		2/14/2011	10	<10	mg/L	Lab blank
02350-017	ATRN-2	2/8/2011	2/14/2011	10	260	mg/L	
02350-018	ATRO	2/8/2011	2/14/2011	10	269	mg/L	
02350-019	ATRF-3	2/8/2011	2/14/2011	10	217	mg/L	
02350-020	ART-2	2/8/2011	2/14/2011	10	1198	mg/L	
02350-021	ULRU-2	2/8/2011	2/14/2011	10	261	mg/L	
02350-022	MZRT-2	2/8/2011	2/14/2011	10	385	mg/L	
02350-023	MZRG-2	2/8/2011	2/14/2011	10	256	mg/L	
02350-019	ATRF-3 - LD	2/8/2011	2/14/2011	10	229	mg/L	Lab duplicate
02351-013	APM-18	2/9/2011	**	**	**	**	
02351-014	APMOB	2/9/2011	**	**	**	**	Note: ** Not analyzed
02351-015	APM-20	2/9/2011	**	**	**	**	for the parameter
02351-016	ATMN	2/9/2011	**	**	**	**	
02351-017	ATMTO	2/9/2011	**	**	**	**	
02351-018	ATMS	2/9/2011	**	**	**	**	

Peer Reviewed By: *EY*
 Date Reviewed: 2/15/12

Approved By: *EY*
 Date Approved: 2/13/12



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Attachment C: TDS Analytical Results Logbook page

GUAM ENVIRONMENTAL PROTECTION AGENCY - EMAS Analytical Program
Total Dissolved Solids (TDS) Dried at 180°C, SM 2540 C.

Date: _____

Analyst: _____

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



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Attachment 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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Attachment E: Balance Calibration Logbook page

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

GEPA Log: QC-10-02

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 $\pm 0.0005\text{g}$.
ACTION: If value is greater than criteria, inform supervisor.
NOTE: Class "S" weights equivalent to ASTM type 1 or 2.

