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SM2510B – Specific Conductance in Water

Access to this SOP shall be available within the laboratory for reference purposes; the official copy of this SOP resides on the official Georgia EPD website at https://epd.georgia.gov/about-us/epd-laboratory-operations. Printed copies of this SOP will contain a watermark indicating the copy is an uncontrolled copy.

1 Scope and Application

1.1. The Orion Star A212 Benchtop Conductivity Meter and the 4 –Electrode conductivity cell 013005MD with a nominal cell constant of 0.475 cm^{-1} are used to measure conductivity, which is the ability of an aqueous solution to carry an electric current. This ability depends on the presence of ions; on their total concentration, mobility, and valence; and on the temperature of measurement. The results are automatically corrected to 25° C.

1.2. Restricted Procedure

1.2.1. This procedure is restricted to use by an analyst experienced in the operation of an Orion Star A212 Benchtop Conductivity Meter and the 4 –Electrode conductivity cell 013005MD. Additionally, the analyst must complete the requirements of the GAEPD Initial Demonstration of Analyst Proficiency prior to the analysis of actual samples. Analysts are further warned that performance of this analysis involves the use of potentially hazardous chemicals; refer to the GAEPD Chemical Hygiene Plan for additional information regarding chemicals required by this method.

2 Definitions

2.1. Refer to Chapter 3 of the Georgia EPD Laboratory Quality Assurance Manual for Quality Control Definitions.

2.2. Primary Source (PS) – A standard that is used to make up the calibration points of a curve.

2.3. Second Source (SS) – A standard made from a manufacturer other than that of the primary source.

2.4. Initial Calibration Verification (ICV) – An ICV is a second source standard that is used to verify the correctness of the primary source calibration curve. The ICV is run at a level equal to that of a Laboratory Control Sample (LCS) or the midpoint on the calibration curve.

2.5. Continuing Calibration Check (CCC) or Continuing Calibration Verification (CCV) – A standard used to verify that the response of the instrument has not changed since initial calibration. The CCC is run at a level equal to that of a Laboratory Control Sample (LCS) or the midpoint on the calibration curve.

2.6. Calibration Blank (CB), Initial Calibration Verification Blank (ICB), Method Blank (MBLK) or Continuing Calibration Blank (CCB) – A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes.

2.7. LCS (Laboratory Control Sample) and LCSD (Laboratory Control Sample Duplicate) are prepared by spiking laboratory reagent water, Ottawa sand or air sampling device with the target analyte or compound. They are used to validate the analytical batch with respect to accuracy and precision.

3 Interferences

3.1. No significant interferences.

4 Safety

4.1. Refer to the EPD Laboratory Safety / Chemical Hygiene Plan & Fire Safety Plan online revision.

5 Apparatus and Equipment

5.1. Sample Container: Half-Gallon plastic container

5.2. Orion Star A212 Benchtop Conductivity Meter
4 –Electrode conductivity cell 013005MD with a nominal cell constant of 0.475 cm^{-1} and automatic temperature compensation that reads directly in µmho/cm and is automatically corrected to 25.0° C.

5.3. 50-150 ml glass beakers

5.4. Class A graduated cylinders, and pipettes

5.5. Balance: analytical, capable of accurately weighing to the nearest 0.1 mg

5.6. Thermometer and/or temperature sensor for automatic compensation

5.7. Lint free tissue

6 Reagents

6.1. Reagent Water:
Purified water which does not contain any measurable quantities of target analytes or interfering compounds for each compound of interest. (Deionized, HPLC, Milli-Q water or equivalent. Milli-Q water has a resistivity of 18.2 [MΩ·cm] @ 25° C and a TOC of 50 µg/L or less).

6.2. Potassium Chloride (KCl):

6.2.1 KCl must be dried in an oven set at 103-105 ° C for one hour before use. Cool in desiccator before use.

6.3 Calibration Standards:

6.3.1. 147 µmho/cm Standard:

6.3.1.1 Measure 74.56 mg of dried anhydrous KCl in a 1 L volumetric flask and dilute to volume using reagent water. Prepare every 3 months. This standard may also be purchased. If purchased the expiration date is the manufacturer's expiration date or 3 months from opening whichever is sooner.

6.3.2. 1410 µmho/cm Standard:

6.3.2.1. Measure 745.6 mg of dried anhydrous KCl in a 1 L volumetric flask and dilute to volume using reagent water. Prepare every 3 months. This standard may also be purchased. If purchased the expiration date is the manufacturer's expiration date or 3 months from opening whichever is sooner.

6.3.3. 12900 µmho/cm Standard:

6.3.3.1. Measure 7.456 g of dried anhydrous KCl in a 1 L volumetric flask and dilute to volume using reagent water. Prepare every 3 months. This standard may also be purchased. If purchased the expiration date is the manufacturer's expiration date or 3 months from opening whichever is sooner.

6.4. 100,000 µmho/cm High Calibration Check Standard:

6.4.1. Measure 57.7984 g of dried anhydrous KCl in a 1 L volumetric flask and dilute to volume using reagent water. Prepare every 3 months. This standard may also be purchased (Fisher PN# R5889300-500A or equivalent). If purchased the expiration date is the manufacturer's expiration date or 3 months from opening whichever is sooner.

6.5. 14,000 µmho/cm ICV Stock Standard Solution:

6.5.1. This standard is purchased (Ricca Chemical PN# R2246141-500A or equivalent). Standard is stable for 3 months. The expiration date is the manufacturer's expiration date or 3 months from opening whichever is sooner.

6.6. 420 umho/cm ICV Solution (LCS/LCSD):

6.6.1. Pipette 30 ml of ICV Stock Standard Solution (14,000 µmho/cm) into 1 L volumetric flask and dilute to volume. Prepare every 3 months.

6.6.2. To prepare from a second source of dried anhydrous KCl, measure 0.2221 g of dried anhydrous KCl(from different lot or manufacturer than used to make calibration standards) in a 1 L volumetric flask and dilute to volume using reagent water. Prepare every 3 months.

7 Sample Collection

7.1. Samples are collected in half-gallon plastic containers.

7.2. Samples should be cooled in ice as soon as possible after collection and stored at 0 – 6 °C (not frozen).

7.3. No chemical preservation is required.

7.4. Holding time is 28 days under refrigeration at 0 – 6 °C (not frozen).

8 Calibration

8.1. Calibration Standards

8.2. The calibration standards used are 147 µmho/cm, 1410 µmho/cm and 12900 µmho/cm.

8.3. Calibration Curve

8.3.1. The conductivity meter is calibrated daily.

8.4. Calibration Verification

8.4.1. The Orion Star A212 Benchtop conductivity meter's calibration is verified using three standards at the following concentrations: 147 µmho/cm, 1410 µmho/cm and 12900 µmho/cm. In addition, a 100,000 umho/cm standard is analyzed.

8.4.1.1. These four standards are analyzed with every batch.

8.4.1.2. Acceptance limits for recovery are 90-110%.

8.4.2. An Initial Calibration Verification (ICV) is analyzed after the calibration standards. The ICV is named as a Laboratory Control Standard (LCS) for LIMS tracking purposes. The ICV is prepared from a stock of a different source than the calibration standards.

8.4.3. The % Drift of the ICV from the true value must be within ±10%.

8.4.4. A Method Blank (MBLK) must also be analyzed after the calibration standards.

8.4.4.1. A method blank is analyzed for every batch.
8.4.4.2. The method blank must be less than the method RL or the run will have to be repeated.
8.4.5. A Continuing Calibration Check (CCC) and a Continuing Calibration Blank (CCB) must be analyzed every 10 samples and at the end of the sample run.
8.4.5.1. The CCB value must be less than the method RL or the samples associated with the out of control CCB will have to be reanalyzed.
8.4.5.2. The CCC acceptance limits for recovery are 90-110%.
8.4.5.3. If the CCC does not meet the acceptance criteria, all samples affected by the out of control CCC must be rerun.
8.4.5.4. The mid-range standard 1410 µmho/cm is used for the CCC.
8.4.6. *Determination of Cell Constant*:
8.4.6.1. The probe has a nominal cell constant of 0.475 cm^{-1}.
8.4.6.2. The cell constant will display after calibration.
8.4.6.3. Record the measured cell constant and the nominal cell constant in the maintenance logbook and on the batch sheet.
8.4.6.4. The % error of the measured cell constant must be within 10%.
8.4.7. A high calibration check standard (100,000 umhos/cm) is analyzed with every batch.
8.4.7.1. Acceptance limits for recovery are 90-110%.

9 Quality Control

9.1. Refer to Table 14.1 for Reporting Limits (RL's), Table 14.2 for Quality Control Acceptance Criteria, and Table 14.3 for Quality Control Procedures associated with this method, and Standard Operating Procedure for control charts and control limits (SOP reference 13.3).
9.2. Refer to GA EPD Laboratory SOP's – Initial Demonstration of Capability SOP 6-001, Rev. 3 or later, or Continuing Demonstration of Capability SOP 6-002, Rev. 2 or later SOPs for training criteria (SOP reference 13.2).
9.3. The default control limits for SM2510B are 90-110% recovery for Conductivity for LCS recoveries. The EPD Laboratory applies LCS recovery limits to LCSDs. Note, unless specified by method, the EPD Laboratory does not validate batch quality based on LCSD recoveries. Note: The LCS for this method is also used for the ICV so both the LCS and LCSD are second source standards.
9.4. By default, the EPD Laboratory sets LCS/LCSD precision limits for this method to be 0-15% RPD.
9.5. LCS/LCSD recovery and precision limits are static (i.e., not adjusted by control chart results).
9.6. 10% of all routine samples must be analyzed in duplicate.
9.7. Batch samples in groups of 20 or less. For each batch, analyze a Sample Duplicate for a minimum of 10% of routine samples.

9.7.1. For batches of 1 to 10 routine samples, one Sample/Sample Duplicate pair must be analyzed.

9.7.2. For batches of 11 to 20 routine samples, two Sample/Sample Duplicate pairs must be analyzed using different samples for each pair.

9.8. By default, the EPD Laboratory sets default sample/duplicate sample precision control limits to be 0 – 15% RPD. These limits are static.

9.9. The control limits are static and appear in Table 14.2. Control charts must still be performed for trend monitoring.

9.10. Because the limits for this method are static, no Appendix A will be generated.

9.11. The 4 –Electrode conductivity cell 013005MD temperature compensation must be verified at least quarterly using a NIST traceable thermometer. The allowable temperature deviation of the conductivity cell temperature sensor compared to a NIST traceable thermometer is ≤ ± 2° C. This verification must be recorded in the Conductivity maintenance log. If temperature is outside temperature requirement, replace probe.

9.11.1. Record manufacturer, serial number, and model number in the maintenance log when conductivity cell is replaced.

9.12. Conductivity analyses are exempt from the requirement to perform MDL studies.

9.13. When analyzing conductivity on a microbiology monthly DI water QC sample, report results using 3 significant figures. Notify the Microbiology Lab if sample value is ≥2.0 µmho/cm. Service may need to be performed on the laboratory's DI water system.

10 Procedure

10.1. Remove the sample bottles, standards, and reagents from cold storage, and allow equilibration to room temperature prior to sample preparation and/or analysis.

10.2. Pour up approximately 100 ml of fresh 147 µmho/cm, 1410 µmho/cm and 12900 µmho/cm conductivity standards into 3 separate 150 mL beakers.

10.3. Press the POWER button on the meter and make sure the middle line indicates the µS/cm icon.

10.3.1. Allow meter to warm up for 15 minutes.

10.4. Make sure the temperature reading changes when placing the probe in a sample or standard to ensure that the automatic temperature compensation is working.

10.5. The temperature sensor must be checked on a quarterly basis (See 9.11.).

10.6. Calibration Procedure:

10.6.1. Press "cal"tab using the F1 function key, rinse probe with reagent water and blot dry with a lint free tissue.

10.7. Place conductivity probe in the 150 mL beaker containing the147 µmho/cm standard. Press start.

10.8. Stir gently using the probe and make sure there is no air in probe.

10.9. Wait for stable reading. Once the reading is stable either accept the value by pressing accept or edit the value by pressing edit and entering the correct number using the keypad on screen. Press Next.

10.10. Rinse probe with reagent water and blot dry with a lint free tissue.

10.11. Place probe in 150 mL beaker filled with the 1410 µmho/cm standard. Press start. Repeat Step 10.9. Press Next.

10.12. Rinse probe with reagent water and blot dry with a lint free tissue.

10.13. Place probe in 150 mL beaker filled with the 12900 µmho/cm standard. Press Start. Repeat Step 10.9. Change value to 12.900 mS/cm which is equivalent to 12900 µS/cm.

10.14. Press "Cal Done" to end calibration.

10.15. The cell constant will be displayed. Record the measured cell constant and the nominal cell 0.475 cm^{-1} found in the probe user guidebook into the maintenance logbook and batch sheet. The measured value should be within 10% of 0.475 cm^{-1} using equation 11.4. Press "Meas" to exit calibration.

10.16. The meter is now in the measure mode and samples can now be analyzed.

10.17. Rinse probe with reagent water and blot dry with lint free tissue.

10.18. Perform a backlog of samples and batch in groups of 20 or less. For each batch, analyze one or two Sample Duplicate pairs for a minimum of 10% of all routine samples.

10.19. The CCC is analyzed after every ten samples and at the end of the sample run.

10.19.1. The CCC must be poured into a ½ gallon sample collection container before it is poured up for measurement. Record lot # of bottle used.

10.20. The CCB is analyzed after every ten samples and at the end of the sample run.

10.20.1. The CCB must be poured into a ½ gallon sample collection container before it is poured up for measurement. Record lot # of bottle used.

10.21. The High Level Calibration Check (100,000 µmho/cm) is to be analyzed once per batch.

10.22. The LCS/LCSD for this method is the second source standard (ICV) so it is equivalent to the ICV.

10.22.1. The 420 µmho/cm ICV Solution is used as the LCS/LCSD for this analysis for tracking purposes.

10.22.2. The LCS and LCSD is analyzed once per batch.

10.23. Pour 100 mL of sample into a 150 mL beaker.

10.24. Immerse the probe into the sample and stir gently using probe.

10.25. Check that the temperature reading is changing and measure the conductivity when it switches from stabilizing to ready. **Note that if value is above a certain threshold, the reading will change to mS/cm. 1000 umho=1 mS/cm.**

10.26. Make sure to rinse the probe with reagent water and blot the end of the probe with a lint free tissue between samples.

11 Calculations

11.1 Relative Percent Difference (%RPD or RPD):

$$\%\text{RPD} = \frac{|X_1 - X_2|}{\frac{(X_1 + X_2)}{2}} * 100$$

11.2 Percent Drift, %Drift:

$$\%\text{Drift} = \frac{(\text{Concentration}_{\text{Calculated}} - \text{Concentration}_{\text{Expected}})}{\text{Concentration}_{\text{Expected}}} * 100$$

11.2.1 Where:

$\text{Concentration}_{\text{Calculated}}$ = Concentration calculated from result

$\text{Concentration}_{\text{Expected}}$ = Theoretical concentration of the standard

11.3 Percent Relative Standard Deviation (%RSD):

$$\%\text{RSD} = \frac{\sigma_{n-1}}{\overline{X}} * 100$$

11.3.1 Where:

σ_{n-1} = Sample Standard Deviation

$\overline{X}$ = Mean of the values

11.4 % ERROR

$$\%\text{Error} = \frac{|\text{N.V.} - X_1|}{\text{T.V.}} * 100$$

11.4.1 Where:

N.V. = nominal cell value

X_1 = measured value

12 Waste Management

12.1. Refer to GA EPD Laboratory SOP- EPD Laboratory Waste Management Standard Operating Procedures, reference 13.4.

12.2. Dispose of samples down laboratory sink drain.

13 References

13.1. Standard Methods Online - SM2510B – Conductivity, 1997: Editorial revision 2011.

13.2. GA EPD Laboratory SOP's – Initial Demonstration of Capability SOP 6-001, online revision or later or Continuing Demonstration of Capability SOP 6-002, online revision.

13.3. GA EPD Laboratory SOP-EPD Laboratory Procedures for Control Charts and Control Limits SOP, SOP 6-025, online revision.

13.4. GA EPD Laboratory SOP-EPD Laboratory Waste Management SOP, SOP 6-015, online revision.

13.5. GA EPD Laboratory – Quality Assurance Manual, online revision.

13.6. GA EPD Laboratory SOP – Laboratory Safety/Chemical Hygiene Plan & Fire Safety Plan, online revision.

14 Reporting Limits (RLs), Precision and Accuracy Criteria, and Quality Control Approach

Table 14.1 RLs for Method SM 2510B

Parameter/Method	Analyte	Matrix (aqueous) RL	Unit
SM 2510B	Conductivity	10	µmho/cm

Table 14.2 Acceptance Criteria for Method SM 2510B

QC Type	Analyte	Accuracy (%R) LCL	UCL	Precision (%RPD)
(LCS)[1]	Conductivity (Specific Conductance)	90	110	15
Sample Duplicate	Conductivity (Specific Conductance)	--		15

[1] *A second source standard is used for the LCS which also satisfies the requirement of an ICV for this analysis therefore control limits are static.*

Table 14.3 Summary of Calibration and QC Procedures for Method SM 2510B

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
SM 2510B	Conductivity	Initial calibration verification using three standards	Prior to every batch	Value must be within 10% of the true value.	Correct problem then repeat initial calibration verification	
		Initial Demonstration of Capability (IDC): Demonstrate ability to generate acceptable accuracy and precision using four analysis of a QC check sample and a MBLK. In addition, the analyst must also prepare one standard.	Once per analyst	QC Acceptance Criteria Table 14.2 and Initial Demonstration SOP (SOP reference 13.2)	Recalculate results: locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Continuing Demonstration: Demonstrate ability to generate acceptable accuracy and precision using a variety of analysis options of a QC sample(s)	Required every six months after IDC for each analyst	QC Acceptance Criteria Table 14.2 and Continuing Demonstration SOP (SOP reference 13.2)	Recalculate results: locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Laboratory Control Sample (LCS/ LCSD) (420 µmho/cm) LCS for this method is the second source standard so it is equivalent to the ICV.	One LCS/LCSD per analytical batch	QC Acceptance Criteria Table 14.2	Correct problem then reanalyze the LCS/LCSD and all samples in the affected batch	If unable to reanalyze, flag with a "J"
		High Level Calibration Check (100,000 µmho/cm)	Once per batch	± 10% of true value	Correct problem than reanalyze High Level Calibration Check and all samples in affected batch	
		Sample Duplicate	10% of samples	QC Acceptance Criteria Table 14.2	Evaluate out of control event and reanalyze all samples in the affected batch.	
		Method Blank (MBLK)	Once per batch	Value must be < RL.	Correct problem then reanalyze blank and all samples processed with the contaminated blank	

Table 14.3 Summary of Calibration and QC Procedures for Method SM 2510B						
Method	**Applicable Parameter**	**QC Check**	**Minimum Frequency**	**Acceptance criteria**	**Corrective Action**	**Flagging Criteria**
SM2510B	Conductivity	Continuing Calibration Check (CCC) (1410 µmho/cm) CCC's are used for LCS/LCSD	After every 10 samples and at the end of the run.	Result must be within 10% of expected value.	Correct problem and reanalyze blank and all samples processed with the contaminated blank.	
		Continuing Calibration Blank (CCB)	After every 10 samples and at the end of the run.	Value must be < RL.	Correct problem and reanalyze blank and all samples processed with the contaminated blank.	
		Conductivity of Microbiology Water (Suitability Testing)	Once per month	Value must be < 2.0 umho/cm *report result using 3 significant figures and send report to Microbiology Lab.	Notify Microbiology Lab and service deionized water system	
		Temperature Compensation Probe check	At least Quarterly	≤ ± 2° C	Correct problem or replace probe	

Updates to SOP
Section 2
Section 13
Table 14.3