

NORTH CAROLINA WASTEWATER/GROUNDWATER LABORATORY CERTIFICATION BRANCH

APPROVED PROCEDURE FOR THE ANALYSIS OF SALINITY (Electrical Conductivity Method)

This document provides an approved procedure for the analysis of Salinity for compliance monitoring per 15A NCAC 02H .0805 (a) (7) and (g) (4).

Holding Time:

- Samples must be analyzed within 28 days of collection based on the holding time of conductivity in 40 CFR Part 136.3 Table II per EPA Region 4.
- Samples not analyzed within 15 minutes of collection must be transported on ice and stored above freezing and $\leq 6^{\circ}\text{C}$, based on the preservation requirements of conductivity in 40 CFR Part 136.3 Table II per EPA Region 4.

General Information:

- Sample duplicates are not a required quality control element for Field parameters.
- Sample results are to be reported in units of parts per thousand (ppt) which is equivalent to Practical Salinity Units (PSU).
- The sample temperature at the time of analysis must be reported in the comment section of the Discharge Monitoring Report (DMR).
- Salinity samples must not be diluted.
- Salinity may be measured directly if the meter and probe allow. If not, the conductivity may be measured and converted to salinity. This Approved Procedure does not address converting conductivity to salinity. The formula may be found in Standard Methods 2520 B-2011.
- Thoroughly rinse probe with one or more portions of sample prior to sample measurement.
- Keep the probe clean when not in use.
- When performing multiple sample analyses, a post-analysis calibration verification must be analyzed at the end of the run. It is recommended that a mid-day or a mid-batch calibration verification be performed when samples are analyzed over an extended period of time. The value obtained for the post-analysis calibration verification check-standard must read within $\pm 10\%$ of the standard's true value. If the obtained value is outside of the $\pm 10\%$ range, corrective action must be taken. If recalibration is necessary, all samples analyzed since the last acceptable calibration verification must be reanalyzed, if possible. If samples cannot be reanalyzed, the data must be qualified.

Operational Range Verification

Before using this method or a new instrument, determine its operational range (upper and lower limits), or at least verify the intended range of use. For each analyte, use standard concentrations that provide increasing instrument or other test response. The minimum reporting level (MRL) is set at or above the lowest standard used in the analysis. Quantitation at the MRL must be verified initially and at least quarterly (preferably daily) by analyzing a QC sample. The standard must read within $\pm 10\%$ of the true value.

Standards:

Potassium Chloride (KCl) conductivity standards may be purchased or prepared according to Table 2510:I of Standard Methods, 2510 A-2011. In addition to the standards listed below, standard seawater, commercially available with a salinity of 35 at 15°C may be purchased. Alternatively, a standard may be prepared at this concentration by dissolving 32.4356 g of KCl in 1 L of deionized water.

A portion of the standard should not be used for more than one calibration. Discard any used standard portions.

Note: The preparation of the standards in Table 2510:I indicates that the relationship between mass of KCl used and standard concentration is not linear; therefore, standards may not be diluted, but must be prepared individually.

Conductivity and Salinity of Potassium Chloride

KCl Molarity (M)	KCl Wt.* (grams)	Conductivity at 25°C ($\mu\text{mho/cm}$)	Salinity at 15°C (ppt)	Salinity at 20°C (ppt)	Salinity at 25°C (ppt)
0.0001	0.00745	14.9	0.01	0.01	0.01
0.0005	0.03728	73.9	0.05	0.04	0.04
0.001	0.07455	146.9	0.09	0.08	0.07

Table continued next page

Conductivity and Salinity of Potassium Chloride (continued)					
KCl Molarity (M)	KCl Wt.* (grams)	Conductivity at 25°C (µmho/cm)	Salinity at 15°C (ppt)	Salinity at 20°C (ppt)	Salinity at 25°C (ppt)
0.005	0.37275	717.5	0.44	0.39	0.35
0.01	0.74550	1,412	0.89	0.79	0.71
0.02	1.49100	2,765	1.8	1.6	1.4
0.05	3.72750	6,667	4.6	4.1	3.6
0.10	7.45500	12,890	9.4	8.3	7.4
0.20	14.9100	24,800	19.1	16.9	15.1
0.44	32.4356	42,900	35	30.9	27.6

*Diluted to 1 L

Calibration:

1. Instruments are to be calibrated according to the manufacturer's calibration procedure prior to analysis of samples each day compliance monitoring is performed. For most meters, this is a one-standard calibration.
2. Thoroughly rinse the probe with one or more portions (three recommended) of the standards prior to calibration and calibration verification.
3. Analyze and document a second-source calibration verification check-standard prior to compliance sample analysis. It is recommended that this standard value approximate the expected range of sample values measured. If the standard concentration is at the reporting limit, this would satisfy the requirement to verify the reporting limit quarterly.
4. The value obtained for the calibration verification check-standard must read within $\pm 10\%$ of the true value of the calibration verification check standard. If the obtained value is outside of the $\pm 10\%$ range, corrective action must be taken.

Analysis:

1. Thoroughly rinse probe with one or more portions of sample.
2. Measure salinity of sample according to salinity meter manufacturer's instructions.
3. Document salinity and temperature results.

Documentation:

The following must be documented in indelible ink whenever sample analysis is performed:

1. Date and time of sample collection. If Salinity measurement is made *in situ*, it must be notated.
2. Date and time of sample analysis to verify the 28-day holding time is met
3. Facility name or permit number, and sample site (ID or location)
4. Collector's/analyst's name or initials
5. True value of the standard used for calibration
6. True value of the calibration verification check-standard
7. Value obtained for the check-standard
8. True value and value obtained for the post-analysis calibration verification(s), when applicable
9. Temperature (with units) of standards
10. Quality control assessments
11. Indication of when the post-analysis calibration verification was performed (e.g., time of analysis, end-of-day analysis, etc.)
12. The final value to be reported
13. Units of measure (ppt)
14. Temperature (with units) of samples at the time of measurement
15. Traceability for chemicals, reagents, standards and consumables
16. Unique instrument identification (serial number preferred)
17. Parameter analyzed
18. Method reference
19. Data qualifiers, when necessary

Refer to <https://www.deq.nc.gov/about/divisions/water-resources/water-sciences/chemistry-laboratory/laboratory-certification-branch/field-approved-procedures-and-technical-assistance> for additional resources.

Ref: Standard Methods 2520 B-2011 and Standard Methods 2510 B-2011