AUTOMATED DETERMINATION OF CONDUCTIVITY USING THE METROHM OMNIS INSTRUMENTATION

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1. SCOPE AND APPLICATION

1.1. This SOP is applicable for the determination of the specific conductivity in micromhos per centimeter (µmhos/cm) of aqueous samples using an automatic PC-Titrate instrument. The method detection limit (MDL) is 3.5 µmhos/cm and the practical quantitation limit (PQL) is 8.0 µmhos/cm. Samples can be analyzed from the MDL to 199.9 mmhos/cm (depending on the specifications of the conductivity probe used). This method is applicable to drinking, surface, ground, and saline waters as well as aqueous wastes.

1.2. The tests covered by this SOP are W-COND and W-COND-F.

2. SUMMARY OF THE METHOD

2.1. This SOP is based on EPA method 120.1 (Rev. 1982).

2.2. The sample conductivity is measured electrometrically using the Metrohm OMNIS instrument. The results are automatically corrected to 25 °C.

2.3. There are no real interferences with conductivity. If readings become erratic or probe response is sluggish, the probe may need re-platinizing. See Section 10 for electrode maintenance.

3. EQUIPMENT AND SUPPLIES

3.1. Instrumentation: Metrohm OMNIS Robot.

3.2. Equipment:

Equipment	Manufacturer/Vendor[1]	Model/Catalog Number
Conductivity Electrode	Metrohm	Conductivity measuring cell – 6.0915.130
Analytical Balance	Mettler Toledo	MS204TS/0
Volumetric Flask, Class A, Assorted	Fisher (SR)	10-212B; 10-210-5D; 10-212E; 10-212-5L; 10-208M
Graduated Cylinder, Class A, Assorted	Fisher (SR)	08-553A; 08-548D
Glass Beaker, Assorted	Fisher (SR)	02-540E; 02-540G; 02-540K; 02-555-20E
Or Equivalent		
[1]**Note:** (SR) designates equipment is typically ordered through the in-house stockroom		

3.3. Consumables and frequently replaced supplies:

Supplies/Consumables	Vendor[1]	Catalog Number
Lithium Grease	Fisher	NC0484446
Centrifuge Tubes, Polypropylene, 50 mL	Fisher (SR)	05-538-68, 05-53855A
Sample Beakers, Polypropylene, Assorted	Fisher (SR)	08-732-119; 08-732-120; 08-732-122
Weigh Dish, Hexagonal, Assorted	Fisher (SR)	02-202-102; 02-202-100
Weigh Dish, Aluminum, 110 mm	Fisher (SR)	08-732-100
Pipet, Transfer, Polyethylene, 7mL	Fisher (SR)	13-711-7M
Parafilm	Fisher (SR)	13-374-12

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Or Equivalent
[1]**Note:** (SR) designates supply is typically ordered through the in-house stockroom

4. REAGENTS AND STANDARDS (SEE DEP SOPS NU-041 and NU-042)

Note: Before preparing reagents or standards, or running samples, please read the safety notes in the safety/hazardous waste section of this SOP and the MSDS for the chemicals used in this analysis.

Note: Standard preparations must be documented in the Laboratory Information Management System (LIMS) 'Standard Preparation Tracker' module. Unique ID numbers for each standard are generated in the module and documented in the reagent and standard logsheet (NU-RGTSTD) submitted with each analysis folder. The reagent and standard logsheet (NU-RGTSTD) template is located at \\floridadep\data\labs\Lab3\Chem_Data\Inorganic\Nutrients\RunData\MetaData\Auto-T\Forms\. Follow preparation and labelling instructions in DEP SOP NU-042. A Certificate of Analysis (CoA) for each purchased reagent and standard is maintained in the CertificatesofAnalysis subfolder within the Auto-T folder on the Nutrients Drive. All containers holding standards or reagents must be labeled with their unique ID and expiration date.

Note: The volumes prepared for the standards may be altered by the analyst as necessary. The final concentrations of the constituents of a standard must not be changed.

4.1. Chemicals:

	Chemical	Alias	Manufacturer[1]	Catalog Number	Purity	Storage
4.1.1.	Potassium Chloride	KCl	Fisher Chemical	P217-500	ACS, ≥99%	Room Temp
4.1.2.	Potassium Chloride Reference Solution, 1408.8 µmhos/cm at 25°C	Conductivity Calibration Standard	Ricca	589532	See CoA	Fridge after opening
4.1.3.	Minerals QC CRM	ALTQC	NSI Lab Solutions	QCI-136	See CoA	Fridge after opening
4.1.4.	DI Water	H_2O	In house	N/A	N/A	N/A
Or Equivalent [1]**Note:** (SR) designates chemical is typically ordered through the in-house stockroom						

Deionized water is replaced daily. All other purchased chemicals are given the expiration date from the CoA and/or chemical container. If there is not an expiration date listed, the expiration date is designated by following the protocol in SOPs NU-041 and NU-042.

4.2. KCl 1 M stock standard (1M COND) –

KCl (4.1.1.)	74.55 g
DI water (4.1.4.)	up to 1000 mL

Place 74.55 g dried KCl (two hours at 105 °C) in a 1 L volumetric flask and dilute to the mark with deionized (DI) water.

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Store in a refrigerator. Holding time, 180 days.

4.3. KCl 0.01 M stock standard –

KCl (4.1.1.)	0.7455 g
DI water (4.1.4.)	up to 1000 mL

Place 0.7455 g dried KCl (two hours at 105 °C) in a 1 L volumetric flask and dilute to the mark with DI water.

Store in a refrigerator. Holding time, 180 days.

4.4. Working standards:

To a 1 L volumetric flask containing approx. 400 mL DI water add the amount of 0.01 M KCl stock standard (4.3.) indicated in the table. Bring to volume with DI water.

Store in a refrigerator. Holding time, 60 days.

Standard Concentration	QC Standard, Alias	Final Volume (mL)	Vol of 0.01 M KCl standard added (mL)
0.001 M KCl (146.9 µmhos/cm)	LFB_CHCK2, CHCK2	1000	100
0.0005 M KCl (73.5 µmhos/cm)	LFB_CHCK4, CHCK4	1000	50

4.5. Conductivity Calibration Standard (4.1.2.) is used for conductivity electrode calibration at the beginning of every run.

4.6. A certified quality control (QC) sample (ALTQC) (4.1.3.). Prepared following the supplier's instructions, if needed. Standard can be stored at room temp, but is generally stored in the refrigerator. The concentration is provided by the manufacturer. There is not a recommended holding time by the manufacturer, but standard is given a 28-day holding time upon opening.

5. SAMPLE HANDLING AND PRESERVATION

5.1. Samples are stored at ≤ 6°C (not frozen) and must be analyzed within 28 days of collection to avoid qualification.

6. SAMPLE ANALYSIS/QUANTITATION (SEE DEP SOP NU-033)

Note: Allow the samples to come to room temperature prior to measuring the conductivity.

6.1. General operation of electrode – The electrode is stored dry automatically and must be conditioned prior to use by running at least one rinse cup prior to the start of analysis. Electrode is fragile; use caution when moving.

6.2. Meter Calibration – Calibrate the meter using the conductivity calibration standard (4.5.).

6.3. Start-up of the Metrohm OMNIS Software – Choose the appropriate method (conductivity alone or in combination with other tests). Set up the run sequence corresponding to the timetable of Metrohm OMNIS software.

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6.4. Loading samples – Pour 60 mL of sample or standard into a glass sample tube. Load the sample tubes onto the autosampler in the sequence determined in section 6.4. It is recommended to run a tap water sample at the very beginning as a rinse to clean up the system and condition the probe.

Example Run Sequence:

Rinse – Tap water
Conductivity Calibration Standard (4.5.)
MDL (blank – DI water)
CHCK4 (4.4)
ALTQC (4.6)
1M COND (4.2.)
Conductivity Calibration Standard (COND CAL) (4.5.)
Blank – DI water
MBLK – Method Blank: DI water (one per batch)
LFB_CHCK4 or **LFB_CHCK2**
Batched samples: one sample run in duplicate (sample ID_D) for each batch of max. 20 samples.
CHCK2 – alternate with CHCK4 every 20 cups
Blank – DI water

6.6. Click on the START button to start the run.

7. CALCULATIONS

7.1. The specific conductance is reported in micromhos per centimeter (µmhos/cm) at 25 °C. The conductivity meter automatically carries out the temperature compensation. The reference temperature is preset to 25 °C. The temperature coefficient is set to 2.00%.

7.2. For general calculations (including relative percent difference (RPD)), refer to the FDEP Quality Manual Section 5.9.

8. QUALITY CONTROL/DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES (SEE DEP SOP NU-038 AND NU-043)

8.1. The conductivity meter is calibrated at the start of every analytical run. The calibration is acceptable if the initial sequence of check standards, as described in 8.2, show recoveries within the specified acceptance limits.

8.2. A blank, a 111,900 µmhos/cm high calibration check standard (1M COND, 4.2), a 73.9 µmhos/cm continuing calibration verification (CCV) standard (CHCK4, 4.4.), and a QC standard (ALTQC, 4.6.) must be analyzed prior to running samples. Acceptance limits for the recovery of the blank are < 3.5 µmhos/cm and > -3.5 µmhos/cm. Acceptance limits for the recovery of the high check standard and the CCV standard are 85 – 115%. Acceptance limits for the recovery of the QC standard must be within the manufacturer's certified limits but not exceed 85 – 115%.

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8.3. Analyze a blank and a CCV (CHCK2 or CHCK4) standard every 20 cups tested and at the end of the analyses. Acceptance limits for the recovery of the blank is < 3.5 µmhos/cm and > -3.5 µmhos/cm, and 85 – 115% for CCVs.

8.4. A method blank (MBLK) and LFB (LFB_CHCK2 or LFB_CHCK4) are also run with every batch (20 samples or less). Acceptance limits for the recovery of the MBLK are < 3.5 µmhos/cm and > -3.5 µmhos/cm, and 90 – 110% for the LFB.

8.5. For every analytical batch of 20 or less samples, a duplicate sample must be analyzed. The RPD must be less than 20%.

8.6. Sample results are compared to the sample's field conductivity shown on the backlog. If the RPD between the lab and field results is not less than 20%, the results must be confirmed.

8.7. For general method performance and acceptance criteria for QC measures and corrective action for out-of-control data and contingencies for handling out-of-control or unacceptable data, refer to DEP SOPs NU-038 and NU-043.

8.8. Calibration – The calibration standard (4.5.) is the Conductivity Calibration Standard (approx. 1409 µmhos/cm at 25 °C) or equivalent. Every day samples are analyzed, a new calibration is performed at the beginning of the analysis.

8.9. The relative error of the calibration is evaluated by analyzing the low check (73.9 µmhos/cm, CHCK4) and the Conductivity Calibration Standard (1408.8 µmhos/cm, COND CAL) following the calibration, prior to samples being analyzed. Acceptance limits for recovery are 85 – 115%.

8.10. Annual MDL verification is performed using the LIMS statistics tracker module (see DEP SOP NU-043). Method blank (MBLK) data is collected and utilized for the ongoing MDL verification.

9. DATA ARCHIVAL

9.1. All analytical results are automatically saved in the database of the instrument software.

9.2. The raw data is archived in the export file used for the LIMS QCM upload as well as the instrument software. A report folder is created for each analytical run. A folder may contain more than one analysis (e.g. Alkalinity, and/or Conductivity) in it if the analyses are part of the same analytical run. The folder is labeled with the test (e.g., ALK and/or W-COND), filename/s (YYMMDDN-O#), and initials of the analyst.

9.3. The export file is used to create a general results report. Follow these steps to create the general result report:

7.3.1. Create a delimited file from the instrument CSV. Open excel, go to file – open – browse – locate the run files folder for that year – set the file type to "All Files" – select the run file. A dialogue box should appear, select "Delimited" – Next – Uncheck "Tab" – Check "Semicolon" – Finish.

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7.3.2. Format the delimited file as a table. Use “Ctrl+A” to select all the data in the file, go to the home tab in Excel and select “Format as Table”, ensure the proper data is selected and press “OK”.

7.3.3. Filter the table to show the data you need. Select the filter drop-down for the column header “Subsample name” and deselect “Subsample name”.

7.3.4. In a new excel sheet, open the form NU-AUTOTSHEET-1. Record the File Name and copy and paste the results from the delimited table into the appropriate form columns. Print the form as a PDF. Sign the form in Adobe using a certificate.

9.4. Each report folder should contain the following items – general results report, calibration report(s), running list report (bench sheet), reagent and standard logsheet(s), and a copy of the data and QC assessments uploaded from QC Manager to LIMS at the time of test authorization.

9.5. The report folders are on file with the nutrients and general chemistry supervisory personnel. Periodically, the files are moved to the State Records Center for long time storage according to DEP SOP LB-006.

9.6. The analytical file can be authorized online if necessary by scanning the entire folder into the correct year on the Nutrients drive at \\floridadep\data\labs\Lab3\Chem_Data\Inorganic\Nutrients\RunData\AnalysisFolders\Auto-T\. Once the authorizer has made comments and digitally signed all items of the pdf after reviewal, the QC report from QCM is saved as “YYMMDDN-A#_Authorization” and digitally signed as well. The signed analytical file and QC report is saved into a folder.

9.7. Method validation data is on file with the supervisor. Digital copy is also stored on the network, under an appropriate sub-folder in \\floridadep\data\labs\Lab5\Chemistry\Inorganic\Nutrient\Validations\Metrohm.

10. MAINTENANCE OF ELECTRODE

10.1. Clean a dirty electrode with detergent and/or dilute nitric acid (1%) by dipping the cell into cleaning solution and agitating for two to three seconds. Grease, oil, fingerprints and other contaminants on the sensing elements can cause erroneous measurements and erratic responses. Refer to electrode instructions for further information on cleaning and re-platinizing conductivity cells.

10.2. Refer to DEP SOP NU-060 Section 5 for other maintenance procedures.

11. SAFETY AND HAZARDOUS WASTE MANAGEMENT/POLLUTION PREVENTION

11.1. Review the laboratory Health and Safety Plan (accessible at https://floridadep.sharepoint.com/dear/SitePages/Lab%20Health%20and%20Safety.aspx).

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11.2. Safety glasses, gloves and lab coats should be worn at all times whilst handling samples and reagents.

11.3. Read the MSDS information for safe reagent and sample handling.

11.4. Dispose of excess samples by flushing them down the sink with plenty of water.

12. REFERENCES/DEFINITIONS

For definitions see section 3 of the FDEP Quality Manual. All abbreviations used throughout the document are defined at least once in the document usually at the first time of use.

12.1. EPA Method 120.1, (Rev. 1982).

12.2. Metrohm Conductivity 2020 Monograph: Conductometry – Conductivity Measurement.

12.3. DEP SOP LB-006, Standard Operating Procedure for Records Maintenance and Storage.

12.4. DEP SOP NU-033, Summary of Analysis Procedures for the General Chemistry and Nutrients Workgroups.

12.5. DEP SOP NU-038, Nutrients and General Chemistry: Quality Assurance and Quality Control.

12.6. DEP SOP NU-041, Reagent Preparation Procedure.

12.7. DEP SOP NU-042, Standard Preparation Procedure.

12.8. DEP SOP NU-043, Nutrients and General Chemistry Quality Assurance and Data Authorization Guidelines.

12.9. OMNIS Software 4.4.1.

12.10. Quality Manual, Bureau of Laboratories, FDEP.

12.11. Laboratory Health and Safety Plan.

12.12. Metrohm OMNIS Instrument Operations Manual.

Appendix of Significant Changes

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