AUTOMATED DETERMINATION OF CONDUCTIVITY USING THE PC-TITRATE INSTRUMENTATION

1. SCOPE AND APPLICATION

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 2.0 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. 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 PC-Titrate instrument. The results are automatically corrected to 25 °C with a preset temperature coefficient of 1.91 %.

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:

Instrument Unique ID	Component	Manufacturer	Model/Catalog Number
PC-TITRATE1	Autosampler	Gilson	AutoMax 197, PC-1000-680
	Burette Module	ManTech	Burivar I/2, PC-1000-1040
	Pump Module	ManTech	TitraRinse/A Pump, PC-1000-408
	Titration Module	ManTech	TitraSip Titration, PC-1300-475
	Titration Module	ManTech	TitraSip Titration, PC-1300-475
	Interface Module	ManTech	Interface Module, PC-1000-102/4
	Conductivity Meter	Jenway	4510 Conductivity Meter, PCA-2000-1015
	Software	ManTech	PC-TITRATE V3
PC-TITRATE2	Autosampler	Gilson	Mega 215 Autosampler, PC-1000-680
	Burette Module	ManTech	Burivar I/2, PC-1104-00
	Pump Module	ManTech	TitraRinse/A Pump, PC-1000-408
	Titration Module	ManTech	TitraSip Titration, PC1300-475
	Interface Module	ManTech	Interface Module, PC-1000-102/4
	Conductivity Meter	Jenway	4510 Conductivity Meter, PCM-1000-991
	Software	ManTech	PC-TITRATE V3

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3.2 Equipment:

Equipment	Manufacturer/Vendor[1]	Model/Catalog Number
Conductivity Electrode	ManTech	PC-96-CT1002
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
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 \\FLDEP1\DEAR\LABS\Lab3\Chem_Data\Inorganic\Nutrients\Reagent_Prep\PCTitrate. Follow preparation and labelling instructions in DEP SOP NU-042. A Certificate of Analysis for each purchased reagent is maintained in the Certificate of Analysis folder within the REAGENT_PREP folder for PC-Titrate analyses on the Nutrients drive. Certificates of Analysis for purchased standards are maintained in the Standards subfolder within the Certificate of Analysis folder for PC-Titrate analyses 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.

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4.1. Chemicals:

	Chemical	Alias	Manufacturer[1]	Catalog Number	Purity	Expiration[2]	Storage
4.1.1.	Potassium Chloride	KCl	Fisher Chemical	P217-500	ACS, ≥99%	5 years (*)	Room Temp
4.1.2.	Potassium Chloride Reference Solution, 1408.8 umho/cm at 25°C	Conductivity Calibration Standard	Ricca	589532	See COA	See COA	Room Temp
4.1.3.	QC Mineral Standard	ALTQC	Inorganic Ventures	QCP-MIN-500	See COA	See COA	Fridge after opening
4.1.4.	DI Water	H_2O	In house	N/A	N/A	Daily	N/A
Or Equivalent [1]**Note:** (SR) designates chemical is typically ordered through the in-house stockroom [2]**Note:** (*) designates manufacturer's suggested retest date. No expiration date given.							

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

KCl (4.1.1.)	74.99 g
DI water (4.1.4.)	up to 1000 mL

Place 74.99 g dried KCl (two hours at 105 °C) in a 1 L volumetric flask and dilute to the mark with deionized (DI) water.

Store in a refrigerator. Holding time, 180 days.

4.3. KCl 0.01 M stock standard –

KCl (4.1.1.)	0.7499 g
DI water (4.1.4.)	up to 1000 mL

Place 0.7499 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 umhos/cm)	LFB_CHCK2, CHCK2	1000	100
0.0005 M KCl (73.5 umhos/cm)	LFB_CHCK4, CHCK4	1000	50

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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.). Check the manufacturer's expiration date on the bottle or the certificate of analysis for the particular lot number and ensure it does not expire within the next 28 days. Holding time, 28 days after opening.

5. SAMPLE HANDLING AND PRESERVATION

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.

This SOP should be used in conjunction with SOP NU-060 **-** Automated PC Titrate Instrument Operating Procedure, which gives a more detailed description of the instrumentation.

6.1. Make sure all sample tubes have their caps removed prior to operation of the instrument or the conductivity probe and sample needles will be damaged

6.2. 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.

6.3. Meter Calibration - Calibrate the meter using conductivity standard (4.5.). Refer to page 11 of Model 4510 Conductivity Meter Operator Manual for details.

6.4. Start-up of the PC-Titrate Software - Log in to the PC-Titrate software; choose the appropriate method (conductivity alone or in combination with other tests). Set up the run sequence corresponding to the timetable of PC-Titrate software (see DEP SOP NU-060 for details).

6.5 Loading samples - Pour or filter 45 - 47 mL of sample or standard into a 50 mL PP centrifuge tube. If samples will only be run for conductivity, only 35-40 mL of sample or standard is needed. Load the sample tubes onto the autosampler in sequence determined in section 6.4. It is recommended to run a DI water sample at the very beginning as a rinse to clean up the system and condition the probe.

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Example Run Sequence:

Rinse – tap water
Conductivity Calibration Standard (4.5.)
Rinse – DI water
MDL (blank-DI water)
CHCK4 (4.4)
ALTQC (4.6)
1M COND (4.2.)
Conductivity Calibration Standard (COND CAL) (4.5.)
Rinse – DI water
Blank- DI water
MBLK- Method blank- filtered 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 or CHCK4- alternate CCV CHCK 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 1.91 %.

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 according to the manufacturer's instructions (see 6.3.). 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 111900 µmhos/cm high calibration check standard (1MCOND, 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 < 2.0 µmhos/cm and > -2.0 µmhos/cm. Acceptance limits for the recovery of the high check standard and the

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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 to 115 %.

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 < 2.0 µmhos/cm and > -2.0 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 < 2.0 µmhos/cm and > -2.0 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 (1409 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 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. Raw data is retrievable with the 'HISTORICAL DATA' function of PC-Titrate software. At the beginning of every month, the raw data is moved to the network drive by the analyst for archiving in the PCT BACKUP SYSTEM 1 (for PC-Titrate 1) or PCT BACKUP SYSTEM 2 (for PC-Titrate 2) folder located at: \\FLDEP1\DEAR\\Labs\Lab3\Chem_Data\Inorganic\Nutrients\PC-TITRATE.

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9.2. A tab delimited text file for each run must be exported to the network drive: \\FLDEP1\DEAR\LABS\Lab3\Chem_Data\Inorganic\Nutrients\PCTitrate or \\FLDEP1\DEAR\LABS\Lab3\Chem_Data\Inorganic\Nutrients\PCTitrate\PCTitrate 2, depending on which instrument is used. The file is stored in the analysis year subfolder for the instrument used. The file name should use the format YYMMDDN-A# where YY is the year, MM is the month, DD is the day, N is the first letter of the test/s conducted, A is auto-titrator, and # is 1 or 2 which indicates what instrument was used (e.g., fluoride and conductivity run = 120206FC-A1). This file is used by QC manager for data processing and uploading into LIMS.

9.3. The raw data is archived in paper format. A report folder is created for each analytical run. A folder may contain more than one analysis (e.g. Alkalinity, and Conductivity, and/or Fluoride) in it if the analyses are part of the same analytical run. The folder is labeled with the test (ALK, W-COND, and/or W-F), filename/s (YYMMDDN-A#), and initials of the analyst.

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 Method validation data is on file with the supervisor. Digital copy is also stored on the network, under an appropriate sub-folder in \\fldep1\dear\Labs\Lab5\Chemistry\Inorganic\Nutrient\Validations\PC Titrate.

10. MAINTENANCE OF ELECTRODE

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.

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 on the intranet at https://www.fldepnet.org/content/dear/bureau-laboratories-health-and-safety-plan).

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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. Model 4310 and Model 4510 Conductivity Meters Operating Manual.

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. DEP SOP NU-060, Automatic PC-Titrate Instrument Operating Procedure.

12.10. Quality Manual, Bureau of Laboratories, FDEP.

12.11. Laboratory Health and Safety Plan.

Appendix of Significant Changes

03/02/2009 Added new high-level check standard to Sections 5.2 and 9.1

02/05/2010 Clarified the Sample Handling and Preservation in Section 6 and the acceptance limits for the ALTQC standard in Section 9.1.

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02/03/2015	Data archival information was clarified in Section 9.
02/04/2016	File paths and web addresses were updated.
02/14/2018	Storage location for the Certificates of Analysis of purchased reagents and standards changed to the network drive. Acceptance limits for the LFB and Cal check standard added.
12/10/2019	The run sequence was clarified in Section 6. Acceptance criteria for quality control measures were updated in Section 8.
12/28/2020	In Section 3, the instrumentation, equipment, and supplies were updated and inputted into table format. In Section 4, chemicals were updated and inputted into table format. Preparation procedures for reagents and standards were reformatted.
01/18/2022	Added statement about the evaluation of relative error of the calibration. Made the low check (CHCK4) required to be run first, rather than the option of CHCK4 or CHCK2.

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