Illinois State Water Survey

Health and Environmental Application Laboratory

Standard Operating Procedure
For The Determination of Conductivity

SOP Number: AN.HEAL.EL.conductivity.15.3 (AN-0019)

Revision 15.3, effective July 20, 2022

NOTE THE HEALTH & SAFETY WARNINGS IN SECTION 4.0

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Revision History

Beginning Revision	Ending Revision	Revision Date	Changes
13.0	14.0	3/9/2020	Updated procedure and also SOP number to the new convention
14.0	15.0	8/2/2021	Removed section 12.4 about reanalysis
15.0	15.1	4/19/2022	removed appendix B (SDS sheet), added appendix A (quality control solution preparation)
15.1	15.2	5/12/2022	Added holding times to appendix A, fixed date on front cover, added revision history table
15.2	15.3	7/20/2022	Formatting and minor editing. Section 16.0 changed to reflect current recordkeeping procedures

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1.0 Scope & Applicability

Samples are measured for conductivity using unfiltered aliquots.

2.0 Summary of Method

2.1 The conductance meter is standardized and calibrated with externally purchased solutions and HEAL in-house prepared solutions every 12 samples for quality assurance purposes. Control charts are generated.

2.2 A Laboratory Information Management System (LIMS) is used to record and track conductivity analysis.

3.0 Definitions

DI	Deionized (water) at 18.2 Mohms-cm or higher
FBYY####	DI Blank
FHYY####	An external quality control high standard 20.0 µS/cm standard
FLYY####	An external quality control low standard 5.0 µS/cm standard
FR50YY##	An in-house prepared quality control synthetic sample targeting the 50th percentile concentration of all precipitation samples analyzed for the NADP/NTN.
FOYY####	An external quality control 84.0 µS/cm standard, used for calculating the correction factor
HDPE	High-Density Polyethylene
LABNO	Laboratory identification number
ISA	Insufficient Sample for Analysis
LIMS	Laboratory Information Management System
LDPE	Low-Density Polyethylene
NA	No Analysis
QA	Quality Assurance
QA/QC	Quality Assurance/Quality Control

4.0 Health & Safety Warnings

4.1 Safety eyewear is required to be worn in the lab beyond the partition when filtration is in progress.

4.2 Safety Data Sheets (SDS) applicable to this SOP are available through the **Division of Research Safety,** and can be found online at http://www.drs.illinois.edu.

4.3 The Illinois State Water Survey Chemical Hygiene Plan covers the ISWS laboratory safety program, including, but not limited to, personal protective equipment used, control equipment inventory and operations (such as vented hoods), employee training programs, medical programs, and safety. The ISWS Chemical Hygiene Plan is available at http://isws.illinois.edu/staffonly/resources/manuals.asp. Procedural notes are included in test methods used (e.g. ASTM International, United States

Environmental Protection Agency (USEPA), or Standard Methods for the Examination of Water and Wastewater).

4.4 The University of Illinois **Division of Research Safety** requirements for chemical safety can be found at http://www.drs.illinois.edu/trainingapp/Quizzer/qdbQuizMain.aspx?hfo=10.

4.5 The HEAL has listed known health and safety warnings for this SOP, but this list should not be assumed to comprise all health and safety issues.

5.0 Cautions

5.1 Contamination Cautions

Personnel need to be extremely organized and detailed in all aspects of the conductivity measurement. Personnel need to be mindful of the low concentration level of the analytes and take care to avoid contamination. Detailed quality assurance data logs are kept and carefully monitored for potential problems.

5.2 Conductance Cell Cautions

5.2.1 The cells in use are YSI 3253. These have built-in thermistors to allow for automatic temperature compensation.

5.2.2 This cell must be stored with DI water in it. When new cells arrive, wrap parafilm around vent holes and fill with DI. Then fill a vial with DI and use as a cap on the cell. Parafilm cap to the cell and store cell in its box until use. New cells must soak at least 24 hours before use.

5.2.3 When daily measurements are finished, store cell with fresh DI in it, and cap it to prevent evaporation.

5.2.4 If a cell dries out, it must be rehydrated with DI a minimum of 4 hours.

6.0 Interferences

None

7.0 Personnel Qualifications

Several days training with qualified staff and successful analysis of internal QA/QC samples.

8.0 Apparatus & Materials

8.1 Equipment

8.1.1 YSI 3200 Conductance Meter

8.1.2 YSI 3253 Conductance Cell

8.1.3: PVC vial support blocks and plexiglass covers

8.1.4 LIMS Preplab Module

8.2 Chemicals and Solutions

8.2.1 Deionized water, with a resistivity of 18.2 Mohms or better

8.2.2 Quality Control FR50 Solution (found on the laboratory benches in room 209 and in cooler # 214).

8.2.3 Quality Control FH (20 μS/cm) Solution (found on the laboratory benches in room 209 and in cooler # 214).

8.2.4 Quality Control FL (5 μS/cm) Solution (found on the laboratory benches in room 209 and in cooler # 214).

8.2.5 Quality Control FO (84 μS/cm) Solution (found on the laboratory benches in room 209 and in cooler # 214).

8.3 Supplies

8.3.1 Conical Polystyrene Sample Cups, 4 mL (found in room 209 in cabinet, labeled vials.)

8.3.2 Nalgene LDPE 500 mL Wash Bottle (found in room 209 on laboratory benches)

8.3.3 Safety Glasses

8.3.4 Parafilm (found in room 209 on laboratory benches)

8.3.5 ULINE Wipers (found in room 209 in cabinet labeled “Uline Wipers”)

8.3.6 Reanalysis Notebook (on bookshelf in room 209)

8.3.7 Room 209 Records Notebook (found on the laboratory benches in room 209)

9.0 Instrument or Method Calibration

9.1 HEAL Conductance Meter Information

9.1.1 The YSI 3200 conductance meter and YSI 3253 conductance cell are used for all conductance measurements. For more detailed instructions on how to work with the YSI 3200 meter, see the manual *YSI Model 3200 - Conductance, Resistance, Salinity, Total Dissolved Solids, and Temperature Instrument Operators Manual* (located on the bookshelf in room 209)

9.1.2 Conductance analysis is always done before pH because the pH electrode filling solution leakage will alter the measured conductance of the samples if done before.

9.1.3 The meter and cell must be standardized and calibrated at the start of analysis. After initial calibration, additional QC solutions are measured every twelfth sample.

9.1.4 When reading conductance values with the YSI 3200 and 3253, wait approximately 5 seconds before recording a measurement. Do not wait to read the display longer than this, as conductance typically drifts continuously during measurements. When first pouring the sample into the cell, the value will rapidly fluctuate to find the correct value, stabilize 5 seconds, and then slowly start to drift steadily in one direction, usually in a downward fashion. The correct reading is when the meter and cell momentarily stabilize.

9.1.5 The YSI 3253 Conductivity cells are dip cells which can be used as cup cells. The hole is covered with parafilm, and the cell inverted. This will change the cell constant somewhat, so a correction factor is used to compensate for the offset. Using the cell as a cup cell allows a small sample volume of only 2 mL for a conductance measurement.

9.1.6 Note the temperature on the meter. This is found on the bottom of the screen. It should be between 20˚C - 25˚C. Room 209 has its own air conditioner and temperature control to ensure this temperature range can be maintained.

9.1.7 Temperature Compensation is found on the right side of the screen and should read 1.50% ˚C (see meter manual for more instructions).

9.2 Standardization of YSI 3200 Meter and YSI 3253 Cell

9.2.1 Start the Milli-Q unit in Room 209. Let the water run until the resistance meter reads a minimum of 18.2 Mohm-cm before using. See section 13.1 if the value is < 18.2 Mohm-cm.

9.2.2 Using a wash bottle, flush the cell with a copious amount of DI water. Flick excess water from the cell and then refill with DI. Use this portion to read the raw conductance value of the DI water. The water should be under 1.0 μS/cm. If it is, continue to standardize. If it is not, see section 13.2.

9.2.3 Fill the cell with an aliquot of 84.0 μS/cm standard. The cell does not need to be filled completely, but the black platinum electrode needs to be completely immersed. This is used as a “conditioning rinse” of the cell. Allow this solution to remain in the cell for several seconds, then discard. Flick excess solution from the cell and refill with a second aliquot. The raw conductance value is read from this second portion.

9.2.4 The 84.0 μS/cm value should read greater than 60 μS/cm and less than 90 μS /cm. If it does, the meter and cell are now standardized. If the value is out of range, see section 13.3.

9.2.5 Using the 84.0 μS/cm value, calculate the correction factor (see section 14.1 for equation). The correction factor correct for the inverted nature of the cell.

10.0 Sample Collection

Samples to be analyzed are stored at 4˚C ± 2˚C in the walk-in cooler, room 214.

11.0 Handling & Preservation

11.1 All samples are to be handled with care, avoiding any direct hand/body contact with the sample or interior of the bottle and lid.

11.2 Keep sample bottles tightly sealed when not being poured. Keep samples and standards covered as much as possible prior to analysis to reduce airborne contamination.

11.3 When pouring samples into the sample tubes, avoid splashing or spillage of sample into an adjoining sample tube.

12.0 Sample Preparation and Analysis

12.1 Setup of LIMS for Conductivity Analysis

12.1.1 Turn on computer and click ***BenchChem*** icon, or you can find BenchChem in programs under NADP.

12.1.2 Select ***Chemistry***, ***Conductivity***, ***Load Sample List***, ***Create List***.

12.1.3 Select ***Sample Range*** tab.

12.1.4 Enter sample range for analysis in the **First Sample ID** and **Last Sample ID** text boxes. Select ***Add to List*** tab then ***Save*** tab.

12.1.5 The sample list will appear. Click on the first sample in your list. The current sample for analysis will be highlighted and appear in the **Current Sample** text box. You will need to select the Serial Port for your meter before sending any values to LIMS. This is located at the bottom of the screen in the **Serial Port** textbox. Each meter has a serial number located on the bottom left corner of the meter.

12.1.6 Enter the correction factor (see section 14.1 for calculation) and select the ***Set Factor*** *tab.* The correction factor will be shown in the **Corr. Factor** text box.

12.2 Measuring Conductivity of QC Samples Using LIMS

12.2.1 After standardization is complete, all QA solutions (FH, FL, FR50 and FB) must be analyzed before precipitation sample analysis can occur. LIMS is used to record these values.

12.2.2 Alternate QC solutions after every 12 samples. After 36 samples, recalibrate with 84.0 standard, and measure a high and a low QC standards.

12.2.3 Scan the selected QC sample. This will appear in the **Current Sample** text box.

12.2.4 Pour an aliquot of the selected QC solution into the cell and allow this to remain in the cell for 5 seconds to stabilize. This aliquot is used to take the first measurement. When solution has stabilized, press the **Send** button on the meter to send the value to the LIMS. The conductivity will not be sent to LIMS automatically.

12.2.5 After sample is sent to LIMS, the correct value should read within the control limits as defined by the quality assurance specialist.

12.2.6 When the value is sent, it will appear on the screen in the **Initial** text box.

12.2.7 Pour second aliquot of same QC solution into the cell and repeat steps 12.2.4 through 12.2.6. The second value will appear in the **Final** text box. Click on the **Save Result** tab. If results are out of control limits, see section 13.4.

12.2.8 After writing the value, the QC sample will disappear from the next box and the next sample in the series will be listed.

12.3 Measuring Conductivity of Samples Using LIMS.

12.3.1 Begin measuring samples after the meter and cell have been calibrated and QC samples have been measured.

12.3.2 Check to make sure the sample ID appearing in the text box matches the correct sample vials.

12.3.3 Samples with volumes of 30 mL or more will have 2 sample vials. Samples with less than 30 mL will have 1 vial.

12.3.4 For samples with 2 vials, pour ½ of the contents of the first vial into the cell. The sample volume does not have to fill the cell entirely, but the black platinum electrode within the cell must be completely covered.

12.3.5 When the meter stabilizes, press **Send** button on the meter to send the value to the LIMS

12.3.6 When the value is sent, it will appear on the screen in the **Initial** text box.

12.3.7 Pour one-half of the remaining contents of the second vial into the cell and repeat steps 12.3.5 through 12.3.6. The second value will appear in the **Final** text box. Click on the **Save Result** tab. The two remaining ½ vials are used for pH analysis (see SOP #AN.HEAL.EL.pH, *Determination of pH*).

12.3.8 After the results are written, the computer will automatically go on to the next sample in the list.

12.3.9 For samples with only one sample vial (4 mL), Take both readings from that vial.

12.3.10 For samples with only ¾ vial (3 mL), pour the entire contents of the vial into the cell and use this measurement as a valid raw value. After obtaining the value, do not discard. Pour the contents from the cell back into the vial to be used for pH analysis.

12.3.11 For samples with only ½ vial (2 mL), conductance cannot typically be done. There is usually not enough volume to immerse the black platinum electrode within the cell with the sample. Leave the ½ vial for pH measurement only and skip to the next sample in the list.

12.3.12 When finished analyzing samples, measure all QC solutions a final time. Store the cell with fresh DI in it, and cap it to prevent evaporation from occurring.

13.0 Troubleshooting

13.1 If the DI Water Resistance Meter on the Millipore unit reads less than 18.2 Mohm-cm notify the quality assurance specialist and stop analysis.

13.2 If the DI water conductivity is greater than 1.0 μS/cm when measuring with the YSI cell before standardization:

13.2.1 Replace the DI water in the wash bottle and reanalyze.

13.2.2 Replace the DI water in the wash water with room 306 DI water to test against the room 209 DI water.

13.2.3 Replace the YSI 3253 conductance cell to test the old cell.

13.2.4 Replace the wash bottle with a new one.

13.2.5 If none of these steps solve the issue, then stop analysis and inform the quality assurance specialist.

13.3 If the 84.0 μS/cm raw conductance (measured) value is not between 60 - 90 μS/cm:

13.3.1 Re-measure the 84.0 μS/cm again.

13.3.2 Replace the bottle with a new bottle of 84.0 μS/cm and re-measure.

13.3.3 Replace the YSI 3253 cell with a new cell.

13.3.4 Use the YSI 3166 Calibrator set to check meter calibration

13.3.5. If none of these steps solve the issue, stop all analysis and inform the quality assurance specialist.

13.4 If the FR50, FH, and FL corrected conductance do not measure within the control limits as determined by quality assurance specialist:

13.4.1 Re-measure the solution out of control;

13.4.2 Recalibrate and then re-measure

13.4.3 If none of these steps solve the issue, stop all analyses and inform the Laboratory Supervisor or Quality Assurance Specialist.

14.0 Data Acquisition, Calculations & Data Reduction

14.1 All raw conductance values must be corrected using a correction factor. The correction factor is calculated from the 84.0 µS/cm theoretical value (84.0 µS/cm) divided by the 84.0 µS/cm measured value (60 - 90 µS/cm).

Theoretical / measured = correction factor.

14.2 The correction factor is calculated every 36 samples after standardization with the 84.0 µS/cm conductivity standard.

14.3 All raw sample values sent to LIMS are corrected automatically by the computer. This corrected value appears below the **Corrected** text box when the data is sent.

15.0 Computer Hardware & Software

For LIMS hardware and software information, see SOP #SS.NEON.0.NEONsoftware, NEON Software manual.

16.0 Data Management & Records Management

16.1 The analyst records all analyzed samples in the logbook daily.

16.2 To find any conductivity value, go to the **Instrumental Chemistry**, select **LIMS**, **Query**, **LIMS Query**. In the **By Sample Number** section, enter the first and the last sample numbers of interest, then click **Retrieve**.

16.3 Information on new conductance cells and replacement/repair information on conductivity meters is recorded in the “Records Notebook.”

17.0 Quality Control and Quality Assurance Section

17.1 Control Charts are generated in the LIMS by the QC data entered.

17.1.1 Select ***BenchChem**, **LIMS**, **New Tables*** from the toolbar.

17.1.2 Select ***QC Samples*** tab from the query options.

17.1.3 QC Charts can be viewed by analyst or date range from one day to the entire year. Select if desired.

17.1.4 Use drop down menu to select desired Sample ID, Analyte, and Date Range. The control chart will appear on the screen.

17.1.5 As a quality control check once a month, use the YSI 3166 calibrator set to check meter calibration per manufacturer’s specifications. If it is out of range, contact Quality Assurance chemist.

17.2 Quality Assurance Replicates

- As you are pouring your samples, select a sample that is at least 50 mL for your replicates.
- After pouring the original sample, pour the sample three additional times and set them aside.
- Two of these samples will be analyzed back-to-back.
- The original sample will be in the sample list labeled # 22XXXXXX.
- The three additional samples will be entered as # 22XXXXXX-Q.
- The replicate samples may serve as a QC sample.

18.0 References

YSI Model 3200 Conductance, Resistance, Salinity, Total Dissolved Solids, and Temperature Instrument, April 1999.

APPENDIX A. Quality Control Samples Preparation

A.1 Calculations

Conductivity, µS/cm = ∑ (Ion Concentration, µeq/L) x $\Lambda_{\pm}$ x 10^{-3}
where:

- (Ion Concentration, µeq/L) are concentrations of K^+ and Cl^-.
- $\Lambda_{\pm}$ = Ionic conductivity, $10^{-4} m^2 S\text{-}mol^{-1}$ as listed in Table *:

Ion	$\Lambda_{\pm}$, $10^{-4} m^2 S/mol$
K^+	73.48
Cl^-	76.31

* From "Ion Conductivity and Diffusion at Infinite Dilution," *CRC Handbook of Chemistry and Physics*, 102nd Ed., 2021-2022, Section 5. Ionic Conductivity and Diffusion at Infinite Dilution (chemnetbase.com).

Calculate the required concentration of KCl to prepare a solution with a certain conductivity using the following formula:

$$\sum \text{(Ion Concentration, µeq/L)} = (\text{Conductivity, µS}\cdot\text{cm}^{-1} / \sum \Lambda_{\pm}) \times 10^3$$

Stock Solution **5000 µS/cm**:

KCl concentration, µeq/L = {5000 / (73.48 + 76.31)} x 10^3 = 33.38 x 10^3 µeq/L (or µM/L)

Conductivity Standard **5 µS/cm**:

KCl concentration, µeq/L = {5 / (73.48 + 76.31)} x 10^3 = 33.38 µeq/L (or µM/L)

Conductivity Standard **20 µS/cm**:

KCl concentration, µeq/L = {20 / (73.48 + 76.31)} x 10^3 = 133.52 µeq/L (or µM/L)

These calculations are based on the fact that the ideal DI water is not dissociated into ions. The slight dissociation of the real DI water must be taken into account. Typically, the conductivity of DI water used in a laboratory ranges between 0.9 - 1.1. Before preparing the stocks and standards, the electrical conductivity of the DI water must be measured and subtracted from the value of the required electrical conductivity.

Example:

DI water conductivity is 1.0 $\mu S \cdot cm^{-1}$

Stock Solution **5000 µS/cm**:

KCl concentration, µeq/L = $\{4999/(73.48 + 76.31)\} \times 10^3 = 33.37 \times 10^3$ µeq/L (or µM/L)

Conductivity Standard **5 µS/cm**:

KCl concentration, µeq/L = $\{4/(73.48 + 76.31)\} \times 10^3 = 26.70$ µeq/L (or µM/L)

Conductivity Standard **20 µS/cm**:

KCl concentration, µeq/L = $\{19/(73.48 + 76.31)\} \times 10^3 = 126.84$ µeq/L (or µM/L)

A.2 Summary of Method (assuming that DI water conductivity is equal to 1 $\mu S \cdot cm^{-1}$)

To prepare KCL Stock Solution **5000 µS/cm**: (33.37×10^3 µM/L KCl):

1. Dry KCl (crystals) > 5 hours in oven at 104 °C.
2. Add 2.4878 g dried KCl to the 1L volumetric flask with 800 mL DI water. Add DI water to 1L volume. Shake carefully.
3. Do not keep longer than 1 year.

To prepare **5 µS/cm** Specific Conductance QC Solution (26.70 µM/L KCl):

1. Add 0.8 mL KCL stock solution to the 1L volumetric flask with 800 mL DI water. Add DI water to 1 L volume. Shake carefully.
2. Analyze pH, conductance, K^+, Cl^-
3. Discard after 6 months.

To prepare **20 µS/cm** Specific Conductance QC Solution (126.84 µM/L KCl):

1. Add 3.8 mL KCL stock solution to the 1L volumetric flask with 800 mL DI water. Add DI water to 1 L volume. Shake carefully.
2. Analyze pH, conductance, K^+, Cl^-
3. Discard after 6 months.

A.3 Quality Control and Quality Assurance

Limits for acceptance of SCS
Specific conductance = 5 ± 0.5 µS/cm
pH = 5.62 ± 0.10 pH units
$[K^+]$ = 1.044 mg K^+/L ± 0.052 (± 5%)
$[Cl^-]$ = 0.9465 mg Cl^-/L ± 0.047 (± 5%)

Specific conductance = 20 ± 2.0 µS/cm
pH = 5.62 ± 0.10 pH units
$[K^+]$ = 4.958 mg K^+/L ± 0.247 (± 5%)
$[Cl^-]$ = 4.496 mg Cl^-/L ± 0.225 (± 5%)

References

Standard Operating Procedure For IPD/CPD Calculations for Atmospheric Deposition Samples DA.HEAL.0.IPD.3.2 (DA-0067.4)

"Ion Conductivity and Diffusion at Infinite Dilution," *CRC Handbook of Chemistry and Physics*, 102nd Ed., 2021-2022

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