# Illinois State Water Survey Health and Environmental Application Laboratory

# **Standard Operating Procedure**For The Determination of Conductivity

(based on Standard Methods 2510 B)

SOP Number: AN.HEAL.EL.conductivity

Revision 16.2, 8 October 2024

### 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
15.3	16.0	9/15/2023	Changed QC scheme and made
			formatting changes
16.0	16.1	7/25/2024	Removed blanks, changed QC
			scheme.
16.1	16.2	10/8/24	Minor updates to address
			comments, corrected duplicate
			acceptance criteria.

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### 1.0 Scope & Applicability

This method is applicable for drinking, surface, ground, and wet deposition water samples.

### 2.0 Summary of Method

- 2.1 The conductance meter is calibrated with externally purchased solutions and HEAL in-house prepared solutions analyzed every 10 samples for quality assurance purposes.
- 2.2 A Laboratory Information Management System (LIMS) is used to record and track conductivity analysis.

### 3.0 Definitions

**ACS** American Chemical Society

**CCV** Continuing Calibration Verification

**LFB** Laboratory Fortified Blank

**DI** Deionized (water) at 18.0 Mohms-cm or higher

ICV Initial Calibration Verification. HEAL uses a simulated rain

sample as an ICV:

FR50- a solution with target analyte concentrations at the

50th percentile of the NTN network results.

MDL Method Detection Limit

HDPE High Density Polyethylene

Laboratory Information Management System

QA Quality Assurance
QC Quality Control

R<sup>2</sup> Coefficient of Determination reflecting the deviation of the

measured data points from the calibration curve

SDS Safety Data Sheet

**SOP** Standard Operating Procedure

### 4.0 Health & Safety Warnings

- 4.1 Always wear eye protection in the laboratory.
- 4.2 Food, drinks, and smoking are not allowed in the laboratory.
- 4.3 Safety Data Sheets (SDS) applicable to this SOP can be found online by using the University of Illinois Division of Research Safety (DRS) website: <a href="https://www.drs.illinois.edu/Programs/SafetyDataSheets">https://www.drs.illinois.edu/Programs/SafetyDataSheets</a>.
- 4.4 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 <a href="https://go.illinois.edu/ISWS-Chemical-Hygiene-Plan">https://go.illinois.edu/ISWS-Chemical-Hygiene-Plan</a>.

- 4.5 The University of Illinois DRS has a laboratory safety guide available at <a href="https://www.drs.illinois.edu/site-documents/LaboratorySafetyGuide.pdf">https://www.drs.illinois.edu/site-documents/LaboratorySafetyGuide.pdf</a>. The ISWS has their own laboratory safety manual, available at <a href="https://go.illinois.edu/ISWS-Laboratory-Safety-Manual">https://go.illinois.edu/ISWS-Laboratory-Safety-Manual</a>.
- 4.6 The HEAL practices pollution prevention, which encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. The quantity of chemicals purchased should be based on the expected usage during its shelf life and disposal cost of unused material.

### 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.

- 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.
- 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

Personnel are trained by qualified staff. Demonstration of capability is performed and approved in accordance with the QAP.

### 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 8.2.3 Quality Control LFB (20 µS/cm) Solution 8.2.4 Quality Control FL (5 µS/cm) Solution 8.2.5 Quality Control FO (84 µS/cm) Solution 8.3 Supplies 8.3.1 Conical Polystyrene Sample Cups, 4 mL (Globe Scientific #110811 or equivalent) 8.3.2 Nalgene LDPE 500 mL Wash Bottle 8.3.3 Safety Glasses 8.3.4 Parafilm 8.3.5 ULINE Wipers, Kimwipes, or similar 8.3.6 Reanalysis Notebook 8.3.7 Room 209 Records Notebook 9.0 **Instrument or Method Calibration** 9.1 **HEAL Conductance Meter Information** The YSI 3200 conductance meter and YSI 3253 conductance cell are used for all 9.1.1 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 may leak and 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 ten samples. 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.

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.

9.1.5

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.
- 9.2.2 Fill the cell with an aliquot of 84.0  $\mu$ S/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.
- 9.2.3 The 84.0  $\mu$ S/cm value should read greater than 60  $\mu$ S/cm and less than 90  $\mu$ S /cm. If it does, the meter and cell are now standardized. If the value is out of range, see section 13.1.
- 9.2.4 Using the 84.0  $\mu$ S/cm value, calculate the correction factor (see section 14.1 for equation). The correction factor corrects 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.
- 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, QC solutions FR50, LFB and FL must be analyzed before precipitation sample analysis can occur. LIMS is used to record these values.
- 12.2.2 QC solutions are analyzed after every 10 samples: FL and duplicate. After 30 samples, calibrate with 84.0 standard, and measure FR50, LFB and FL.
- 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.
- 12.2.8 After writing the value, the QC sample will disappear 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  $\frac{1}{2}$  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  $\frac{1}{2}$  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 84.0  $\mu$ S/cm raw conductance (measured) value is not between 60 to 90  $\mu$ S/cm:
- 13.1.1 Re-measure the 84.0 μS/cm again.
- 13.1.2 Replace the bottle with a new bottle of 84.0  $\mu$ S/cm and re-measure.
- 13.1.3 Replace the YSI 3253 cell with a new cell.
- 13.1.4 Use the YSI 3166 Calibrator set to check meter calibration
- 13.1.5. If none of these steps solve the issue, stop all analysis and inform the quality assurance specialist.
- 13.2 If the FR50, LFB, and FL corrected conductance do not measure within the control limits as determined by quality assurance specialist:
- 13.2.1 Re-measure the solution out of control;
- 13.2.2 Recalibrate and then re-measure
- 13.2.3 Make new QC solutions and repeat step 13.2.2.

### 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  $\mu$ S/cm theoretical value (84.0  $\mu$ S/cm) divided by the 84.0  $\mu$ S/cm measured value (60 - 90  $\mu$ S/cm).

### Theoretical / measured = correction factor.

- The correction factor is calculated every 36 samples after standardization with the  $84.0~\mu\text{S/cm}$  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.
- 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

- 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 guery 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.

Table 17-1. Quality Control Summary

QC Name	QC Type	Target Value	Acceptance Criteria
FR50	ICV	9.34 μS/cm	+/- 1.0 μS/cm
FL	CCV	5 μS/cm	+/- 5%
LFB	LFB	20	+/- 5%
Duplicate	DUP	-	+/- 10% if >10*MDL

# 18.0 References 18.1 YSI Model 3200 Conductance, Resistance, Salinity, Total Dissolved Solids, and Temperature Instrument, April 1999. 18.2 Standard Operating Procedure For IPD/CPD Calculations for Atmosphere Deposition Samples DA.HEAL.0.IPD.3.2 (DA-0067.4). 18.3 "Ion Conductivity and Diffusion at Infinite Dilution," CRC Handbook of Chemistry and Physics, 102<sup>nd</sup> Ed., 2021-2022. 18.4 Standard Methods for the Examination of Water and Wastewater, 22<sup>nd</sup> Ediiton, 2012, 2510 B. Laboratory Method, Washington, DC.

### **APPENDIX A. Quality Control Samples Preparation**

### A.1 Calculations

Conductivity,  $\mu$ S/cm =  $\sum$  (Ion Concentration,  $\mu$ eq/L) x  $\Lambda_{\pm}$  x 10<sup>-3</sup> where:

- (Ion Concentration, μeq/L) are concentrations of K<sup>+</sup> and Cl-.
- Λ<sub>±</sub> = Ionic conductivity, 10<sup>4</sup>m<sup>2</sup>S-mol<sup>-1</sup> as listed in Table \*:

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

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

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

$$\Sigma$$
 (Ion Concentration,  $\mu$ eg/L) = (Conductivity,  $\mu$ S·cm<sup>-1</sup> /  $\Sigma$   $\Lambda$ <sub>+</sub>) x 10<sup>3</sup>

Stock Solution 5000 µS/cm:

KCl concentration,  $\mu eq/L = \{5000 / (73.48 + 76.31)\} \times 10^3 = 33.38 \times 10^3 \mu eq/L \text{ (or } \mu \text{M/L)}$ 

Conductivity Standard 5 µS/cm:

KCl concentration,  $\mu eq/L = \{5 / (73.48 + 76.31)\} \times 10^3 = 33.38 \mu eq/L (or <math>\mu M/L$ )

Conductivity Standard 20 µS/cm:

KCI concentration,  $\mu eq/L = \{20 / (73.48 + 76.31)\} \times 10^3 = 133.52 \mu eq/L (or <math>\mu M/L$ )

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 µS⋅cm<sup>-1</sup>

Stock Solution **5000 µS/cm**:

KCI concentration,  $\mu eq/L = \{4999/(73.48 + 76.31)\} \times 10^3 = 33.37 \times 10^3 \mu eq/L (or <math>\mu M/L$ )

Conductivity Standard **5 µS/cm**:

KCl concentration,  $\mu eq/L = \{4 / (73.48 + 76.31)\} \times 10^3 = 26.70 \mu eq/L \text{ (or } \mu \text{M/L)}\}$ 

Conductivity Standard 20 µS/cm:

KCl concentration,  $\mu eq/L = \{19 / (73.48 + 76.31)\} \times 10^3 = 126.84 \mu eq/L (or <math>\mu M/L$ )

**A.2** Summary of Method (assuming that DI water conductivity is equal to 1 μS·cm<sup>-1</sup>)

To prepare KCL Stock Solution **5000 μS/cm**: (33.37 x 10<sup>3</sup> 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 KCI):

- 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<sup>+</sup>, Cl<sup>-</sup>
- 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<sup>+</sup>, Cl<sup>-</sup>
- 3. Discard after 6 months.

### A.3 Quality Control and Quality Assurance

```
Limits for acceptance of SCS Specific conductance = 5 \pm 0.5 \mu \text{S/cm} pH = 5.62 \pm 0.10 \mu \text{pH} units [K<sup>+</sup>] = 1.044 \mu \text{mg} \text{ K}^{+}/\text{L} \pm 0.052 (\pm 5\%) [Cl<sup>-</sup>] = 0.9465 \mu \text{mg} \text{ Cl}^{-}/\text{L} \pm 0.047 (\pm 5\%)
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```
Specific conductance = 20 \pm 2.0 \mu \text{S/cm}
pH = 5.62 \pm 0.10 \text{ pH} units
[K<sup>+</sup>] = 4.958 \text{ mg K}^+/\text{L} \pm 0.247 (\pm 5\%)
[Cl<sup>-</sup>] = 4.496 \text{ mg Cl}^-/\text{L} \pm 0.225 (\pm 5\%)
```

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

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