



Use of ThermoOrion Chloride/Chloride Combination Electrodes

ThermoOrion chloride (94-17A, 94-17B) and chloride combination (96-17B) electrodes provide a simple, rapid, accurate, and cost-effective means of measuring chloride ions in aqueous solution. Read-out can be accomplished by specific ion meters sold by Orion or by various pH/mV meters such as the Accumet Models 825MP and XL25, both of which are available for use at E-Town. Copies of the relevant instructions for electrode preparation and testing, obtained from the Instruction Manual, are included here. Users are encouraged to consult the Instruction Manual for more information on the theory of electrode operation (pp. 28-29); electrode characteristics such as response, reproducibility, temperature effects, and interferences (pp. 24-27); and common analytical procedures, including direct calibration, incremental techniques (known addition, known subtraction, analyte addition, analyte subtraction), and titrations (pp. 7-17).

BASIC OPERATING PROCEDURES:

1. Prepare the Chloride/Chloride Combination Electrode for Use

- a. Based on the model that you are using (*i.e.*, Model 94-17A, 94-17B, or 96-17B), follow the appropriate instructions included on page 4. Note that the 94-17A and 94-17B require little preparation, but they must be used with a reference electrode (Model 90-02), which you will prepare in Step 2. Make certain to check carefully that you are using the correct filling solution(s).
- b. NOTE: While using filling solution, replace the long-term storage cap with a flip top cap for easier dispensing. When you are finished, be certain to replace the long-term storage cap, and wash the flip top thoroughly with deionized water.

2. Prepare the Reference Electrode for Use (if using Model 94-17A or 94-17B)

- a. Follow the instructions included on page 5 (new style) or 6 (older style) to prepare the double junction reference electrode (Model 90-02). Again, make certain to check carefully that you are using the correct filling solution(s).

3. Prepare a Meter for Read-Out

- a. Determine whether you have a BNC connector or an older-style connector on your chloride electrode.



BNC
Connector

- b. If you have a BNC-style connector, connect your chloride electrode (and reference electrode if needed) to an Accumet XL25 Meter, and safely position your electrode(s) in the attached arm. (You will first need to remove the shorting cap on the meter's BNC connector. Twist the BNC shorting cap to unlock it, and then gently disconnect it. Do the reverse to properly attach a BNC connector.)
- c. If you have an older-style connector, connect your chloride electrode (and reference electrode if needed) to an Accumet Model 825MP, and safely position your electrode(s) in the attached arm.
- d. Submerge your prepared electrodes (with protective caps removed) in deionized water until you are ready to proceed with measurements, and then plug in the meter. Once plugged in,
 - i. the 825MP should be in standby mode. Press the STBY/MEAS switch to toggle from standby to measurement mode in millivolts (mV). The reading in mV should now be displayed.
 - ii. the XL25 will run a self test, briefly display the Fisher catalog cover, and then display the main desktop screen. To verify that the correct channel has been assigned to your electrode, use the stylus (upper right hand-corner of the meter) to select MODE. If they appear, touch CH2 and CH3 to deselect them. On CH1, assure that mV mode is selected by touching it with the stylus, and then touch SINGLE CHANNEL to return to the read-out screen. The reading in mV should now be displayed.

4. Check Electrode Operation (Slope)

- a. To confirm proper operation of your chloride electrode (and reference electrode if in use), follow the instructions on page 7. Be certain to use the appropriate ionic strength adjusted (ISA'd) standard solutions. If you do not obtain the expected results, consult the Troubleshooting section in the Instruction Manual (pp. 20-22) to see if you can pinpoint an obvious problem that can be fixed. If you still have difficulty, notify Dr. Kneas.
- b. NOTE: If using the Model 94-17B electrode, you can stir the solution continuously while recording measurements. The Model 96-17B electrode, however, is not designed to operate while the solution is being stirred. It will be necessary to stir after each addition to the solution before the measurement is recorded.
- c. NOTE: Rinse electrodes with deionized water between measurements.

5. Conduct your Analytical Measurements

- a. Proceed with the appropriate analytical procedure (*i.e.*, direct calibration, incremental technique, or titration) to determine the amount of chloride in solution.
- b. Unplug the meter when you have completed your measurements, and prepare the electrodes for storage as described in the next step.

6. Prepare the Electrodes for Storage

- a. The Model 94-17B chloride electrode should be rinsed thoroughly, dried carefully with a KimWipe (using caution not to damage the sensing element on its tip), and stored with its protective cap. For long term storage, the electrode should be returned to the box from which it came.

- b. The Model 96-17B can be stored for short periods of time (in between measurements and up to one week) in a 0.01 M chloride standard solution. For longer term storage, the electrode should be drained, flushed inside with deionized water, dried carefully, and stored with its protective cap in the box from which it came.
- c. The Model 90-02 double junction reference electrode can be stored for very short periods of time (in between sample measurements and up to one hour) in air. For short periods of time (up to one week), it should be stored in its filling solution or deionized water. Make certain that the solution inside the electrode does not evaporate and result in crystallization. For longer term storage, the electrode should be drained, flushed with deionized water, dried carefully, and stored with its protective cap in the box from which it came.
- d. Reminder: When finished with filling solutions, replace the flip top cap with the long term storage cap, and wash the flip top thoroughly with deionized water. Return the filling solution and the clean flip top cap to the box from which they came.

7. Dispose of Waste, Clean up Station

- a. Store samples and standards or dispose of them properly. They should not be left near the potentiometric work stations. Assure that all waste and storage bottles are labeled appropriately.
- b. Return shared equipment and reagents to the designated locations and stock equipment to your drawer.
- c. Using a damp sponge, clean the area where you were working.
- d. If you are the last to use a particular work station, wrap cords around the base of the meter, stir plate, etc.

Chloride (94-17B) and Chloride Combination (96-17B) Electrodes

Electrode Preparation

Model 94-17B

1. Remove the rubber cap covering the electrode tip.
2. Fill reference electrode Model 90-02 according to instructions in the reference electrode instruction manual. Fill the inner chamber with Cat. No. 900002 and the outer chamber with Cat. No. 900003.

Model 96-17B — Filling Solution

The filling solution (Cat. No. 900017) supplied with this electrode is designed to minimize junction potentials and chloride ion contamination of the sample, and can be used for all chloride measurements. Orion now offers an additional filling solution (Cat. No. 900001) designed for use in samples more concentrated than 10^{-2} M chloride. The electrode potential characteristics of the reference electrode portion, when using the Cat. No. 900001 solution, match those of a conventional saturated KCl calomel reference electrode. ***Use of other filling solutions will void the warranty on the electrode.***

Filling Instructions

The electrode is shipped without filling solution in the reference chamber. To fill from the flip-spout bottle:

1. Lift the spout to a vertical position. **This refers to the interchangeable white cap on the bottle of the fill solution.**
2. Insert the spout into the filling hole in the outer sleeve and add a small amount of filling solution to the chamber. Tip the electrode to moisten the O-ring and return electrode to a vertical position.
3. Holding the electrode by the barrel with one hand, use the thumb to push down on the electrode cap, allowing a few drops of filling solution to drain to wet the inner cone.
4. Release sleeve. If sleeve does not return to its original position immediately, check to see if the O-ring is moist enough and repeat steps 2-4 until the sleeve has returned to original position. Add filling solution up to the filling hole.

Add filling solution each day before using electrode. The filling solution level should be at least one inch above the level of sample in the beaker to ensure a proper flow rate. If the filling solution is less than one inch above the sample solution level, electrode potentials may be erratic.

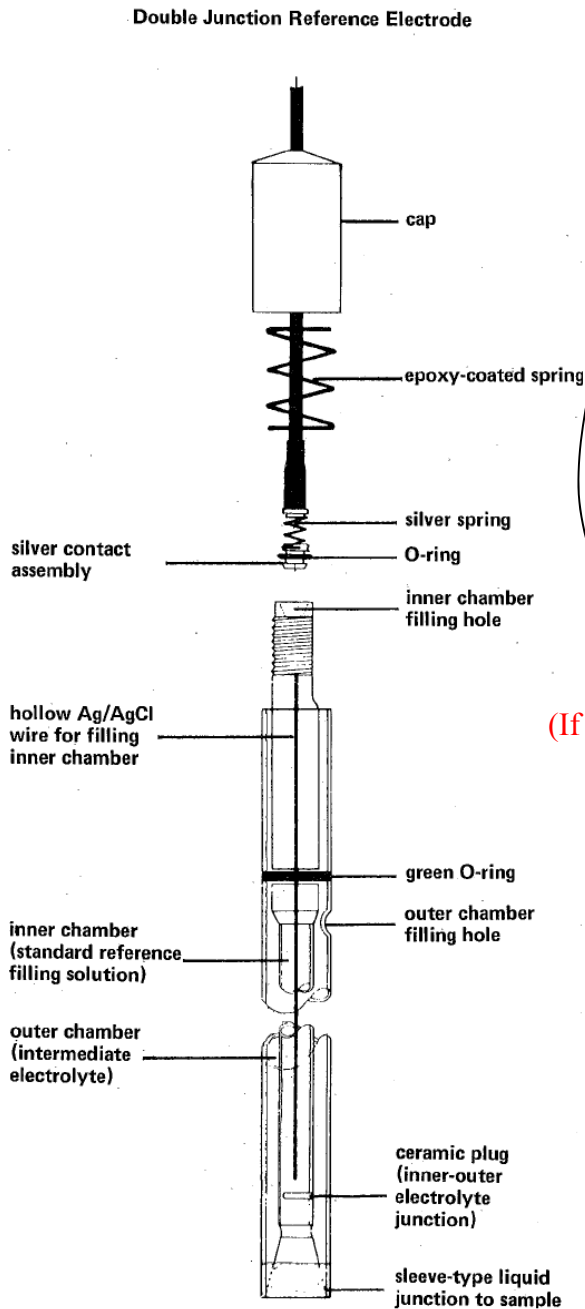
Double Junction Reference Electrode (Model 90-02, Newer Style)

Filling Instructions

To fill from the flip-spout bottle, lift the spout to the vertical position, place the spout in the electrode-filling hole, and squeeze the bottle firmly.

1. **Inner Chamber:** Use Orion 900002 only! Unscrew the electrode cap and spring up the cable. Push down on top of the inner chamber until cone at bottom end can be grasped using a lint-free tissue. Grasp cone and pull inner chamber free of outer sleeve. Slide rubber sleeve at top of inner chamber down to uncover filling hole. Using the flip-spout bottle, fill inner chamber up to fill hole and slide rubber sleeve back up. (If having trouble filling inner chamber, add some solution and shake electrode down like a clinical thermometer, repeat until filled.) Wipe excess filling solution off inner chamber surfaces and slide inner chamber completely up into outer sleeve. Place the spring back on inner chamber and screw cap on finger-tight.
2. **Outer Chamber:** Select an appropriate outer chamber filling solution. Using a flip-spout bottle or "medicine dropper," add a small amount of filling solution through the filling hole in the outer sleeve. Tip the electrode to moisten the green O-ring on the electrode body. Holding the electrode by the cap in one hand, push the outer sleeve up into the cap with the other hand, allowing the filling solution to wet the inner cone. Release the sleeve, check to see that the end of the sleeve is flush with the bottom surface of the cone, and fill the outer chamber up to the filling hole if the sleeve does not return to the correct position, push it gently down into place.

Double Junction Reference Electrode (Model 90-02, Older Style)



filling instructions

The Model 90-02 Double Junction Reference Electrode is shipped dry and must be filled with two different solutions before use. To fill from the flip-spout bottle, lift the spout to the vertical position, place the spout in the electrode filling hole, and squeeze the bottle firmly.

- 1. Inner chamber:** Use only Orion's 90-00-02 colored filling solution. Unscrew electrode cap and remove the green epoxy-coated spring. Remove silver spring and contact assembly. Using the flip-spout bottle, fill at the small hole in the center of the silver plug. Cover vent hole with an absorbent tissue to catch any overflow. Fill inner chamber up to the vent hole, place spring(s) on the top of the electrode body, and screw cap on finger-tight.
- 2. Outer chamber:** Select an appropriate outer chamber filling solution. Using a flip-spout bottle or "medicine dropper," add a small amount of filling solution through the filling hole in the outer sleeve. Tip the electrode to moisten the green O-ring on the electrode body. Holding the electrode by the cap in one hand, push the outer sleeve up into the cap with the other hand, allowing the filling solution to wet the inner cone. Release the sleeve, check to see that the end of the sleeve is flush with the bottom surface of the cone, and fill the outer chamber up to the filling hole. If the sleeve does not return to the correct position, push it gently down into place.

(If Connected to Base)

Chloride (94-17B) and Chloride Combination (96-17B) Electrodes

Checking Electrode Operation (Slope)

These are general instructions which can be used with most meters to check electrode operation. See individual meter instruction manuals for more specific information.

This procedure measures electrode slope. Slope is defined as the change in millivolts observed with every tenfold change in concentration. Obtaining the slope value provides the best means for checking electrode operation.

1. If electrodes have been stored dry, prepare the electrodes as described under the section entitled **Electrode Preparation**.
2. Connect electrodes to the meter by inserting the reference electrode pin-tip connector and the sensing electrode connector into appropriate jacks on the digital pH/mV meter or specific ion meter. Non-ORION meters may require special adaptors. Consult your meter instruction manual.
3. Place 100 ml distilled water in a 150 mL beaker. Add 2 mL ISA, Orion Cat No. 940011. Stir thoroughly. Set the function switch of the meter to the mV mode.
4. Rinse electrodes with distilled water and place in the solution prepared in step 3 above.
5. Select either 0.1 M Chloride, 1000 ppm Chloride, 10% Sodium Chloride, or .1% Sodium Chloride Standard. Pipet 1 mL of the standard into the beaker. Stir thoroughly. When a stable reading is displayed, record the electrode potential in millivolts.
6. Pipet 10 mL of the same standard into the same beaker. Stir thoroughly. When a stable reading is displayed, record the electrode potential in millivolts.
7. The difference between the first and second potential reading is defined as the slope of the electrode. The difference should be in the range of 54-60 mV/decade when the solution temperature is 25 °C. If the difference in potential is not within this range, refer to **TROUBLESHOOTING**.