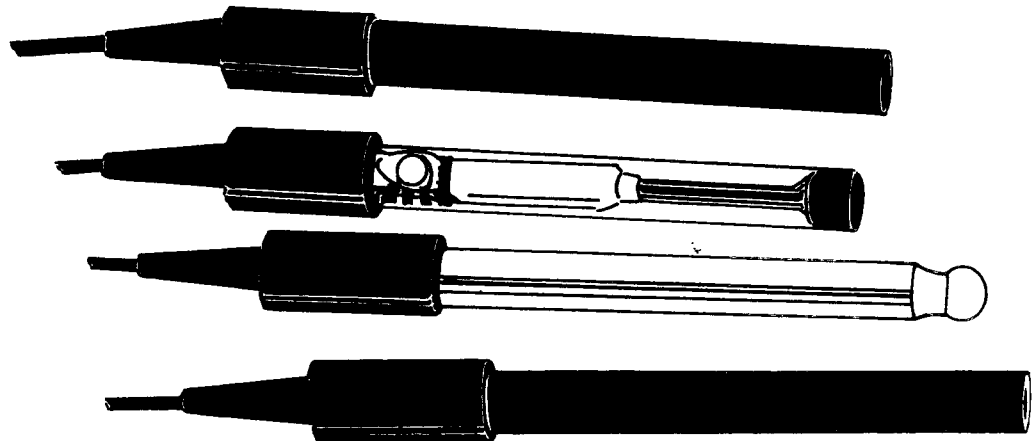


ISE-8760 & ISE-8770

Chloride Ion Selective Electrodes



Operator's Manual
M791/0892

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SECTION 1 GENERAL DESCRIPTION

The OMEGA® Chloride Ion Electrodes are used to quickly, simply and accurately measure chloride ions in aqueous solutions.

SECTION 2 PREPARATION FOR MEASUREMENT

2.1 REQUIRED EQUIPMENT

1. A pH/mV meter or an ion meter, either line operated or portable.
2. Semi-logarithmic 4-cycle graph paper for preparing calibration curves when using the meter in the mV mode.
3. A magnetic stirrer.
4. The OMEGA Chloride Ion Electrode, ISE-8760, (reference electrode necessary), or the OMEGA Chloride Combination Ion Electrode, ISE-8770.
5. The OMEGA Reference Electrode, PHE-3211, filled with Salt Bridge Solution, PHFS-B250, to be used with the ISE-8770.
6. Polishing paper or jeweler's rouge to polish dirty or etched electrode membranes.

2.2 REQUIRED SOLUTIONS

1. Deionized or distilled water for solution and standard preparation.
2. OMEGA Ionic Strength Adjustor (ISA), 5M NaNO₃, ISE-8720-R1. This solution provides a constant background ionic strength. To prepare ISA from your own stock, half fill a 100 mL volumetric flask with distilled water and add 42.5 grams of reagent grade sodium nitrate. Swirl the flask to dissolve the solid. Finally, fill the flask to the mark with distilled water.

Prepare standard solutions with a composition similar to the samples if the samples have an ionic strength above 0.1M.

3. OMEGA Chloride Standard, 0.1M NaCl, ISE-8770-S1. To prepare this solution from your own stock, half fill a 1 liter volumetric flask with distilled water and add 5.84 grams of reagent grade NaCl. Swirl the flask gently to dissolve the solid. Fill the flask to the mark with distilled water.
4. OMEGA Chloride Standard, 1000 ppm Cl⁻¹, ISE-8770-S2. To prepare this solution from your own stock, half fill a 1 liter volumetric flask with distilled water and add 1.65 grams of reagent grade NaCl. Swirl the flask to dissolve the solid. Fill to the mark with distilled water.

2.3 ELECTRODE PREPARATION

Remove the rubber cap covering the electrode tip and the rubber band covering the filling hole of the reference electrode.

Connect the electrode to the proper terminals as recommended by the meter manufacturer.

NOTE

Do not be alarmed if white crystals form at end of electrode. This is normal with pH electrodes. These crystals are potassium chloride. Remove the shipping cap and rinse with distilled water to dissolve the crystals.

2.3.1 Electrode Slope Check (with standard pH/mV meter)

Check electrode on a daily basis. To a 150 mL beaker, add 100 mL of distilled water and 2 mL of ISA. Place the beaker on a magnetic stirrer and begin stirring at a constant rate. After assuring that the meter is in the mV mode, lower the electrode tip into the solution.

Using a pipet, add 1 mL of 0.1M or 1000 ppm chloride standard to the beaker. When the reading is stable, record the millivolt reading.

Using a pipet, add 10 mL of the 0.1M or 1000 ppm chloride standard to the beaker. When the reading has stabilized, record the millivolt reading.

Determine the difference between the two readings. A difference of 57 ± 2 mV indicates correct electrode operation, assuming the solution temperature is between 20° and 25°C. See the Troubleshooting section if the potential change is not within this range.

Slope is defined as the change in potential observed when the concentration changes by a factor of 10.

2.3.2 Electrode Slope Check (with ion meter)

Prepare standard chloride solutions whose concentrations vary by tenfold. Use either the 0.1M NaCl or the 1000 ppm stock solutions. Use the serial dilution method for this preparation.

To a 150 mL beaker, add 100 mL of the lower value standard and 2 mL of ISA. Place the beaker on the magnetic stirrer and begin stirring at a constant rate. Lower the electrode tip into the solution.

Assure that the meter is in the concentration mode.

Adjust the meter to the concentration of the standard and fix the value in the memory according to the meter manufacturer's instructions.

Rinse the electrode with distilled water and blot dry.

To a 150 mL beaker, add 100 mL of the higher value standard and 2 mL of ISA. Place the beaker on a magnetic stirrer and begin stirring at a constant rate. Lower the electrode tip into the solution.

Adjust the meter to the concentration of the standard and fix the value in the memory.

Read the electrode slope according to the meter manufacturer's instructions. Correct electrode operation is indicated by a slope of 93-100%. See Section 5, Troubleshooting, if the potential is not within this range.

SECTION 3 MEASUREMENT PROCEDURE

3.1 MEASURING HINTS

All samples and standards should be at the same temperature for precise measurement.

Constant, but not violent, stirring is necessary for accurate measurement. Magnetic stirrers can generate sufficient heat to change the solution temperature. To counteract this effect, place a piece of insulating material, such as styrofoam sheet or asbestos sheet, between the stirrer and beaker.

Always rinse the electrodes with distilled water and blot them dry between measurements. Use a clean, dry tissue to prevent cross-contamination.

For samples with high ionic strength, prepare standards whose composition is similar to the sample:

A slow-responding electrode may point to deposits on the membrane. Polishing paper or jeweler's rouge should be used to restore performance. If using polishing paper, cut off a small strip and place it on the bench top. Hold the electrode perpendicular to the paper and, with circular motions, polish the electrode tip against the paper. To use jeweler's rouge, put a small amount of the rouge on a piece of cotton and wet the powder with a drop of distilled water. Polish the electrode tip in the same manner as with the polishing paper. Rinse the electrode and blot dry. Soak the electrode in a standard solution for about 5 minutes before use.

3.2 SAMPLE REQUIREMENTS

Inorganic solutions will not affect the epoxy electrode body. Infrequent measurements in solutions containing methanol, benzene, or acetone are permitted. Please check with OMEGA before using this electrode in other organic solvents.

The temperature of the sample solution and of the standard solutions should be the same and below 100°C.

3.3 UNITS OF MEASUREMENT

Chloride concentrations are measured in units of parts per million, equivalents per liter, moles per liter, or any other convenient concentration unit. Table 3-1 indicates some of the concentration units

TABLE 3-1
Concentration Unit Conversion Factors

<u>ppm Cl⁻¹</u>	<u>moles/liter Cl⁻¹</u>
355	1.0 X 10 ⁻²
35.5	1.0 X 10 ⁻³
3.55	1.0 X 10 ⁻⁴

3.4 DIRECT MEASUREMENT (using a standard pH/mV meter)

Direct measurement is a simple procedure for measuring a large number of samples. A single meter reading is all that is required for each sample. The ionic strength of samples and standards should be made the same by adjustment with ISA for all chloride solutions. The temperature of both sample solution and of standard solutions should be the same.

1. By serial dilution, prepare 10⁻², 10⁻³ and 10⁻⁴M or 100 and 10 ppm chloride standards. Add 2 mL of ISA per 100 mL of standard. Prepare samples with a composition similar to the samples if the samples have an ionic strength above 0.1 M.
2. Place the most dilute solution (10⁻⁴M or 10 ppm) on the magnetic stirrer and begin stirring at a constant rate. After assuring that the meter is in the mV mode, lower the electrode tips into the solution. When the reading has stabilized, record the mV reading.
3. Place the midrange solution (10⁻³ M or 100 ppm) on the magnetic stirrer and begin stirring. After rinsing the electrodes in distilled water, blot them dry and immerse the electrode tips in the solution. When the reading has stabilized, record the mV reading.
4. Place the most concentrated solution (10⁻²M or 1000 ppm) on the magnetic stirrer and begin stirring. After rinsing the electrodes in distilled water, blot them dry and immerse the electrode tip in the solution. When the reading has stabilized, record the mV reading.
5. Using the semi-logarithmic graph paper, plot the mV reading (linear axis) against the concentration (log axis). A typical calibration curve can be found in Figure 3-1. Extrapolate the curve down to about 2.0 x 10⁻⁴ M or 7 ppm.

6. To a clean, dry 150 mL beaker, add 100 mL of sample and 2 mL of ISA. Place the beaker on the magnetic stirrer and begin stirring. Place the electrode tip in the solution. When the reading has stabilized, record the mV reading. Determine the concentration directly from the calibration curve.
7. The calibration should be checked every two hours. Assuming no change in ambient temperature, place the electrode tip in the midrange standard. After the reading has stabilized, compare it to the original reading recorded in Step 3. A reading differing by more than 0.5 mV or a change in the ambient temperature will necessitate the repetition of Steps 2-5. A new calibration curve should be prepared daily.

3.5 DIRECT MEASUREMENT (using an ion meter)

1. By serial dilution of the 0.1M or 1000 ppm chloride standard, prepare two chloride standards whose concentration is near the expected sample concentration. Measure out 100 mL of each standard into individual 150 mL beakers and add 2 mL of ISA to each.
2. Place the most diluted solution on the magnetic stirrer and begin stirring at a constant rate. Assure that the meter is in concentration mode.
3. Lower the electrode tip into the solution.
4. Adjust the meter to the concentration of the chloride standard and fix the value in the memory according to the meter manufacturer's instructions after stabilization of the reading.
5. Rinse the electrode with distilled water and blot dry.
6. Place the most concentrated solution on the magnetic stirrer and begin stirring at a constant rate.
7. Lower the electrode tip into the solution.
8. Adjust the meter to the concentration of the chloride standard and fix the value in the memory according to the meter manufacturer's instructions after stabilization of the reading.
9. For low level measurements, place the rinsed, dried electrodes into the solution containing 100 mL of distilled water and 2 mL of ISA. After stabilization, fix the blank value in the meter according to the meter manufacturer's instructions.
10. Place 100 mL of the sample and 2 mL of ISA in a 150 mL beaker, place it on the magnetic stirrer, and begin stirring.

11. Immerse the electrode tip in the solution and wait for the reading to stabilize. Read the concentration directly from the meter display.
12. The calibration should be checked every two hours. Assuming no change in ambient temperature, place the electrode tip in the first chloride standard. After the reading has stabilized, compare it to the original reading in Step 4. A reading differing by more than 0.5 mV or a change in the ambient temperature will necessitate the repetition of Steps 2-8. The meter should be re-calibrated daily.

3.6 Low Level Determinations (using a standard pH/mV meter)

This procedure is recommended for solutions with ionic strengths less than $1.0 \times 10^{-2}M$. If the solution is high in ionic strength, but low in chloride, use the same procedure, but prepare a calibration solution with a composition similar to the sample.

1. Using 20 mL of standard ISA, dilute to 100 mL with distilled water. This low level ISA is added at the rate of 1 mL low level ISA to each 100 mL of sample. The background ionic strength will be $1.0 \times 10^{-2}M$.
2. Dilute 10 mL of 0.1M standard to 100 mL to prepare a $1.0 \times 10^{-2}M$ standard solution for measurements in moles per liter. Use the 1000 ppm standard for measurements in ppm. Standards should be prepared fresh daily.
3. Add 1 mL of the low level ISA to a 100 mL volumetric flask and fill to the mark with distilled water. Pour this solution into a 150 mL beaker and place the beaker on the magnetic stirrer. Begin stirring at a constant rate.
4. Place the electrode tip in the solution. Assure that the meter is in the mV mode.
5. Add increments of the $1.0 \times 10^{-2}M$ or 1000 ppm standard as given in Table 3-2.
6. After the reading has stabilized, record the mV reading after each addition.

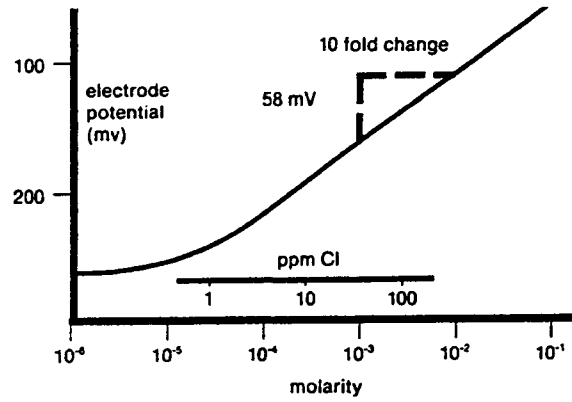


Figure 3-1 Typical Chloride Electrode Calibration Curve

TABLE 3-2
Stepwise Calibration for Low Level Chloride Measurements

<u>Step</u>	<u>Pipet</u>	<u>Added Volume (mL)</u>	<u>Concentration</u>	
			<u>M</u>	<u>ppm</u>
1	A	0.1	1.0×10^{-5}	1.0
2	A	0.1	2.0×10^{-5}	2.0
3	A	0.2	4.0×10^{-5}	4.0
4	A	0.2	6.0×10^{-5}	6.0
5	A	0.4	9.9×10^{-5}	9.9
6	B	2	2.9×10^{-4}	29
7	B	2	4.8×10^{-4}	48

Pipet A = 1 mL graduated pipet

Pipet B = 2 mL pipet

Solutions: additions of 1000 ppm or 1.0×10^{-2} M standard to 100 mL of ISA as prepared in Step 3.

7. On semi-logarithmic graph paper, plot the millivolt reading (linear axis) against the concentration (log axis) as in Figure 3-1.
8. Rinse the electrode and blot dry.
9. Measure out 100 mL of the sample into a 150 mL beaker, add 1 mL of low level ISA, and place the beaker on the magnetic stirrer. Begin stirring. Lower the electrode tip into the solution. After the reading has stabilized, record the mV reading and determine the concentration from the low level calibration curve.
10. Prepare a new low level calibration curve daily. Check the calibration curve every two hours by repeating Steps 2-7.

SECTION 4 ELECTRODE CHARACTERISTICS

4.1 REPRODUCIBILITY

Direct electrode measurements reproducible to $\pm 2\%$ can be obtained if the electrode is calibrated every hour. Factors such as temperature fluctuations, drift, and noise limit reproducibility. Reproducibility is independent of concentration within the electrode's operating range.

4.2 INTERFERENCES

All chloride samples must be free of mercury. Insoluble salts of silver may be deposited on the membrane, causing electrode malfunction, if high levels of ions forming these salts are present in the sample. A layer of silver may form on the electrode surface in the presence of strongly reducing solutions. Proper performance can be restored by polishing.

Solutions containing oxidizing agents such as MnO^{-1} , Fe^{+3} , and Cu^{+2} , may be measured without problem.

The maximum allowable ratio of interfering ion to chloride ion is given in Table 4-1. This ratio is expressed as the ratio of the interfering ion molarity to the chloride molarity. Readings will be in error if this ratio is exceeded. Neither accuracy of the measurement nor surface of the electrode membrane will be affected if the ratio is less than that listed in the table.

TABLE 4-1
Maximum Allowable Ratio of Interfering Ion to Chloride Ion

<u>Interferences</u>	<u>Interfering Ion (M)</u> <u>Chloride Ion (M)</u>
OH^{-1} (1)	80
NH_3 (2)	1.2×10^{-1}
$\text{S}_2\text{O}_3^{2-}$ (2)	1.0×10^{-2}
BR^{-1} (3)	3.0×10^{-3}
S^{-2} (4)	1.0×10^{-6}
I^{-1} (3)	5.0×10^{-7}
CN^{-1} (4)	2.0×10^{-7}

- (1) Acidity with 1 M HNO_3 to pH=4 to remove hydroxide interferences.
- (2) These substances represent complexing species whose maximum level can be exceeded without electrode damage. Value shown represents a 1% error.
- (3) Add CISA to solutions containing mixed halides to remove interferences.
- (4) Add CISA or a solution of Ni^{+2} to remove sulfide or cyanide interferences.

4.3 REMOVAL OF INTERFERENCES WITH CISA

CISA is an oxidizing agent which will oxidize up to a 100-fold excess of CN^{-1} over Cl^{-1} , 100 mg/liter NH_3 , 100 mg/liter BR^{-1} or I^{-1} , or 500 mg/liter S^{-2} . Chloride measurement interferences may be removed by using CISA. The reagents used to prepare CISA are strong oxidizing agents and should be handled in a fume hood.

To prepare CISA, add approximately 800 mL of distilled water to a 1 liter volumetric flask. Add 15.1 grams of NaBrO_3 to the flask and swirl to dissolve the solid. Slowly add 75 mL of concentrated nitric acid (70% w/w or 15.9N), mix, and dilute to the mark with distilled water.

To use CISA, mix equal amounts of CISA and sample. Solutions should be allowed to stand for ten minutes before measuring. Since chloride will be oxidized upon prolonged standing, all standards mixed with CISA should be discarded after measuring. A fresh mixture of CISA and standard should be prepared for each calibration. After adding CISA, follow the procedures for direct measurement.

4.4 TEMPERATURE INFLUENCES

Samples and standards should be within $\pm 1^\circ\text{C}$ of each other, since electrode potentials are influenced by changes in temperature. Because of the solubility equilibria on which the electrode depends, the absolute potential of the reference electrode changes slowly with temperature. The slope of the electrode, as indicated by the factor "S" in the Nernst equation, also varies with temperature. Table 4-2 gives values for the "S" factor in the Nernst equation for each ion.

TABLE 4-2
Temperature vs. Values for the Electrode Slope

<u>Temp</u> ($^\circ\text{C}$)	<u>S</u> "
0	54.2
10	56.2
20	58.2
25	59.2
30	60.1
40	62.1
50	64.1

If changes in temperature occur, the electrodes should be re-calibrated.

The temperature range for the Chloride Ion Selective Electrodes is $0^\circ\text{--}100^\circ\text{C}$, provided that temperature varies substantially from room temperature, equilibrium times up to one hour are recommended.

Only intermittent use is recommended at temperatures above 80°C .

4.5 ELECTRODE RESPONSE

Plotting the electrode mV potential against the chloride concentration on semi-logarithmic paper results in a straight line with a slope of about 58 mV per decade. Refer to Figure 4-1.

The time needed to reach 99% of the stable electrode potential reading, the electrode response time, varies from several seconds in highly concentrated solutions to several minutes near the detection limit. Refer to Figure 4-1.

4.6 COMPLEXATION

Total concentration (C_t) consists of free ions (C_f) and complexed or bound ions (C_c) in solution:

$$C_t = C_f + C_c$$

Since the electrode only responds to free ions, any complexing agent in the solution reduces the measured concentration of ions.

Chloride ions complex with some metal ions. Table 4-3 lists the levels of complexing metals causing a 10% error at $1.0 \times 10^{-4}M$ chloride.

TABLE 4-3
Levels of Complexing Agents Causing a 10% Error at
 $1.0 \times 10^{-4}M$ Chloride

<u>Ion</u>	<u>Concentration</u>
i^{+3}	$4.0 \times 10^{-4}M$ (80 ppm)
Cd^{+2}	$2.0 \times 10^{-3}M$ (200 ppm)
Mn^{+2}	$2.0 \times 10^{-2}M$ (1100 ppm)
Pb^{+2}	$2.0 \times 10^{-3}M$ (400 ppm)
Sn^{+2}	$6.0 \times 10^{-3}M$ (700 ppm)
Tl^{+3}	$4.0 \times 10^{-5}M$ (8 ppm)

4.7 ELECTRODE STORAGE

The electrode is stored dry after rinsing well and blotting dry. The reference electrode is stored according to directions given with the specific electrode.

4.8 THEORY OF OPERATION

The Chloride Ion Electrode is composed of a membrane bonded into an epoxy body. When an electrode potential develops across the membrane, the membrane is in contact with a solution containing chloride ions and is capable of measuring free chloride ions and is capable of measuring free chloride ions. This electrode potential is measured against a constant reference potential, using a standard pH/mV meter or an ion meter. The level of chloride ions, corresponding to the measured potential, is described by the Nernst equation:

$$E = E_o + S \log X$$

where:

- E = measured electrode potential
- E_o = reference potential (a constant)
- S = electrode slope (-58 mV)
- X = level of chloride ions in solution

The activity, X, represents the effective concentration of the ions in solution. The activity is related to the free ion concentration, C_f , by the activity coefficient, f_i , by:

$$X = C_f$$

Activity coefficients vary, depending on total ionic strength, I, defined as:

$$I = 1/2 \sum C_x Z_x^2$$

where:

C_x = concentration of ion X

Z_x = charge of ion X

Σ = sum of all of the types of ions in the solution.

In the case of high and constant ionic strength relative to the sensed ion concentration, the activity coefficient, f_i , is constant and the activity, X , is directly proportional to the concentration.

To adjust the background ionic strength to a high and constant value, ionic strength adjustor is added to samples and standards. The recommended ISA for chloride is NaNO_3 . Solutions other than this may be used as ionic strength adjustors as long as ions that they contain do not interfere with the electrode's response to chloride ions. Samples with high ionic strength (greater than 0.1M) do not need ISA added and standards for these solutions should be prepared with a composition similar to the samples.

The reference electrode must also be considered. When two solutions of different composition are brought into contact with one another, liquid junction potentials arise. Millivolt potentials occur from the interdiffusion of ions in the two solutions. Electrode charge will be carried unequally across the solution boundary resulting in a potential difference between the two solution, since ions diffuse at different rates. When making measurements, it is important to remember that this potential be the same when the reference is in the standardizing solution as well as in the sample solution or the change in liquid junction potential will appear as an error in the measured electrode potential.

The composition of the liquid junction filling solution in the reference electrode is most important. The speed with which the positive and negative ions in the filling solution should be equitransferent. No junction potential can result if the rate at which positive and negative charge carried into the sample is equal.

Strongly acidic (pH=0-2) and strongly basic (pH=12-14) solutions are particularly troublesome to measure. The high mobility of hydrogen and hydroxide ions in samples make it impossible to mask their effect on the junction potential with any concentration of an equitransferent salt. One must either calibrate the electrodes in the same pH range as the sample or use a known increment method for ion measurement.

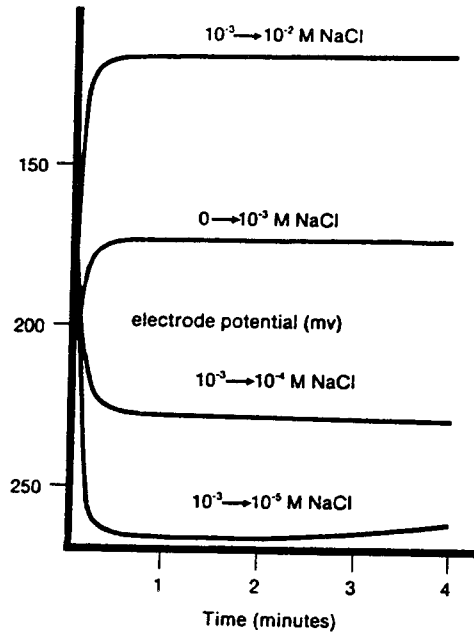


Figure 4-1 Typical Electrode Response to Step Changes in NaCl

SECTION 5 TROUBLESHOOTING

SYMPTOM	POSSIBLE CAUSES	NEXT STEP
Out-of-range reading	Defective meter	Check meter with shorting strap
	Electrode not plugged in properly	Reseat electrode
	Reference electrode junction is dry	Hold cap and lift outer sleeve to expel a few drops of fill solution
	Reference electrode not filled	Be sure inner and outer chambers of double junction electrode are filled
	Calibration control not turned far enough	Continue turning calibration control
	Meter or stirrer not grounded	Ground meter or stirrer
	Static electricity	Wipe plastic meter face with detergent
"Incorrect answer" (but calibration curve is good)	Reference electrode junction clogged	Clean out junction
	Incorrect scaling of semi-log paper	Plot millivolts on the linear axis. On the log axis, be sure concentration numbers within each decade are increasing with increasing concentration.
	Incorrect sign	Be sure to note sign correctly
	Bad samples	Be sure ISA is added to sample

SYMPTOM	POSSIBLE CAUSES	NEXT STEP
	Incorrect standards	Prepare fresh standards
	Wrong units used	Apply correct conversion factor: $10^{-3} \text{ M} = 35.5 \text{ ppm Cl}^{-1}$
Drift (reading changing in one direction)	Samples and standards at different temperature	Allow solutions to come to the same temperature before measurement
	Incorrect reference filling	Use recommended fill solution in outer chamber of reference electrode
	Membrane dirty or etched	Polish membrane
Low slope or no slope	Standards contaminated or incorrectly made	Prepare fresh standards
	Standard used as ISA	Use ISA

SECTION 6 SPECIFICATIONS

CONCENTRATION RANGE: 10 to 5×10^{-5} M (35,000 to 1.8 ppm)

pH RANGE: 2 to 12

TEMPERATURE RANGE: 0° - 80°C

RESISTANCE: <1 Mohm

SIZE:
110 mm in Length
12 mm in Diameter
1 m Cable Length



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