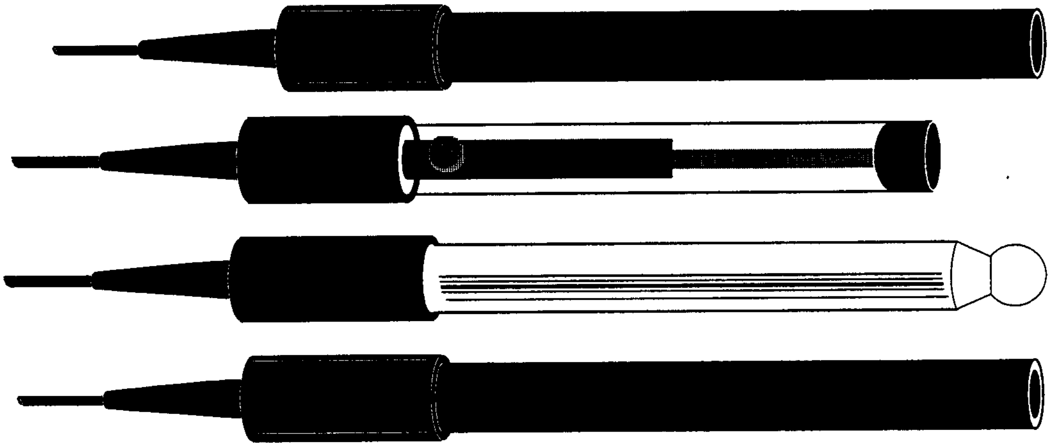
ISE-8765 & ISE-8775

Sodium Ion Selective Electrodes



OMEGA®

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Operator's Manual

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SECTION 1 GENERAL DESCRIPTION

The OMEGA® Sodium Ion Electrodes are used to quickly, simply, accurately, and economically measure sodium 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 Sodium Ion Electrode, ISE-8775, (reference electrode necessary), or Sodium Combination Ion Electrode, ISE-8765.
5. The Double Junction Reference Electrode, PHE-3211, with filling solution, PHFS-8710 in the outer junction.

2.2 REQUIRED SOLUTIONS

1. Deionized or distilled water for solution and standard preparation.
2. ISA (ionic strength adjuster) to adjust the pH and to keep a constant background ionic strength present in the solution, ISE-8765-R1. To prepare the ISA, add 20.0 grams of reagent grade ammonium chloride (NH_4Cl) to a 100 mL volumetric flask about half-full of distilled water. Add 27.0 mL of concentrated ammonium hydroxide (NH_4OH) and dilute to the mark with distilled water.
3. Electrode storage solution for sodium ion electrodes, ISE-8745-R1. To prepare from your own stock, add 29.2 grams of reagent grade sodium chloride (NaCl) to 100 mL of distilled water to produce a 5M NaCl stock solution. To each 100 mL of storage solution, add 2 mL of ISA. ELECTRODES MUST NOT BE STORED IN DISTILLED WATER OR AIR.
4. Dilute ISA for electrode rinsing. To a 1 liter volumetric flask, add 20 mL of ISA and dilute to the mark with distilled water. Use this solution to rinse the electrode between measurements. DO NOT RINSE WITH DISTILLED WATER.
5. Sodium Standard, 0.1M NaCl, ISE-8765-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.

6. Sodium Standard, 1000 ppm Na^+, ISE-8765-S2. To prepare this solution from your own stock, half fill a 1 liter volumetric flask with distilled water and add 2.542 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. Fill the combination electrode or the reference electrode salt bridge with the appropriate filling solution. Soak the electrode tip overnight in the electrode storage solution.

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 the 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. If drifting or instability is observed, see the Troubleshooting section.

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

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

Determine the difference between the two readings. A difference of 56 +/-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 a factor of 10.

2.3.2 Electrode Slope Check (with ion meter)

Prepare standard sodium solutions whose concentrations vary by tenfold. Use either the 0.1M NaCl or the 1000 ppm Na^+ standard 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 the electrode rinse solution 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 90-100%. See the Troubleshooting section if the slope 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, preferably ambient temperature. The electrodes should not be used above 70°C.

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 or asbestos sheet, between the stirrer and the beaker.

Always rinse the electrode with electrode rinse solution from a wash bottle to prevent solution carryover. Never use distilled water. Store the electrode in electrode storage solution between measurements. Do not store in air or distilled water. Always soak new electrodes overnight in electrode storage solution prior to first use. When making low level sodium measurements, use a dilute sodium chloride storage solution, adjusting the pH by adding ISA, for a storage medium.

Plastic labware should be used for low level measurements.

All measurements should be made in basic solution. All samples and standards should be adjusted to a pH above 9 with ISA.

DO NOT ATTEMPT TO ETCH THE ELECTRODE BULB WITH AMMONIUM BIFLUORIDE UNDER ANY CIRCUMSTANCES.

3.2 SAMPLE REQUIREMENTS

The sample measuring range is pH 9-12. Use the ISA recommended to adjust the pH for best accuracy. Make sure that the samples and standards are at the same temperature. The glass electrode sensing bulb will not be attacked by most organic solvents.

3.3 UNITS OF MEASUREMENT

Sodium 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

ppm Na^+	moles/liter Na^+
229.90	1×10^{-2}
22.99	1×10^{-3}
2.30	1×10^{-4}

3.4 DIRECT MEASUREMENT (using 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 sodium 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^{-4} M or 100 and 10 ppm sodium standards. Add 2 mL of ISA per 100 mL of standard.
2. Place the most dilute solution (10^{-4} M 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 tip 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 electrode with electrode rinse solution, blot dry and immerse the electrode tip into the solution. When the reading is stabilized, record the mV reading.

4. Place the most concentrated solution (10^{-2} M or 1000 ppm) on the magnetic stirrer and begin stirring. After rinsing the electrode with electrode rinse solution, blot 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.

A calibration curve is constructed on semi-logarithmic paper when using a pH/mV meter in the direct measurement procedure. The measured electrode potential in mV (linear axis) is plotted against the standard concentration (log axis). In the linear region of the curve, only three standards are necessary to determine a calibration curve. In the non-linear region, additional points must be measured. The direct measurement procedures given are for the linear portion of the curve. The non-linear portion of the curve requires the use of low level sodium determination procedure.

Extrapolate the curve down to about 5×10^{-5} M or 1 ppm. For measurements below this level, follow the instructions for low level measurement.

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 electrode should be re-calibrated every 1-2 hours. Repeat steps 2-5.

3.5 DIRECT MEASUREMENT (using an ion meter)

1. By serial dilution of the 0.1 M or 1000 ppm sodium standard, prepare two sodium 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 dilute solution on the magnetic stirrer and begin stirring at a constant rate. Assure that the meter is in the concentration mode.

3. Lower the electrode tip into the solution.

4. Adjust the meter to the concentration of the sodium 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 electrode rinse solution and blot dry.

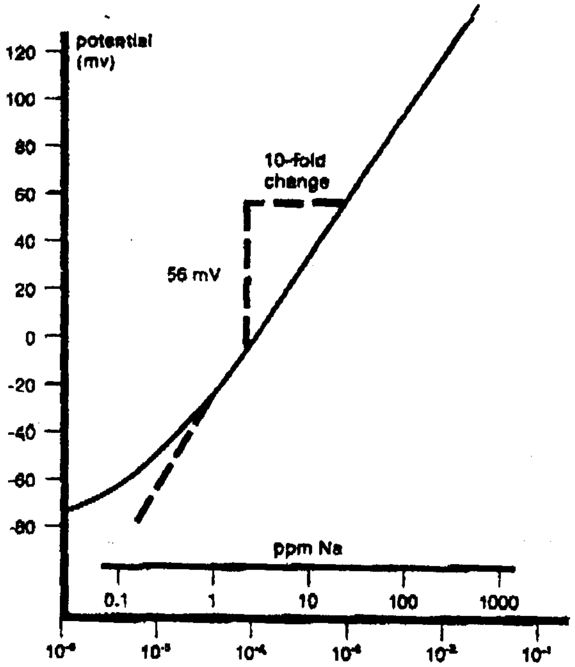

Figure 3-1 Typical Sodium Electrode Calibration Curve

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 sodium standard and fix the value in the memory according to the meter instructions, after stabilization of the reading.

9. For low level measurements, place the rinsed, dried electrode into a 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 electrode should be recalibrated every 1-2 hours. Repeat steps 2-9.

3.6 LOW LEVEL SODIUM DETERMINATION

This procedure is recommended for solutions with ionic strength less than 1×10^{-2} M. If the solution is high in ionic strength, but low in sodium, use the same procedure, but prepare a calibration solution with a composition similar to the sample. Use plastic labware for low sodium measurements.

1. Using 20 mL of standard ISA, dilute to 100 mL with distilled water.

2. Dilute 20 mL of the outer chamber filling solution to 100 mL with distilled water and fill the reference electrode if using a double junction reference electrode.

3. Dilute 1 mL of the 0.1 M standard to 100 mL to prepare a 1×10^{-3} M standard solution for measurements in moles per liter. Dilute 10 mL of the 1000 ppm standard solution to prepare a 100 ppm standard solution for measurements in ppm. Add 1 mL of low level ISA to each 100 mL of standard. Standards should be prepared fresh daily.

4. To a 150 mL plastic beaker, add 100 mL of distilled water and 1 mL of low level ISA. Add NH_4OH, if necessary, to adjust the pH above 9. Place the beaker on the magnetic stirrer and begin stirring at a constant rate.

5. Place the electrode tip in the solution. Assure that the meter is in the mV mode.

6. Add increments of the 1 x 10^{-3} M or 100 ppm standard as given in Table 3-2 below.

7. After the reading has stabilized, record the mV reading after each addition.

TABLE 3-2
Stepwise Calibration for Low Level Sodium Measurements

Step	Pipet	Added Volume (mL)	Concentration M	ppm
1	A	0.1	1 x 10-6	0.10
2	A	0.1	2 x 10-6	0.20
3	A	0.2	4 x 10-6	0.40
4	A	0.2	6 x 10-6	0.60
5	A	0.4	9.9 x 10-6	0.99
6	B	2.0	2.9 x 10-5	2.91
7	B	2.0	4.8 x 10-5	4.76

Pipet A = 1 mL graduated pipet
Pipet B = 2 mL pipet
Solution = additions of 1 x 10-3 M or 100 ppm standard to 100 mL of distilled water and 1 mL of low level ISA.

8. On semi-logarithmic graph-paper, plot the millivolt reading (linear axis) against the concentration (log axis) as in Figure 3-1.

9. Rinse the electrode in electrode rinse solution and blot dry.

10. Measure out 100 mL of the sample into a 150 mL plastic beaker, and add 1 mL of low level ISA. Place the beaker on the magnetic stirrer, and begin stirring. Adjust the pH, if necessary, to above 9. 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.

11. Prepare a new low level calibration curve daily. Check the calibration curve every two hours by repeating steps 3-8.

For low level sodium determination using an ion meter, follow the procedure given for normal sodium determination using an ion meter and the blank correction procedure. Use plastic labware for low level sodium measurement.

SECTION 4 ELECTRODE CHARACTERISTICS

4.1 REPRODUCIBILITY

Direct electrode measurements reproducible to +/-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

Table 4-1 lists some common cations that, if present in high enough levels, will cause electrode interferences and measurement errors or electrode drift when using the sodium ion electrode.

Most samples do not contain or contain very little of the cations shown in Table 4-1. The ammonium ion (NH_4^+), found in the recommended ISA, will not result in an error if all samples and standards have the same level of ISA present.

Electrode drift and slow response could indicate the presence of high interference from the ions listed. Soak the electrodes in electrode storage solution when this happens, to restore proper response.

TABLE 4-1
Levels of Interfering Ions Resulting in a 10% Error at Specified Levels of Sodium

Interference	1 x 10^{-4} M (1 ppm) Na^+	1 x 10^{-3} M (10 ppm) Na^+	1 x 10^{-2}M (100 ppm) Na^+
Li^+	5 x 10^{-4} M (2 ppm)	5 x 10^{-3} M (1.5 x 10^1 ppm)	5 x 10^{-2} M (1.5 x 10^2 ppm)
K^+	1 x 10^{-2} M (1.7 x 10^2 ppm)	1 x 10^{-1} M (1.7 x 10^3 ppm)	1 M (1.7 x 10^4 ppm)
Rb^+	3 x 10-1 M (1.1 x 10^4 ppm)	3M (1.1 x 10^5 ppm)	-
NH^+4	3 x 10-1 M (1.8 x 10^3 ppm)	3M (1.8 x 10^4 ppm)	-
Ag^+	3 x 10^{-9} M (1 x 10^{-4} ppm)	3 x 10^{-8} M (1 x 10^{-7} ppm)	3 x 10^{-7} M (1 x 10^{-2} ppm)
Tl^+	5 x 10^{-2} M (4.5 x10^3 ppm)	5 x 10^{-1} M (4.5 x 10^4 ppm)	-

4.3 TEMPERATURE INFLUENCES

Samples and standards should be at the same temperature, since electrode potentials are influenced by changes on temperature.

Provided that temperature equilibria has occurred, the sodium ion electrode can be used at temperatures from -5°C to 70°C. Room temperature measurements are recommended, since measurements at temperatures markedly different from room temperature may require equilibrium time up to one hour. The electrode should not be used at temperatures above 70°C, since damage to the membrane may result. Table 4-2 indicates the variation of theoretical slope with temperature.

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

Temp.°C	"S"
0	54.20
10	56.18
20	58.16
25	59.16
30	60.15
40	62.13
50	64.11

4.4 ELECTRODE RESPONSE

Plotting the electrode mV potential against the sodium concentration on semi-logarithmic paper results in a straight line with a slope of about 56 mV per decade. Refer to Figure 3-1.

The time needed to reach 99% of the stable electrode potential reading, the electrode response time, varies from one minute or less for sodium concentration above 1×10^{-5} M to several minutes near the detection limit. Refer to Figure 4-1.

A hydrated layer forms on the surface of the sodium sensitive glass, which results in increased response time, after prolonged use. The electrode should be stored in electrode storage solution overnight to restore normal response.

4.5 ELECTRODE STORAGE

The sodium ion electrode should be stored in the electrode storage solution, never in air or distilled water. A more dilute sodium chloride solution, with pH adjusted through the use of ISA, may be used for storage before low level measurements.

4.6 LIMITS OF DETECTION

Free sodium ion concentration down to 1×10^{-6} M can be measured in basic solutions. For measurements below 10^{-5} M, use plastic labware, since a significant pickup of sodium may occur from glassware due to desorption from container walls.

4.7 pH EFFECTS

The electrode response to sodium ions is greatly influenced by the pH of the solution. Hydrogen ion interferes with measurements of low level sodium ion measurements, although the electrode can be used over a wide pH range. See Figure 4-2.
The edge of the shaded area (the straight line) indicates a minimum pH at which dilute sodium measurements can be made with less than 10% hydrogen ion interference.

The pH should be adjusted to a pH greater than 9 by the addition of ISA to all standards and samples for optimal results over the entire concentration range of sodium. Additional ammonium hydroxide may be necessary to adjust the pH to the desired level in some cases.

4.8 THEORY OF OPERATION

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

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

where:

E = measured electrode potential
Eo = reference potential (a constant)
S = electrode slope
X = level of sodium in solution

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

$$X = \gamma \; Cf$$

Activity coefficients may vary, depending on the total ionic strength, I, determined as:

$$I = 1/2 \; \Sigma \; C_x Z_x 2$$

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 ionic concentration, the activity coefficient, γ is the 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 adjuster is added to samples and standards. The recommended ISA for sodium is an ammonium chloride-ammonia buffer. Solutions other than this may be used as ionic strength adjusters as long as ions that they contain do not interfere with the electrode's response to sodium ions.

The reference electrode must also be considered. When two solutions of different compositions are brought into contact with one another, liquid junction potentials arise. Millivolt potentials occur from the interdiffusion of ions into the two solutions. Electrode charge will be carried unequally across the solution boundary resulting in a potential difference between the two solutions, 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 solutions diffuse into the sample should be as nearly equal as possible; that is, the filling solution should be equitransferent. No junction potential can result if the rate at which positive and negative charge carried in the sample is equal.

Strongly acid (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 makes it impossible to mask their effect on the junction potential with any concentration of an equitransferent salt. One must either calibrate the electrode in the same pH range as the samples or use a known increment method for ion measurement.

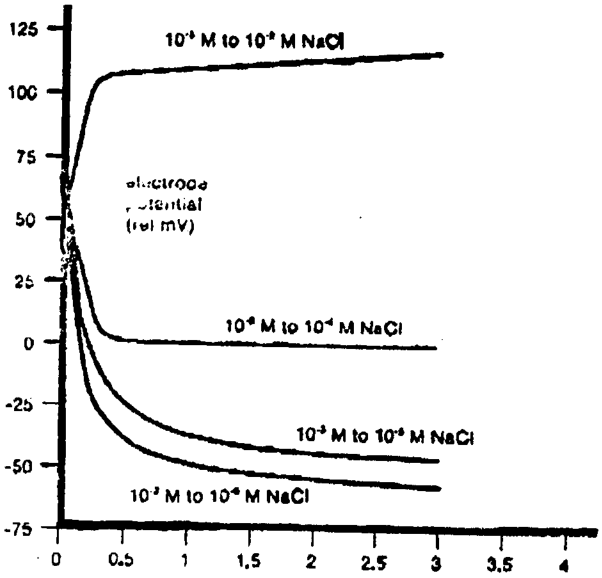

Figure 4-1 Typical Electrode Response to Step Changes in Sodium Chloride Concentration

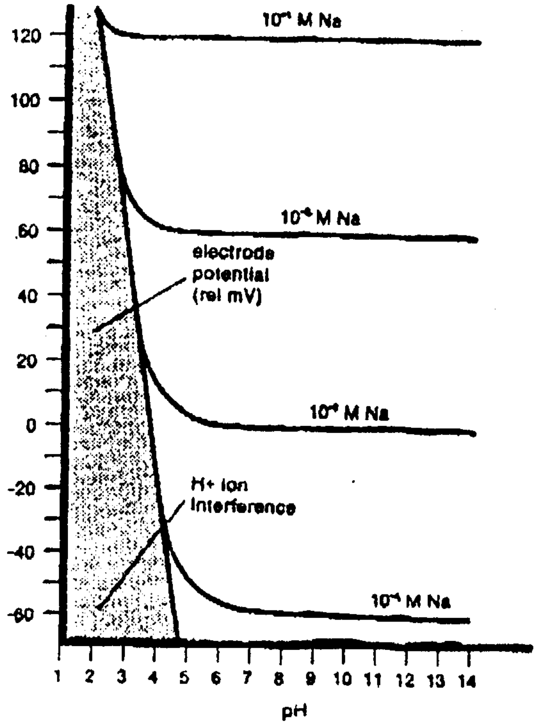

Figure 4-2 Sodium Electrode Potential Behavior vs. Solution pH in Pure NaCl Solution at 25°C

SECTION 5 TROUBLESHOOTING

SYMPTOM	**POSSIBLE CAUSES**	**NEXT STEP**
Out-of-range reading	Defective meter	Check meter with shorting strap (see meter manual).
	Electrode not plugged in properly	Unplug electrode and reseat.
	Reference electrode junction is dry	Replenish reference filling solution.
	Reference electrode not filled	Be sure electrode is filled.
"Incorrect answer" (but calibration curve is good)	Incorrect scaling of semilog paper	Plot millivolts on the linear axis. On the log axis, be sure concentration numbers within each decade are increasing with increasing concentrations.
	Incorrect sign	Be sure to note sign of millivolt reading correctly.
	Incorrect standards	Prepare fresh standards.
	Wrong units	Apply correct conversion factor: 10^{+3} M = 23.0 ppm.
	Sample pH too low	Adjust pH above 9.

Drift (reading slowly changing in one direction)	Samples and standards at different temp.	Allow solutions to come to room temperature before measurement.
	Incorrect reference filling solution	Use recommended filling solution.
	Membrane hydrated or exposed to interferences	Soak overnight in electrode storage solution.
	pH too low	Adjust pH.
	ISA not used	Use recommended ISA.
Low slope or no slope	Standards contaminated or incorrectly made	Prepare fresh standards.
	ISA not used	Use recommended ISA.
	Standard used as ISA	Use ISA.
	pH too low	Adjust pH.
	Membrane hydrated or exposed to interferences	Soak overnight in electrode storage solution.
Noisy or unstable readings	Defective meter	Check meter with shorting strap.
	ISA not used	Use recommended ISA.
	Meter or stirrer not grounded	Ground meter or stirrer.
Meter cannot be calibrated	Calibration control not turned far enough	Continue turning the calibration control.

SECTION 6 SPECIFICATIONS

Concentration Range:	Saturated to 1 x 10^{-6} M (0.02 ppm)
pH Range:	3 to 12 (dependent on Na^+ level)
Temperature Range:	-5° to 70°C
Resistance:	< 200 Mohm
Reproducibility:	+/-2%
Size:	4-1/2" (length) 0.470" (diameter) 3 ft. (standard cable length)
Storage:	Store in 5M NaCl with added ISA

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