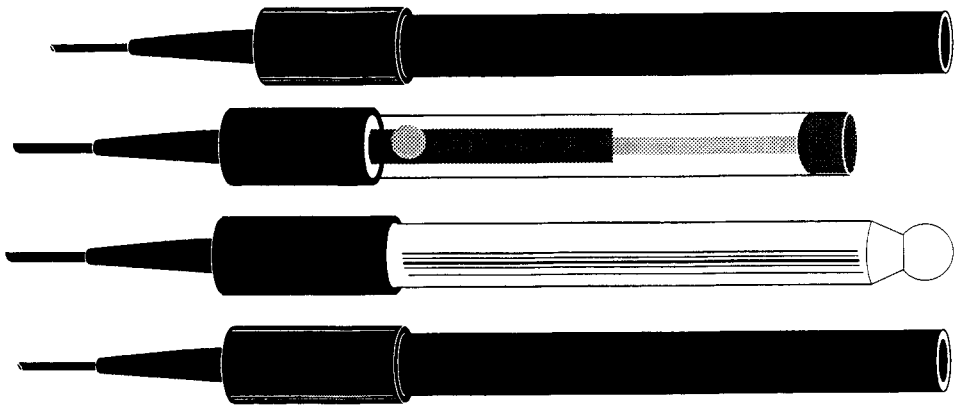
ISE-8720, ISE 8715

Bromide/Iodide
Ion Selective Electrodes



OMEGA®

An OMEGA Technologies Company

Operator's Manual

SECTION 1 GENERAL DESCRIPTION

The OMEGA® Bromide Ion Electrodes and Iodide Ion Electrodes are used to quickly, simply, accurately, and economically measure free bromide and free iodide ion 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, with a readability to 0.1mV.

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 Bromide Ion Electrode, ISE-8720 (reference electrode necessary) or Iodide Ion Electrode, ISE-8715 (reference electrode necessary).

5. The OMEGA Reference Electrode, PHE-3211, filled with Salt Bridge Solution, PHFS-KNO_3.

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 $NaNo_3$, ISE-8720-R1. This solution provides a constant background ionic strength. To prepare ISA from your own stock, half fill a 100mL volumetric flask with distilled water and add 42.5 grams of reagent grade sodium nitrate. Swirl the flask to dissolve solid. Fill the flask to the mark with distilled water, cap, and upend the flask several times to mix the solution.

3. Bromide Standards:
   a) OMEGA Bromide Standard, 0.1M NaBr, ISE-8720-S1. To prepare this solution from your own stock, half fill a one liter volumetric flask with distilled water and add 10.3 grams of reagent grade sodium bromide, NaBr. Swirl the flask gently to dissolve the solid. Fill the flask with distilled water, cap, and upend several times to mix the solution.

b) OMEGA Bromide Standard, 1000ppm Br^{-1}, BROASO2 To prepare this solution from your own stock, half fill a 1 liter volumetric flask with distilled water and add 1.29 grams of reagent grade NaBr. Swirl the flask gently to dissolve the solid. Fill the flask with water, cap, and upend several times to mix the solution.

4. Iodide Standards:
   a) OMEGA Iodide Standards, 0.1M NaI, ISE-8715-S1. To prepare this solution from your own stock, half fill a one liter volumetric flask with distilled water and add 15.0 grams of reagent grade sodium iodide, NaI. Swirl the flask gently to dissolve the solid. Fill the flask with distilled water, cap, and upend the flask several times to mix the solution.

   b) OMEGA Iodide Standard, 1000ppm I^{-1}, ISE-8715-S2. To prepare this solution from your own stock, half fill a volumetric flask with distilled water and add 1.18 grams of reagent grade NaI. Fill the flask with distilled water, cap, and upend several times to mix the solution.

2.3 ELECTRODE PREPARATION

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

Connect the electrodes 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)

The electrodes should be checked 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 the appropriate standard to the beaker. For the bromide ion electrode, use either the 0.1M or the 1000 ppm bromide standard. When the reading is stable, record the millivolt reading.

Using a pipet, add 10 mL of the same standard to the beaker. When the reading is stabilized, record the millivolt reading.

Determine the difference between the two readings. A difference of 58 ± 3 mV indicates correct electrode operation, assuming the solution temperature is 25°C. See Section 5, Troubleshooting, 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 an ion meter)

Prepare standard bromide/iodide solutions whose concentrations vary by tenfold. Use either the 0.1M or the 1000 ppm bromide standard for the bromide ion electrode. Use the 0.1M or the 1000 ppm iodide standard for iodide ion electrode. 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 a 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. a 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 slope is not within this range.

SECTION 3 MEASUREMENT PROCEDURE

3.1 MEASURING HINTS

All samples and standards should be within 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 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.
Dilute concentrated samples (70.1M) before measurement.

Use fresh standards for calibration.

Use 2 mL of ISA for each 100 mL of sample or standard.

Check calibration every two hours by placing the electrode in a fresh 100 mL of the first standard with two mL of ISA added. If the calibration has changed, re-calibrate the electrodes.

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

The temperature of the sample solutions and of the standard solutions should be the same and below 80°C. Intermittent use is permitted from 80°-100°C. About a 2% measurement error will occur for every 1°C temperature difference.

3.3 UNITS OF MEASUREMENT

Bromide and iodide ions can be measured in units of moles/liter, parts/million, or any other convenient concentration units. Table 3-1 indicates some of the concentration units.

TABLE 3-1
CONCENTRATION UNIT CONVERSION FACTORS

moles/liter	$ppmBr^{-1}$	$ppmI^{-1}$
1.0×10^{-4}	8.0	12.7
1.0×10^{-3}	79.9	127
1.0×10^{-2}	799	1,270
1.0×10^{-1}	7,990	12,700

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. The temperature of both sample solutions 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 standards for the ion in question. Add 2mL of ISA per 100 mL of each standard. Prepare standards with a composition similar to the samples if the samples have an ionic strength above 0.1M.

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 100ppm) on the magnetic stirrer and begin stirring. After rinsing the electrode in distilled water, blot them dry and immerse the electrode tip in the solution. When the reading has 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 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 for Bromide can be found in Figure 3-1. A calibration curve for Iodide can be found in Figure 3-2.

6. To a clean, dry 150 mL beaker, add 100 mL of the 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.

3.5 DIRECT MEASUREMENT (using an ion meter)

1. By serial dilution of the 0.1M or 1000 ppm standards for the ion in question, prepare two standards whose concentration is near the expected sample concentration. Measure 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 standard and fix the value in the memory according to the meter manufacturer's instructions after stabilization of the reading.

5. Rinse the electrodes 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 standard and fix the value in the memory according to the meter manufacturer's instructions after stabilization of the reading.

9. For low level bromide 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 into the meter according to the meter manufacturer's instructions. No low level measurement are given for the iodide electrodes, since the electrodes show a linear response down to 2.0×10^{-8}M.

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.

3.6 LOW LEVEL DETERMINATIONS

For the Iodide electrodes no low level determinations are given. Linear response is shown down to 2.0×10^{-8}M.

For the bromide electrodes, low level procedures are suggested if the total ionic strength is less than 1.0 X 10^{-2}M and if the bromide concentration ion is less than 2.0X10-6M (0.2ppm). For samples with high ionic strength, prepare standards with backgrounds similar to the sample.

1. Using 20 mL of the standard ISA, dilute to 100 mL with distilled water. This low level (1.0M $NaNo_3$) 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 X 10^{2}M.

2. Dilute 1 mL of 0.1M standard to 1000 mL to prepare a 1.0 X 10^{-}M standard solution for measurements in moles per liter. Dilute 10 mL of 1000 ppm standard to prepare a 10 ppm standard solution for measurements in ppm.

3. Add 100 mL of distilled water to a 150 mL beaker. Add 1 mL of low level ISA, place the beaker on the magnetic stirrer, and begin stirring at a constant rate.

4. Place the rinsed, dried electrode tip in the solution and assure that the meter is in the mV mode.

5. Add increments of the 1.0 X 10^{-4}M or 10 ppm standard as given in Table 3-2 below.

6. After the reading has stabilized, record the mV reading after each addition. Electrode response time is much larger at these levels. Allow adequate time for the electrode to stabilize.

TABLE 3-2
STEPWISE CALIBRATION FOR LOW LEVEL BROMIDE MEASUREMENTS

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

Pipet A = 1mL graduated pipet
Pipet B = 2mL pipet
Solutions: additions of 1.0 X 10^{-4}M or 10 ppm standard to 100 mL of solutions 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 them dry.

9. Measure out 100 mL of 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.

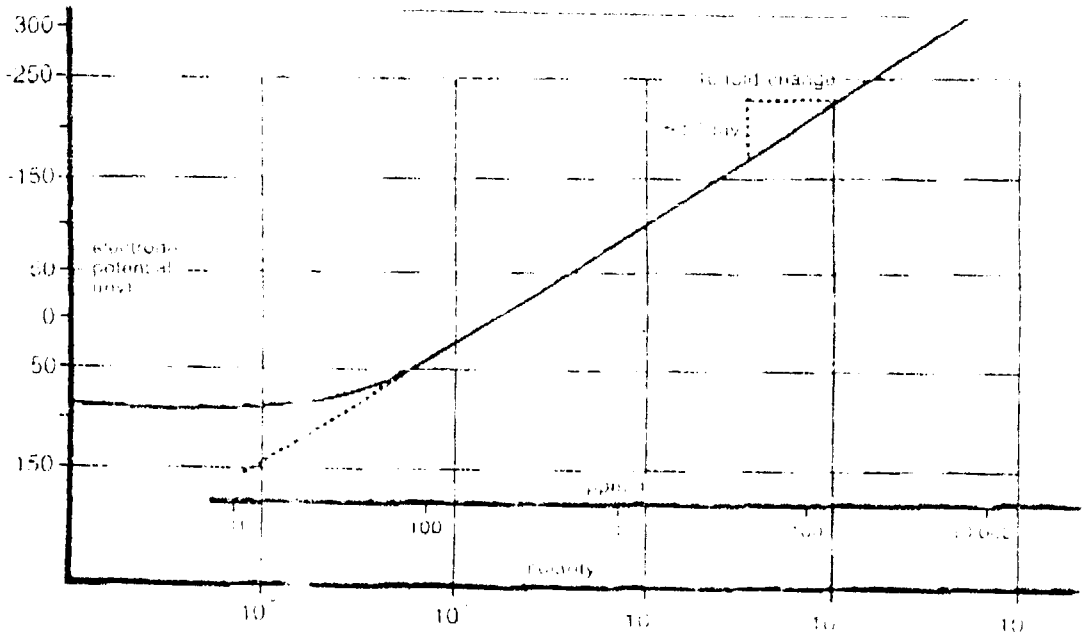


Figure 3-1 Typical Iodide Electrode Calibration Curve

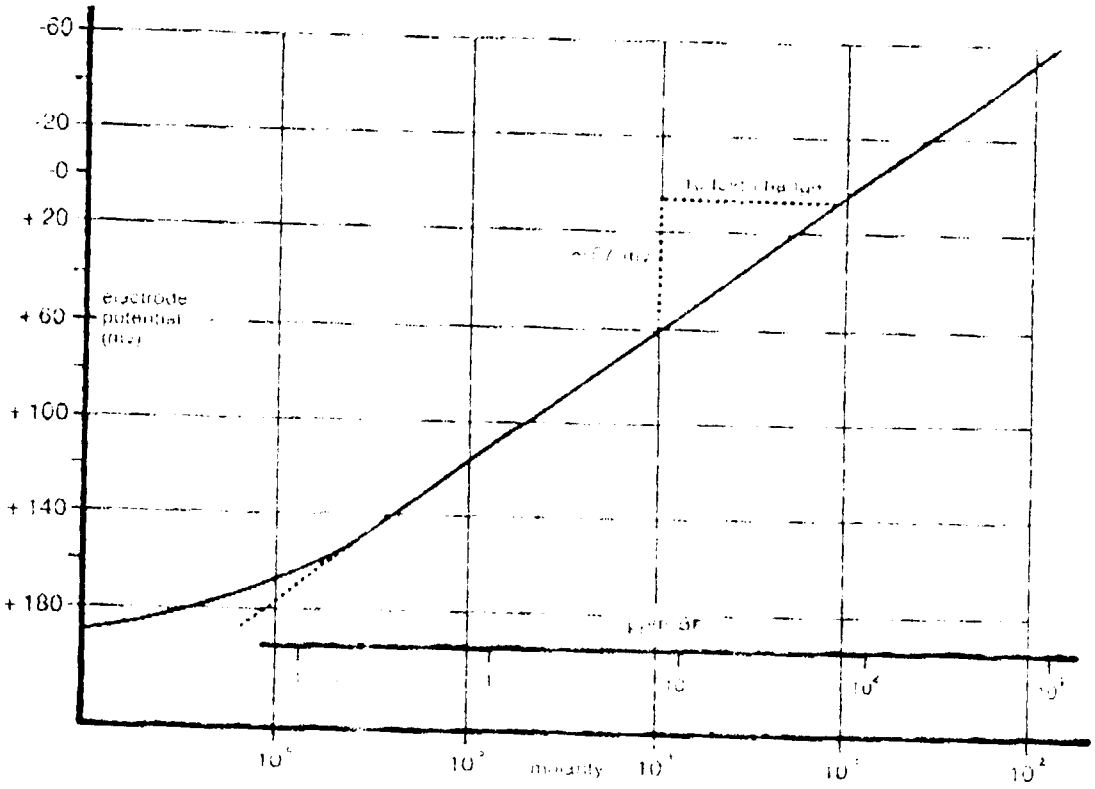


Figure 3-2 Typical Bromide Electrode Calibration Curve

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 bromide or iodide 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_4^{-1}, Fe^{+3}, and Cu-2, may be measured without problem.

The maximum allowable ratio of interfering ion to bromide and to iodide ion is given in Table 4-1. This ratio is expressed as the ratio of the interfering ion molarity to the halide 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 HALIDE ION

Interference		Maximum Ratio for Bromide	Maximum Ratio for Iodide
Cl^{-1}	(1)	4.0×10^{2}	1.0×10^{6}
OH^{-1}	(2)	3.0×10^{4}	–
NH_3	(3)	2.0	–
$S_2O_3^{-2}$	(3)	2.0×10^{1}	1.0×10^{5}
Br^{-1}	(1)	–	5.0×10^{3}
S^{-2}	(4)	1.0×10^{-6}	1.0×10^{-6}
I^{-1}	(1)	2.0×10^{-4}	–
CN^{-1}	(4)	8.0×10^{-5}	4.0×10^{-1}

(1) Gran's plot titration can be used to measure mixed halides in solution.

(2) Activity with HNO_3 to pH=4 to remove hydroxide interference.

(3) These substances represent complexing species whose maximum level can be exceeded without electrode damage. Values shown represents a 1% error.

(4) Add Ni^{+2} to remove sulfide or cyanide interferences.

As an example of Table 4-1's use when using the bromide ion electrode: What is the maximum level of iodide tolerable in a sample whose bromide concentration is 10^{-2}M?

Using Table 4-1, the maximum ratio is:

$$\frac{[I^{-1}]}{[Br^{-1}]} = 2 \times 10^{-4}$$

or $$[I^{-1}] = 2 \times 10^{-4} [Br^{-1}]$$

$$= 2 \times 10^{-4} (1 \times 10^{-2})$$

$[I^{-1}] = 2 \times 10^{-6}$ M maximum iodide concentration for no interference

4.3 TEMPERATURE INFLUENCES

Samples and standards should be within $\pm 1°C$ of each other, since electrode potentials are influenced by changes in temperature. A 1°C difference in temperature results in a 2% error at the 1.0×10^{-3}M concentration level. Because of solubility equilibria on which the electrode depends, the absolute potential of the reference electrode changes slowly with the 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 of the "S" factor in the Nernst equation for the bromide and for the iodide ions.

TABLE 4-2
TEMPERATURE VS. VALUES FOR THE ELECTRODE SLOPE

Temp (°C)	"S"
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 of the Bromide and Iodide Ion Electrodes is 0°-80°C, provided that temperature equilibrium has occurred.

Only intermittent use is recommended at temperatures from 80°-100°C.

4.4 ELECTRODE RESPONSE

Plotting the electrode mV potential against the bromide or iodide concentration on semi-logarithmic paper results in a straight line with a slope of about 58mV per decade. Refer to Figures 3-1 and 3-2.

The time needed to reach 9% 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 for iodide concentrations.

If the electrode response time increases considerably or the mV potential drifts, the membrane may need polishing. Refer to Section 3.1.

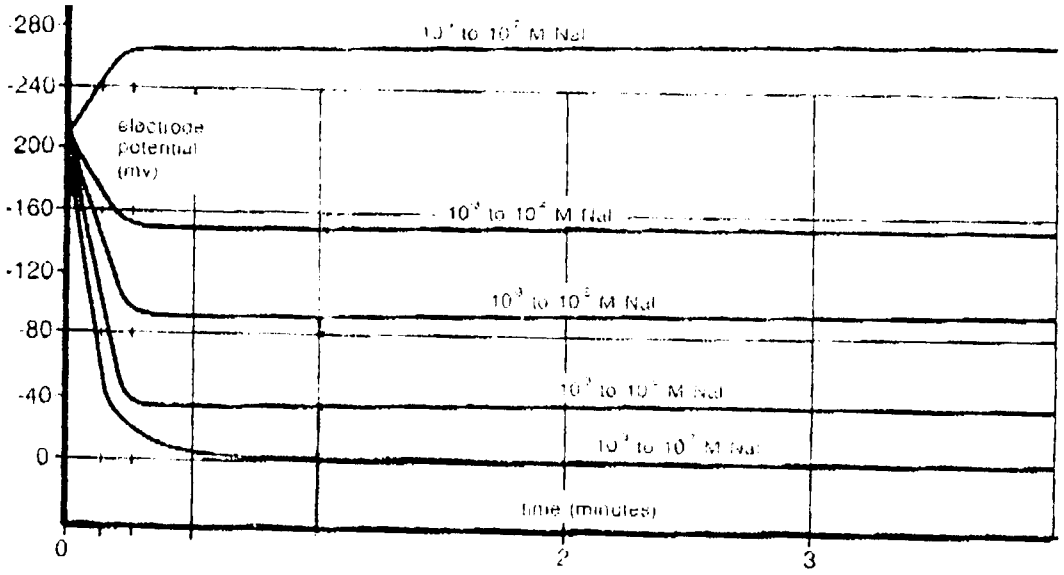


Figure 4-1 Typical Electrode Response to Step Changes in Iodide Concentration

4.5 LIMITS OF DETECTION

Due to the very slight water solubility of the electrode membranes, the electrode respond not only to the halides in the sample, but also to the ions dissolved from the membranes. The solid line (actual response) in Figures 3-1 and 3-2 and the dashed line (theoretical response) show a discrepancy due to response of the electrode to the dissolved membrane at low levels. See Section 3.6 for measurement procedures in the low level range.

4.6 COMPLEXATION

Total concentration (Ct) consists of free ions (Ct) and complexed or bound ions (Cc) in solutions:

$$Ct=Cf + Cc$$

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

Bromide and iodide ions complex with some metal ions. Table 4-3 lists the levels of complexing metals causing a 20% error.

TABLE 4-3
LEVELS OF COMPLEXING AGENTS CAUSING A 20% ERROR

Ion	Concentration	Iodide
Bi^{+3}	4.0×10^{-4}M (80ppm)	2.0×10^{-5}M (4ppm)
Cd^{+2}	2.0×10^{-3}M (200ppm)	5.0×10^{-4}M (50ppm)
Pb^{+2}	8.0×10^{-3}M (1600ppm)	5.0×10^{-3}M (1000ppm)
Sn^{+2}	2.0×10^{-2}M (2400ppm)	
Tl^{+3}	2.0×10^{-5}M (4ppm)	

4.7 ELECTRODE STORAGE

Do not store the electrodes in distilled water.

Between samples, store the bromide and iodide ion electrodes dry in air. For long term storage, rinse the electrode, blot dry, and use the rubber cap to protect the membrane.

4.8 THEORY OF OPERATION

The Bromide Ion Electrodes and the Iodide Ion Electrodes are composed of a sensing membrane bonded into a glass or an epoxy body. The membrane is composed of silver bromide/silver sulfide or silver iodide/silver sulfide. When an electrode potential develops across the membrane, the electrode is in contact with solution containing bromide or iodide ions and is capable of measuring free bromide or iodide ions. This electrode potential is measured against a constant reference potential, using a standard pH/mV meter or an ion meter. The level of the halide 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= measured potential (a constant)
S= electrode slope (-58mV)
X= Level of halide ions 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, f_i, by:

$$x = Cf$$

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

$$I = 1/2 \; \Sigma \; CxZ_x^2$$

where:

Cx= concentration of ion x
Zx= charge of ion x
Σ = sum if all 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 halide ions 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 the halide 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.

SECTION 5 TROUBLESHOOTING

SYMPTOM	POSSIBLE CAUSES	NEXT STEP
Out of Range reading	Defective meter	Perform meter checkout procedure
	Electrodes not plugged in properly	Unplug electrodes and reseat
	Reference electrode junction is dry	Hold reference electrode and push cap to expel a few drops of filling a solution
	No reference electrode	Use OMEGA Reference Electrode
	Reference electrode not filled	Be sure reference electrode is filled
	Electrode not in solution	Put electrodes in solution
"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 concentration
	Incorrect sign	Be sure to note sign of millivolt value correctly
	Incorrect standards	Prepare fresh standards
	Wrong units used	Apply correct conversion factor: 10^{-3}M bromide = 79.9 ppm 10^{-3}M iodide = 127 ppm
	Complexing Agents in sample	Remove complexing agents
	Interference present	Remove interferences

SYMPTOM	POSSIBLE CAUSE	NEXT STEP
Noisy or unstable readings	Defective meter	Perform meter checkout
	Meter or stirrer improperly grounded	Check meter and stirrer for grounding
	Wrong reference electrode	Do not use calomel or Ag/AgCl (frit-or-fiber-type) reference electrode
	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
Drift (Reading slowly changing in one direction)	Samples and standards at different temperature	Allow solutions to come to room temperature before measurement
	Membrane dirty or etched	Polish membrane
	Incorrect reference filling solution	Use recommended filling solution

SECTION 6 SPECIFICATIONS

CONCENTRATION RANGE:	Bromide 1.0 to $5.0X10^{-6M}$ 79.900 to 0.4ppm
pH RANGE:	0-14
TEMPERATURE RANGE:	0° - 80°C (80°-100°C intermittent use)
RESISTANCE:	<0.1 megohm
REPRODUCIBILITY:	$\pm$2%
SIZE:	120mm in length 12mm in diameter 1m cable length

NOTES

NOTES

MADE IN USA

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- ☑ Benchtop/Laboratory Meters
- ☑ Controllers, Calibrators, Simulators & Pumps
- ☑ Industrial pH & Conductivity Equipment

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- ☑ Data Acquisition and Engineering Software
- ☑ Communications-Based Acquisition Systems
- ☑ Plug-in Cards for Apple, IBM & Compatibles
- ☑ Datalogging Systems
- ☑ Recorders, Printers & Plotters

HEATERS

- ☑ Heating Cable
- ☑ Cartridge & Strip Heaters
- ☑ Immersion & Band Heaters
- ☑ Flexible Heaters
- ☑ Laboratory Heaters

ENVIRONMENTAL MONITORING AND CONTROL

- ☑ Metering & Control Instrumentation
- ☑ Refractometers
- ☑ Pumps & Tubing
- ☑ Air, Soil & Water Monitors
- ☑ Industrial Water & Wastewater Treatment
- ☑ pH, Conductivity & Dissolved Oxygen Instruments

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