PHOENIX ELECTRODE COMPANY BROMIDE ION ELECTRODES INSTRUCTION MANUAL

GENERAL INSTRUCTIONS

Introduction

The pHoenix Bromide Ion Electrodes are used to quickly, simply, accurately, and economically measure free bromide ion in aqueous solutions.

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 pHoenix Bromide Ion Electrode, Cat. No. BR01501, (reference electrode necessary), the pHoenix Bromide Ion Combination Glass Electrode, Cat. No. BR01502, or the pHoenix Bromide Ion Combination Epoxy Electrode, Cat. No. BR01503.
- 5. The pHoenix Double Junction Reference Electrode, Cat. No. 5731429 (for use with the BR01501) with pHoenix Filling Solution, Cat. No. R001011, in the inner junction and with pHoenix Filling Solution, Cat. No. R001015, in the outer junction.
- 6. pHoenix Polishing Paper, Cat. No. 948201, to polish dirty or etched electrode membranes.

Required Solutions

- 1. Deionized or distilled water for solution and standard preparation.
- 2. pHoenix Ionic Strength Adjuster (ISA), 5M NaNO3, Cat. No. BR0IS01. To prepare this solution from your own laboratory stock, half-fill a 1000 ml volumetric flask with distilled water and add 425 grams of reagent-grade sodium nitrate. Swirl the flask gently to dissolve the solid. Fill to the mark with distilled water, cap, and upend the flask several times to mix the solution.

- 3. pHoenix Bromide Standard, 0.1M NaBr, Cat. No. BROASO1. To prepare this solution from your own laboratory 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 to the mark with distilled water, cap, and upend several times to mix the solution.
- 4. pHoenix Bromide Standard, 1000 ppm Br⁻¹, Cat. No. BROASO2. To prepare this solution from your own laboratory stock, half-fill a one 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 to the mark with distilled water, cap, and upend several times to mix the solution.

GENERAL PREPARATION

Electrode Preparation

Remove the rubber caps covering the electrode tips and the rubber insert covering the filling hole of the combination electrode or of the reference electrode. Fill the reference electrode or the combination electrode with the filling solution shipped with the electrode to a level just below the fill hole. No preparation is required with a sealed reference electrode.

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

- 1. 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 millivolt mode, lower the electrode tips into the solution.
- 2. Using a pipet, add 1 ml of either the 0.1M or the 1000 ppm standard to the beaker. When the reading is stable, record the mV reading.
- 3. Using a pipet, add 10 ml of the same standard used above to the beaker. When the reading has stabilized, record the mV reading.
- 4. Determine the difference between the two readings. The electrode is operating correctly if the mV potential has

changed by a difference of 57"3 mV, assuming the solution temperature is 25° C. See the **TROUBLESHOOTING** sections if the potential change is not within this range.

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

Electrode Slope Check (with an ion meter) (check electrodes each day)

- 1. Prepare standard bromide solutions whose concentrations vary by tenfold. Use either the 0.1M or the 1000 ppm bromide standard. Use the serial dilution method for this preparation.
- 2. 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 tips into the solution. Assure that the meter is in the concentration mode.
- 3. Adjust the meter to the concentration of the standard and fix the value in the memory according to the meter manufacturer's instructions.
- 4. Rinse the electrodes with distilled water and blot dry.
- 5. To another 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 tips into the solution.
- 6. Adjust the meter to the concentration of the standard and fix the value in the memory.
- 7. 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 sections if the slope is not within this range.

MEASUREMENT

Measuring Hints

All samples and standards should be at the same temperature for precise measurement. A difference of $1^{\circ}C$ in temperature will result in about a 2% measurement error.

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, between the stirrer and beaker.

Always rinse the electrodes with distilled water and blot 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 (>0.1M) before measurement.

Use fresh standards for calibration.

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

Always check to see that the membrane is free from air bubbles after immersion into the standard or sample.

Sample Requirements

All samples must be aqueous and not contain organics which can dissolve the epoxy electrode body and/or the cement bonding the sensing crystal to the electrode body. Infrequent measurements in solutions containing methanol, benzene, or acetonitrile are permitted. Highly polar solvents slowly attack the electrode. Please check with pHoenix Electrode Company 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 .

The pH range for the bromide ion electrode is 2-14. Neutralize samples outside this range with acid or base to bring them in range.

Interferences should be absent. If they are present, use the procedure found in the **Interference** and **Electrode Response** sections to remove them.

Units of Measurement

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

TABLE 1: Concentration Unit Conversion Factors

moles/liter	ppm Br ⁻¹
1.0X10 ⁻⁴	8.0
1.0X10 ⁻³	79.9
1.0X10 ⁻²	799.0
1.0X10 ⁻¹	7,990.0

MEASUREMENT PROCEDURE

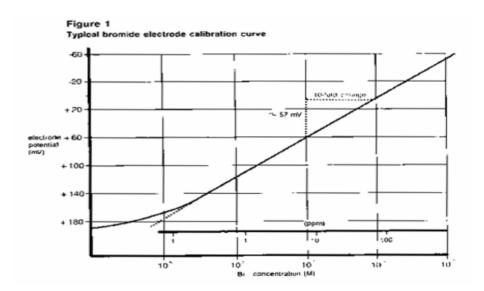
Direct Measurement

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 bromide solutions. The temperature of both sample solutions and of standard solutions should be the same.

Direct Measurement of Bromide (using a pH/mV meter)

- 1. By serial dilution prepare three standard solutions from the 0.1M or 1000 ppm standard. The resultant concentrations should be 10^{-2} , 10^{-3} , and 10^{-4} M or 1000, 100 and 10 ppm bromide standards. Add 2 ml of ISA to each 100 ml of 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 (1.0X10⁻⁴M or 10 ppm) in a 150 ml beaker 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 mid-range solution (1.0X10⁻³M or 100 ppm) in a 150 ml beaker on the magnetic stirrer and begin stirring at a constant rate. After rinsing the electrodes in distilled water, blot dry and immerse the electrode tips in the solution. When the reading has stabilized, record the mV reading.
- 4. Place the most concentrated solution (1.0X10⁻²M or 1000 ppm) in a 150 ml beaker on the magnetic stirrer and begin stirring at a constant rate. After rinsing the

- electrodes in distilled water, blot dry and immerse the electrode tips in the solution. When the reading has stabilized, record the mV reading.
- 5. Using semi-logarithmic graph paper, plot the mV reading (linear axis) against the concentration (log axis). Extrapolate the curve down to about $1.0 \times 10^{-5} \text{M}$ or 1 ppm. A typical calibration curve for bromide can be found in Figure 1.



A calibration curve is constructed on semi-logarithmic paper when using a pH/mV meter in the millivolt mode. 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 procedures.

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 at a constant rate. Rinse the electrode tips with distilled water, blot dry and lower into the solution. When the reading has stabilized, record the mV reading. Using the

- calibration curve, 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 tips in the mid-range standard. After the reading has stabilized, compare it to the original reading recorded in Step 3 above. A reading differing by more than 0.5 mV or a change in the ambient temperature will necessitate the repetition of Steps 2-5 above. A new calibration curve should be prepared daily.

Direct Measurement of Bromide (using an ion meter)

- 1. By serial dilution of the 0.1M or 1000 ppm bromide standard, 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 TSA to each.
- 2. Place the more dilute solution on the magnetic stirrer and begin stirring at a constant rate. Assure that the meter is in the concentration mode. Lower the electrode tips into the solution.
- 3. 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.
- 4. Rinse the electrodes with distilled water and blot dry.
- 5. Place the more concentrated solution on the magnetic stirrer and begin stirring at a constant rate. Lower the electrode tips into the solution.
- 6. 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.
- 7. For low level bromide measurements, place the rinsed, dried electrodes 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.
- 8. 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.

- 9. Immerse the electrode tips in the solution and wait for the reading to stabilize. Read the concentration directly from the meter display.
- 10. The calibration should be checked every two hours. Assuming no change in ambient temperature, place the electrode tips in the first bromide standard. After the reading has stabilized, compare it to the original reading in Step 3 above. A reading differing by more than 0.5 mV or a change in the ambient temperature will necessitate the repetition of steps 2-6 above. The meter should be re-calibrated daily.

Low Level Bromide Determinations (using a pH/mV meter)

This procedure is recommended for solutions containing less than $2.0 \times 10^{-6} \text{M}$ (0.2ppm). Use the following low level bromide measurement procedure in the non-linear portion of the calibration curve. (See Figure 1.)

- 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 $1.0 \times 10^{-2} M$.
- 2. Dilute 1 ml of 0.1M standard to 1000 ml to prepare a $1.0 \times 10^{-4} \rm M$ standard solution for measurements in moles per liter. Dilute 10 ml of 1000 ppm standard to 1000 ml 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 tips in the solution and assure that the meter is in the mV mode.
- 5. Add increments of the $1.0 \times 10^{-4} \mathrm{M}$ or 10 ppm standard as given in Table 2 below.
- 6. After the reading has stabilized, record the mV reading after each addition. Electrode response time is much longer at these levels. Allow adequate time for the electrodes to stabilize.

TABLE 2: Step-wise Calibration for Low Level Bromide Measurements

		Added	Concer	ntration
Step	Pipette	Volume	(ml) M	ppm
1	A	0.1	1.0X10 ⁻⁷	0.01
2	A	0.1	2.0×10^{-7}	0.02
3	A	0.2	4.0×10^{-7}	0.04
4	A	0.2	6.0×10^{-7}	0.06
5	A	0.4	9.9X10 ⁻⁷	0.99
6	В	2.0	2.9×10^{-6}	2.90
7	В	2.0	4.8X10 ⁻⁶	4.80

Pipet A = 1 ml graduated pipet

Pipet B = 2 ml pipet

Solutions: additions of 1.0X10⁻⁴M or 10 ppm standard to 100 ml of solution as prepared in Step 3 above.

- 7. On semi-logarithmic graph paper, plot the mV reading (linear axis) against the concentration (log axis) as in Figure 1.
- 8. Rinse the electrodes in distilled water and blot dry.
- 9. Measure out 100 ml of sample into 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. Lower the electrode tips 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 1-2 hours by repeating Steps 2-7 above.

ELECTRODE CHARACTERISTICS

Reproducibility

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.

Interferences

A layer of silver may form on the electrode surface in the presence of strong reducing agents. 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. Proper performance can be restored by polishing. See the section **Electrode Response** for proper polishing procedure.

Solutions containing oxidizing agents such as MnO_4^{-1} , Fe^{+3} , and Cu^{+2} , may be measured without problem. All samples must be free of mercury.

The maximum allowable ratio of interfering ion to bromide ion is given in Table 3. This ratio is expressed as the ratio of the interfering ion molarity to the bromide 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 3: Maximum Allowable Ratio of Interfering Ion to Bromide Ion

Interference	Maximum Ratio for Bromide
OH ⁻¹ (2)	30000
Cl^{-1} (1)	400
$S_2O_3^{-2}$ (3)	20
NH ₃ (3)	2
I ⁻¹ (1)	2.0×10^{-4}
CN^{-1} (4)	8.0×10^{-5}
S ⁻² (4)	1.0X10 ⁻⁶

- (1) Gran's plot titration can be used to measure mixed halides in solution.
- (2) Acidify with $1M \ HNO_3$ 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 3's use when using the bromide ion electrode:

What is the maximum level of iodide tolerable in a sample whose bromide concentration is $10^{-2}\mathrm{M}$?

Using Table 3, the maximum ratio is:

$$= 2X10^{-4}$$
[Br⁻¹]

or
$$[I^{-1}] = 2X10^{-4} [Br^{-1}]$$

 $[I^{-1}] = 2X10^{-4} (1X10^{-2})$

 $[I^{-1}] = 2X10^{-6}M$ maximum iodide concentration for no interference

Complexation

Total concentration (Ct) consists of free ions (Cf) 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 ions complex with some metal ions. Table 4 lists the levels of complexing metals causing a 20% error at $1.0 \times 10^{-4} M$ bromide.

Ion	Concentration	
Bi ⁺³	4.0X10 ⁻⁴ M (80 ppm)	
Cđ ⁺²	$2.0 \times 10^{-3} M (200 ppm)$	
Pb ⁺²	$8.0 \times 10^{-3} M (1600 ppm)$	
Sn ⁺²	$2.0 \text{X} 10^{-2} \text{M} \text{ (2400 ppm)}$	
Tl ⁺³	$2.0 \times 10^{-5} M (4 ppm)$	

Temperature Influences

Samples and standards should be at the same temperature since electrode potentials are influenced by changes in temperature. A 1°C difference in temperature results in a 2% error at the $1.0\text{X}10^{-3}\text{M}$ 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 5 gives values of the "S" factor in the Nernst equation for the bromide ion.

TABLE 5: Temperature vs. Values for the Electrode Slope

Temp (°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

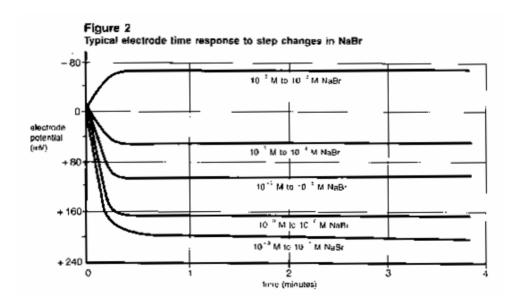
If changes in temperature occur, the electrodes should be recalibrated.

The temperature range of the pHoenix Bromide Ion Electrodes is $0^{\circ}-80^{\circ}\text{C}$, provided that temperature equilibrium has occurred. If the temperature varies substantially from room temperature, equilibrium times up to one hour are recommended.

Electrode Response

Plotting the electrode mV potential against the bromide concentration on semi-logarithmic paper results in a straight line with a slope of about 57 mV per decade. (Refer to Figure 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 2.)



A drifting potential reading or a decrease in electrode slope may mean that the electrode membrane needs polishing.

To polish the membrane:

- 1. If using polishing paper, cut off a 1-2" piece and place it face up on the lab bench.
- 2. Put a few drops of distilled or deionized water in the center of the paper.
- 3. Holding the paper (cotton) steady with one hand, bring the membrane of the electrode down perpendicular to the paper and, with a slight swirling motion, gently polish the tip of the electrode against the surface of the polishing paper (cotton) for a few seconds.
- 4. Rinse the electrode surface with distilled or deionized water and soak the electrode tip in standard solution for about five minutes before use.
- 5. If using jeweller's rouge, place a cotton ball on the table top and flatten it using the bottom of a beaker.
- 6. Put 1-2 drops of distilled or deionized water in the center of the cotton pad.
- 7. Add a small amount of jeweller's rouge to the damp cotton.
- 8. Continue with Steps 3 and 4 above.

Limits of Detection

The upper limit of detection in pure sodium bromide solutions is 1M. In the presence of other ions, the upper limit of detection is above $1.0 \times 10^{-1} \text{M}$ bromide, but the possibility of a liquid junction potential developing at the reference electrode and the "salt extraction effect" are two limiting factors. Some salts may infuse into the electrode membrane at high salt concentrations, causing deviation from the theoretical response. Either dilute samples between 1M and $1.0 \times 10^{-1} \text{M}$ or calibrate the electrode at 4 or 5 intermediate points.

The lower limit of detection is influenced by the slight water solubility of the electrode pellet. Refer to Figure 1 for a comparison of the theoretical response to the actual response at low levels of bromide. Bromide measurements below $10^{-4} \rm M~Br^{-1}$ should employ low level procedures.

pH Effects

Hydroxide ion interferes with measurements of low levels of bromide although the electrode can be used over a reasonable pH range. Table 3 should be used to determine the minimum pH at which low level bromide measurements can be made without more than a 10% error due to hydroxide ion interference.

Electrode Life

The bromide electrode will last six months in normal laboratory use. On-line measurements might shorten operational lifetime to several months. In time, the response time will increase and the calibration slope will decrease to the point calibration is difficult and electrode replacement is required.

Electrode Storage

The pHoenix Bromide Electrodes may be stored for short periods of time in $1.0 \times 10^{-2} \mathrm{M}$ bromide solution. For longer storage (longer than two weeks), rinse and dry the sensing pellet and cover the membrane tip with any protective cap shipped with the electrode. The reference portion of the combination electrode (or the outer chamber of the reference electrode) should be drained of filling solution, if refillable, and the rubber insert placed over the filling hole.

ELECTRODE THEORY

Electrode Operation

The pHoenix Bromide Ion Electrode consists of a sensing membrane bonded into a glass or an epoxy body. The membrane is composed of silver bromide/silver sulfide. When an electrode potential develops across the membrane, the electrode is in contact with solution containing bromide ions and is capable of measuring free bromide 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 bromide 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 (-57mV/decade)

X = level of bromide 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, a, by:

X = aCf

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

 $I = 2 OC_x Z_x^2$

where:

 C_x = concentration of ion x Z_x = charge of ion x

O = 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, a, 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 adjuster is added to samples and standards. The recommended ISA for bromide ions is NaNO3. 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 the bromide 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 another, liquid junction potentials arise. potentials occur from the inter-diffusion of ions in the two Electrode charge will be carried unequally across the solutions. 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 solution diffuse into the sample 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.

TROUBLESHOOTING GUIDE

The goal of troubleshooting is the isolation of a problem through checking each of the system components in turn: the meter, the glass-ware, the electrodes, the standards and reagents, the sample, and the technique.

Meter

The meter may be checked by following the check-out procedure in the instrument instruction manual.

Glass-ware

Clean glass-ware is essential for good measurement. Be sure to wash the glass-ware well with a mild detergent and rinse very well with distilled or deionized water. Clean glass-ware will drain without leaving water droplets behind.

Electrodes

The electrodes may be checked by using the procedure found in the sections entitled **Electrode Slope Check**.

- 1. Be sure to use distilled or deionized water when following the procedures given in **Electrode Slope Check**.
- 2. If the electrode fails to respond as expected, see the sections **Measuring Hints** and **Electrode Response**. Repeat the slope check.
- 3. If the electrodes still fail to respond as expected, substitute another bromide ion electrode that is known to be in good working order for the questionable electrode. If the problem persists and you are using an electrode pair, try the same routine with a working reference electrode.
- 4. If the problem persists, the standards and/or reagents may be of poor quality, interferences in the sample may be present or the technique may be faulty. (See Standards & Reagents, Sample, and Technique sections below.)
- 5. If another electrode is not available for test purposes, or if the electrode in use is suspect, review the instruction manual and be sure to:
 - Clean and rinse the electrodes thoroughly.
 - Prepare the electrodes properly.

- Use the proper filling solution.
- Adjust the pH and the ionic strength of the solution by the use of the proper ISA.
- Measure correctly and accurately.
- Review TROUBLESHOOTING HINTS.

Standards & Reagents

Whenever problems arise with the measuring procedure that has been used successfully in the past, be sure to check the standard and reagent solutions. If in doubt about the credibility of any of the solutions, prepare them again. Errors may result from contamination of the ISA, incorrect dilution of standards, poor quality distilled/deionized water, or a simple mathematical miscalculation.

Sample

Look for possible interferences, complexing agents, or substances which could affect the response or physically damage the sensing electrode (or the reference electrode) if the electrodes work perfectly in the standard, but not in the sample.

Try to determine the composition of the samples prior to testing to eliminate a problem before it starts. (See Measuring Hints, Sample Requirements, and Interferences.)

Technique

Be sure that the electrode's limit of detection has not been exceeded. Be sure that the analysis method is clearly understood and is compatible with the sample.

Refer to the instruction manual again. Reread ${\tt GENERAL}$ ${\tt PREPARATION}$ and ${\tt ELECTRODE}$ ${\tt CHARACTERISTICS}$.

If trouble still persists, call pHoenix Electrode Company at 1-800-522-7920 and ask for the Technical Services Department.

TROUBLESHOOTING HINTS

Symptom	Possible Causes	Next Step
Out of Range Reading	defective meter	check meter with shorting strap (see meter instruction manual)
	defective electrode	check electrode operation
	electrodes not plugged in properly	unplug electrodes and reseat
	reference electrode not filled	be sure reference electrode is filled
	air bubble on membrane	remove bubble by redipping electrode
	electrodes not in solution	put electrodes in solution
Noisy or Unstable Readings (readings continuously or rapidly changing)	defective meter	check meter with shorting strap
	air bubble on membrane	remove bubble by re-dipping electrode
	electrode exposed to interferences	soak electrode in bromide standard
	defective electrode	replace electrode
	ISA not used	use recommended ISA
	meter or stirrer not grounded	ground meter or stirrer
Drift (reading slowly changing in one direction)	samples and standards at different temperatures	allow solutions to come to room temper- ature before measurement
	electrode exposed to complexing agents	check section entitled Complexation
	incorrect reference	use recommended

	filling solution	filling solution
Low Slope or No Slope	standards contamin- ated or incorrectly made	prepare fresh standards
	ISA not used	use recommended ISA
	standard used as ISA	use ISA
	electrode exposed to complexing agents	check section entitled Complexation
	air bubble on membrane	remove bubble by re-dipping probe
"Incorrect Answer" (but calibration curve is good)	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 of millivolt number correctly
	incorrect standards	prepare fresh standards
	wrong units used	apply correct conversion factor: $10^{-3}M = 79.9 \text{ ppm as}$ Br ⁻¹
	complexing agents in sample	check section entitled Complexation

SPECIFICATIONS

Concentration Range: $1M \text{ to } 5 \text{ x } 10^{-6}M$

79,900 ppm to 0.4 ppm

pH Range: 2-14

Temperature Range: 0E to 80EC

Resistance: <1 Mohm

Reproducibility: "2%

Samples: Aqueous sloutions only;

avoid organic solvents

Size: 110 mm in length

12 mm in diameter 1 m cable length

Storage: Store in bromide solution

ORDERING INFORMATION

P/N	DESCRIPTION
BR01501	Bromide Ion Electrode, mono (reference electrode necessary), epoxy body
BR01502	Bromide Ion Electrode, combination, glass body
BR01503	Bromide Ion Electrode, combination, epoxy body
5731429	Reference Electrode, double junction, sleeve, epoxy body, for use with the BR01501
BR0AS01	Bromide Standard, 0.1M NaBr
BR0AS02	Bromide Standard, 1000 ppm NaBr
BR0IS01	Ionic Strength Adjuster (ISA), $5M$ NaNO $_3$
R001011	Electrode Filling Solution, 4M KCl/AgCl, for the 5731429 inner chamber
R001015	Electrode Filling Solution, $1M\ KNO_3$, for the 5731429 outer chamber and the BR01502 electrode
R001046	Electrode Filling Solution, 1M $\ensuremath{\text{KNO}_3/0.1M}$ KCl, for the BR01503 electrode

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