pHoenix Electrode Company
FLUOROBORATE ION ELECTRODES
INSTRUCTION MANUAL

GENERAL INSTRUCTIONS

Introduction

The pHoenix Electrode Company Fluoroborate Ion Electrodes are used to quickly, simply, accurately, and economically measure fluoroborate 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. Plastic beakers and storage bottles for all fluoroborate standards and samples. The hydrofluoric acid present in fluoroborate solutions will etch glass.

5. The pHoenix Electrode Company Fluoroborate Ion Electrode, Cat. No. BF41502, or BF41508.

Required Solutions

1. Deionized or distilled water for solution and standard preparation.

2. pHoenix Ionic Strength Adjuster (ISA), 2M $(NH_4)_2SO_4$, Cat. No. BF4IS01. To prepare this solution from your own laboratory stock, half fill a 1000 ml volumetric flask with distilled water and add 264 grams of reagent-grade ammonium sulfate, $((NH_4)_2SO_4$. Swirl the flask to dissolve the solid. Fill the flask to the mark with distilled water, cap, and invert the flask several times to mix the contents. ISA is added at the rate of 2 ml of ISA to each 100 ml of standard or sample to adjust the ionic strength to about 0.12M.

3. pHoenix Electrode Filling Solution, 0.1M $(NH_4)_2SO_4$, Cat. No. R001044.

4. pHoenix Fluoroborate Standard, 0.1M $NaBF_4$, Cat. No. BF4AS01. To prepare this solution from your own laboratory stock, add 10.98 grams of reagent-grade $NaBF_4$ to a one liter volumetric flask about half full with distilled water. Swirl the flask gently to dissolve the solid. Fill to the mark with distilled water, cap and upend several times to mix the solution. Store the solution in polyethylene bottles and discard after one week to avoid errors introduced by hydrolysis of fluoroborate.

5. pHoenix Fluoroborate Standard, 1000 ppm BF_4^{-1}, Cat. No. BF4AS02. To prepare this solution from your own laboratory stock, add 1.27 grams of reagent-grade NaBF4 to a one liter volumetric flask about half full with distilled water. Swirl the flask gently to dissolve the solid. Fill to the mark with distilled water, cap and upend several times to mix the solution. Store the solution in polyethylene bottles and discard after one week to avoid errors introduced by hydrolysis of fluoroborate.

GENERAL PREPARATION

Electrode Preparation

Remove the rubber cap covering the electrode tip and any rubber insert covering the filling hole of the reference electrode. Fill the combination electrode with the filling solution shipped with the electrode to a level just below the fill hole. Gently shake the electrode downward in the same manner as a clinical thermometer to remove any air bubbles which may be trapped behind the fluoroborate membrane. Prior to first usage, or after long-term storage, immerse the fluoroborate membrane in fluoroborate standard for thirty minutes. The electrode is now ready for use.

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

Electrode Slope Check (with pH/mV meter)
(check electrode each day)

1. To a 150 ml beaker, add 100 ml of distilled water and two 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 tip into the solution.

2. Using a pipet, add 1 ml of 0.1M or 1000 ppm fluoroborate standard to the beaker. When the reading has stabilized, record the mV reading.

3. Using a pipet, add 10 ml of the same fluoroborate 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 operation correctly if the millivolt potential has changed by 56 ± 2 mV, assuming the solution temperature is between 20° and 25°C. See the **TROUBLESHOOTING** sections 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.

Electrode Slope Check (with ion meter)
(check electrode each day)

1. Prepare standard fluoroborate solutions whose concentrations vary by a factor of ten. Use either the 0.1M or 1000 ppm fluoroborate 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 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.

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 electrode 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 the magnetic stirrer and begin stirring at a constant rate. Lower the electrode tip 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** section if the slope is not within this range.

MEASUREMENT

Measuring Hints

The sensing membrane is normally subject to water uptake and might appear milky. This has no effect on performance.

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

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 electrode 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.

A slow responding electrode may be caused by interferences to the electrode. To restore proper performance, soak the electrode in distilled water for about 5 minutes to clean the membrane, rinse, and soak in standard solution for about 5 minutes.

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

Sample Requirements

All samples must be aqueous and not contain organics which can dissolve in the membrane or extract out the liquid ion exchanger.

The temperature of the standard solutions and of the sample solutions should be the same and below 40°C. About a 2% error will be introduced for a 1°C difference in temperature.

Interferences should be absent. If they are present, use the procedures found in the **Interferences** section to remove them.

The pH range for the fluoroborate ion electrode is 2.5-11. Neutralize samples outside this range with acid or base to bring them in range. Fluoroborate sample with either high or low pH must be analyzed immediately after preparation or hydrolysis of fluoroborate to BF_3OH^{-1}, $BF_2(OH)_2^{-1}$, and $BF(OH)_3^{-1}$ will occur. Convert boric acid or borate ion to fluoroborate by addition of HF.

Units of Measurement

Fluoroborate concentrations are measured in units of ppm as boron, ppm as fluoroborate, moles per liter, or any other convenient concentration unit. Table 1 indicates some of the concentration units.

TABLE 1: Concentration Unit Conversion Factors

ppm B	ppm BF_4^{-1}	moles/liter
108.9	868.0	1.0×10^{-2}
10.8	86.8	1.0×10^{-3}
1.1	8.7	1.0×10^{-4}

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

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

1. By serial dilution, prepare three standard solutions from the 0.1M or 1000 ppm stock standard. The resultant concentrations should be 10^{-2}, 10^{-3}, and 10^{-4}M or 100 and 10 ppm fluoroborate 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. Use plastic lab ware.

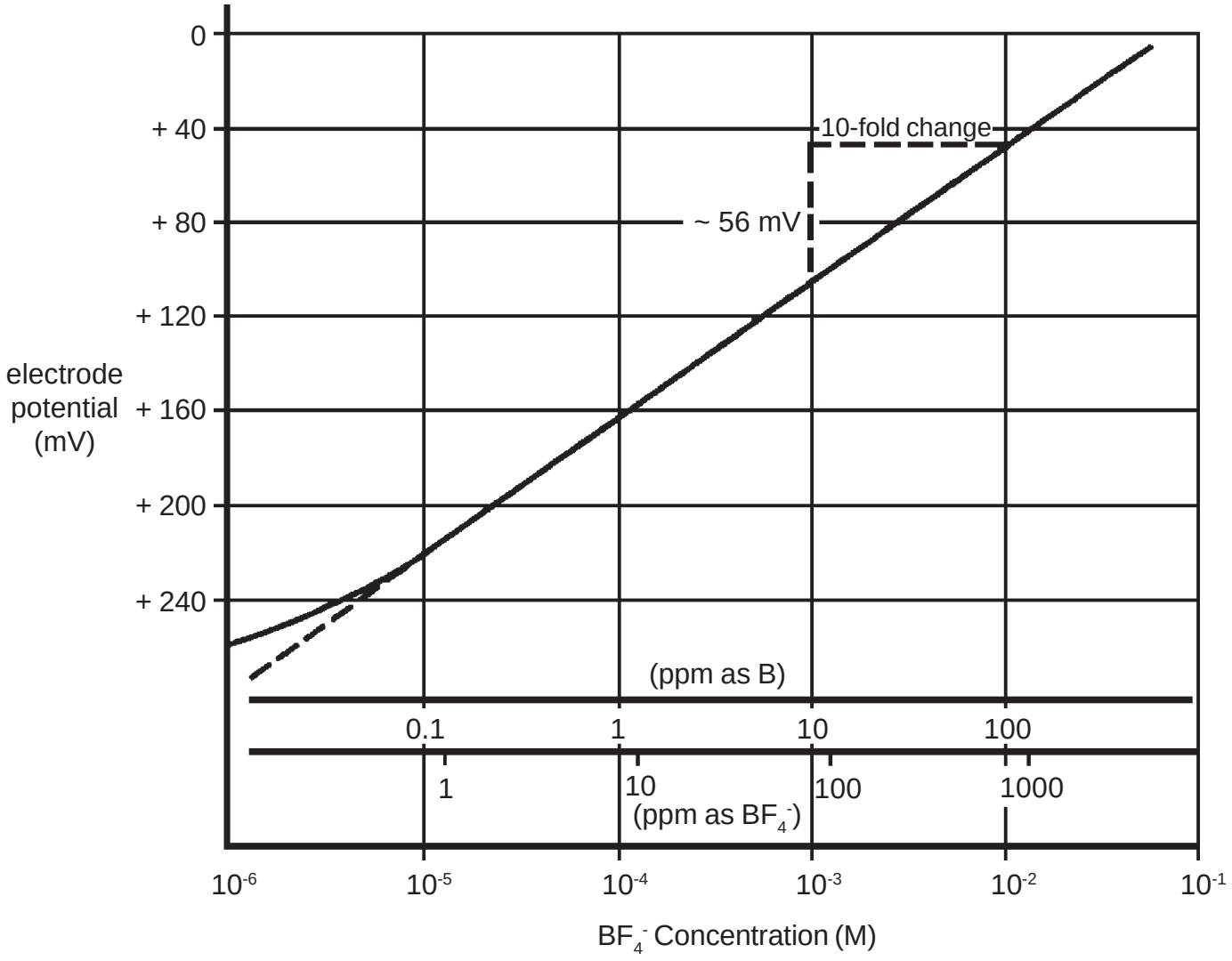
2. Place the most dilute solution (10^{-4}M or 10 ppm) in a 150 ml beaker on the magnetic stirrer and begin stirring at a constant rate. After assuring that the meter is in the mV mode, lower the electrode tip into the solution. After the reading has stabilized, record the mV reading.

3. Place the mid-range solution (10^{-3}M or 100 ppm) in a 150 ml beaker on the magnetic stirrer and begin stirring. After rinsing the electrode with distilled water, blot dry and immerse the electrode tip in the solution. When the reading has stabilized, record the mV value.

4. Place the most concentrated solution (10^{-2}M or 1000 ppm) in a 150 ml beaker on the magnetic stirrer and begin stirring. After rinsing the electrode in distilled water, 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). Extrapolate the calibration curve down to about $1.0X10^{-5}$M (1.0 ppm NO_3^{-1}). A typical calibration curve can be found in Figure 1.

Figure 1
Typical Fluoroborate Electrode Calibration Curve



A calibration curve is constructed on semi-logarithmic paper when using the 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 tip with distilled water, blot dry, and lower the electrode tip into the solution. When the reading has stabilized, record the mV reading. Using the calibration curve, determine the sample concentration.

7. The calibration should be checked every two hours. Assuming no change in ambient temperature, place the electrode tip 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 Fluoroborate (using an ion meter)

1. By serial dilution of the 0.1M or 1000 ppm fluoroborate standard, prepare two fluoroborate 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 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 tip into the solution.

3. Adjust the meter to the concentration of the fluoroborate standard and fix the value in the memory according to the meter manufacturer's instructions after stabilization of the reading.

4. Rinse the electrode 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 tip into the solution.

6. Adjust the meter to the concentration of the nitrate standard and fix the value in the memory according to the meter manufacturer's instructions after stabilization of the reading.

7. For low level 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 in 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 the beaker on the magnetic stirrer and begin stirring.

9. Immerse the electrode tip 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 tip in the first fluoroborate 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 ambient temperature will necessitate the repetition of Steps 2-6 above. The meter should be re-calibrated daily.

Low Level Fluoroborate Measurements (using a pH/mV meter)

This procedure is recommended for solutions with ionic strengths less than $1.0X10^{-2}M$. If the solution is high in ionic strength, but low in fluoroborate, 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 [$0.4M\ (NH_4)_2SO_4$] is added at the rate of 1 ml low level ISA to each 100 ml of sample. The background ionic strength will be $4.0X\ 10^{-3}M$.

2. Dilute 1 ml of 0.1M standard to 100 ml to prepare a $1.0X10^{-3}M\ BF_4^{-1}$ solution for measurements in moles per liter. Use the 1000 ppm standard for preparing a 100 ppm NO_3^{-1} standard by diluting 10 ml of the 1000 ppm standard to 100 ml. 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 $1.0X10^{-3}M$ or 100 ppm standards as given in Table 2 below.

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

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

Step	Pipet	Added Volume (ml)	Concentration $M\ NO_3^{-1}$	$ppm\ NO_3^{-1}$
1	A	0.1	1.0×10^{-6}	0.1
2	A	0.1	2.0×10^{-6}	0.2
3	A	0.2	4.0×10^{-6}	0.4
4	A	0.2	6.0×10^{-6}	0.6
5	A	0.4	9.9×10^{-6}	1.0
6	B	2.0	2.9×10^{-5}	2.9
7	B	2.0	4.8×10^{-5}	4.8

Pipet A = 1 ml graduated pipet
Pipet B = 2 ml pipet
Solutions: additions of $1.0X10^{-3}M$ or 100 ppm standard to 100 ml of ISA as prepared in Step 3 above.

7. On semi-logarithmic graph paper, plot the millivolt reading (linear axis) against the concentration (log axis) as in Figure 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. Place the beaker on the magnetic stirrer and 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.

Low Level Fluoroborate Determination (using an ion meter)

Follow the procedure given for normal fluoroborate determinations using an ion meter and the blank correction procedure.

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

Certain anions are electrode interferences and will cause electrode malfunction, drift or measurement errors if present in high enough levels. The level of interfering common anions that will cause a 10% error at three levels of nitrate is given in Table 3.

TABLE 3: Concentration of Possible Interferences Causing a 10% Error at Various Levels of NFluoroborate; Background Ionic Strength of 0.12M $(NH_4)_2SO_4$.

Interferences (moles/liter)	10^{-2} M	10^{-3} M	10^{-4} M BF_4^{-1}
PO_4^{-3}	8.0×10^{-1}	8.0×10^{-2}	8.0×10^{-3}
HPO_4^{-2}	8.0×10^{-1}	8.0×10^{-2}	8.0×10^{-3}
$H_2PO_4^{-1}$	8.0×10^{-1}	8.0×10^{-2}	8.0×10^{-3}
Cl^{-1}	5.0×10^{-1}	5.0×10^{-2}	5.0×10^{-3}
CO_3^{-2}	3.0×10^{-1}	3.0×10^{-2}	3.0×10^{-3}
HCO_3^{-1}	3.0×10^{-1}	3.0×10^{-2}	3.0×10^{-3}
NO_3^{-1}	5.0×10^{-2}	5.0×10^{-3}	5.0×10^{-4}
NO_2^{-1}	1.0×10^{-2}	1.0×10^{-3}	1.0×10^{-4}
Br^{-1}	1.0×10^{-2}	1.0×10^{-3}	1.0×10^{-4}
CN^{-1}	5.0×10^{-3}	5.0×10^{-4}	5.0×10^{-5}
ClO_3^{-1}	5.0×10^{-4}	5.0×10^{-5}	5.0×10^{-6}
I^{-1}	5.0×10^{-5}	5.0×10^{-6}	5.0×10^{-7}
ClO_4^{-1}	5.0×10^{-6}	5.0×10^{-7}	5.0×10^{-8}

Interferences (ppm)	100 ppm N	10 ppm N	1 ppm N
PO_4^{-3}	$7.0x10^4$	$7.0x10^3$	$7.0x10^2$
HPO_4^{-2}	$7.1x10^4$	$7.1x10^3$	$7.1x10^2$
$H_2PO_4^{-1}$	$7.2x10^4$	$7.2x10^3$	$7.2x10^2$
Cl^{-1}	$1.6x10^4$	$1.6x10^3$	$1.6x10^2$
CO_3^{-2}	$1.7x10^4$	$1.7x10^3$	$1.7x10^2$
HCO_3^{-1}	$1.7x10^4$	$1.7x10^3$	$1.7x10^2$
NO_3^{-1}	$2.9x10^3$	$2.9x10^2$	$2.9x10^1$
NO_2^{-1}	$4.2x10^2$	$4.2x10^1$	4.2
Br^{-1}	$7.4x10^2$	$7.4x10^1$	7.4
CN^{-1}	$1.2x10^2$	$1.2x10^1$	1.2
ClO_3^{-1}	$4.0x10^1$	4.0	$4.0x10^{-1}$
I^{-1}	6.0	$6.0x10^{-1}$	$6.0x10^{-2}$
ClO_4^{-1}	$5.0x10^{-1}$	$5.0x10^{-2}$	$5.0x10^{-3}$

Interferences such as phosphate, bromide, iodide and cyanide can be removed by precipitation with 0.5 grams of silver sulfate added to 100 ml of sample. Carbonate and bicarbonate, which are weak interferences, can be removed by acidifying the sample to pH 4.5 with sulfuric acid. Nitrite interference can be removed by adding 0.3 grams of sulfamic acid to 100 ml of sample.

The above interference removal procedures require similar treatment of standards as well as samples.

If the electrode is exposed to high levels of interfering ions which cannot be removed, the electrode reading may drift and the response may become sluggish. Restore performance by soaking in distilled water for 30 minutes followed by soaking in fluoroborate standard for 30 minutes.

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 10^{-3}M level. 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 fluoroborate electrode, as indicated by the factor "S" in the Nernst equation, also varies with temperature. Table 4 gives values for the "S" factor in the Nernst equation for the fluoroborate ion.

The operating range of the fluoroborate ion electrode is 0°-40°C, provided that temperature equilibrium has occurred.

If the temperature varies substantially from room temperature, equilibrium times up to one hour are recommended.

TABLE 4: Temperature vs. Values for the Electrode Slope

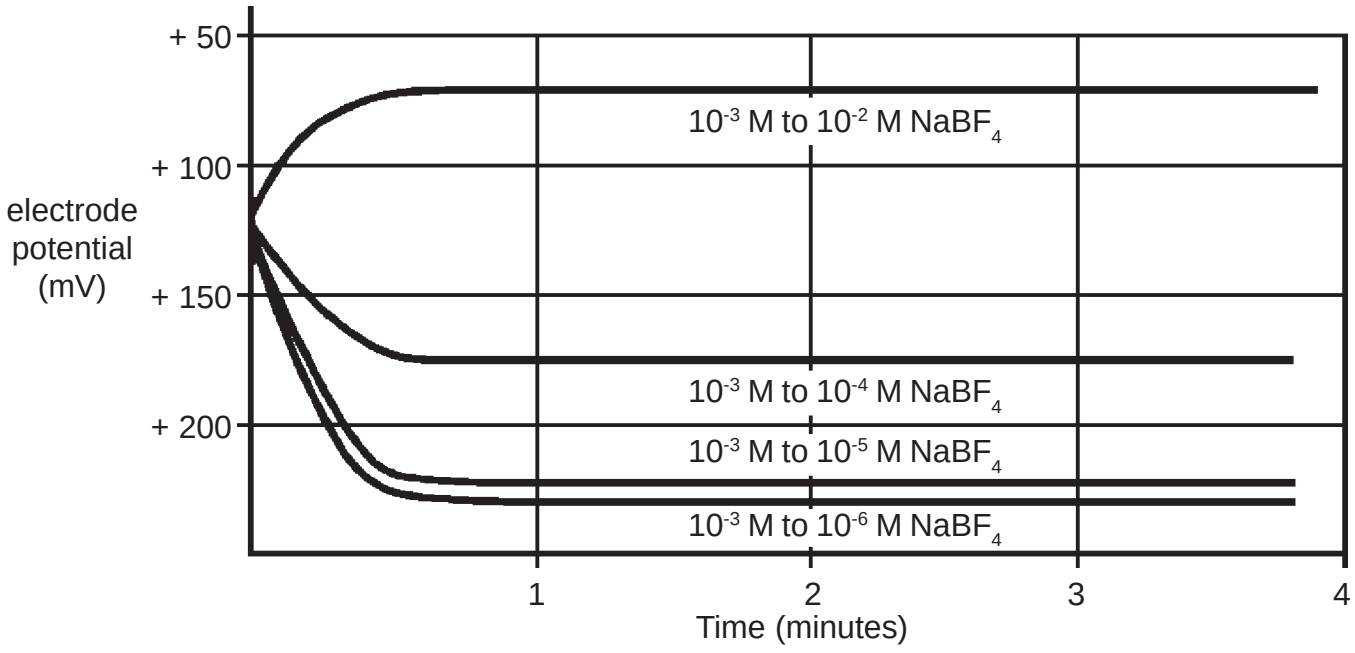
Temp (°C)	"S"
0	54.20
10	56.18
20	58.18
25	59.16
30	60.15
40	62.13
50	64.11

Electrode Response

Plotting the mV potential against the fluoroborate concentration on semi-logarithmic paper results in a straight line with a slope of about 56 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 one minute or less in highly concentrated solutions to several minutes near the detection limit. (Refer to Figure 2.)

Figure 2
Typical Electrode Time Response to Step Changes in $NaBF_4$



Limits of Detection

The upper limit of detection in pure sodium fluoroborate solutions is 1M. In the presence of other ions, the upper limit of detection is above 10^{-1}M fluoroborate, but two factors influence this upper limit. Both the possibility of a liquid junction potential developing at the reference electrode and the salt extraction effect influence this upper limit. Some salts may infuse into the electrode membrane at high salt concentrations, causing deviation from the theoretical response. Either dilute samples between 1M and 10^{-1}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 ion exchanger used in the sensing portion of the electrode. Refer to Figure 1 for a comparison of the theoretical response to the actual response at low levels of fluoroborate. Fluoroborate measurements below 10^{-5}M BF_4^{-1} (0.87 ppm as BF_4^{-1} or 0.11 ppm as B)) should employ low level procedures.

pH Effects

The operating range of the fluoroborate electrode is from pH 2.5 to pH 11.

Electrode Life

The fluoroborate electrode will last six months in normal laboratory use. On-line measurement 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 fluoroborate electrodes may be stored for short periods of time in 10^{-2}M fluoroborate solution. For longer storage (longer than two weeks), rinse and dry the fluoroborate membrane and cover the tip with any protective cap shipped with the electrodes. The reference portion of the combination electrode should be drained of filling solution, and the rubber insert placed over the filling hole.

ELECTRODE THEORY

Electrode Operation

The fluoroborate electrode consists of an electrode body

containing a liquid internal filling solution in contact with a gelled organophilic membrane containing a fluoroborate ion exchanger. When the membrane is in contact with a solution containing free fluoroborate ions, an electrode potential develops across the membrane. This electrode potential is measured against a constant reference potential, using a standard pH/mV meter or an ion meter. The level of fluoroborate ions, corresponding to the measured potential, is described by the Nernst equation:

$$E = E' - S \log X$$

where:

E = measured electrode potential
E'= reference potential (a constant)
S = electrode slope (~56 mV/decade)
X = level of nitrate ions in solution

The activity, X, represents the effective concentration of the ions in solution. The total fluoroborate ion concentration, C_t, is the sum of free fluoroborate ion, C_f, and complexed or bound fluoroborate ion, C_b. The electrode is able to respond to only the free ions, whose concentration is :

$$C_f = C_t - C_b$$

Since fluoroborate ions form very few stable complexes, the free ion concentration may be equated to the total ion concentration.

The activity is related to the free ion concentration, C_f, by the activity coefficient, ã, by:

$$X = \tilde{a}\ C_f$$

Activity coefficients vary, depending on total ions strength, I, defined 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 ion concentration, the activity coefficient, ã, 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 (ISA) is added to samples and

standards. The recommended ISA for fluoroborate is $(NH_4)_2SO_4$. 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 nitrate ions.

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 inter-diffusion of ions in 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 samples 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 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 equitransferent salt. One must either calibrate the electrodes in the same pH range as the sample or use a known incremental 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 plastic-ware, the electrode, the standards & reagents, the sample, and the technique.

Meter

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

Plastic-ware

Clean plastic-ware is essential for good measurement. Be sure to wash the plastic-ware well with a mild detergent and rinse very well with distilled or deionized water.

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 section **Measuring Hints**. Repeat the slope check.

3. If the electrode still fails to respond as expected, substitute another fluoroborate 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 or reagent 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 electrode thoroughly.
   - Prepare the electrode 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 **GENERAL PREPARATION** and **ELECTRODE 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
	electrode not plugged in properly	unplug electrode and reseat
	reference electrode not filled	be sure reference electrode is filled
	air bubble on membrane	remove air bubble by re-dipping electrode
	electrode not in solution	put electrode in solution
Noisy or Unstable Readings (readings continuously or rapidly changing)	defective meter	check meter with shorting strap
	air bubble on membrane	remove air bubble by re-dipping electrode
	ISA not used	use recommended ISA
	meter or stirrer not grounded	ground meter or stirrer
	defective electrode	replace electrode
	electrode exposed to interferences	soak electrode in fluoroborate standard
	outer filling solution level too low	fill electrode to level just below the fill hole
Drift (reading slowly changing in one direction)	samples and standards at different temperatures	allow solutions to come to room temperature before measurement
	electrode exposed to interferences	soak electrode in fluoroborate standard
	incorrect reference filling solution	use recommended filling solution

Symptom	Possible Causes	Next Step
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
	electrode exposed to interferences	soak electrode in fluoroborate standard
	air bubble on membrane	remove bubble by re-dipping electrode
	defectice electrode	check electrode operation
"Incorrect Answer" (but calibration 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: 1.0×10^{-3}M = 86.8 ppm as BF_4^{-1} = 10.8 ppm as Boron
	complexing agents in sample	use decomplexing procedure
	sample carryover	rinse electrodes thoroughly between samples

SPECIFICATIONS

Concentration Range:	1M to 7 x 10^{-6}M (1.1 x 10^{4} to 9.0 x 10^{-2} ppm as B)
pH Range:	2.5 to 11
Temperature Range:	0° to 40°C
Resistance:	100 megohms
Reproducibility:	±2%
Samples:	aqueous solutions only no organic solvents
Size:	110 mm in length 12 mm in diameter 1 m cable length
Storage:	Store in dilute fluoroborate solution

ORDERING INFORMATION

P/N	DESCRIPTION
BF41502	Fluoroborate Electrode, combination, glass body
BF41508	Fluoroborate Electrode, combination, epoxy body
BF4AS01	Fluoroborate Standard, 0.1M $NaBF_4$
BF4AS02	Fluoroboate Standard. 1000 ppm BF_4^{-1}
BF4IS01	Fluoroboate ISA (Ionic Strength Adjustor), 2M $(NH_4)_2SO_4$
R001044	BF41502 and BF41508 Combination Electrode Filling Solution, 0.1M $(NH_4)_2SO_4$

TABLE OF CONTENTS