

NO₂⁻

Orion 93-46,
Orion 97-46, ionplus®

Orion Nitrite Electrode

INSTRUCTION MANUAL



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ORION Series A meters and 900A printer are protected by U.S. patents 5,108,578, 5,198,093 and German patents D334,208 and D346,753.

Sure-Flow electrodes are protected by European Patent 278,979 and Canadian Patent 1,286,720.

ionplus electrodes and Optimum Results solutions are protected by US Patent 5,830,338.

ROSS Ultra electrodes have patents pending.

ORION ORP Standard is protected by US Patent 6,350,367.

ORION Series A conductivity meters are protected by US Patent 5,872,454.

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The specifications, descriptions, drawings, ordering information and part numbers within this document are subject to change without notice.

This publication supersedes all previous publications on this subject.

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GENERAL INFORMATION

Introduction

The Orion 93-46 Half-Cell and Orion 97-46 ionplus® Nitrite Electrodes measure nitrite in aqueous solutions simply, accurately, and economically.

The Orion 97-46 ionplus Nitrite Electrode offers additional benefits from its Sure-Flow® Combination reference design. With this electrode, a separate reference electrode is unnecessary, making it convenient to use with small sample volumes. The free-flowing Sure-Flow junction assures stable, drift-free potentials. When measuring dirty samples that would clog conventional electrode junctions, the Sure-Flow junction can be cleaned by pressing the electrode cap.

General analytical procedures, required solutions, electrode characteristics, and electrode theory are discussed in this manual. Operator instructions for Orion meters are outlined in the individual meter's instruction manual.

Consult Technical Service Chemists for assistance and troubleshooting advice. Please refer to **Troubleshooting** for information or contacting Thermo Electron Corporation.

Required Equipment

Meter

ISE meters, such as Orion EA 940, 920A, 920Aplus, 720A, 720Aplus, 710A, 710Aplus, 290A, or 290Aplus, offering direct concentration readout for specific ions are the easiest to use. If unavailable, use a pH/mV meter with readability to 0.1 mV, such as Orion 420A, 420Aplus, 520A, 520Aplus, 525A or 525Aplus.

Reference Electrode

Orion

For use with Orion 93-46:

Orion 90-01 Single Junction
Reference Electrode

900100

For use with Orion 97-46:

No separate reference electrode required

NA

ionplus® Stirring Accessory or Stir Bars, and Magnetic Stirrer

Stir bar or ionplus stirring accessory, Orion 900060 which slides over the combination electrode body, to mix solution. Micro-stir bars are recommended for small volume measurements.

Graph Paper

4 cycle semi-logarithmic paper for preparing calibration curves (for use with digital pH/mV laboratory meters).

Required Solutions

Distilled or Deionized Water

To prepare all solutions and standards.

Reference Electrode Filling Solution

Required for a complete measuring system

Filling Solutions

Orion

For Orion 97-46 & Orion 90-01:

Optimum Results™ F Filling Solution

900046

Note: Do not use the outer filling solution shipped with the Orion 90-01 reference electrode.

Nitrite Stock Calibration Standards

To prepare daily calibration solutions.

Standards

Orion

0.1 M Nitrite Concentration Standard

954606

100 ppm Nitrite as Nitrogen Standard

Customer
Prepared

To prepare a 100 ppm Nitrite as Nitrogen standard, pipet 71.4 mL of the 0.1 M Nitrite standard, Orion 954606, into a 1 L volumetric flask, and dilute to volume with distilled water.

Additional Solutions

Orion

Nitrite Interference Suppressor Solution (NISS™)

934610

For removal of a variety of interfering anions, including chloride ion, present in samples such as drinking and waste water as well as to buffer the solutions. See **Interferences & pH Effects**

Nitrite Storage Solution (Mixture of 0.1 M Nitrite Concentration Standard and NISS)

Customer
Prepared

Prepare by adding 0.1 mL of Orion 954606 and 50 mL of Orion 934610 to a 100 mL volumetric flask and dilute to volume with distilled water. Alternately, store the nitrite electrode in the lowest concentration of nitrite standard with NISS that was used during calibration.

BEFORE USING THE ELECTRODE

Electrode Assembly and Preparation

Orion 93-46 Nitrite Half Cell Electrode:

Remove the sensing module from the vial. Make sure the rubber electrode washer on the sensing module is in place. See **Figure 1**. Screw the sensing module into the electrode body until finger tight. To ensure electrical continuity, shake down the electrode like a clinical thermometer. The membrane surface should look dark and homogeneous, with no bubbles on the inner surface. Rinse the nitrite electrode with distilled water, then soak in Nitrite Storage Solution, see page 3, for 1 to 2 hours prior to initial use. **Do not immerse the electrode past the rubber electrode washer.** See **Figure 1**.

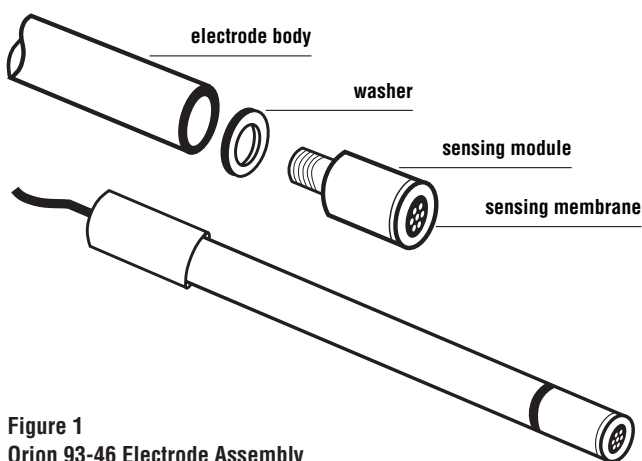


Figure 1
Orion 93-46 Electrode Assembly

Orion 90-01 Single Junction Reference Electrode:

Required for use with Orion 93-46 Nitrite Half Cell Electrode. Fill the reference electrode according to instructions in the reference manual, using Optimum Results™ F, Orion 900046, instead of the Filling Solution provided with the electrode. **Do not use the filling solution shipped with the 90-01 reference electrode because it will interfere with your nitrite measurements.**

Orion 97-46 ionplus® Nitrite Combination Electrode:

This electrode consists of two parts, the sensing module and the electrode handle. See **Figure 2**. **The assembly is different than other Orion electrodes.**

Be careful not to touch the sensing membrane or reference pellet during assembly!

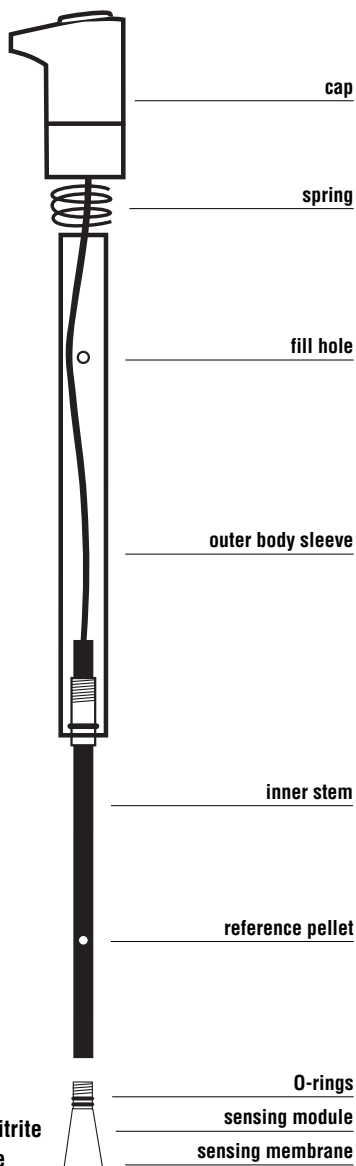


Figure 2
Orion 97-46 ionplus Nitrite
Combination Electrode

1

Remove the sensing module from the vial. Make sure both O-rings are in place. Take the handle from the box.

2

Take the outer body sleeve, with the fill hole end towards the white cap, and gently push the inner stem through the outer body.

3

Slide the outer body sleeve, spring, and cap down the electrode cable until the outer body sleeve is beyond the inner stem.

4

With one hand grasp the middle of the inner stem **without touching the reference pellet**. With your other hand, screw the sensing module onto the stem until it stops and the module is flush against the stem. Then tighten an additional one-quarter turn and stop. **Do not continue to over tighten**. The module should be firmly attached to the stem.

5

Holding the electrode cable, slide the outer body, spring and cap over the inner stem.

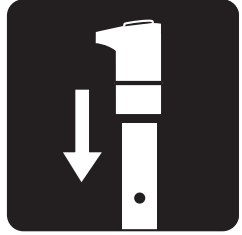
6

Grasp the outer body sleeve, **do not touch the sensing membrane**.

With your other hand, pull on the cable and gently screw the cap onto the inner stem. **Stop** when an opposite force is felt. **Do not over tighten or continue to turn the cap!** The cap will not completely stop! If the inner body turns at all, the cap is too tight. Remove the cap and reassemble.

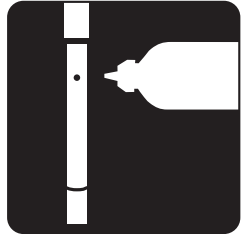
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Hold the electrode with one hand. Press on the top of the cap with your thumb to make sure the electrode has a smooth flushing motion and reseats back onto the module.



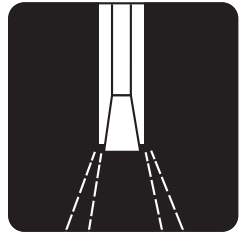
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Fill the outer body with Optimum Results™ F filling solution, Orion 900046, to approximately 1/4 full.



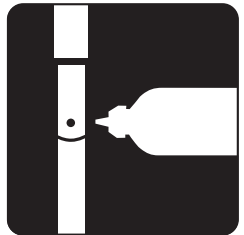
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Press cap to flush out the solution. Release the cap and ensure that the outer body sleeve returns to its original position.

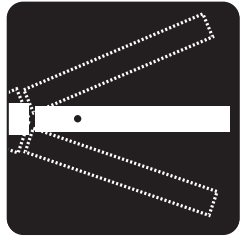


10

Refill the electrode with Optimum Results F filling solution until the fluid level is just below the fill hole.



To ensure electrical continuity, grasp the outer body and cap and shake the sensing module end firmly. Check to make sure the membrane surface is dark and homogeneous with no bubbles on the inner surface.



Electrode reference filling solution should be added each day before use. The filling solution should be no lower than 1 inch from the fill hole and must be above the reference pellet. The filling solution level should always remain 1 inch above the sample level to ensure proper flow rate. Rinse the nitrite electrode with distilled water, then soak in Nitrite Storage Solution, see page 3, for 1 to 2 hours prior to initial use.

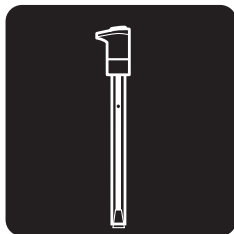
Checking Electrode Operation (Slope)

Use these general instructions to check electrode operation. See individual meter instruction manuals for more specific information.

This procedure measures electrode slope. Slope is defined as the change in millivolts observed with every tenfold (decade) change in concentration. Obtaining the slope value provides the best means for checking electrode operation.

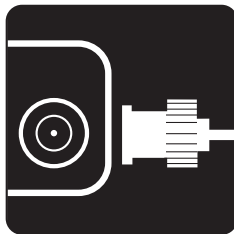
1

If electrode(s) has been stored dry, prepare the electrode(s) as described under the section entitled **Electrode Assembly and Preparation**.



2

Connect electrode(s) to the meter as described in the meter instruction manual.



3

Place 25 mL distilled water into a 150 mL beaker. Add 25 mL NISS™, Orion 934610. Stir thoroughly.



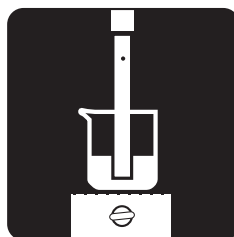
4

Set the meter to the mV mode.



5

Rinse electrode(s) with distilled water, shake dry, and place in the solution prepared in step 3 above.



6

Pipet 0.5 mL of the 100 ppm Nitrite as Nitrogen standard into the beaker. Stir thoroughly.



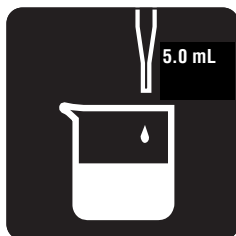
7

When a stable reading is displayed, within 4 to 5 minutes, record the electrode potential in millivolts.



8

Pipet 5.0 mL of the same standard into the same beaker.
Stir thoroughly.



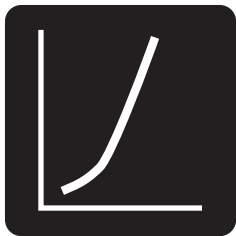
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When a stable reading is displayed, with in 4 to 5 minutes, record the electrode potential in millivolts.



10

The difference between the first and second potential reading is defined as the slope of the electrode. The difference should be in the range of -50 to -60 mV/decade when the solution temperature is 25 ± 5 °C. If the slope is not within this range, resoak the electrode as described under the section entitled **Electrode Assembly and Preparation**. For other troubleshooting techniques refer to the **Troubleshooting** section.



Recommendations for Optimum Results™

Units of Measurement

Measure nitrite in units of moles per liter, parts per million as nitrite, parts per million as nitrogen or any other convenient unit (see **Table 1**).

Table 1
Concentration Unit Conversion Factors

moles per liter	ppm as NO ₂ ⁻	ppm as N
10 ⁻⁴	4.60	1.40
10 ⁻³	46.0	14.0
10 ⁻²	460	140

Sample Requirements

Samples must be aqueous and must not contain organic solvents. Consult Technical Service Chemists for using the electrode in specific applications.

Sample temperature must be less than 40 °C, with samples and standards at the same temperature (within ± 0.5 °C). At the 1.4 ppm as N or 10⁻⁴ M NO₂⁻ level, a 1°C difference in temperature produces about a 2% error (Orion 93-46) and 0.5% error (Orion 97-46). For highly accurate results, use a water bath to control temperature variances.

Dilute concentrated samples, those greater than 200 ppm as N or 0.01 M NO₂⁻, before analysis.

Interferences not removed by the addition of NISS™ should be absent. See section entitled **Interferences** for a list of possible interferences.

Important ISE Measurement Techniques

- Stir all standards and samples at a uniform rate during measurement. Magnetic stirrers may generate sufficient heat to change solution temperature. Place a piece of insulating material such as cork, cardboard or styrofoam between the stir plate and sample beaker.
- Always use fresh standards for calibration.
- Always rinse electrode(s) with distilled water thoroughly between measurements. Shake electrode after rinsing to prevent solution carryover. **Do not wipe or rub the sensing membrane, as you may contaminate and damage the surface.**
- Store the electrode(s) in Nitrite Storage Solution (see **Required Solutions**) or in the lowest standard, containing NISS™, between measurements.
- Allow all standards and samples to come to room temperature (± 0.5 °C) for precise measurements.
- After immersion in solution, check the nitrite electrode for any air bubbles on the membrane surface. Remove air bubbles at the electrode surface by gently tapping the electrode.
- The Orion 93-46 Nitrite Half-Cell Electrode should be submerged approximately half the length of the nitrite module. **DO NOT submerge the nitrite electrode above the rubber electrode washer.** Submerge the reference electrode to the same depth as the nitrite electrode.

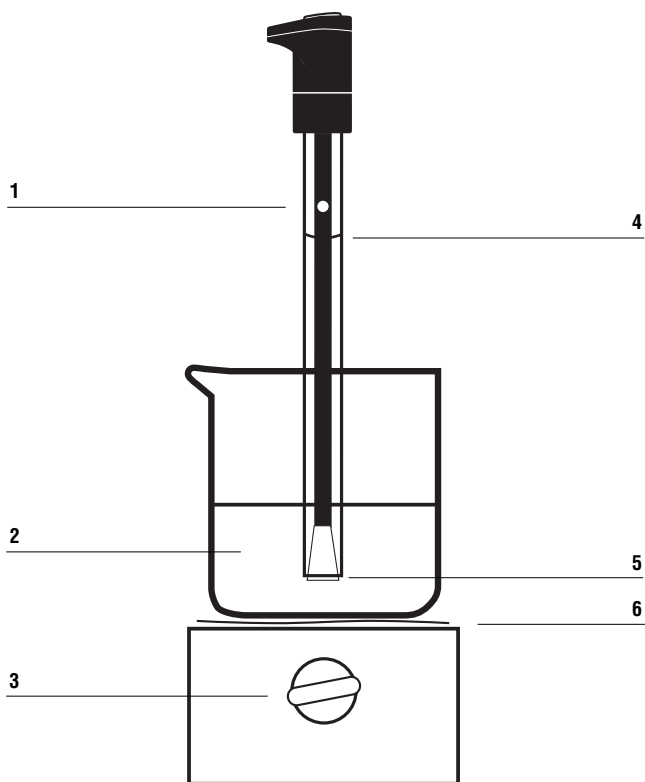


Figure 3

1. Ensure fill hole is not covered during measurement.
2. Use fresh standard.
3. Stir all samples and standards.
4. Filling solution level must be higher than sample level, and at least 1 inch above the reference pellet.
5. Immerse reference junction.
6. Place insulation between stirrer and beaker.

NOTE: Do not submerge nitrite module past the rubber electrode washer (Orion 93-46 only).

Roadway to ionplus® Success!

Guide to measuring techniques

	Direct Measurement	Small Volume Direct Measurement	Low-Level Measurement	Known Addition
Recommended Concentration Range	0.05 to 200 ppm nitrite as N	0.05 to 200 ppm nitrite as N	< 0.5 ppm nitrite as N	0.05 to 200 ppm nitrite as N
Large # of samples	X	X (Orion 97-46 only)	X	X
Small sample volume		X (Orion 97-46 only)		X
Reduced chemical usage		X (Orion 97-46 only)		
Field Measurements	X	X (Orion 97-46 only)		X
Ionic Strength >0.1 M				X
Occasional sampling				X
see page #	18	24	29	35

A variety of analytical techniques are available to the analyst. The best technique is dependent upon the sample matrix. The following section describes the recommended techniques for nitrite determination.

Direct Measurement is a simple procedure for measuring a large number of samples. This method requires only one meter reading for each sample. Calibration is performed in a series of standards. The concentration of the samples is determined by comparison to the standards. Addition of NISS™ to all solutions ensures that samples and standards have similar ionic strength, proper pH, and reduces the effect of interfering ions. When measuring small sample volumes or to reduce chemical usage, follow the **Small Volume Direct Measurement** method, using the Orion 97-46 ionplus Nitrite Electrode.

Low-Level Measurement is similar to Direct Measurement. Use this method when the expected sample concentration is less than 0.5 ppm as N. Using a minimum of three calibration standards compensates for the electrode's non-linear response at low concentrations. This procedure describes the best means of preparing low-level calibration standards.

Known Addition is an alternate method useful when measuring only a few samples, when samples have a high (> 0.1 M) ionic strength, or have a complicated background matrix. Refer to **Theory of Operation** for an explanation of these effects. The electrodes are immersed in the sample solution and an aliquot of a nitrite solution is added to the sample. From the change in potential before and after the addition, the original sample concentration is determined. As in direct calibration, any convenient concentration unit can be used.

MEASUREMENT PROCEDURES

Direct Measurement

The following direct measurement procedures are recommended for “high-level” measurements, when all samples fall within the electrode’s linear range, greater than 0.5 ppm as N or 4×10^{-5} M NO_2^- . A two point calibration is sufficient, though more points can be used if desired. Using ISE meters, such as the Orion 920A, 920Aplus, 720A, 720Aplus, 710A, 710Aplus, 290Aplus, or 290A, read sample concentrations directly from the meter. Refer to the meter’s instruction manual for calibration details. When using a mV meter, prepare a calibration curve on semi-logarithmic graph paper, or a linear regression can be performed at the user’s discretion using a spreadsheet or graphing program.

For Improved Accuracy

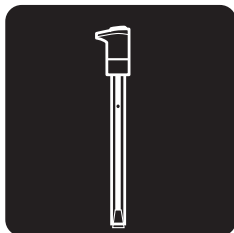
- Bracket standard concentrations around the expected sample concentration.
- Always dilute samples and standards in a 1:1 ratio with NISS™. For example, 25 mL of sample and 25 mL of NISS.
- Verify direct measurement procedure by measuring a standard of known concentration as an unknown or by spiking a sample with nitrite standard.
- For high ionic strength, samples having an ionic strength of 0.1 M or greater, prepare standards with a composition similar to that of the samples, measure the samples using the known addition method, or dilute the samples.
- During calibration, measure the least concentrated standard first, and work up to the most concentrated.
- The best method for preparation of standards is serial dilution. This procedure involves preparing an initial standard that is diluted to prepare a second standard solution, using volumetric glassware. The second is similarly diluted to prepare a third standard, and so on, until the desired range of standards has been prepared.
- Review section entitled **Important ISE Measurement Techniques**.

Direct Measurement Procedure using an ISE meter or a mV meter

See individual meter instruction manuals for more specific calibration information.

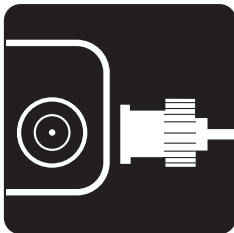
1

Prepare the electrode(s) as described in **Electrode Assembly and Preparation**.



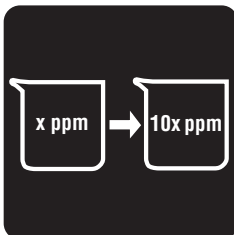
2

Connect electrode(s) to the meter, and adjust the meter to measure concentration for an ISE meter or mV for a mV meter.



3

Prepare at least two standards that bracket the expected sample range and differ in concentration by a factor of ten. Standards can be prepared in any concentration unit to suit the particular analysis requirement. All standards should be at the same temperature as the samples. For details on temperature effects on electrode performance, refer to **Temperature Effects**.



4

Measure 50 mL of each standard and sample into separate 150 mL beakers. Add 50 mL of NISS™ to each standard and sample. **NOTE: Other solution volumes may be used as long as the ration of solution to NISS remains 1:1.** Stir thoroughly.



5

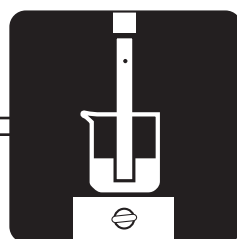
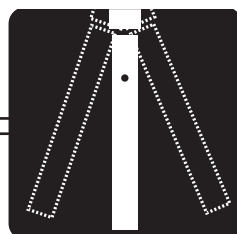
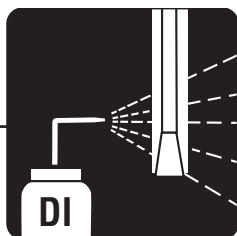
For a ISE meter: Rinse electrode(s) with distilled water, shake dry, and place into the beaker containing the most dilute standard. Wait for a stable reading, calibrate the meter to display the value of the standard as described in the meter instruction manual.

For a mV meter: Rinse electrode(s) with distilled water, shake dry, and place into the beaker containing the most dilute standard. When a stable reading is displayed, record the mV value and corresponding standard concentration.

6

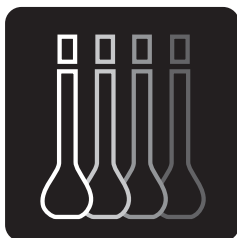
For a ISE meter: Rinse electrode(s) with distilled water, shake dry, and place into the beaker with the next standard. Wait for a stable reading, then adjust the meter to display the value of the second standard, as described in the meter instruction manual.

For a mV meter: Rinse electrode(s) with distilled water, shake dry, and place into the beaker containing the next standard. When a stable reading is displayed, record the mV value and corresponding standard concentration.



7

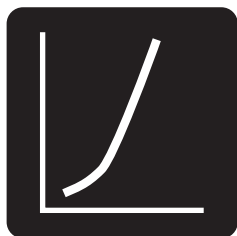
Repeat step 6 for all standards, working from the least concentrated to most concentrated standard.



8

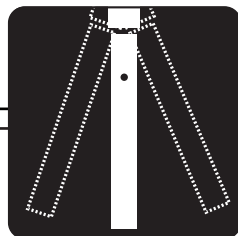
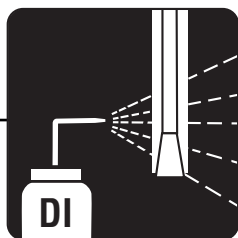
For an ISE meter: Calibration information will be calculated and stored automatically.

For a mV meter: Using semi-logarithmic graph paper, prepare a calibration curve by plotting the millivolt values on the linear axis and the standard concentration values on the logarithmic axis. See **Figure 4**.



9

Rinse electrode(s) with distilled water, shake dry, and place into sample.



10

For an ISE meter: When the electrode stabilizes, the meter will display the sample concentration.

For a mV meter: When the electrode stabilizes, the meter will display the mV value for the sample. Using the calibration curve prepared in step 8, determine the unknown sample concentration.



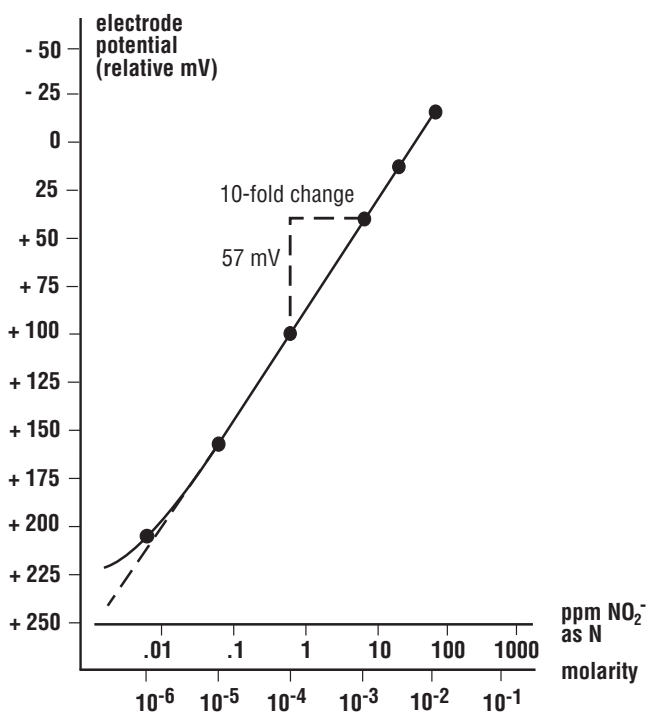


Figure 4
Typical Nitrite Electrode Calibration Curve

During the direct measurement procedure, a calibration curve is constructed automatically by the ISE meter. Alternately, a calibration curve may be plotted by hand using semi-logarithmic paper. Measured electrode potentials of standard solutions are plotted on the linear axis against their concentrations on the log axis. In the linear regions of the curves, only two standards are needed to determine a calibration curve. In non-linear regions, more points must be taken for accuracy. The direct measurement procedures in the manual are given for concentrations in the region of linear electrode response. When measuring in the non-linear region follow the low-level measurement procedure. This curve serves as an example only. Actual mV values may differ.

Small Volume Direct Measurement (Orion 97-46 ionplus® Nitrite Electrode only)

Using the Sure-Flow reference design, the Orion 97-46 ionplus Nitrite Electrode allows measurement of sample volumes as small as 5 mL with a modified direct measurement procedure. This technique is applicable to any sample where reduced chemical usage of standards and Nitrite Interference Suppressor Solution is important. This small volume measurement is well-suited for field testing as the combination reference electrode conveniently reduces equipment, set-up and sampling time. All samples should be greater than 0.5 ppm as N or 4×10^{-5} M NO_2^- . As with the previously described **Direct Measurement** procedure, a two point calibration is sufficient, though more points can be used if desired. Use a direct concentration meter (ISE meter) or a pH/mV meter with 0.1 mV resolution. The following procedure recommends using 10 mL of sample. Smaller sample volumes can be used, as long as the final volume of solution is sufficient to cover the reference junction of the Orion 97-46 electrode. Do not allow the sensing membrane to touch the sample container.

For Improved Accuracy

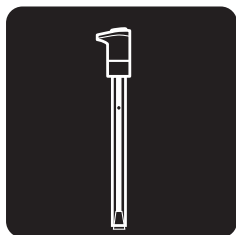
- Use Orion 97-46 ionplus Nitrite Electrode.
- Bracket standard concentrations around the expected sample concentration.
- Always dilute samples and standards in a 1:1 ratio with NISS™.
- Verify this procedure by measuring a standard of known concentration as an unknown or by spiking a sample with nitrite standard.
- For high ionic strength samples, having an ionic strength of 0.1 M or greater, prepare standards with a composition similar to that of the samples, measure the samples using the known addition method, or dilute the samples.
- During calibration, measure the least concentrated standard first, and work up to the most concentrated.
- The best method for preparation of standards is serial dilution. This procedure involves preparing an initial standard that is diluted to prepare a second standard solution, using volumetric glassware. The second is similarly diluted to prepare a third standard, and so on, until the desired range of standards has been prepared.
- Review section entitled **Important ISE Measurement Techniques**.

Small Volume Direct Measurement Procedure using an ISE meter or a mV meter and Orion 97-46 ionplus® Nitrite Electrode

See individual meter instruction manuals for more specific calibration information.

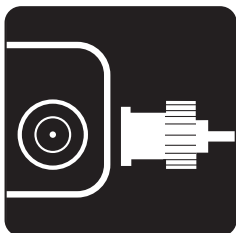
1

Prepare the Orion 97-46 ionplus Nitrite Electrode as described in **Electrode Assembly and Preparation**.



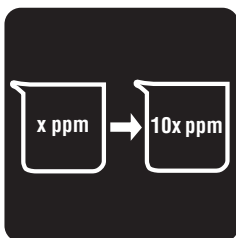
2

Connect the electrode to the meter, and adjust the meter to measure concentration for an ISE meter and mV for a mV meter.



3

Prepare at least two standards that bracket the expected sample range and differ in concentration by a factor of ten. Standards can be prepared in any concentration unit to suit the particular analysis requirement. All standards should be at the same temperature as the samples. For details on temperature effects on electrode performance, refer to **Temperature Effects**.



4

Measure 10 mL of each standard and sample into separate 150 mL beakers. Add 10 mL of NISS™ to each standard and sample.

NOTE: *Other solution volumes may be used, as long as the ratio of solution to NISS remains 1:1.*

Stir thoroughly.



5

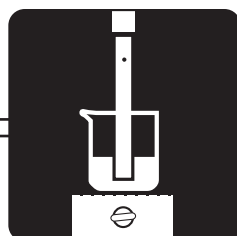
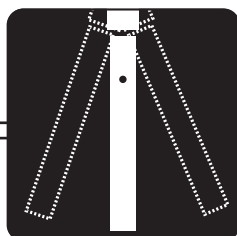
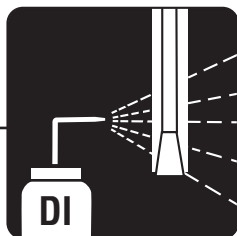
For an ISE meter: Rinse Orion 97-46 ionplus® Nitrite Electrode with distilled water, shake dry, and place into the beaker containing the most dilute standard. Wait for a stable reading, calibrate the meter to display the value of the standard as described in the meter instruction manual.

For a mV meter: Rinse Orion 97-46 ionplus Nitrite Electrode with distilled water, shake dry, and place into the beaker containing the most dilute standard. Wait for a stable reading, record the mV value and corresponding standard concentration.

6

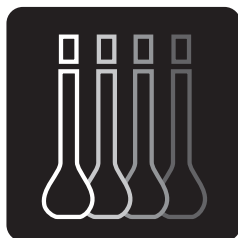
For an ISE meter: Rinse Orion 97-46 ionplus Nitrite Electrode with distilled water, shake dry, and place into the beaker with the next standard. Wait for a stable reading, then adjust the meter to display the value of the second standard, as described in the meter instruction manual.

For a mV meter: Rinse Orion 97-46 ionplus Nitrite Electrode with distilled water, shake dry, and place into the beaker containing the next concentrated standard. Wait for a stable reading, record the mV value and corresponding standard concentration.



7

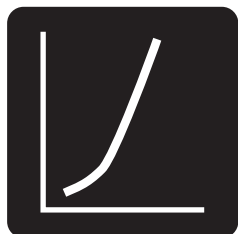
Repeat step 6 for all standards, working from the least concentrated to most concentrated standard.



8

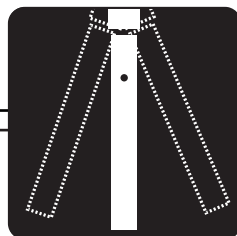
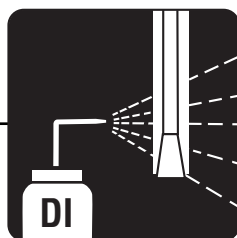
For an ISE meter: Calibration information will be calculated and stored automatically.

For a mV meter: Using semi logarithmic graph paper, prepare a calibration curve by plotting the millivolt values on the linear axis and the standard concentration values on the logarithmic axis. See **Figure 4**.



9

Rinse Orion 97-46 ionplus® Nitrite Electrode with distilled water, shake dry, and place into the sample.



10

For an ISE meter: When the electrode stabilizes, the meter will display the sample concentration.

For a mV meter: When the electrode stabilizes, the meter will display the mV value for the sample. Using the calibration curve prepared in step 8, determine the unknown sample concentration.

RESULT

Low Level Measurements By Direct Measurement

Use this method when measuring solutions with a nitrite concentration of less than 0.5 ppm as N or 4×10^{-5} M NO_2^- , those within the non-linear range of the nitrite electrode. Low-level measurements require at least three standards to compensate for the electrode's non-linearity. Electrode response slows as the ions become more dilute.

For Improved Accuracy

- If some samples have low-level concentrations, and some have higher concentrations, dilute the higher concentrations down to the low-level range. The electrode's time response at low-levels is faster, when it is not also exposed to high concentrations.
- The choice of calibration standard concentrations is important for obtaining the best electrode performance and most rapid analysis time. Here are some guidelines:

Ideally, calibration standard concentrations should bracket the expected sample concentrations.

If some samples have very low concentrations, for example less than 0.1 ppm as N or 7×10^{-6} M NO_2^- , bracketing is not always practical. Generally, the lowest standard concentration should not be much below 0.1 ppm as N or 7×10^{-6} M NO_2^- , since response time is slow at such low levels.

The best results are obtained when the concentration of the highest calibration standard is ten to one hundred times the lowest calibration standard concentration. Space additional standards equally within the range.

If the expected sample concentrations fall within a narrow range (less than one order of magnitude), a ratio of highest to lowest standard concentration of ten should be used.

When measuring sub-ppm levels with Orion 920A, 720A, 710A, or 290A, take advantage of the autoblack feature. It does not require a zero standard, but can perform blank correction as long as the lowest standard concentration is in the non-linear range of the electrode. Electrodes are very slow in the absence of a measurable concentration and a multipoint calibration generally will be less accurate when “zero” is included as a standard. Standard concentrations should be chosen such that the lowest standard value is larger than the blank value obtained, and the second lowest standard should be at least twice that of the lowest. See your A-Series meter operations manual for additional information on blank correction.

When not using an ISE meter, a calibration curve can be drawn on semi-logarithmic graph paper or the data can be processed by means of a spreadsheet or graphing program with a non-linear curve fitting feature.

When using the Orion 920A, 920Aplus, 720A, 720Aplus, 710A, 710Aplus, 290A or 290Aplus, with the autoblack feature, three calibration points are sufficient. If a calibration curve is prepared manually, additional points may be helpful to facilitate drawing the curve.

- Remember to stir all standards and samples at a uniform rate.
- Typical response time for this electrode is approximately 3 minutes. Low-level measurements may take longer to stabilize. For measurements below 0.5 ppm as N or 4×10^{-4} M NO_2^- , wait for 6 minutes or the meter’s “ready” signal, whichever takes longer, before calibrating the meter or recording the sample value. For samples with less than 0.1 ppm nitrite as nitrogen, the analysis time could be as long as 10 minutes.
- After testing samples with high concentrations of nitrite, rinse electrode with distilled water and then soak in Nitrite Storage Solution for 8 - 12 hours before measuring in very low nitrite containing samples.

Low-Level Measurement Procedure using an ISE meter or a mV meter

1

Prepare electrode(s) as described in **Electrode Assembly and Preparation**.



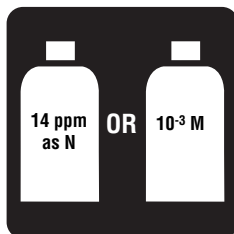
2

Connect the electrode(s) to the meter, and adjust the meter to measure concentration for an ISE meter or mV for a mV meter.



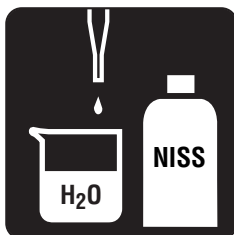
3

Dilute the 0.1 M nitrite standard, Orion 954606, to 14 ppm as N or 10^{-3} M NO_2^- .



4

Measure 25 mL distilled water into 150 mL beaker. Add 25 mL NISS™. Stir thoroughly.



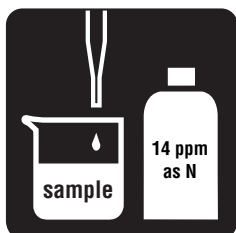
5

Rinse the electrodes with distilled water, shake dry, and place into beaker.



6

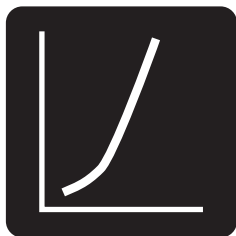
Add increments of the standard to the beaker using steps outlined in **Table 2**.



7

For an ISE meter: Follow meter instruction manual for detailed calibration instructions.

For a mV meter: Record stable millivolt reading after each increment. On semi-logarithmic paper, plot the concentration (log axis) against the millivolt potential (linear axis), see **Figure 4**. Prepare a new low-level calibration curve with fresh standards each day.



8

Measure 25 mL of sample into a beaker. Add 25 mL of NISS™.



9

Rinse the electrode(s) with distilled water, shake dry, and place into the sample. Stir thoroughly.



10

For an ISE meter: When the electrode stabilizes, the meter will display the sample concentration.

For a mV meter: When the electrode stabilizes, the meter will display the sample mV value. Determine the sample concentration corresponding to the measured potential using the low-level calibration curve prepared in step 7.

RESULT

Table 2
Preparing a Calibration Curve For Low-Level Measurements Using a Meter with mV Readout

Step	Graduated			
	Pipet Size	Added Volume	Concentration ppm N	Concentration Molarity
1	1 mL	0.1 mL	0.056	4.0×10^{-6}
2	1 mL	0.2 mL	0.167	1.2×10^{-5}
3	1 mL	1.0 mL	0.710	5.1×10^{-5}
4	2 mL	2.0 mL	1.73	1.2×10^{-4}

Additions of 14 ppm as N or 10^{-3} M NO_2^- standard to 25 mL distilled water, plus 25 mL NISS™.

Known Addition

Known addition, KA, is a convenient technique for measuring samples in the linear response range, greater than 0.5 ppm as N or 4×10^{-5} M NO_2^- , because no calibration curve is needed. Use this method to verify the results of a direct measurement or to minimize existing matrix effects. The sample potential is measured before and after addition of a standard solution. Many meters, such as the Orion 920A and 930 Ionalyzer[®], have the known addition algorithms pre-programmed. This programming makes multiple standard additions to the sample, resulting in more precise results. Having the ability to read the sample concentration result directly from these meters provides a great convenience. Accurate measurement requires that the following conditions be met.

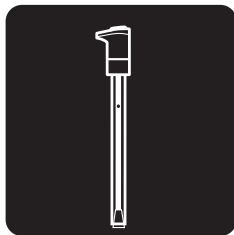
For Improved Accuracy

- Sample concentration should be known to within a factor of three.
- Concentration should approximately double as a result of the addition.
- With double or multiple known addition, the final addition should be 10 to 100 times the sample concentration.
- All samples and standards should be at the same temperature.
- In general, either no complexing agents or a large excess of the complexing agents may be present.
- Standard addition volume should be less than 10% of the sample volume, or standard should be pre-treated with NISS[™] in a 1:1 ratio.
- Dilute samples in a 1:1 ratio with NISS before analysis.
- Review section entitled **Important ISE Measurement Techniques**.

Set-up for Known Addition with all meters

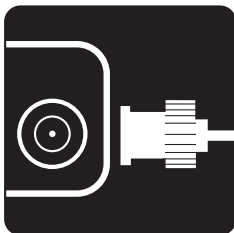
1

Prepare electrode(s) as described in **Electrode Assembly and Preparation**.



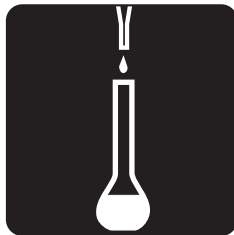
2

Connect electrode(s) to the meter.



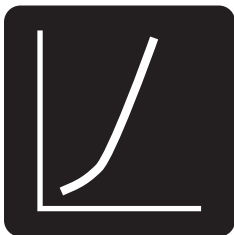
3

Prepare a standard solution that, upon addition to the sample, will cause the concentration of the nitrite to double. Refer to **Table 3** as a guideline.



4

Determine the slope of the nitrite electrode by performing the procedure under **Checking Electrode Operation (Slope)**.



Single Known Addition Measurement Procedure using an ISE meter with KA program

See individual meter instruction manuals for more specific information.

1

Set up the meter to measure in the known addition mode.



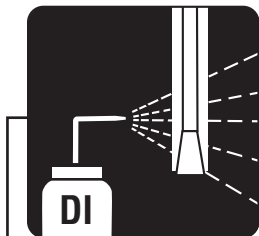
2

Measure 25 mL of the sample into a beaker. Add 25 mL NISS™. Stir thoroughly.



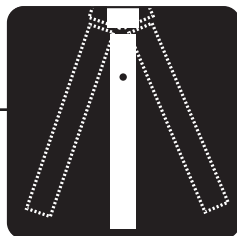
3

Rinse electrode(s) with distilled water, shake dry, and place into sample solution.



4

When a stable reading is displayed, program the meter as described in the meter instruction manual.



5

Pipet the appropriate amount of the standard solution into the beaker. Stir thoroughly.



6.

When a stable reading is displayed, record the sample concentration.



Known Addition Measurement Procedure using a mV meter

1. Set the meter to millivolt mode.
2. Measure 25 mL of the sample into a 150 mL beaker. Add 25 mL NISS™. Stir thoroughly.
3. Rinse electrode(s) with distilled water, blot dry and place into sample solution.
4. When a stable reading is displayed, record the mV value as E_1 .
5. Pipet the appropriate amount of standard solution into the beaker. See **Table 3**. Stir thoroughly.
6. When a stable reading is displayed, record the mV value as E_2 . Subtract the first reading from the second to find ΔE .
7. From **Table 5**, find the Q value that corresponds to the change in potential, ΔE . To determine the original sample concentration, multiply Q by the concentration of the added standard times two:

$$C_{\text{sam}} = QC_{\text{std}}$$

where:

C_{std} = standard concentration

C_{sam} = sample concentration

Q = reading from known addition table

The table of Q values is calculated for a 10% total volume change for electrodes with slopes of -57.2, -58.2, -59.2, and -60.1 mV/decade.

The equation for the calculation of Q for different slopes and volume changes is given below:

$$Q = \frac{p \cdot r}{((1 + p)10^{\Delta E/S}) - 1}$$

where:

ΔE = $E_2 - E_1$

S = slope of the electrode

p = (volume of standard)/(volume of sample & NISS)

r = (volume of sample & NISS) / (volume of sample)

Table 3

Volume of Addition	Concentration of Standard Before Adding NISS
0.5 mL	50 x sample concentration
2.5 mL	10 x sample concentration
5 mL*	5 x sample concentration

*Most convenient volume to use, valid for Q Tables

If it is more convenient, a simple spreadsheet can be set up to calculate known addition results, using any ratios of sample and addition. A typical worksheet is shown in **Table 4**. The numbers shown are examples, but the formulas and their locations should be copied exactly.

Table 4
Calculating known addition for nitrite samples using Lotus, Excel or Quattro Spreadsheet

A	B	C
1	Enter Values	
2	VOL. OF SAMPLE & NISS™, ML	50
3	VOL. OF ADDITION, ML	5
4	CONCENTRN. OF ADDITION	10
5	VOL. OF SAMPLE	25
6	INITIAL MV READING	-45.3
7	FINAL MV READING	-63.7
8	ELECTRODE SLOPE	-59.2
9		
10		DERIVED VALUES
11	ΔE	+C7-C6
12	p TERM	+C3/C2
13	ANTILOG TERM	+10^(C11/C8)
14	r TERM	+C2/C5
15	Q TERM	+C12*C14/{[(1+C12)*C13]-1}
16	CALCULATED INITIAL CONC. IN SAME UNIT AS ADDITION	+C15*C4

NOTE: for Excel, use = instead of + at start of formula

Table 5**Known Addition for an added volume one-tenth the sample volume.
Slopes (in the column headings) are in units of mV/decade**

ΔE	Q, Concentration Ratio				
	Monovalent	-57.2	-58.2	-59.2	-60.1
5.0		0.2917	0.2957	0.2996	0.3031
5.2		0.2827	0.2867	0.2906	0.2940
5.4		0.2742	0.2781	0.2820	0.2854
5.6		0.2662	0.2700	0.2738	0.2772
5.8		0.2585	0.2623	0.2660	0.2693
6.0		0.2512	0.2550	0.2586	0.2619
6.2		0.2443	0.2480	0.2516	0.2548
6.4		0.2377	0.2413	0.2449	0.2480
6.6		0.2314	0.2349	0.2384	0.2416
6.8		0.2253	0.2288	0.2323	0.2354
7.0		0.2196	0.2230	0.2264	0.2295
7.2		0.2140	0.2174	0.2208	0.2238
7.4		0.2087	0.2121	0.2154	0.2184
7.6		0.2037	0.2070	0.2102	0.2131
7.8		0.1988	0.2020	0.2052	0.2081
8.0		0.1941	0.1973	0.2005	0.2033
8.2		0.1896	0.1927	0.1959	0.1987
8.4		0.1852	0.1884	0.1914	0.1942
8.6		0.1811	0.1841	0.1872	0.1899
8.8		0.1770	0.1801	0.1831	0.1858
9.0		0.1732	0.1762	0.1791	0.1818
9.2		0.1694	0.1724	0.1753	0.1779
9.4		0.1658	0.1687	0.1716	0.1742
9.6		0.1623	0.1652	0.1680	0.1706
9.8		0.1590	0.1618	0.1646	0.1671
10.0		0.1557	0.1585	0.1613	0.1638
10.2		0.1525	0.1553	0.1580	0.1605
10.4		0.1495	0.1522	0.1549	0.1573
10.6		0.1465	0.1492	0.1519	0.1543
10.8		0.1437	0.1463	0.1490	0.1513
11.0		0.1409	0.1435	0.1461	0.1485
11.2		0.1382	0.1408	0.1434	0.1457
11.4		0.1356	0.1382	0.1407	0.1430
11.6		0.1331	0.1356	0.1381	0.1404
11.8		0.1306	0.1331	0.1356	0.1378

ΔE	Q, Concentration Ratio			
	Monovalent	-57.2	-58.2	-59.2
12.0	0.1282	0.1307	0.1331	0.1353
12.2	0.1259	0.1283	0.1308	0.1329
12.4	0.1236	0.1260	0.1284	0.1306
12.6	0.1214	0.1238	0.1262	0.1283
12.8	0.1193	0.1217	0.1240	0.1261
13.0	0.1172	0.1195	0.1219	0.1239
13.2	0.1152	0.1175	0.1198	0.1218
13.4	0.1132	0.1155	0.1178	0.1198
13.6	0.1113	0.1136	0.1158	0.1178
13.8	0.1094	0.1117	0.1139	0.1159
14.0	0.1076	0.1098	0.1120	0.1140
14.2	0.1058	0.1080	0.1102	0.1121
14.4	0.1041	0.1063	0.1084	0.1103
14.6	0.1024	0.1045	0.1067	0.1086
14.8	0.1008	0.1029	0.1050	0.1069
15.0	0.0992	0.1012	0.1033	0.1052
15.5	0.0953	0.0973	0.0994	0.1012
16.0	0.0917	0.0936	0.0956	0.0974
16.5	0.0882	0.0902	0.0921	0.0938
17.0	0.0850	0.0869	0.0887	0.0904
17.5	0.0819	0.0837	0.0856	0.0872
18.0	0.0790	0.0808	0.0825	0.0841
18.5	0.0762	0.0779	0.0797	0.0813
19.0	0.0736	0.0753	0.0770	0.0785
19.5	0.0711	0.0727	0.0744	0.0759
20.0	0.0687	0.0703	0.0719	0.0734
20.5	0.0664	0.0680	0.0696	0.0710
21.0	0.0642	0.0658	0.0673	0.0687
21.5	0.0621	0.0637	0.0652	0.0666
22.0	0.0602	0.0617	0.0631	0.0645
22.5	0.0583	0.0597	0.0612	0.0625
23.0	0.0564	0.0579	0.0593	0.0606
23.5	0.0547	0.0561	0.0575	0.0588
24.0	0.0530	0.0544	0.0558	0.0570
24.5	0.0514	0.0528	0.0541	0.0553

ΔE	Q, Concentration Ratio			
	Monovalent	-57.2	-58.2	-59.2
25.0	0.0499	0.0512	0.0525	0.0537
25.5	0.0484	0.0497	0.0510	0.0522
26.0	0.0470	0.0483	0.0495	0.0507
26.5	0.0456	0.0469	0.0481	0.0492
27.0	0.0443	0.0455	0.0468	0.0479
27.5	0.0431	0.0443	0.0455	0.0465
28.0	0.0419	0.0430	0.0442	0.0452
28.5	0.0407	0.0418	0.0430	0.0440
29.0	0.0395	0.0407	0.0418	0.0428
29.5	0.0385	0.0396	0.0407	0.0417
30.0	0.0374	0.0385	0.0396	0.0406
30.5	0.0364	0.0375	0.0385	0.0395
31.0	0.0354	0.0365	0.0375	0.0384
31.5	0.0345	0.0355	0.0365	0.0374
32.0	0.0335	0.0345	0.0356	0.0365
32.5	0.0327	0.0336	0.0346	0.0355
33.0	0.0318	0.0328	0.0337	0.0346
33.5	0.0310	0.0319	0.0329	0.0337
34.0	0.0302	0.0311	0.0320	0.0329
34.5	0.0294	0.0303	0.0312	0.0321
35.0	0.0286	0.0295	0.0305	0.0313
35.5	0.0279	0.0288	0.0297	0.0305
36.0	0.0272	0.0281	0.0290	0.0298
36.5	0.0265	0.0274	0.0282	0.0290
37.0	0.0258	0.0267	0.0275	0.0283
37.5	0.0252	0.0260	0.0269	0.0276
38.0	0.0246	0.0254	0.0262	0.0270
38.5	0.0240	0.0248	0.0256	0.0263
39.0	0.0234	0.0242	0.0250	0.0257
39.5	0.0228	0.0236	0.0244	0.0251
40.0	0.0223	0.0230	0.0238	0.0245
40.5	0.0217	0.0225	0.0232	0.0239
41.0	0.0212	0.0219	0.0227	0.0234
41.5	0.0207	0.0214	0.0221	0.0228
42.0	0.0202	0.0209	0.0216	0.0223

ΔE	Q, Concentration Ratio				
	Monovalent	-57.2	-58.2	-59.2	-60.1
42.5		0.0197	0.0204	0.0211	0.0218
43.0		0.0192	0.0199	0.0206	0.0213
43.5		0.0188	0.0195	0.0202	0.0208
44.0		0.0183	0.0190	0.0197	0.0203
44.5		0.0179	0.0186	0.0192	0.0198
45.0		0.0175	0.0181	0.0188	0.0194
45.5		0.0171	0.0177	0.0184	0.0190
46.0		0.0167	0.0173	0.0179	0.0185
46.5		0.0163	0.0169	0.0175	0.0181
47.0		0.0159	0.0165	0.0171	0.0177
47.5		0.0156	0.0162	0.0168	0.0173
48.0		0.0152	0.0158	0.0164	0.0169
48.5		0.0148	0.0154	0.0160	0.0166
49.0		0.0145	0.0151	0.0157	0.0162
49.5		0.0142	0.0147	0.0153	0.0158
50.0		0.0139	0.0144	0.0150	0.0155
50.5		0.0135	0.0141	0.0146	0.0151
51.0		0.0132	0.0138	0.0143	0.0148
51.5		0.0129	0.0135	0.0140	0.0145
52.0		0.0126	0.0132	0.0137	0.0142
52.5		0.0124	0.0129	0.0134	0.0139
53.0		0.0121	0.0126	0.0131	0.0136
53.5		0.0118	0.0123	0.0128	0.0133
54.0		0.0116	0.0120	0.0125	0.0130
54.5		0.0113	0.0118	0.0123	0.0127
55.0		0.0110	0.0115	0.0120	0.0125
55.5		0.0108	0.0113	0.0118	0.0122
56.0		0.0106	0.0110	0.0115	0.0119
56.5		0.0103	0.0108	0.0113	0.0117
57.0		0.0101	0.0106	0.0110	0.0114
57.5		0.0099	0.0103	0.0108	0.0112
58.0		0.0097	0.0101	0.0105	0.0110
58.5		0.0095	0.0099	0.0103	0.0107
59.0		0.0093	0.0097	0.0101	0.0105
59.5		0.0091	0.0095	0.0099	0.0103
60.0		0.0089	0.0093	0.0097	0.0101

ELECTRODE STORAGE

Orion 93-46 Nitrite Half-Cell Electrode

The nitrite sensing module should be kept in the glass vial until used. The assembled electrode can be stored in Nitrite Storage Solution. For long periods of time (over 2-3 days), disassemble the nitrite electrode, rinse thoroughly with distilled water, blot dry, and store the module in its vial. **NOTE: Over time NISS™ will discolor the membrane. As long as the slope of the electrode remains between -50 to -60 mV/decade the electrode performance is not compromised.**

Orion 97-46 ionplus® Nitrite Electrode

The solution in the Orion 97-46 ionplus Combination Nitrite Electrode should not be allowed to evaporate and crystallize around the junction.

For short periods of time (2-3 days):

Store the assembled electrode in Nitrite Storage Solution (see **Required Solutions**).

For storage longer than 2-3 days:

Drain the reference compartment of the electrode and flush it with distilled water. Disassemble the electrode, see **Figure 2**, as follows to remove the nitrite sensing module:

1. Grasp the outer body sleeve. With your other hand, unscrew the electrode cap. Allow cap and spring assembly to slide down the electrode cable.
2. Push the inner stem of the electrode handle out through the outer electrode sleeve, exposing the sensing module.
3. Rinse the inner stem and module well with distilled water. Blot dry gently in order not to damage the sensing membrane.
4. Carefully unscrew the sensing module from the inner stem, taking care not to touch the sensing membrane.

5. Place the nitrite sensing module into the glass vial until it is to be used again.
6. Gently dry the inside of the inner stem and O-ring area with a lint-free tissue and reassemble the electrode handle. Store dry.

NOTE: Over time NISS™ will discolor the membrane. As long as the slope of the electrode remains between -50 to -60 mV/decade the electrode performance is not compromised.

Orion 90-01 Single Junction Reference Electrode

The Orion 90-01 Reference Electrode may be stored in air between sample measurements (up to 1 hour).

For short periods of time (2-3 days):

The Orion 90-01 may be stored in its filling solution or distilled water. Do not allow the solution inside the electrode to evaporate and crystallize.

For storage longer than 2-3 days:

Drain the reference electrode completely, rinse with distilled water, and store dry.

TROUBLESHOOTING

Troubleshooting Checklist

Symptom	Possible Causes
Off-scale or Over-range reading	Defective meter
	Defective sensing module
	Electrodes not plugged in properly
	Module not installed properly
	Reference electrode junction is dry (Orion 97-46 & 90-01)
	No reference electrode (Orion 93-46)
	Reference electrode chamber not filled (Orion 97-46 & 90-01)
	Interior of membrane not thoroughly wetted
	Air bubble on membrane
	Electrodes not in solution
Noisy or unstable readings (readings continuously or rapidly changing)	Defective meter
	Meter or stirrer improperly grounded
	Module not installed properly
	Air bubble on membrane
	Interior of membrane not thoroughly wetted
	Wrong reference electrode
	NISS™ not used
	Orion 97-46 Electrode Cap on too tight

Solution

Check meter with shorting cap (See meter instruction manual)

Refer to **Troubleshooting Guide**

Unplug electrodes and reseal

Check electrode assembly

Through the reference junction, expel a few drops of filling solution

Use Orion 90-01 Reference Electrode
(with Orion 93-46 Nitrite Electrode)

Be sure reference electrode is filled with correct solution.
See **Electrode Assembly and Preparation**.

Tap module gently or shake down like a clinical thermometer

Remove air bubble by gently tapping electrode

Put electrodes in solution

Check meter with shorting cap. (See meter instruction manual)

Check meter and stirrer for grounding

Check **Before Using the Electrode**

Remove air bubble by gently tapping electrode

Tap module gently or shake down like a clinical thermometer.

Use Orion 90-01 Single Junction Reference Electrode
(with Orion 93-46 Nitrite Electrode) Do not use calomel or Ag/AgCl
(frit-or fiber-type) reference electrode.

Use recommended NISS™, Orion 934610

Reassemble electrode. See **Electrode Assembly and Preparation**.

Troubleshooting Checklist (cont.)

Symptom	Possible Causes
Drift (Reading slowly changing in one direction)	Samples and standards at different temperatures
	Electrode exposed to interference
	Incorrect reference filling solution
	Membrane may contain a surface layer of contaminant
	Orion 97-46 Electrode Cap on too tight
Low slope or No slope	Electrodes not properly conditioned
	Standards contaminated or incorrectly made
	NISS™ not used
	Standard used as NISS
	Orion 97-46 Electrode Cap on too tight
	Defective sensing module
	Electrode exposed to interferences
“Wrong Answer” (But calibration curve is OK)	Incorrect scaling of semi-log paper
	Incorrect standards
	Incorrect sign
	Wrong units used
	Complexing agents in sample

Solution

Allow solutions to come to room temperature $\pm 0.5^\circ\text{C}$ before measurement.

See **Electrode Assembly and Preparation**. Use NISS™, Orion 934610.
See **Interferences**.

Use recommended filling solution. See **Electrode Assembly and Preparation**.

Rinse electrode with distilled water and soak in Nitrite Storage Solution for 1 hour

Reassemble electrode. See **Electrode Assembly and Preparation**.

See **Electrode Assembly and Preparation**

Prepare fresh standards

Use NISS, Orion 934610

Use NISS!

Reassemble electrode. See **Electrode Assembly and Preparation**.

Refer to **Troubleshooting Guide**

See **Interferences**. Use NISS, Orion 934610

Plot millivolts on the linear axis. On the log axis, be sure concentration numbers within each decade are increasing with increasing concentration

Prepare fresh standards

Be sure to note sign of millivolt value correctly

Apply correct conversion factor: $10^{-3}\text{ M} = 46\text{ ppm as NO}_2^- = 14\text{ ppm as N}$

Use known addition, or a decomplexing procedure

For additional information on blank correction with your A-Series meter, see your meter operations manual.

Troubleshooting Guide

The most important principle in troubleshooting is to isolate the components of the system and check each in turn. The components of the system are: 1) Meter 2) Electrodes 3) Standard 4) Sample and 5) Technique. See also **Important ISE Measurement Techniques and For Improved Accuracy**.

Meter

The meter is the easiest component to eliminate as a possible cause of error. Thermo Orion meters are provided with an instrument checkout procedure in the instruction manual and a shorting cap for convenience in troubleshooting. Consult the manual for complete instructions and verify that the instrument operates as indicated and is stable in all steps.

Electrodes

1. Rinse electrodes thoroughly with distilled water.
2. Check electrode operation (slope), see **Checking Electrode Operation**.
3. If electrode fails this procedure, resoak nitrite electrode as directed in **Electrode Assembly and Preparation**.

Clean reference electrode as described in reference electrode instruction manual (used with Orion 93-46 only).

4. Repeat step 2.
- 5a. For the 93-46 Nitrite Half-Cell Electrode

If the electrodes still do not perform as described, determine whether the nitrite or reference electrode is at fault. To do this, substitute a known working electrode for the electrode in question and repeat the slope check.

5b. For the 97-46 ionplus® Nitrite Electrode:

Disassemble and reassemble the electrode, taking care not to overtighten the cap.

If the electrode still does not perform as described, replace the sensing module and repeat the slope check. If the slope check still fails, replace the electrode handle.

6. If the stability and slope check out properly, but measurement problems persist, the sample may contain interferences or complexing agents, or the technique may be in error. See **Standard**, **Sample**, and **Technique** sections.
7. Before replacing a “faulty” electrode, or if another electrode is not available for test purposes, review the instruction manual and be sure to:
 - Clean the electrode thoroughly
 - Prepare the electrode properly
 - Check that Orion 97-46 Electrode Cap is not too tight
 - Use proper filling solution, NISS™, and standards
 - Measure correctly
 - Review **Troubleshooting Checklist**

Standard

The quality of results depends greatly upon the quality of the standards. Always prepare fresh standards when problems arise as it could save hours of frustrating troubleshooting! Error may result from contamination of prepared standards, quality of dilution, distilled water, or a numerical error in calculating the concentrations.

The best method for preparation of standards is by serial dilution. This procedure involves preparing an initial standard that is diluted to prepare a second standard solution, using volumetric glassware. The second is similarly diluted to prepare a third standard, and so on, until the desired range of standards has been prepared.

Sample

If the electrodes work properly in the standards but not in the sample, look for possible interferences, complexing agents, or substances that could affect response or physically damage the sensing electrode or the reference electrode. If possible, determine the composition of the samples and check for problems. See **Sample Requirements, Interferences, and Specifications**.

Technique

Check the method of analysis for compatibility with your sample. Direct measurement may not always be the method of choice. If a large amount of complexing agents is present, known addition may be best. If working at low levels, be sure to follow the low level measurement technique. Also, be sure that the expected concentration of the ion of interest is within the electrode's limits of detection. If problems persist, review operational procedures and instruction manuals to be sure that proper technique has been followed. Read **Important ISE Measurement Techniques and Measurement Procedures**.

Assistance

After troubleshooting all components of your measurement system, contact The Technical EdgeSM for Orion products. Within the United States call 1.800.225.1480, outside the United States call 978.232.6000 or fax 978.232.6031. In Europe, the Middle East and Africa, contact your local authorized dealer. For the most current contact information, visit www.thermo.com.

ELECTRODE CHARACTERISTICS

Electrode Response

The electrode potential plotted against concentration on semi-logarithmic paper results in a straight line until concentration reaches 0.7 ppm as N or 5×10^{-5} M NO_2^- , with a slope of about -50 to -60 mV per decade. See **Figure 4**.

The electrode exhibits good time response (98% in one minute or less) for nitrite concentrations above 10^{-4} M NO_2^- . Below this value response times vary from 2 to 5 minutes. Below 0.1 ppm as N or 7×10^{-6} M NO_2^- response times may vary from 5 to 10 minutes. See **Figure 5**.

Limits of Detection

In pure sodium nitrite solutions, the upper limit of detection is 1.4×10^{-2} M. At nitrite concentrations greater than 10^{-2} M, dilute the sample. The lower limit of detection is determined by the slight water solubility of the ion exchanger, which causes deviation from theoretical response. **Figure 4** shows the theoretical response at low levels of nitrite compared to the actual response. If nitrite measurements are made below 0.5 ppm as N or 4.0×10^{-5} M NO_2^- a low-level measurement procedure is recommended.

Reproducibility

Reproducibility is limited by factors such as temperature fluctuations, drift, and noise. Within the electrode operating range, reproducibility is independent of concentration. With calibration every hour, direct electrode measurements reproducible to $\pm 2\%$ can be obtained.

Temperature Effects

Since electrode potentials are affected by changes in temperature, samples and standard solutions should be within $\pm 0.5^\circ\text{C}$ ($\pm 1^\circ\text{F}$) of each other. At the 10^{-4} M level, a 1°C difference in temperature results in a 0.5% error with Orion 97-46 ionplus® Nitrite Electrode and 2.0% with Orion 93-46 Nitrite Electrode.

The absolute potential of the reference electrode changes slowly with temperature because of the solubility equilibria on which the electrode depends. The slope of the nitrite electrode also varies with temperature, as indicated by the factor "S" in the Nernst equation.

Values for the change in slope for nitrite ion are given in **Table 6**. If temperature changes occur, meter and electrodes should be recalibrated.

The electrode can be used at temperatures from 0 to 40°C, provided that temperature equilibrium has occurred. For use at temperatures substantially different from room temperature, equilibrium times of up to one hour are recommended.

The isopotential concentration, C_{iso} , for Orion 97-46 ionplus® Nitrite Electrode is approximately 4.0×10^{-3} M.

Table 6
Values of Electrode Slope vs. Temperature

T°C	S	T°C	S
0	-54.20	30	-60.15
10	-56.18	40	-62.13
20	-58.16	50	-64.11
25	-59.16		

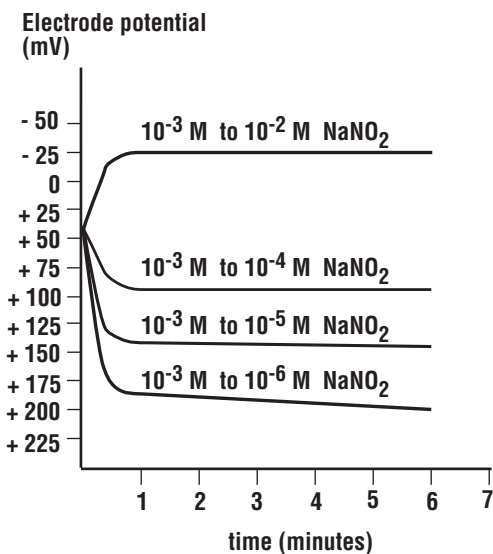


Figure 5
Typical Electrode Response to NaNO₂

Interferences

Some anions, if present at high enough levels, are electrode interferences and will cause measurement errors. **Table 7** indicates levels of common anions that will cause 10% errors at different concentrations of nitrite.

In many samples anions listed in **Table 7** are absent or insignificantly low. Many of the interferences can be removed or minimized by the following procedures (letters refer to letters in **Table 7**):

- a. These interferences can be minimized by precipitation with silver. Use NISS™ in samples to effect removal of these interferences. 0.02 M of the interfering ion is removed by a 1:1 addition, in the absence of other interfering ions.
- b. Bicarbonate can be removed by acidifying the sample with NISS. 0.01 M of the interfering ion is removed by a 1:1 addition in the absence of other interfering ions.

NOTE: Use of any of the above procedures require similar treatment of standards as well as samples.

Thermo Orion manufactures a Nitrite Interference Suppressor Solution (Orion 934610), recommended for the removal of a variety of interfering anions and to buffer the sample and standards.

The Nitrite Interference Suppressor Solution is mixed in an equal volume with samples and also with standards. For example, 25 mL of sample would be mixed with 25 mL of the Nitrite Interference Suppressor Solution. This procedure ensures that samples and standards have a similar background, and that no correction factor is needed for the dilution.

If the electrode is exposed to high levels of interfering ions, it may become drift and sluggish in response. When this happens, restore normal performance as mentioned in **For Improved Accuracy** for low level measurement.

When the level of interferences in samples is constant, it is sometimes possible to measure nitrite accurately when interference levels are higher than those in **Table 7**. Call or write Technical Service Chemists for more information. See **Assistance**.

Electrode Life

Each sensing module should last at least six months in normal laboratory use. In time, electrode slope will decrease and readings will start to drift, indicating that the module should be changed. Before replacement, refer to **Troubleshooting** Checklist, to make sure that the difficulties are caused by the sensing module.

Table 7
Levels of Possible Interferences Causing a 10% Error

Interferences to NO₂⁻			
Moles/Liter	10⁻⁴ M	10⁻³ M	10⁻² M
Salicylate	7.15 x 10 ⁻⁵	7.15 x 10 ⁻⁴	7.15 x 10 ⁻³
(a) Iodide	1.65 x 10 ⁻⁴	1.65 x 10 ⁻³	1.65 x 10 ⁻²
(a) Bromide	9.9 x 10 ⁻³	9.9 x 10 ⁻²	9.9 x 10 ⁻¹
Chlorate	2.7 x 10 ⁻²	2.7 x 10 ⁻¹	2.7
Acetate	1.7 x 10 ⁻²	1.7 x 10 ⁻¹	1.7
(b) Bicarbonate	2.0 x 10 ⁻²	2.0 x 10 ⁻¹	2
Nitrate (as N)	2.0 x 10 ⁻²	2.0 x 10 ⁻¹	2
Sulfate	1.5 x 10 ⁻²	1.5 x 10 ⁻¹	1.5
(a) Chloride	1.2 x 10 ⁻²	1.2 x 10 ⁻¹	1.2
Perchlorate	1.2 x 10 ⁻²	1.2 x 10 ⁻¹	1.2
Fluoride	1.1 x 10 ⁻²	1.1 x 10 ⁻¹	1.1

Interferences NO₂⁻ as N			
ppm	1 ppm	10 ppm	100 ppm
Salicylate	7	70	700
(a) Iodide	15	150	1,500
(a) Bromide	565	5,650	56,500
Chlorate	1,629	16,290	162,900
Acetate	717	7,170	71,700
(b) Bicarbonate	873	8,730	87,300
Nitrate (as N)	203	2,030	20,300
Sulfate	1,059	10,590	105,900
(a) Chloride	315	3,150	31,500
Perchlorate	834	8,340	83,400
Fluoride	150	1,500	15,000

Letters refer to corresponding letter in **Interferences** section.

Chemistry of Nitrite

The following considerations should be kept in mind when sampling or storing nitrite solutions.

1. Nitrite ion forms a weak acid in solutions below about pH 4. The highly acidic solutions decompose slowly, resulting in a loss of nitrite. The rate is a few percentages per hour, and is increased by exposure to air, or rapid stirring. Alkaline nitrite solutions are much more stable.
2. Nitrite can be oxidized to nitrate by many common oxidizing reagents, such as hydrogen peroxide, chlorine or bromine, and ozone.
3. Many reducing agents are oxidized by nitrite, with the nitrite being converted to NO. Examples are iodine, ferrous ion, sulfite or sulfur dioxide, and stannous tin.
4. Amines react with nitrous acid to form nitrogen and nitrosamines; sulfamic acid reacts rapidly to form nitrososulfamic acid.
5. Nitrite has been reported to form complexes with mercury, copper, cadmium, lead and some other metals at high levels, but at lower levels (100 ppm for copper and cadmium, and 1 ppm for lead) these complexes are not significant, and need not be of concern.

pH Effects

Although the electrode can be used in a pH range of 4 to 8, the optimum pH range for the nitrite electrode is pH 4 to 5. All samples and standards should be adjusted with the Nitrite Interference Suppressor Solution (NISS™) in a 1:1 ratio to buffer solutions to this pH range. A constant pH must be maintained on both samples and standards.

It is not recommended to use this electrode at the extreme pH ranges since the sensitivity will be limited. At high pH, the hydroxide interference will become more pronounced, and at low pH the membrane will degrade. For best results, all samples and standards should be buffered or diluted to the recommended pH range of pH 4 to 5.

Theory of Operation

The nitrite electrode consists of an electrode body and a replaceable pretested sensing module. The sensing module contains a liquid internal filling solution in contact with a gelled organophilic membrane containing a nitrite selective ion exchanger. See **Figure 6**.

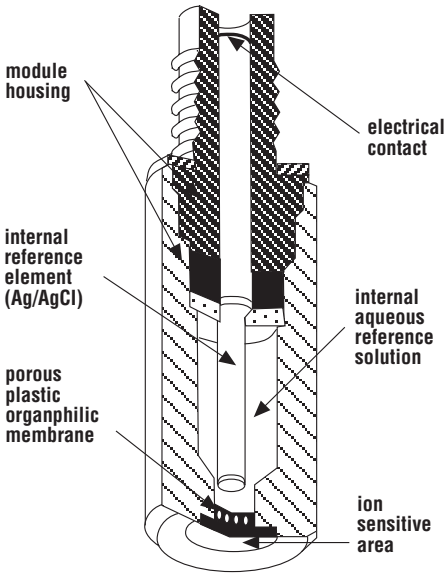


Figure 6
Example of Ion Sensing Module

When the membrane is in contact with a nitrite solution, an electrode potential develops across the membrane. This potential, which depends on the level of free nitrite ion in solution, is measured against a constant reference potential with a pH/mV meter or specific ion meter. The measured potential corresponding to the level of nitrite ion in solution is described by the Nernst equation:

$$E = E_0 + S \log (A)$$

where:

- E = measured electrode potential
- E_0 = reference potential (a constant)
- A = nitrite ion level in solution
- S = electrode slope (about -57 mV per decade)

The level of nitrite ion, A , is the activity of “effective concentration” of free nitrite ion in solution. The total nitrite concentration, C_t , includes some bound or complexed ions as well as free ions, whose concentration is:

$$C_t = C_f + C_b$$

where:

C_b = concentration of nitrite ions in all bound or complexed forms

C_f = concentration of free nitrite ions

The nitrite ion activity is related to free nitrite ion concentration by the activity coefficient:

$$A = \gamma C_f$$

Ionic activity coefficients are variable and largely depend on total ionic strength. Ion strength is defined as:

$$\text{Ionic strength} = 1/2 \sum (C_i Z_i^2)$$

C_i = concentration of ion i

Z_i = charge of ion i

If the background ionic strength is high and constant relative to the sensed ion concentration, the activity coefficient is constant and activity is directly proportional to concentration.

For the nitrite electrode use of Nitrite Interference Suppressor Solution (NISS™) is recommended. Use of NISS in a 1:1 ratio will provide a background ionic strength that will be constant relative to variable concentrations of nitrite ion.

Reference electrode conditions must also be considered. Liquid junction potentials arise any time two solutions of different composition are brought into contact. The potential results from the interdiffusion of ions in the two solutions. Since ions diffuse at different rates, the electrode charge will be carried unequally across the solution boundary resulting in a potential difference between the two solutions. In making electrode measurements, it is important that this potential be the same when the reference is in the standardizing solution as well as in the sample solution; otherwise, the change in liquid junction potential will appear as an error in the measured specific ion electrode potential.

The most important variable which analysts have under their control is the composition of the liquid junction filling solution. The filling solution should be equitransferent. That is, the speed with which the positive and negative ions in the filling solution diffuse into the sample should be as nearly equal as possible. If the rate at which positive and negative charge is carried into the sample solution is equal, then no junction potential can result.

WARRANTY

For the most current warranty information, visit www.thermo.com.

The Thermo Electron Corporation, Orion products warranty covers failures due to manufacturer's workmanship or material defects from the date of purchase by the user. User should return the warranty card and retain proof of purchase. Warranty is void if product has been abused, misused, or repairs attempted by unauthorized persons.

Warranties herein are for product sold/installed by Thermo or its authorized dealers.

Any product sold by a U.S. or Canadian distributor must be returned to Thermo for any warranty work. Please contact our Technical Service department for further information. A Return Authorization Number must be obtained from The Technical EDGESM for Orion Products before returning any product for in-warranty repair or replacement.

In the event of failure within the warranty period, Thermo will at the company's option, repair or replace product not conforming to this warranty. There may be additional charges, including freight, for warranty service performed in some countries. For service, call Thermo or its authorized dealer outside the United States and Canada. Thermo reserves the right to ask for proof of purchase, such as the original invoice or packing slip.

Field Service is available on Orion BOD AutoEZTM, EZ Flash[®] GC Accessory and TEA Analyzer[®]. Contact our Field Service department for details on quotations, service and other field service-related activities.

The following products are warranted to be free from defects in material and workmanship in the period listed below from the date of purchase from the user or from the date of shipment from Thermo, whichever is earlier, provided use is in accordance with the operating limitations and maintenance procedures in the instruction manual and when not having been subjected to accident, alteration, misuse, abuse or breakage of electrodes:

Thirty-six months from date of purchase by the user (or forty-two months from date of shipment from Thermo)

- Waterproof Meters (Orion 630, 635, 830A, 835A, 260A, 261S, 265A, 266S, 130A, 131S, 135A and 136S), Conductivity Meters (Orion 105Aplus, 115Aplus, 125Aplus, 145Aplus, 150Aplus and 162A), PerpHect[®] pH/ISE Meters (Orion 310, 320, 330, 350, 370) pH/ISE Meters (Orion 210Aplus, 230Aplus, 250Aplus, 290Aplus, 410Aplus, 420Aplus, 520Aplus, 525Aplus, 710Aplus, 720Aplus and 920Aplus), pHuture MMSTM Meters (Orion 535A and 555A), pH/Conductivity Meter (Orion 550A), Dissolved Oxygen Meters (Orion 805Aplus, 810Aplus, 850Aplus and 862A).

Twenty-four months from date of purchase by the user (or thirty-six months from date of shipment from Thermo)

- Orion ROSS Ultra® Electrodes, Orion AQUAfast® IV Colorimeters, Orion AQUAfast® IV Turbidimeter, Orion 925 Flash Titrator™, Series 100 DuraProbe™ Conductivity Cells and Series 800 Dissolved Oxygen Probes.

Twelve months from date of purchase by the user (or eighteen months from date of shipment from Thermo)

- Laboratory pH Meters, (Orion 301, 611 and 940), SensorLink®, pHuture™ pH Meters (Orion 610 and 620), Sage® Pumps, Cahn® Balances, 930 Ionalyzer®, 950 ROSS™ FAST QC™ Titrator, 960 Titrator PLUS®, Karl Fischer Titrators, Autosamplers, Liquid Handling Devices, Liquid Handling Automation Workstations (Orion AS2000, AS2500 and AS4000), Pumps (Orion SP201, SP201-HR, SP201-S, Peristaltic and Rinse), pHuture® Conversion Box, Wine Master®, 607 Switchbox, rf link™, AQUAfast® II Colorimeters, Vacuum Degasser and Flowmeter.
- Orion EZ Flash® GC Accessory, Orion TEA Analyzer® 610 and 510 excluding consumable items carry twelve months warranty only.
- Orion Ion Selective Electrodes, ionplus® Electrodes, ROSS™ Electrodes, Sure-Flow® Electrodes, PerpHecT® Electrodes, AquaPro Professional Electrodes, No Cal™ pH electrodes, Standard Line pH Electrodes, Tris pH Electrodes, KNiPHE® electrode, ORP Triode™ (Orion 9180BN), pHuture™ pH Probes (Orion 616500) and pHuture MMS™ Quatrode™ and Triode™ (Orion 616600 and 617900), Orion 97-08 DO Probe, Series 100 Conventional Conductivity Cells, temperature probes and compensators (except those products noted).
- Orion 93 and 97 ionplus Series sensing modules are warranted to give six months of operation if placed in service before the date indicated on the package, except 93-07 and 97-07 Nitrate modules are warranted to give ninety days of operation if placed in service before the date indicated on the package.

Six months from date of purchase by the user (or twelve months from date of shipment from Thermo)

- Orion Flash Titration™ Probe (Orion 092518), pHuture™ Electrode (Orion 615700), pHuture MMS™ Pentrode™ (Orion 617500), Quatrode™ (Orion 617800) and Triode™ (Orion 615800), Low Maintenance Triode™ (Orion 9107BN), ORP Low Maintenance Triode™ (Orion 9179BN), and PerpHecT® Low Maintenance Triode™ (Orion 9207BN), Waterproof Triode™ (Orion 9107WP, 9107WL, 9109WL and 9109WP), QuiKcheK® Meters and Micro Electrodes.

Three months from date of purchase by the user (or six months from date of shipment from Thermo)

- Economy Line Electrodes, Orion 91-05, 91-06, 91-15, 91-16, 91-25, 91-26, 91-35, 91-36, 92-06. Warranty also includes failure for any reason (excluding breakage), except abuse, provided the electrode is not used in solutions containing silver, sulfide, perchlorate, or hydrofluoric acid; or in solutions more than one (1) Molar in strong acid or base at temperatures above 50 °C.

“Out-of-Box” Warranty - Should any of the following products fail to work when first used, contact Thermo immediately for replacement.

- Orion Solutions, Standards, Reagents, Cables, Ferrules, Tubing, Line adapters, Printers, Software, Cases, Stands, Probe Membranes, AQUAfast® Test Strips, EZ Flash® columns, Liquid Handling Probes, Adapter Plates and Racks and general accessories.

For products in the catalog not listed in this warranty statement, please visit our website at: www.thermo.com.

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ORDERING INFORMATION

Orion	Description
9346BN	Nitrite Plastic Membrane Half-Cell Electrode, BNC Connector
934600	Nitrite Plastic Membrane Half-Cell Electrode, U.S. Standard Connector
9746BN	ionplus® Nitrite Combination Plastic Membrane Electrode, BNC Connector
090032	BNC Electrode to U.S. Standard Meter Adapter
900100	Single Junction Sure-Flow® Reference Electrode
900046	Optimum Results™ F Reference Filling Solution, 5 x 60 mL Bottles
954606	Nitrite Standard Solution (0.1 M), 475 mL
934610	Nitrite Interference Suppressor Solution, NISS™, 475 mL
974601	Replacement Sensing Module for Orion 97-46 ionplus Nitrite Electrode, 1 each
934601	Replacement Electrode Module for Orion 93-46 Nitrite Electrode, package of 3
9700BN	Replacement ionplus Electrode Handle, with BNC Connector
9300BN	Replacement Electrode Body, with BNC Connector
930000	Replacement Electrode Body, with U.S. Standard Connector
900060	ionplus Stirring Accessory

SPECIFICATIONS

Concentration Range

3.6×10^{-6} M to 1.4×10^{-2} M NO_2^-
0.05 to 200 ppm NO_2^- as N

pH Range

4 to 5 pH

Temperature Range

0 to 40°C

Electrode Resistance

1 to 5 megohms

Reproducibility

± 2%

Sample

Aqueous solutions only

Module Life

Six months under normal laboratory conditions

Size	93-46	97-46
Electrode Length (Body with Module)	135 mm	110 mm
Cap Length	30 mm	30 mm Cap
Diameter	16 mm	16 mm
Electrode Diameter	12 mm	13 mm Cable
Length	1 meter	1 meter
Maximum Immersion Depth	22 mm	Up to fill hole

Environmental Instruments

Water Analysis

North America

166 Cummings Center
Beverly, MA 01915 USA
Tel: 978-232-6000
Dom. Fax: 978-232-6015
Int'l. Fax: 978-232-6031

Europe

12-16 Sedgeway Business Park
Witchford, Cambridgeshire
England, CB6 2HY
Tel: 44-1353-666111
Fax: 44-1353-666001

Far East

Room 904, Federal Building
369 Lockhart Road
Wanchai, Hong Kong
Tel: 852-2836-0981
Fax: 852-2834-5160

Customer Support

Toll Free: 800-225-1480
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For updated contact information, visit www.thermo.com

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