

NO_3^-

Orion 9307,
Orion 9707 ionplus®

Orion Nitrate 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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This publication supersedes all previous publications on this subject.

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GENERAL INFORMATION

Introduction

The Orion 9307 and Orion 9707 ionplus® Nitrate Electrodes measure nitrate in aqueous solutions simply, accurately, and economically.

The Orion 9707 ionplus® Nitrate 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 Orion's Technical Service Chemists for assistance and troubleshooting advice. Please refer to **Troubleshooting** for information on contacting Orion.

Required Equipment

Meter

ISE meters, such as Orion EA 940, 920A, 720A, 710A, or 290A, 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, 520A, or 525A.

Reference Electrode

Orion No.

For use with Orion 9307:

Orion 90-02 Double Junction
Reference Electrode

900200

For use with Orion 9707:

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 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 Solution

Orion No.

For Orion 9707 & Outer chamber of Orion 90-02:

Optimum Results™ F Filling Solution 900046

NOTE: Do not use the outer filling solution shipped with the reference electrode.

For Orion 90-02:

Inner Chamber Filling Solution 900002

Nitrate Stock Calibration Standards

To prepare daily calibration solutions.

Standards

Orion No.

0.1 M Nitrate Concentration Standard 920706

1000 ppm as N Standard 920707

100 ppm as N Standard 930707

Ionic Strength Adjustor (ISA)

Orion 930711

To adjust ionic strength of samples and standards.

Nitrate Interference Suppressor Solution

Orion 930710

For removal of a variety of interfering anions, including chloride ion, present in samples such as drinking water, wastewaters, and soils. See **Interferences**.

Preservative Solution

Customer Prepared

Prepare a 1 M boric acid preservative solution by dissolving 6.2 g reagent-grade boric acid in 100 mL boiling water. Let cool. Add 1 mL preservative solution to 100 mL of all standards and samples to prevent biological degradation of the solutions.

BEFORE USING THE ELECTRODE

Electrode Assembly and Preparation

Orion 9307 Nitrate 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. Rinse the nitrate electrode with distilled water, then soak in Nitrate Standard, 100 ppm (or 10^{-2} M) for 1 to 2 hours prior to initial use. **Do not immerse the electrode past the rubber electrode washer.** See **Figure 1**.

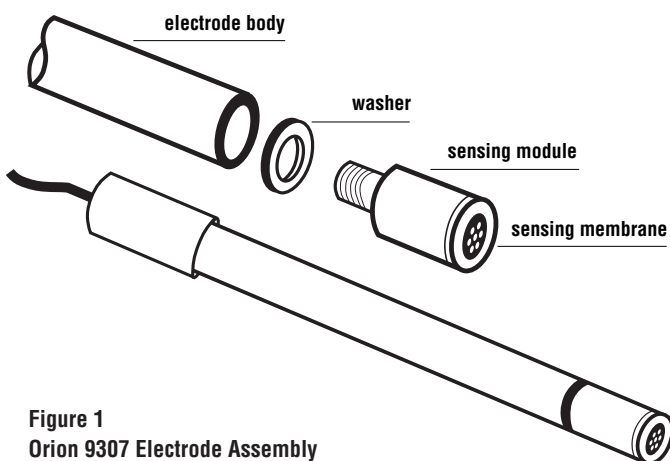


Figure 1
Orion 9307 Electrode Assembly

Orion 90-02 Double Junction Reference Electrode:

Required for use with Orion 9307 Nitrate Half Cell Electrode. No assembly is required. Fill the reference electrode according to instructions in the reference electrode instruction manual, using Orion 900002 Filling Solution in the inner chamber. Use Optimum Results F, Orion 900046, instead of the Outer Chamber Filling Solution provided with the electrode. **Do not use the outer chamber filling solution shipped with the 90-02 reference electrode because it will interfere with your nitrate measurements.**

Orion 9707 ionplus® Nitrate 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!

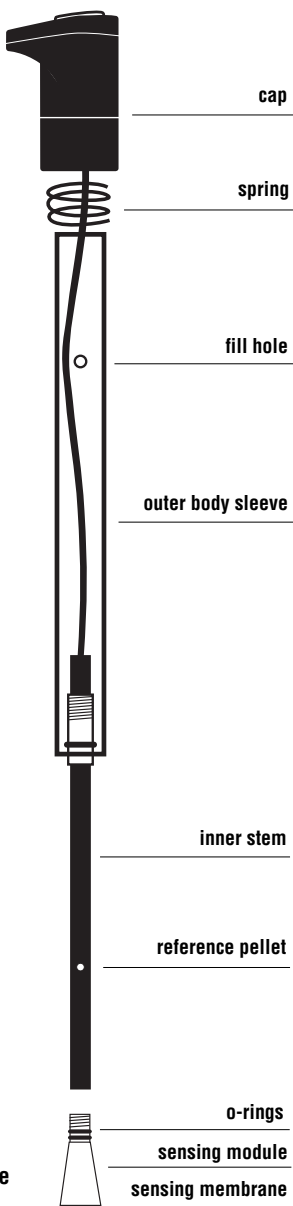


Figure 2
Orion 9707 ionplus® Nitrate
Combination Electrode

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 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 tighten**. The module should be firmly attached to the stem.

5

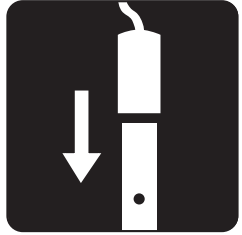
Holding the electrode cable, slide the outer body, spring and cap over the inner stem.

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.

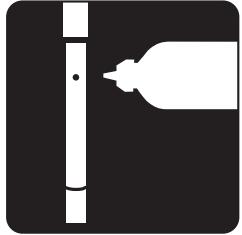
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Hold the electrode with one hand. Press on the top of the cap with your thumb to make sure the electrode has a smooth flushing motion and reseats back onto the module.



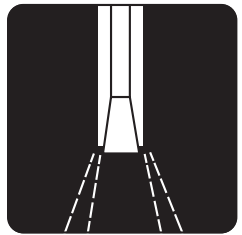
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Fill the outer body with Optimum Results F filling solution, Orion 900046, to approximately 1/4 full.



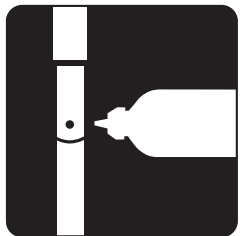
9

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

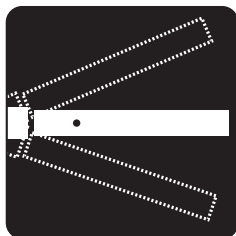


10

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 nitrate electrode with distilled water, then soak in Nitrate Standard, 100 ppm (or 10^{-2} M) for 1 to 2 hours prior to initial use.

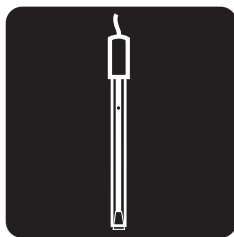
Checking Electrode Operation (Slope)

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.

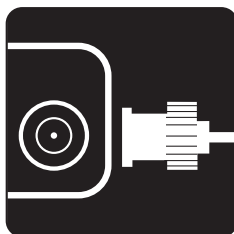
1

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



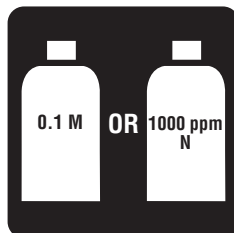
2

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



3

Select either 0.1 M or 1000 ppm nitrate standard.



4

Place 100 mL distilled water into a 150 mL beaker. Add 2 mL ISA, Orion 930711. Stir thoroughly.



5

Set the meter to the mV mode.



6

Rinse electrode(s) with distilled water, blot dry, and place in the solution prepared in step 4 above.



7

Pipet 1.0 mL of the standard into the beaker. Stir thoroughly.



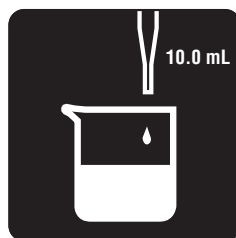
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When a stable reading is displayed, record the electrode potential in millivolts.



9

Pipet 10.0 mL of the same standard into the same beaker. Stir thoroughly.



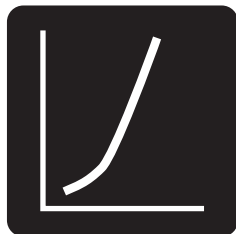
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When a stable reading is displayed (within 4 to 5 minutes), record the electrode potential in millivolts.



11

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 -54 to -60 mV/decade when the solution temperature is $25 \pm 5^\circ\text{C}$. If the slope is not within this range, re-soak 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 nitrate in units of moles per liter, parts per million as nitrate, 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_3^-	ppm as N
10^{-4}	6.20	1.40
10^{-3}	62.0	14.0
10^{-2}	620	140

Sample Requirements

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

Sample temperature must be less than 40°C, with samples and standards all at the same temperature. At the 14 ppm as N or 10^{-3} M NO_3^- level, a 1°C difference in temperature produces about a 1.5% error. For highly accurate results, use a water bath to control temperature variances.

Interferences should be absent. See section entitled **Interferences** for a list of possible interferences. If interferences are present in the sample and cannot be removed, use Nitrate Interference Suppressor Solution, Orion 930710, instead of ISA solution.

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, then blot dry. **Do not wipe or rub the sensing membrane, as you may contaminate and damage the surface.**
- Allow all standards and samples to come to room temperature for precise measurements.
- After immersion in solution, check the nitrate electrode for any air bubbles on the membrane surface. Remove air bubbles at the electrode surface by gently tapping the electrode.
- The Orion 9307 Nitrate Half-Cell Electrode should be submerged approximately half the length of the nitrate module. **DO NOT submerge the nitrate electrode above the rubber electrode washer.** Submerge the reference electrode to the same depth as the nitrate electrode.
- Nitrate solutions are a culture medium for bacteria and algae. Dilute samples and standards cannot be kept unless they are treated to inhibit biological growth. Preservation Solution should thus be added to samples when they are collected (1 mL for every 100 mL of sample).
- If interferences are present, use Nitrate Interference Suppressor Solution, Orion 930710. Samples such as soils and plant tissues may have a variety of interfering ions present. See **Interferences**.

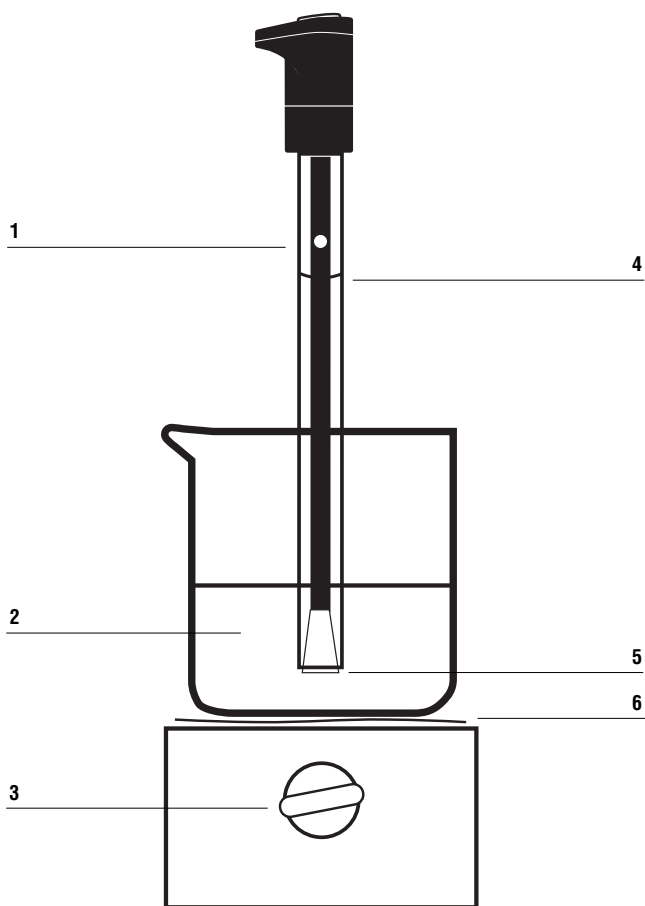


Figure 3

1. Uncover fill hole during measurement (Orions 90-02 and 9707).
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 nitrate module past rubber electrode washer (Orion 9307 only).

Roadway to ionplus® Success!

Guide to measuring techniques

	Direct Measurement	Small Volume Direct Measurement	Low-Level Measurement	Known Addition
Recommended Concentration Range	0.1 to 14000 ppm as N	0.1 to 14000 ppm as N	<1.4 ppm as N	1.4 to 14000 ppm as N
Large # of samples	X	X (Orion 9707 only)	X	X
Small sample volume		X (Orion 9707 only)		X
Reduced chemical usage		X (Orion 9707 only)		
Field Measurements	X	X (Orion 9707 only)		X
Ionic Strength >0.1 M				X
Occasional sampling				X
see page #	18	24	29	34

A variety of analytical techniques are available to the analyst. The best technique is dependent upon sample matrix. The following section describes the recommended techniques for nitrate 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 with a series of standards. The concentration of the samples is determined by comparison to the standards. Addition of ISA to all solutions ensures that samples and standards have similar ionic strength, proper pH, and reduces the effect of interfering ions. Alternatively, Nitrate Interference Suppressor Solution is specially formulated to eliminate interfering ion effects in typical sample matrices. When measuring small sample volumes or to reduce chemical usage, follow the **Small Volume Direct Measurement** method, using the Orion 9707 ionplus[®] Nitrate Electrode.

Low-Level Measurement is similar to Direct Measurement. Use this method when the expected sample concentration is less than 1.4 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 nitrate standard 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 1.4 ppm as N or 10^{-4} M NO_3^- . A two point calibration is sufficient, though more points can be used if desired. Using ISE meters, such as the Orion 920A, 720A, 710A, 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

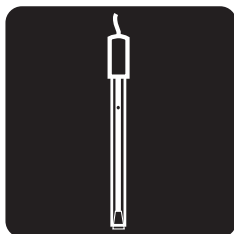
- Bracket standard concentrations around the expected sample concentration.
- Always dilute samples and standards in a 50:1 ratio with ISA. For example, 100 mL of sample and 2 mL of ISA.
- Alternatively, Nitrate Interference Suppressor Solution can be used in a 1:1 dilution ratio. Do not use ISA when using Nitrate Interference Suppressor Solution.
- Verify this procedure by measuring a standard of known concentration as an unknown or by spiking a sample with nitrate 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 meters or a mV meter

See individual meter instruction manuals for more specific calibration information.

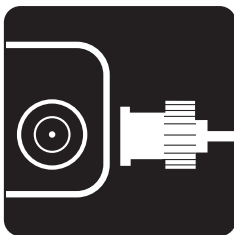
1

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



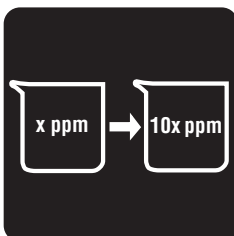
2

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 100 mL of each standard and sample into separate 150 mL beakers. Add 2 mL ISA to each standard and sample.

NOTE: Other solution volumes may be used, as long as the ratio of solution to ISA remains 50:1.

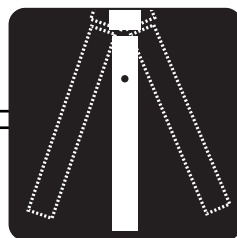
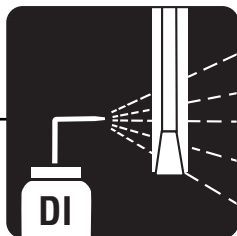
If sample matrix contains interfering ions, dilute sample and standards in a larger beaker, in a 1:1 ratio, with Nitrate Interference Suppressor Solution instead of ISA. Stir thoroughly.



5

For an 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

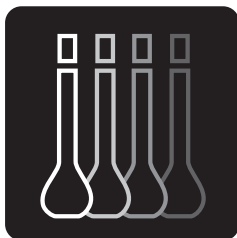
For an ISE meter: Rinse electrode(s) with distilled water, shake dry, and place into the beaker with the next standard. Wait for a stable reading, then adjust the meter to display the value of the second standard, as described in the meter instruction manual.

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

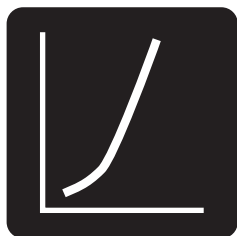
Repeat step 6 for all standards, working from the least concentrated to most concentrated standard.



8

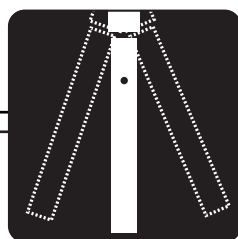
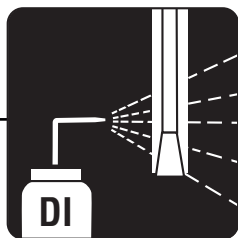
For an ISE meter: Calibration information will be calculated and stored automatically.

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 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.



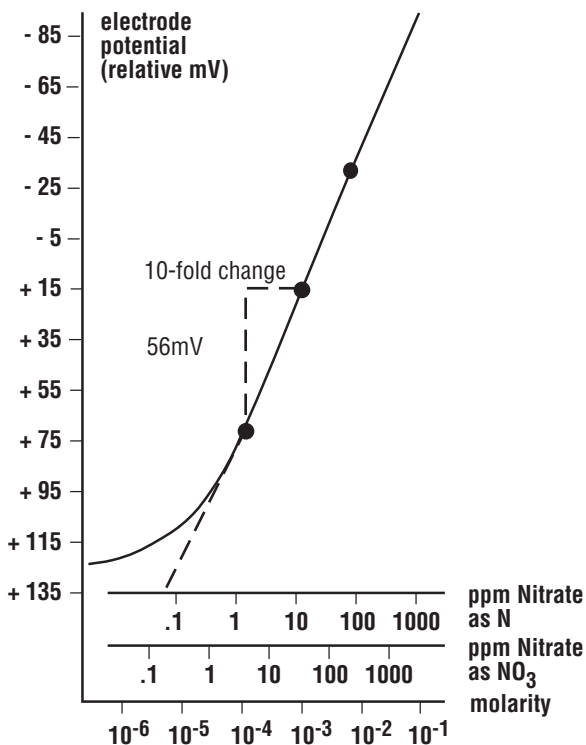


Figure 4
Typical Nitrate Electrode Calibration Curve

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 accurate results. 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 9707 ionplus® Nitrate Electrode only)

Using the Sure-Flow reference design, the Orion 9707 ionplus® Nitrate 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, Nitrate Interference Suppressor, and ISA 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 1.4 ppm as N or 10^{-4} M NO_3^- . 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 25 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 9707 ionplus® electrode. Do not allow the sensing membrane to touch the sample container.

For Improved Accuracy

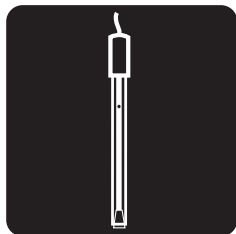
- Use Orion 9707 ionplus® Nitrate Electrode.
- Bracket standard concentrations around the expected sample concentration.
- Always dilute samples and standards in a 50:1 ratio with ISA, or in a 1:1 ratio with Nitrate Interference Suppressor.
- Verify this procedure by measuring a standard of known concentration as an unknown or by spiking a sample with nitrate 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 9707 ionplus® Nitrate Electrode

See individual meter instruction manuals for more specific calibration information.

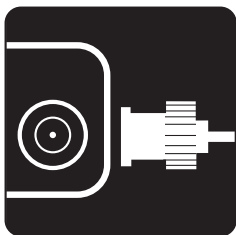
1

Prepare the Orion 9707 ionplus® Nitrate electrode as described in **Electrode Preparation**.



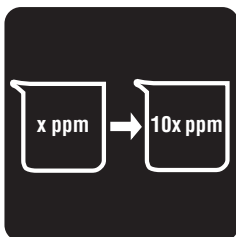
2

Connect the electrode 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 25 mL of each standard and sample into separate 150 mL beakers. Add 0.5 mL ISA to each standard and sample.

NOTE: Other solution volumes may be used, as long as the ratio of solution to ISA remains 50:1.

If sample matrix contains interfering ions, dilute sample and standards in a larger beaker, in a 1:1 ratio, with Nitrate Interference Suppressor Solution instead of ISA. Stir thoroughly.



5

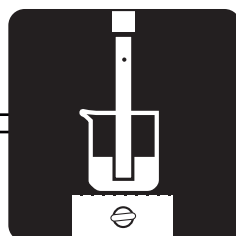
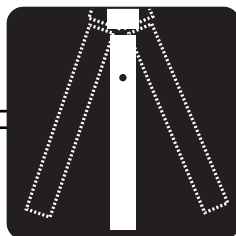
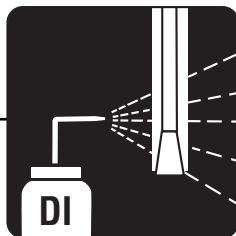
For an ISE meter: Rinse Orion 9707 ionplus® Nitrate Electrode with distilled water, shake dry, and place into the beaker containing the most dilute standard. Wait for a stable reading, calibrate the meter to display the value of the standard as described in the meter instruction manual.

For a mV meter: Rinse Orion 9707 ionplus® Nitrate 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

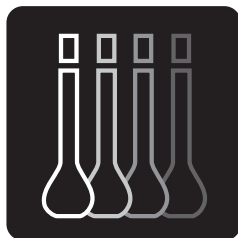
For an ISE meter: Rinse ionplus® Nitrate Electrode with distilled water, shake dry, and place into the beaker with the next standard. Wait for a stable reading, then adjust the meter to display the value of the second standard, as described in the meter instruction manual.

For a mV meter: Rinse ionplus® Nitrate Electrode with distilled water, shake dry, and place into the beaker containing the next concentrated standard. When a stable reading is displayed, record the mV value and corresponding standard concentration.



7

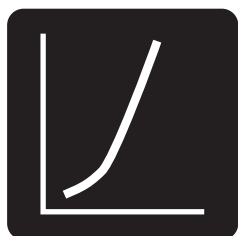
Repeat step 6 for all standards, working from the least concentrated to most concentrated standard.



8

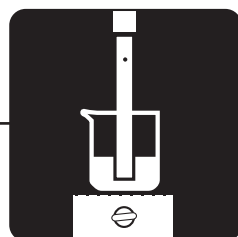
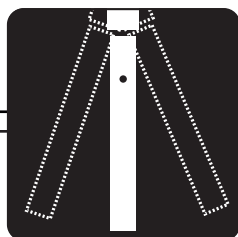
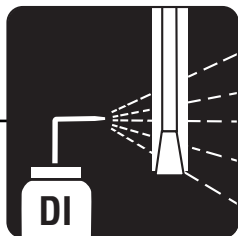
For an ISE meter: Calibration information will be calculated and stored automatically.

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 ionplus® Nitrate 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.



Low-Level Measurements By Direct Measurement

Use this method when measuring solutions with a nitrate concentration of less than 1.4 ppm as N or 10^{-4} M NO_3^- , those within the non-linear range of the nitrate electrode. Low-level measurements require at least three standards to compensate for the electrode's non-linearity.

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 response time at low-levels is faster when it is not 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.

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 instruction 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.

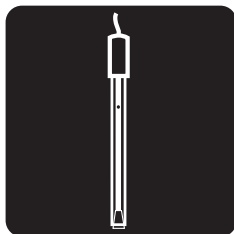
When using the Orion 920A, 720A, 710A or 290A, with the autoblack feature, three calibration points are sufficient. If a calibration curve is prepared manually, additional points may be helpful to facilitate drawing the curve.

- Remember to stir all standards and samples at a uniform rate.
- Review section entitled **Important ISE Measurement Techniques**.

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

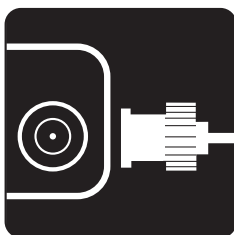
1

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



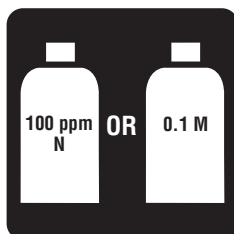
2

Connect the electrode(s) to the meter. Set the meter to read concentration for an ISE meter or mV for a mV meter.



3

Select a standard solution. Use either 100 ppm N, Orion 930707, or dilute the 0.1 M Nitrate Standard, Orion 920706, to 10^{-3} M.



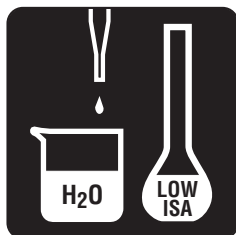
4

Prepare a low-level ISA solution by diluting 20 mL of the Nitrate ISA, Orion 930711, to 100 mL with distilled water. **Use this low-level ISA for low-level measurements only.** Certain sample matrices may require Nitrate Interference Suppressor Solution, Orion 930710, instead of low-level ISA. A solution to sample ratio of 1:9 is recommended for most matrices.



5

Measure 100 mL distilled water into 150 mL beaker. Add 1 mL low-level ISA.



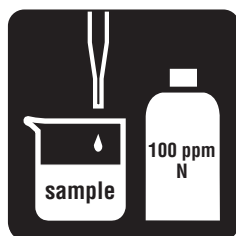
6

Rinse the electrode(s) with distilled water, place into beaker. Stir thoroughly.



7

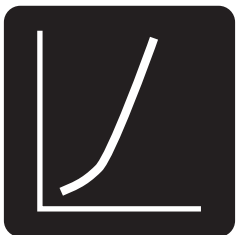
Add increments of the standard to the beaker using steps outlined in **Table 2**.



8

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.



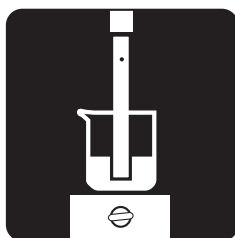
9

Measure 100 mL of sample into a beaker. Add 1 mL of low-level ISA.



10

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



11

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 8.



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	0.1	10^{-6}
2	1 mL	0.1 mL	0.2	2.0×10^{-6}
3	1 mL	0.2 mL	0.4	3.9×10^{-6}
4	1 mL	0.2 mL	0.6	5.9×10^{-6}
5	1 mL	0.4 mL	1.0	9.8×10^{-6}
6	2 mL	2.0 mL	2.9	2.9×10^{-5}
7	2 mL	2.0 mL	4.7	4.7×10^{-5}

Additions of 100 ppm as N or 10^{-3} M NO_3^- standards to 100 mL distilled water, plus 1 mL low-level ISA.

Known Addition

Known addition is a convenient technique for measuring samples in the linear response range, greater than 1.4 ppm as N or 10^{-4} M NO_3^- , 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. These direct-reading meters provide a great convenience. Accurate measurement requires that the following conditions be met.

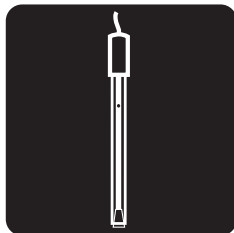
For Improved Accuracy

- 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 ISA in a 50:1 ratio or a 1:1 ratio with Nitrate Interference Suppressor Solution.
- Dilute samples in a 50:1 ratio with ISA or a 1:1 ratio with Nitrate Interference Suppressor Solution 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 Preparation**.



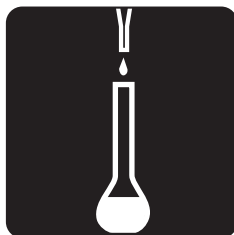
2

Connect electrode(s) to the meter.



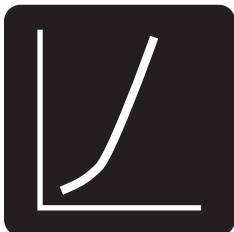
3

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



4

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



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.



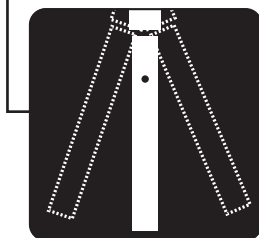
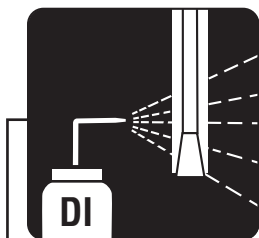
2

Measure 50 mL of the sample into a beaker. Add 1 mL ISA or 50 mL Nitrate Interference Suppressor Solution. Stir thoroughly.



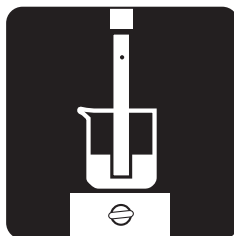
3

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 50 mL of the sample into a 150 mL beaker. Add 1 mL ISA or 50 mL Nitrate Interference Suppressor Solution. 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**, or by calculation, 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:

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

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:

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

where:

$$\Delta E = E_2 - E_1$$

$$S = \text{slope of the electrode}$$

$$p = (\text{volume of standard}) / (\text{volume of sample \& ISA})$$

$$r = (\text{volume of sample \& ISA}) / (\text{volume of sample})$$

Table 3

Volume of Addition	Concentration of Standard Before Adding ISA
1 mL	100 x sample concentration
5 mL	20 x sample concentration
10 mL*	10 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 nitrate samples using Lotus,
Excel or Quattro Spreadsheet

A	B	C
1		Enter Values
2	VOL. OF SAMPLE & ISA, ML.	51
3	VOL. OF ADDITION, ML	10
4	CONCENTRN. OF ADDITION	10
5	VOL. OF SAMPLE	50
6	INITIAL MV READING	-45.3
7	FINAL MV READING	-63.7
8	ELECTRODE SLOPE	-59.2
9		
10		DERIVED VALUES
11	DELTA E	+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			
		Slope		
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
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

ΔE	Q, Concentration Ratio			
	Monovalent	Slope		
	-57.2	-58.2	-59.2	-60.1
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
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

ΔE	Q, Concentration Ratio				
	Monovalent	-57.2	-58.2	Slope	-59.2
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	
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 9307 Nitrate Half-Cell Electrode

The nitrate sensing module should be kept in the glass vial until used. The assembled electrode can be stored in nitrate standard. For long periods of time (over 2-3 days), disassemble the nitrate electrode, rinse thoroughly with distilled water, blot dry, and store the module in its vial.

Orion 9707 ionplus® Nitrate Electrode

The solution in the Orion 9707 ionplus® Combination Nitrate 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 nitrate standard, such as 100 ppm as N.

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 nitrate 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 nitrate 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.

Orion 90-02 Double Junction Reference Electrode

The Orion 90-02 Reference Electrode may be stored in air between sample measurements (up to 1 hour).

For short periods of time (up to one week):

The Orion 90-02 may be stored in its filling solution or distilled water. Do not allow the solution inside the electrode to evaporate and crystallize.

For long periods of time (over one week):

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 (Orions 9707 & 90-02) No reference electrode (Orion 9307) Reference electrode chamber not filled (Orions 9707 & 90-02) 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 ISA or Nitrate Interference Suppressor Solution not used Orion 9707 Electrode Cap on too tight
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 contaminants Orion 9707 Electrode Cap on too tight

Solution

Check meter with shorting strap (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 90-02 Reference Electrode

(with Orion 9307 Nitrate 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 on electrode by gently tapping it

Put electrodes in solution

Check meter with shorting strap (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 90-02 Double Junction Reference Electrode

(with Orion 9307 Nitrate Electrode) Do not use calomel or Ag/AgCl (frit- or fiber-type) reference electrode

Use recommended ISA, Orion 930711 or Nitrate Interference

Suppressor Solution, Orion 930710

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

Allow solutions to come to room temperature before measurement

See **Electrode Assembly and Preparation**

Use nitrate interference suppressor solution, 930710. See **Interferences**

Use recommended filling solution.

See **Electrode Assembly and Preparation**

Rinse electrode with distilled water and soak in nitrate standard (10^{-2} M) for 1 hour

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

Troubleshooting Checklist (cont.)

Symptom	Possible Causes
Low slope or No slope	Electrodes not properly conditioned Standards contaminated or incorrectly made ISA or Nitrate Interference Suppressor Solution not used Standard used as ISA or Nitrate Interference Suppressor Solution Orion 9707 Electrode Cap on too tight Defective sensing module Electrode exposed to interferences
“Wrong Answer” (But calibration curve is OK)	Incorrect scaling of semilog paper Incorrect standards Incorrect sign Wrong units used

Solution

See **Electrode Assembly and Preparation**

Prepare fresh standards

Use recommended ISA, Orion 930711 or Nitrate Interference Suppressor Solution, Orion 930710

Use ISA or Nitrate Interference Suppressor Solution!

Reassemble electrode. See **Electrode Assembly and Preparation**
Refer to **Troubleshooting Guide**

See **Interferences**. Use Nitrate Interference Suppressor Solution, Orion 930710

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:

$10^{-3} \text{ M} = 62 \text{ ppm as NO}_3^- = 14 \text{ ppm as N}$

For additional information on blank correction with your meter, see meter instruction 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) Electrode(s) 3) Standard 4) Sample and 5) Technique. See also **Important ISE Measurement Techniques** and **For Improved Accuracy** sections.

Meter

The meter is the easiest component to eliminate as a possible cause of error. 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.

Electrode(s)

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

Clean Orion 90-02 reference electrode as described in reference electrode instruction manual (used with Orion 9307 only).

4. Repeat step 2.
- 5a. For the 9307 Nitrate Half-Cell Electrode:

If the electrodes still do not perform as described, determine whether the nitrate 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 9707 ionplus® Nitrate 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 below.
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 the Orion 9707 Electrode cap is not too tight
 - Use proper filling solution, ISA, 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 – 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 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 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 the ionic strength varies markedly from sample to sample, 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 with a slope of about -54 to -60 mV per decade until concentration reaches 10^{-4} M NO_3^- . See **Figure 4**.

The electrode exhibits good time response (98% in one minute or less) for nitrate concentrations above 10^{-3} M NO_3^- . Below this value response times vary from 2 to 5 minutes. See **Figure 5**.

Limits of Detection

In pure sodium nitrate solutions, the upper limit of detection is 1 M. When possible, dilute the sample to the linear working range of the electrode. If this is not possible, the possibility of a liquid junction potential developing at the reference electrode and the “salt extraction effect” need to be considered. At high salt concentrations, some salts may be extracted into the electrode membrane, causing deviation from theoretical response. To measure samples between 10^{-1} and 1 M, calibrate the electrode at 4 or 5 intermediate points, or 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 nitrate compared to the actual response. If nitrate measurements are made below 10^{-4} M (1.4 ppm as N), 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 $\pm 1^\circ\text{C}$ ($\pm 2^\circ\text{F}$) of each other. At the 10^{-3} M level, a 1°C difference in temperature results in a 1.5% error. 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 nitrate electrode also varies with temperature, as indicated by the factor “S” in the Nernst equation. Values for the change in slope for nitrate 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 9707 ionplus® electrode is approximately 3.2×10^{-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		

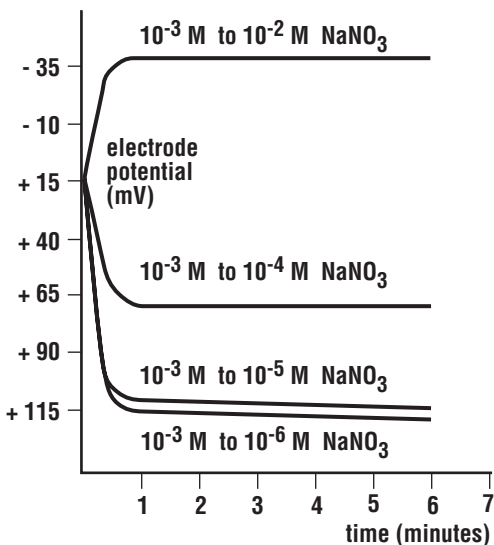


Figure 5
Typical Electrode Response to NaNO₃

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% error at various concentrations of nitrate.

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. Carbonate and bicarbonate can be removed by acidifying the sample to pH 4.5 with sulfuric acid, converting the ions to carbon dioxide.
- b. These interferences can be minimized by precipitation with silver. Dissolve solid silver sulfate in samples to effect removal.
- c. Nitrite can be removed by adding sufficient sulfamic acid to samples.
- d. These interferences cannot be removed. Use Orion Nitrate Test Kit, Orion 700005, to convert nitrate to ammonia. Measure using ammonia electrode, Orion 9512. As an alternate method, convert nitrate to nitrite with a reduction column and measure nitrite levels with a nitrite electrode (Orion 9746 or 9346). For more information contact Orion Technical Service.
- e. Many organic (carboxylic) anions also interfere with the nitrate electrode. These anions can be removed by using a 1 M ISA solution.

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

Thermo offers a Nitrate Interference Suppressor Solution (Orion 930710), recommended for the removal of a variety of interfering anions present in samples such as soils or plant tissues.

The Nitrate 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 Nitrate 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 drifty and sluggish in response. When this happens, restore normal performance as outlined in **Troubleshooting**.

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

Electrode Life

Each sensing module should last at least three 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 Moles/Liter	10⁻⁴ M	10⁻³ M	10⁻² M
(d) ClO ₄ ⁻	1 x 10 ⁻⁸	1 x 10 ⁻⁷	1 x 10 ⁻⁶
(b) I ⁻	5 x 10 ⁻⁷	5 x 10 ⁻⁶	5 x 10 ⁻⁵
(d) ClO ₃ ⁻	5 x 10 ⁻⁶	5 x 10 ⁻⁵	5 x 10 ⁻⁴
(b) CN ⁻	1 x 10 ⁻⁵	1 x 10 ⁻⁴	1 x 10 ⁻³
(b) Br ⁻	7 x 10 ⁻⁵	7 x 10 ⁻⁴	7 x 10 ⁻³
(c) NO ₂ ⁻	7 x 10 ⁻⁵	7 x 10 ⁻⁴	7 x 10 ⁻³
(b) HS ⁻	1 x 10 ⁻⁴	1 x 10 ⁻³	1 x 10 ⁻²
(a) HCO ₃ ⁻	1 x 10 ⁻³	1 x 10 ⁻²	0.1
(a) CO ₃ ²⁻	2 x 10 ⁻³	2 x 10 ⁻²	0.2
(b) Cl ⁻	3 x 10 ⁻³	3 x 10 ⁻²	0.3
(b) H ₂ PO ₄ ⁻	5 x 10 ⁻³	5 x 10 ⁻²	0.5
(b) HPO ₄ ²⁻	5 x 10 ⁻³	5 x 10 ⁻²	0.5
(b) PO ₄ ³⁻	5 x 10 ⁻³	5 x 10 ⁻²	0.5
(e) OAc ⁻	2 x 10 ⁻²	0.2	2
F ⁻	6 x 10 ⁻²	0.6	6
SO ₄ ²⁻	0.1	1.0	10

Letters in parentheses refer to comments in the **Interferences** section.

Interferences to NO₃⁻ as N

ppm	1 ppm	10 ppm	100 ppm
(d) ClO ₄ ⁻	7 x 10 ⁻⁴	7 x 10 ⁻³	7 x 10 ⁻²
(b) I ⁻	4 x 10 ⁻²	0.4	4
(d) ClO ₃ ⁻	0.3	3	30
(b) CN ⁻	0.2	2	20
(b) Br ⁻	4	40	400
(c) NO ₂ ⁻	2	23	230
(b) HS ⁻	2	23	230
(a) HCO ₃ ⁻	44	440	4400
(a) CO ₃ ²⁻	86	860	8600
(b) Cl ⁻	76	760	7600
(b) H ₂ PO ₄ ⁻	346	3464	34640
(b) HPO ₄ ²⁻	343	3430	34300
(b) PO ₄ ³⁻	339	3390	33900
(e) OAc ⁻	1042	10420	104200
F ⁻	814	8140	81400
SO ₄ ²⁻	6857	68570	685700

Letters in parentheses refer to comments in the **Interferences** section.

Theory of Operation

The nitrate 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 nitrate selective ion exchanger. See **Figure 6**.

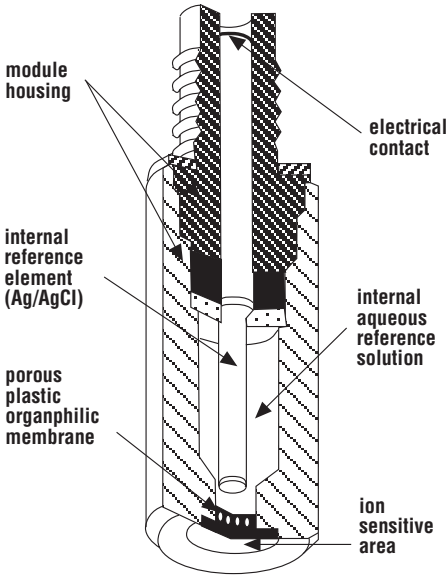


Figure 6
Example of Ion Sensing Module

When the membrane is in contact with a nitrate solution, an electrode potential develops across the membrane. This potential, which depends on the level of free nitrate 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 nitrate 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 = nitrate ion level in solution

S = electrode slope (about -56 mV per decade)

The level of nitrate ion, A , is the activity or “effective concentration” of free nitrate ion in solution. The total nitrate 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 nitrate ions in all bound or complexed forms

C_f = concentration of free nitrate ions

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

$$A = \gamma C_f$$

Ionic activity coefficients are variable and largely depend on total ionic strength. Ionic 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.

Ionic strength adjustor (ISA) is added to nitrate standards and samples so that the background ionic strength is high and constant relative to variable concentrations of nitrate ion. For the nitrate electrode, $(\text{NH}_4)_2\text{SO}_4$ is the recommended ISA. Nitrate Interference Suppression Solution, a specific solution for removal of nitrate-interfering ions, is recommended for samples with competing ions. Other solutions can be used as long as they do not contain ions that would interfere with the electrode’s response to nitrate 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. Using the Optimum Results F filling solution will minimize junction potential in most samples.

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, 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, 136S, 1230, 142 and 842), Conductivity Meters (Orion 105AplusTM, 115AplusTM, 125AplusTM, 145AplusTM, 150AplusTM and 162A), PerpHect[®] pH/ISE Meters (Orion 310, 320, 330, 350, 370) pH/ISE Meters (Orion 210AplusTM, 230AplusTM, 250AplusTM, 290AplusTM, 410AplusTM, 420AplusTM, 520AplusTM, 525AplusTM, 710AplusTM, 720AplusTM and 920AplusTM), pHuture MMSTM Meters (Orion 535A and 555A), pH/Conductivity Meter (Orion 550A), Dissolved Oxygen Meters (Orion 805AplusTM, 810AplusTM, 850AplusTM 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, AQUAfast® IV Colorimeters, 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), Smart Chek™ meters, 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 No.	Description
9307BN	Orion 9307 Nitrate Plastic Membrane Half-cell Electrode, BNC connector
930700	Orion 9307 Nitrate Plastic Membrane Half-cell Electrode, U.S. Standard connector
9707BN	Orion 9707 ionplus® Nitrate Combination Plastic Membrane Electrode, BNC connector
090032	BNC Electrode to U.S. Standard Meter Adapter
900200	Orion 90-02 Double Junction Sure-Flow® Reference Electrode
900046	Optimum Results™ F Reference Filling Solution, 5 x 60 mL bottles
900002	Inner Chamber Filling Solution for Orion 90-02 Reference Electrode, 5 x 60 mL bottles
920706	Nitrate Standard Solution (0.1 M), 475 mL
920707	Nitrate Standard Solution (1000 ppm as N), 475 mL
930707	Nitrate Standard Solution (100 ppm as N), 475 mL
930711	Nitrate Ionic Strength Adjustor Solution, 475 mL
930710	Nitrate Interference Suppressor Solution, 475 mL
970701	Replacement Sensing Module for Orion 9707 ionplus® Nitrate electrode, each
930701	Replacement Electrode Module for Orion 9307 Nitrate electrode, package of 3
930702	Replacement Electrode module (one) for Orion 9307 Nitrate electrode.
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

7×10^{-6} M to 1 M NO_3^-
0.1 to 14,000 ppm NO_3^- as N

pH Range

2.5 to 11 pH
Low-level measurements may be influenced by hydrogen or hydroxide ion interferences

Temperature Range

0 to 40°C

Electrode Resistance

1 to 5 megohms (9307)
0.1 to 5 megohms (9707)

Reproducibility

$\pm 2\%$

Sample

Aqueous solutions only

Module Life

Three months under normal laboratory conditions

Size	9307	9707
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

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