# Measuring Nitrate in Water and Wastewater using the Thermo Scientific Orion Dual Star pH/ISE Meter

Water and Lab Products, Thermo Fisher Scientific

#### **Key Words**

Thermo Scientific Orion Dual Star pH/ISE meter, nitrate ion selective electrode (ISE), nitrate, wastewater, water

## Goal

This technical note explains how to accurately measure the level of nitrate in general water and wastewater samples using the Thermo Scientific<sup>™</sup> Orion<sup>™</sup> Dual Star<sup>™</sup> pH/ISE dual channel benchtop meter.



#### Introduction

Nitrate is a colorless, odorless and tasteless compound found in some natural water bodies. Accurate testing of the level of nitrate in water and wastewater is important to determine the proper course of treatment. The following technical note describes how the Orion Dual Star pH/ISE meter paired with an Orion nitrate combination electrode can help you measure the nitrate levels in water and wastewater.

## **Required Equipment**

- Orion Dual Star pH/ISE meter with electrode stand (Cat. No. 2115000)
- Nitrate combination electrode (Cat. No. 9707BNWP)
- Automatic Temperature Compensation (ATC) probe (Cat. No. 927007MD)
- Stirrer probe (Cat. No. 096019) or magnetic stir plate and bar

- 100 ppm nitrate as nitrogen standard (Cat. No. 930707)
- Nitrate interference suppressor solution (NISS) (Cat. No. 930710)
- Thermo Scientific<sup>™</sup> Orion<sup>™</sup> Optimum Results<sup>™</sup> F electrode filling solution (Cat. No. 900046)
- Deionized water
- 100 mL volumetric flasks
- 50 mL beakers
- 50 mL and 10 mL pipettes
- 25 mL graduated cylinders or 10 mL automatic pipette



#### Nitrate Electrode Setup

Note: Add filling solution each day before using the electrode. The filling solution level should be at least one inch above the level of sample to ensure a proper flow rate. The fill hole should always be open when taking measurements.

See Figure 1 for labeled electrode components.

- 1. Remove the sensing module from the vial package. Make sure that both O-rings are in place below the threads on the sensing module. Remove the electrode handle from the box.
- 2. Unscrew the electrode cap. Slide the cap and spring down the electrode cable.
- 3. Hold the outer sleeve and gently push the inner stem through the outer sleeve. Slide the outer sleeve down the cable.
- 4. Grasp the inner stem without touching the reference pellet. With the other hand, unscrew and remove the red storage tip. Screw the sensing module onto the inner stem until it stops and is flush against the stem. Do not overtighten the module. The module should be firmly attached to the inner stem.
- 5. Hold the electrode cable and slide the outer sleeve, spring and cap over the inner stem.



Sensing membrane

- 6. Grasp the outer sleeve without touching the sensing module. With the other hand, pull on the cable and gently screw the cap onto the inner stem. Stop when resistance is felt. The cap will not completely stop. If the inner body turns at all, the cap is too tight.
- 7. Insert the spout of the Optimum Results F bottle into the electrode fill hole, add a small amount of filling solution to the chamber, tip the electrode upsidedown to wet the cap O-ring and return the electrode to its original, vertical position.
- 8. Hold the electrode body and use your thumb to push down on the electrode cap to allow a few drops of filling solution to drain out and wet the junction.
- 9. Release the electrode cap and allow the outer sleeve to return to its original position. If the outer sleeve does not return to its original position immediately, check if the O-ring is moist and repeat step 8.
- 10. Add filling solution to the electrode up to the bottom of the electrode fill hole.
- 11. Soak the electrode in a 100 ppm or  $10^{-2}$  M nitrate standard for 1 to 2 hours prior to use.

#### **Calibration Standard Preparation**

 Prepare a 50 ppm nitrate standard by pipetting 50 mL of the 100 ppm nitrate standard into a 100 mL volumetric flask. Dilute to the mark with deionized water. Mix the solution well.



2. Prepare a 10 ppm nitrate standard by pipetting 10 mL of the 100 ppm nitrate standard into a 100 mL volumetric flask. Dilute to the mark with deionized water. Mix the solution well.



3. Prepare a 1 ppm nitrate standard by pipetting 10 mL of the 10 ppm nitrate standard into a 100 mL volumetric flask. Dilute to the mark with deionized water. Mix the solution well.



4. Pipette 10 mL each of the 50 ppm, 10 ppm and 1 ppm standards into separate 50 mL beakers and label the beakers. Add 10 mL of NISS to each beaker. Gently stir the standard and NISS to mix the solution.



## **Sample Preparation**

1. Using a graduated cylinder or automatic pipette, measure and add 10 mL of the sample into a 50 mL beaker and label the beaker. Add 10 mL of NISS to the beaker. Gently stir the sample and NISS to mix the solution.



2. Repeat step 1 for additional samples.

## **Meter Preparation**

The Orion Dual Star meter has two BNC, reference and ATC connections. These connections are labeled as Channel 1 or Channel 2 on the ridge above the connections.



1. Prepare the power adapter by selecting the appropriate wall outlet plug and sliding the plug plate into the groove on the back of the adapter.



2. Connect the power adapter to the meter and then to the wall outlet. See *Figure 2*. Connect the nitrate electrode to one of the BNC inputs on the meter and note which channel (channel 1 or channel 2) was selected. See *Figure 3*. Connect the ATC probe to the 8 pin MiniDIN input on the meter and note which channel was selected. See *Figure 4*. Connect the stirrer probe to the stir jack input on the meter. See *Figure 5*.



## **Meter Setup**

Note: It is highly recommended that the EZ Startup menu be completed the first time that the meter is used. The EZ Startup menu sets important meter parameters, such as the displayed language, date and time, measurement mode and read type for each channel, and data output settings. To access the EZ Startup menu from the measurement mode, press the *setup* key, press the  $\blacktriangle$  /  $\checkmark$  keys to highlight <u>EZ Startup</u> and press the f2 (select) key.

- 1. In the measurement mode, press the *setup* key.
- Press the ▲ / ▼ keys to highlight <u>Channel 1</u> or <u>Channel 2</u>, depending on which BNC input the nitrate electrode was connected to, and press the *f2 (select)* key.
- 3. Press the ▲ / ▼ keys to highlight <u>Measure Mode</u> and press the *f2 (select)* key. Press the ▲ / ▼ keys to highlight <u>ISE</u> and press the *f2 (accept)* key. Press the ▲ / ▼ keys to highlight <u>ppm</u> (or <u>mg/L</u>, depending on your preference or method) and press the *f2 (accept)* key.
- Press the ▲ / ▼ keys to highlight <u>Electrode ID</u> and press the *f*2 (*select*) key. Press the ▲ / ▼ keys to highlight <u>NO</u><sub>3</sub><sup>-</sup> and press the *f*2 (*accept*) key.
- Press the ▲ / ▼ keys to highlight <u>Resolution</u> and press the *f2 (select)* key. Press the ▲ / ▼ keys to highlight <u>3 Significant Figures (1.00)</u> and press the *f2 (accept)* key.

- Press the ▲ / ▼ keys to highlight <u>Temperature Input</u> and press the *f*2 (*select*) key. Press the ▲ / ▼ keys to highlight <u>ATC1</u> or <u>ATC2</u>, depending on which 8 pin MiniDIN input the ATC probe was connected to, and press the *f*2 (*accept*) key.
- Press the ▲ / ▼ keys to highlight <u>Calibration Setup</u> and press the f2 (select) key. Press the ▲ / ▼ keys to highlight <u>Autoblank</u> and press the f2 (select) key. Press the ▲ / ▼ keys to highlight On and press the f2 (accept) key. Verify that the <u>Low Level Stability</u> and <u>Isopotential</u> options are set to <u>Off</u>. Press the f1 (back) key.
- Press the ▲ / ▼ keys to highlight <u>Read Type</u> and press the *f2 (select)* key. Press the ▲ / ▼ keys to highlight <u>On Ready</u> and press the *f2 (select)* key.
- 9. Press the *f1 (back)* key.
- 10. Press the  $\blacktriangle / \lor$  keys to highlight <u>Instrument</u> <u>Parameters</u> and press the *f*2 (*select*) key.
- 11. Press the ▲ / ▼ keys to highlight Export Data (PC/ Printer/Log) and press the f2 (select) key. Press the ▲ / ▼ keys to highlight Export Trigger and press the f2 (select) key. Press the ▲ / ▼ keys to highlight Channel 1 or Channel 2, depending on which BNC input the nitrate electrode was connected to, and press the f2 (accept) key. Press the ▲ / ▼ keys to highlight Data Log and press the f2 (select) key. Press the ▲ / ▼ keys to highlight Data Log On/Off and press the f2 (select) key. Press the ▲ / ▼ keys to highlight On and press the f2 (accept) key.
- 12. Press the *f1* (back) key twice.
- 13. Press the ▲ / ▼ keys to highlight <u>Stirrer Speed</u> and press the *f*2 (*select*) key. Press the ▲ / ▼ keys to highlight <u>1</u> and press the *f*2 (*accept*) key. Press the *f*1 (*back*) key.
- 14. Press the *f1 (back)* key. The meter will return to the measurement mode.

## **Calibration Procedure**

1. Prepare and condition the nitrate electrode. Connect the nitrate electrode, ATC probe and stirrer probe to the meter (note which channel the nitrate electrode is connected to) and place the electrode and probes in the electrode stand. Prepare the 50 ppm, 10 ppm and 1 ppm standards.



2. In the measurement mode, press the *cal* key.



 Dual channel display only: Press the ▲ / ▼ keys to highlight <u>Channel 1</u> or <u>Channel 2</u> as the channel to calibrate and press the *f2 (accept)* key.



4. Rinse the nitrate electrode, ATC probe and stirrer probe with deionized water, blot dry and place into the 1 ppm calibration standard.



5. When the electrode and standard are ready, press the *f3 (start)* key to begin the calibration.



6. Wait for the concentration value to stop flashing and then use the numeric keypad and the *decimal* key to enter the concentration of the first standard as 1.00 and press the *f2 (accept)* key.



7. Press the *f*2 (*next*) key to proceed to the next calibration standard.



8. Rinse the nitrate electrode, ATC probe and stirrer probe with deionized water, blot dry and place into the 10 ppm calibration standard.



9. When the electrode and standard are ready, press the *f3 (start)* key.



10. Wait for the concentration value to stop flashing and then use the numeric keypad and the *decimal* key to enter the concentration of the second standard as 10.0 and press the *f2 (accept)* key.



11. Press the *f*2 (*next*) key to proceed to the next calibration standard.



12. Rinse the electrode, ATC probe and stirrer probe with deionized water, blot dry and place into the 50 ppm calibration standard.



13. When the electrode and standard are ready, press the *f3 (start)* key.



14. Wait for the concentration value to stop flashing and then use the numeric keypad and the *decimal* key to enter the concentration of the third standard as 50.0 and press the *f2 (accept)* key.



15. Press the *f3 (cal done)* key. A summary of the calibration will be displayed. The slope should be -54 mV per decade to -65 mV per decade.



16. Press the *f*2 (*log/print*) key to save and end the calibration and export the calibration data to the calibration log.



### **Sample Measurement Procedure**

1. Calibrate the nitrate electrode and meter (note which channel the nitrate electrode is connected to) and make sure that the electrode and probes are in the electrode stand. Prepare the nitrate samples.



2. Rinse the nitrate electrode, ATC probe and stirrer probe with deionized water, blot dry and place into the sample. Press the *stirrer* key to turn on the stirrer probe.



3. The meter display will flash <u>stabilizing</u> and then show <u>ready</u> once the measurement is stable. Record the concentration and temperature of the sample when the meter display shows <u>ready</u>. When <u>ready</u> is shown on the display, the meter will export the measurement to the data log.



4. Press the *stirrer* key to turn off the stirrer probe. Remove the nitrate electrode, ATC probe and stirrer probe from the sample.



5. Repeat steps 2 through 4 for all of the samples.



 When all of the samples have been measured, store the equipment. Between samples, store the nitrate electrode in a 1:1 NISS to deionized water solution. For overnight storage, store the nitrate electrode in the 100 ppm nitrate standard. Store the ATC probe and stirrer probe dry. Visit www.thermoscientific.com/water to view the full line of Thermo Scientific Orion products and download product literature, user guides and manuals and software updates. For any questions or technical support, contact your local Technical Sales Representative or our Technical Support Specialists by phone at 1-978-232-6000 or 1-800-225-1480 (US toll-free), or by email at wai.techservbev@thermofisher.com.

# **Ordering Information**

To purchase an Orion Dual Star meter, electrodes and other related products, please contact your local equipment distributor and reference the part numbers listed below.

Product	Description	Part Number
Meter	Orion Dual Star pH/ISE Dual Channel Benchtop Meter	2115000
Electrodes	Orion Nitrate Combination Electrode	9707BNWP
	Orion Stainless Steel ATC Temperature Probe	927007MD
Solutions	Orion 100 ppm Nitrate As Nitrogen (N) Standard, 475 mL	930707
	Orion Nitrate Interference Suppressor Solution (NISS), 475 mL	930710
	Orion Optimum Results F Electrode Filling Solution, 5 x 60 mL	900046
Accessories	Orion Stirrer Probe	096019
	Swing Arm Electrode Stand	090043

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