Technical Bulletin 602

Low Level Nitrite Measurements For Water Samples

Overview

This application is designed to measure low-levels of nitrite in drinking and wastewater samples. Use this method for samples with nitrite concentrations between 0.5 ppm to 0.02 ppm as N. The Orion 9346 Nitrite Electrode measures nitrite in aqueous solution simply, accurately, and economically. Nitrite Interference Suppressor Solution (NISS) is added to all solutions to ensure that samples and standards have similar ionic strength, proper pH, and to reduce the effect of interfering ions. In Table 1, the Orion 920A ISE meter was used to calculate the slope and determine the sample concentration.

Table 1
Typical Results of Samples Run in EPA Sequence

Standard Calibration - Nitrite As N

1. 0.056 mg/L
2. 0.167 mg/L
3. 0.710 mg/L
4. 1.730 mg/L

Slope: -57.5 mV/d

Sequence Order	Meter Reading	Expected Reading	% Recovery
Calibration			
Blank:	0.0059 ppm (Below Detection)	<0.05 ppm	
Reference Standard:	0.553 ppm	0.549 ppm	101%
Blank:	0.0012 ppm (Below Detection)	<0.05 ppm	
Spike Blank:	0.110 ppm	0.112 ppm	98%
Midpoint Verification:	0.572 ppm	0.549 ppm	104%
Blank:	0.002 ppm (Below Detection)	<0.05 ppm	
Sample 1:	0.000 ppm (Below Detection)		
Sample 2:	0.0316 ppm (Below Detection)		
Sample 3:	0.107 ppm		
Sample 3 Duplicate:	0.112 ppm		(4.6%D)
Sample 3:	0.114 ppm		
Sample 3 Spike:	0.253 ppm	0.226 ppm	124%*
Sample 3 Spike Duplicate:	0.249 ppm	0.226 ppm	121%*
Sample 4:	0.000 ppm (Below Detection)		
Sample 5:	0.0048 ppm (Below Detection)		
Blank:	0.0029 ppm (Below Detection)	<0.05 ppm	
Midpoint Verification:	0.579 ppm	0.549 ppm	105%

Sample Origin
Sample 1 Well water from St. Matthews, S.C., Groundwater 30 feet
Sample 2 Abandoned open well water (30 feet deep) in central S.C., near St. Matthews
Sample 3 Holding Pond of a waste-stabilization land application system, S.C.
Sample 4 Tap water from Corsiville, Iowa
Sample 5 Secondary effluent water, Mass.
*The spike recovery is calculated with concentration of sample 3 equals to 0.114 ppm.

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Apparatus

1. Direct Concentration (ISE) meters such as Orion 920A, 720A, 710A, or 290A. If an ISE meter is unavailable, use a pH/mV meter with readability to 0.1 mV such as Orion 420A, 520A or 525A.
2. ORION Nitrite-Electrode
3. ORION Single Junction Reference Electrode
4. Volumetric flasks
5. Beakers
6. Pipettes
7. Magnetic Stirrer & Stir Bars

Required Solutions

1. Optimum Results F Reference Filling Solution
2. 0.1 M Nitrite Calibration Standard
3. Nitrite Interference Suppressor Solution (NISS)
4. Nitrite Storage Solution
5. Sodium Nitrite Reference Standard

Electrode Assembly

Nitrite Electrode: Remove the sensing module from the vial. Make sure the rubber electrode washer on the sensing module is in place. Screw the sensing module into the electrode body until finger tight. To ensure electrical continuity, shake down the electrode like a clinical thermometer. The membrane surface should look dark and homogeneous, with no bubbles on the inner surface.

Reference Electrode: Assemble and fill electrode in accordance to the reference electrode instruction manual. Use Orion 900046 Optimum Results reference filling solution.

Electrode Preparation

Rinse the nitrite electrode with distilled water, then soak in Nitrite Storage Solution for 1-2 hours before measurement.
Do not immerse electrode past the rubber electrode washer.

Nitrite Storage Solution Preparation

Prepared by adding 0.1 mL of 0.1 M Nitrite Calibration Standard and 50 mL of NISS to a 100 mL volumetric flask and dilute to volume with distilled water. Alternately, store the nitrite electrode in the lowest concentration of nitrite standard with NISS that was used during calibration.

Analysis Procedure

Calibration

Measure 25 mL distilled water into a 150 mL beaker and add 25 mL NISS. Rinse the electrodes with distilled water, blot dry, and place into beaker. Stir thoroughly. Add increments of the 14 ppm as N or 10^{-3} M NO_2^- standard to the beaker using steps outlined in Table 2. Wait for stable reading after each increment. If using an ISE meter, calibrate meter following meter instruction manual. If using a pH/mV meter, record stable mV reading after each increment and plot calibration curve on semilogarithmic paper.

Table 2

Preparing a Calibration Curve For Low-Level Nitrite Measurements

Step	Graduated Pipette Size	Added Volume	Concentration ppm as N	Concentration Molarity
1	1 mL	0.1 mL	0.056	$2.0X10^{-6}$
2	1 mL	0.2 mL	0.167	$1.2X10^{-5}$
3	1 mL	1.0 mL	0.710	$5.1X10^{-5}$
4	2 mL	2.0 mL	1.73	$1.2X10^{-4}$

Sample Measurement
Measure 25mL of sample into a beaker. Add 25mL NISS. Rinse the electrodes with distilled water, blot dry, and place into the sample. Stir thoroughly. When a stable reading is displayed, record the sample concentration directly from the meter.

Important Notes

1. Prepare a calibration curve with fresh standards each day.
2. Samples must be aqueous; not containing organic solvents.
3. Temperature must be less than 40 °C. Samples and standards should be at the same temperature (within ±0.5 °C). At the 1 ppm as N or 10^{-4} M NO_2^- level, a 1 °C difference in temperature will give rise to about a 2% error. For highly accurate results, use of a water bath may become necessary to control temperature variances. This particular application does not use temperature control.
4. After immersion in solution, check nitrite electrode for any air bubbles on membrane surface. Remove air bubbles at the electrode surface by gently tapping electrodes.
5. The electrode should be submerged approximately half the length of the nitrite electrode module.
6. Reference standard is made fresh with sodium nitrite salt.

Ordering Information

Description	Orion
ORION *ionplus* Nitrite Combination Electrode with/BNC Connector	9746BN
ORION Nitrite Electrode w/BNC Connector	9346BN
ORION Nitrite Electrode w/U.S. Standard Connector	934600
Replacement Nitrite Modules	934601
ORION Single Junction Reference Electrode	900100
Optimum Results F Reference Filling Solution	900046
Nitrite Interference Suppressor Solution (NISS)	934610
Nitrite Calibration Standard	954606

Note: When using the ORION *ionplus* Nitrite Combination Electrode a Reference Electrode ***is not*** necessary.

FOR ADDITIONAL INFORMATION CALL THE TECHNICAL EDGE FOR ORION PRODUCTS at 1-800-225-1480.

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