



Meter Log # 82

Parameter and Sample Type

Nitrite in Surface Water and Wastewater by Colorimetry

Introduction

The Thermo Scientific Orion AQUA fast® AQ3700 Colorimeter comes programmed with methods for testing nutrients. This application note covers testing nitrite in wastewater and surface water using Orion AC2046 Tablets. Nitrite ions react with a reagent tablet and undergoe diazotization to produce a highly colored azo dye. The method range is $0.01-0.5~\text{mg NO}_2$ -N/L. The Orion AC2046 Method is accepted by U.S. EPA for wastewater and drinking water compliance monitoring.

References

- 1. Thermo Orion Method AC2046 Nitrite Nitrogen Test Kit Method, rev 5, 04/24/2002; EPA July 24, 2003 (drinking water) and June 7, 2004 (wastewater).

 www.thermoscientific.com/water
- Method 4500 NO₂- B, Colorimetric Method. Standard Methods for the Examination of Water and Wastewater. APHA, AWWA, & WEF, Washington, D.C. www.standardmethods.org

Recommended Equipment

Orion AQ3700 Colorimeter; Orion AC2046, Nitrite Test Kit; Orion AC2V24 24mm vials: timer.

Required Solutions

Orion 954606 0.1 M Nitrite standard (1400 mg/L as N); intermediate nitrite standard, 14 mg NO₂-N/L; calibration verification nitrite standard, 0.21 mg NO₂-N/L; deionized (DI) water.

Solutions Preparation

- 1. 14 mg NO₂-N/L standard: pipet 1.0 mL of 0.1 M nitrite standard into a 100-mL volumetric flask. Dilute to the mark w/ DI.
- 2. 0.21, NO₂-N/L calibration verification standard: pipet 1.5 mL of 14 mg NO₂-N/L standard into a 100-mL volumetric flask. Dilute to the mark with DI.

Meter Setup

Before starting the analysis, read carefully Method 270 (Nitrite with Tablet) and Section 1.2 of the AQ3700 User Guide, Important Notes.

Turn on the meter, select method 270 for "Nitrite with Tablets" testing. Fill a clean vial to the line with DI water. Cap and wipe w/lint-free wiper until dry and free of smears. Handling the vial by the cap, insert the vial into the meter aligning the mark on the vial with the mark on the meter. Zero the meter on the DI water.

Sample Storage and Preparation

Store samples refrigerated (<6°C). Test samples within 48 hours.

Sample Vial Storage and Cleaning

Clean and store vials per instructions in the user guide. Do not allow reacted samples to remain in the vials overnight.

Meter Performance Check/Calibration Verification

Check meter performance by testing a reagent blank and the 0.21 mg NO₂⁻-N/L standard as follows: fill one clean vial with 10.0 mL of DI water for the reagent blank and another clean vial with 10.0 mL of 0.21 mg/L calibration verification standard. Use a clean pipet for best accuracy. Add reagent and test each vial according to the "Testing Procedure" paragraph below. The reagent blank should read <0.01 mg/L ("underrange" will be displayed) and the 0.21 mg/L standard should read within +/- 10%. If the meter performance check fails, take corrective actions as described in the Appendix of this note.

Calibration

The meter is shipped precalibrated. The meter performance is very stable and does not require frequent calibration. If a standard reading is not within criteria, see corrective actions above. If the corrective actions fail, perform 1-point calibration according to User calibration Method of the AQ3700 User Guide.

Sample Preparation

Fill a clean vial with 10 mL of sample. Use a pipet for best accuracy. (Note: If the sample is colored or turbid, zero the meter with the filled sample vial to compensate. Remember to re-zero the meter with DI water in a sample vial before testing a clear, colorless sample).

Testing Procedure

Tear open the foil pouch containing the AC2046 Tablet and add the tablet into the vial without touching the tablet. (See illustration in the Appendix). Crush the tablet with the tamping rod. Close the vial tightly with the cap and swirl until the tablet is completely dissolved. Wipe the vial until clean and dry. Insert into the meter aligning marks. Press the test key. After 10 minutes, the meter will automatically read the sample and display the nitrite reading in mg/l as nitrogen. For multiple samples, use a timer to count 10 minutes, then test samples sequentially, bypassing the meter timer by pressing the arrow/8 key when the meter countdown starts.

Quality Control (QC)

Recommended QC procedures include: calibration verification, reagent blank analysis, QC samples, sample duplicates & spikes.





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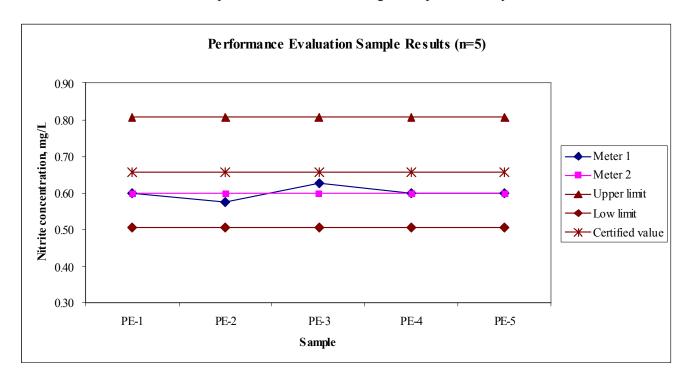
Nitrite Results:

Reagent blanks are clean, standard checks are accurate, sample replicates are reproducible, spike recovery is good, and spike duplicates show good agreement.

Parameter	Replicates	Criteria	AQ3700 Orion 1	AQ3700 Orion 2
Reagent Blanks	4	< 0.03 mg/L	<0.03 (underrange)	<0.03 (underrange)
0.03 mg/L Std	4	90 - 110% R	100.0%	100.0%
0.20 mg/L Std	4	90 - 110% R	100.0%	105.0%
0.45 mg/L Std	4	90 - 110% R	97.8%	100.0%
MDL (n=7)	7	< 0.03 mg/L	< 0.01 mg/L	< 0.01 mg/L
Wastewater Effluent	6		0.07	0.07
STDEV			0.004	0.000
%CV		< 15% RSD	5.7%	0.0%
spike (0.14 mg/L)	1	85 - 115% R	91.7%	100.0%
spike dupl (0.14 mg/L)	1	85 - 115% R	91.7%	100.0%
Surface Water	2		<0.03 (underrange)	<0.03 (underrange)
STDEV			not applicable	not applicable
%CV		< 15% RSD	not applicable	not applicable
spike (0.21 mg/L)	1	85 - 115% R	85.7%	85.7%
spike dupl (0.21 mg/L)	1	85 - 115% R	90.5%	90.5%

Performane Evaluation (PE) Standard Results:

PE standard results are within acceptance criteria and show good reproducibility.







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Appendix:

Correct use of tablet reagents:

The tablet reagents should be added to a sample straight from the foil without touching them with the fingers as shown on the images below; a clean tamping rod should be used to crush the tablets





Corrective Actions:

If the meter performance check fails, take corrective actions as follows: 1) wipe the vial carefully with a lint-free wipe to remove all fingerprints and liquid drips from the exterior, handle the vial by the cap only, and remeasure; 2) if the tablet is not white and does not hold its form, use a fresh tablet or another lot of tablets; 3) using a clean vial, rezero the meter with DI water; using the same vial, fill with 0.21 mg/L standard or DI, add reagent tablets, and retest; 4) prepare a fresh standard and retest; 5) if the both the standard retest and the reagent blank retest read high, apply a reagent blank correction by zeroing the meter on the fresh reagent blank, and re-read the standard.