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Application Note

Meter Log #83

Parameter and Sample Type
Nitrate in Surface Water and Wastewater by Colorimetry

Introduction
This application note covers the procedure to enter a User Concentration Method for testing nitrate in surface and wastewater on the Thermo Scientific Orion AQUAfast® AQ3700 Colorimeter. Nitrate is determined using Orion AC2007 Tablets. The first tablet reduces nitrate to nitrite using zinc as the reducing agent, and then nitrite ion reacts with a second reagent tablet to produce a highly colored azo dye. The method range is 0.30 – 9.0 mg NO_3^--N/L. The Orion AC2007 Method is similar to the cadmium reduction method accepted by U.S. EPA for drinking water and wastewater compliance monitoring. Consult the Method Modifications guidelines in 40 CFR Part 136.6(b)(2)(i) for monitoring use requirements.

References

1. ASTM D3867-09 Standard Test Methods for Nitrite-Nitrate in Water www.astm.org.
2. 2. Method 4500 NO3- E, Cadmium Reduction 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 AC2007, Nitrate Test Kit; Orion AC2V24 24mm vials; timer.

Required Solutions
Orion 920707 1000 ppm Nitrate as N standard; intermediate nitrate standard, 100 mg/L NO_3^--N/L; calibration standards, 0.30, 1.0, 3.0, 6.0, and 9.0 mg/L NO_3^--N/L; calibration verification standard, 4.0 mg/L $NO3^-$-N/L; deionized water.

Solutions Preparation
1) 100 mg NO_3^--N/L intermediate nitrite standard: pipet 10.0 mL of 1000 ppm nitrate standard into a 100-mL volumetric flask and dilute to the mark with DI.
2) 0.30, 1.0, 3.0, 6.0, and 9.0 mg/L NO_3^--N/L calibration standards: pipet 0.30, 1.0, 3.0, 6.0, and 9.0 mL of 100 mg $NO3^-$-N/L standard each into the respective 100-mL volumetric flasks and dilute to the mark with DI.
3) 4.0 mg/L NO_3^--N/L calibration verification standard: pipet 4.0 mL of 100 mg NO_3^--N/L standard into a 100-mL volumetric flask and dilute to the mark with DI.

Calibration Curve Preparation
Prepare reagent blank (0 mg/L NO_3^--N/L): fill a clean vial with 10 mL of DI water. Prepare 0.30, 1.0, 3.0, 6.0, and 9.0 mg/L NO_3^--N/L calibration standards: for each standard, pipette 5.0 mL of DI water and 5.0 mL of calibration standard solution into a clean vial.

Meter Setup
Turn on the meter. Follow the steps described in Section 2.4.7 User Operations, User Concentration Methods of the AQ3700 User Guide to start the calibration procedure. When prompted, enter a new method number in the range from 850 to 859, wavelength of 530 nm, resolution of 0.01, and units of mg/L. Use a clean vial filled with DI water to Zero the meter.

Generate the Calibration Curve
After zeroing with DI, start with the reagent blank as the first standard (S1) as follows: add reagent according to the "Reagent Addition" paragraph below. Repeat for all standards in order of increasing concentration. When color has developed exactly 10 minutes for the reagent blank, insert into the meter and enter the concentration of S1 as 0 mg/L, then press TEST. (Note**:** create a written record of the concentration and absorbance data for each calibration point as it is displayed. See Appendix for details). When all required standards are entered and measured, press Store key.

Calibration Verification
Check meter accuracy by testing a nitrate standard at 4.0 mg NO3--N/L and a reagent blank. The reagent blank should read <0.10 (under range) and the 4.0 mg/L standard should read within +/- 10%. If not, take corrective action as details on page 2.

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.

Sample Preparation
Fill a clean, dry vial with 5.0 mL of sample and 5.0 mL DI water. Use a pipette for best accuracy.

Reagent Addition
Before testing, read carefully Section 1.2 Important Notes in the AQ3700 User Guide. Add one nitrate HR 1 tablet straight from the foil to the vial. Crush the tablet with a clean tamping rod. Mix the vial well to dissolve the tablet completely. Then add one nitrate HR 2 tablet straight from the foil to this vial. Crush the tablet with the same tamping rod. Mix the vial well to dissolve the tablet completely. Cap the vial tightly and wipe the vial exterior to ensure that it is clean and dry. Record the time when the second tablet is dissolved and wait exactly 10 minutes for color development. While waiting, start preparing the next standard using the same test procedure. After 10 minutes exactly, place the vial in the meter and press TEST key.

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

Results for Wastewater and Surface Water Testing:

Reagent blanks are clean, standard checks are accurate, sample replicates are reproducible, matrix spike recovery is good, spike duplicates show good agreement, and results for the commercially-available performance evaluation (PE) sample are within acceptance criteria.

Parameter	Replicates	Criteria*	AQ3700 Orion 1	AQ3700 Orion 2
Reagent Blank	4	<0.10 mg/L	0.01	0.00
Standards				
3.0 mg/L	4	88 - 114% R	107.6%	108.2%
0.50 mg/L	3	88 - 114 % R	94.7%	93.3%
4.0 mg/L	3	88 - 114 % R	108.6%	110.8%
8.0 mg/L	3	88 - 114 % R	98.6%	97.0%
MDL	7**	<0.30 mg/L	0.09	0.10
Wastewater Effluent	8	--	6.95	6.91
STDEV	--	--	0.75	0.71
%CV	--	22% RSD	10.8%	10.3%
spike (3.0 mg/L)	1	77 - 121% R	85.8%	86.5%
spike dupl (3.0 mg/L)	1	77 - 121% R	93.8%	90.5%
%D	2	22% RPD	3.9%	2.0%
Surface Water	4	--	0.27	0.28
STDEV	--	--	0.026	0.030
%CV	--	14% RSD	9.6%	10.8%
spike (5.0 mg/L)	1	87 - 115% R	106.2%	104.5%
spike dupl (5.0 mg/L)	1	87 - 115% R	99.0%	97.5%
%D	2	14% RPD	6.7%	6.6%
PE Sample	6	5.02 - 6.46	5.35	5.29
STDEV	--	--	0.25	0.22
%CV	--	13% RSD	4.8%	4.2%

* Criteria from EPA-821-B-98-002, Protocol for EPA Approval of Alternate Test Procedures for Organic and Inorganic Analytes in Wastewater and Drinking Water.

** Method Detection Limit (MDL) determined on 7 replicates of 0.30 mg/L standard

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 check standard or DI, add reagent tablet, 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.

Appendix:

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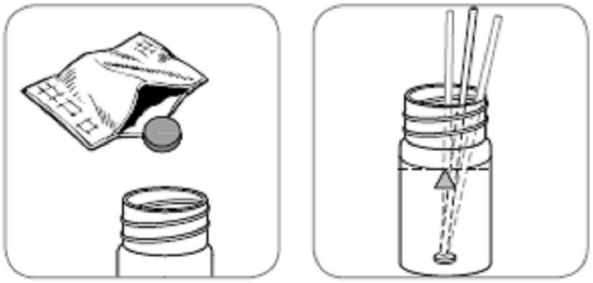
Correct use of tablet reagents:

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.