Nitrate

DOC316.53.01465

Cadmium Reduction Method **Method 8039**

0.4 to 30.0 mg/L NO_3^-N (HR) **Powder Pillows or AccuVac® Ampuls**

Scope and application: For water, wastewater and seawater.

Test preparation

Before starting

For the best results, measure the reagent blank value for each new lot of reagent. Replace the sample with deionized water in the test procedure to determine the reagent blank value. Subtract the reagent blank value from the sample results.
This method is technique-sensitive. Shaking time and technique influence the color development. For most accurate results, use a standard solution that is within the test range and run the test several times. Increase or decrease the shaking time to get the expected result. Use the adjusted shaking time for sample measurements.
The reagents that are used in this test contain cadmium. Rinse the sample cell immediately after use to remove all cadmium particles. Collect the reacted samples for safe disposal.
A deposit of unoxidized metal will remain at the bottom of the sample cell after the reagent dissolves. The deposit will not affect results.
Always do tests in sample cells or AccuVac© Ampuls. Do not put the instrument in the sample or pour the sample into the cell holder.
Make sure that the sample cells are clean and there are no scratches where the light passes through them.
Rinse the sample cell and cap with the sample three times before the sample cell is filled.
Make sure that there are no fingerprints or liquid on the external surface of the sample cells or AccuVac© Ampuls. Wipe with a lint-free cloth before measurement.
Cold waters can cause condensation on the sample cell or bubbles in the sample cell during color development. Examine the sample cell for condensation or bubbles. Remove condensation with a lint-free cloth. Invert the sample cell to remove bubbles.
Install the instrument cap over the cell holder before ZERO or READ is pushed.
Rinse the sample cell immediately after use to remove metal particles.
The AccuVac Ampul Snapper makes AccuVac Ampul tests easier to do. The AccuVac Ampul Snapper keeps the broken tip of the ampul, prevents exposure to the sample and provides controlled conditions for filling the ampule.
Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.
Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

Powder pillows

Description	Quantity
NitraVer® 5 Nitrate Reagent Powder Pillow, 10-mL	1
Sample cells, 25-mm (10 mL)	2

Refer to Consumables and replacement items on page 7 for order information.

AccuVac Ampuls

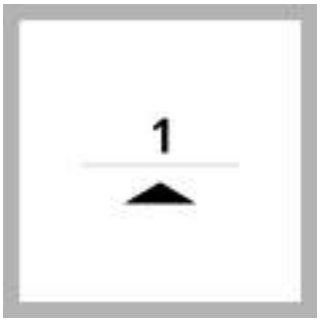
Description	Quantity
NitraVer® 5 Nitrate Reagent AccuVac® Ampul	1
Beaker, 50-mL	1
Stopper for 18-mm tubes and AccuVac Ampuls	1
Sample cell, 25-mm (10 mL)	1

Refer to Consumables and replacement items on page 7 for order information.

Sample collection and storage

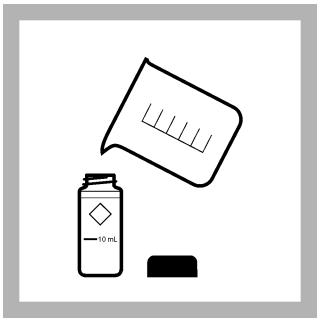
- Collect samples in clean glass or plastic bottles.
- Analyze the samples as soon as possible for best results.
- If immediate analysis is not possible, immediately filter and keep the samples at or below 6 °C (43 °F) for a maximum of 48 hours.
- To preserve samples for a maximum of 28 days, adjust the sample pH to 2 or less with concentrated sulfuric acid (approximately 2 mL per liter) and keep at or below 6 °C (43 °F). The test results then include nitrate and nitrite.
- Let the sample temperature increase to room temperature before analysis.
- Before analysis, adjust the pH to 7 with 5 N sodium hydroxide solution.
- Correct the test result for the dilution caused by the volume additions.

Powder pillow procedure

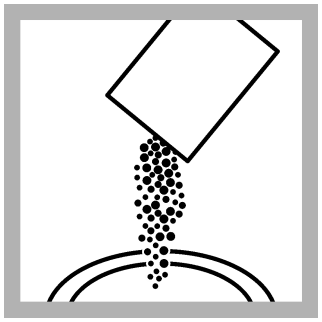


1. Set the instrument to channel 1.

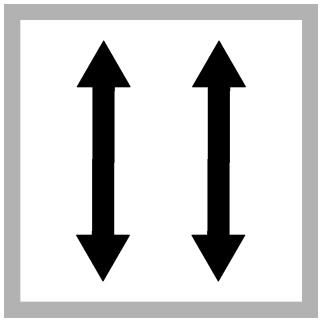
For DR300, push the up arrow button. For PCII, push the menu button, checkmark button, then the menu button again.



2. Prepare the sample: Rinse a sample cell and cap three times with sample. Fill the sample cell to the 10-mL mark with sample.



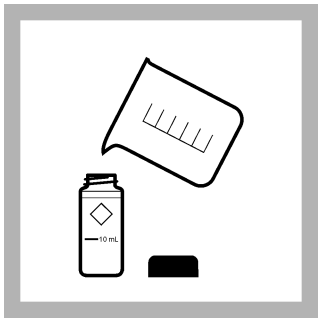
3. Add the contents of one NitraVer 5 Nitrate Reagent Powder Pillow to the cell.



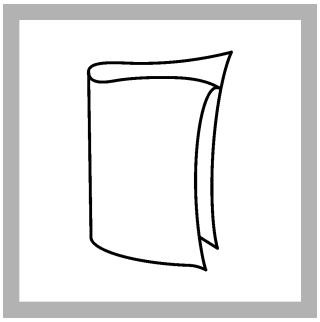
4. Put the stopper on the sample cell. Shake vigorously for 1 minute. Undissolved powder will not affect results..



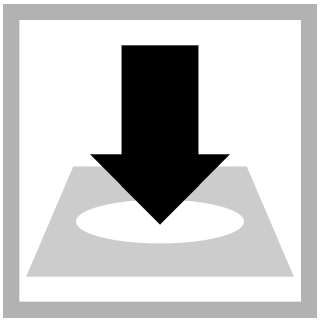
5. Set and start a timer for 5 minutes. A 5-minute reaction time starts.



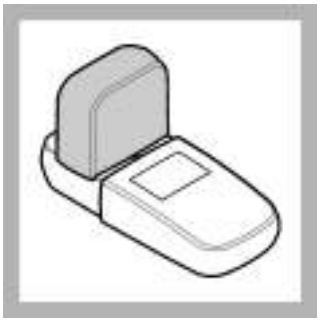
6. Prepare the blank: Rinse a sample cell and cap three times with sample. Fill the sample cell to the 10-mL mark with sample. Close the sample cell.



7. Clean the blank sample cell.



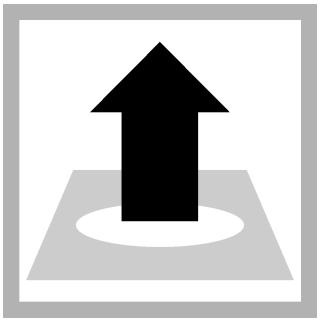
8. Insert the blank into the cell holder. Point the diamond mark on the sample cell toward the keypad.



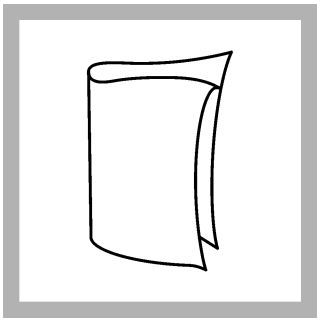
9. Install the instrument cap over the cell holder.



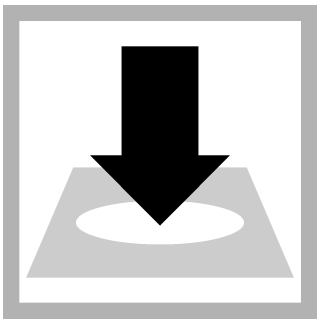
10. Push **ZERO**. The display shows “0.0”.



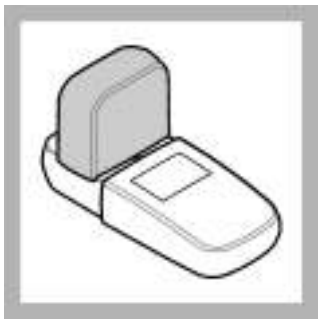
11. Remove the sample cell from the cell holder.



12. Clean the prepared sample cell.



13. Within 1 minute after the timer expires, insert the prepared sample into the cell holder. Point the diamond mark on the sample cell toward the keypad.

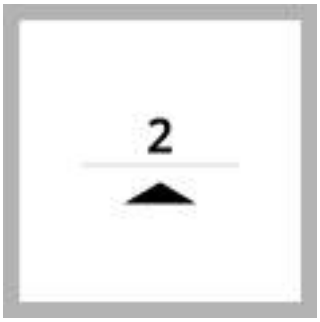


14. Install the instrument cap over the cell holder.



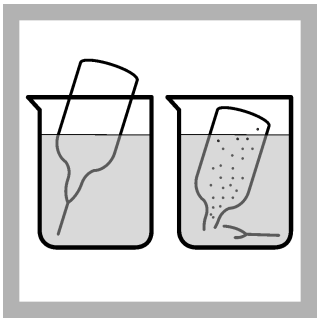
15. Push **READ**. Results show in mg/L nitrate as nitrogen (NO_3^-–N). To change the test results to mg/L nitrate (NO_3^-), multiply the test result by 4.4.

AccuVac® Ampul procedure

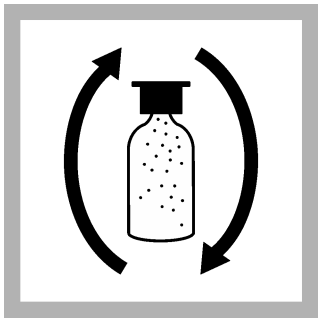


1. Set the instrument to channel 2.

For DR300, push the up arrow button. For PCII, push the menu button, checkmark button, then the menu button again.



2. Prepare the sample: Collect at least 40 mL of sample in a 50-mL beaker. Fill the AccuVac Ampul with sample. Keep the tip immersed while the AccuVac Ampul fills completely.

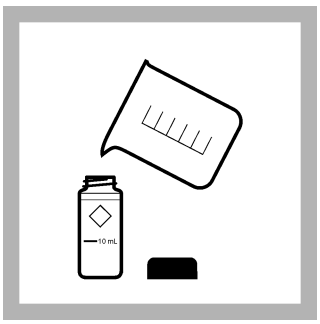


3. Quickly invert the AccuVac Ampul several times to mix.
Invert for 1 minute (approximately 48 to 52 times). Undissolved powder will not affect results.

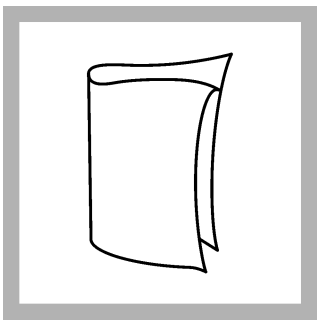


4. Set and start a timer for 5 minutes. A 5-minute reaction time starts.

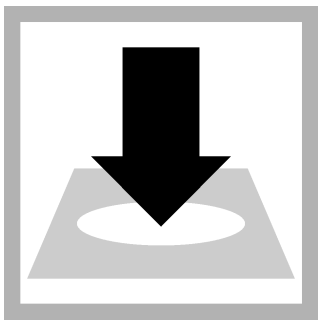
Keep the sample still while the timer counts down. An amber color will show if nitrate is in the sample.



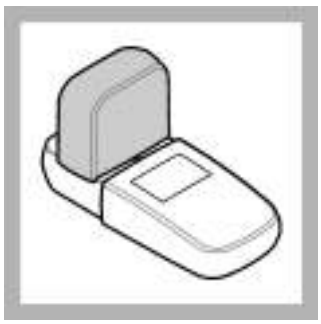
5. Prepare the blank: Rinse a sample cell and cap three times with sample. Fill the sample cell to the 10-mL mark with sample. Close the sample cell.



6. Clean the blank sample cell.



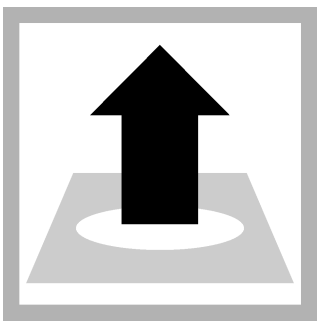
7. Insert the blank into the cell holder. Point the diamond mark on the sample cell toward the keypad.



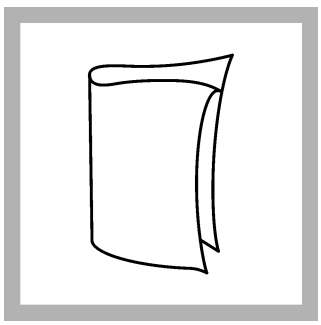
8. Install the instrument cap over the cell holder.



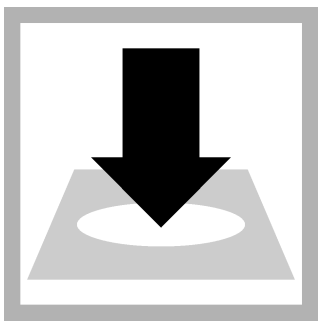
9. Push **ZERO**. The display shows “0.0”.



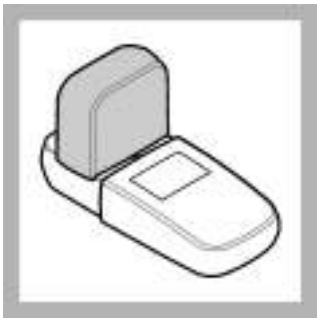
10. Remove the sample cell from the cell holder.



11. Clean the AccuVac Ampul.



12. Within 1 minute after the timer expires, insert the prepared sample AccuVac Ampul into the cell holder.



13. Install the instrument cap over the cell holder.



14. Push **READ**. Results show in mg/L nitrate as nitrogen (NO_3^-–N). To change the test results to mg/L nitrate (NO_3^-), multiply the test result by 4.4.

Interferences

Interfering substance	Interference level
Chloride	Chloride concentrations above 100 mg/L cause low results. The test can be used at high chloride concentrations (seawater) if a calibration is made with standards that have the same chloride concentration as the samples (refer to Seawater calibration on page 5).
Ferric iron	Interferes at all levels
Nitrite	Interferes at all levels Compensate for nitrite interference as follows: **1.** Add 30-g/L Bromine Water by drops to the sample until a yellow color remains. **2.** Add 1 drop of 30-g/L Phenol Solution to remove the color. **3.** Use the test procedure to measure the concentration of the treated sample. Report the results as total nitrate and nitrite.
Highly buffered samples or extreme sample pH	Can prevent the correct pH adjustment (of the sample) by the reagents. Sample pretreatment may be necessary.
Strong oxidizing and reducing substances	Interfere at all levels

Pollution prevention and waste management

Reacted samples contain cadmium and must be disposed of as a hazardous waste. Dispose of reacted solutions according to local, state and federal regulations.

Seawater calibration

Chloride concentrations above 100 mg/L cause low results. To use this method for samples with high chloride concentrations, calibrate the instrument with nitrate standard solutions that contain the same amount of chloride as the samples.
Prepare calibration standards that contain chloride and 1.0, 3.0, 5.0 and 10.0 mg/L nitrate (as NO_3^-–N) as follows:

1. Prepare 1 liter of chloride water that has the same chloride concentration as the samples.

a. Weigh the applicable amount of ACS-grade sodium chloride: (chloride concentration of samples in g/L) x (1.6485) = g of NaCl per liter.

Note: *18.8 g/L is the typical chloride concentration of seawater.*

b. Add the sodium chloride to a 1-liter volumetric flask.

c. Dilute to the mark with deionized water. Mix thoroughly. Use this water as the dilution water to prepare the nitrate standard solutions.

2. Use a pipet to add 1.0, 3.0, 5.0 and 10.0 mL of a 100 mg/L nitrate-nitrogen (NO_3^-–N) standard solution into four different 100-mL Class A volumetric flasks.
3. Dilute to the mark with the prepared chloride water. Mix thoroughly.
4. Complete the test procedure for each of the standard solutions and for the prepared chloride water (for a 0-mg/L standard solution).
5. Measure the absorbance of the standard solutions and enter a user-prepared calibration into the instrument.

Accuracy check

Standard additions method

Use the standard additions method to validate the test procedure, reagents and instrument and to find if there is an interference in the sample.

Items to collect:

- Nitrate Nitrogen Standard Solution, 500 mg/L NO_3^-–N, 10-mL Voluette® Ampules
- Ampule breaker
- Pipet, TenSette®, 0.1–1.0 mL and tips

1. Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the standard solution, respectively, to three 10-mL portions of fresh sample. Mix well.

   Note: *For AccuVac® Ampuls, add 0.4 mL, 0.8 mL and 1.2 mL of the standard solution to three 50-mL portions of fresh sample.*
2. Use the test procedure to measure the concentration of each of the spiked samples. Start with the smallest sample spike. Measure each of the spiked samples in the instrument.
3. Compare the expected result to the actual result. The expected nitrate-nitrogen concentration increase is 5.0 mg/L for each 0.1 mL of standard that is added. For AccuVac Ampuls, the expected nitrate-nitrogen concentration increase is 4.0 mg/L for each 0.4 mL of standard that is added.

Standard solution method

Use the standard solution method to validate the test procedure, the reagents and the instrument.

Items to collect:

- Nitrate Nitrogen Standard, Solution, 10.0-mg/L NO_3^-–N

1. Use the test procedure to measure the concentration of the standard solution.
2. Compare the expected result to the actual result.

   Note: *The factory calibration can be adjusted slightly with the standard calibration adjust option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are small variations in the reagents or instruments.*

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a DR300 and a Pocket Colorimeter II during ideal test conditions. Users can get different results under different test conditions.

Precision (95% confidence interval)
10.0 ± 3.0 mg/L NO_3^-N (powder pillow); 10.0 ± 0.8 mg/L NO_3^-N (AccuVac Ampul)

Summary of method

Cadmium metal reduces nitrate in the sample to nitrite. The nitrite ion reacts in an acidic medium with sulfanilic acid to form an intermediate diazonium salt. The salt couples with gentisic acid to form an amber colored solution.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
NitraVer® 5 Nitrate Reagent Powder Pillow, 10 mL	1	100/pkg	2106169
OR			
NitraVer® 5 Nitrate Reagent AccuVac® Ampul	1	25/pkg	2511025

Required apparatus (powder pillows)

Description	Quantity/test	Unit	Item no.
Sample cells, 10-mL round, 25 mm x 60 mm	2	6/pkg	2427606

Required apparatus (AccuVac Ampul)

Description	Quantity/Test	Unit	Item no.
Sample cell, 10-mL round, 25 mm x 60 mm	1	6/pkg	2427606
Beaker, 50 mL	1	each	50041H
Stoppers for 18-mm tubes and AccuVac Ampuls	2	6/pkg	173106

Recommended standards

Description	Unit	Item no.
Nitrate Nitrogen Standard Solution, 10.0-mg/L NO_3–N	500 mL	30749
Nitrate Nitrogen Standard Solution, Voluette® Ampule, 500-mg/L NO_3–N	16/pkg	1426010
Wastewater Influent Standard Solution, Mixed Parameter, for NH_3-N, NO_3-N, PO_4, COD, SO_4, TOC	500 mL	2833149

Optional reagents and apparatus

Description	Unit	Item no.
AccuVac® Ampul Snapper	each	2405200
Bromine Water, 30 g/L	29 mL	221120
Mixing cylinder, graduated, 50 mL	each	2088641
Pipet, TenSette®, 0.1–1.0 mL	each	1970001
Pipet, volumetric, Class A, 0.5 mL	each	1451534
Pipet, volumetric, Class A, 1.00 mL	each	1451535
Pipet, volumetric, Class A, 2 mL	each	1451536
Pipet, volumetric, Class A, 3 mL	each	1451503
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	50/pkg	2185696
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	1000/pkg	2185628
Phenol Solution, 30-g/L	29 mL	211220
Pipet filler, safety bulb	each	1465100
Sodium Hydroxide Solution, 5 N	50 mL	245026
Sulfuric Acid, ACS	500 mL	97949
Flask, volumetric, Class A, 100 mL, glass	each	1457442

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HACH COMPANY
WORLD HEADQUARTERS
Telephone: (970) 669-3050
FAX: (970) 669-2932

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