Nitrate

DOC316.53.01066

Cadmium Reduction Method

Method 8039

0.3 to 30.0 mg/L NO_3^-N (HR)

Powder Pillows or AccuVac® Ampuls

Scope and application: For water, wastewater and seawater.

Test preparation

Instrument-specific information

Table 1 shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests. Table 2 shows sample cell and adapter requirements for AccuVac Ampul tests. The tables also show all of the instruments that have the program for this test.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information for reagent addition

Instrument	Sample cell orientation	Sample cell
DR 6000 DR 3800 DR 2800 DR 2700 DR 1900	The fill line is to the right.	2495402 
DR 5000 DR 3900	The fill line is toward the user.	
DR 900	The orientation mark is toward the user.	2401906 

Table 2 Instrument-specific information for AccuVac Ampuls

Instrument	Adapter	Sample cell
DR 6000 DR 5000 DR 900	—	2427606 
DR 3900	LZV846 (A)	
DR 1900	9609900 or 9609800 (C)	
DR 3800 DR 2800 DR 2700	LZV584 (C)	2122800 

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Before starting

Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.
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 automatically with the reagent blank adjust option.
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.
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. (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	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
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	1
Stopper for 18-mm tubes and AccuVac Ampuls	1

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

Sample collection and storage

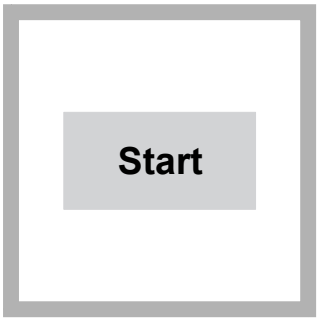
- Collect samples in clean glass or plastic bottles.
- Analyze the samples as soon as possible for best results.
- If immediate analysis is not possible, immediately filter and keep the samples at or below 6 °C (43 °F) for a maximum of 48 hours.
- To preserve samples for a maximum of 28 days, adjust 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.

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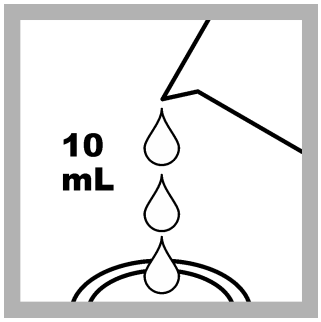
Powder pillow procedure

⚠ CAUTION

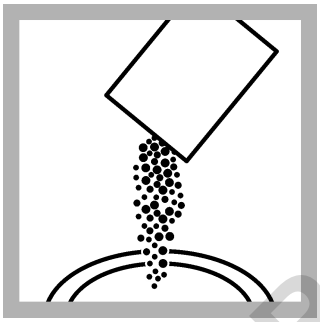
Hazardous waste exposure. Prepared samples contain cadmium. Refer to the SDS for safe handling and disposal instructions. Obey all local and regional disposal regulations.



1. Start program **355 N, Nitrate HR PP**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.



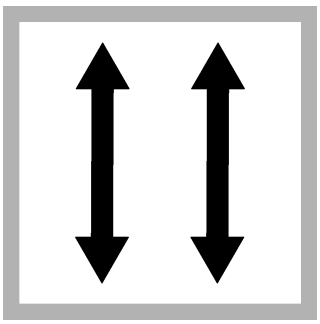
2. Prepare the sample: Fill a sample cell with 10 mL of sample.



3. Add the contents of one NitraVer 5 Nitrate Reagent Powder Pillow. Put the stopper on the sample cell.



4. Start the instrument timer. A 1-minute reaction time starts.

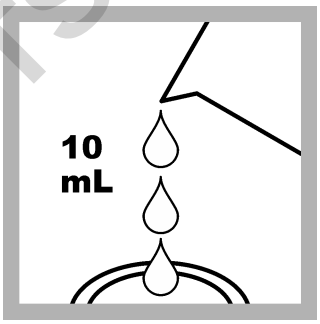


5. Put the stopper on the sample cell. Shake the cell vigorously until the timer expires. Some powder may not dissolve. Undissolved powder will not affect results.

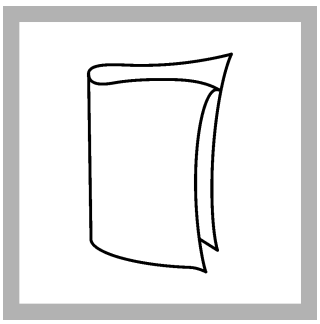


6. Start the instrument timer. A 5-minute reaction time starts.

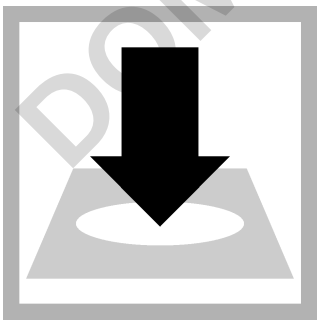
An amber color shows if nitrate is present.



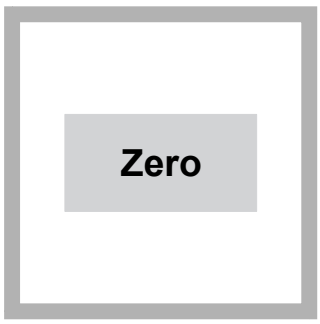
7. Prepare the blank: When the second timer expires, fill a second sample cell with 10 mL of sample.



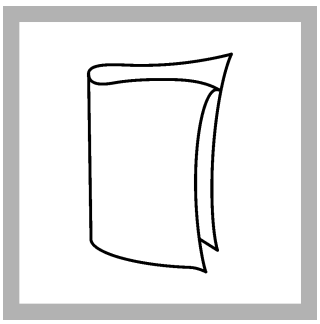
8. Clean the blank sample cell.



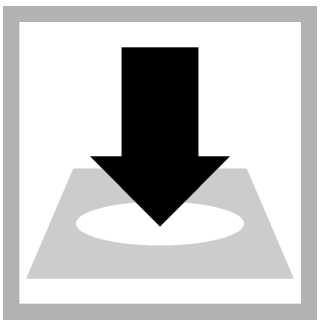
9. Insert the blank into the cell holder.



10. Push **ZERO**. The display shows 0.0 mg/L NO_3^-–N.

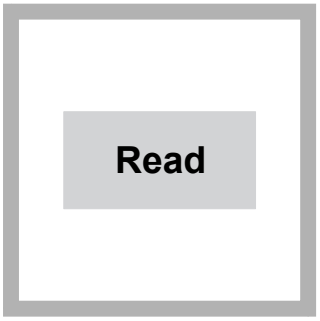


11. Clean the prepared sample cell.



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

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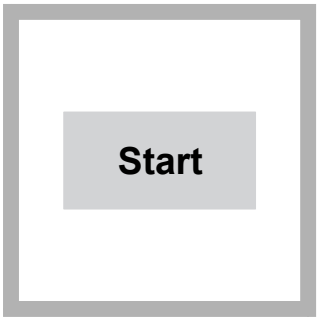


13. Push **READ**. Results show in mg/L NO_3^-–N.

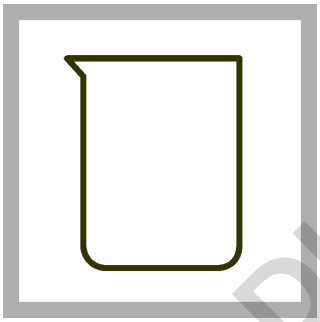
AccuVac Ampul procedure

⚠CAUTION

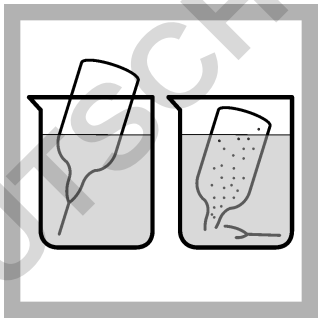
Hazardous waste exposure. Prepared samples contain cadmium. Refer to the SDS for safe handling and disposal instructions. Obey all local and regional disposal regulations.



1. Start program **361 N, Nitrate HR AV**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.



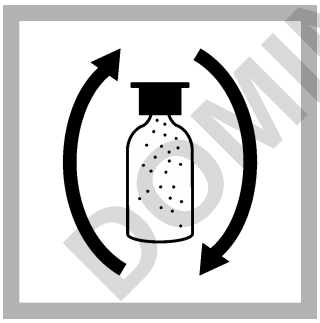
2. Prepare the sample: Collect at least 40 mL of sample in a 50-mL beaker.



3. Tap the bottom of a NitraVer 5 Nitrate AccuVac Ampul to dislodge the powder. Fill the Ampul with sample. Keep the tip immersed while the Ampul fills completely.



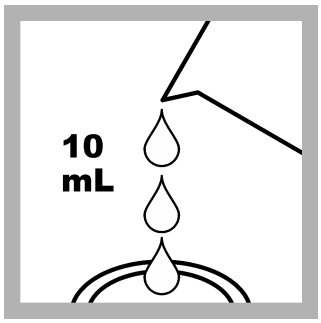
4. Start the instrument timer. A 1-minute reaction time starts.



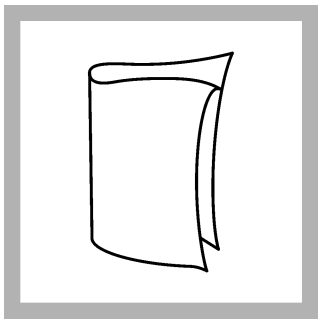
5. Invert the Ampul 48 to 52 times as the timer counts down.



6. Start the instrument timer. A 5-minute reaction time starts.
Keep the sample still while the timer counts down. An amber color shows if nitrate is present.

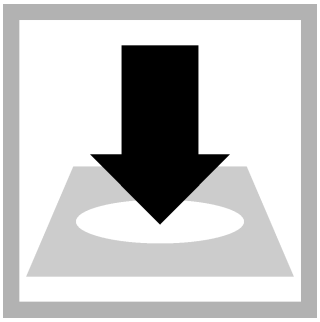


7. Prepare the blank: When the second timer expires, fill a sample cell with 10 mL of sample.

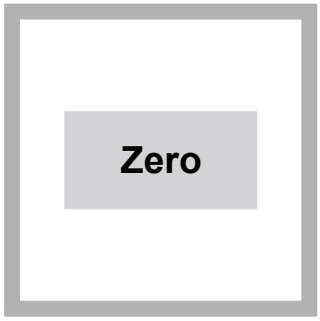


8. Clean the blank sample cell.

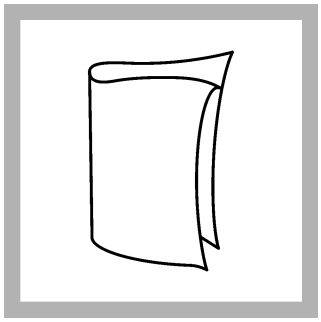
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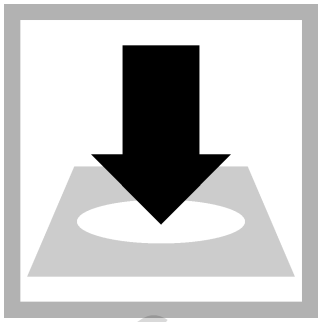
9. Insert the blank into the cell holder.



10. Push **ZERO**. The display shows 0.0 mg/L NO_3^-–N.



11. Clean the AccuVac Ampul.



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



13. Push **READ**. Results show in mg/L NO_3^-–N.

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

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.

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Prepare calibration standards that contain chloride and 1.0, 3.0, 10.0 and 30.0 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, 10 and 30.0 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 calibration into the instrument.
6. Use the user program to measure samples that contain high concentrations of chloride.

Accuracy check

Standard additions method (sample spike)

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

Items to collect:

- 1000 mg/L Nitrate Nitrogen (NO_3^-–N) Standard Solution
- 100-mL volumetric flask, Class A
- 25-mL volumetric pipet, Class A and pipet filler
- Deionized water
- Pipet, TenSette®, 0.1–1.0 mL and tips

1. Prepare a 250 mg/L nitrate-nitrogen standard solution as follows:
   a. Use a pipet to add 25 mL of a 1000 mg/L nitrate nitrogen standard solution into a 100-mL volumetric flask.
   b. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
2. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
3. Go to the Standard Additions option in the instrument menu.
4. Select the values for standard concentration, sample volume and spike volumes.
5. Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the prepared 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 prepared standard solution to three 50-mL portions of fresh sample.*
6. 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.
7. Select **Graph** to compare the expected results to the actual results.
   Note: *If the actual results are significantly different from the expected results, make sure that the sample volumes and sample spikes are measured accurately. The sample volumes and sample spikes that are used should agree with the selections in the standard additions menu. If the results are not within acceptable limits, the sample may contain an interference.*

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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 spectrophotometer during ideal test conditions. Users can get different results under different test conditions.

Program	Standard	Precision (95% Confidence Interval)	Sensitivity Concentration change per 0.010 Abs change
355	10 mg/L NO_3^-–N	9.3–10.7 mg/L NO_3^-–N	0.3 mg/L at 0 ppm, 0.5 mg/L at 10 ppm, 0.8 mg/L at 30 ppm NO_3^-–N
361	10 mg/L NO_3^-–N	9.3–10.7 mg/L NO_3^-–N	0.5 mg/L at 0 ppm, 0.6 mg/L at 10 ppm, 0.8 mg/L at 30 ppm NO_3^-–N

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. The measurement wavelength is 500 nm for spectrophotometers or 520 nm for colorimeters.

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.

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.
Stopper, Neoprene, solid, size #1	2	12/pkg	1480801
OR			
Stoppers for 18-mm tubes and AccuVac Ampuls	2	6/pkg	173106

Required apparatus (AccuVac)

Description	Quantity/test	Unit	Item no.
Beaker, 50 mL	1	each	50041H

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Recommended standards

Description	Unit	Item no.
Nitrate Nitrogen Standard Solution, 10.0-mg/L NO_3–N	500 mL	30749
Nitrate Nitrogen Standard Solution 1000-mg/L NO_3–N	500 mL	1279249
Wastewater Influent Standard Solution, Mixed Parameter, for NH_3-N, NO_3-N, PO_4, COD, SO_4, TOC	500 mL	2833149
Water, deionized	4 L	27256

Optional reagents and apparatus

Description	Unit	Item no.
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, volumetric, Class A, 25 mL	each	1451540
Pipet filler, safety bulb	each	1465100
AccuVac® Ampul Snapper	each	2405200
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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