Nitrate

DOC316.53.01067

Cadmium Reduction Method

Method 8192

0.01 to 0.50 mg/L NO_3^-–N (LR)

Powder Pillows

Scope and application: For water, wastewater and seawater.

Test preparation

Instrument-specific information

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

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

Table 1 Instrument-specific information

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 

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.
UV light changes the color of the prepared sample to yellow. Keep the prepared sample out of direct sunlight.
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.

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Items to collect

Description	Quantity
NitraVer® 6 Nitrate Reagent Powder Pillow, 10-mL	1
NitriVer® 3 Nitrite Reagent Powder Pillow, 10-mL	1
Cylinder, graduated mixing, 25-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 6 for order information.

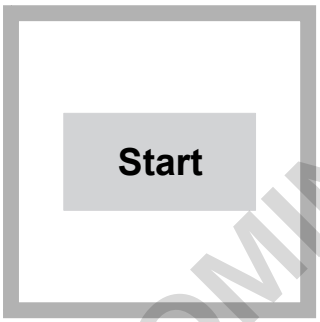
Sample collection and storage

- 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.

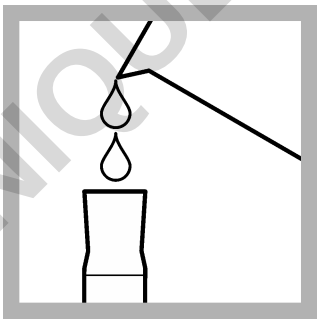
Powder pillow procedure

⚠ CAUTION

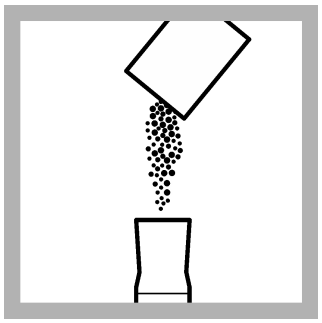
Hazardous waste exposure. Prepared samples contain cadmium. Refer to the SDS for safe handling and disposal instructions. Obey all local and regional disposal regulations.



1. Start program **351 N, Nitrate LR**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.



2. Fill the mixing cylinder with 15 mL of sample.

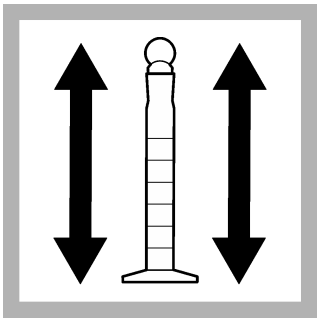


3. Add the contents of one NitraVer 6 Reagent Powder Pillow to the cylinder. Close the cylinder.



4. Start the instrument timer. A 3-minute reaction time starts.

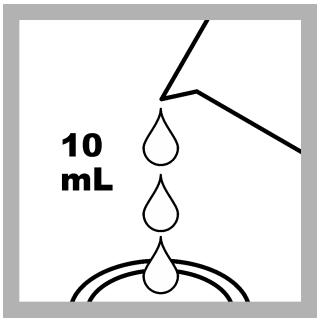
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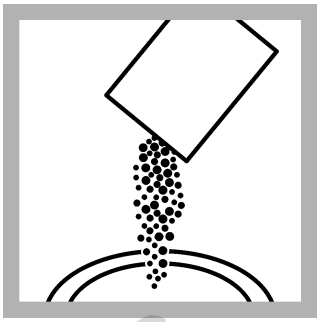
5. Shake the cylinder vigorously during the reaction period. Some powder may not dissolve.



6. When the timer expires, start the timer again. A 2-minute reaction time starts.



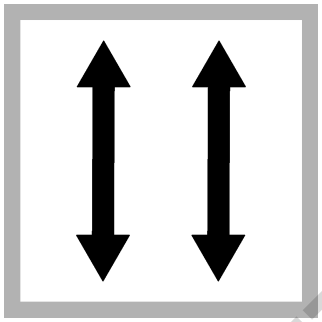
7. Prepare the sample: When the timer expires, carefully pour 10 mL of sample into a sample cell. Do not transfer cadmium particles to the sample cell.



8. Add the contents of one NitriVer 3 Reagent Powder Pillow to the prepared sample cell.



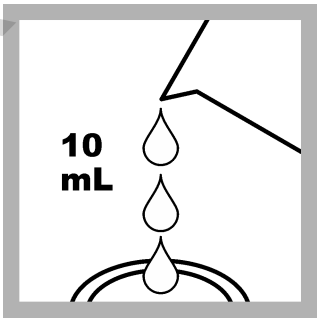
9. Start the instrument timer. A 30-second reaction time starts.



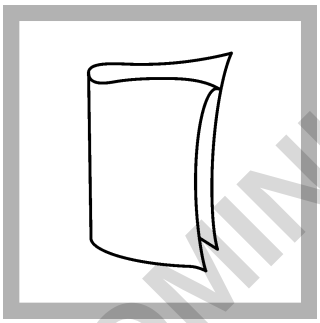
10. Put the stopper on the sample cell. Shake the sample cell gently during the 30-second timer. A pink color shows if nitrate is present in the sample.



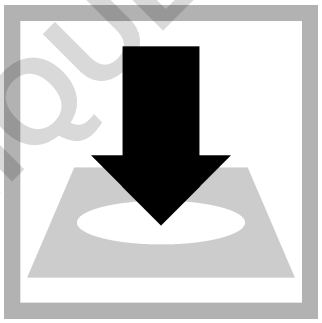
11. Start the instrument timer. A 15-minute reaction time starts.



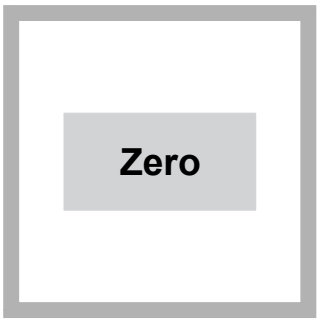
12. Prepare the blank: When the timer expires, fill a second sample cell with 10 mL of the original sample.



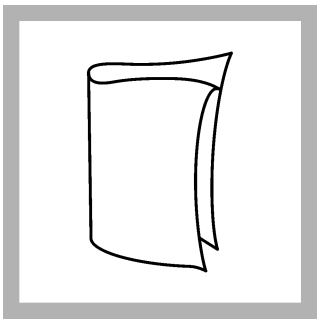
13. Clean the blank sample cell.



14. Insert the blank into the cell holder.

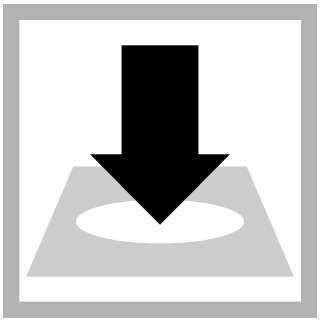


15. Push **ZERO**. The display shows 0.00 mg/L NO_3^-–N.



16. Clean the prepared sample cell.

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17. Insert the prepared sample into the cell holder.



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

Interferences

Interfering substance	Interference level
Calcium	100 mg/L
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 4).
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
Interference from direct sunlight	UV light changes the color of the prepared sample to yellow. Keep the prepared sample out of direct sunlight.

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 0.05, 0.1, 0.25 and 0.50 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 0.5, 1.0, 2.5 and 5.0 mL of a 10 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.

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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:

- Nitrate Nitrogen Standard Solution, 100-mg/L NO_3^-–N
- 50-mL volumetric flask, Class A
- 6-mL volumetric pipet, Class A and pipet filler
- Deionized water
- Pipet, TenSette®, 0.1–1.0 mL and tips
- Mixing cylinders, 25 mL (3)

1. Prepare a 12 mg/L nitrate nitrogen standard solution as follows:
   a. Use a pipet to add 6.0 mL of a 100 mg/L NO_3^-–N standard solution into a 50-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 15-mL portions of fresh sample. Mix well.
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.*

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-mg/L NO_3^-–N
- 100-mL volumetric flask, Class A
- 4-mL volumetric pipet, Class A and pipet filler
- Deionized water

1. Prepare a 0.40 mg/L nitrate nitrogen standard solution as follows:
   a. Use a pipet to add 4.00 mL of 10 mg/L nitrate nitrogen standard solution into the volumetric flask.
   b. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.

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2. Use the test procedure to measure the concentration of the prepared standard solution.
3. 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
351	0.40 mg/L NO_3^-–N	0.35–0.45 mg/L NO_3^-–N	0.003 mg/L 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 chromotropic acid to form a pink-colored product. The measurement wavelength is 507 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.
Nitrate Reagent Set, low range, 10 mL	1	100/pkg	2429800
Includes:			
NitraVer® 6 Nitrate Reagent Powder Pillow, 10 mL	1	100/pkg	2107249
NitriVer® 3 Nitrite Reagent Powder Pillow, 10 mL	1	100/pkg	2107169

Required apparatus

Description	Quantity/test	Unit	Item no.
Mixing cylinder, graduated, 25 mL with stopper	1	each	2088640
Stoppers for 18-mm tubes and AccuVac Ampuls	2	6/pkg	173106

Recommended standards and apparatus

Description	Unit	Item no.
Flask, volumetric, Class A, 100 mL, glass	each	1457442
Nitrate Nitrogen Standard Solution, 10.0-mg/L NO_3–N	500 mL	30749
Nitrate Nitrogen Standard Solution, 100-mg/L NO_3–N	500 mL	194749
Water, deionized	4 L	27256

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Optional reagents and apparatus

Description	Unit	Item no.
Bromine Water, 30 g/L	29 mL	221120
Pipet, TenSette®, 0.1–1.0 mL	each	1970001
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
Pipet, volumetric, Class A, 4.00 mL	each	1451504
Flask, volumetric, 50 mL	each	1457441
Pipet filler, safety bulb	each	1465100
Phenol Solution, 30-g/L	29 mL	211220
Sodium Hydroxide Standard Solution, 5.0 N	1 L	245053
Sulfuric Acid, concentrated, ACS	500 mL	97949
Sodium Chloride, ACS	454 g	18201H
Pipet, TenSette®, 1.0–10.0 mL	each	1970010
Pipet tips for TenSette® Pipet, 1.0–10.0 mL	50/pkg	2199796

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