

Cadmium Reduction Method

Method 8171

MR (0.1 to 10.0 mg/L NO<sub>3</sub><sup>-</sup>-N)

Powder Pillows or AccuVac® Ampuls

Scope and Application: For water, wastewater and seawater



## Test preparation

## How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

**Table 1 Instrument-specific information**

Instrument	Powder pillows		AccuVac Ampuls	
	Sample cell	Cell orientation	Sample cell	Adapter
DR 5000	2495402	Fill line faces user	2427606	—
DR 3900	2495402	Fill line faces user	2427606	LZV846 (A)
DR 3800, DR 2800, DR 2700	2495402	Fill line faces right	2122800	LZV584 (C)

### Before starting the test:

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample.

A deposit of unoxidized metal will remain at the bottom of the cell after the NitraVer® 5 dissolves. The deposit will not affect results.

This method is technique-sensitive. Shaking time and technique influence color development. For most accurate results, make successive tests on a 10.0-mg/L Nitrate Nitrogen Standard solution. Adjust shaking times to obtain the correct result.

Rinse the sample cell immediately after use to remove all cadmium particles. Retain the used sample for proper hazardous waste disposal for cadmium.

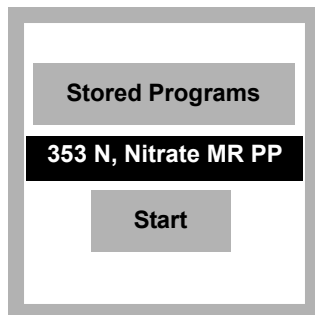
Prepared samples will contain cadmium and must be disposed of according to Federal, State and local hazardous waste regulations. Refer to the current MSDS for safe handling and disposal instructions.

## Collect the following items:

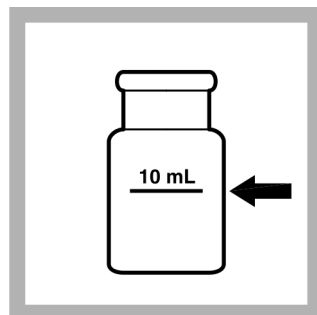
Description	Quantity
Powder Pillow Test:	
NitraVer® 5 Nitrate Reagent Powder Pillow	1
Sample Cells (see <a href="#">Instrument-specific information</a> )	2
Stopper, Neoprene, #1, solid	2
AccuVac Test:	
NitraVer® 5 Nitrate Reagent AccuVac® Ampul	1
Beaker, 50-mL	1
Sample Cell for blank (see <a href="#">Instrument-specific information</a> )	1

See [Consumables and replacement items](#) for reorder information.

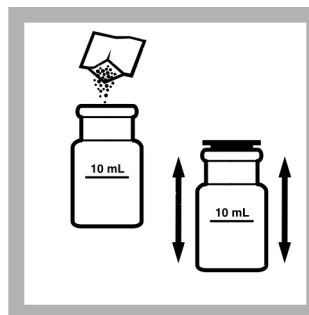
## Cadmium reduction method for powder pillows



1. Select the test.  
Insert an adapter if required (see [Instrument-specific information](#)).  
Refer to the user manual for orientation.



2. Fill a sample cell with 10 mL of sample.



3. **Prepared Sample:**  
Add the contents of one NitraVer 5 Nitrate Reagent Powder Pillow. Insert a stopper into the cell.



4. Start the instrument timer.  
A one-minute reaction period will begin.  
Shake the cell vigorously until the timer expires.  
**Note:** Some solid material will not dissolve.

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**Cadmium reduction method for powder pillows (continued)**

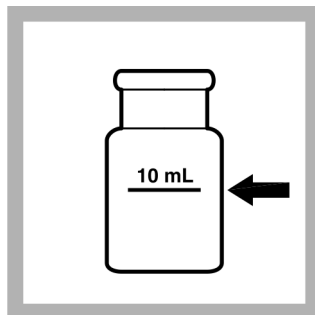
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5. When the timer expires, start the timer again.

A five-minute reaction period will begin.

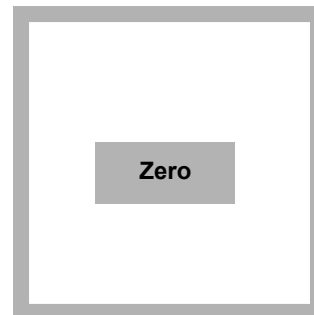
An amber color will develop if nitrate is present.



6. **Blank Preparation:** When the timer expires, fill a second sample cell with 10 mL of sample.



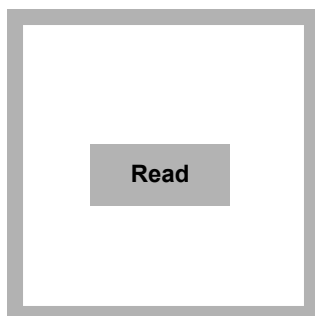
7. Wipe and insert the blank into the cell holder.



8. **ZERO** the instrument. The display will show: 0.0 mg/L NO<sub>3</sub><sup>-</sup>-N

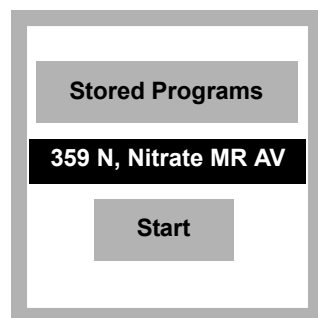


9. Within two minutes after the timer expires, wipe and insert the prepared sample into the cell holder.

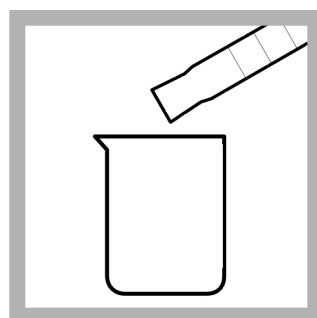


10. **READ** the results in mg/L NO<sub>3</sub><sup>-</sup>-N. Refer to the user manual to display other chemical forms.

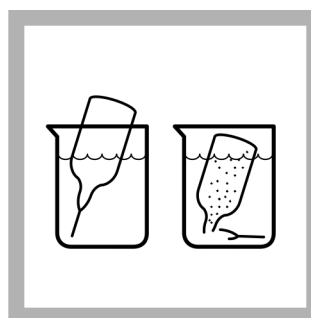
## Cadmium reduction method for AccuVac® Ampuls



1. Select the test.  
Insert an adapter if required (see [Instrument-specific information](#)).  
Refer to the user manual for orientation.



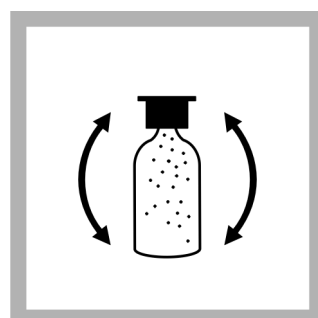
2. **Prepared Sample:**  
Collect at least 40 mL of sample in a 50-mL beaker.



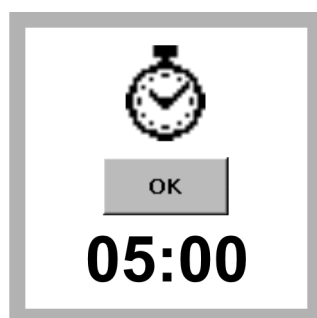
3. Fill a NitraVer 5 Nitrate AccuVac® Ampul with sample. Keep the tip immersed while the Ampul fills completely. Place a stopper over the Ampul tip.



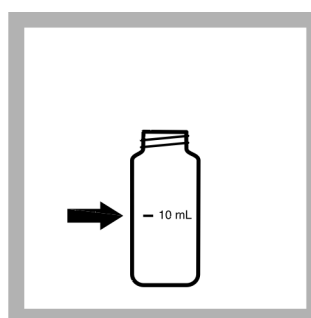
4. Start the instrument timer.  
A one-minute reaction period will begin.



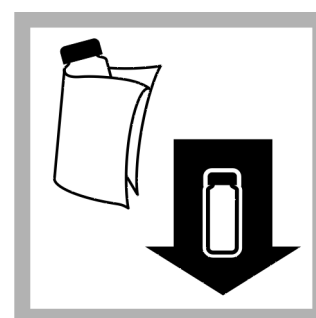
5. Invert the Ampul 48–52 times as the timer counts down.



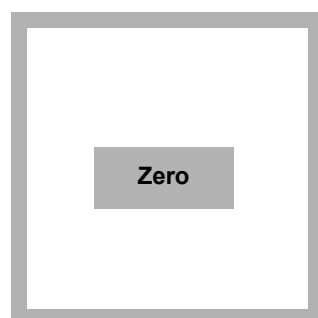
6. When the timer expires, start the timer again.  
A five-minute reaction period will begin. An amber color will develop if nitrate is present.



7. **Blank Preparation:**  
When the timer expires, fill a round sample cell with 10 mL of sample.



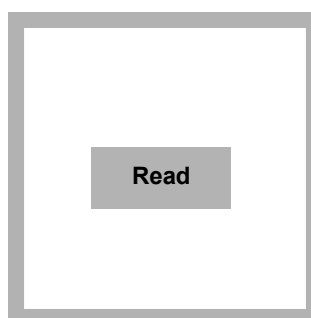
8. Wipe the blank and insert it into the cell holder.



9. **ZERO** the instrument.  
The display will show:  
0.0 mg/L NO<sub>3</sub><sup>-</sup>-N



10. Within two minutes after the timer expires, wipe the Ampul and insert it into the cell holder.



11. **READ** the results in mg/L NO<sub>3</sub><sup>-</sup>-N.

## Interferences

**Table 2 Interfering substances**

Interfering substance	Interference level
Chloride	Chloride concentrations above 100 mg/L will cause low results. The test may be used at high chloride concentrations (seawater) but a calibration must be done using standards spiked to the same chloride concentration (see <a href="#">Seawater calibration</a> ).
Ferric iron	Interferes at all levels
Nitrite	Interferes at all levels Compensate for nitrite interference as follows: <ol style="list-style-type: none"> <li>1. Add 30-g/L Bromine Water<sup>1</sup> drop-wise to the sample until a yellow color remains.</li> <li>2. Add one drop of 30-g/L Phenol Solution<sup>1</sup> to destroy the color.</li> <li>3. Proceed with Step 2 of the test. Report the results as total nitrate and nitrite.</li> </ol>
pH	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.
Strong oxidizing and reducing substances	Interfere at all levels

<sup>1</sup> See [Optional reagents and apparatus](#).

## Seawater calibration

Chloride concentrations above 100 mg/L will cause low results. To perform this test in water with high interference level, calibrate the water using standards spiked to the same chloride concentrations as the required samples. To prepare calibration standards containing 1.0, 3.0, 5.0 and 10.0 mg/L nitrate as NO<sub>3</sub>-N:

1. Prepare a 1 L volume of chloride water that matches the concentration of the samples, using the following equation:
  - a. Add necessary Chloride concentration (g/L) x (1.6485) = g of ACS grade NaCl to 1 L of deionized water.

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

  - b. Mix this solution thoroughly to make sure that it is a homogeneous solution. Use this water as the dilution water instead of the deionized water when preparing the nitrate standards.
2. Use Class A glassware or a Tensette Pipet to pipet 1.0, 3.0, 5.0, and 10.0 mL of the 100 mg/L Nitrogen-Nitrate as NO<sub>3</sub><sup>-</sup>-N (NIST) Standard Solution (Catalog Number 194749) into four different 100 mL Class A volumetric flasks.
3. Dilute to the mark with the prepared chloride water. Mix thoroughly.
4. Use the prepared chloride water for the 0-mg/L nitrate as NO<sub>3</sub>-N standard.

## Sample collection, preservation and storage

- Most reliable results are obtained when samples are analyzed as soon as possible after collection. If prompt analysis is impossible, store samples in clean plastic or glass bottles for up to 24 hours at 4 °C. To preserve samples for longer periods, add 2 mL of Concentrated Sulfuric Acid (H<sub>2</sub>SO<sub>4</sub>)\* per liter and store at 4 °C. The results are reported as total nitrate and nitrite.
- Before analysis, warm the sample to room temperature and adjust the pH to 7 with 5.0 N Sodium Hydroxide Standard Solution\*. Do not use mercury compounds as preservatives.

\* See [Optional reagents and apparatus](#).

- Correct the test result for volume additions by dividing the total volume (acid + base + sample) by the original sample volume and multiplying the test result by this factor.

### Accuracy check

#### Standard additions method (sample spike)

Required for accuracy check:

- Nitrate Nitrogen Standard, 100-mg/L  $\text{NO}_3^-$ -N
  - TenSette Pipet and Pipet Tips
1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
  2. Select **Options>More>Standard Additions** from the instrument menu.
  3. Accept the default values for standard concentration, sample volume and spike volumes. After the values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
  4. Open the standard solution bottle.
  5. Use the TenSette Pipet to prepare spiked samples: add 0.1 mL, 0.2 mL and 0.3 mL of standard to three 10-mL portions of fresh sample.
  6. Follow the [Cadmium reduction method for powder pillows](#) test procedure for each of the spiked samples, starting with the 0.1 mL sample spike. Measure each of the spiked samples in the instrument.
  7. Select **GRAPH** to view the results. Select **IDEAL LINE** (or best-fit) to compare the standard addition results to the theoretical 100% recovery.

#### Standard additions method for AccuVac Ampuls (sample spike)

Required for accuracy check:

- 500 mg/L Nitrate Nitrogen Ampule Standard Solution
  - Ampule breaker
  - TenSette Pipet and Pipet Tips
  - Mixing cylinder, 50-mL (3)
1. Fill three mixing cylinders each with 50-mL of sample and spike with 0.1 mL, 0.2 mL and 0.3 mL of 500 mg/L Nitrate Nitrogen Ampule Standard Solution.
  2. Transfer 40 mL from each of the three mixing cylinders to three 50-mL beakers.
  3. Analyze each standard addition sample as described in the [Cadmium reduction method for AccuVac® Ampuls](#).
  4. Accept each standard additions reading. Each addition should reflect approximately 100% recovery. Standard solution method

*Note: Refer to the instrument user manual for specific software navigation instructions.*

#### Standard Solution Method

Required for accuracy check:

- 5.0-mg/L Nitrate Nitrogen Standard Solution (prepared)
- 100-mg/L Nitrate Nitrogen Standard
- Deionized water
- 100-mL volumetric flask

- 5-mL Volumetric pipet and pipet filler
- OR
- TenSette Pipet and Pipet Tips
1. Prepare a 5.0-mg/L nitrate nitrogen standard solution as follows:
    - a. Pipet 5.0 mL of 100-mg/L Nitrate Nitrogen Standard, into a 100-mL volumetric flask.
    - b. Dilute to the mark with deionized water. Mix well.
  2. Use this 5.0 mg/L nitrate nitrogen standard solution in place of the sample. Follow the [Cadmium reduction method for powder pillows](#) test procedure.
  3. To adjust the calibration curve using the reading obtained with the standard solution, select **Options>More>Standard Adjust** from the instrument menu.
  4. Turn on the Standard Adjust feature and accept the displayed concentration. If an alternate concentration is used, enter the concentration and adjust the curve to that value.

## Method performance

Program	Standard	Precision 95% Confidence Limits of Distribution	Sensitivity Concentration change per 0.010 Abs change
353	5.0 mg/L NO <sub>3</sub> <sup>-</sup> -N	4.8–5.2 mg/L NO <sub>3</sub> <sup>-</sup> -N	0.04 mg/L NO <sub>3</sub> <sup>-</sup> -N
359	5.0 mg/L NO <sub>3</sub> <sup>-</sup> -N	4.6–5.4 mg/L NO <sub>3</sub> <sup>-</sup> -N	0.05 mg/L NO <sub>3</sub> <sup>-</sup> -N

## Summary of method

Cadmium metal reduces nitrates 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. Test results are measured at 400 nm.

## Consumables and replacement items

### Required reagents

Description	Quantity/Test	Unit	Catalog number
NitraVer® 5 Nitrate Reagent Powder Pillows (for 10 mL sample)	1	100/pkg	2106169
OR			
NitraVer® 5 Nitrate Reagent AccuVac® Ampul	1	25/pkg	2511025

### Required apparatus (powder pillows)

Description	Quantity	Unit	Catalog number
Sample cell, 10 mL square, matched pair	2	2/pkg	2495402
Stopper, Neoprene, solid, size #1	2	12/pkg	1480801

# Nitrate

## Required apparatus (AccuVac)

Description	Quantity	Unit	Catalog number
Beaker, 50-mL	1	each	50041H
Sample cell, 10 mL round, 25 x 54 mm	1	each	2122800
Sample cell, 10 mL round, 25 x 60 mm	1	6/pkg	2427606

## Recommended standards

Description	Unit	Catalog number
Mixed Parameter Drinking Water Standard, for F, NO <sub>3</sub> -N, PO <sub>4</sub> , SO <sub>4</sub>	500 mL	2833049
Nitrate Nitrogen Standard Solution, 100-mg/L NO <sub>3</sub> <sup>-</sup> -N	500 mL	194749
Nitrate Nitrogen Standard Solution, 500 mg/L NO <sub>3</sub> -N, 10-mL ampules	16/pkg	1426010
Water, deionized	4 L	27256

## Optional reagents and apparatus

Description	Unit	Catalog number
Ampule breaker for 10 mL ampules	each	2196800
Bromine Water, 30-mg/L	29 mL	221120
Cylinder, mixing, 50 mL	each	2088641
Flask, volumetric, 100-mL	each	1457442
Pipet, TenSette, 0.1–1.0 mL	each	1970001
Pipet Tips, for TenSette Pipet 1970001	50/pkg	2185696
Pipet Tips, for TenSette Pipet 1970001	1000/pkg	2185628
Pipet, volumetric, Class A, 5.0 mL	each	1451537
Pipet, volumetric, Class A, 1.0 mL	each	1451535
Pipet, volumetric, Class A, 3.0 mL	each	1451503
Pipet, volumetric, Class A, 10.0 mL	each	1451538
Pipet Filler, safety bulb	each	1465100
Phenol Solution, 30-g/L	29 mL	211220
5.0 N Sodium Hydroxide Standard Solution	1 L	245053
Sulfuric Acid, concentrated	500 mL	97949



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