

Cadmium Reduction Method

Method 8039

HR (0.3 to 30.0 mg/L NO₃⁻-N)

Powder Pillow 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. Subtract the reagent blank value from the final results or perform a reagent blank adjust.

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-mg/L Nitrate Nitrogen Standard solution. Adjust shaking time and technique to obtain the correct result. Use this technique for all the samples.

Rinse the sample cell immediately after use to remove all cadmium particles. 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:

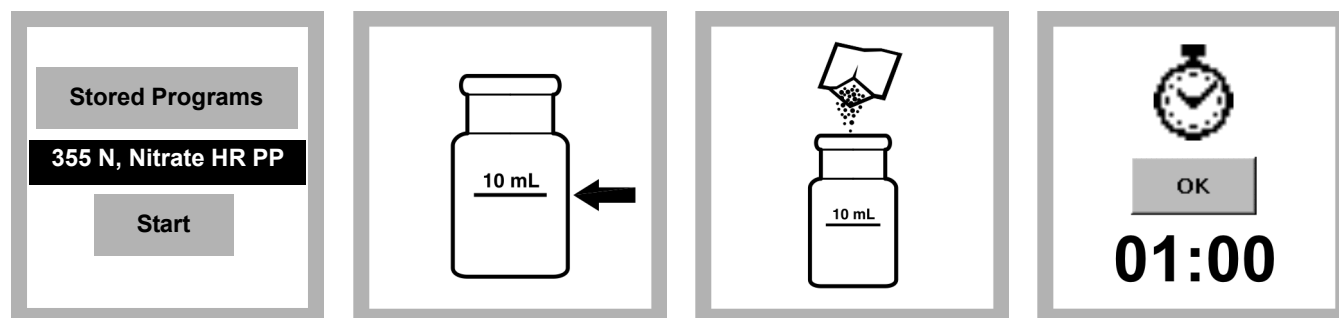
| Description | Quantity |
|--------------------------------------------|----------|
| Powder Pillow Test: | |
| NitraVer® 5 Nitrate Reagent Powder Pillow | 1 |
| Sample Cells, 1-inch, 10-mL, with stopper | 2 |
| AccuVac Test: | |
| NitraVer® 5 Nitrate Reagent AccuVac® Ampul | 1 |
| Beaker, 50-mL | 1 |
| Sample Cell, for blank | 1 |

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

Cadmium Reduction Method for powder pillows

CAUTION

Hazardous waste exposure. Prepared samples contain cadmium. Refer to the current MSDS for safe handling and disposal instructions. Follow all federal, state and local hazardous waste disposal regulations.



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.

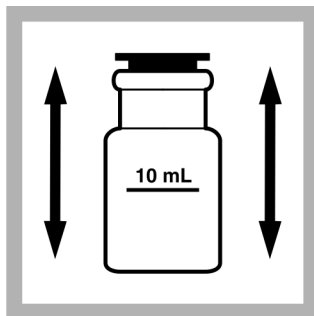
3. Prepared Sample:
Add the contents of one NitraVer 5 Nitrate Reagent Powder Pillow. Stopper.

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

Cadmium Reduction Method for powder pillows (continued)

CAUTION

Hazardous waste exposure. Prepared samples contain cadmium. Refer to the current MSDS for safe handling and disposal instructions. Follow all federal, state and local hazardous waste disposal regulations.



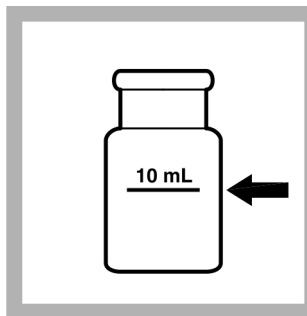
5. Shake the cell vigorously until the timer expires.

Note: Some solid materials may not dissolve.



6. When the timer expires, start the timer again. A five-minute reaction period will begin.

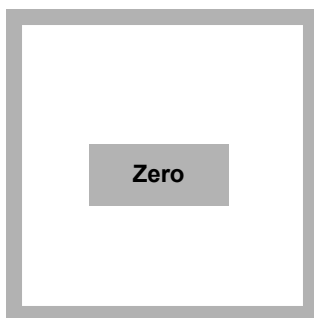
An amber color will develop if nitrate is present.



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



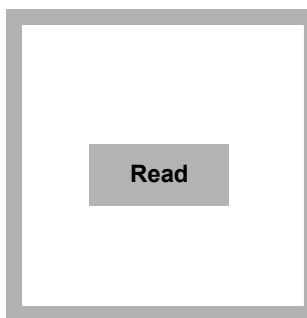
8. Wipe the blank and insert it into the cell holder.



9. ZERO the instrument.
The display will show:
0.0 mg/L NO₃⁻-N



10. Within one minute after the timer expires, wipe the prepared sample and insert it into the cell holder.



11. READ the results in mg/L NO₃⁻-N.
To display other chemical forms, refer to the user manual.

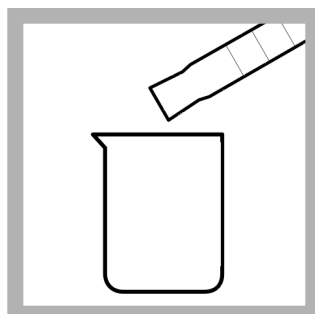
Cadmium Reduction Method for AccuVac® Ampuls

CAUTION

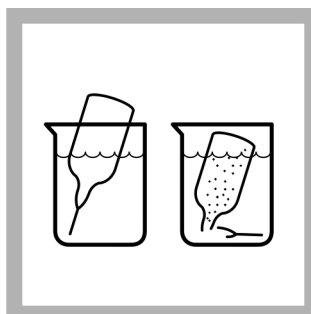
Hazardous waste exposure. Prepared samples contain cadmium. Refer to the current MSDS for safe handling and disposal instructions. Follow all federal, state and local hazardous waste disposal regulations.



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. Tap the bottom of a NitraVer 5 Nitrate AccuVac® Ampul on a hard surface to dislodge powder. Fill the Ampul with sample. Keep the tip immersed while the Ampul fills completely. Insert a cap over the Ampul tip.



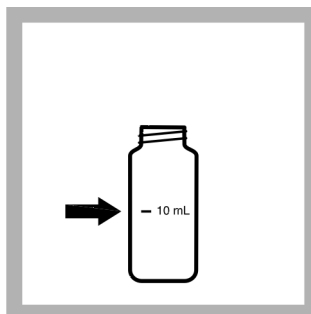
4. Start the instrument timer.
A one-minute reaction period will begin.



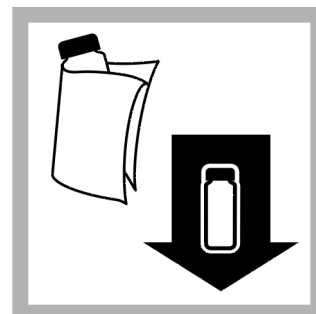
5. Invert the Ampul 48–52 times as the timer counts down.



6. When the timer expires, start the timer again. A five-minute reaction period will begin. Do not agitate or disturb the sample during this time.
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.

Cadmium Reduction Method for AccuVac® Ampuls (continued)

CAUTION

Hazardous waste exposure. Prepared samples contain cadmium. Refer to the current MSDS for safe handling and disposal instructions. Follow all federal, state and local hazardous waste disposal regulations.



9. ZERO the instrument.

The display will show:

0.0 mg/L NO_3^- -N

10. Within one minute after the timer expires, wipe the Ampul and insert into the cell holder.

11. READ the results in mg/L NO_3^- -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. Refer to Seawater calibration . |
| Ferric iron | Interferes at all levels |
| Nitrite | Interferes at all levels Compensate for nitrite interference as follows: Before performing step 3, add 30-g/L Bromine Water ¹ dropwise to the sample until a yellow color remains. Add one drop of 30-g/L Phenol Solution ¹ to destroy the color. Proceed with step 3. Report the results as total nitrate and nitrite. |
| 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 |

¹ 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 5.0, 10.0, 20.0 and 30.0 mg/L nitrate as NO_3^- -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. 18.8 g/L is a typical seawater chloride concentration.
 - b. Mix this solution thoroughly to get 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 0.5, 1.0, 2.0, and 3.0 mL of the 1000 mg/L Nitrogen-Nitrate as NO_3^- -N (NIST) Standard Solution (Catalog Number 1279249) 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_3^- -N standard.

Sample collection, preservation and storage

- More 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_2SO_4)* per liter and store at 4 °C. The results are reported as total nitrate plus 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.
- 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 for powder pillows (sample spike)

Required for accuracy check:

- Nitrate Nitrogen Standard solution, 1000-mg/L NO_3^- -N
 - Mixing cylinders
 - 25-mL Volumetric pipet
 - TenSette Pipet and Pipet Tips
 - Pipet filler
 - 100 mL Volumetric Flask
1. Prepare a 250-mL nitrate nitrogen standard solution by pipetting 25 mL of a 1000 mg/L Nitrate Nitrogen standard solution into a 100 mL volumetric flask. Dilute the solution to the required volume with deionized water and mix thoroughly.
 2. After reading test results, leave the sample cell (unspiked sample) in the instrument.
 3. Select **Options>More>Standard Additions** from the instrument menu.

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

- 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.
- 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.
- Follow the [Cadmium Reduction Method for powder pillows](#) test procedure for each of the spiked samples using the powder pillows, starting with the 0.1 mL sample spike. Measure each of the spiked samples in the instrument.
- 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)

- Fill three mixing cylinders each with 50-mL of sample and spike with 0.4 mL, 0.8 mL and 1.2 mL of 250-mg/L standard.
- Transfer 40 mL from each of the three mixing cylinders to three 50-mL beakers.
- Analyze each standard addition sample as described in the [Cadmium Reduction Method for AccuVac® Ampuls](#).
- 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.

Required for accuracy check:

- 10.0-mg/L Nitrate Nitrogen Standard Solution
- Use the 10.0-mg/L Nitrate Nitrogen Standard Solution in place of the sample. Follow the [Cadmium Reduction Method for powder pillows](#) and [Cadmium Reduction Method for AccuVac® Ampuls](#) test procedures.
 - To adjust the calibration curve using the reading obtained with the standard solution, select **Options>More>Standard Adjust** from the instrument menu.
 - 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 | |
|---------|-----------------------------------------|-------------------------------------------------------|-------------------------------------------------------------|------------------------------------------|
| | | | Point of curve | Concentration |
| 355 | 10 mg/L NO ₃ ⁻ -N | 9.3–10.7 mg/L NO ₃ ⁻ -N | 0 ppm | 0.3 mg/L NO ₃ ⁻ -N |
| | | | 10 ppm | 0.5 mg/L NO ₃ ⁻ -N |
| | | | 30 ppm | 0.8 mg/L NO ₃ ⁻ -N |
| 361 | 10 mg/L NO ₃ ⁻ -N | 9.3–10.7 mg/L NO ₃ ⁻ -N | 0 ppm | 0.5 mg/L NO ₃ ⁻ -N |
| | | | 10 ppm | 0.6 mg/L NO ₃ ⁻ -N |
| | | | 30 ppm | 0.8 mg/L NO ₃ ⁻ -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 500 nm.

Consumables and replacement items

Required reagents

| Description | Quantity/Test | Unit | Catalog number |
|---------------------------------------------------------------------|---------------|---------|----------------|
| NitraVer® 5 Nitrate Reagent Powder Pillows (for 10-mL sample) OR | 1 | 100/pkg | 2106169 |
| 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 or | 2 | 12/pkg | 1480801 |
| Stopper | 2 | 6/pkg | 173106 |

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 |
|-----------------------------------------------------------------------------------------------------------------------------------------|--------|----------------|
| 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, 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 | Catalog number |
|-----------------------------------------|--------|----------------|
| Bromine Water, 30 g/L | 29 mL | 221120 |
| Cylinder, mixing, 50 mL | each | 2088641 |
| Pipet, TenSette®, 0.1 to 1.0 mL | each | 1970001 |
| Pipet, Volumetric, Class A, 0.5 mL | each | 1451534 |
| Pipet, Volumetric, Class A, 1.0 mL | each | 1451535 |
| Pipet, Volumetric, Class A, 2.0 mL | each | 1451536 |
| Pipet, Volumetric, Class A, 3.0 mL | each | 1451503 |
| Pipet Tips, for TenSette Pipet 19700-01 | 50/pkg | 2185696 |

Optional reagents and apparatus (continued)

| Description | Unit | Catalog number |
|-------------------------------------------|-------------|-----------------------|
| Pipet Tips, for TenSette Pipet 19700-01 | 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 Snapper | each | 2405200 |
| Sodium Hydroxide Standard Solution, 5.0 N | 50 mL SCDB | 245026 |
| Sulfuric Acid, concentrated, ACS | 500 mL | 97949 |
| Flask, volumetric, Class A, 100 mL | each | 1457442 |



FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:

In the U.S.A. – Call toll-free 800-227-4224

Outside the U.S.A. – Contact the HACH office or distributor serving you.

On the Worldwide Web – www.hach.com; E-mail – techhelp@hach.com

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