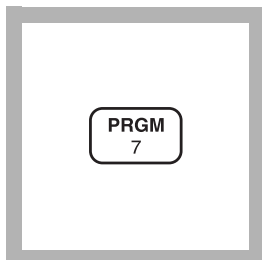


# NITRATE, High Range (0 to 30.0 mg/L $\text{NO}_3^-$ -N) For water, wastewater, and seawater\*

## Cadmium Reduction Method (Using Powder Pillows or AccuVac Ampuls)

### Using Powder Pillows



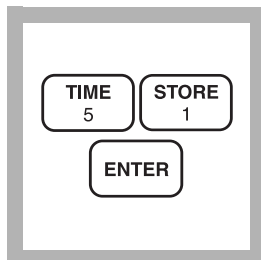
1. Enter the stored program number for high range nitrate nitrogen ( $\text{NO}_3^-$ -N) powder pillows.

Press: **PRGM**

The display will show:

**PRGM ?**

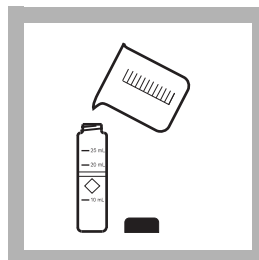
*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **51 ENTER**

The display will show **mg/L,  $\text{NO}_3$ -N** and the **ZERO** icon.

*Note: For alternate forms ( $\text{NO}_3$ ), press the **CONC** key.*



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

*Note: Adjust the pH of stored samples before analysis.*

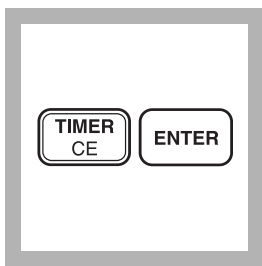


4. Add the contents of one NitraVer 5 Nitrate Reagent Powder Pillow to the sample cell (the prepared sample). Cap the sample cell.

*Note: It is important to remove all of the powder from the foil pillow. Tap the pillow until no more powder pours out.*

\* Seawater requires a manual calibration; see Interferences.

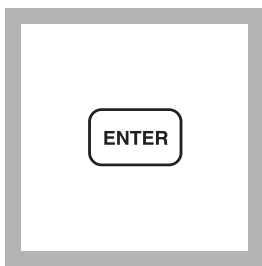
## NITRATE, High Range, continued



5. Press:  
**TIMER ENTER**

A one-minute reaction period will begin. Shake the sample cell vigorously until the timer beeps.

***Note:** It is important to shake the cell vigorously. Shaking time and technique influence color development. For most accurate results, do successive tests on a standard solution and adjust the shaking time to obtain the correct result.*



6. After the timer beeps, the display will show:  
**5:00 TIMER 2**

Press: **ENTER**

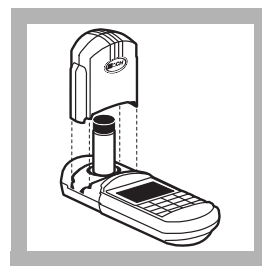
A five-minute reaction period will begin.

***Note:** A deposit will remain after the reagent dissolves and will not affect test results.*

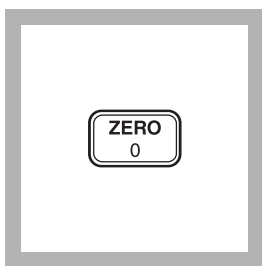
***Note:** An amber color will develop if nitrate nitrogen is present.*



7. Fill another cell with 10 mL of sample (the blank). Wipe off any fingerprints or liquid.



8. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

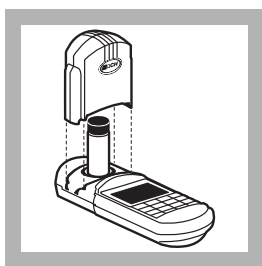


9. When the timer beeps, press **ZERO**.

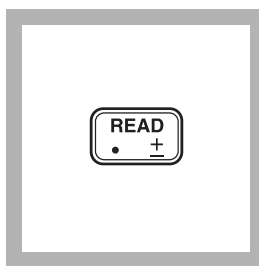
The cursor will move to the right, then the display will show:

**0.0 mg/L NO<sub>3</sub>-N**

***Note:** If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



10. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



11. Press: **READ**

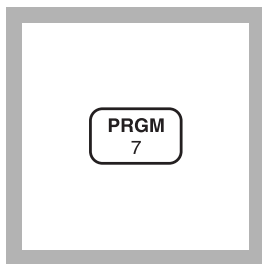
The cursor will move to the right, then the result in mg/L NO<sub>3</sub>-N (or alternate form) will be displayed.

***Note:** Use of the Standard Adjust feature for each new lot of reagent is highly recommended. See Accuracy Check.*

***Note:** Rinse the sample cell immediately after use to remove all cadmium particles. Save the spent sample for proper hazardous waste disposal for cadmium.*

# NITRATE, High Range, continued

## Using AccuVac Ampuls



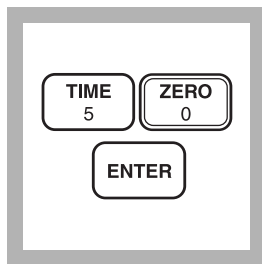
1. Enter the stored program number for high range nitrate nitrogen ( $\text{NO}_3^- - \text{N}$ ) AccuVac Ampuls.

Press: **PRGM**

The display will show:

**PRGM ?**

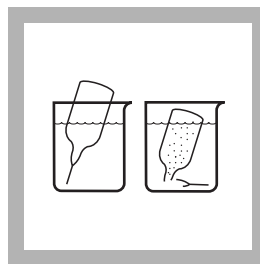
*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **50 ENTER**

The display will show **mg/L,  $\text{NO}_3 - \text{N}$**  and the **ZERO** icon.

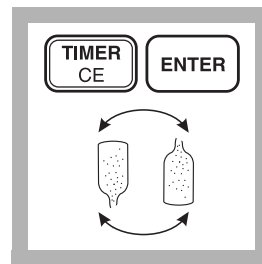
*Note: For alternate forms ( $\text{NO}_3$ ), press the **CONC** key.*



3. Collect at least 40 mL of sample in a 50-mL beaker. Fill a NitraVer 5 Nitrate AccuVac Ampul with sample. Place a stopper over the tip of the ampul.

*Note: Keep the tip immersed while the ampul fills. The ampul will not fill completely.*

*Note: Adjust the pH of stored samples before analysis.*

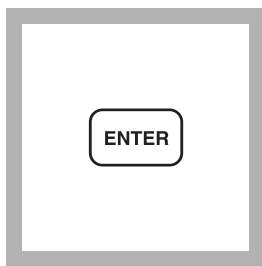


4. Press:

**TIMER ENTER**

A one-minute mixing period will begin. Invert the ampul repeatedly back and forth until the timer beeps. Wipe off any liquid or fingerprints.

*Note: Mixing time and technique influence color development. For most accurate results, do successive tests on a standard solution and adjust the mixing time to obtain the correct result.*



5. The display will show: **5:00 TIMER 2**

Press: **ENTER**

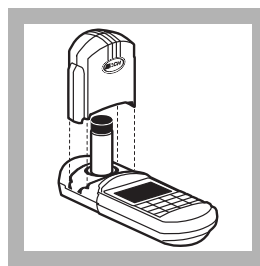
A five-minute reaction period will begin.

*Note: A deposit will remain after the reagent dissolves and will not affect results.*

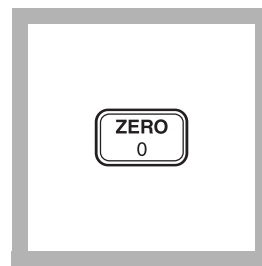
*Note: An amber color will develop if nitrate nitrogen is present.*



6. Fill a sample cell with at least 10 mL of sample (the blank).



7. When the timer beeps, place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.



8. Press: **ZERO**

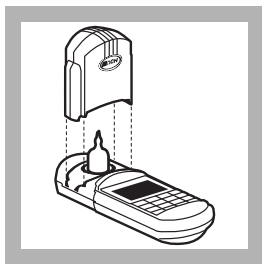
The cursor will move to the right, then the display will show:

**0.0 mg/L  $\text{NO}_3 - \text{N}$**

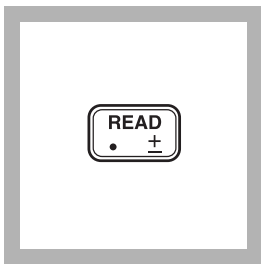
*Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*

## NITRATE, High Range, continued

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**9.** Place the AccuVac Ampul into the cell holder. Tightly cover the ampul with the instrument cap.



**10.** Press: **READ**

The cursor will move to the right, then the result in mg/L  $\text{NO}_3\text{-N}$  (or alternate form) will be displayed.

***Note:** Use of the Standard Adjust feature for each new lot of reagent is highly recommended. See Accuracy Check.*

***Note:** See Pollution Prevention and Waste Management for proper disposal of cadmium.*

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## Sampling and Storage

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. For longer storage periods, adjust sample pH to 2 or less with sulfuric acid, ACS (about 2 mL per liter). Sample refrigeration is still required.

Before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution.

Do not use mercury compounds as preservatives.

Correct the test result for volume additions; see *Correction for Volume Additions (Section 1)* for more information.

## Accuracy Check

### Standard Additions Method

- a) Fill three 25-mL mixing cylinders with 25 mL of sample.
- b) Snap the neck off a Nitrate Nitrogen Ampule Standard, 500 mg/L nitrate nitrogen.
- c) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of Nitrate Nitrogen Standard Solution to the three samples. Stopper and mix thoroughly.
- d) For AccuVac analysis, transfer the solutions to clean, dry 50-mL beakers. For analysis with powder pillows, transfer only 10 mL of solution to clean, dry sample cells.
- e) Analyze each sample as described above. The nitrate nitrogen ( $\text{NO}_3^-$ -N) concentration should increase 2.0 mg/L for each 0.1 mL of standard added.
- f) If these increases do not occur, see *Standard Additions (Section 1)* for more information.

### Standard Solution Method

Use a Hach Nitrate-Nitrogen Standard Solution, 10.0 mg/L  $\text{NO}_3^-$ -N, listed under Optional Reagents as the sample and perform the procedure as described above.

### Standard Adjust

To adjust the calibration curve using the reading obtained with the 10.0-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **10.0** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the curve adjustment. See *Section 1, Standard Curve Adjustment* for more information. If you are using a reagent blank correction, the blank correction should be entered before the Standard Adjust value is entered.

## Method Performance

### Precision

In a single laboratory using standard solutions of 25.0 mg/L nitrate nitrogen ( $\text{NO}_3^-$ -N) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.3$  mg/L nitrate nitrogen for program #50 and  $\pm 1.7$  mg/L nitrate nitrogen for program # 51.

## NITRATE, High Range, continued

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### Estimated Detection Limit

The estimated detection limit for program 50 is 0.5 mg/L  $\text{NO}_3^-$ -N and 0.8 mg/L  $\text{NO}_3^-$ -N for program 51. For more information on the estimated detection limit, see *Section 1*.

### Interferences

Interfering Substance	Interference Levels and Treatments
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.
Ferric iron	All levels
Nitrite	All levels Compensate for nitrite interference as follows: Add 30-g/L Bromine Water dropwise to the sample in Step 3 until a yellow color remains. Add one drop of 30-g/L Phenol Solution to destroy the color. Proceed with Step 4. 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.

### Summary Of Method

Cadmium metal reduces nitrates present in the sample to nitrite. The nitrite ion reacts in an acidic medium with sulfanilic acid to form an intermediate diazonium salt which couples to gentisic acid to form an amber-colored product.

### Pollution Prevention and Waste Management

NitraVer 5 contains cadmium metal. Both samples and reagent blanks will contain cadmium (D006) at a concentration regulated as hazardous wastes by the Federal RCRA. Do not pour these solutions down the drain. See *Section 3* for more information on proper disposal of these materials.

## NITRATE, High Range, continued

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### REQUIRED REAGENTS & APPARATUS (Using Powder Pillows)

Description	Quantity Required		Unit	Cat. No.
	Per Test			
NitraVer 5 Nitrate Reagent Powder Pillows.....	1 pillow	.....	100/pkg	.....21061-69
Sample Cell, 10-20-25 mL, w/cap .....	2	.....	6/pkg	.....24019-06

### REQUIRED REAGENTS (Using AccuVac Ampuls)

NitraVer 5 Nitrate Reagent AccuVac Ampul .....	1 ampul	.....	25/pkg	.....25110-25
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### REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL .....	1	.....	each	.....500-41H
Stopper .....	1	.....	6/pkg	.....1731-06

### OPTIONAL REAGENTS

Bromine Water 30 g/L .....	29 mL *	.....	2211-20
Nitrate Nitrogen Standard Solution, 10.0 mg/L as (NO <sub>3</sub> <sup>-</sup> -N) .....	500 mL	.....	307-49
Nitrate Nitrogen Standard Solution, 1000 mg/L as (NO <sub>3</sub> <sup>-</sup> -N) .....	500 mL	.....	12792-49
Nitrate Nitrogen Standard Solution, PourRite ampule, 500 mg/L as NO <sub>3</sub> <sup>-</sup> -N, 2 mL .....	20/pkg	.....	14260-20
Phenol Solution .....	29 mL	.....	2112-20
Sodium Hydroxide Standard Solution, 5.0 N.....	50 mL*	.....	2450-26
Sulfuric Acid, ACS .....	500 mL*	.....	979-49
Water, deionized .....	4 L	.....	272-56

### OPTIONAL APPARATUS

AccuVac Snapper Kit .....	each	.....	24052-00
Cylinder, graduated, mixing, 25 mL .....	each	.....	1896-40
Dropper, for 29-mL bottle .....	each	.....	2258-00
pH Indicator Paper, 1 to 11 pH.....	5 rolls/pkg	.....	391-33
pH Meter, <i>sension</i> <sup>™</sup> 1, portable, with electrode.....	each	.....	51700-10
Pipet Filler, safety bulb .....	each	.....	14651-00
Pipet, serological, 2 mL.....	each	.....	532-36
Pipet, TenSette, 0.1 to 1.0 mL .....	each	.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg	.....	21856-96
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg	.....	21856-28
PourRite Ampule Breaker .....	each	.....	24846-00
Thermometer, -20 to 110 °C, non-mercury .....	each	.....	26357-02

### *For Technical Assistance, Price and Ordering*

In the U.S.A. call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

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\* Contact Hach for larger sizes.