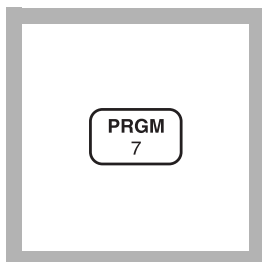


NITRATE, Mid Range (0 to 5.0 mg/L NO₃⁻-N) For water, wastewater and seawater***Cadmium Reduction Method (Using Powder Pillows or AccuVac Ampuls)****Using Powder Pillows**

1. Enter the stored program number for medium range nitrate nitrogen using powder pillows.

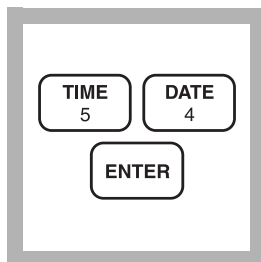
Press: **PRGM**

The display will show:

PRGM ?

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

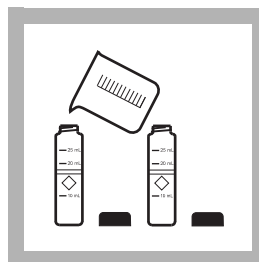
***Note:** Adjust the pH of stored samples before analysis.*



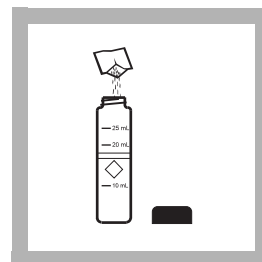
2. Press: **54 ENTER**

The display will show **mg/L, NO₃-N** and the **ZERO** icon.

***Note:** For alternate form (NO₃), press the **CONC** key.*



3. Fill two sample cells with 10 mL of sample each. One cell will be the prepared sample, the other is the blank. Set the blank aside.

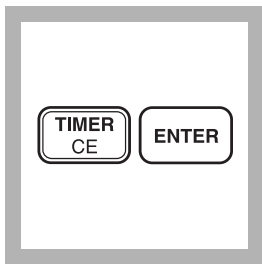


4. Add the contents of one NitraVer 5 Nitrate Reagent Powder Pillow to one cell (the prepared sample). Cap the cell.

***Note:** It is necessary to remove all the powder from the foil pouch by tapping repeatedly until no more powder comes out.*

* Seawater requires a manual calibration; see *Interferences*.

NITRATE, Mid Range, continued

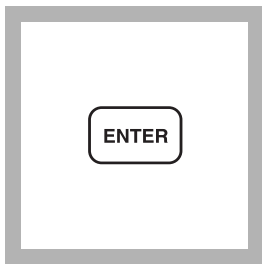


5. Press:

TIMER ENTER

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

***Note:** Shaking time and technique influence color development. Low results usually occur if shaking is not vigorous enough. For most accurate results, do successive tests on a standard solution and adjust the shaking time by ± 1 minute to obtain the correct result. See the Accuracy Check section for more information.*



6. After the timer beeps, the display will show:

5:00 TIMER 2

Press: **ENTER**

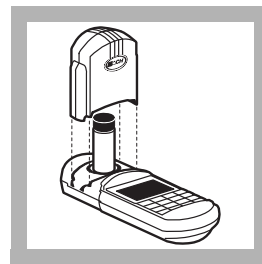
A five-minute reaction period will begin.

***Note:** A cadmium deposit will remain after the NitraVer 5 Nitrate Reagent Powder dissolves and will not affect test results.*

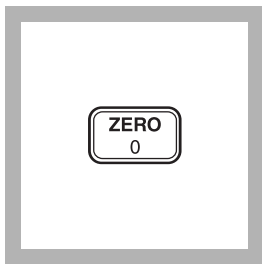
***Note:** An amber color will develop if nitrate nitrogen is present.*



7. After the timer beeps, wipe off any liquid or fingerprints.



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

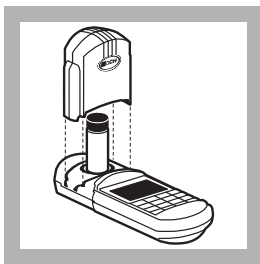


9. Press: ZERO

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

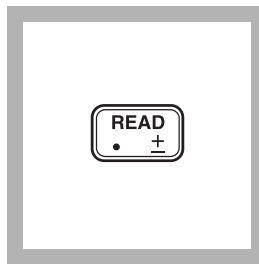
0.0 mg/L NO₃-N

***Note:** If Reagent Blank Correction is on, the display may flash “limit”.*



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

***Note:** Read the sample within two minutes after the timer beeps.*



11. Press: READ

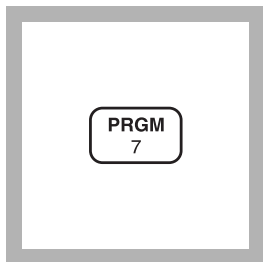
The cursor will move to the right, then the result in mg/L NO₃-N (or NO₃) will be displayed.

***Note:** Use of the standard adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

***Note:** Rinse the sample cell immediately after use to remove all the cadmium particles. See Pollution Prevention and Waste Management following these steps for disposal of cadmium particles.*

NITRATE, Mid Range, continued

Using AccuVac Ampuls



1. Enter the stored program number for medium range nitrate nitrogen using AccuVac Ampuls.

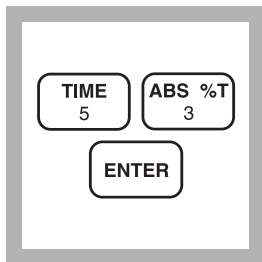
Press: **PRGM**

The display will show:

PRGM ?

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

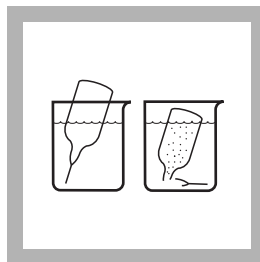
***Note:** Adjust the pH of stored samples before analysis.*



2. Press: **53 ENTER**

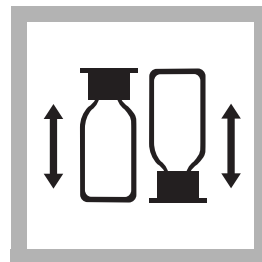
The display will show **mg/L, NO₃-N** and the **ZERO** icon.

***Note:** For alternate form (NO₃), 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 to allow room for mixing.*



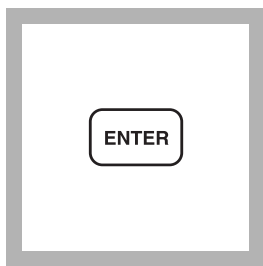
4. Press:

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 after mixing.

***Note:** Mixing speed and technique influence color development. For most accurate results, do successive tests on a standard solution and increase or decrease the mixing time to obtain the correct result. See Accuracy Check for more information.*

NITRATE, Mid Range, continued



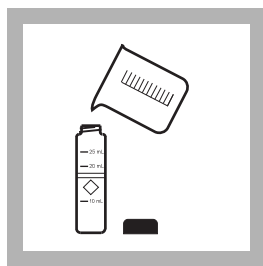
5. After the timer beeps, the display will show:
05:00 Timer 2

Press: **ENTER**

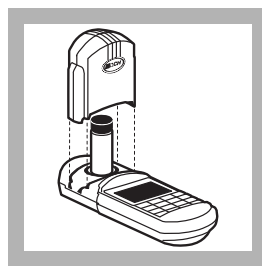
A five-minute reaction period will begin.

Note: A cadmium deposit will remain after the NitraVer 5 Nitrate Reagent Powder dissolves and will not affect results.

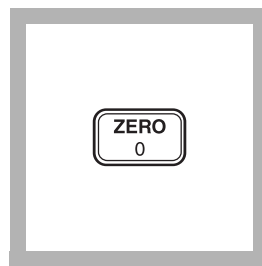
Note: An amber color will develop if nitrate nitrogen is present.



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



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

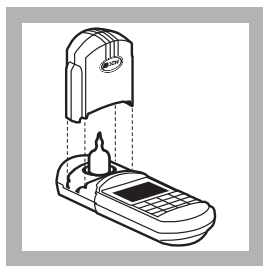


8. Press: **ZERO**

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

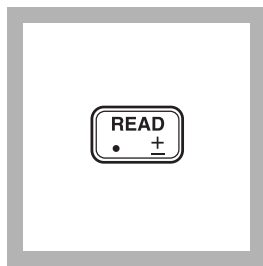
0.0 mg/L NO₃-N

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



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

Note: Read the sample within two minutes after the timer beeps.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L NO₃-N (or NO₃) will be displayed.

Note: Use of the standard adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.

NITRATE, Mid Range, continued

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 graduated mixing cylinders with 25 mL of sample.
- b) Snap the neck off a Nitrate Nitrogen Ampule Standard Solution, 100 mg/L NO_3^- -N.
- c) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of the standard to the three samples. Stopper and mix well.
- d) For analysis with AccuVac Ampuls, transfer the solutions to dry, clean 50 mL beakers. For analysis with powder pillows, transfer only 10 mL of the solution to dry, clean sample cells.
- e) Analyze each sample as described above. The nitrate nitrogen (NO_3^- -N) concentration should increase 0.4 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

A 1.0 mg/L Nitrate Nitrogen Standard Solution is available from Hach. Use this standard in place of sample in the above procedure.

Standard Adjust

To adjust the calibration curve using the reading obtained with the 1.00-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 **1.0** to edit the

NITRATE, Mid Range, continued

standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment . See *Section 1, Standard Curve Adjustment* for more information.

Method Performance

Precision

In a single laboratory using a standard solution of 3.0 mg/L nitrate nitrogen (NO_3^- -N) and two representative lots of powder pillows with the instrument, a single operator obtained a standard deviation of ± 0.2 mg/L nitrate nitrogen.

In a single laboratory using a standard solution of 3.0 mg/L NO_3^- -N and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ± 0.1 mg/L nitrate nitrogen.

Estimated Detection Limit

The estimated detection limit for programs 53 and 54 is 0.2 mg/L NO_3^- -N. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substances and Suggested Treatments

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 interfere. Compensate for nitrite interference as follows: 1. Add 30-g/L Bromine Water dropwise to the sample in Step 3 until a yellow color remains. 2. Add one drop of 30-g/L Phenol Solution to destroy the color. 3. 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.

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.

NITRATE, Mid Range, continued

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 waste by the Federal RCRA. Do not pour these solutions down the drain. See *Section 3* for more information on proper disposal of these materials.

REQUIRED REAGENTS AND APPARATUS (Using Powder Pillows)

Description	Qty/ Test	Unit	Cat. No.
NitraVer 5 Nitrate Reagent Powder Pillows	1 pillow	100/pkg.....	21061-69
Sample Cell, 10-20-25 mL, w/ caps	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.....	500-41
Stopper	1	6/pkg.....	1731-06

OPTIONAL REAGENTS

Bromine Water 30 g/L	29 mL*.....	2211-20
Drinking Water Standard, Inorganics, (Fe ⁻ , NO ₃ ⁻ , SO ₄ ²⁻ , PO ₄ ³⁻)	500 mL.....	28330-49
Nitrate Nitrogen Standard Solution, 1.0 mg/L as NO ₃ ⁻ -N.....	500 mL.....	2046-49
Nitrate Nitrogen Standard Solution, 100 mg/L as NO ₃ ⁻ -N.....	500 mL.....	1947-49
Nitrate Nitrogen Standard Solution, PourRite Ampule, 100 mg/L as NO ₃ ⁻ -N, 2 mL	20/pkg.....	1947-20
Phenol Solution, 30 g/L	29 mL.....	2112-20
Sodium Hydroxide Standard Solution, 5.0 N	50 mL SCDB*.....	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.....	20886-40
Dropper, for 1-oz bottle	each.....	2258-00
pH Paper, 1 to 11 pH units.....	5 rolls/pkg.....	391-33
pH Meter, <i>sension</i> TM 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

* Contact Hach for larger sizes.