Cadmium Reduction Method





1. Enter the stored program number for low range nitrate nitrogen (NO₃⁻-N).

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: 55 ENTER

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

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



3. Fill a 25-mL graduated mixing cylinder to the 15-mL mark with sample.

Note: Adjust the pH of stored samples before analysis.



For water, wastewater and seawater*

4. Add the contents of one NitraVer 6 Nitrate Reagent Powder Pillow to the cylinder. Stopper.

Note: It is necessary to remove all the powder from the foil pillow. Tap the pillow until no more powder pours out. Be sure to remove powder from the corners of the pillow.

^{*} Seawater requires a manual calibration; see Interferences.



5. Press: TIMER ENTER

A 3-minute reaction period will begin. Shake the cylinder vigorously throughout this three minute period.

Note: Shaking time and technique influence color development. For most accurate results, analyze a standard solution several times and adjust the shaking time to obtain the correct result.



6. When the timer beeps, the display will show: 2:00 TIMER 2 Press: ENTER

A 2-minute reaction period will begin.

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

7. When the timer

beeps, pour 10 mL of the sample into a sample cell.

Note: Do not transfer any cadmium particles.

|--|

8. Add the contents of one NitriVer 3 Nitrite Reagent Powder Pillow to the sample cell (the prepared sample). Cap the cell and shake gently for 30 seconds.

Note: A pink color will form if nitrate is present.



9. The display will show: 15:00 TIMER 3 Press: ENTER

A 15-minute reaction period will begin.

Fill another sample cell (the blank) with 10 mL of sample.



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



11. Press: ZERO

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

0.00 mg/L NO3-N

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



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

NITRATE, Low Range, continued

Note: See Pollution Prevention and Waste

Management for proper

disposal of cadmium.



13. Press: READ

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

Note: Standard Adjust may be performed using a prepared standard (see Section 1).

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

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 15 mL of sample.
- **b**) Snap the neck off a Nitrate Nitrogen Ampule Standard Solution, 12.0 mg/L NO₃⁻N.
- c) Using the TenSette Pipet, add 0.1, 0.2, and 0.3 mL of the standard to the three samples. Stopper and mix well.
- d) Analyze each sample as described above. The nitrate nitrogen concentration should increase 0.08 mg/L for each 0.1 mL of standard added.
- e) If these increases do not occur, see *Standard Additions* (Section 1) for more information.

Standard Solution Method

Prepare a 0.20 mg/L nitrate nitrogen standard by diluting 2.00 mL of a

10.0 mg/L Nitrate Nitrogen Standard Solution to 100.0 mL with deionized water. Use this standard in place of sample in Step 3.

Standard Adjust

To adjust the calibration curve using the reading obtained with the 0.20-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 **0.20** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the curve adjustment. If you are using a reagent blank correction, the blank correction should be entered before the Standard Adjust feature is entered. See *Section 1*, *Standard Curve Adjustment* for more information.

Method Performance

Precision

In a single laboratory using a standard solution of 0.25 mg/L nitrate nitrogen (NO₃⁻-N) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ± 0.03 mg/L nitrate nitrogen.

Estimated Detection Limit

The estimated detection limit for program 55 is 0.01 mg/L NO₃⁻N. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Calcium	100 mg/L
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: This method measures both the nitrate and nitrite in the sample. If nitrite is present, the nitrite nitrogen test Program 60 should be done on the sample. Pretreat the nitrate nitrogen sample with the following pretreatment. Then subtract the amount of nitrite found from the results of the LR nitrate nitrogen test using the pretreated sample. 1. Add 30-g/L Bromine Water dropwise to the sample in Step 3 until a yellow color remains. Mix after each drop. 2. Add one drop of 30-g/L Phenol Solution to destroy the yellow color. 3. Proceed with the LR Nitrate procedure.
рН	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 chromotropic acid to form a pink-colored product.

Pollution Prevention and Waste Management

NitaVer 6 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.

REQUIRED REAGENTS

Low Range Nitrate Reagent Set (100 tests)	
Includes: (1) 21071-69, (1) 21072-49	
	Quantity Required

Description	Per Test	Unit	Cat. No.
NitriVer 3 Nitrite Reagent Powder Pillows	1 pillow.	100/pkg	.21071-69
NitraVer 6 Nitrate Reagent Powder Pillows	1 pillow .	100/pkg	.21072-49

REQUIRED APPARATUS

Cylinder, graduated, mixing, 25 mL	1	each	1896-40
Sample Cell, 10-20-25 mL, w/ cap		6/pkg	24019-06

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Bromine Water, 30 g/L		2211-20
Nitrate Nitrogen Standard Solution, 10.0 mg/L as NO ₃ ⁻ -N	500 mL	
Nitrate Nitrogen Standard Solution, Voluette ampule,		
12 mg/L as NO ₃ ⁻ -N, 10 mL	16/pkg	14333-10
Phenol Solution, 30 g/L	29 mL	
Pretreatment Kit, contains: (1) 2112-20, (1) 2211-20	each	
Sodium Hydroxide Standard Solution, 5.0 N	. 50 mL* SCDB	
Sulfuric Acid, ACS	500 mL*	
Water, deionized	4 L	

OPTIONAL APPARATUS

Ampule Breaker	each	21968-00
Dropper, for 29-mL bottle	each	
Flask, volumetric, Class A, 100 mL	each	14574-42
pH Indicator Paper, 1 to 11 pH	5-roll/pkg	
pH Meter, <i>sension</i> [™] <i>I</i> , portable, with electrode	each	51700-10
Pipet, serological, 2 mL	each	
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
Pipet, volumetric, Class A, 2.00 mL	each	14515-36
Pipet Filler, safety bulb	each	14651-00
Thermometer, -20 to 110 °C	each	
Nitrate at these levels can also be determined directly using the Nitra	ate Ion Select	ive Electrode
(Cat. No. 23488-00).		

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.

^{*} Contact Hach for larger sizes