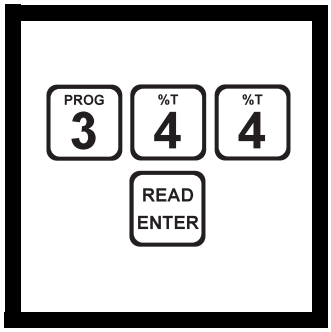
ANALYTICAL PROCEDURES
For DR/2000 and DR/3000 Instruments

Method 10020

NITRATE, High Range, Test 'N Tube™ *

(0 to 30.0 mg/L NO_3^-–N)

Chromotropic Acid Method **For water and wastewater**



1. Enter the stored program number for Test 'N Tube nitrate nitrogen (NO_3^-–N).

Press: **344 READ/ENTER**

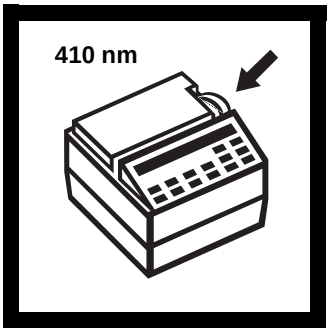
The display will show:

DIAL nm TO 410

Note: *See Instrument Setup following these steps to enter the method into the DR/2000.*

Note: *DR/2000s with software versions 3.0 and greater will display* **P** *and the program number.*

Note: *Instruments with software versions 3.0 and greater will not display* **DIAL nm TO** *message if the wavelength is already set correctly. The display will show the message in step 3. Proceed with step 4.*

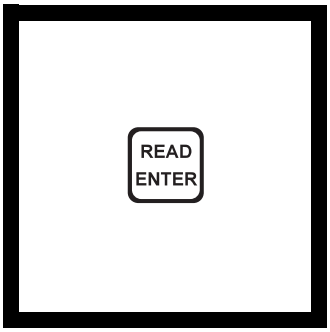


2. Rotate the wavelength dial until display shows:

410 nm

Note: *For DR/3000s, set wavelength to 410 nm and press* **CLEAR**.

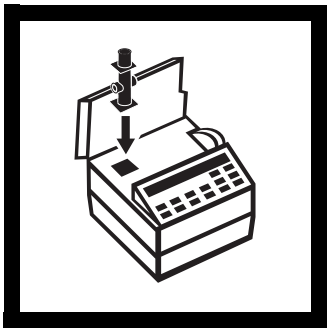
Note: *If sample cannot be analyzed immediately, see Sampling and Storage on page 7.*



3. Press: **READ/ENTER**

The display will show:

mg/l NO_3^- N Vial



4. Place the COD Vial Adapter into the cell holder with the marker to the right.

Note: *For DR/3000s, the groove in the adapter faces the front of the instrument.*

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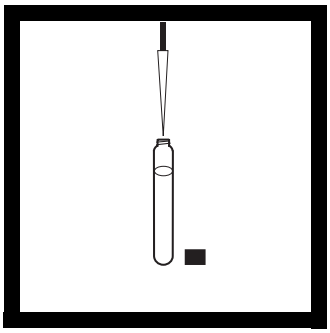
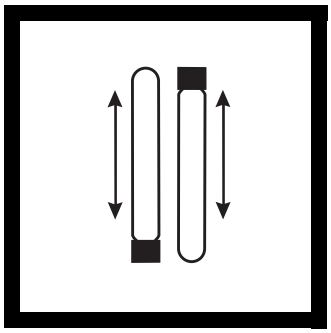


5. Remove the cap from a Nitrate Pretreatment Solution Vial and add 1 mL of sample (this will be the prepared sample).

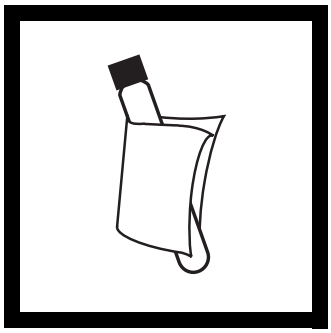
Note: *For proof of accuracy, use a 10 mg/L Nitrate Nitrogen Standard Solution in place of the sample (see OPTIONAL REAGENTS on page 9).*

Note: *Run a reagent blank for this test. Use deionized water in place of the sample. Subtract this result from all test results run with this lot of NitraVer X Reagent B. Determine a new reagent blank when the reagent lot of NitraVer® X Reagent B changes.*



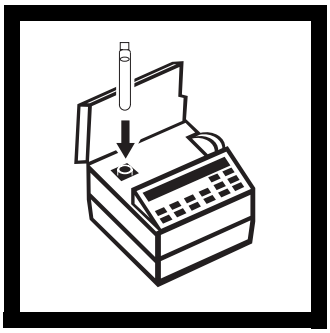
6. Cap the tube and invert it 10 times to mix.

Note: *This test is technique sensitive. If these instructions are not followed, low results may occur. Hold the tube in a vertical position with the cap pointing up. Invert the vial so the cap now points down. Wait for all of the solution to flow to the cap end. Pause. Return the vial to the original position. Wait for all the solution to flow to the vial bottom. This process equals 1 inversion. Do this 10 times. This should take 30 seconds.*

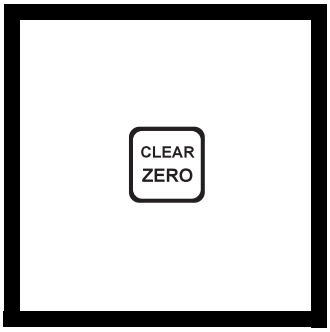


7. Clean the outside of the vial with a towel.

Note: *Wipe with a damp towel, and follow with a dry one, to remove fingerprints and other marks.*



8. Place the sample into the vial adapter with the Hach logo facing the front of the instrument. Place the cover on the adapter.



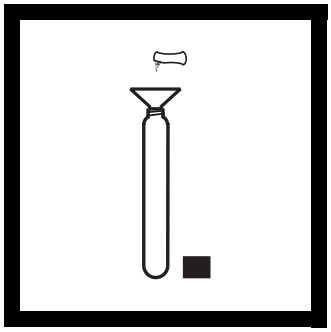
9. Press: **ZERO**

The display will show:

WAIT

then: **0.0 mg/l NO_3^- N Vial**

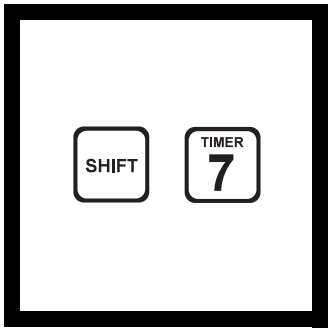
Note: *For DR/3000s, press* **MANUAL PROGRAM**. *Then press* **ZERO**. *Press* **ZERO** *again if the display does not show* **0.000**. *Enter the concentration factor (***20.51***) and press* **CONC FACTOR**. *Then press* **1 CONC**.



10. Using a funnel, add the contents of 1 NitraVer X Reagent B Powder Pillow to the vial. Cap and invert 10 times to mix. (This will be the prepared sample.)

Note: *See step 6 for inversion instructions.*

Note: *A small amount of solid matter will not dissolve.*



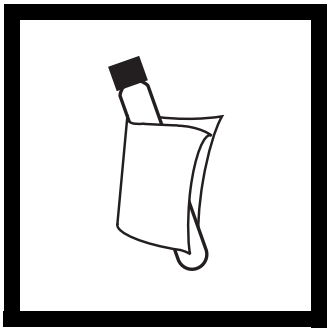
11. Press: **SHIFT TIMER**

A five-minute reaction period will begin. Do not shake the vial again.

Note: *A yellow color will develop if nitrate nitrogen is present.*

Note: *Complete steps 12–14 within five minutes after the timer beeps.*

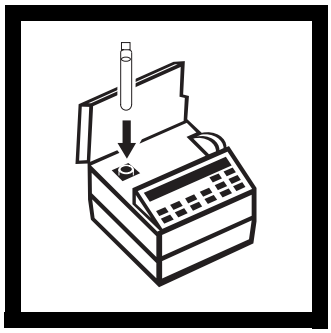
Note: *For DR/3000s, press* **5 TIMER**.



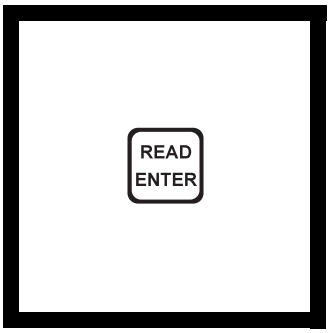
12. When the timer beeps, the display will show:

0.0 mg/l NO_3^- N Vial

Clean the outside of the vial with a towel.



13. Place the sample into the vial adapter with the Hach logo facing the front of the instrument. Place the cover on the adapter.



14. Press: **READ/ENTER**

The display will show:

WAIT

then the result in mg/L nitrate expressed as nitrogen (**NO_3^- N**) will be displayed.

Instrument Setup

DR/2000 with 1.265 through 1.27 software
Enter the calibration as an operator-programmed calibration.
Follow the steps in the Instrument Operation section of the *Instrument Manual*.
Store the method as follows:

nm = 410

Decimal = 000.0

Units = mg/l

Symbol = NO_3^- N Vial

Timer 1 = 5:00

Enter the calibration with 0.000 absorbance values for zero and # 1 standard. To do this, do not place anything in the sample cell compartment. Begin by storing standards 0 and 1 as the concentrations shown in the table below. Accept 0.000 Abs as the absorbance value for all standards. Store the calibration values by pressing **SHIFT READ/ENTER**.

Next, edit the absorbance values for the standards to the values given below. Follow the steps given in the Instrument Operation section of the *Instrument Manual*.

Std.	Conc	Abs
# 0	0.0	0.000
# 1	33.0	1.600

The method is now stored as an operator-programmed method number between 950 and 999. Record the method number for future reference when using this method.

DR/2000 with Software Versions 2.0 and 2.2
Enter the calibration as an update to Hach-stored programs.

1. Press: 

2. Press: 

3. Press: 

Within 3 seconds, press: 

The display will show: **ENTER nm**

4. Press: 

Note: *If you make an error, press* **SHIFT CLEAR** *and re-enter the number. When the number is correct, press* **READ/ENTER**.

The display will show: **DECIMAL? 00.00**

5. Use the arrow keys to correctly position the decimal point. Press the **DOWN ARROW** key once. The display will show: **DECIMAL? 000.0**

6. Press: **READ/ENTER**. The display will show: **UNITS?**

7. Use the arrow keys to select the appropriate unit of measure. Press the down arrow key once. The display will show: **mg/l**

8. Press **READ/ENTER** when the correct unit of measure is displayed. The display will show: **SYMBOL?**

9. Construct the correct symbol display: **NO_3^- N Vial**

a. Select letters and regular numbers by scrolling to the correct symbol with the arrow keys.

b. To make a letter uppercase, press the **SHIFT** key.

c. To enter subscript numbers, enter the digit on the numeric keypad.

d. To enter superscript characters, enter the digit on the numeric keypad number then make it a superscript by pressing **SHIFT**.

e. The space is the character displayed after one press of the **DOWN ARROW**.

f. Accept each symbol by pressing **READ/ENTER**.

g. After entering the last character and pressing **READ/ENTER** to accept it, the instrument will leave symbol entry mode.

10. When the instrument is out of symbol entry mode, the display will show: **TIMER?**

11. This method has one timed step, so press **SHIFT TIMER**. The display will show: **MM:SS TIME 1?**

12. Enter a timer value of 5 minutes.

13. Press: 

14. Press **READ/ENTER** to accept the timer value. The display will show: **MM:SS TIME 2?**

15. Press **READ/ENTER** to complete the timer entry. The display will show: **#1 Data**

16. Enter the following 12 numbers as shown. Complete each number entry by pressing the **READ/ENTER** key.

# 1 Data	0
# 2 Data	6682
# 3 Data	6426
# 4 Data	6682
# 5 Data	6426
# 6 Data	6682
# 7 Data	6681
# 8 Data	6911
# 9 Data	65535
#10 Data	9362
#11 Data	512
Checksum	9173

The final number is a check value which determines if the data sequence was correctly entered. If an error was made during number entry, the display will return to the prompt for data # 1 and the entire sequence must be re-entered. If all numbers are correctly entered, the display will return to the method prompt and is ready for use.

DR/2000 with Software Version 3.0 and 3.1

1. Turn the instrument on. Press **SHIFT METHOD** to enter configuration mode. The display will show: **MOMENTARY** or **CONSTANT ON**

2. Press the **UP ARROW** key twice to select **HACH UPDATE**. Press **READ/ENTER**. The display will show: **ENTER #:**

3. Press: 

The display will show: **P344 ENTER nm**

4. Press: 

Note: *If you make an error, press* **SHIFT CLEAR** *and re-enter the number. When the number is correct, press* **READ/ENTER**.

The display will show: **P344 DECIMAL? 00.00**

5. Use the arrow keys to correctly position the decimal point. Press the **DOWN ARROW** key once. The display will show: **DECIMAL? 000.0**

6. With the decimal point correctly positioned, press **READ/ENTER**.
The display will show: **P344 UNITS?**

7. Use the arrow keys to select the appropriate unit of measure. Press the **DOWN ARROW** key once. The display will show: **P344 mg/l**

8. Press **READ/ENTER** when the correct unit of measure is displayed.
The display will show: **P344 mg/l _**

9. Construct the display to read the correct symbol. The symbol must be entered exactly as shown, including dashes and spaces between characters: **NO_3^- N Vial**

   The 3 is subscript; the – is superscript. There is a space after the superscript – and before Vial.

   a. Select letters and numbers by scrolling to the correct character with the arrow keys.

   b. To make a letter uppercase, press the **SHIFT** key.

   c. Make a number or sign superscript, subscript or regular by pressing **SHIFT** until the symbol is correct.

   d. The space is the character displayed after one press of the down arrow.

   e. Make sure to enter the display line EXACTLY as shown, including all spaces.

   f. Accept each symbol by pressing **READ/ENTER**.

   g. After entering the last character and pressing **READ/ENTER** to accept it, the instrument will leave symbol entry mode.

10. When the instrument is out of symbol entry mode,
the display will show: **P344 TIMER?**

11. This method has one timed step, so press **SHIFT TIMER**.
The display will show: **MM:SS TIME 1?**

12. Enter a timer value of 5 minutes. Press: 

13. Press **READ/ENTER** to accept the timer value.
The display will show: **MM:SS TIME 2?**

14. Press **READ/ENTER** to complete the timer entry.
The display will show: **#0 STANDARD**

15. Press **READ/ENTER** to display the zero data pair.
The display will show: **0.000 Abs 000.0 mg/l**

16. Press **READ/ENTER**. The display will show: **# 1 STANDARD**

17. Press **READ/ENTER**. The display will prompt for entry of the first concentration point: **# 1 000.0 mg/l**

18. Enter concentration point #1 from the table below by pressing:

so that the display shows: **# 1 033.0 mg/l**

19. Press **READ/ENTER**. The display will prompt for entry of the first absorbance point: **# 1 0.000 Abs**

20. Enter absorbance point #1 from the table below by pressing:

so that the display shows: **# 1 1.600 Abs**

21. Press **READ/ENTER**. The display will show the first data pair: **1.600 Abs 033.0 mg/l**

22. Press **READ/ENTER** to accept the first data pair.
The display will show: **# 2 STANDARD**

23. The data pair values from the table below are now entered.

Standard	Concentration	Absorbance
# 0	[0.0] mg/L	[0.000] Abs
# 1	[33.0] mg/L	[1.600] Abs

24. When the last point pair is entered the display will show: **# 2 STANDARD**

25. Press **SHIFT READ/ENTER** to complete data point entry. The display will show: **#:**

26. Enter the validation number:

so that the display shows: **#: 3718**

27. Press **READ/ENTER**. The display will show: **COMPLETED**
then: **P344 mg/l NO_3^- N Vial**

Note: *If the display shows:* **INCORRECT #** *then prompts again for the validation number, you may have made an error during data entry. Make sure that the validation number is correct. If so, then the error occurred during some other portion of the method entry. You must press* **METH** *and respond to the* **ABORT?** *message by pressing* **READ/ENTER**, *then re-enter the method.*

The instrument is now ready for use with method 344.

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 (up to 14 days), 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 in the *Procedures Manual.*

Accuracy Check

Standard Additions Method

a. Snap the neck off a fresh High Range Nitrate Nitrogen Standard Voluette™ Ampule, 500 mg/L NO_3^-–N.

b. Use the TenSette® Pipet to add 0.1, 0.2, and 0.3 mL of standard to three 25-mL samples, respectively. Mix each thoroughly.

c. Analyze each sample as described in the procedure. The nitrogen concentration should increase 2.0 mg/L for each 0.1 mL of standard added.

d. If these increases do not occur, see Standard Additions in the *Procedures Manual* for more information.

Standard Solution Method
Use a 10.0 mg/L Nitrate Nitrogen Standard Solution listed under *OPTIONAL REAGENTS* to check test accuracy. Or, this can be prepared by diluting 1.00 mL of solution from a High Range Nitrate Nitrogen Voluette Ampule Standard Solution, 500 mg/L NO_3^-–N, to 50.0 mL with deionized water.

Precision

DR/2000: In a single laboratory, using a standard solution of 20.0 mg/L nitrate nitrogen (NO_3^-–N) and 2 representative lots of reagent with the DR/2000, a single operator obtained a standard deviation of ± 0.2 mg/L N.

DR/3000: In a single laboratory, using a standard solution of 20.0 mg/L nitrate nitrogen (NO_3^-–N) and 2 representative lots of reagent with the DR/3000, a single operator obtained a standard deviation of ± 0.1 mg/L N.

Interferences

Interfering Substance	Interference Level
Barium	A negative interference at concentrations greater than 1 mg/L.
Chloride	Does not interfere below 1000 mg/L.
Copper	Causes a positive interference at all levels.
Hardness	Does not interfere.
Nitrite	A positive interference at concentrations greater than 12 mg/L. Remove nitrite interference up to 100 mg/L by adding 400 mg of urea (one full 0.5 g Hach measuring spoon) to 10 mL of sample. Swirl to dissolve. Proceed with the nitrate test as usual.

Summary of Method

Nitrate in the sample reacts with chromotropic acid under strongly acidic conditions to yield a yellow product with a maximum absorbance at 410 nm.

Reagents and Apparatus

REQUIRED REAGENTS

	Cat. No.
NitraVer® X Nitrate, High Range Test 'N Tube™ Reagent Set (50 vials)	26053-45

Includes: (1) 26055-46, (1) 272-42, (50) Nitrate Pretreatment Solution Vials*

Description	Quantity Required Per Test	Unit	Cat. No.
Nitrate Pretreatment Solution Vials	1	50/pkg	*
NitraVer® X Reagent B Powder Pillows	1	50/pkg	26055-46
Water, deionized	varies	100 mL	272-42

REQUIRED APPARATUS

COD Vial Adapter, DR/2000 and DR/3000	1	each	44799-00
Funnel, micro	1	each	25843-35
Pipet, TenSette® 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for 19700-01 TenSette® Pipet	1	50/pkg	21856-96
Test Tube Rack	1–3	each	18641-00

OPTIONAL REAGENTS

Nitrate Nitrogen Standard Solution, 10 mg/L NO_3^-–N	500 mL	307-49
Nitrate Nitrogen Standard Solution, Voluette™ Ampules, 500 mg/L	16/pkg	14260-10
Sodium Hydroxide Standard Solution, 5.0 N	50 mL	2450-26
Sulfuric Acid, ACS, concentrate	500 mL	979-49
Urea, ACS	100 g	11237-26
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Flask, volumetric class A, 50 mL	each	14574-41
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
Pipet, serological, 2 mL	each	532-36
Spoon, measuring, 0.5 g	each	907-00

* Not available separately. Please order the complete set (Cat. No. 26053-45) as a replacement.

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