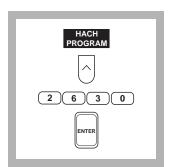
Method 10019

Diazotization Method

LR (0 to 0.500 mg/L NO_2 --N)

Test 'N TubeTM Vials

Scope and Application: For water, wastewater and seawater. The estimated detection limit for program number 2630 is 0.0013 mg/L NO_2 -N.



1. Press the soft key under *HACH PROGRAM*.

Select the stored program for the Test 'N Tube Nitrite method by pressing **2630** with the numeric keys.

Press: **ENTER**

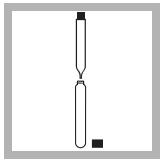
Note: If sample cannot be analyzed immediately, see Sample Collection, Storage and Preservation following these steps.



2. The display will show: HACH PROGRAM: 2630 Nitrite, TNT

The wavelength (λ) , **507 nm**, is automatically selected.

Note: For best results, determine a reagent blank for each new lot of reagent as follows. Prepare a reagent blank by repeating Steps 3 through 10, using deionized water as the sample. Zero the instrument on deionized water by pressing the soft key under **ZERO**. Insert the reagent blank and the blank value will be displayed. Correct for the reagent blank by pressing the soft keys under OPTIONS, (MORE), and then BLANK:OFF. Enter the reagent blank value and press ENTER. Repeat for each new lot of reagent.



3. Fill a Test 'N Tube NitriVer 3 Nitrite vial with 5 mL of sample. Cap and shake to dissolve powder. This is the prepared sample.

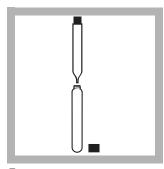
Note: For proof of accuracy, use a 0.250-mg/L nitrite nitrogen standard solution (preparation given in the Accuracy Check section) in place of the sample.

Note: A pink color will develop if nitrite nitrogen is present.



4. Press the soft key under *START TIMER*. A 20-minute reaction period will begin.

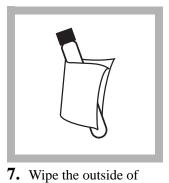
NITRITE, continued



5. When the timer beeps, fill an empty Test 'N Tube vial with 5 mL of sample (the blank).



6. Insert the Test 'N Tube Adapter into the sample cell module by sliding it under the thumb screw and into the alignment grooves. Fasten with the thumb screw.



the vials with a towel. **Note:** Wiping with a damp towel, followed by a dry one, removes fingerprints and other marks.



8. Place the blank into the cell holder.



9. Press the soft key under **ZERO**.

The display will show:

$0.0000 \, mg/L \, NO_2$ --N

Note: If you are using a reagent blank correction, the display will show the correction.

Note: For alternate concentration units, press the soft key under OPTIONS. Then press the soft key under UNITS to scroll through the available options. Press ENTER to return to the read screen.



10. Place the prepared sample into the cell holder. The result in mg/L nitrite expressed as nitrogen will be displayed.

Note: The results can be expressed as NO₂⁻. Press the soft keys under METHOD OPTIONS, then FORM: to scroll through the available options. Press ENTER to return to the read screen.

Interferences

| Interfering Substance | Interference Levels and Treatments | |
|--|---|--|
| Antiminous ions | Interfere by causing precipitation | |
| Auric ions | Interfere by causing precipitation | |
| Bismuth ions | Interfere by causing precipitation | |
| Chloroplatinate ions | Interfere by causing precipitation | |
| Cupric ions | Cause low results | |
| Ferric ions | Interfere by causing precipitation | |
| Ferrous ions | Cause low results | |
| Lead ions | Interfere by causing precipitation | |
| Mercurous ions | Interfere by causing precipitation | |
| Metavanadate ions | Interfere by causing precipitation | |
| Nitrate | Very high levels of nitrate (>100 mg/L nitrate as N) appear to undergo a slight amount of reduction to nitrite, either spontaneously or during the course of the test. A small amount of nitrite will be found at these levels. | |
| Silver ions | Interfere by causing precipitation | |
| Strong oxidizing and reducing substances | Interfere at all levels | |

Sample Collection, Storage and Preservation

Collect samples in clean plastic or glass bottles.

Store at $4 \,^{\circ}\text{C}$ (30 $^{\circ}\text{F}$) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test.

Accuracy Check

Standard Solution Method

Preparing nitrite standards is difficult. A standard should be prepared by a trained chemist. Hach recommends using the standard preparation instructions in *Standard Methods for the Examination of Water and Wastewater*, 18th ed., under the headings "Stock nitrite solution:," "Intermediate nitrite solution:," and "Standard nitrite solution:." These can be found on pp. 4–86. Prepare a 0.250-mg/L standard. Perform the nitrite test on the standard solution.

Method Performance

Precision

Standard: 0.2500 mg/L NO₂⁻–N

| Program | 95% Confidence Limits | |
|---------|--------------------------------------|--|
| 2630 | 0.2493-0.2507 mg/L NO ₂ N | |

For more information on determining precision data and method detection limits, refer to Section 1.5.

Estimated Detection Limit

| Program | EDL | |
|---------|-------------------------------|--|
| 2630 | 0.0013 mg/L NO ₂ N | |

For more information on derivation and use of Hach's estimated detection limit, see Section 1.5.2. To determine a method detection limit (MDL) as defined by the 40 CFR part 136, appendix B, see Section 1.5.1.

Sensitivity

Program Number: 2630

| Program | Δ Abs | ∆Concentration | |
|--------------|--------------|----------------|--|
| Entire Range | 0.010 | 0.0035 mg/L | |

See Section 1.5.3 Sensitivity Explained for more information.

Calibration Standard Preparation

Preparing nitrite standards is difficult. Calibration should be performed by a trained chemist. Hach recommends using the standard preparation instructions in *Standard Methods for the Examination of Water and Wastewater*, 18th ed., under the headings "Stock nitrite solution:," "Intermediate nitrite solution:," and "Standard nitrite solution". These can be found on pp. 4–86.

Using the standards prepared above and the analysis procedure, generate a calibration curve.

Summary of Method

Nitrite in the sample reacts with sulfanilic acid to form an intermediate diazonium salt. This couples with chromotropic acid to produce a pink colored complex directly proportional to the amount of nitrite present.

Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the *Material Safety Data Sheet* for information specific to the reagents used. For additional information, refer to Section 1.

Pollution Prevention and Waste Management

For information on pollution prevention and waste management, refer to Section 1.

| REQUIRED EQUIPMENT AND SUPPLIES | | | |
|---|-------------------|--------|----------|
| | Quantity Required | | |
| Description | | Unit | |
| DR/4000 Test Tube Adapter | | | |
| NitriVer 3 Nitrite Vials | | | |
| Pipet, TenSette, 1.0 to 10.0 mL | 1 | each | 19700-10 |
| Pipet Tips, for 19700-10 TenSette Pipet | varies | 50/pkg | 21997-96 |
| Test 'N Tube Vials | | | |
| Test 'N Tube Vial caps | | | |
| OPTIONAL REAGENTS AND STANDARDS | | | |
| Sodium Nitrite, ACS | | 454 g | 2452-01 |
| Water, deionized | | | |
| Water, deionized | | | |
| OPTIONAL EQUIPMENT AND SUPPLIES | | | |
| Balance, analytical, 110 VAC | | each | 26103-00 |
| Balance, analytical, 220 VAC | | | |
| Flask, volumetric, 1000-mL | | | |
| Test Tube Rack | | | |

^{*} Items not sold separately

