Method 8153

Ferrous Sulfate Method*

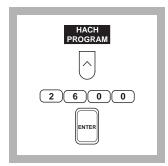
HR (0 to 250 mg/L NO₂-)

Powder Pillows

Scope and Application: For water and wastewater.

The estimated detection limit for program number 2600 is 1 mg/L NO_2^- .

* Adapted from McAlpine, R. and Soule, B., Qualitative Chemical Analysis, New York, 476, 575 (1933)



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

Select the stored program number for high range nitrite by pressing **2600** with the numeric keys.

Press: **ENTER**

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

Note: The Flow Cell and Sipper Modules cannot be used with this procedure.



2. The display will show:

HACH PROGRAM: 2600 Nitrite, HR

The wavelength (λ), **585 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 9, 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 sample cell with 10 mL of sample.

Note: For proof of accuracy, use a 200-mg/L nitrite solution in place of the sample. See the Accuracy Check section for preparation.



4. Add the contents of one NitriVer 2 Nitrite Reagent Powder Pillow, stopper and shake to dissolve (the prepared sample).

Note: A greenish-brown color will develop if nitrite is present.



5. Press the soft key under **START TIMER**.

A 10-minute reaction period will begin. It is critical to leave the sample undisturbed on a flat surface for the reaction period or low results may occur.



6. Fill another sample cell with 10 mL of sample (the blank). Place it into the cell holder.



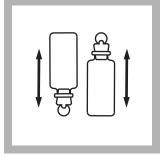
7. Press the soft key under *ZERO*.

The display will show:

$0~mg/L~NO_2^-$

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.



8. Gently invert the prepared sample twice. Remove the stopper.

Note: Avoid excessive mixing or low results may occur.



9. Place the prepared sample into the cell holder. Close the light shield. Results in mg/L NO₂⁻ (or chosen units) will be displayed.

Note: The results can be expressed as nitrite nitrogen (NO₂⁻–N) or as sodium nitrite (NaNO₂). Press the soft keys under **METHOD OPTIONS** and then **FORM:** to scroll through the available options.

Interferences

This test does not measure nitrates nor is it applicable to glycol-based samples. Dilute glycol-based samples and follow the Low Range Nitrite procedure.

Sample Collection, Storage and Preservation

Collect samples in clean plastic or glass bottles.

The following storage instructions are necessary only when prompt analysis is impossible. 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.

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*. Prepare a 200 mg/L standard.

Method Performance

Precision

Standard: 200 mg/L NO₂-

Program	95% Confidence Limits	
2600	199–201 mg/L NO ₂ -	

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

Estimated Detection Limit

Program	EDL
2600	1 mg/L NO ₂ -

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: 2600

Portion of Curve	Δ Abs	∆Concentration	
Entire Range	0.010	1.4 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

The method uses ferrous sulfate in an acidic medium to reduce nitrite to nitrous oxide. Ferrous ions combine with the nitrous oxide to form a greenish-brown complex in direct proportion to the 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 REAGENTS AND STANDARDS			
	Quantity Required		
Description		Unit	
NitriVer 2 Nitrite Reagent Powder Pillows	1 pillow	100/pkg	21075-69
REQUIRED EQUIPMENT AND SUPPLIES			
DR/4000 1-Inch Cell Adapter	1	each	48190-00
Sample Cells, matched pair, 1-inch, glass, with stoppers	2	pair	26126-02
OPTIONAL REAGENTS AND STANDARDS			
Sodium Nitrite, ACS		454 g	2452-01
Water, deionized			
OPTIONAL EQUIDMENT AND CURRING			
OPTIONAL EQUIPMENT AND SUPPLIES		•	26102.00
Balance, analytical, 110 VAC			
Balance, analytical, 220 VAC		each	26103-02
DR/4000 Carousel Module Kit		each	48070-02
Flask, volumetric, 1000-mL		each	547-53
Pipet, serological, 10-mL		each	532-38
Pipet Filler safety bulb		each	14651-00

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