Nitrite DOC316.53.01533

Diazotization Method¹

Method 10296

2 to 90 mg/L NO_2^- -N or 7 to 295 mg/L NO_2^- (UHR)

TNTplus[™] 841

Scope and application: For wastewater, Anammox, surface water and process water.

¹ Adapted from Standard Methods for the Examination of Water and Wastewater.



Test preparation

Instrument-specific information

Table 1 shows all of the instruments that have the program for this test. The table also shows the adapter and light shield requirements for the applicable instruments that can use TNTplus vials.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information for TNTplus vials

Instrument	Adapters	Light shield
DR 6000	_	_
DR 3900	_	LZV849
DR 1900	9609900 or 9609800 (A)	_

Before starting

DR3900: Install the light shield in Cell Compartment #2 before this test is started.

Review the safety information and the expiration date on the package.

The recommended sample pH is 3-10.

The recommended temperature for samples is 15–40 °C (59–104 °F).

The recommended temperature for reagent storage is 15-25 °C (59-77 °F).

DR 1900: Go to All Programs>LCK or TNTplus Methods>Options to select the TNTplus number for the test. Other instruments automatically select the method from the barcode on the vial.

Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.

Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

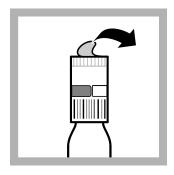
Description	Quantity
Nitrite UHR TNT841 Reagent Set	1
Pipet, adjustable volume, 0.2–1.0 mL	1
Pipet tips, for 0.2–1.0 mL pipet	1

Refer to Consumables and replacement items on page 4 for order information.

Sample collection and storage

- Collect samples in clean glass or plastic bottles.
- To preserve samples for later analysis, keep the samples at or below 6 °C (43 °F) for up to 48 hours.
- Let the sample temperature increase to room temperature before analysis.

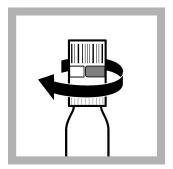
Test procedure



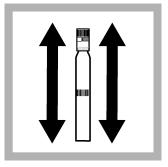
 Carefully remove the lid from the DosiCap[™] Zip cap. Remove the cap from the test vial.



2. Use a pipet to add 0.2 mL of sample to the test vial. Immediately continue to the next step.



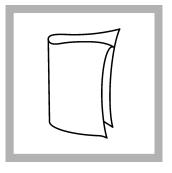
3. Turn the DosiCap Zip over so that the reagent side goes on the test vial. Tighten the cap on the vial.



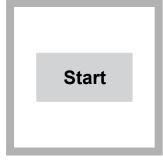
4. Shake the vial 2–3 times to dissolve the reagent in the cap.
Look through the open end of the DosiCap to make sure that the reagent has dissolved.



5. Start the reaction time of 15 minutes.



6. When the timer expires, clean the vial.



7. DR 1900 only: Select program 841. Refer to Before starting on page 1.



8. Insert the vial into the cell holder. DR 1900 only: Push **READ**. Results show in mg/L NO_2^- –N.

Reagent blank correction

For the best results, measure the reagent blank value for each new lot of reagent. Replace the sample with deionized water in the test procedure to determine the reagent blank value. Subtract the reagent blank value from the sample results automatically with the reagent blank adjust option. Measure the reagent blank value when a new lot of reagent is used.

- **1.** Use deionized water as the sample in the test procedure to measure the reagent blank value.
- 2. Set the reagent blank function to on. The measured reagent blank value is shown.
- 3. Accept the blank value. The reagent blank value is then subtracted from all results until the reagent blank function is set to off or a different method is selected.

 Note: As an alternative, record or enter the reagent blank value at a different time. Push the highlighted reagent blank box and use the keypad to enter the value.

Sample blanks

Samples with color or turbidity can cause high results. Samples without color or turbidity do not require sample blanks. To adjust for color or turbidity, use the steps that follow to find the sample blank.

- 1. Do the test procedure, but do not remove the foil lid from the vial.
- 2. Put the cap on the vial.
- **3.** Subtract the value from the final procedure step from the initial sample value to get the corrected sample concentration.

Interferences

Table 2 shows the ions that were individually examined to the given concentrations and do not cause interference. No cumulative effects or influences of other ions were found. Chromium (VI) ions interfere with the determination. Copper (II) ions interfere with the determination even at concentrations below 1 mg/L.

Table 2 Interfering substances

Interfering substance	Interference level
Sn ⁴⁺	10 mg/L
Ni ²⁺ , Ag ⁺	20 mg/L
Co ²⁺ , Zn ²⁺ , Cd ²⁺ , Mn ²⁺	50 mg/L
Cr ³⁺ , Hg ²⁺	100 mg/L
Mg ²⁺	200 mg/L
Fe ²⁺	300 mg/L
Fe ³⁺	600 mg/L
K ⁺ , NO ₃ ⁻ , Ca ²⁺ , Cl ⁻ , NH ₄ ⁺	2000 mg/L
SO ₄ ²⁻	4000 mg/L
PO ₄ ³ -	5000 mg/L

Accuracy check

Standard solution method

Use the standard solution method to validate the test procedure, the reagents and the instrument.

Items to collect:

- 250-mg/L NO₂ –N Standard Solution
- Volumetric flask, Class A, 25 mL
- Pipet, 1.0 to 10.0 mL
- Pipet tips
- Deionized water
- Make a nitrite standard solution at a nitrite concentration level near the sample concentration. Add the applicable volume of the stock standard solution to the volumetric flask.
 - 10 mg/L as N—Add 1.0 mL of stock standard solution
 - 40 mg/L as N—Add 4.0 mL of stock standard solution
 - 70 mg/L as N—Add 7.0 mL of stock standard solution
- 2. Fill the volumetric flask to the mark with deionized water (25 mL).
- 3. Put a stopper in the flask. Invert to mix.

- **4.** Use the test procedure to measure the concentration of the prepared standard solution.
- **5.** Compare the expected result to the actual result.

Note: The factory calibration can be adjusted slightly with the standard calibration adjust option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are small variations in the reagents or instruments.

Summary of Method

Nitrite in the sample reacts with a primary aromatic amine in acidic solution to form a diazonium salt. This couples with an aromatic compound to form a complex with color that is directly proportional to the amount of nitrite in the sample. The measurement wavelength is 605 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
Nitrite UHR TNT841 Reagent Set	1	25/pkg	TNT841

Required apparatus

Description	Quantity/test	Unit	Item no.
Pipet, adjustable volume, 0.2–1.0 mL	1	each	BBP078
Pipet tips, for 0.2–1.0 mL pipet	2	100/pkg	BBP079
Adapter set for DR 1900 spectrophotometer	1	set of 4	9609900 or 9609800
Light shield, DR 3900	1	each	LZV849

Recommended standards

Description	Unit	Item no.
Nitrite Standard Solution, 250 mg/L as N	500 mL	2340249

Optional reagents and apparatus

Description	Unit	Item no.
Sampling bottle with cap, low density polyethylene, 500-mL	12/pkg	2087079
Test tube rack, polyethylene, for 13-mm OD vials, 90 holes	each	2497900
Water, deionized	4 L	27256
Flask, volumetric, Class A, 25 mL	each	1457440
Pipet, TenSette [®] , 1.0–10.0 mL	each	1970010
Pipet tips for TenSette® Pipet, 1.0–10.0 mL	50/pkg	2199796

