Nitrate

DOC316.53.01070

Dimethylphenol Method

Method 10206[1]

0.23 to 13.50 mg/L NO_3^-–N or 1.00 to 60.00 mg/L NO_3^- (LR)

TNTplus 835

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

[1] USEPA approved for water and wastewater analysis, 40 CFR part 136; and drinking water, 40 CFR part 141.23.

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
DR6000, DR5000	—	—
DR3900	—	LZV849
DR3800, DR2800	—	LZV646
DR1900	9609900 or 9609800 (A)	—

Before starting

DR3900, DR3800, DR2800: 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 sample temperature must be 20–23 °C (68–73 °F) for accurate results.
The recommended temperature for reagent storage is 15–25 °C (59–77 °F).
DR1900: 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

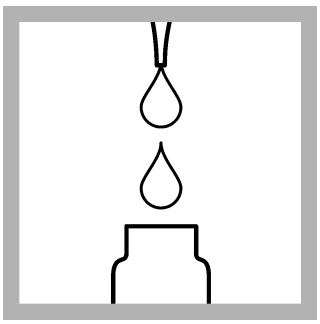
Description	Quantity
Nitrate LR TNTplus Reagent Set	1
Pipet, adjustable volume, 0.1–1.0 mL	1
Pipet tips, for 0.1–1.0 mL pipet	1

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

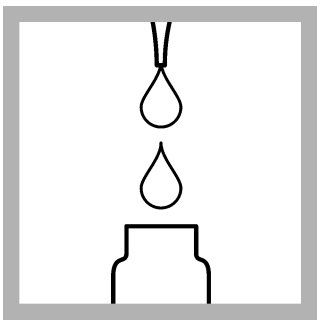
Sample collection and storage

- Collect samples in clean glass or plastic bottles.
- Analyze the samples as soon as possible for best results.
- If immediate analysis is not possible, immediately filter and keep the samples at or below 6 °C (43 °F) for a maximum of 48 hours.
- To preserve samples for a maximum of 14 days, adjust the sample pH to 2 or less with concentrated sulfuric acid (approximately 2 mL per liter) and keep at or below 6 °C (43 °F). The test results then include nitrate and nitrite.
- Let the sample temperature increase to room temperature before analysis.
- Before analysis, adjust the pH to 7 with 5 N sodium hydroxide solution.
- Correct the test result for the dilution caused by the volume additions.

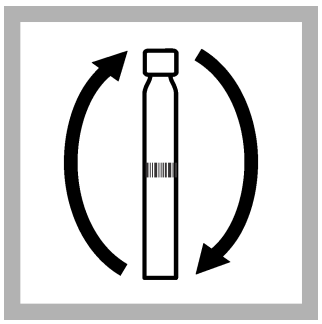
Test procedure



1. Use a pipet to add 1.0 mL of sample to the test vial.

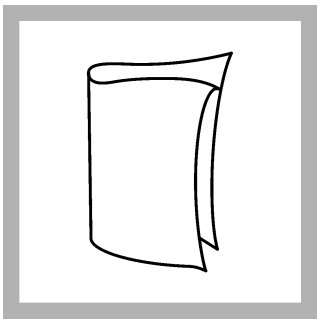


2. Use a pipet to add 0.2 mL of Solution A to the test vial.

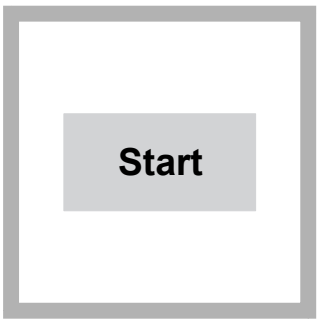


3. Tighten the cap on the vial and invert until completely mixed.

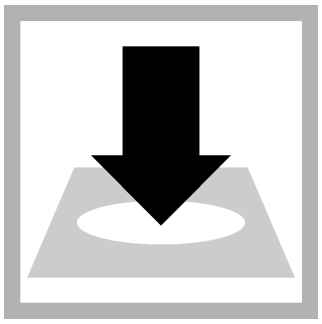
4. Start the reaction time of 15 minutes.



5. When the timer expires, clean the vial.



6. DR1900 only: Select program 835. Refer to Before starting on page 1.



7. Insert the vial into the cell holder. DR1900 only: Push **READ**.
Results show in mg/L NO_3^-–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

If the sample has color or turbidity, measure a sample blank to correct the test result for the interference.

1. Use deionized water as the reagent in the test procedure to measure the sample blank value:
   a. Use a pipet to add 1.0 mL of sample to the test vial.
   b. Use a pipet to add 0.2 mL of deionized water to the test vial.
   c. Tighten the cap on the vial and invert until completely mixed.
   d. Start the reaction time of 15 minutes.
   e. When the timer expires, clean the vial.
   f. DR1900 only: Select program 835.
   g. Insert the vial into the cell holder. DR1900 only: Push READ. Results show in mg/L NO_3^-–N.
2. Subtract the sample blank value from the test result (with reagent) to get the corrected sample concentration.

Interferences

Table 2 shows that the ions were individually examined to the given concentrations and do not cause interference. No cumulative effects or influences of other ions were found.

The cumulative effects and influence of other ions have not been found. High loads of oxidizable organic substances (COD) cause the reagent to change color and to give high-bias results. The test can thus only be used for wastewater analyses if the COD is less than 200 mg/L. Verify measurement results with sample dilutions or standard additions.

Nitrite concentrations of more than 2.0 mg/L interfere (high-bias results). Add 50 mg of sulfamic acid (amidosulfonic acid) to 5.0 mL of sample, dissolve and wait for 10 minutes. Analyze the prepared sample as described in the procedure above.

Table 2 Interfering substances

Interfering substance	Interference level
Cl^-, K^+, Na^+	500 mg/L
Ag^+	100 mg/L
Ca^{2+}, Cd^{2+}, Cu^{2+}, Fe^{3+}, Ni^{2+}, Pb^{2+}, Sn^{2+}, Zn^{2+}	50 mg/L
Co^{2+}, Fe^{2+}	10 mg/L
Cr^{6+}	5 mg/L
NO_2^-	2 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:

- Nitrate-Nitrogen Standard Solution, 10.0 mg/L NO_3^-–N or Wastewater Influent Standard Solution, Mixed Parameter

1. Use the test procedure to measure the concentration of the standard solution.
2. Compare the expected result to the actual result.

   Note: *The factory calibration can be adjusted slightly with the standard 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

Nitrate ions in solutions that contain sulfuric and phosphoric acids react with 2,6-dimethylphenol to form 4-nitro-2,6-dimethylphenol. The measurement wavelength is 345 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
Nitrate LR TNTplus Reagent Set	1	25/pkg	TNT835

Required apparatus

Description	Quantity/test	Unit	Item no.
Pipet, adjustable volume, 0.1–1.0 mL	1	each	BBP078
Pipet tips, for 0.1–1.0 mL pipet	2	100/pkg	BBP079
Light shield, DR3800, DR2800, DR2700	1	each	LZV646
Light shield, DR3900	1	each	LZV849

Recommended standards

Description	Unit	Item no.
Nitrate Nitrogen Standard Solution, 10.0-mg/L NO_3–N	500 mL	30749
Nitrate Nitrogen Standard Solution 1000-mg/L NO_3–N	500 mL	1279249
Wastewater Influent Standard Solution, Mixed Parameter, for NH_3-N, NO_3-N, PO_4^{3-}, COD, SO_4^{2-}, TOC	500 mL	2833149

Optional reagents and apparatus

Description	Unit	Item no.
Filter membrane, 0.45 micron, 25 mm	100/pkg	2514101
Sampling bottle with cap, low density polyethylene, 500 mL	12/pkg	2087079
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	245032
Sulfamic Acid, 454 g	each	234401
Sulfuric Acid, concentrated, ACS	500 mL	97949
Test tube rack, polyethylene, for 13-mm OD vials, 90 holes	each	2497900
Water, deionized	4 L	27256

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