Nitrogen, Total

DOC316.53.01088

Persulfate Digestion Method

Method 10208

5 to 40 mg/L N (HR)

TNTplus™ 827

Scope and application: For 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 5000	—	—
DR 3900	—	LZV849
DR 3800, DR 2800	—	LZV646
DR 1900	9609900 or 9609800 (A)	—

Before starting

DR 3900, DR 3800, DR 2800: 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 3–12.
The sample temperature must be 15–25 °C (59–77 °F) for accurate results.
The recommended temperature for reagent storage is 15–25 °C (59–77 °F).
Important: Make sure to close each reagent bottle immediately after each use.
The 20-mm reaction tube can be used for 7 tests. After each use, clean the tube thoroughly with a brush and water, then rinse well with high-quality distilled water and let dry.
Use only high-quality deionized water or organic-free water for standards preparation, sample dilutions or reagent blanks.
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
Nitrogen, Total, HR TNTplus Reagent Set	1
DRB200 reactor with 20-mm wells	1
Pipet, adjustable volume, 1.0–5.0 mL	1

Items to collect (continued)

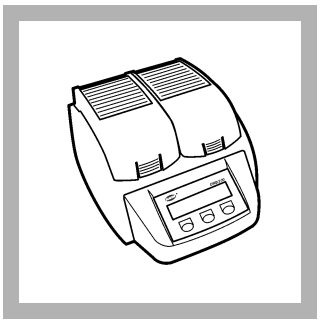
Description	Quantity
Pipet, adjustable volume, 0.2–1.0 mL	1
Pipet tips	1
Test tube rack	1

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

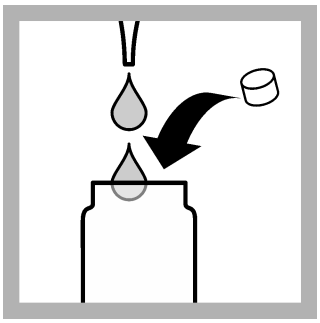
Sample collection and storage

- Collect samples in clean glass or plastic bottles.
- Analyze the samples as soon as possible for best results.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated sulfuric acid (approximately 2 mL per liter). No acid addition is necessary if the sample is tested immediately.
- Keep the preserved samples at or below 6 °C (43 °F) for a maximum of 28 days.
- 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.

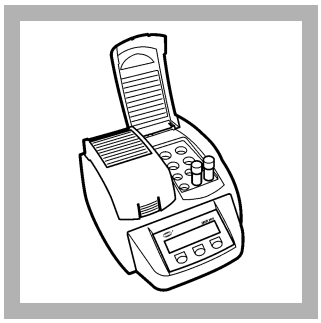
Test procedure



1. Set the DRB200 reactor power to on. Set the temperature to 120 °C.

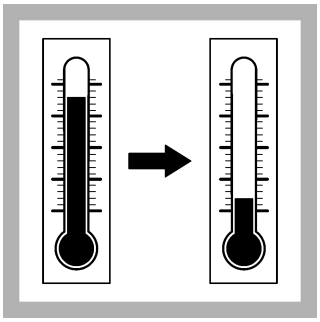


2. Add 0.5 mL of sample, 2.0 mL of Solution A and 1 Reagent B tablet in quick succession to a dry 20-mm reaction tube.
Close the reaction tube immediately. Do not invert.

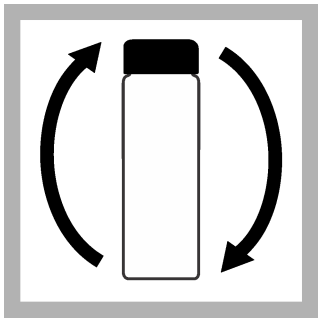


3. Insert the reaction tube in the preheated DRB200 reactor. Close the lid.

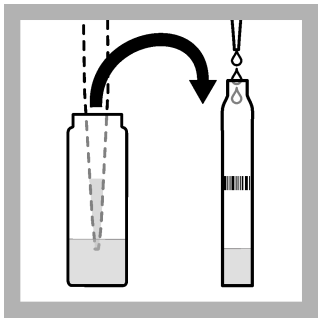
4. Keep the reaction tubes in the reactor for 30 minutes.



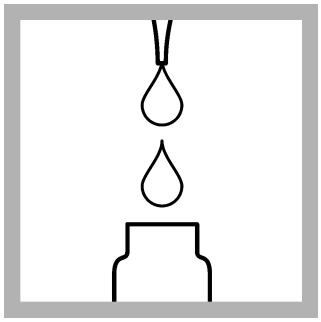
5. When the timer expires, carefully remove the reaction tube from the reactor. Let the temperature of the reaction tube decrease to room temperature.



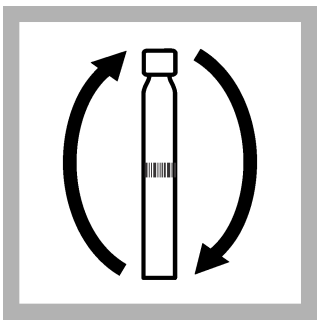
6. Invert the reaction tube 2–3 times.



7. Use a pipet to add 0.5 mL of the digested sample from the 20-mm reaction tube into a test vial.

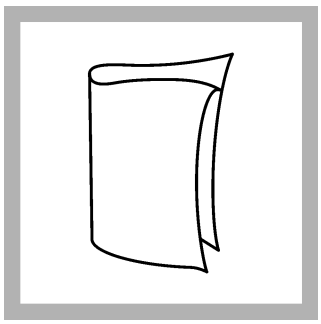


8. Use a pipet to add 0.2 mL of Solution D to the test vial.

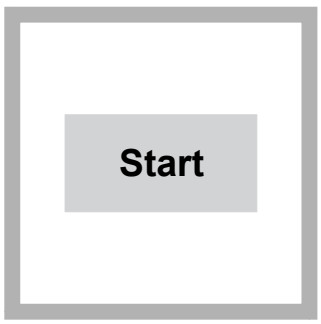


9. Quickly tighten the cap on the vial and invert until completely mixed.

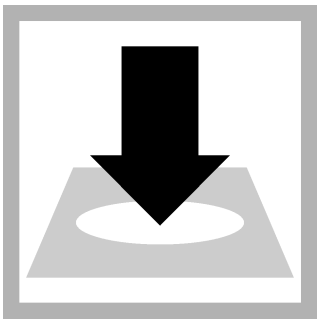
10. Start the reaction time of 15 minutes.



11. When the timer expires, clean the vial.



12. DR 1900 only: Select program 827. Refer to Before starting on page 1.



13. Insert the vial into the cell holder. DR 1900 only: Push **READ**.
Results show in mg/L 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.*

Interferences

A light pink color may develop during the reaction. This color does not interfere with the analysis. 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.

Table 2 Interfering substances

Interfering substance	Interference level
COD	1000 mg/L
Chloride	2000 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:

- Nitrogen, Ammonia Standard Solution, 10-mg/L NH_3–N

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

Inorganic and organic nitrogen compounds are digested with peroxodisulfate and oxidized to nitrate. The nitrate ions react with 2,6-dimethylphenol in a solution of sulfuric and phosphoric acid to form a nitrophenol. The measurement wavelength is 345 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
Nitrogen, Total, HR TNTplus Reagent Set	1	25/pkg	TNT827

Required apparatus

Description	Quantity/test	Unit	Item no.
DRB 200 Reactor, 115 VAC option, 9 x 13 mm + 2 x 20 mm, 1 block	1	each	DRB200-01
DRB 200 Reactor, 230 VAC option, 9 x 13 mm + 2 x 20 mm, 1 block	1	each	DRB200-05
Pipet, adjustable volume, 1.0–5.0 mL	1	each	BBP065
Pipet tips, for 1.0–5.0 mL pipet	1	75/pkg	BBP068
Pipet, adjustable volume, 0.2–1.0 mL	1	each	BBP078
Pipet tips, for 0.2–1.0 mL pipet	2	100/pkg	BBP079
Test tube rack for 20-mm vials, 20 holes	1	each	2497912

Required apparatus (continued)

Description	Quantity/test	Unit	Item no.
Test tube rack for 20-mm vials, 40 holes	1	each	2497902
Light shield, DR 3800, DR 2800, DR 2700	1	each	LZV646
Light shield, DR 3900	1	each	LZV849

Recommended standards

Description	Unit	Item no.
Nitrogen Ammonia Standard Solution, 10-mg/L NH_3–N	500 mL	15349

Optional reagents and apparatus

Description	Unit	Item no.
Brush, test tube	each	69000
Sampling bottle with cap, low density polyethylene, 500-mL	12/pkg	2087079
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	245032
Sulfuric Acid, concentrated, ACS	500 mL	97949
Water, deionized	4 L	27256
Water, organic-free	500 mL	2641549

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