Nitrogen, Simplified TKN (s-TKN™)

DOC316.53.01258

s-TKN[™] Method Method 10242^{1,2} 1 to 16 mg/L TKN TNTplus[®] 880

Scope and application: For water and wastewater. Digestion is required.

- Revision 1.2, March 2022
- ² This method is USEPA equivalent under 40 CFR 136.6 to EPA Method 351.2 and Hach Method 10242, Revision 1.1, January 10, 2013 as published at 40 CFR part 136.



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 sample temperature must be 15–25 $^{\circ}\text{C}$ (59–77 $^{\circ}\text{F}) for accurate results.$

Set the reactor temperature to 120 °C (248 °F). Keep the reactor tube in the reactor for 30 minutes for digestion. Lower temperatures or shorter digestion time will give incorrect results.

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

Samples must be preserved with acid as specified in Sample collection and storage on page 2. Make sure to adjust the pH and temperature before the analysis.

The sample pH must be 3-10 for accurate results.

A small amount of turbidity does not interfere. If there is a large amount of turbidity, let the turbidity settle or filter samples through a 0.45-µm filter.

Sodium Hydroxide Solution A and Oxidant Tablet B—Make sure to close each 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.

The nitrite concentration can be determined with nitrite reagents on samples that have not been preserved. Identify the nitrite concentration (TNT839/TNT840) and subtracted the value from the s-TKN result.

The total nitrogen concentration must be between 1 and 16 mg/L N. The combined nitrate/nitrite concentration must be between 0.23 and 13.5 mg/L N. Dilute the sample if necessary.

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

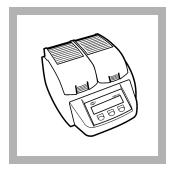
Description	Quantity
s-TKN TNTplus [®] Reagent Set	1
DRB200 reactor with 20-mm wells	1
Pipet, adjustable volume, 1.0–5.0 mL	1
Pipet, adjustable volume, 0.1–1.0 mL	1
Pipet tips	1
Test tube rack	1

Refer to Consumables and replacement items on page 5 for order information.

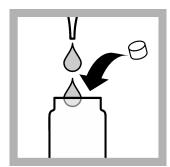
Sample collection and storage

- Collect samples in clean glass or plastic bottles.
- Adjust the sample pH to less than 2 with concentrated sulfuric acid (approximately 2 mL per liter).
- 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 (248 °F).



2. Add 1.3 mL of sample, 1.3 mL of Solution A and 1 Reagent B tablet in quick succession to a dry 20-mm reaction tube.

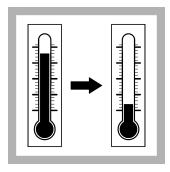
Close the reaction tube immediately. Do not invert.



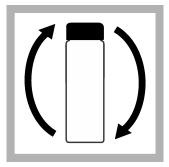
3. Insert the reaction tube in the preheated DRB200 reactor. Close the lid.



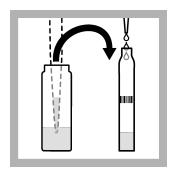
4. Keep the reaction tube in the reactor for 30 minutes.



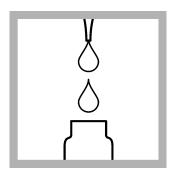
5. When the timer expires, carefully remove the reaction tube from the reactor. Let the temperature of the reaction tube decrease to room temperature.



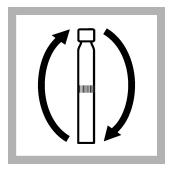
6. Invert the reaction tube 2-3 times.



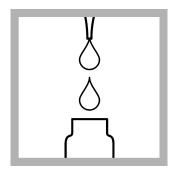
7. Use a pipet to slowly add 0.5 mL of the digested sample from the 20-mm reaction tube into a test vial 1 (red label).



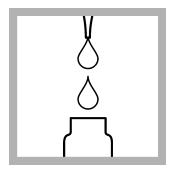
8. Use a pipet to slowly add 0.2 mL of Solution D to test vial 1 (red label).



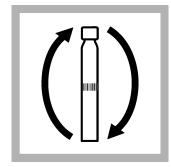
9. Quickly tighten the cap on the vial and invert until completely mixed. Immediately continue to the next step.



10. Use a pipet to slowly add 1.0 mL of undigested sample to a test vial 2 (green label).



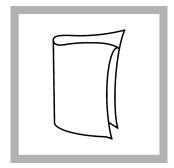
11. Use a pipet to slowly add 0.2 mL of Solution D to test vial 2 (green label).



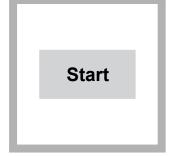
12. Quickly tighten the cap on the vial and invert until completely mixed.



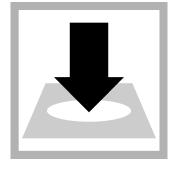
13. Start the reaction time of 15 minutes.



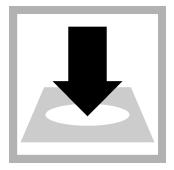
14. When the timer expires, clean the vials.



15. DR1900 only: Select program 880. Refer to Before starting on page 1.



16. Insert the test vial 1 (red label) into the cell holder. DR1900 only: Push **READ1**. Immediately continue to the next step.



17. Insert the test vial 2 (green label) into the cell holder. DR1900 only: Push READ2.
Results show in mg/L Total N, mg/L NO₃–N + NO₂–N and mg/L TKN.

Interferences

High levels of oxidizable organic substances (COD) have an effect on the reagent color and give high results. Use this test procedure for wastewater only when the COD level is less than 500 mg/L COD. A light pink color may develop during the reaction. The pink color does not cause interference 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 Non-interfering substances

Interfering substance	Interference level
Cd ²⁺	50 mg/L
Ca ²⁺	50 mg/L
CI ⁻	500 mg/L
Cr ⁶⁺	5 mg/L
Co ²⁺	10 mg/L
Cu ²⁺	50 mg/L
Fe ²⁺	10 mg/L
Fe ³⁺	50 mg/L
Pb ²⁺	50 mg/L
Ni ²⁺	50 mg/L
NO ₂ -	2 mg/L
K ⁺	500 mg/L
Ag ⁺	100 mg/L
Na ⁺	500 mg/L
Sn ²⁺	50 mg/L
Zn ²⁺	50 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:

- Wastewater Effluent Standard Solution, Mixed Parameter (expected result: 7.56 mg/L Total N, 4 mg/L NO₃–N + NO₂–N, 3.56 mg/L TKN)
- **1.** Use the test procedure to measure the concentration of the standard solution.
- 2. Compare the expected result to the actual result. The Wastewater Effluent Standard Solution contains a component that adds 1.56 mg/L N to the Total N and TKN values. This is in addition to the 2 mg/L NH₃-N and 4 mg/L NO₃-N shown on the label.

Summary of Method

Total Kjeldahl Nitrogen (TKN) is the sum of organic nitrogen and ammonia. In the simplified TKN method, inorganic and organic nitrogen are oxidized to nitrate by digestion with peroxodisulfate. The nitrate ions react with 2,6-dimethylphenol in a solution of sulfuric and phosphoric acid to form a nitrophenol. Oxidized forms of nitrogen in the original sample (nitrite + nitrate due to sample preservation) are determined in the second test vial and then subtracted, which results in TKN.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
Simplified TKN (s-TKN) TNTplus® reagent set	1	25/pkg	TNT880

Required apparatus

Description	Quantity/test	Unit	Item no.
DRB200 Reactor, 115 VAC option, 9 x 13 mm + 2 x 20 mm, 1 block	1	each	DRB200-01
DRB200 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, 0.1–1.0 mL	1	each	BBP078
Pipet tips, for 0.1–1.0 mL pipet	2	100/pkg	BBP079
Test tube rack for 20-mm vials, 20 holes	1	each	2497912
Test tube rack for 20-mm vials, 40 holes	1	each	2497902
Light shield, DR3800, DR2800, DR2700	1	each	LZV646
Light shield, DR3900	1	each	LZV849

Recommended standards

Description	Unit	Item no.
Wastewater Effluent Standard Solution, Mixed Parameter, for NH ₃ -N, NO ₃ -N, PO ₄ ³⁻ , COD, SO ₄ ²⁻ , TOC	500 mL	2833249

Optional reagents and apparatus

Description	Unit	Item no.
Filter membrane, 0.45 micron, 25 mm	100/pkg	2514101
Filter holder, 25-mm, for Luer-type syringe	each	246800
Sampling bottle with cap, low density polyethylene, 500 mL	12/pkg	2087079
Sodium Hydroxide Standard Solution, 5.0 N	1 L	245053

Optional reagents and apparatus (continued)

Description	Unit	Item no.
Sulfuric Acid, concentrated, ACS	500 mL	97949
Syringe, 10 cc, Luer-Lock tip	each	2202400
Digestion vials, 20 mm	5/pkg	LZP065