

**s-TKN™ Method**  
**0 to 16 mg/L TKN**

**Method 10242<sup>1</sup>**  
**TNTplus® 880**

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

<sup>1</sup> USEPA approved for water and wastewater analysis, 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 °C (59–77 °F) for accurate 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.

Important: Make sure to close each reagent bottle immediately after each use.

The 20-mm reaction tube can be used for 13 tests. After each use, clean the tube thoroughly with a brush and water, then rinse well with high-quality distilled water and let dry.

If a large amount of turbidity forms after the addition of MicroCap C, let the turbidity settle, then go to the next step. A small amount of turbidity does not interfere.

The nitrite concentration can be determined with nitrite reagents on samples that have not been preserved. The nitrite concentration must then be subtracted 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

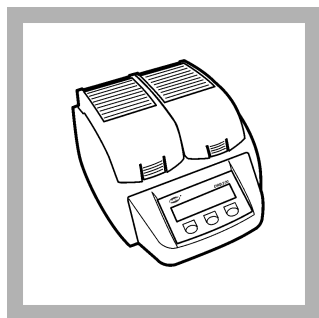
Description	Quantity
s-TKN TNTplus <sup>®</sup> Reagent Set	1
DRB200 reactor with 20-mm wells	1
Pipet, adjustable volume, 1.0–5.0 mL	1
Pipet, adjustable volume, 0.2–1.0 mL	1
Pipet tips	1
Test tube rack	1

Refer to [Consumables and replacement items](#) on page 5 for order information.

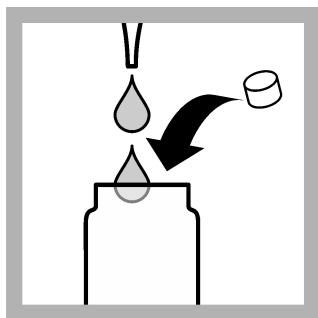
## Sample collection and storage

- 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.

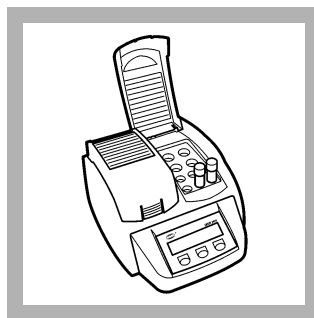
## Test procedure



**1.** Set the DRB200 reactor power to on. Set the temperature to 100 °C.



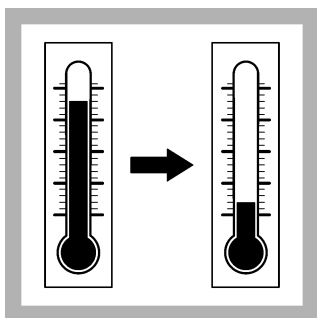
**2.** Add 1.3 mL of sample, 1.3 mL of Solution A and 1 Reagent B tablet in quick succession to a dry 20-mm reaction tube. Close the reaction tube immediately. Do not invert.



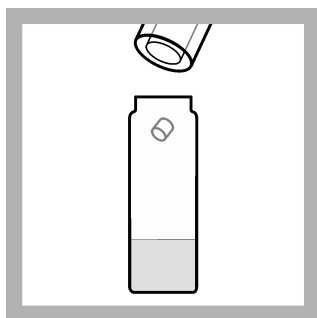
**3.** Insert the reaction tube in the preheated DRB200 reactor. Close the lid.



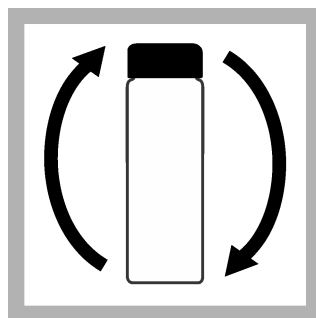
**4.** Keep the reaction tube in the reactor for 1 hour.



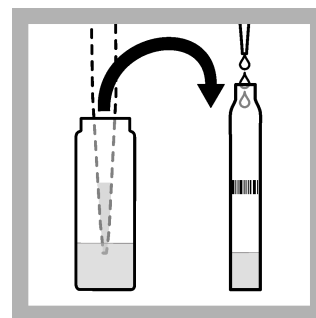
**5.** When the timer expires, carefully remove the reaction tube from the reactor. Let the temperature of the reaction tube decrease to room temperature.



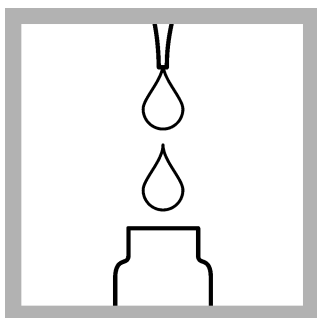
**6.** When cool, add 1 Micro Cap C to the reaction tube.



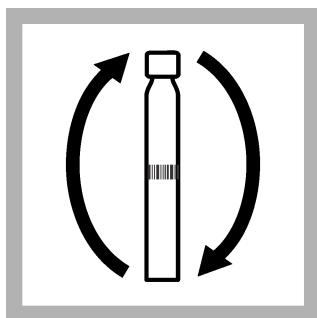
**7.** Tighten the cap on the reaction tube and invert until completely mixed.



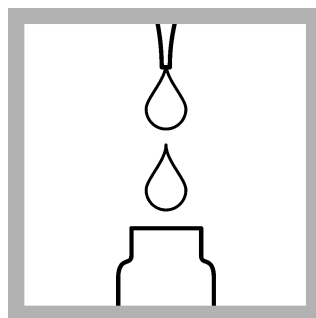
**8.** Use a pipet to add 0.5 mL of the digested sample from the 20-mm reaction tube into a test vial 1 (red label).



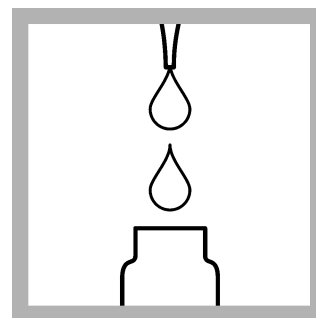
**9.** Use a pipet to add 0.2 mL of Solution D to the test vial.



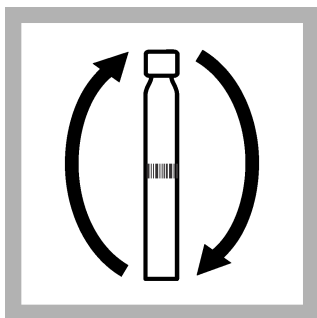
**10.** Quickly tighten the cap on the vial and invert until completely mixed. Immediately continue to the next step.



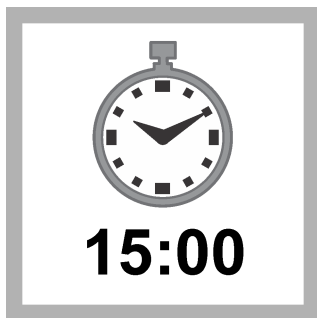
**11.** Use a pipet to add 1.0 mL of undigested sample to a test vial 2 (green label).



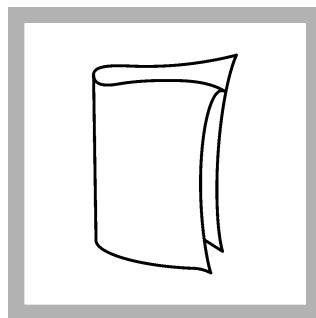
**12.** Use a pipet to add 0.2 mL of Solution D to the test vial.



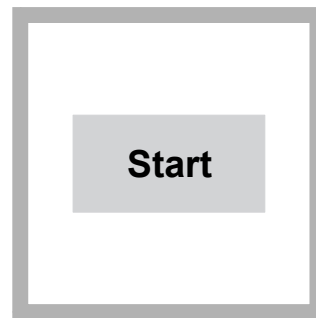
**13.** Quickly tighten the cap on the vial and invert until completely mixed.



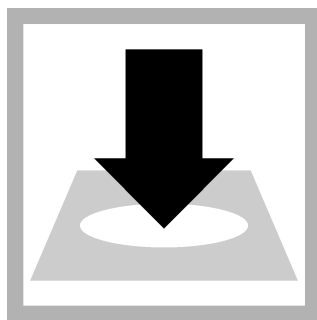
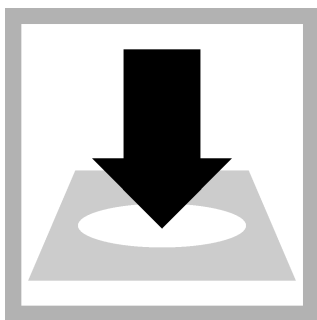
**14.** Start the reaction time of 15 minutes.



**15.** When the timer expires, clean the vials.



**16.** DR1900 only: Select program 880. Refer to [Before starting](#) on page 1.



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

**18.** Insert the test vial 2 (green label) into the cell holder. DR1900 only: Push **READ2**.  
Results show in mg/L Total N, mg/L  $\text{NO}_3\text{-N}$  +  $\text{NO}_2\text{-N}$  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.

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
$\text{Cd}^{2+}$	50 mg/L
$\text{Ca}^{2+}$	50 mg/L
$\text{Cl}^-$	500 mg/L
$\text{Cr}^{6+}$	5 mg/L
$\text{Co}^{2+}$	10 mg/L
$\text{Cu}^{2+}$	50 mg/L
$\text{Fe}^{2+}$	10 mg/L
$\text{Fe}^{3+}$	50 mg/L
$\text{Pb}^{2+}$	50 mg/L
$\text{Ni}^{2+}$	50 mg/L
$\text{NO}_2^-$	2 mg/L
$\text{K}^+$	500 mg/L
$\text{Ag}^+$	100 mg/L
$\text{Na}^+$	500 mg/L
$\text{Sn}^{2+}$	50 mg/L
$\text{Zn}^{2+}$	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 NO<sub>3</sub>-N + NO<sub>2</sub>-N, 3.56 mg/L TKN)
- Use the test procedure to measure the concentration of the standard solution.
  - 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 NH<sub>3</sub>-N and 4 mg/L NO<sub>3</sub>-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 <sup>®</sup> 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, 0.2–1.0 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
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 <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> <sup>3-</sup> , COD, SO <sub>4</sub> <sup>2-</sup> , 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

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**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



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