# Nitrogen, Ammonia

# Salicylate Method

TNTplus<sup>™</sup> 831

**Method 10205** 

Scope and application: For surface waters, municipal and industrial wastewaters.



**Test preparation** 

1 to 12 mg/L  $NH_3$ -N (LR)

# 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 4-8.

The temperature of the samples and reagents must be 20–23 °C (68–73 °F) for accurate results.

The recommended temperature for reagent storage is 2-8 °C (35-46 °F).

Analyze the samples as soon as possible for best results.

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

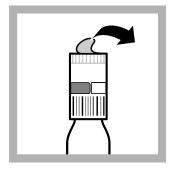
Description	Quantity
Nitrogen, Ammonia LR TNTplus Reagent Set	1
Pipet, adjustable volume, 0.2–1.0 mL	1
Pipet tips, for 0.2–1.0 mL pipet	1

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

# Sample collection and storage

- Collect samples in clean glass or plastic bottles.
- 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 hydrochloric acid. 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.

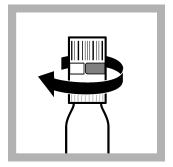
# **Test procedure**



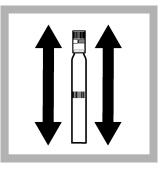
 Carefully remove the lid from the DosiCap<sup>™</sup> Zip cap. Remove the cap from the test vial.



**2.** Use a pipet to add 0.5 mL of sample to the test vial. Immediately continue to the next step.



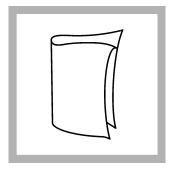
**3.** Turn the DosiCap Zip over so that the reagent side goes on the test vial. Tighten the cap on the vial.



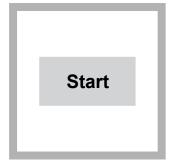
**4.** Shake the vial 2–3 times to dissolve the reagent in the cap. Look through the open end of the DosiCap to make sure that the reagent has dissolved.



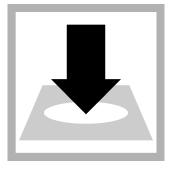
**5.** Start the reaction time of 15 minutes.



6. Clean the vial.



**7.** DR1900 only: Select program 831. Refer to Before starting on page 1.



**8.** Insert the vial into the cell holder. DR1900 only: Push **READ**. Results show in NH<sub>3</sub>–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 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.

Primary amines are found and cause high-bias results. A 10,000-fold excess of urea does not interfere. All reducing agents interfere and cause low-bias results.

**Note:** An analyte concentration that is larger than the stated range adversely has an effect on color formation, which results in a false reading within the method range.

Verify measurement results with sample dilutions or standard additions.

Distillation is necessary for samples with severe interferences. Complete the distillation procedure with the Hach General Purpose Distillation Set.

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Interfering substance	Interference level
CI <sup>-</sup> , SO <sub>4</sub> <sup>2-</sup>	1000 mg/L
K <sup>+</sup> , Na <sup>+</sup> , Ca <sup>2+</sup>	500 mg/L
CO <sub>3</sub> <sup>2-</sup> , NO <sub>3</sub> <sup>-</sup> , Fe <sup>3+</sup> , Cr <sup>3+</sup> , Cr <sup>6+</sup> , Zn <sup>2+</sup> , Cu <sup>2+</sup> , Co <sup>2+</sup> , Ni <sup>2+</sup> , Hg <sup>2+</sup>	50 mg/L
Fe <sup>2+</sup>	25 mg/L
Sn <sup>2+</sup>	10 mg/L
Pb <sup>2+</sup>	5 mg/L
Ag <sup>+</sup>	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:

- Nitrogen, Ammonia Standard Solution, 10-mg/L NH<sub>3</sub>-N or Wastewater Effluent 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 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**

Ammonium ions react at pH 12.6 with hypochlorite ions and salicylate ions in the presence of sodium nitroprusside as a catalyst to form indophenol. The amount of color formed is directly proportional to the ammonia nitrogen present in the sample. The measurement wavelength is 694 nm.

# Pollution prevention and waste management

The ammonia salicylate reagent contains sodium nitroferricyanide which, when digested, is converted to total cyanide and can have an effect on total cyanide limits in the effluent. Dispose of reacted solutions according to local, state and federal regulations.

# Consumables and replacement items

### Required reagents

Description	Quantity/Test	Unit	Item no.
Nitrogen, Ammonia LR TNTplus Reagent Set	1	25/pkg	TNT831

### 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
Light shield, DR3800, DR2800, DR2700	1	each	LZV646
Light shield, DR3900	1	each	LZV849

# **Recommended standards**

Description	Unit	Item no.
Nitrogen Ammonia Standard Solution, 10-mg/L NH <sub>3</sub> -N	500 mL	15349
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.
MICRO DIST <sup>®</sup> distillation block, digital, 110 VAC	each	A17102
OR		
DRB 200 Reactor, 115 VAC option, 8 x 20 mm + 12 x 13 mm, 1 block		DRB20004
DRB 200 adapter sleeves for MICRO DIST®	8/pkg	LZT144
MICRO DIST <sup>®</sup> tubes, user fill option for Ammonia	50/pkg	A17517A
MICRO DIST <sup>®</sup> tubes, user fill option for Ammonia	100/pkg	A17117A
Hydrochloric Acid, concentrated	500 mL	13449
Sampling bottle with cap, low density polyethylene, 500 mL	12/pkg	2087079
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	245032
Test tube rack, polyethylene, for 13-mm OD vials, 90 holes	each	2497900
Water, deionized	4 L	27256



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