Nitrogen, Total Inorganic

DOC316.53.01090

Titanium Trichloride Reduction Method

Method 10021

0.2 to 25.0 mg/L N

Test 'N Tube™ Vials

Scope and application: For water, wastewater and seawater.

Test preparation

Instrument-specific information

Table 1 shows all of the instruments that have the program for this test. The table also shows adapter and light shield requirements for the instruments that use them.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information for test tubes

Instrument	Adapters	Light shield
DR 6000, DR 5000	—	—
DR 3900	—	LZV849
DR 3800, DR 2800, DR 2700	—	LZV646
DR 1900	9609900 (D[1])	—
DR 900	4846400	Cover supplied with the instrument

[1] The D adapter is not available with all instrument versions.

Before starting

Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.
DR 3900, DR 3800, DR 2800 and DR 2700: Install the light shield in Cell Compartment #2 before this test is started.
For safety, wear gloves to break open the reagent ampules.
The reagents that are used in this test contain sodium nitroferricyanide. **Keep cyanide solutions at pH > 11 to prevent exposure to hydrogen cyanide gas.** Collect the reacted samples for proper disposal.
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
Total Inorganic Nitrogen Pretreatment Reagent Set ($TiCl_3$ Reduction Method)	1
Test 'N Tube AmVer™ Nitrogen-Ammonia Reagent Set	1
Centrifuge	1
Funnel, micro	1
Light shield or adapter (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	1
Pipet, TenSette®, 0.1- to 1.0-mL, with pipet tips	1

Items to collect (continued)

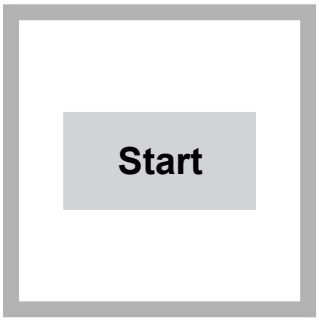
Description	Quantity
Pipet, volumetric, Class A, 1.00-mL	1
Test tube rack	1
Water, deionized	1 mL

Refer to Consumables and replacement items on page 6 for order information.

Sample collection and storage

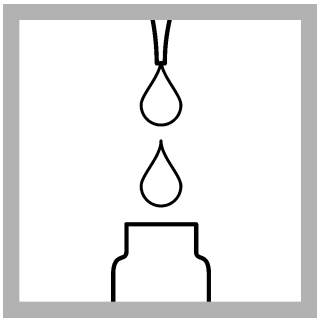
- Collect samples in clean glass or plastic bottles.
- Analyze the samples as soon as possible for best results.
- If the sample contains chlorine, add one drop of 0.1 N sodium thiosulfate to 1 liter of sample to remove each 0.3 mg/L of chlorine.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated hydrochloric acid (about 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 5.0 N sodium hydroxide standard solution.
- Correct the test result for the dilution caused by the volume additions.

Test 'N Tube procedure

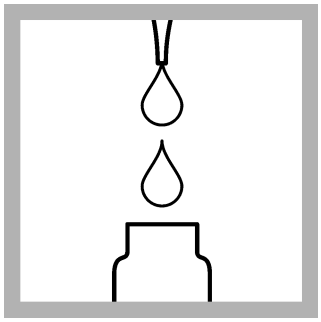


1. Start program **346 N Inorganic TNT**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

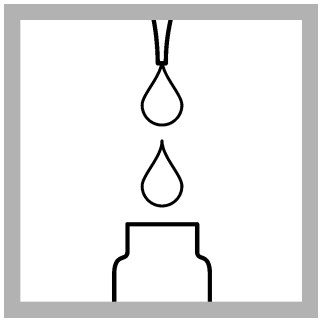
Note: *Although the program name can be different between instruments, the program number does not change.*



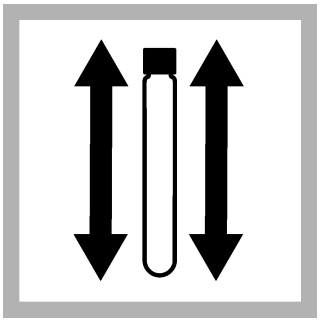
2. Add 1 mL of Total Inorganic Nitrogen Pretreatment Base concentrate into two Total Inorganic Nitrogen Pretreatment Diluent vials.



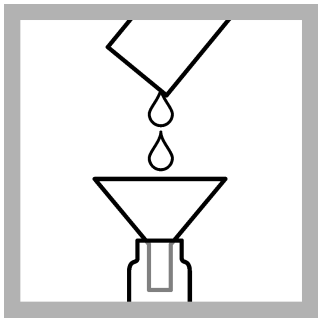
3. Prepare the sample: Add 1 mL of sample to one of the vials.



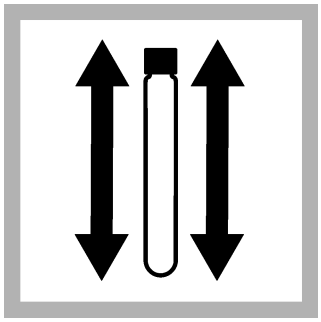
4. Prepare the blank: Add 1 mL of deionized water to the second vial.



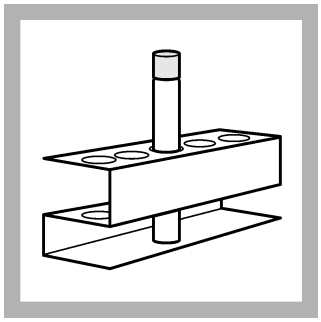
5. Put the caps on both vials. Shake for 30 seconds to mix.



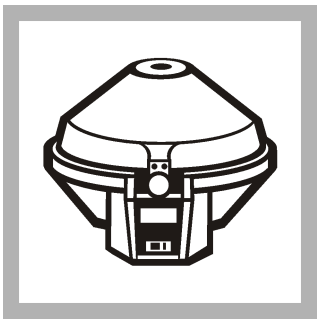
6. Add the contents of one Total Inorganic Nitrogen Reductant ampule to the prepared sample vial. Add the contents of another Total Inorganic Nitrogen Reductant ampule to the blank vial.
A black precipitate will form immediately.



7. Put the caps on both vials. Shake gently for at 30 seconds to mix.
The precipitate should be black after shaking. Excessive shaking will cause the precipitate to change to a white color and give low test results.



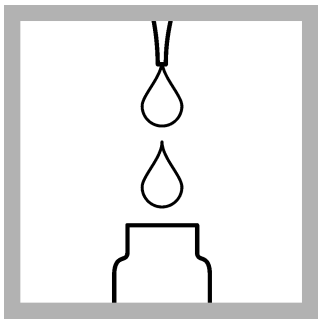
8. Let the vials stand for at least one minute.



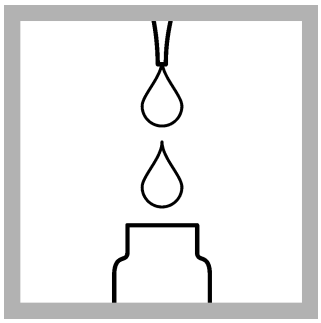
9. Put the vials in a centrifuge.

10. Start the instrument timer. A 3-minute reaction time starts.

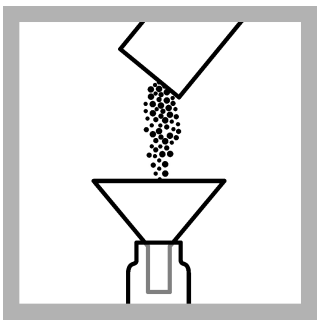
Put the vials in a centrifuge and run the centrifuge to for three minutes. If no centrifuge is available, let the vials sit for 30 minutes so the solids settle at the bottom of the vials.



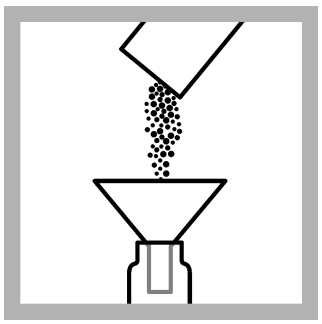
11. After the timer expires, add 2 mL of prepared sample from the centrifuge to an AmVer Diluent Reagent Test 'N Tube for Low Range Ammonia Nitrogen.
Do not disturb the sediment in the bottom of the vials.



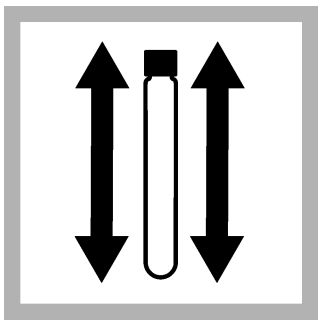
12. Add 2 mL of blank from the centrifuge to a second AmVer Diluent Reagent Test 'N Tube for Low Range Ammonia Nitrogen.



13. Add the contents of one Ammonia Salicylate Reagent Powder Pillow (for 5-mL samples) to each vial.

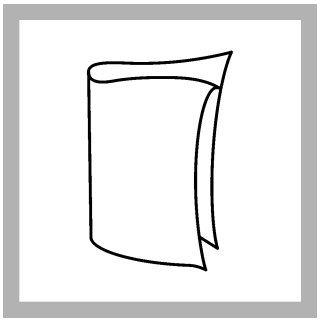


14. Add the contents of one Ammonia Cyanurate Reagent Powder Pillow)for 5-mL samples) to each vial.

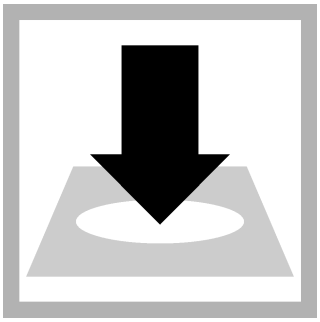


15. Put the caps on both vials. Shake to dissolve the powder completely. A green color shows if nitrogen is present.

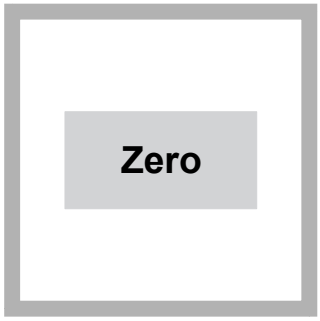
16. Start the instrument timer. A 20-minute reaction time starts.



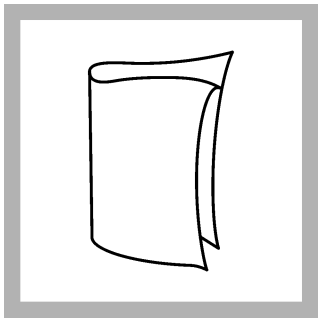
17. When the timer expires, clean the blank vial.



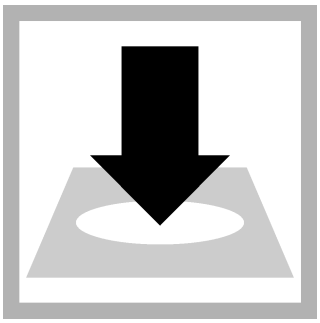
18. Insert the blank vial into the 16-mm cell holder.



19. Push **ZERO**. The display shows 0.0 mg/L N.



20. Clean the sample vial.



21. Insert the sample vial into the 16-mm cell holder.

22. Push **READ**. Results show in mg/L N.

Interferences

The substances in Table 2 may interfere when present. The substances in Table 3 do not interfere below the levels listed.

Table 2 Interfering substances

Interfering substance	Interference level
Calcium	Causes a positive interference at 1000 mg/L as $CaCO_3$
Manganese (IV)	Causes a negative interference at 3 mg/L
Magnesium	Causes a positive interference at 1000 mg/L as $CaCO_3$
Sulfide	Causes a negative interference at 3 mg/L
Sulfate	Causes a negative interference at 250 mg/L

Table 3 Non-interfering substances

Interfering substance	Interference level
Al^{3+}	8 mg/L
Ba^{2+}	40 mg/L
Cu^{2+}	40 mg/L
Fe^{3+}	8 mg/L
Zn^{2+}	80 mg/L
F^-	40 mg/L
PO_4^{3-}-P	8 mg/L
SiO_2	80 mg/L
EDTA	80 mg/L

Accuracy check

Standard additions method (sample spike)

Use the standard additions method (for applicable instruments) to validate the test procedure, reagents and instrument and to find if there is an interference in the sample.

Items to collect:

- Nitrate Nitrogen PourRite Ampule Standard, 500-mg/L NO_3^-–N
- Ampule breaker
- Pipet, TenSette®, 0.1–1.0 mL and tips
- 25-mL mixing cylinders (3)

1. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
2. Go to the Standard Additions option in the instrument menu.
3. Select the values for standard concentration, sample volume and spike volumes.
4. Open the standard solution.
5. Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the standard solution, respectively, to three 25-mL portions of fresh sample. Mix well.
6. Use the test procedure to measure the concentration of each of the spiked samples. Start with the smallest sample spike. Measure each of the spiked samples in the instrument.
7. Select **Graph** to compare the expected results to the actual results.

   Note: *If the actual results are significantly different from the expected results, make sure that the sample volumes and sample spikes are measured accurately. The sample volumes and sample spikes that are used should agree with the selections in the standard additions menu. If the results are not within acceptable limits, the sample may contain an interference.*

Standard solution method

Use the standard solution method to validate the test procedure, the reagents and the instrument.

Items to collect:

- 10.0-mg/L Nitrate Nitrogen Standard Solution

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 slight variations in the reagents or instruments.*

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a spectrophotometer during ideal test conditions. Users can get different results under different test conditions.

Program	Standard	Precision (95% confidence interval)	Sensitivity Concentration change per 0.010 Abs change
346	20.0 mg/L NO_3^-–N	19.6–20.4 mg/L NO_3^-–N	0.2 mg/L NO_3^-–N

Species recovery

The total inorganic nitrogen test is designed to provide an estimate of the total nitrite, nitrate and ammonia nitrogen load in a water or wastewater sample. This test is most applicable to the monitoring of samples taken from an industrial process stream or a wastewater treatment stream where it is important to track the inorganic nitrogen load as it passes through the treatment process. The test does show different recoveries of each

of the three nitrogen species, as shown in Table 4. The test is not recommended for use when quantifying only one of the three species. In that case, specific procedures for each particular analyte would be more appropriate.

Table 4 Species recovery

Nitrogen form	Percent recovery
NH_3–N	112%
NO_3^-–N	100%
NO_2^-–N	77%

Summary of method

Titanium (III) ions reduce nitrate and nitrite to ammonia in a basic environment. After centrifugation to remove solids, the ammonia is combined with chlorine to form monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate. The 5-aminosalicylate is oxidized in the presence of a sodium nitroprusside catalyst to form a blue colored compound. The blue color is masked by the yellow color from the excess reagent present to give a final green colored solution. The measurement wavelength is 655 nm for spectrophotometers or 610 nm for colorimeters.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Nitrogen, Total Inorganic, Pretreatment Reagent Set (TiCl3 Reduction Method)	—	25 tests	2604945
Nitrogen Ammonia, Reagent Set, Low Range Test 'N Tube™ AmVer™	2	25 tests	2604545
Water, deionized	varies	100 mL	27242

Required apparatus

Description	Quantity/test	Unit	Item no.
Centrifuge, 115 VAC, 6 x 15 mL	1	each	2676500
OR			
Centrifuge, 220 VAC, 6 x 15 mL	1	each	2676502
Funnel, micro, poly	1	each	2584335
Pipet, TenSette®, 0.1–1.0 mL	1	each	1970001
Pipet Tips, for TenSette® Pipet, 0.1–1.0 mL	2	50/pkg	2185696
Test tube rack	1	each	1864100
Gloves, nitrile, large (other sizes available)	1 pair	100/pkg	2550503

Recommended standards and apparatus

Description	Unit	Item no.
Flask, volumetric, 50-mL	each	1457441
Nitrate Nitrogen Standard Solution, 10.0-mg/L NO_3–N	500 mL	30749
Nitrate Nitrogen Standard Solution, 2-mL PourRite® Ampule, 500 mg/L	20/pkg	1426020
Pipet filler, safety bulb	each	1465100
Pipet, TenSette®, 0.1–1.0 mL	each	1970001

Recommended standards and apparatus (continued)

Description	Unit	Item no.
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	50/pkg	2185696
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	1000/pkg	2185628
Pipet tips for TenSette® Pipet, 1.0–10.0 mL	250/pkg	2199725
Water, deionized	4 L	27256

Optional reagents and apparatus

Description	Unit	Item no.
Mixing cylinder, graduated, 25-mL	each	2088640
Hydrochloric Acid, concentrated	500 mL	13449
Sodium Hydroxide Solution, 5 N	50 mL	245026
Sodium Thiosulfate, 0.1 N	100 mL	32332
Pipet, volumetric, Class A, 1.00-mL	each	1451535
PourRite® Ampule Breaker, 2-mL	each	2484600
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800
Nitrate Nitrogen Standard Solution, 1-mg/L NH_3-N	500 mL	204649
Nitrate Nitrogen Standard Solution, 100-mg/L NH_3-N	500 mL	194749
Nitrate Nitrogen Standard Solution, 1000-mg/L NH_3-N	500 mL	1279249
Nitrate Nitrogen Standard Solution, 15-mg/L NH_3-N	100 mL MDB	2415132

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