Nitrogen, Ammonia

DOC316.53.01079

Salicylate Method

Method 10031

0.4 to 50.0 mg/L NH_3–N (HR)

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 the adapter and light shield requirements for the applicable instruments that can use Test 'N Tube 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 Test 'N Tube vials

Instrument	Adapters	Light shield
DR6000, DR5000	—	—
DR3900	—	LZV849
DR3800, DR2800, DR2700	—	LZV646
DR1900	9609900 (D[1])	—
DR900	4846400	Cover supplied with the instrument

Before starting

Install the instrument cap on the DR900 cell holder before ZERO or READ is pushed.
DR3900, DR3800, DR2800 and DR2700: Install the light shield in Cell Compartment #2 before this test is started.
Small sample sizes (such as 0.1 mL) may not be representative of the entire sample. Mix the sample well before the test or use a different portion of the sample to repeat the test.
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 safe disposal.
Keep the samples sealed at all times to prevent ammonia contamination from the air.
To prevent airborne cross-contamination of the blank, complete the preparation of the blank before samples and standards are opened. If the sample or standard containers are open, move to a separate area of the lab to prepare the blank.
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
Light shield (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	1
High Range Test 'N Tube™ AmVer® 3 Nitrogen Ammonia Reagent Set	2

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

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Items to collect (continued)

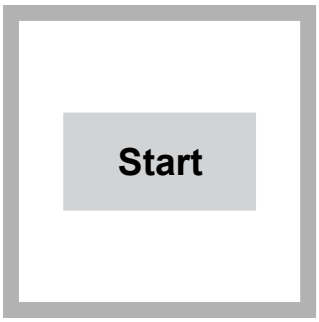
Description	Quantity
Funnel, micro (for reagent addition)	1
Pipet, TenSette® 0.1 to 1.0 mL, with tips	varies

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

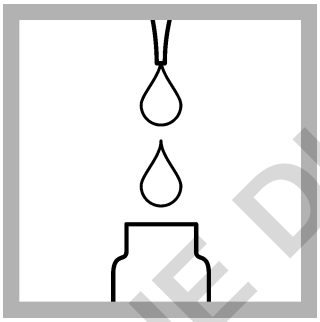
Sample collection and storage

- Collect samples in clean glass or plastic bottles.
- Analyze the samples as soon as possible for best results.
- If chlorine is known to be present, add one drop of 0.1 N Sodium Thiosulfate to 1 L of sample for each 0.3 mg/L Cl_2.
- To preserve samples for later analysis, adjust the sample pH to less than 2. No pH adjustment 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.

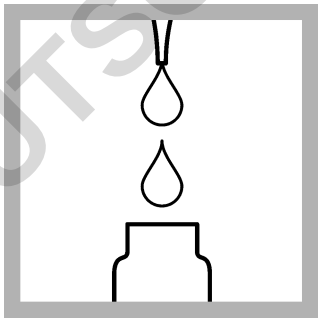
Test 'N Tube procedure



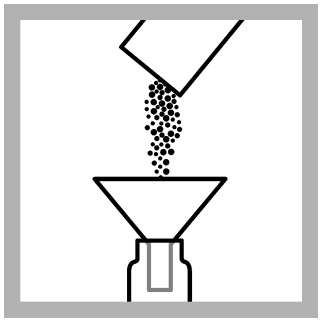
1. Start program **343 N, Ammonia HR TNT**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.



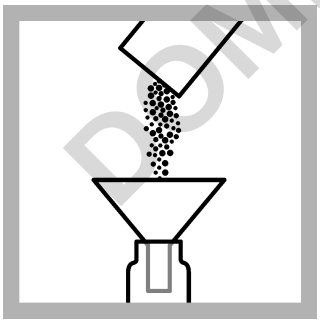
2. Prepare the blank: Add 0.1 mL of **ammonia-free water** to one AmVer™ Diluent Reagent Test 'N Tube for High Range Ammonia Nitrogen.



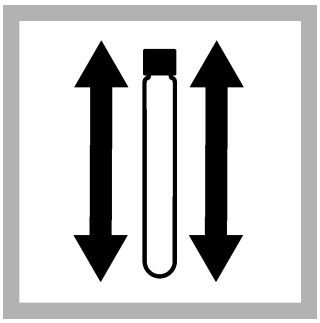
3. Prepare the sample: Add 0.1 mL of **sample** to one AmVer™ Diluent Reagent Test 'N Tube for High Range Ammonia Nitrogen.



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



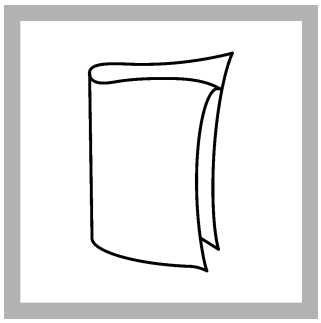
5. Add the contents of one Ammonia Cyanurate Reagent Powder Pillow to each vial.



6. Put the caps on both vials. Shake thoroughly to dissolve the powder.

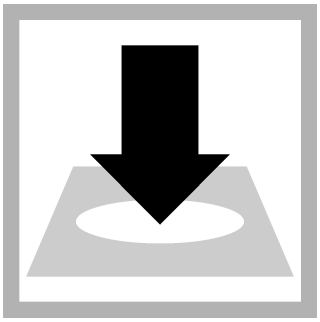


7. Start the instrument timer. A 20-minute reaction time starts.

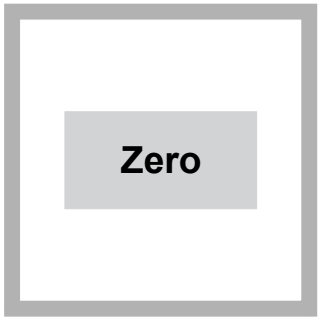


8. Clean the blank vial.

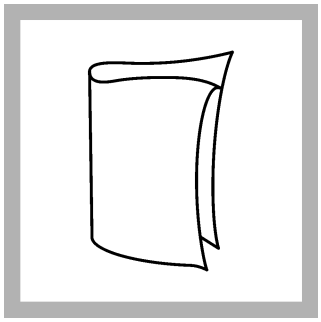
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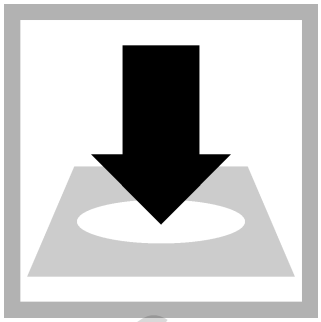
9. Insert the blank vial into the 16-mm cell holder.



10. Push **ZERO**. The display shows 0.0 mg/L NH_3–N.



11. Clean the sample vial.



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



13. Push **READ**. Results show in mg/L NH_3–N.

Interferences

Interfering substance	Interference level
Calcium	50,000 mg/L as $CaCO_3$
Iron	All levels. Correct for iron interference as follows: **1.** Use one of the Iron, Total procedures to measure the iron concentration of the sample. **2.** Use an iron standard solution to add iron to the deionized water blank so that the blank has the same iron concentration as the sample. The iron interference will be zeroed out from the test result.
Magnesium	300,000 mg/L as $CaCO_3$
Monochloramine	Monochloramine that is in chloraminated drinking water interferes directly at all levels and gives high results. Use a Free Ammonia and Monochloramine method to determine free ammonia in these sample matrices.
Nitrate	5000 mg/L as NO_3^-–N
Nitrite	600 mg/L as NO_2^-–N
pH	Adjust acidic or basic samples to approximately pH 7. Use 1 N sodium hydroxide standard solution for acidic samples and 1 N hydrochloric acid standard solution for basic samples.
Phosphate	5000 mg/L as PO_4^{3-}–P
Sulfate	6000 mg/L as SO_4^{2-}

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Interfering substance	Interference level
Sulfide	Sulfide will intensify the color. Remove sulfide interference as follows: **1.** Measure approximately 350 mL of sample in a 500-mL Erlenmeyer flask. **2.** Add the contents of one Sulfide Inhibitor Reagent Powder Pillow. Swirl to mix. **3.** Filter the sample through a folded filter paper and filter funnel. **4.** Use the filtered sample in the test procedure.
Other substances	Less common interferences such as hydrazine and glycine cause intensified colors in the prepared sample. Turbidity and color will give incorrect high values. Samples with severe interferences require distillation. Use the distillation procedure that is supplied with the distillation set.

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:

- Nitrogen, Ammonia Ampule Standard, 150-mg/L NH_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 0.2 mL, 0.4 mL and 0.6 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:

- 100-mg/L Ammonia Nitrogen standard
- 50-mL volumetric flask, Class A
- 20-mL volumetric pipet, Class A and pipet filler safety bulb
- Deionized water

1. Prepare a 40.0 mg/L ammonia nitrogen standard solution as follows:
   a. Use a pipet to add 20.0 mL of 100 mg/L ammonia nitrogen standard solution into the volumetric flask.
   b. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
2. Use the test procedure to measure the concentration of the prepared standard solution.

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3. 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.*

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
343	40.00 mg/L NH_3–N	38.1–41.9 mg/L NH_3–N	0.312 mg/L NH_3–N

Summary of method

Ammonia compounds combine 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 green-colored solution. The measurement wavelength is 655 nm for spectrophotometers or 610 nm for colorimeters.

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, Reagent Set, High Range Test 'N Tube™ AmVer™	2	25 tests	2606945

Required apparatus

Description	Quantity/test	Unit	Item no.
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

Recommended standards

Description	Unit	Item no.
Nitrogen, Ammonia Standard Solution, 10-mg/L NH_3-N	500 mL	15349
Nitrogen, Ammonia Standard Solution, 100-mg/L NH_3-N	500 mL	2406549
Nitrogen, Ammonia Standard Solution, 10-mL Voluette® Ampules, 150 mg/L	16/pkg	2128410
Nitrogen Ammonia Standard Solution, 10-mL Voluette® Ampule, 50-mg/L NH_3–N	16/pkg	1479110
Wastewater Effluent Standard Solution, Mixed Parameter, for NH_3-N, NO_3-N, PO_4^{3-}, COD, SO_4^{2-}, TOC	500 mL	2833249
Water, deionized	4 L	27256

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Optional reagents and apparatus

Description	Unit	Item no.
Mixing cylinder, graduated, 25 mL	each	2088640
Distillation apparatus set, general purpose	each	2265300
Filter paper, folded, 3–5-micron, 12.5 cm	100/pkg	69257
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	1000/pkg	2185628
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800
Funnel, poly, 65 mm	each	108367
Hydrochloric Acid Standard Solution, 1 N	1000 mL	2321353
Sodium Hydroxide Standard Solution, 1.0 N	100 mL MDB	104532
Sulfide Inhibitor Reagent Powder Pillows	100/pkg	241899
Sodium Hydroxide Solution, 5 N	50 mL	245026
Sodium Thiosulfate, 0.1 N	100 mL	32332

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