Nitrogen, Ammonia

DOC316.53.01077

Salicylate Method[1]

Method 8155

0.01 to 0.50 mg/L NH_3–N

Powder Pillows

Scope and application: For water, wastewater and seawater.

[1] Adapted from Clin. Chim. Acta., 14, 403 (1966).

Test preparation

Instrument-specific information

Table 1 shows all of the instruments that have the program for this test. The table also shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests.

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

Table 1 Instrument-specific information

Instrument	Sample cell orientation	Sample cell
DR 6000 DR 3800 DR 2800 DR 2700 DR 1900	The fill line is to the right.	2495402 
DR 5000 DR 3900	The fill line is toward the user.	
DR 900	The orientation mark is toward the user.	2401906 

Before starting

Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.
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.
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
Ammonia Cyanurate Reagent Powder Pillow, 10-mL	2
Ammonia Salicylate Reagent Powder Pillow, 10-mL	2

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Items to collect (continued)

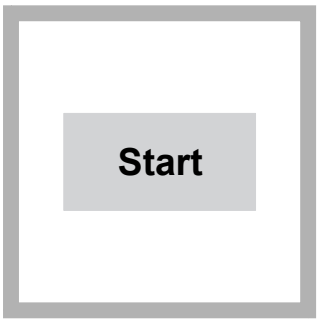
Description	Quantity
Stoppers for 18-mm tubes and AccuVac Ampuls	2
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

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

Sample collection and storage

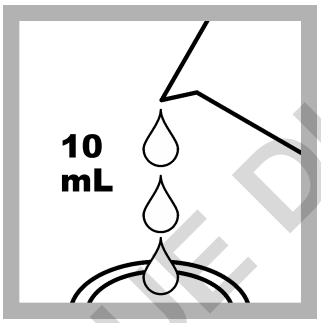
- Collect samples in clean glass or plastic bottles.
- If the sample contains chlorine, add one drop of 0.1 N sodium thiosulfate to 1 liter of sample to remove each 0.3 mg/L of chlorine.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated sulfuric acid (approximately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

Powder pillow procedure

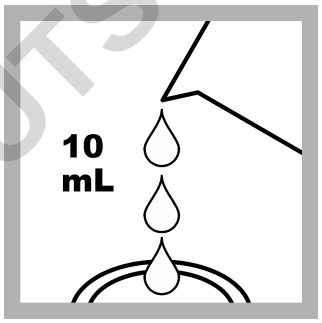


1. Start program **385 N, Ammonia, Salic**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

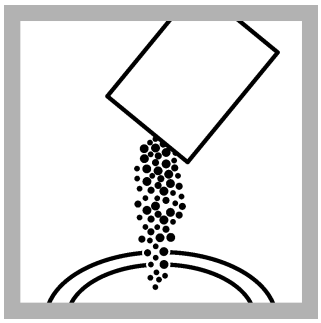
Note: *Although the program name can be different between instruments, the program number does not change.*



2. Prepare the blank: Fill a sample cell with 10 mL of deionized water.

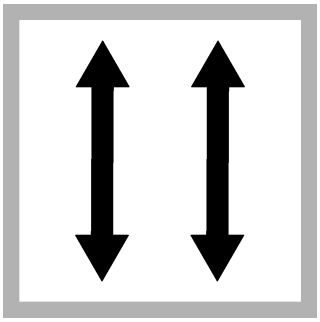


3. Prepare the sample: Fill a second sample cell with 10 mL of sample.



4. Add the contents of one Ammonia Salicylate powder pillow to each sample cell.

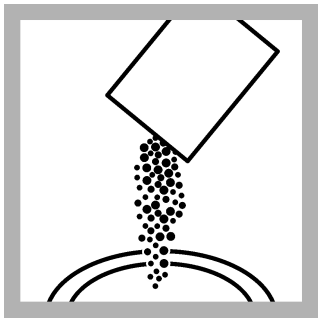
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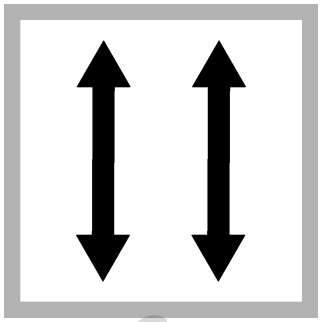
5. Put the stopper on the sample cell. Shake to dissolve the reagent.



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



7. After the timer expires, add the contents of one Ammonia Cyanurate powder pillow to each sample cell.

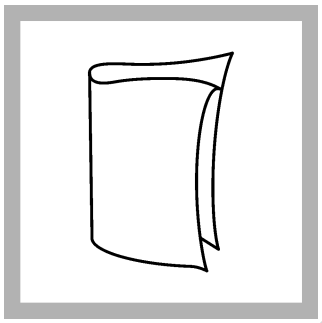


8. Put the stopper on the sample cell. Shake to dissolve the reagent.



9. Start the instrument timer. A 15-minute reaction time starts.

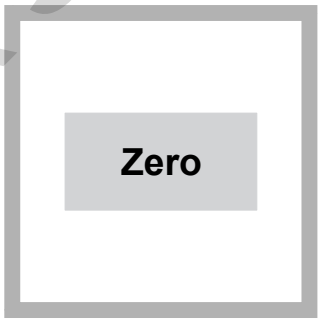
A green color shows when ammonia-nitrogen is present.



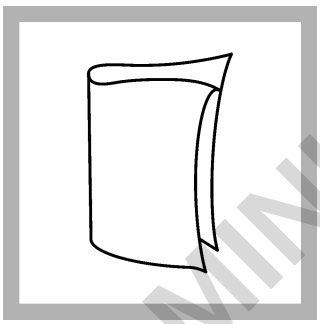
10. When the timer expires, clean the blank sample cell.



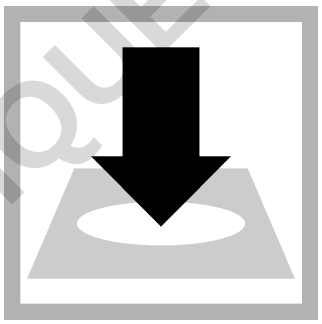
11. Insert the blank into the cell holder.



12. Push **ZERO**. The display shows 0.00 mg/L NH_3–N.



13. Clean the prepared sample cell.



14. Insert the prepared sample into the cell holder.



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

Interferences

Interfering substance	Interference level
Calcium	1000 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	6000 mg/L as $CaCO_3$

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Interfering substance	Interference level
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	100 mg/L as NO_3^-–N
Nitrite	12 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	100 mg/L as PO_4^{3-}–P
Sulfate	300 mg/L as SO_4^{2-}
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:

- Ammonia Nitrogen Standard Solution, 10 mg/L as NH_3–N
- 25-mL mixing cylinders (3)
- Pipet, TenSette®, 0.1–1.0 mL and tips

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:

- Ammonia Nitrogen Standard Solution, 10 mg/L as NH_3–N

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- 100-mL volumetric flask, Class A
- 4-mL volumetric pipet, Class A and pipet filler safety bulb
- Deionized water

1. Prepare a 0.40 mg/L ammonia nitrogen standard solution as follows:
   a. Use a pipet to add 4 mL of the 10 mg/L ammonia nitrogen standard solution into the volumetric flask. *(Alternate preparation: pipet 0.8 mL of a 50-mg/L ammonia nitrogen standard solution into a 100-mL 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.
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
385	0.40 mg/L NH_3–N	0.38–0.42 mg/L NH_3–N	0.004 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 to give a final 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, 10 mL, includes:	—	100 tests	2668000
Ammonia Cyanurate Reagent Powder Pillow, 10 mL	2	100/pkg	2653199
Ammonia Salicylate Reagent Powder Pillow, 10 mL	2	100/pkg	2653299

Recommended standards and apparatus

Description	Unit	Item no.
Flask, volumetric, Class A, 100 mL, glass	each	1457442
Nitrogen Ammonia Standard Solution, 10-mg/L NH_3–N	500 mL	15349
Nitrogen Ammonia Standard Solution, 10-mL Voluette® Ampule, 50-mg/L NH_3–N	16/pkg	1479110
Pipet, TenSette®, 0.1–1.0 mL	each	1970001

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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, volumetric, Class A, 4.00 mL	each	1451504
Stoppers for 18-mm tubes and AccuVac Ampuls	6/pkg	173106
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

Optional reagents and apparatus

Description	Unit	Item no.
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800
Mixing cylinder, graduated, 25-mL	each	2088640
Distillation heater and support for apparatus set, 115 VAC option	each	2274400
Distillation apparatus set, general purpose	each	2265300
Flask, Erlenmeyer, 500 mL	each	50549
Funnel, poly, 65 mm	each	108367
Distillation heater and support for apparatus set, 230 VAC option	each	2274402
Filter Paper, folded, 2–3-micron, pleated, 12.5-cm	100/pkg	189457
Pipet, serological, 2 mL	each	53236
Sodium Hydroxide Solution, 5 N	50 mL	245026
Sulfide Inhibitor Reagent Powder Pillows	100/pkg	241899
Sulfuric Acid, concentrated, ACS	500 mL	97949

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