Nitrogen, Ammonia

DOC316.53.01466

Salicylate Method[1]

Method 8155

0.01 to 0.80 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

Before starting

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.
Always do tests in sample cells. Do not put the instrument in the sample or pour the sample into the cell holder.
Make sure that the sample cells are clean and there are no scratches where the light passes through them.
Rinse the sample cell and cap with the sample three times before the sample cell is filled.
Make sure that there are no fingerprints or liquid on the external surface of the sample cells. Wipe with a lint-free cloth before measurement.
Cold waters can cause condensation on the sample cell or bubbles in the sample cell during color development. Examine the sample cell for condensation or bubbles. Remove condensation with a lint-free cloth. Invert the sample cell to remove bubbles.
Install the instrument cap over the cell holder before ZERO or READ is pushed.
After the test, immediately empty and rinse the sample cell. Rinse the sample cell and cap three times with deionized water.
If the test result is over-range, dilute the sample with high quality, ammonia-free deionized water and repeat the test. Multiply the result by the dilution factor. Refer to Sample dilution on page 2.
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
Sample cells, 25-mm (10 mL)	2

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

Sample collection and storage

- Collect samples in clean glass or plastic bottles.
- If the sample contains chlorine, add 1 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.

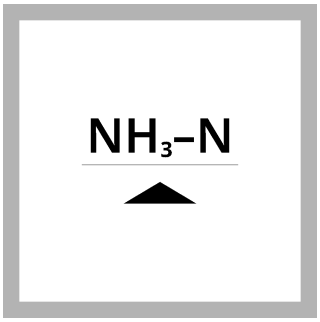
Sample dilution

Select the applicable sample volume from Table 1. The sample volume depends on the starting concentration of the sample. Put the sample in a graduated mixing cylinder, then dilute the sample to 25 mL with deionized water and mix fully.

Table 1 Sample volumes for dilution

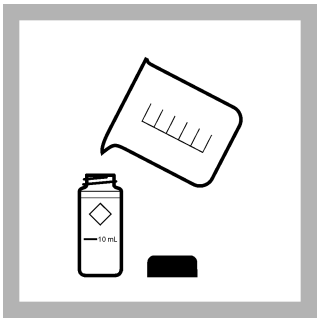
Starting concentration (mg/L NH_3-N)	Sample volume (mL)	Dilution factor
≤ 0.8	Dilution is not necessary.	—
≤ 2	10.0 mL	2.5
≤ 4	5.0 mL	5.0
≤ 8	2.5 mL	10.0
≤ 20	1.0 mL	25.0

Powder pillow procedure

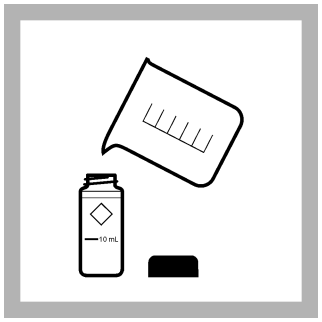


1. Set the instrument to NH_3–N.

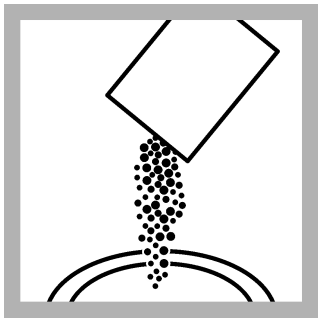
For DR300, push the up arrow button. For PCII, push the menu button, checkmark button, then the menu button again.



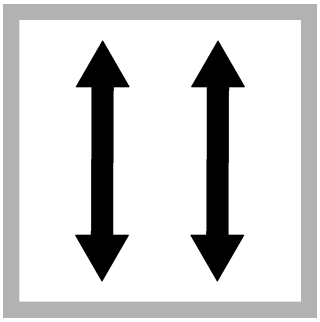
2. Prepare the blank: Fill a sample cell to the 10-mL mark with deionized water.



3. Prepare the sample: Fill a sample cell to the 10-mL mark with sample or diluted sample. Refer to Sample dilution on page 2.

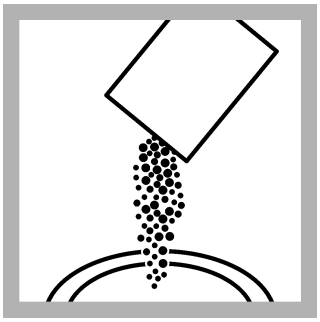


4. Add the contents of one Ammonia Salicylate Powder Pillow to each sample cell.

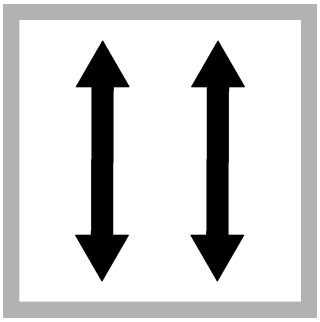


5. Put the stopper on the sample cell. Shake to dissolve the reagent.

6. Set and start a timer for 3 minutes. A 3-minute reaction time starts.

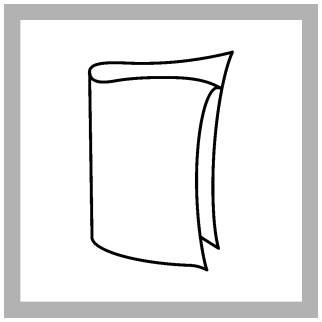


7. After the timer expires, add the contents of one Ammonia Cyanurate Powder Pillow to each sample cell.

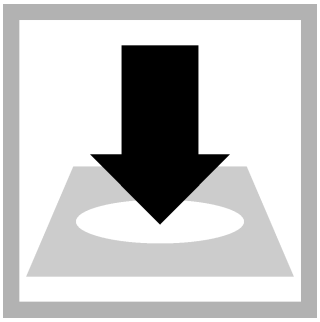


8. Put the stopper on the sample cell. Shake to dissolve the reagent.

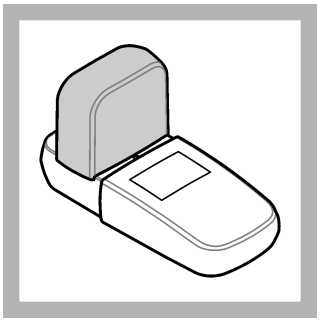
9. Set and start a timer for 15 minutes. A 15-minute reaction time starts.



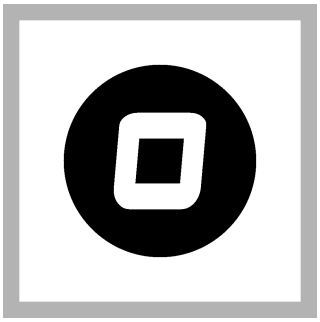
10. When the timer expires, clean the blank sample cell.



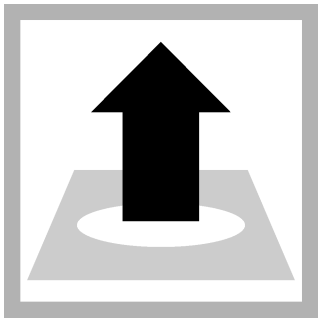
11. Insert the blank into the cell holder. Point the diamond mark on the sample cell toward the keypad.



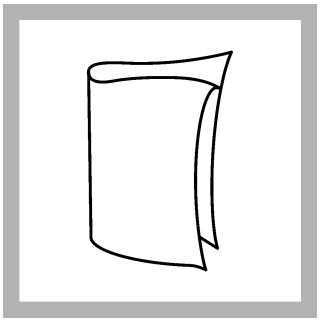
12. Install the instrument cap over the cell holder.



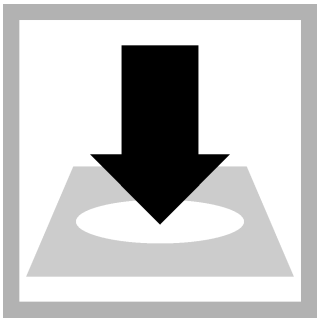
13. Push **ZERO**. The display shows “0.00”.



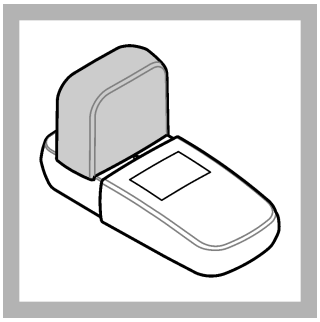
14. Remove the sample cell from the cell holder.



15. Clean the prepared sample cell.

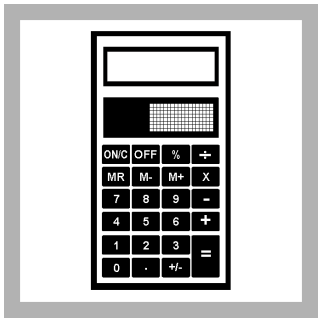


16. Insert the prepared sample into the cell holder. Point the diamond mark on the sample cell toward the keypad.



17. Install the instrument cap over the cell holder.

18. Push **READ**. Results show in mg/L ammonia as nitrogen (NH_3–N).



19. If the sample was diluted, multiply the result by the applicable dilution factor from Table 1 on page 2.

Note: *To change the results to mg/L ammonia (NH_3) , multiply the result by 1.22. To change the results to mg/L ammonium (NH_4^+), multiply the result by 1.29.*

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 $CaCO_3$
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.

Pollution prevention and waste management

The ammonia salicylate reagent contains sodium nitroferricyanide and must be disposed of as a hazardous waste. Dispose of reacted solutions according to local, state and federal regulations.

Accuracy check

Standard additions method

Use the standard additions method 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
- Mixing cylinders, 25-mL (3)
- Pipet, TenSette®, 0.1–1.0 mL and tips

1. 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.
2. 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.
3. Compare the expected result to the actual result. The expected ammonia nitrogen concentration increase is 0.08 mg/L for each 0.2 mL of standard that is added (with no sample dilution).

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

1. Prepare a 0.40 mg/L ammonia nitrogen standard solution as follows:
   a. Use a pipet to add 4.0 mL of 10 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.
3. 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.*

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a DR300 and a Pocket Colorimeter II during ideal test conditions. Users can get different results under different test conditions.

Precision (95% confidence interval)
0.60 ± 0.05 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.

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

Required apparatus

Description	Quantity/test	Unit	Item no.
Sample cells, 10-mL round, 25 mm x 60 mm	2	6/pkg	2427606

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
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
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

Optional reagents and apparatus (continued)

Description	Unit	Item no.
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
Sodium Thiosulfate, 0.1 N	100 mL	32332
Sulfide Inhibitor Reagent Powder Pillows	100/pkg	241899
Sulfuric Acid, concentrated, ACS	500 mL	97949
Sulfuric Acid Standard Solution, 1 N	100 mL MDB	127032

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