

Nitrogen, Ammonia

DOC316.53.01077

Salicylate Method¹**Method 8155****0.01 to 0.50 mg/L NH₃-N****Powder Pillows****Scope and Application:** For water, wastewater and seawater¹ Adapted from *Clin. Chim. Acta.*, 14, 403 (1966)

Test preparation

How to use instrument-specific information

The [Instrument-specific information](#) table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 1 Instrument-specific information

Instrument	Sample cell	Cell orientation	Adapter
DR 5000	2495402	Fill line faces user	A23618
DR 2800	2495402	Fill line faces right	—
DR 2700	2495402	Fill line faces right	—
DR/2500	2427606	—	—
DR/2400	2427606	—	—

Before starting the test:

A green color will develop if ammonia nitrogen is present.

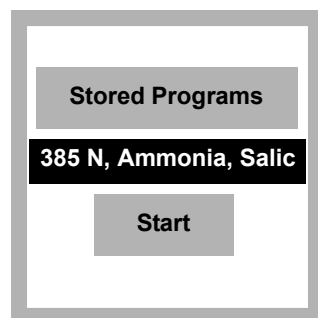
The ammonia salicylate reagent contains sodium nitroferricyanide. Cyanide solutions are regulated as hazardous wastes by the Federal RCRA. Collect cyanide solutions for disposal as reactive (D001) waste. Make sure that cyanide solutions are stored in a caustic solution with pH>11 to prevent cyanide gas. Refer to the current MSDS and local regulatory agencies for further information in safe disposal of these materials.

Collect the following items:

Description	Quantity
Ammonia Cyanurate Reagent pillows	2
Ammonia Salicylate Reagent pillows	2
Sample Cells (see Instrument-specific information)	2
Stopper for sample cells	2

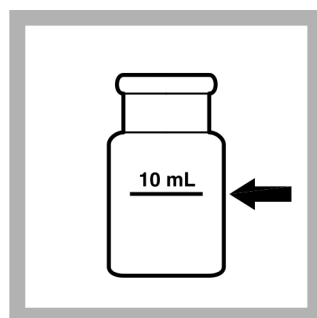
See [Consumables and replacement items](#) for reorder information.

Salicylate method for powder pillows

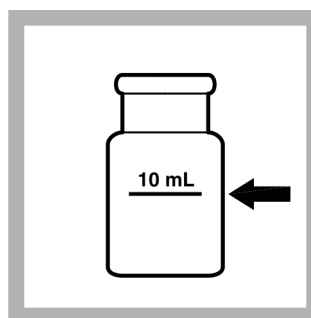


1. Select the test.
Insert an adapter if required (see [Instrument-specific information](#)).

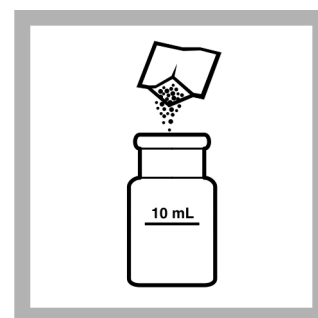
Refer to the user manual for orientation.



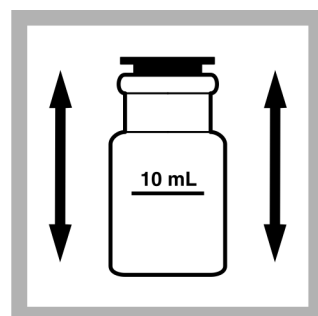
2. **Prepared Sample:**
Fill a sample cell to the 10-mL mark with sample.



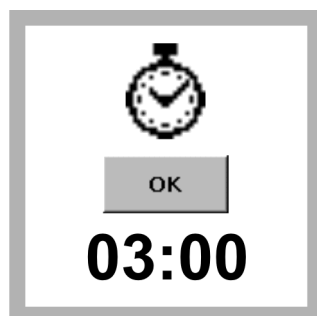
3. **Blank Preparation:**
Fill a second sample cell to the 10-mL mark with deionized water.



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

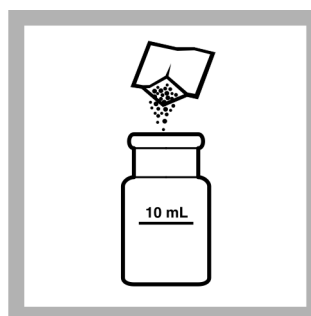


5. Insert the stopper or cap the cell and shake to dissolve.

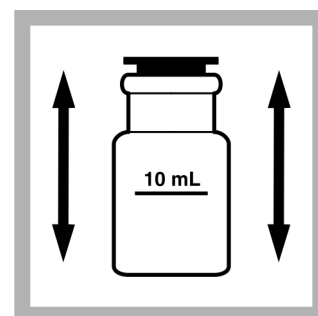


6. Start the instrument timer.

A three-minute reaction period will begin.



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



8. Stopper or cap and shake to dissolve.



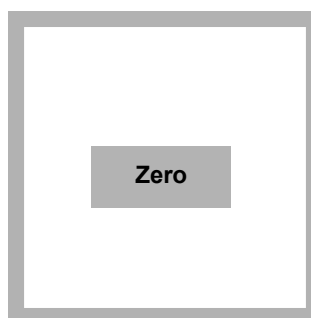
9. Start the instrument timer.

A 15-minute reaction period will begin.

A green color will develop if ammonia-nitrogen is present.



10. When the timer expires, wipe the blank and insert it into the cell holder



11. **ZERO** the instrument.
The display will show: 0.00 mg/L NH₃-N.



12. Wipe the sample and insert it into the cell holder.
READ the results in mg/L NH₃-N.

Interferences

Table 2 Interfering substances

Interfering substance	Interference level
Calcium	Greater than 1000 mg/L as CaCO ₃
Iron	All levels. Correct for iron interference as follows: <ol style="list-style-type: none"> 1. Determine the amount of iron present in the sample by following one of the Iron, Total, procedures. 2. Add the same iron concentration to the ammonia-free water in step 2. The interference will be successfully blanked out.
Magnesium	Greater than 6000 mg/L as CaCO ₃
Monochloramine	Monochloramine present in chloraminated drinking water interferes directly at all levels, giving high results. Use Method 10200, Free Ammonia and Monochloramine, to determine free ammonia in these sample matrices.
Nitrate	Greater than 100 mg/L as NO ₃ ⁻ -N
Nitrite	Greater than 12 mg/L as NO ₂ ⁻ -N
Phosphate	Greater than 100 mg/L as PO ₄ ³⁻ -P
Sulfate	Greater than 300 mg/L as SO ₄ ²⁻
Sulfide	Sulfide will intensify the color. Eliminate sulfide interference as follows: <ol style="list-style-type: none"> 1. Measure about 350 mL of sample in a 500-mL Erlenmeyer flask¹. 2. Add the contents of one Sulfide Inhibitor Reagent¹ Powder Pillow. Swirl to mix. 3. Filter the sample through a Folded Filter Paper¹ and Filter Funnel¹. 4. Use the filtered solution in step 3.
Other Substances	Less common interferences such as hydrazine and glycine will cause intensified colors in the prepared sample. Turbidity and color will give erroneous high values. Samples with severe interferences require distillation. Use the distillation procedure with the General Purpose Distillation Set.

¹ See [Optional reagents and apparatus](#).

Sample collection, preservation and storage

- Collect samples in clean plastic or glass bottles. Most reliable results are obtained when samples are analyzed as soon as possible after collection.
- If chlorine is known to be present, add one drop of 0.1 N Sodium Thiosulfate* for each 0.3 mg/L Cl₂ in a one-liter sample.
- Adjust the pH to 2 or less with concentrated (about 2 mL per liter) Sulfuric Acid.
- Store samples at 4 °C or less. Samples preserved in this manner can be stored up to 28 days.
- Just before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution.
- Correct the test result for volume additions.

* See [Optional reagents and apparatus](#).

Accuracy check

Standard additions method (sample spike)

Required for accuracy check:

- Ammonia Nitrogen Standard Solution, 10-mg/L as $\text{NH}_3\text{-N}$
 - TenSette Pipet and Pipet Tips
 - 25-mL Mixing cylinders (3)
1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
 2. Select standard additions from the instrument menu:

Instrument	Navigate to:
DR 5000	OPTIONS>MORE>STANDARD ADDITIONS
DR 2800	OPTIONS>MORE>STANDARD ADDITIONS
DR 2700	OPTIONS>MORE>STANDARD ADDITIONS
DR/2500	OPTIONS>STANDARD ADDITIONS
DR/2400	OPTIONS>STANDARD ADDITIONS

3. Accept the default values for standard concentration, sample volume and spike volumes. After the values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Open the standard solution.
5. Use the TenSette Pipet to prepare spiked samples: add 0.2 mL, 0.4 mL and 0.6 mL of standard to three 25-mL portions of fresh sample in three mixing cylinders.
6. Follow the [Salicylate method for powder pills](#) test procedure for each of the spiked samples, starting with the 0.2 mL sample spike. Measure each of the spiked samples in the instrument.
7. Select **GRAPH** to view the results. Select **IDEAL LINE** (or best-fit) to compare the standard addition results to the theoretical 100% recovery.

Standard solution method

Note: Refer to the instrument user manual for specific software navigation instructions.

Required for accuracy check:

- Ammonia Nitrogen Standard Solution, 10-mg/L
- OR
- Ammonia Nitrogen Voluette® Standard Solution, 50-mg/L as $\text{NH}_3\text{-N}$
 - Deionized water
 - 100 mL Class A volumetric flask
 - 4-mL Class A volumetric pipet
- OR
- TenSette Pipet, 0.1–1.0 mL and Pipet Tips
1. Prepare a 0.40 mg/L ammonia nitrogen standard solution as follows:
 - Dilute 4.00 mL of Ammonia Nitrogen Standard Solution, 10-mg/L to 100 mL with deionized water.

OR

- Use the TenSette® Pipet to dilute 0.8 mL of an Ammonia Nitrogen Voluette Standard Solution, 50-mg/L as NH₃-N, to 100 mL with deionized water
2. Use the 0.40-mg/L solution in place of the sample. Follow the [Salicylate method for powder pillows](#) test procedure.
 3. To adjust the calibration curve using the reading obtained with the standard solution, navigate to Standard Adjust in the software.

Instrument	Navigate to:
DR 5000	OPTIONS>MORE>STANDARD ADJUST
DR 2800	OPTIONS>MORE>STANDARD ADJUST
DR 2700	OPTIONS>MORE>STANDARD ADJUST
DR/2500	OPTIONS>STANDARD ADJUST
DR/2400	OPTIONS>STANDARD ADJUST

4. Turn on the Standard Adjust feature and accept the displayed concentration. If an alternate concentration is used, enter the concentration and adjust the curve to that value.

Method performance

Program	Instrument	Standard	Precision—95% Confidence Limits of Distribution	Sensitivity—DConcentration per 0.010 DAbs
385	DR 5000	0.40 mg/L NH ₃ -N	0.38–0.42 mg/L NH ₃ -N	0.004 mg/L NH ₃ -N
	DR 2800	0.40 mg/L NH ₃ -N	0.38–0.42 mg/L NH ₃ -N	0.004 mg/L NH ₃ -N
	DR 2700	0.40 mg/L NH ₃ -N	0.38–0.42 mg/L NH ₃ -N	0.004 mg/L NH ₃ -N
	DR/2500	0.30 mg/L NH ₃ -N	0.25–0.35mg/L NH ₃ -N	0.004 mg/L NH ₃ -N
	DR/2400	0.30 mg/L NH ₃ -N	0.25–0.35mg/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 present to give a final green-colored solution. Test results are measured at 655 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
Ammonia Nitrogen Reagent Set for 10-mL samples (100 tests), includes:	—	—	2668000
Includes:			
(2) Ammonia Cyanurate Reagent Powder Pillows	2	100/pkg	2653199
(2) Ammonia Salicylate Reagent Powder Pillows	2	100/pkg	2653299

Nitrogen, Ammonia

Recommended standards and apparatus

Description	Unit	Catalog number
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	147910
Wastewater, Effluent Inorganics, for NH ₃ -N, NO ₃ -N, PO ₄ , COD, SO ₄ , TOC	500 mL	2833249
Pipet, TenSette® 0.1 - 1.0 mL	each	1970001
Pipet Tips, for TenSette Pipet 1970001	50/pkg	2185696
Pipet Tips, for TenSette Pipet 1970001	1000/pkg	2185628
Flask, volumetric, Class A, 100 mL	each	1457442
Pipet, volumetric, Class A, 4.00 mL	each	1451504
Stopper, 18 mm	each	6/pkg
Water, deionized	4 L	27256

Optional reagents and apparatus

Description	Unit	Catalog number
Cylinder, mixing, 25-mL	each	2088640
Distillation Set, general purpose	each	2265300
Erlenmeyer Flask, 500 mL	each	50549
Filter Funnel, Analytical PP, 65 mm	each	108367
Filter Paper, folded, 12.5 cm	100/pkg	189457
Heater and support apparatus; 115 Vac, 60 Hz	each	2274400
Heater and support apparatus, 230 Vac, 50 Hz	each	2274402
Pipet, 2 mL Seriological	each	53236
Ampule breaker, Voluette	each	2196800
Sodium Hydroxide Standard Solution, 5.0 N	50 mL SCDB	245026
Sulfide Inhibitor Reagent Powder Pillow	100/pkg	241899
Sulfuric Acid, Conc	500 mL	97949



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