# 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<br>DR 3800<br>DR 2800<br>DR 2700<br>DR 1900 | The fill line is to the right. | 2495402<br>  |
| DR 5000<br>DR 3900 | The fill line is toward the user. | |
| DR 900 | The orientation mark is toward the user. | 2401906<br>  |

### 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 |

## Items to collect (continued)

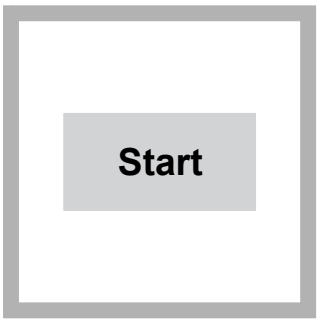
| Description | Quantity |
|---|---|
| Stoppers for 18-mm tubes and AccuVac Ampuls | 2 |
| Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.) | 2 |

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

## Sample collection and storage

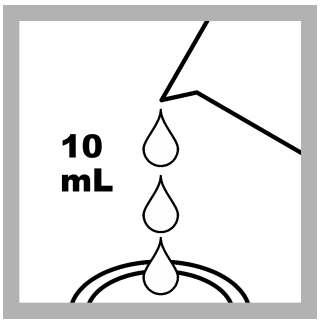
- Collect samples in clean glass or plastic bottles.
- If the sample contains chlorine, add one drop of 0.1 N sodium thiosulfate to 1 liter of sample to remove each 0.3 mg/L of chlorine.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated sulfuric acid (approximately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

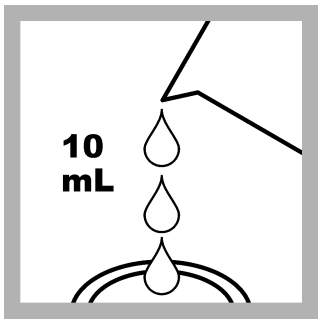



**1.** Start program **385 N, Ammonia, Salic**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

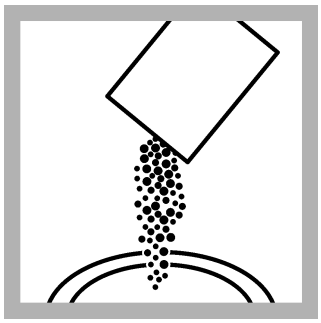
***Note:*** *Although the program name can be different between instruments, the program number does not change.*



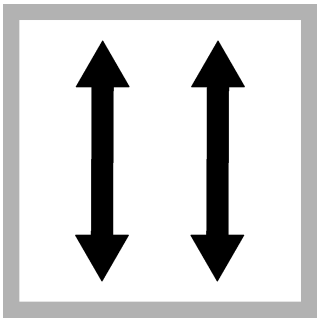

**2. Prepare the blank:** Fill a sample cell with 10 mL of deionized water.




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

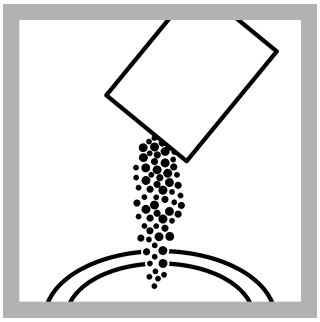


**4.** Add the contents of one Ammonia Salicylate powder pillow to each sample cell.

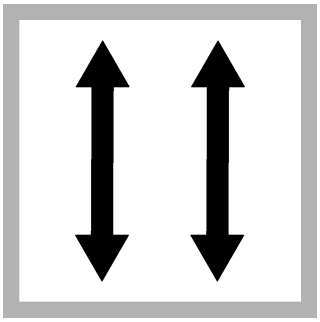


**5.** Put the stopper on the sample cell. Shake to dissolve the reagent.


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



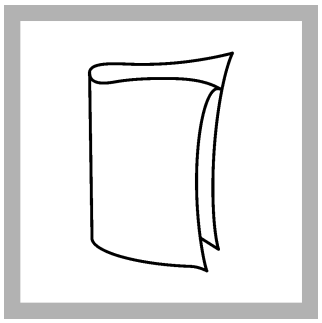
**7.** After the timer expires, add the contents of one Ammonia Cyanurate powder pillow to each sample cell.



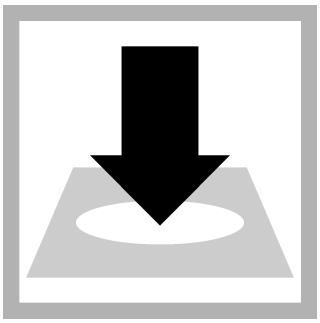
**8.** Put the stopper on the sample cell. Shake to dissolve the reagent.


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

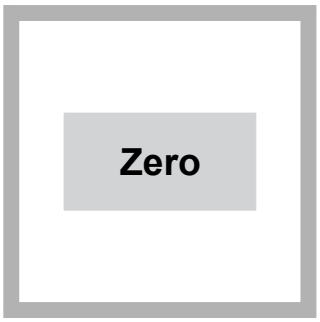
A green color shows when ammonia-nitrogen is present.



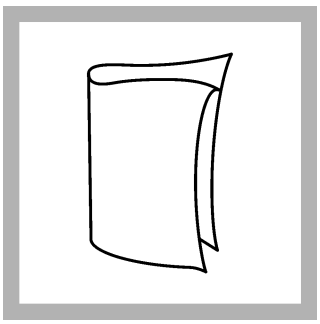
**10.** When the timer expires, clean the blank sample cell.



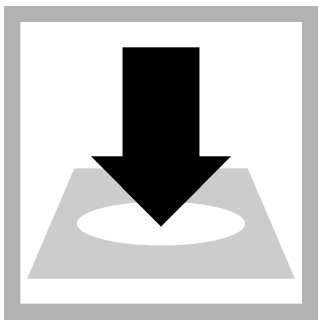
**11.** Insert the blank into the cell holder.




**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:<br>**1.** Use one of the Iron, Total procedures to measure the iron concentration of the sample.<br>**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 $CaCO_3$ |

| 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:<br>**1.** Measure approximately 350 mL of sample in a 500-mL Erlenmeyer flask.<br>**2.** Add the contents of one Sulfide Inhibitor Reagent Powder Pillow. Swirl to mix.<br>**3.** Filter the sample through a folded filter paper and filter funnel.<br>**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

- 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<br>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 |

**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 |

**FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:**
In the U.S.A. – **Call toll-free 800-227-4224**
Outside the U.S.A. – **Contact the HACH office or distributor serving you.**
On the Worldwide Web – **www.hach.com**; E-mail – **techhelp@hach.com**

**HACH** COMPANY
WORLD HEADQUARTERS
Telephone: (970) 669-3050
FAX: (970) 669-2932



09/2015, Edition 10