Nitrogen, Ammonia

DOC316.53.01078

USEPA[1] Nessler Method[2]

Method 8038

0.02 to 2.50 mg/L NH_3–N

Reagent Solution

Scope and application: For water, wastewater and seawater. Distillation is required for wastewater and seawater.

[1] USEPA accepted for wastewater analysis (distillation required), Method 350.2.
[2] Adapted from *Standard Methods for the Examination of Water and Wastewater,* 4500-NH_3 B & C, 15th Edition.

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 specific instruments.

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.	

Before starting

Hold the reagent droppers and dropper bottles vertically, not at an angle, when the reagent is added.
The reagents that are used in this test contain mercury. Collect the reacted samples for safe disposal.
If the Pour-Thru Cell is used, clean the cell periodically. To clean, add several crystals of sodium thiosulfate pentahydrate into the cell funnel. Add deionized water to dissolve the crystals. Rinse fully with deionized water.
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

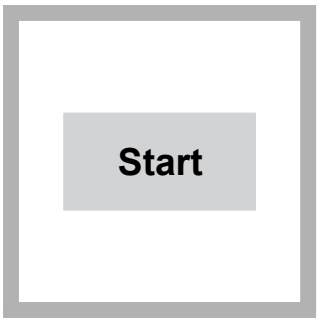
Description	Quantity
Ammonia Nitrogen Reagent Set	1
Water, deionized	25 mL
Pipet, serological, 1-mL	1
Mixing cylinder, graduated, 25 mL, glass stopper	2
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

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

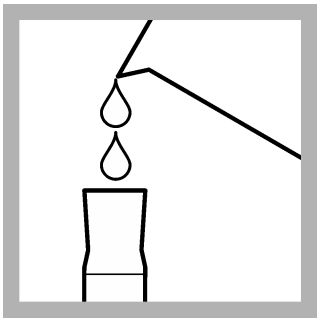
Sample collection and storage

- Collect samples in clean glass or plastic bottles.
- If the sample contains chlorine, add one drop of 0.1 N sodium thiosulfate for each 0.3 mg/L chlorine in 1 liter of sample.
- 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.

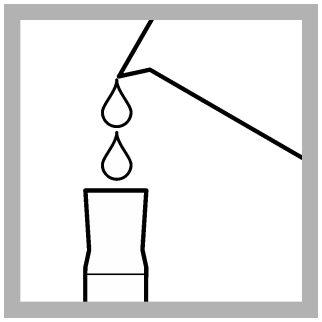
Test procedure



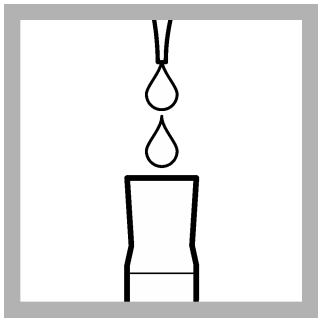
1. Start program **380 N, Ammonia, Ness**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.



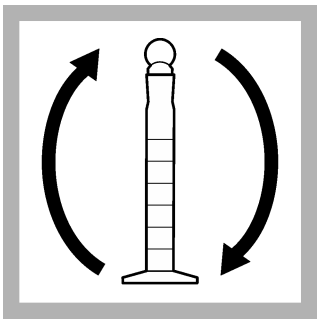
2. Prepare the sample: Fill a mixing cylinder to the 25-mL line with sample.



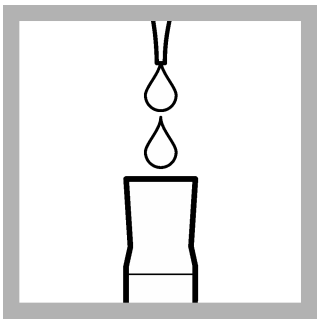
3. Prepare the blank: Fill a mixing cylinder to the 25-mL line with deionized water.



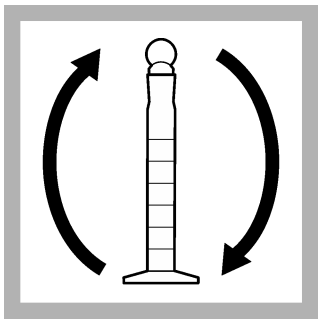
4. Add 3 drops of Mineral Stabilizer to each mixing cylinder.



5. Put the stopper on the mixing cylinders. Invert the mixing cylinders several times to mix.

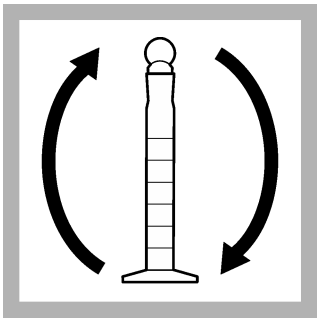


6. Add 3 drops of Polyvinyl Alcohol Dispersing Agent to each mixing cylinder.



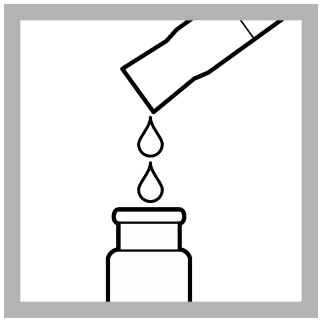
7. Put the stopper on the mixing cylinders. Invert the mixing cylinders several times to mix.

8. Use a pipet to add 1.0 mL of Nessler Reagent to each mixing cylinder.

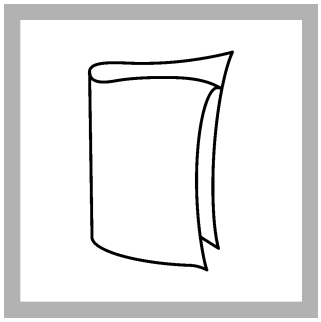


9. Put the stopper on the mixing cylinders. Invert the mixing cylinders several times to mix.

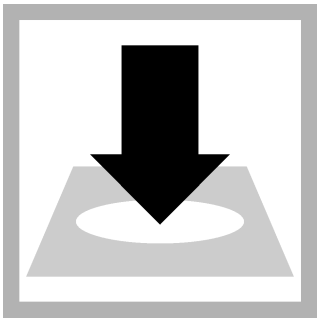
10. Start the instrument timer. A 1-minute reaction time starts.



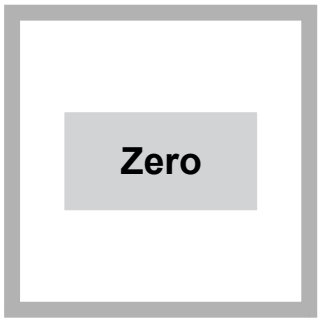
11. Pour 10 mL from the blank cylinder into a sample cell.



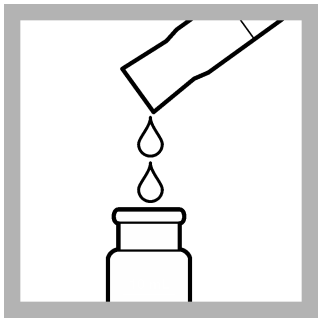
12. When the timer expires, clean the blank sample cell.



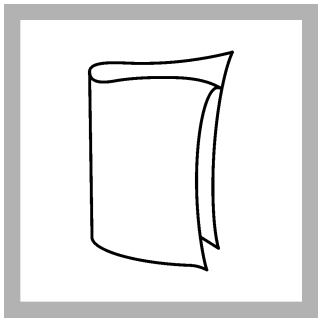
13. Insert the blank into the cell holder.



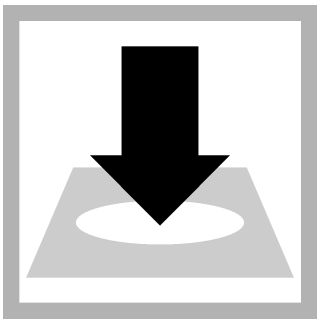
14. Push **ZERO**. The display shows 0.00 mg/L NH_3–N.



15. Pour 10 mL from the sample cylinder into a second sample cell.



16. Clean the prepared sample cell.



17. Insert the prepared sample into the cell holder.

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

Interferences

Table 2 Interfering substances

Interfering substance	Interference level
Chlorine	Remove residual chlorine from a 250 mL sample by adding 1 drop of sodium thiosulfate for each mg/L chlorine (Cl_2). Sodium arsenite can be used instead of sodium thiosulfate. Refer to Sample collection and storage on page 2.
Hardness	A solution containing a mixture of 500 mg/L $CaCO_3$ and 500 mg/L Mg as $CaCO_3$ does not interfere. If the hardness concentration is more than these concentrations, add extra Mineral Stabilizer.
Iron	Interferes at all levels by causing turbidity with Nessler Reagent.

Table 2 Interfering substances (continued)

Interfering substance	Interference level
Seawater	Add 1.0 mL (27 drops) of Mineral Stabilizer to the sample before analysis. This complexes the high magnesium concentrations found in sea water, but the sensitivity of the test is reduced by 30% due to the high chloride concentration. For best results, make a calibration with standards that contain the same chloride concentration as seawater, or distill the sample.
Sulfide	Interferes at all levels by causing turbidity with Nessler Reagent.
Glycine, various aliphatic and aromatic amines, organic chloramines, acetone, aldehydes and alcohols	May cause greenish or other off colors or turbidity. Distill the sample if these compounds are present.

Pollution prevention and waste management

The Nessler reagent contains mercuric iodide. The reacted samples and blanks will contain mercury and must be disposed of as a hazardous waste. Dispose of reacted solutions according to local, state and federal regulations.

Distillation

To eliminate most interferences, distill the sample, then use the distilled sample in the test procedure.

1. Set up the distillation apparatus for general purpose distillation. Refer to the Distillation Apparatus manual for proper assembly.
2. Measure 250 mL of sample into a 250-mL graduated cylinder.
3. Pour the sample into a 400-mL beaker. If the sample contains chlorine, add 1 drop of 0.1 N sodium thiosulfate solution for each 1 mg/L Cl_2 to remove the chlorine.
4. Add 25 mL of borate buffer solution and mix. Adjust the pH to approximately 9.5 with 1 N sodium hydroxide solution. Use a pH meter to monitor the pH.
5. Pour the solution into the distillation flask.
6. Add a magnetic stir bar and 5 glass beads.
7. Use a graduated cylinder to measure 25 mL of deionized water into a 250-mL Erlenmeyer flask. Add the contents of one Boric Acid Powder Pillow. Mix thoroughly.
8. Set the flask under the distillation apparatus drip tube. Elevate the flask so that the end of the tube is immersed in the solution.
9. Set the stirrer power to on. Set the stir control to 5.
10. With the thermometer inserted, set the heat control to 10. The yellow pilot lamp is an indication that the heater is on.
11. Turn on the water and adjust to maintain a steady flow through the condenser.
12. When 150 mL of distillate has been collected, turn the heater off. Immediately remove the collection flask. Measure the distillate to make sure 150 mL was collected (total volume = 175 mL).
13. Adjust the pH to approximately 7 with 1 N sodium hydroxide solution. Use a pH meter to monitor the pH.
14. Quantitatively transfer the distillate into a 250-mL volumetric flask. Dilute to the mark with deionized water. Mix well. Use the diluted distillate in the test procedure.

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:

- 50-mg/L Nitrogen-Ammonia Standard Solution

- Mixing cylinders, 25-mL (3x)
- TenSette Pipet and pipet 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 0.1 mL, 0.2 mL and 0.3 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:

- 1-mg/L Nitrogen-Ammonia Standard Solution

1. Use the test procedure to measure the concentration of the standard solution.
2. 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
380	1.00 mg/L NH_3–N	0.99–1.01 mg/L NH_3–N	0.02 mg/L NH_3–N

Summary of Method

The Mineral Stabilizer complexes hardness in the sample. The Polyvinyl Alcohol Dispersing Agent helps the color formation in the reaction of Nessler Reagent with ammonia and certain other amines. A yellow color is formed that is proportional to the ammonia concentration. The measurement wavelength is 425 nm.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Ammonia Nitrogen Reagent Set, includes:	—	—	2458200
Nessler Reagent	2 mL	500 mL	2119449
Mineral Stabilizer	6 drops	50 mL SCDB	2376626

Consumables and replacement items (continued)

Description	Quantity/test	Unit	Item no.
Polyvinyl Alcohol Dispersing Agent	6 drops	50 mL SCDB	2376526
Water, deionized	varies	4 L	27256

Required apparatus

Description	Quantity/test	Unit	Item no.
Mixing cylinder, graduated, 25 mL with stopper	1	each	2088640
Pipet, serological, 1 mL, glass	1	50/pkg	2093135
Pipet filler, safety bulb	1	each	1465100

Recommended standards

Description	Unit	Item no.
Nitrogen Ammonia Standard Solution, 1.0-mg/L NH_3–N	500 mL	189149
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

Optional reagents and apparatus

Description	Unit	Item no.
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800
Distillation apparatus set, general purpose	each	2265300
Distillation heater and support for apparatus set, 115 VAC option	each	2274400
Distillation heater and support for apparatus set, 230 VAC option	each	2274402
Pipet, TenSette®, 0.1–1.0 mL	each	1970001
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	50/pkg	2185696
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	245032
Sodium Thiosulfate, 0.1 N	100 mL	32332
Sulfuric Acid, ACS	500 mL	97949

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