NITROGEN, Ammonia

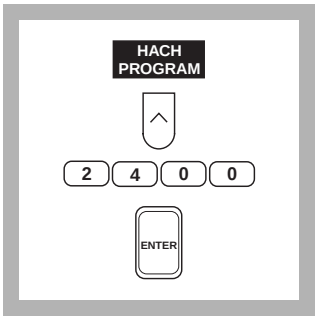
✔ **Method 8038**

Nessler Method*

(0 to 2.500 mg/L NH_3–N)

Scope and Application: *For water, wastewater, seawater; distillation is required for wastewater and seawater; USEPA accepted for wastewater analyses (distillation is required). See Distillation following this procedure. The estimated detection limit for program number 2400 is 0.017 mg/L.*

* Adapted from *Standard Methods for the Examination of Water and Wastewater 4500-NH_3 B & C.*



1. Press the soft key under ***HACH PROGRAM***.

Select the stored program for low range ammonia nitrogen (NH_3-N) by pressing **2400** with the numeric keys.

Press: **ENTER**

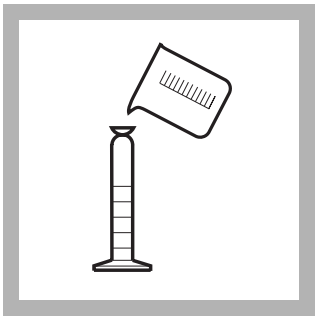
Note: *If samples cannot be analyzed immediately, see Sample collection, Storage and Preservation following these steps. Adjust the pH of preserved samples before analysis.*

Note: *The Flow Cell and Sipper Modules can be used with this procedure. Periodically clean the cells by pouring a few sodium thiosulfate pentahydrate crystals into the cell funnel. Flush it through the funnel and cell with enough deionized water to dissolve. Rinse out the crystals.*

2. The display will show:

HACH PROGRAM: 2400 N, Ammonia Nessler

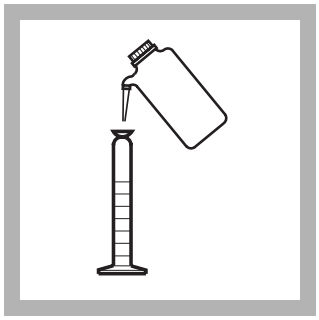
The wavelength (λ), **425 nm**, is automatically selected.



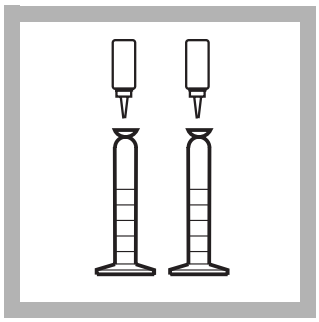
3. Fill a 25-mL mixing graduated cylinder (the prepared sample) to the 25-mL mark with sample.

Note: *For proof of accuracy, use a 1.0-mg/L Ammonia Nitrogen Standard Solution (listed under OPTIONAL REAGENTS AND STANDARDS) in place of the sample.*

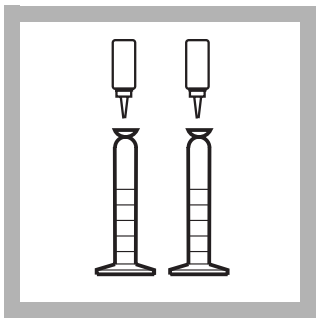
Note: *For non-preserved samples with extreme pH, see the Interferences section.*



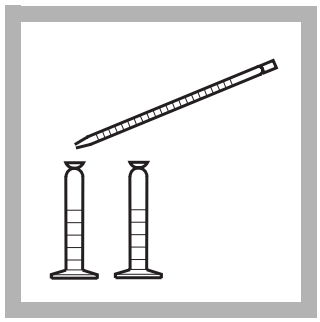
4. Fill another 25-mL mixing graduated cylinder (the blank) with deionized water.



5. Add three drops of Mineral Stabilizer to each cylinder. Stopper. Invert several times to mix.



6. Add three drops of Polyvinyl Alcohol Dispersing Agent to each cylinder by holding the dropping bottle vertically. Invert several times to mix.

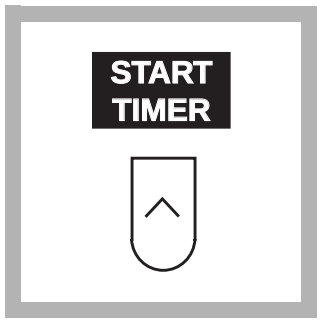


7. Pipet 1.0 mL of Nessler Reagent into each cylinder. Stopper. Invert several times to mix.

Note: *Nessler Reagent is toxic and corrosive. Pipet carefully and use a pipet filler.*

Note: *A yellow color will develop if ammonia is present. (The reagent will cause a faint yellow color in the blank.)*

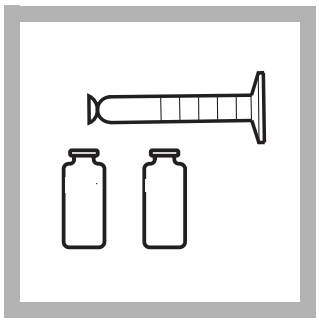
Note: *Do not wait more than 15 minutes after reagent addition (Step 7) before performing Step 12.*



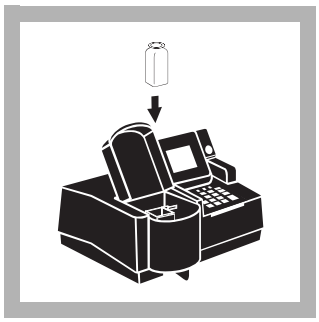
8. Press the soft key under ***START TIMER***.

A one-minute reaction period will begin.

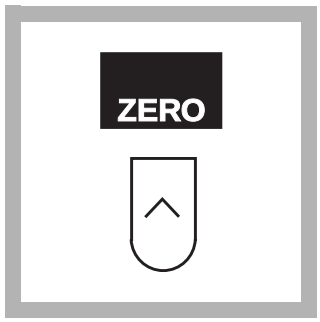
Note: *Continue with Step 9 while timer is running.*



9. Pour each solution into a sample cell.



10. When the timer beeps, place the blank into the cell holder. Close the light shield.



11. Press the soft key under ***ZERO***.

The display will show:

0.000 mg/L N NH_3

Note: *For alternate concentration units press the soft key under* ***OPTIONS****. Then press the soft key under* ***UNITS*** *to scroll through the available options. Press* **ENTER** *to return to the read screen.*

12. Place the prepared sample into the cell holder. Close the light shield. Results in mg/L ammonia expressed as nitrogen (NH_3–N) (or chosen units) will be displayed.

Note: *The results may be expressed as mg/L ammonia (NH_3) or mg/L ammonium (NH_4^+). Press the soft keys under* ***OPTIONS****, then* ***FORM:*** *to scroll through the available options. Press* **ENTER** *to return to the read screen.*

Interferences

Interfering Substance	Interference Levels and Treatments
Chlorine	Remove residual chlorine by adding 2 drops of sodium arsenite for each mg/L Cl from a 250 mL sample. Sodium thiosulfate can be used in place of sodium arsenite. See *Sample collection, Storage and Preservation* below.
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 exceeds these concentrations, add extra Mineral Stabilizer.
Iron	Interferes at all levels by causing turbidity with Nessler Reagent.
Seawater	May be analyzed by adding of 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 percent due to the high chloride concentration. For best results, perform a calibration, using standards spiked to the equivalent chloride concentration, or distill the sample as described below.
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.

Sample collection, Storage and Preservation

Collect samples in clean glass or plastic bottles. If chlorine is present, add one drop of 0.1 N Sodium Thiosulfate for each 0.3 mg/L Cl_2 in a 1-liter sample. Preserve the sample by reducing the pH to 2 or less with sulfuric acid (at least 2 mL). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Warm samples to room temperature and neutralize with 5 N Sodium Hydroxide before analysis. Correct the test result for volume additions; see Section *1.2.2 Correcting for Volume Additions*.

Accuracy Check

Standard Additions Method

a. Leave the unspiked sample in the sample compartment. Verify that the units displayed are in mg/L. Select standard additions mode by pressing the soft keys under ***OPTIONS, (MORE)*** and then ***STD ADD***.

b. Press **ENTER** to accept the default sample volume (mL), 25.

c. Press **ENTER** to accept the default standard concentration (mg/L), 50.

d. Press the soft key under ***ENTRY DONE***.

e. Snap the neck off a Nitrogen Ammonia Voluette Ampule Standard, 50-mg/L NH_3–N.

f. Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively to three 25-mL samples and mix each thoroughly.

g. Analyze each standard addition sample as described above. Accept the standard additions reading by pressing the soft key under ***READ*** each time. Each addition should reflect approximately 100% recovery.

h. After completing the sequence, the display will show the extrapolated concentration value and the “best-fit” line through the standard additions data points, accounting for matrix interferences.

i. See Section *1.4.1 Standard Additions* for more information.

Standard Solutions Method
To check accuracy, use a 1.0-mg/L Nitrogen Ammonia Standard Solution listed under *OPTIONAL REAGENTS AND STANDARDS*. Or, prepare a 1.0-mg/L ammonia nitrogen standard solution by pipetting 1.00 mL of Nitrogen Ammonia Voluette Ampule Standard, 50-mg/L, into a 50-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily. Perform the Nessler procedure as described above.

To adjust the calibration curve using the reading obtained with the 1.0-mg/L Nitrogen Ammonia Standard Solution, press the soft keys under ***OPTIONS, MORE*** then ***STD: OFF***. Press **ENTER** to accept the displayed concentration, the value of which depends on the selected units. If an alternate concentration is used, enter the actual concentration and press **ENTER** to return to the read screen. See Section *1.5.5 Adjusting the Standard Curve* for more information.

Method Performance

Precision
Standard: 1.00 mg/L NH_3–N

Program	95% Confidence Limits
2400	0.99–1.01 mg/L NH_3–N

For more information on determining precision data and method detection limits, refer to Section *1.5*.

Estimated Detection Limit

Program	EDL
2400	0.017 mg/L NH_3–N

For more information on derivation and use of Hach's estimated detection limit, see Section *1.5.2*. To determine a method detection limit (MDL) as defined by the 40 CFR part 136, Appendix B, see Section *1.5.1*.

Sensitivity
Program Number: 2400

Portion of Curve	ΔAbs	ΔConcentration
Entire Range	0.010	0.01717 mg/L

See Section *1.5.3 Sensitivity Explained* for more information.

Distillation

a. Measure 250 mL of sample into a 250-mL graduated cylinder and pour into a 400-mL beaker. Destroy chlorine, if necessary, by adding 2 drops of Sodium Arsenite Solution per mg/L Cl_2.

b. Add 25 mL of Borate Buffer Solution and mix. Adjust the pH to about 9.5 with 1 N Sodium Hydroxide solution. Use a pH meter.

c. Set up the general purpose distillation apparatus as shown in the *Hach Distillation Apparatus Manual*. Pour the solution into the distillation flask. Add a stir bar.

d. 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. Place the flask under the still drip tube. Elevate so the end of the tube is immersed in the solution.

e. Turn on the heater power switch. Set the stir control to 5 and the heat control to 10. Turn on the water and adjust to maintain a constant flow through the condenser.

f. Turn off the heater after collecting 150 mL of distillate. Immediately remove the collection flask to avoid sucking solution into the still. Measure the distillate to ensure 150 mL was collected (total volume = 175 mL).

g. Adjust the pH of the distillate to about 7 with 1 N Sodium Hydroxide. Use a pH meter.

h. Pour the distillate into a 250-mL volumetric flask; rinse the erlenmeyer with deionized water. Add the rinsings to the volumetric. Dilute to the mark. Stopper. Mix thoroughly. Analyze as described above.

Calibration Standard Preparation

To perform an ammonia calibration using the Nessler method, prepare standards containing 0.5, 1.0 and 2.0 mg/L ammonia-nitrogen as follows:

a. Into three different 100-mL volumetric flasks pipet 0.5, 1.0 and 2.0 mL of the 100-mg/L Nitrogen Ammonia Standard Solution (Cat No. 24065-49) using Class A glassware.

b. Dilute to the mark with deionized ammonia-free water. Mix thoroughly.

c. Using the Nessler method and the calibration procedure described in the *User-Entered Programs* section of the *DR/4000 Spectrophotometer Instrument Manual*, generate a calibration curve from the standard prepared above.

Summary of Method

The Mineral Stabilizer complexes hardness in the sample. The Polyvinyl Alcohol Dispersing Agent aids the color formation in the reaction of Nessler Reagent with ammonium ions. A yellow color is formed proportional to the ammonia concentration.

Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the *Material Safety Data Sheet* for information specific to the reagents used. For additional information, refer to Section *1*.

Pollution Prevention and Waste Management

Nessler Reagent contains mercuric iodide. Both the sample and the blank will contain mercury (D009) at a concentration regulated as a hazardous waste by the Federal RCRA. Do not pour these solutions down the drain. See Section *1* for further information on proper disposal of these materials.

REQUIRED REAGENTS AND STANDARDS

Ammonia Nitrogen Reagent Set 24582-00
Includes: (1) 21194-49, (1) 23766-26, (1) 23765-26

Description	Quantity Required Per Test	Unit	Cat. No.
Nessler Reagent	2 mL	500 mL	21194-49
Mineral Stabilizer	6 drops	50 mL*	SCDB 23766-26
Polyvinyl Alcohol Dispersing Agent	6 drops	50 mL	SCDB 23765-26
Water, deionized	25 mL	4 liters	272-56

REQUIRED EQUIPMENT AND SUPPLIES

Cylinder, graduated, mixing, 25-mL	2	each	21190-40
DR/4000 1-Inch Cell Adapter	1	each	48190-00
Pipet, serological, 1-mL	2	each	532-35
Pipet Filler, safety bulb	1	each	14651-00

OPTIONAL REAGENTS AND STANDARDS

Borate Buffer Solution	1000 mL		14709-53
Boric Acid Powder Pillows	50/pkg		14817-66
Nitrogen, Ammonia Standard Solution, 1-mg/L NH_3-N	500 mL		1891-49
Nitrogen, Ammonia Standard Solution, 100-mg/L NH_3–N	500 mL		24065-49
Nitrogen, Ammonia Standard Solution, 0-mL Voluette Ampule, 50-mg/L NH_3–N	16/pkg		14791-10
Sodium Arsenite Solution, 5.0-g/L	100 mL	MDB	1047-32
Sodium Hydroxide Standard Solution, 5.0 N	100 mL*	MDB	2450-32
Sodium Hydroxide Standard Solution, 1.0 N	100 mL*	MDB	1045-32
Sodium Thiosulfate Standard Solution, 0.1 N	100 mL*	MDB	323-32
Sulfuric Acid, ACS, concentrated	500 mL*		979-49

* Contact Hach for larger sizes.

OPTIONAL EQUIPMENT AND SUPPLIES

Description	Unit	Cat. No.
Ampule Breaker Kit	each	21968-00
Beaker, 400-mL	each	500-48
Cylinder, graduated, 25-mL	each	508-40
Cylinder, graduated, 250-mL	each	508-46
Distillation Apparatus, general purpose accessories	each	22653-00
Distillation heater and support apparatus set, 115 VAC	each	22744-00
Distillation heater and support apparatus set, 230 VAC	each	22744-02
Dropper, plastic, 0.5- and 1.0-mL marks	10/pkg	21247-10
DR/4000 Carousel Module Kit	each	48070-02
DR/4000 Flow-Thru Cell Module	each	48090-04
DR/4000 Sipper Cell Module, 1-inch	each	48090-03
Flask, Erlenmeyer, 250-mL	each	505-46
Flask, volumetric, 50-mL	each	547-41
Flask, volumetric, 250-mL	each	547-46
pH Meter, *sens**ion**™1*, portable	each	51700-00
Pipet, serological, 2-mL	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, Class A, 1-mL	each	14515-35
Thermometer, -10 to 110 °C	each	1877-01

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