



Method 8155

Salicylate Method*

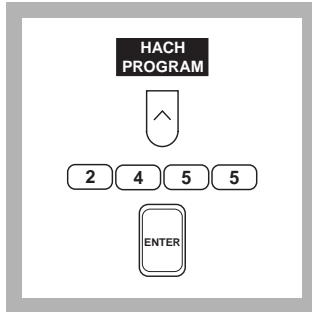
Powder Pillows

(0 to 0.80 mg/L NH₃-N)

Scope and Application: For water, wastewater, and seawater.

The estimated detection limit for program number 2455 is 0.09 mg/L NH₃-N.

* Adapted from *Clin. Chim. Acta.*, 14, 403 (1966)



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

Select the stored program number for ammonia nitrogen (NH₃-N) by pressing **2455** with the numeric keys.

Press: **ENTER**

Note: If samples cannot be analyzed immediately, see *Sample Collection, Storage and Preservation* following these steps.

Note: The Flow Cell and Sipper Modules can be used with this procedure.



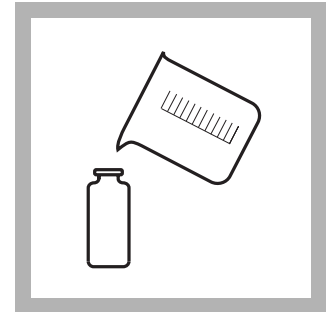
2. The display will show: **HACH PROGRAM: 2455 N, Ammonia Salic.**

The wavelength (λ), **655 nm**, is automatically selected.

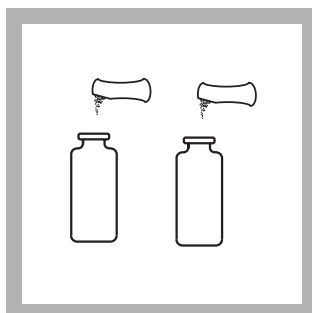


3. Fill a glass-stoppered sample cell to the 25-mL mark with sample.

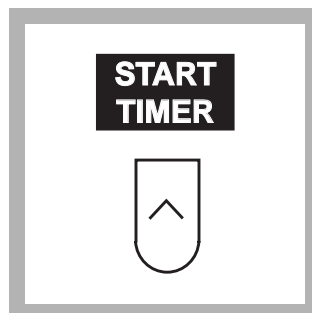
Note: For proof of accuracy, a 0.60-mg/L NH₃-N solution (preparation given in the *Accuracy Check* section) can be used in place of the sample.



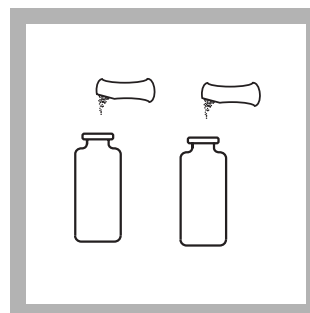
4. Fill another glass-stoppered sample cell to the 25-mL mark with deionized water (the blank).



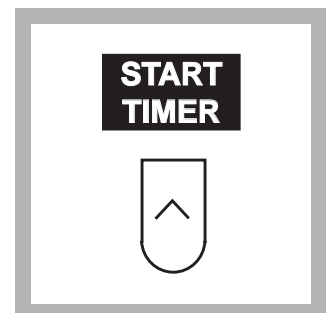
5. Add the contents of one Ammonia Salicylate Powder Pillow to each cell. Stopper and shake to dissolve the powder.



6. Press the soft key under **START TIMER**. A 3-minute reaction period will begin.



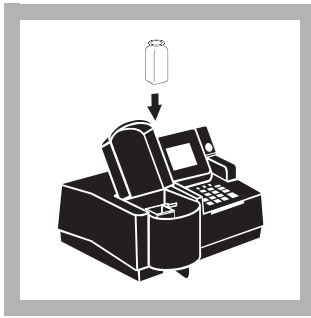
7. When the timer beeps, add the contents of one Ammonia Cyanurate Reagent Powder Pillow to each cell. Stopper and shake to dissolve the reagent.



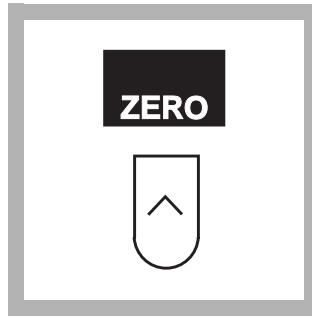
8. Press the soft key under **START TIMER**. A 15-minute reaction period will begin.

Note: A green color will develop if ammonia nitrogen is present.

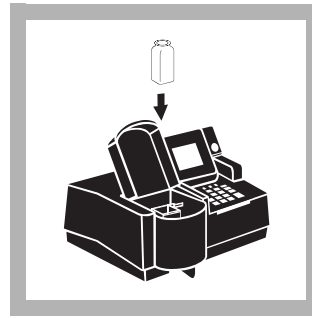
NITROGEN, Ammonia, continued



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



10. Press the soft key under **ZERO**.
The display will show:
0.00 mg/L NH₃-N



11. Place the prepared sample into the cell holder. Close the light shield. Result in mg/L ammonia as nitrogen (NH₃-N) (or chosen units) will be displayed.

Note: Results may be expressed as mg/L ammonia (NH₃) or mg/L ammonium (NH₄⁺). Press the soft keys under **OPTIONS**, then **FORM**: to scroll through the available options. Press **ENTER** to return to the read screen.

Interferences

Table 1 Interfering Substances and Suggested Treatments

Interfering Substance	Interference Levels
Calcium	Greater than 1000 mg/L as CaCO ₃
Iron	All levels. Correct for iron interference as follows: <ol style="list-style-type: none"> Determine the amount of iron present in the sample by following one of the Iron, Total, procedures. Add the same iron concentration to the ammonia-free water in Step 3. The interference will be successfully blanked out.
Magnesium	Greater than 6000 mg/L as CaCO ₃
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"> Measure about 350 mL of sample in a 500-mL erlenmeyer flask. Add the contents of one Sulfide Inhibitor Reagent Powder Pillow. Swirl to mix. Filter the sample through a folded filter paper. 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. Hach recommends the distillation procedure using the Hach General Purpose Distillation Set. See <i>OPTIONAL REAGENTS AND STANDARDS</i> .

Sample Collection, Storage and Preservation

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, the sample must be treated immediately with sodium thiosulfate. Add one drop of Sodium Thiosulfate Standard Solution, 0.1 N, for each 0.3 mg of chlorine present in a one-liter sample.

For longer storage, adjust the pH to 2 or less with concentrated sulfuric acid (about 2 mL per liter). 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 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), 10.
- d. Press the soft key under **ENTRY DONE**.
- e. Open an Ammonia Nitrogen Standard Solution, 10-mg/L as NH₃-N.
- f. Use the TenSette Pipet to add 0.2 mL, 0.4 mL and 0.6 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 addition 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 Solution Method

Prepare a 0.60-mg/L ammonia nitrogen standard by diluting 6.00 mL of the Ammonia Nitrogen Standard Solution, 10-mg/L, to 100 mL with deionized water. Or, using the TenSette Pipet, prepare a 0.60-mg/L ammonia nitrogen standard by diluting 1.2 mL of a Ammonia Nitrogen Volumetric Standard Solution, 50-mg/L as NH₃-N, to 100 mL with deionized water.

To adjust the calibration curve using the reading obtained with the 0.60-mg/L 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: 0.60 mg/L NH₃-N

Program	95% Confidence Limits
2455	0.54–0.66 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
2455	0.09 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: 2455

Portion of Curve	ΔAbs	ΔConcentration
Entire Range	0.010	0.004 mg/L

See Section 1.5.3 *Sensitivity Explained* for more information.

Calibration Standard Preparation

To perform an nitrogen calibration using the salicylate method, prepare a 10 mg/L ammonia nitrogen stock solution by pipetting 10 mL of a 100-mg/L Nitrogen Ammonia Standard Solution (Cat. No. 24065-49) into a 100-mL volumetric flask using Class A glassware. Dilute to the mark with deionized water and mix thoroughly. Or use the 10 mg/L Nitrogen Ammonia Standard Solution from Hach (Cat. No. 153-49).

Prepare calibration standards containing 0.20, 0.50, and 0.80 mg/L ammonia nitrogen as follows:

- a. Into three different 100-mL volumetric flasks, pipet 2.00, 5.00, and 8.00 mL of the 10-mg/L Nitrogen Ammonia Standard Solution using Class A glassware.
- b. Dilute to the mark with deionized water. Mix thoroughly.
- c. Using the salicylate 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 standards prepared above.

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.

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

For information on pollution prevention and waste management, refer to Section 1.

REQUIRED REAGENTS AND STANDARDS

Description	Cat. No
Ammonia Nitrogen Reagent Set, (100 tests).....	22437-00
Includes: (4) 23955-66, (4) 23953-66	

Description	Quantity Required		Cat. No.
	Per Test	Unit	
Ammonia Cyanurate Reagent Powder Pillows	2	25/pkg	23955-66
Ammonia Salicylate Reagent Powder Pillows	2	25/pkg	23953-66

REQUIRED EQUIPMENT AND SUPPLIES

Description	Quantity Required		Cat. No.
	Per Test	Unit	
Clippers, for opening powder pillows	1	each	968-00
DR/4000 1-Inch Cell Adapter	1	each	48190-00
Sample Cells, matched pair, 1 inch, w/ stopper	2	pair	26126-02

OPTIONAL REAGENTS AND STANDARDS

Ammonia Nitrogen Standard Solution, 10-mg/L as NH ₃ -N	500 mL	153-49
Ammonia Nitrogen Standard Solution, 10-mL Voluette Ampule, 50-mg/L as NH ₃ -N	16/pkg	14791-10
Ammonia Nitrogen Standard Solution, 100-mg/L as NH ₃ -N	500 mL	24065-49
Sodium Hydroxide Standard Solution, 1.00 N.....	100 mL MDB	1045-32
Sodium Hydroxide Standard Solution, 5.0 N.....	59 mL DB	2450-26
Sodium Thiosulfate Standard Solution, 0.1 N.....	100 mL MDB	323-32
Sulfide Inhibitor Reagent Powder Pillows	100/pkg	2418-99
Sulfuric Acid, concentrated, ACS	500 mL	979-49
Sulfuric Acid Standard Solution, 1.000 N.....	100 mL MDB	1270-32
Water, deionized	4 liters	272-56

NITROGEN, Ammonia, continued

OPTIONAL EQUIPMENT AND SUPPLIES

Cylinder, graduated, polypropylene, 500-mL	each.....	1081-49
Distillation Heater and Support Apparatus, 115 VAC.....	each.....	22744-00
Distillation Heater and Support Apparatus, 230 VAC.....	each.....	22744-02
Distillation Apparatus Set, general purpose.....	each.....	22653-00
DR/4000 Carousel Module Kit	each.....	48070-02
DR/4000 Flow Cell Module Kit, 1-inch	each.....	48070-04
DR/4000 Flow Cell Module Kit, 1-cm.....	each.....	48070-05
DR/4000 Sipper Module Kit, 1-inch.....	each.....	48090-03
Filter Paper, folded, 12.5-cm.....	100.....	1894-57
Flask, Erlenmeyer, polypropylene, 500-mL.....	each.....	1082-49
Flask, volumetric, Class A, 100-mL	each.....	14574-42
Funnel, poly, 65-mm	each.....	1083-67
pH Meter, <i>sensio</i> TM 1, portable.....	each.....	517000-00
Pipet Filler, safety bulb.....	each.....	14651-00
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, 2.00-mL.....	each.....	14515-36
Pipet, volumetric, Class A, 5.00-mL.....	each.....	14515-37
Pipet, volumetric, Class A, 6.00-mL.....	each.....	14515-06
Pipet, volumetric, Class A, 8.00-mL.....	each.....	14515-08
Thermometer, -10 to 110 °C.....	each.....	1877-01



FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:

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Outside the U.S.A. – Contact the HACH office or distributor serving you.

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