



Method 10023

Salicylate Method*

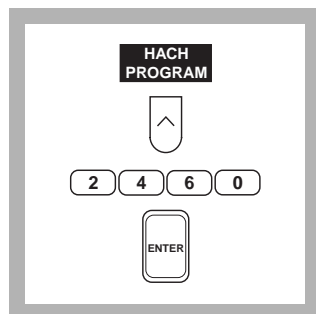
Test 'N Tube™ Vials

LR (0 to 2.500 mg/L NH₃-N)

Scope and Application: For water, wastewater and seawater.

The estimated detection limit for program number 2460 is 0.031 mg/L NH₃-N.

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



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

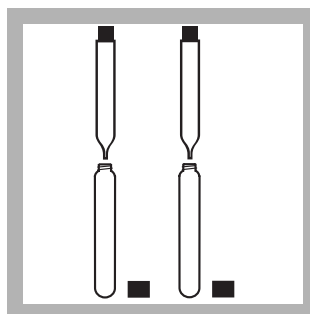
Select the stored program number for Low Range Nitrogen-Ammonia, Test 'N Tube method, by pressing **2460** with the numeric keys.

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.

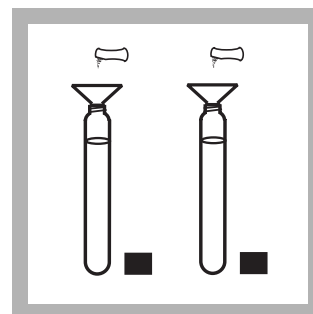


2. The display will show: **HACH PROGRAM: 2460 N, Ammonia LR TNT**
The wavelength (λ), **655nm**, is automatically selected.

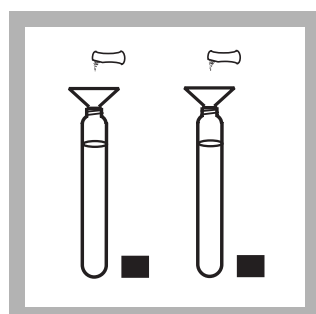


3. Remove the caps from two AmVer Diluent Reagent Test 'N Tubes for Low Range Ammonia Nitrogen. Add 2.0 mL of sample to one vial (the sample). Add 2.0 mL of ammonia-free water to the other vial (the blank).

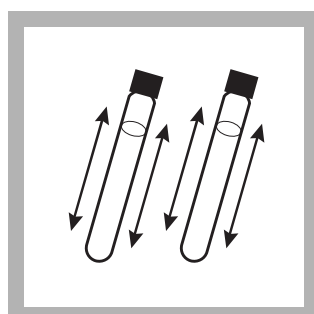
Note: For non-preserved samples with extreme pH, see the *Interferences* section.



4. Using a funnel, add the contents of one Ammonia Salicylate Reagent Powder Pillow for 5 mL sample to each vial.

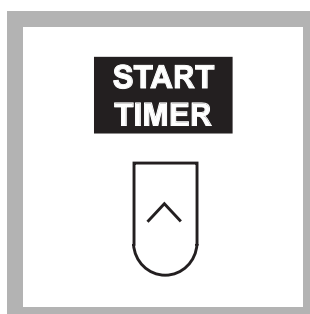


5. Add the contents of one Ammonia Cyanurate Reagent Powder Pillow (for 5-mL sample) to each vial.



6. Cap the vials tightly and shake thoroughly to dissolve the powder.

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

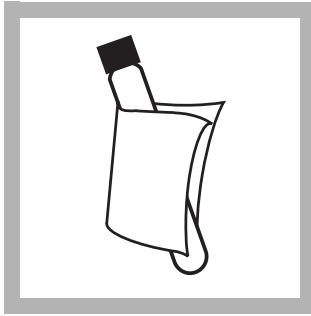


7. Press the soft key under **START TIMER**. A 20-minute reaction period will begin.



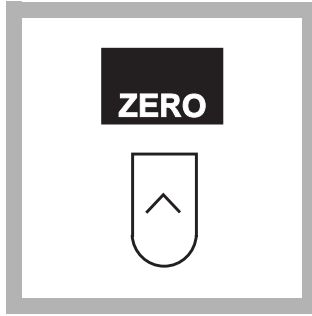
8. Insert the Test Tube Adapter into the sample cell module by sliding it under the thumb screw and into the alignment grooves. Fasten with the thumb screw.

NITROGEN, Ammonia, continued



9. When the timer beeps, clean the outside of the vial with a towel, and place the blank into the cell holder. Close the light shield.

Note: Wiping with a damp cloth followed by a dry one removes fingerprints and other marks.

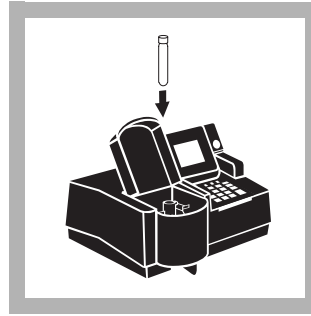


10. Press the soft key under **ZERO**.

The display will show:

0.000 mg/L NH₃-N

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.



11. Place the prepared sample into the cell holder and close the light shield. The result in mg/L ammonia nitrogen (or chosen units) will be displayed.

Note: The results can be expressed as NH₃-N ammonia (NH₃). Press the soft keys under **OPTIONS**, then **FORM**: to scroll through the available options.

Interferences

Interfering Substance	Interference Levels and Treatments
Calcium	2,500 mg/L as CaCO ₃
Iron	<ol style="list-style-type: none"> Determine the amount of iron present in the sample following one of the Iron, Total, procedures. Add the same iron concentration to the ammonia-free water in Step 4. The interference will then be successfully blanked out.
Magnesium	15,000 mg/L as CaCO ₃
Nitrite	30 mg/L as NO ₂ -N
Nitrate	250 mg/L as NO ₃ -N
Orthophosphate	250 mg/L as PO ₄ ³⁻ -P
pH	Acidic or basic samples should be adjusted to approximately pH 7. Use 1 N Sodium Hydroxide Standard Solution for acidic samples and 1 N Hydrochloric Acid Standard Solution for basic samples.
Sulfate	300 mg/L as SO ₄ ²⁻
Sulfide	<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	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> at the end of this procedure.

Sample Collection, Storage and Preservation

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis. 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. Preserve the sample by reducing the pH to 2 or less with hydrochloric acid (at least 2 mL). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Before analysis, warm samples to room temperature and neutralize with 5.0 N Sodium Hydroxide. 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), 25.0.
- c. Press **ENTER** to accept the default standard concentration (mg/L), 50.000.
- d. Press the soft key under **ENTRY DONE**.
- e. Snap the neck off an Ammonia Nitrogen Ampule Standard, 50-mg/L as NH₃-N.
- f. Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard to three 25-mL samples. Mix thoroughly.
- g. Analyze each spiked 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 Solution Method

To check accuracy, use the Nitrogen Ammonia Standard Solution, 1.0-mg/L listed under *OPTIONAL REAGENTS AND STANDARDS*. Or, dilute 1 mL of 50-mg/L Nitrogen Ammonia Standard Solution to 50 mL with deionized water in a 50-mL volumetric flask.

Method Performance

Precision

Standard: 1.000 mg/L NH₃-N

Program	95% Confidence Limits
2460	0.985–1.014 mg/L NH ₃ -N

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

NITROGEN, Ammonia, continued

Estimated Detection Limit

Program	EDL
2460	0.031 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: 2460

Portion of Curve	Δ Abs	Δ Concentration
Entire Range	0.010	0.01604 mg/L

See Section 1.5.3 *Sensitivity Explained* for more information.

Calibration Standard Preparation

To perform an ammonia calibration using the Test 'N Tube LR salicylate method, prepare calibration standards containing 0.5, 1.0, 1.5, 2.0, and 2.5 mg/L NH₃-N as follows:

- a. Into five different 1000-mL Class A volumetric flasks, pipet 5, 10, 15, 20, and 25 mL of a 100-mg/L Ammonia Nitrogen Standard (Cat. No 24065-49) using Class A glassware.
- b. Dilute to the mark with deionized water. Mix thoroughly.
- c. Using the salicylate method and the calibration 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

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. Be sure cyanide solutions are stored in a caustic solution with pH >11 to prevent release of hydrogen cyanide gas. See Section 1 for further information in proper disposal of these materials.

REQUIRED REAGENTS AND STANDARDS

Low Range Test 'N Tube Nitrogen-Ammonia AmVer Reagent Set (25 tests)	Cat. No.
Includes:(50) AmVer Diluent LR Vials*, (1) 23952-66, (1) 23954-66, (1) 272-42	26045-45

	Quantity Required		
Description	per test	Unit	Cat. No.
AmVer Diluent Reagent Test 'N Tube, Low Range.....	2 vials	50/pkg.....	*
Ammonia Cyanurate Reagent Powder Pillows, (5-mL sample) ...	2 pillows	50/pkg.....	23954-66
Ammonia Salicylate Reagent Powder Pillows, (5-mL sample) ...	2 pillows	50/pkg.....	23952-66

REQUIRED EQUIPMENT AND SUPPLIES

DR/4000 Test Tube Adapter.....	1	each.....	48189-00
Funnel, micro, poly	1	each.....	25843-35
Pipet, TenSette, 0–10 mL.....	1	each.....	19700-10
Pipet Tips, for TenSette Pipet 19700-10	varies.....	50/pkg.....	21997-96
Test Tube Rack	1–3	each.....	18641-00

OPTIONAL REAGENTS AND STANDARDS

Nitrogen Ammonia Standard Solution, 1.0-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, 2-mL PourRite Ampule, 50-mg/L NH ₃ -N..	20/pkg.....	14791-20
Nitrogen Ammonia Standard Solution, 10-mL PourRite Ampule, 50-mg/L NH ₃ -N	16/pkg.....	14791-10
Hydrochloric Acid, ACS	500 mL.....	134-49
Sodium Hydroxide Standard Solution, 5.0 N.....	50 mL SCDB.....	2450-26
Sodium Thiosulfate Standard Solution, 0.1 N.....	100 mL MDB.....	323-32

	Unit	
Description		Cat. No.
Sulfide Inhibitor Reagent Powder Pillows	100/pkg.....	2418-99
Water, deionized.....	4 L.....	272-56

OPTIONAL EQUIPMENT AND SUPPLIES

Ampule Breaker Kit, Voluette	each.....	21968-00
Ampule Breaker Kit, PourRite	each.....	24846-00
Distillation Apparatus Set	each.....	22653-00
Distillation Heater & Support Apparatus Set, 120 VAC	each.....	22744-00
Distillation Heater & Support Apparatus Set, 230 VAC	each.....	22744-02
Filter Paper, folded	100/box.....	1894-57
Flask, Erlenmeyer, 500-mL.....	each.....	505-49
Flask, volumetric, Class A, 50.0-mL	each.....	14574-41
Flask, volumetric, Class A, 1000-mL	each.....	14574-53
Funnel, analytical (for filtration).....	each.....	1083-68
pH Indicator Paper, pH 1.0 to 11.0.....	5 rolls/pkg.....	391-33
Pipet, TenSette, 0.1–1.0 mL	each.....	19700-01
Pipet Tips, for TenSette Pipet 19700-01	50/pkg.....	21856-96
Pipet, volumetric, Class A, 5.00-mL.....	each.....	14515-37
Pipet, volumetric, Class A, 10.00-mL.....	each.....	14515-38
Pipet, volumetric, Class A, 15.00-mL.....	each.....	14515-39
Pipet, volumetric, Class A, 20.00-mL.....	each.....	14515-20
Pipet, volumetric, Class A, 25.00-mL.....	each.....	14515-40
Pipet Filler	each.....	12189-00
Thermometer, -20 to 100 °C.....	each.....	566-01
Thermometer, -10 to 260 °C.....	each.....	20959-26

* Not sold separately.



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