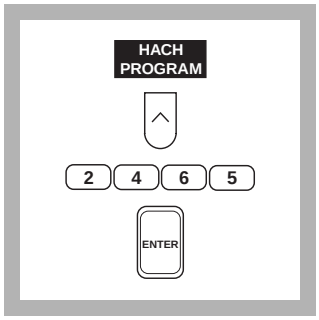
NITROGEN, Ammonia

Method 10031 **Salicylate Method**

Test 'N Tube™ Vials **HR (0 to 50.0 mg/L NH_3–N)**

Scope and Application: *For water, wastewater, and seawater.*
The estimated detection limit for program number 2465 is 0.6 mg/L NH_3–N.



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

Select the stored program number for the High Range Nitrogen Ammonia Test 'N Tube method by pressing **2465** 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.*

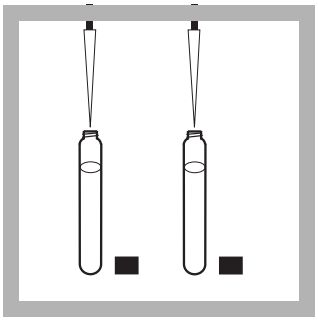
Note: *The Flow Cell and Sipper Modules cannot be used with this method.*

2. The display will show:

HACH PROGRAM: 2465 N, Ammonia HR TNT

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

Note: *For proof of accuracy, use a 10-mg/L Nitrogen Ammonia standard in place of the sample.*

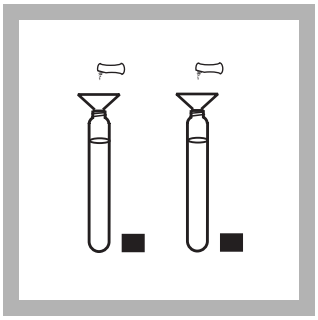


3. Remove the caps from two AmVer Diluent Reagent High Range vials. Add 0.1 mL of ammonia-free water to the other vial (the blank).

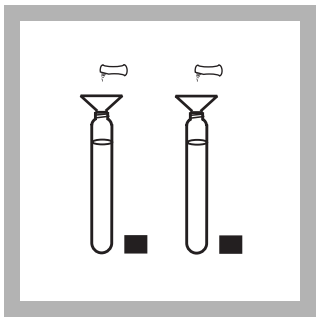
Add 0.1 mL of sample to one vial (the sample).

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

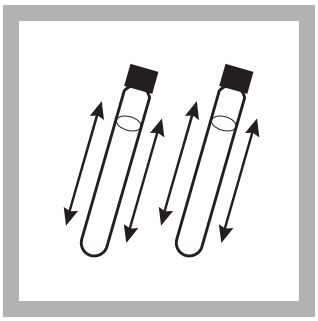
Note: *Small sample sizes may not be representative of the entire sample. Mix the sample well before testing or repeat the test, sampling from different portions of the sample.*



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

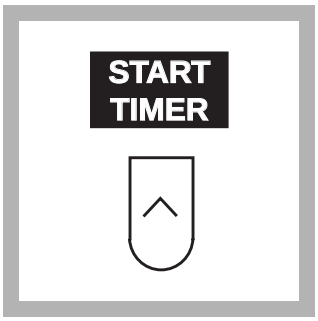


5. Add the contents of one Ammonia Cyanurate Reagent Powder Pillow (for 5-mL sample) to each vial.



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

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



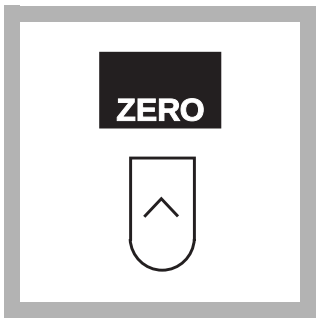
7. Press the soft key under ***START TIMER.***

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.

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.*

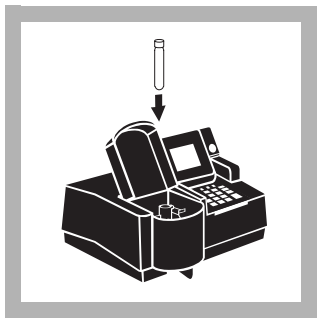


10. Press the soft key under ***ZERO*.**

The display will show:

0.0 mg/L NH_3–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 ammonia (NH_3). Press the soft keys under* ***METHOD OPTIONS****, then* ***FORM:*** *to scroll through the available options. Press* **ENTER** *to return to the read screen.*

Interferences

In some lab environments, airborne cross contamination of the blank is possible. Complete preparation of the blank before opening or handling any samples or standards to avoid transfer of ammonia. If sample or standard containers have already been open, move to a separate area of the lab to prepare the blank.

Substance	Concentration and Suggested Treatments
Acidic or basic samples	Adjust to approximately pH 7. Use 1 N Sodium Hydroxide Standard Solution for acidic samples and 1 N Hydrochloric Acid Standard Solution for basic samples.
Calcium	50,000 mg/L as $CaCO_3$
Glycine, hydrazine	Will cause intensified colors in the prepared sample.
Magnesium	300,000 mg/L as $CaCO_3$
Iron	Eliminate iron interference as follows: **1.** Determine the amount of iron present in the sample using one of the total iron procedures. **2.** Add the same iron concentration to the deionized water in step 4. **3.** The interference will then be successfully blanked out.
Nitrite	600 mg/L as NO_2^-–N
Nitrate	5,000 mg/L as NO_3^-–N
Orthophosphate	5,000 mg/L as PO_4^{3-}–P
Sulfate	6,000 mg/L as SO_4^{2-}

Substance	Concentration and Suggested Treatments
Sulfide	Sulfide will intensify the color. Eliminate sulfide interference as follows: **1.** Measure about 350 mL of sample in a 500 mL erlenmeyer flask. **2.** Add the contents of one Sulfide Inhibitor Reagent Powder Pillow. Swirl to mix. **3.** Filter the sample through folded filter paper. Use the solution in step 4.
Turbidity and color	Give erroneous high values. Samples with severe interferences require distillation. Hach recommends the distillation procedure using the Hach General Purpose Distillation Set.

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 Cl_2 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. Warm samples to room temperature. Neutralize with 5.0 N Sodium Hydroxide before analysis. Correct the test result 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 NH_3–N), 150.0.

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

e. Snap the neck off a Nitrogen, Ammonia Voluette Ampule Standard, 150-mg/L NH_3–N.

f. Use the TenSette Pipet to add 0.2, 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 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 10-mg/L Nitrogen, Ammonia Standard Solution or a 50-mg/L Nitrogen, Ammonia Voluette Ampule Standard listed under *OPTIONAL REAGENTS AND STANDARDS*.

Alternatively, prepare a 40.0-mg/L ammonia nitrogen standard solution by pipetting 4.00 mL of 100-mg/L Ammonia Nitrogen standard into a 100-mL, Class A volumetric flask. Dilute to the mark with deionized water.

To adjust the calibration curve using the reading obtained with the 40.0 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: 10.0 mg/L NH_3–N

Program	95% Confidence Limits
2465	9.6–10.4 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
2465	0.6 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: 2465

Portion of Curve	ΔAbs	ΔConcentration
Entire Range	0.010	0.38 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 HR Salicylate method, prepare calibration standards containing 10.0, 30.0, and 50.0 mg/L NH_3–N as follows:

a. Into three different 50-mL Class A volumetric flasks, pipet 5.00, 15.00, and 25.00 mL of a 100-mg/L Nitrogen Ammonia Standard Solution (Cat. No. 24065-49) using Class A glassware.

b. Dilute to the mark with deionized water. Mix thoroughly.

c. Using the Test 'N Tube HR 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 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

	Cat. No.
High Range Test 'N Tube AmVer Nitrogen Ammonia Reagent Set (50 tests)	26069-45
Includes: (50) HR TNT AmVer Diluent Vials*, (1) 23952-66, (1) 23954-66, (1) 272-42	

Description	Quantity Required per test	Unit	Cat. No.
AmVer Reagent HR TNT Vials	2 vials	50/pkg	*
Ammonia Salicylate Reagent Powder Pillows	2 pillows	50/pkg	23952-66
Ammonia Cyanurate Reagent Powder Pillows	2 pillows	50/pkg	23954-66

REQUIRED EQUIPMENT AND SUPPLIES

DR/4000 Test Tube Adapter	1	each	48189-00
Funnel, micro (for adding reagent)	1	each	25843-35
Test Tube Rack	1	each	18641-00
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	varies	50/pkg	21856-96

OPTIONAL REAGENTS AND STANDARDS

Hydrochloric Acid, ACS	500 mL	134-49
Nitrogen Ammonia Standard Solution, 10-mg/L NH_3–N	500 mL	153-49
Nitrogen Ammonia Standard Solution, 100-mg/L NH_3–N	500 mL	24065-49
Nitrogen Ammonia Standard Solution, 150-mg/L NH_3–N, 10-mL Voluette Ampules	16/pkg	21284-10
Sodium Hydroxide Standard Solution, 5.0 N	50 mL	2450-26
Sodium Thiosulfate Standard Solution, 0.1 N	100 mL	323-32
Sulfide Inhibitor Powder Pillows	100/pkg	2418-99
Water, deionized	4 L	272-56

* Item not sold separately

OPTIONAL EQUIPMENT AND SUPPLIES

Description	Unit	Cat. No.
Distillation Apparatus Set, general purpose	each	22653-00
Distillation Heater & Support Apparatus Set, 115 VAC	each	22744-00
Distillation Heater & Support Apparatus Set, 230 VAC	each	22744-02
Filter Paper, folded, 12.5-cm dia.	100/pkg	692-57
Flask, Erlenmeyer, 500-mL	each	505-49
Flask, volumetric, Class A, 100-mL	each	14574-42
Funnel, analytical, plastic, for filtering	each	1083-67
pH Paper, 1.0 to 11.0 pH units	5 rolls/pkg	391-33
Pipet, serological, 2.00-mL	each	532-36
Pipet, volumetric, Class A, 4.00-mL	each	14515-04
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, 25.00-mL	each	14515-40
Pipet Filler	each	12189-00
Pipet, TenSette, 1.0 to 10.0 mL	each	19700-10
Pipet Tips, for TenSette Pipet 19700-10	50/pkg	21997-96
Thermometer, pocket, -10 to 110 °C	each	1877-01

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