ANALYTICAL PROCEDURES
For DR/2000 and DR/3000 Instruments

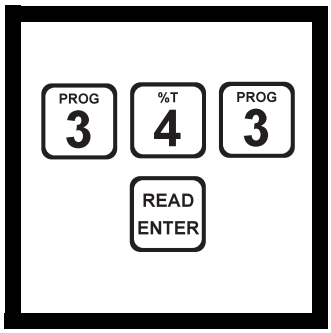
Method 10031

NITROGEN, AMMONIA, High Range Test 'N Tube™ *

(0 to 50.0 mg/L NH_3–N)

Salicylate Method**

For water, wastewater, and seawater



1. Enter the stored program for High Range Test 'N Tube™ Nitrogen, Ammonia.

Press: **3 4 3 READ/ENTER**

The display will show:

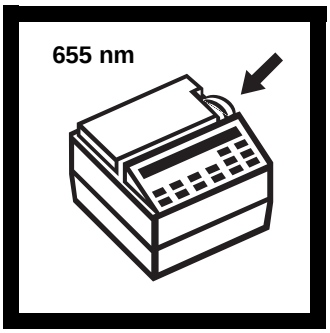
Dial nm to 655

Note: *See Instrument Setup on page 4 to enter this method into the DR/2000.*

Note: *DR/2000s with software versions 3.0 and greater will display* **P** *and the program number.*

Note: *DR/2000s with software versions 3.0 and greater will not display* **DIAL TO** *message if the wavelength is already set correctly. The display will show the message in step 3. Proceed with step 4.*

Note: *If sample cannot be analyzed immediately, see Sampling and Storage on page 8.*

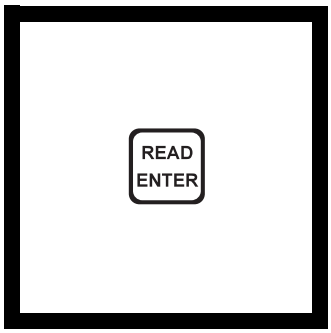


2. Rotate the wavelength dial until the display shows:

655 nm

Note: *For DR/3000 instruments, set the wavelength to 655 nm and press* **CLEAR**.

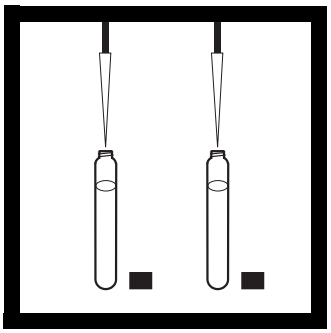
Note: *For proof of accuracy, run a 10 mg/L NH_3–N standard through the analysis.*



3. Press: **READ/ENTER**

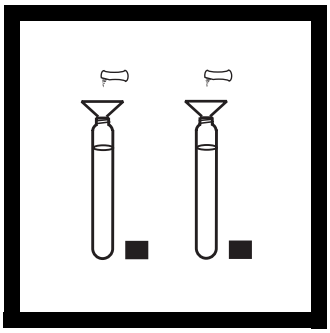
The display will show:

mg/l N Vial HR

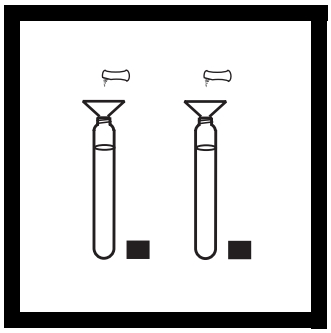


4. Remove the caps from 2 AmVer™ Diluent Reagent High Range Test 'N Tube vials. Add 0.1 mL of deionized water to 1 vial (the blank). Add 0.1 mL of sample to the other (the sample).

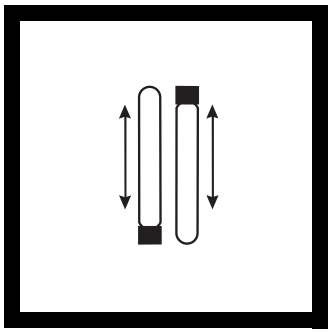
* AmVer™, PourRite™, TenSette®, Test 'N Tube™, and Voluette™ are trademarks of Hach Company.
** Adapted from *Clin. Chim. Acta* 14: 403 (1966).



5. Add the contents of 1 Ammonia Salicylate Reagent Powder Pillow for 5 mL Sample to each vial.



6. Add the contents of 1 Ammonia Cyanurate Reagent Powder Pillow for 5 mL Sample to each vial.



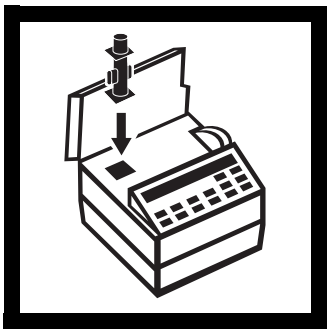
7. Cap the vials tightly and shake thoroughly to dissolve the powder.

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

8. Press: **SHIFT TIMER**

A 20-minute reaction period will begin.

Note: *For DR/3000s, press* **20 TIMER**.



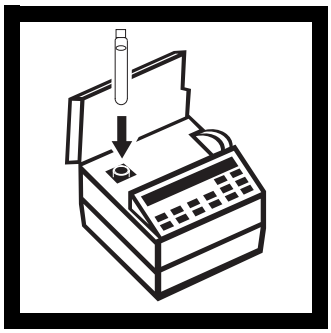
9. Place the COD vial adapter into the cell holder with the marker to the right. When the timer beeps the display will show:

mg/l N Vial HR

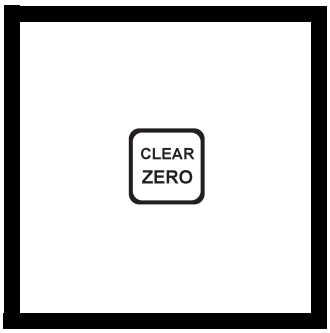
Note: *For DR/3000s, the groove in the adapter faces the front of the instrument.*

10. Clean the outside of both vials with a towel.

Note: *Wipe with a damp cloth and follow by a dry one to remove fingerprints and other marks.*



11. Place the blank into the vial adapter with the Hach logo facing the front of the instrument. Place the cover on the adapter.



12. Press: **ZERO**

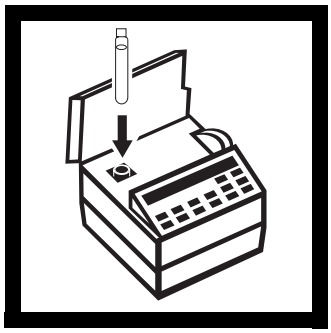
The display will show:

WAIT

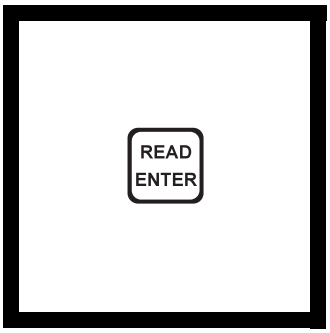
then:

0.0 mg/l N Vial HR

Note: *For DR/3000s, press* **MANUAL PROGRAM**. *Then press* **ZERO**. *Press* **ZERO** *again if the display does not show* **0.000**. *Enter the concentration factor (***35.42***) and press* **CONC FACTOR**. *Then press* **1 CONC**.



13. Place the prepared sample into the vial adapter with the Hach logo facing the front of the instrument. Place the cover on the adapter.



14. Press: **READ/ENTER**

The display will show:

WAIT

then the result in mg/L ammonia nitrogen (N) will be displayed.

Note: *In the constant on mode, pressing* **READ/ENTER** *is not required.* **WAIT** *will not appear. When the display stabilizes, read the result.*

Note: *The result can be expressed as mg/L ammonia (NH_3) by multiplying the mg/L N result by 1.22.*

Instrument Setup

DR/2000 with 1.261 or 1.27 software
Enter the calibration as an operator-programmed calibration. Follow the steps in the Instrument Operation section of the *Instrument Manual*. Store the method as follows:

nm = 655

Decimal = 000.0

Units = mg/l

Symbol = N Vial HR

Timer 1 = 20:00

Enter the calibration with 0.000 absorbance values for zero and #1 standard. To do this, do not place anything in the sample cell compartment. Begin by storing standards 0 and 1 as the concentrations shown in the table below. Accept 0.000 Abs as the absorbance value for all standards. Store the calibration values by pressing **SHIFT READ/ENTER**.

Next, edit the absorbance values for the standards to the values given below.
Follow the steps given in the Instrument Operation section of the *Instrument Manual*.

Std	Conc	Abs
#0	0.0	0.000
#1	55.0	1.553

The method is now stored as an operator-programmed method number between 950 and 999. Record the method number for future reference when using this method.

DR/2000 with software version 2.0 or 2.2
Enter the calibration as an update to Hach-stored programs.

1. Press: 

2. Press: 

3. Press: 

4. Within 3 seconds, press: 

The display will show: **ENTER nm**

5. Press: 

The display will show: **DECIMAL? 00.00**

6. Use the arrow keys to correctly position the decimal point. Press the down arrow key once. The display will show: **DECIMAL? 000.0**

7. Press **READ/ENTER**. The display will show: **UNITS?**

8. Use the arrow keys to select the appropriate unit of measure. Press the down arrow key twice. The display will show: **mg/l**

9. Press **READ/ENTER** when the correct unit of measure is displayed. The display will show: **SYMBOL?**

10. Construct the correct symbol display: **N Vial HR**

    a. Select a letter by scrolling to the correct symbol with the arrow keys.

    b. To make a letter upper case, press the **SHIFT** key.

    c. The space is the character displayed after one press of the down arrow key.

    d. Accept each symbol by pressing **READ/ENTER**.

    e. To end symbol entry, press **READ/ENTER** a second time after accepting the last character.

    When the instrument is out of symbol entry mode, the display will show: **TIMER?**

11. This method has 1 timed step, so press **SHIFT TIMER**. The display will show: **MM:SS TIME 1?**

12. Enter a timer value of 20 minutes. Press: 

13. Press **READ/ENTER** to accept the timer value. The display will show: **MM:SS TIME 2?**

14. Press **READ/ENTER** to complete the timer entry. The display will show: **#1 Data**

15. Enter the following 12 numbers as shown. Complete each entry by pressing the **READ/ENTER** key.

Display	Number Entry
#1 Data	0
#2 Data	11309
#3 Data	11308
#4 Data	11309
#5 Data	11308
#6 Data	11309
#7 Data	11308
#8 Data	11519
#9 Data	65535
#10 Data	5461
#11 Data	512
Checksum	45730

The final number is a check value that determines if the data sequence was entered correctly. If an error was made during number entry, the display will return to the prompt for #1 Data and the entire sequence must be re-entered. If all numbers are correctly entered, the display will return to the method prompt and the instrument is ready for use.

DR/2000 with software version 3.0 or above

1. Turn the instrument on. Press **SHIFT METHOD** to enter the configuration mode. The display will show: **MOMENTARY** or **CONSTANT ON**

2. Press the up arrow key twice to select **HACH UPDATE**. Press **READ/ENTER**. The display will show: **ENTER #:**

3. Press: 

The display will show: **P343 ENTER nm**

4. Press: 

Note: *If you make an error, press* **SHIFT CLEAR** *and re-enter the number. When the number is correct, press* **READ/ENTER.**

   The display will show: **P343 Decimal? 00.00**

5. Use the arrow keys to correctly position the decimal point. Press the down arrow key once. The display will show: **DECIMAL? 000.0**

6. Press: **READ/ENTER.** The display will prompt for the selection of a unit: **P343 UNITS?**

7. Use the arrow keys to select the appropriate unit of measure. Press the down arrow key twice. The display will show: **P343 mg/l**

8. Press **READ/ENTER** when the correct unit of measure is displayed. The display will show: **P343 mg/l _**

9. Construct the display to read the correct symbol. The symbol must be entered exactly as shown including dashes and spaces between characters: **N Vial HR**

   a. Select letters and numbers by scrolling to the correct character with the arrow keys.

   b. To make a letter uppercase, press the **SHIFT** key.

   c. The space is the character displayed after one press of the down arrow.

   d. Make sure to enter the display line EXACTLY as shown, including the spaces. Do not enter trailing spaces.

   e. Accept each symbol by pressing **READ/ENTER.**

   f. When the last character of the symbol is accepted with the **READ/ENTER** key, press **READ/ENTER** a second time to end display entry mode.

   When the instrument is out of symbol entry mode, the display shows: **P343 TIMER?**

10. This method has one timed step, so press **SHIFT TIMER**. The display will show: **MM:SS TIME 1?**

11. Enter a timer value of 20 minutes. Press: 

12. Press **READ/ENTER** to accept the timer value. The display will show: **MM:SS TIME 2?**

13. Press **READ/ENTER** to complete the timer entry. The display will show: **# 0 STANDARD**

14. Press **READ/ENTER** to display the zero data pair. The display will show: **0.000 Abs 000.0 mg/l**

15. Press **READ/ENTER**. The display will show: **#1 STANDARD**

16. Press **READ/ENTER**. The display will prompt for entry of the first concentration point: **#1 000.0 mg/l**

17. Enter concentration point #1 from the table below by pressing **0550** so that the display shows: **# 1 055.0 mg/l**

18. Press **READ/ENTER**. The display will prompt for entry of the first absorbance point: **# 1 0.000 Abs**

19. Enter the absorbance point #1 from the table below by pressing **1553** so that the display shows: **# 1 1.553 Abs**

20. Press **READ/ENTER**. The display will show the first data pair: **1.553 Abs 055.0 mg/l**

21. Press **READ/ENTER** to accept the first data pair. The data pair values from the table below are now entered.

Standard	Concentration	Absorbance
#0	[0.0] mg/l	[0.000] Abs
#1	[55.0] mg/l	[1.553] Abs

When the last data pair is entered the display will show: **#2 STANDARD**

22. Press **SHIFT READ/ENTER** to complete data point entry. The display will show: **#:**

23. Enter the validation number: **4995** so that the display shows: **#: 4995**

24. Press **READ/ENTER**. The display will show: **COMPLETED**

then: **P343 mg/l N Vial HR**

Note: *If the display shows* **INCORRECT #***, then prompts again for the validation number, you may have made an error during data entry. Make sure the validation number is correct. If so, then the error occurred during some other portion of the method entry. Press* **METH** *and respond to the* **ABORT?** *message by pressing* **READ/ENTER**, *then re-enter the method.*

The instrument is now ready for use with method 343.

Sampling and Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis. If chlorine is known to be present, add 1 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 concentrated 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.

Accuracy Check

Standard Solution Method

To check accuracy, use a Nitrogen, Ammonia Standard Solution, 10 mg/L. Or a Nitrogen, Ammonia Voluette™ Ampule Standard, 50 mg/L.

Precision

DR/2000: In a single laboratory, using a standard solution of 25 mg/L ammonia nitrogen (NH_3–N) and 2 representative lots of reagent with the DR/2000 Spectrophotometer, a single operator obtained a standard deviation of ± 0.5 mg/L N.

DR/3000: In a single laboratory, using a standard solution of 25 mg/L ammonia nitrogen (NH_3–N) and 2 representative lots of reagent with the DR/3000 Spectrophotometer, a single operator obtained a standard deviation of ± 0.4 mg/L N.

Interferences

The following ions may interfere when present in concentrations exceeding those listed below

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.

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 yellow color from the excess reagent present will mask the blue and cause the solution to look green.

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 the *Procedures Manual*.

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.

Reagents and Apparatus

REQUIRED REAGENTS

AmVer™ Reagent Set for Nitrogen, Ammonia HR Test 'N Tube™ (50 vials) 26069-45
Includes: (1) 23952-66, (1) 23954-66, (1) 272-42, (50) AmVer HR Vials*

Description	Unit	Cat. No.
AmVer™ HR Reagent Test 'N Tube™ Vials	50/pkg	*
Ammonia Salicylate Reagent Powder Pillows, 5 mL Sample	50/pkg	23952-66
Ammonia Cyanurate Reagent Powder Pillows, 5 mL Sample	50/pkg	23954-66
Water, deionized	100 mL	272-42

REQUIRED APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
COD Vial Adapter, DR/2000, DR/3000	1	each	44799-00
Funnel, micro (for reagent addition)	1	each	25843-35
Pipet, TenSette®, 0.1 to 1.0	1	each	19700-01
Pipet Tips for 19700-01	1	50/pkg	21856-96
Test Tube Rack	1–3	each	18641-00

OPTIONAL REAGENTS

Hydrochloric Acid, ACS	500 mL	134-49
Hydrochloric Acid, 1.0 N	1000 mL	23213-53
Nitrogen, Ammonia Standard Solution, 10 mg/L NH_3–N	500 mL	153-49
Nitrogen, Ammonia Standard Solution, Voluette™ Ampules, 50 mg/L NH_3–N, 10 mL	16/pkg	14791-10
Nitrogen, Ammonia Standard Solution, PourRite™ Ampules, 50 mg/L NH_3–N, 2 mL	20/pkg	14791-20
Sodium Hydroxide Standard Solution, 5.0 N	50 mL	2450-26
Sodium Hydroxide Standard Solution, 1.0 N	100 mL	1045-32
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

OPTIONAL APPARATUS

Cylinder, graduated, 500 mL, poly	each	1081-49
Distillation Apparatus Set, general purpose	each	22653-00
Filter Paper, folded	100/pkg	1894-57
Flask, Erlenmeyer, 500 mL	each	1082-49
Flask, volumetric, 50 mL	each	14574-41
Funnel, analytical (for filtering)	each	1083-68
Heater and Support Apparatus (for distillation), 115 Vac	each	22744-00
Heater and Support Apparatus (for distillation), 230 Vac	each	22744-02
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
Pipet, serological, 2 mL	each	532-36

* This item is not sold separately. Please order the complete set (Cat. No. 26069-45) as a replacement.

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