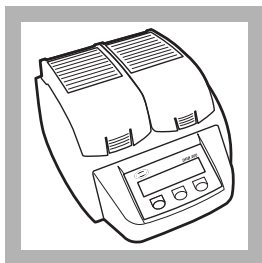


NITROGEN, TOTAL, HR, Test 'N Tube™ (10.0 to 150.0 mg/L N)

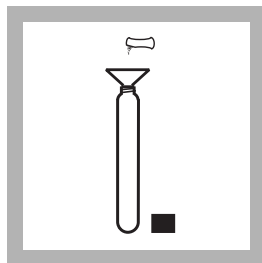
TNT Persulfate Digestion Method

For water and wastewater



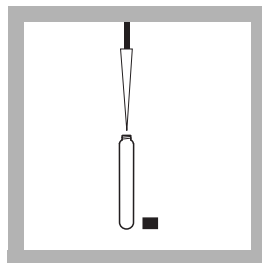
1. Turn on the DRB 200 Reactor. Heat to 103-106 °C (optimum temperature is 105 °C).

Note: For proof of accuracy, run a 125 mg/L NH₃-N standard through digestion and analysis.



2. Prepare a reagent blank: Using a funnel, add the contents of one Total Nitrogen Persulfate Reagent Powder Pillow to one HR Total Nitrogen Hydroxide Digestion Vial.

Note: Wipe off any reagent that gets on the lid or the tube threads.



3. Add 0.5 mL of organic-free water to the vial. Cap the vial and shake vigorously for about 30 seconds.

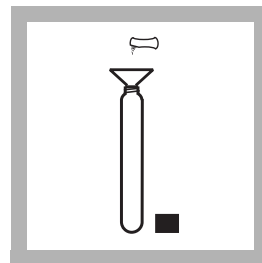
Process this reagent blank exactly the same as the sample, including digestion and color finish. Proceed to step 6.

Note: Alternate water must be free of all nitrogen-containing species.

Note: The persulfate reagent may not dissolve completely after shaking.

Note: One reagent blank is sufficient for each set of samples using the same lots of reagents.

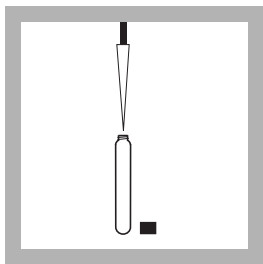
Note: The reagent blank is stable for as long as seven days when stored in the dark; see Blanks for Colorimetric Measurement following this procedure.



4. Prepare a sample: Using a funnel, add the contents of one Total Nitrogen Persulfate Reagent Powder Pillow to one HR Total Nitrogen Hydroxide Digestion Vial.

Note: Wipe off any reagent that gets on the lid or the tube threads.

NITROGEN, TOTAL, HR, Test 'N Tube, continued

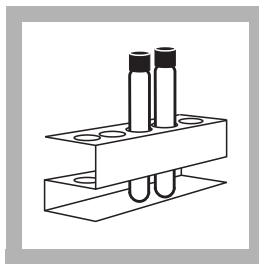


5. Add 0.5 mL of sample to the vial. Cap the vial and shake vigorously for about 30 seconds.

Note: The persulfate reagent may not dissolve completely after shaking.

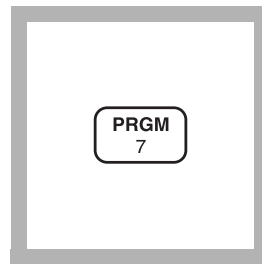


6. Place the vials in the Reactor. Heat for 30 minutes.



7. Using finger cots or gloves, remove the hot vials from the reactor and allow to cool to room temperature.

Note: It is very important to remove the vials from the Reactor after exactly 30 minutes.

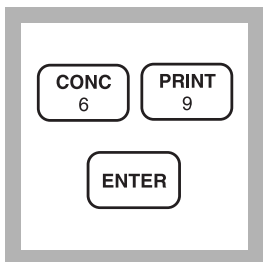


8. Enter the stored program number for Test 'N Tube HR Total Nitrogen.

Press: **PRGM**

The display will show:

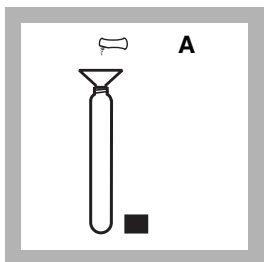
PRGM ?



9. Press: 69 ENTER

The display will show **mg/L, N** and the **ZERO** icon.

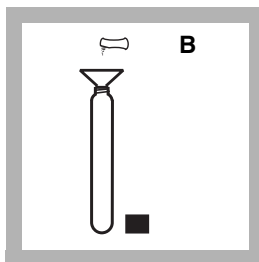
*Note: For alternate forms (NH_3 , NO_3), press the **CONC** key.*



10. Add the contents of one Total Nitrogen Reagent A Powder Pillow to the vial containing the digested blank or sample. Cap the vial and shake for 15 seconds.

Press: **TIMER ENTER** after shaking.

A three-minute reaction period will begin.



11. After the timer beeps, add one Total Nitrogen Reagent B Powder Pillow to the vial. Cap the vial and shake for 15 seconds.

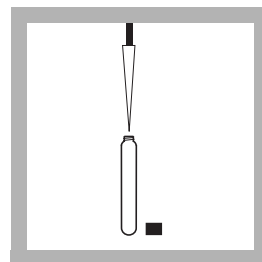
The display will show:

02:00 Timer 2

Press **ENTER** after shaking.

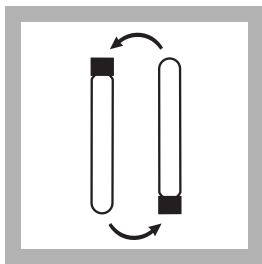
A two-minute reaction period will begin.

Note: The reagent will not completely dissolve. The solution will begin to turn yellow.



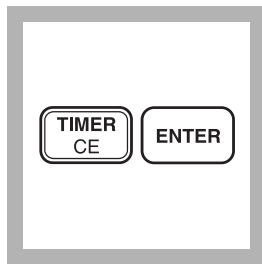
12. After the timer beeps, remove the cap from one Total Nitrogen Reagent C Vial. Add 2 mL of digested, treated sample (or reagent blank) to the vial. The vial will be warm.

NITROGEN, TOTAL, HR, Test 'N Tube, continued



13. Cap and invert slowly 10 times to mix. The vial will be warm.

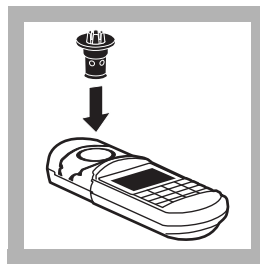
Note: Proper mixing is important for complete recovery. Hold the vial vertical with the cap up. Invert the vial and wait for all of the solution to flow to the cap end. Pause. Return the vial to the upright position and wait for all of the solution to flow to the vial bottom. This is one inversion (10 inversions = 30 seconds).



14. The display will show: **05:00 Timer 3**
Press: **ENTER**

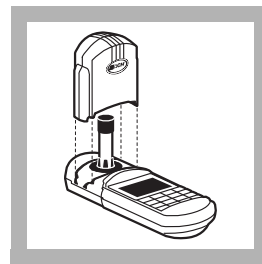
A five-minute reaction period will begin. Do not invert the vial again.

Note: The yellow color will intensify.



15. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

Note: For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.



16. When the timer beeps, wipe the outside of the Total Nitrogen Reagent C vial containing the reagent blank.

Place the vial into the adapter with the Hach logo facing the front of the instrument.

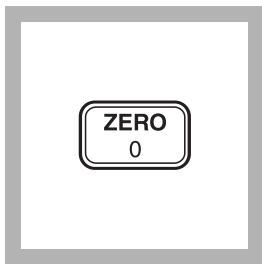
Push straight down on the top of the vial until it seats solidly into the adapter.

Tightly cover the vial with the instrument cap.

Note: Do not move the vial from side to side during insertion, as this can cause errors.

Note: Wipe with a damp towel, followed by a dry one, to remove fingerprints or other marks.

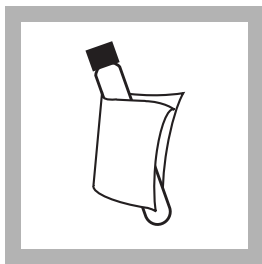
NITROGEN, TOTAL, HR, Test 'N Tube, continued



17. Press: ZERO

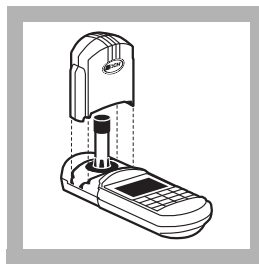
The cursor will move to the right, then the display will show:

0 mg/L N



18. Wipe the Total Nitrogen Reagent C vial containing the sample.

Note: Wipe with a damp towel, followed by a dry one, to remove fingerprints or other marks.



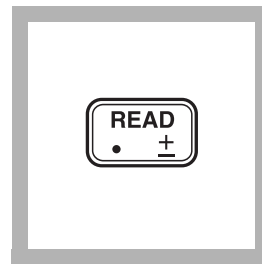
19. Place the vial into the adapter with the Hach logo facing the front of the instrument.

Push straight down on the top of the vial until it seats solidly into the adapter.

Tightly cover the vial with the instrument cap.

Note: Do not move the vial from side to side during insertion, as this can cause errors.

Note: Multiple samples may be read after zeroing on one reagent blank.



20. Press: READ

The cursor will move to the right, then the result in mg/L nitrogen (N) will be displayed.

Note: Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1 of the Procedures Manual).

*Note: If the display flashes **Limit**, dilute the sample and repeat the digestion and the colorimetric finish. The digestion must be repeated for accurate results; diluting and repeating the color finish does not yield complete results. Multiply the result by the dilution factor; see SECTION 1.*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis.

Preserve the sample by reducing the pH to 2 or less with concentrated sulfuric acid (at least 2 mL/L). 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 *Correcting for Volume Additions* in Section 1.

NITROGEN, TOTAL, HR, Test 'N Tube, continued

Accuracy Check

This method generally yields 95-100% recovery on organic nitrogen standards. For proof of accuracy Hach offers a set of three Primary Standards for Kjeldahl Nitrogen.

1. Prepare one or more of the following three solutions. Each preparation is for an equivalent 120 mg/L N standard. Use water that is free of all organic and nitrogen-containing species.
 - a) Weigh 1.6208 g of Ammonium p-Toluenesulfonate (PTSA). Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.
 - b) Weigh 2.1179 g of Glycine p-Toluenesulfonate. Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.
 - c) Weigh 2.5295 g of Nicotinic p-Toluenesulfonate. Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.
2. Analyze each of these solutions using the test procedure above. Calculate the percent recovery for each using this formula:

$$\% \text{ recovery} = \frac{\text{measured concentration}}{120} \times 100$$

The percent recovery should be:

Compound	Lowest Expected % Recovery
Ammonia-PTSA	95%
Glycine-PTSA	95%
Nicotinic-PTSA	95%

Hach analysts have found Ammonia-PTSA to be the most difficult to digest. Other compounds may yield different percent recoveries.

Standard Solution Method

For proof of accuracy, substitute 0.5 mL of a 125 mg/L ammonia nitrogen standard solution for the sample in the procedure. To prepare a 125-mg/L standard, use a 25-mL Class A pipet to transfer 25.00 mL of a 1000-mg/L Ammonia Nitrogen Standard

NITROGEN, TOTAL, HR, Test 'N Tube, continued

(see *OPTIONAL REAGENTS* on page 398) to a 200-mL Class A volumetric flask. Dilute to the line with organic-free water.

Standard Additions Method

- a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- b) Snap the neck off an Ammonia Nitrogen Voluette™ Ampule Standard Solution, 1000 mg/L as NH₃-N.
- c) Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to the three mixing cylinders.
- d) Stopper each cylinder and mix thoroughly.
- e) Add 0.5 mL of each prepared solution, respectively, to three HR Total Nitrogen Hydroxide Digestion Vials.
- f) Analyze each standard addition sample as described in the procedure. The nitrogen concentration should increase 4 mg/L N for each 0.1 mL of standard added.
- g) If these increases do not occur, see *Standard Additions in Section 1* for troubleshooting information.

Blanks for Colorimetric Measurement

The reagent blank may be used repeatedly for measurements using the same lots of reagents. Store the reagent blank in the dark at room temperature (18–25 °C) for a maximum of seven days. If a small amount of white floc appears prior to the end of one week, discard the reagent blank and prepare a new one.

Method Performance

Precision

In a single laboratory, using a standard solution of 125 mg/L N and two lots of reagent with the instrument, a single operator obtained a standard deviation of less than 3 mg/L N. For more information on Hach's precision statement, see *Section 1*.

Estimated Detection Limit

The estimated detection limit for program 69 is 7 mg/L N. For more information on the estimated detection limit, see *Section 1*.

NITROGEN, TOTAL, HR, Test 'N Tube, continued

Interferences

Interfering substances that resulted in a concentration change of $\pm 10\%$:

Substance	Level and Effect
Bromide	> 240 ppm; positive interference
Chloride	≥ 3000 ppm; positive interference

The substances in the following table have been tested and found **not** to interfere up to the indicated levels:

Substance	Maximum Level Tested (mg/L)
Barium	10.4
Calcium	1200
Chromium (3+)	2
Iron	8
Lead	26.4 ppb
Magnesium	2000
Organic Carbon	600
pH	13 pH units
Phosphorus	400
Silica	600
Silver	3.6
Tin	6.0

The large amounts of nitrogen-free organic compounds in some samples may decrease digestion efficiency by consuming some of the persulfate reagent. Samples known to contain high levels of organics should be diluted and re-run to verify digestion efficiency.

Summary of Method

An alkaline persulfate digestion converts all forms of nitrogen to nitrate. Sodium metabisulfite is added after the digestion to eliminate halogen oxide interferences. Nitrate then reacts with chromotropic acid under strongly acidic conditions to form a yellow complex with an absorbance maximum near 420 nm.

NITROGEN, TOTAL, HR, Test 'N Tube, continued

REQUIRED REAGENTS

Test 'N Tube HR Total Nitrogen Reagent Set (50 vials) 27141-00
Includes: (1) 26718-46, (1) 26719-46, (1) 26720-46, *(50) Hydroxide Digestion Vials,
*(50) Acid Solution Vials

Description	Quantity Required		Unit	Cat. No.
	Per Test			
HR Total Nitrogen Hydroxide Digestion Vials.....	1 vial	50/pkg		*
Total Nitrogen Persulfate Reagent Powder Pillows....	1 pillow	50/pkg		26718-46
Total Nitrogen Reagent A, Bisulfite Powder Pillows .1	1 pillow	50/pkg		26719-46
Total Nitrogen Reagent B, Indicator Powder Pillows.1	1 pillow	50/pkg		26720-46
Total Nitrogen Reagent C Vials, Acid Solution.....	1 vial	50/pkg		*

REQUIRED APPARATUS

DRB 200 Reactor, 110 V, 15 x 16 mm tubes			LTV082.53.40001	
DRB 200 Reactor, 220 V, 15 x 16 mm tubes			LTV082.52.40001	
COD/TNT Adapter	1	each	48464-00	
Funnel, micro	1	each	25843-35	
Pipet, TenSette, 0.1 to 1.0 mL.....	1	each	19700-01	
Pipet Tips for 19700-01	2	50/pkg	21856-96	
Test Tube Rack, for cooling vials	1-3	each	18641-00	

OPTIONAL REAGENTS

Nitrogen, Ammonia, 1000 mg/L NH ₃ -N.....	1 L		23541-53	
Nitrogen, Ammonia, Voluette Ampule, 1000 mg/L NH ₃ -N, 10 mL	16/pkg		23541-10	
Sulfuric Acid, ACS	500 mL		979-49	
Primary Standards for Kjeldahl Nitrogen	set of 3		22778-00	
Ammonium p-Toluenesulfonate	25 g		22779-24	
Glycine p-Toluenesulfonate	25 g		22780-24	
Nicotinic Acid p-Toluenesulfonate	25 g		22781-24	
Sodium Hydroxide Standard Solution, 5.0 N	50 mL	MDB	2450-26	
Wastewater Influent Standard, Inorganics (NH ₃ -N, NO ₃ -N, PO ₄ , COD, SO ₄ , TOC).....	500 mL		28331-49	
Water, organic-free.....	500 mL		26415-49	

* These items are not sold separately. Please order the complete set (Cat. No. 27141-00) as a replacement.

NITROGEN, TOTAL, HR, Test 'N Tube, continued

OPTIONAL APPARATUS

Description	Unit	Cat. No.
Ampule Breaker Kit	each.....	21968-00
Balance, analytical, 115 Vac.....	each.....	28014-01
Balance, analytical, 230 Vac	each.....	28014-02
Cots, finger	2/pkg.....	14647-02
Cylinder, graduated, mixing, 25 mL	3..... each.....	26363-40
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm.....	LTV082.53.42001	
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm.....	LTV082.52.42001	
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm.....	LTV082.53.30001	
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm.....	LTV082.52.30001	
Flask, volumetric, Class A, 1000 mL	3..... each.....	14574-53
Flask, volumetric, Class A, 200 mL.....	each.....	14574-45
Pipet, volumetric, Class A, 25 mL	2..... each.....	14515-40
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg.....	21856-28
pH Paper, 1 to 11 pH units	5 rolls/pkg.....	391-33

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224. Out side the U.S.A— Contact the Hach office or distributor serving you.

Outside the U.S.A.—Contact the Hach office or distributor serving you.