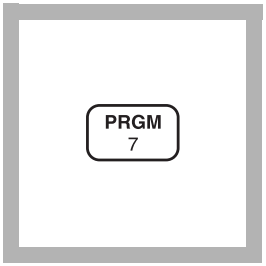
NITROGEN, Total Inorganic, Test 'N Tube™ (0 to 25.0 mg/L N)

Titanium Trichloride Reduction Method

For water, wastewater, and seawater

Requires Centrifuge

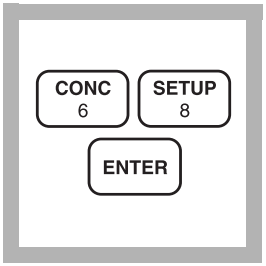


1. Enter the stored program number for Test 'N Tube Total Inorganic Nitrogen.

Press: **PRGM**

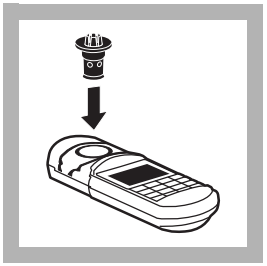
The display will show:

PRGM ?



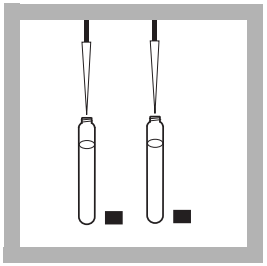
2. Press: **68 ENTER**

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

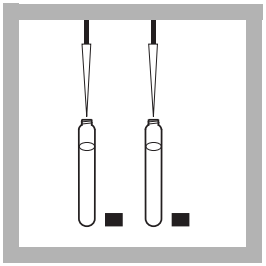


3. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert.

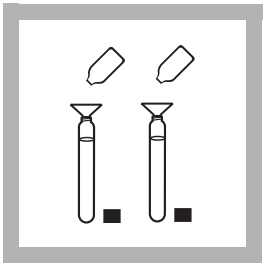
Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



4. Pipet 1 mL of Total Inorganic Nitrogen Pretreatment Base Concentrate into each of 2 Total Inorganic Nitrogen Pretreatment Diluent Vials.



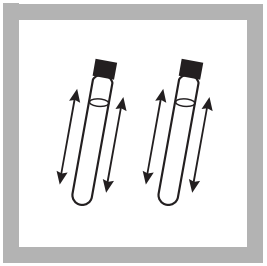
5. Pipet 1 mL of sample into 1 TIN Diluent Vial (the sample). Pipet 1 mL of deionized water into the other vial (the blank). Cap the vials and shake for 30 seconds to mix.



6. Snap the necks off two Total Inorganic Nitrogen Reductant ampules and pour the contents of one into the TIN Diluent Vial containing sample. Repeat for the second vial, the blank.

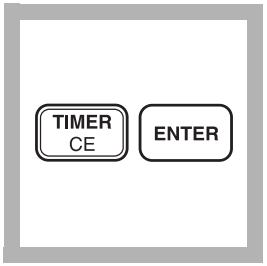
Note: *For safety, wear gloves while breaking the ampules.*

Note: *A black precipitate will form immediately.*



7. Cap the vials. Shake gently for 30 seconds to mix the reagents. Allow the vials to sit for at least one minute.

Note: *The precipitate should remain black after shaking. Excessive shaking will cause a white precipitate and low results.*

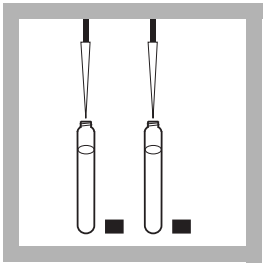


8. Centrifuge the vials for 3 minutes or until the solids settle to the bottom of the vial.

Press: **TIMER ENTER**

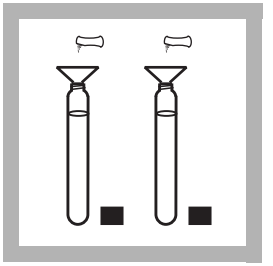
immediately after starting the centrifuge.

Note: *The precipitate will settle without using a centrifuge, but it may take up to 30 minutes.*

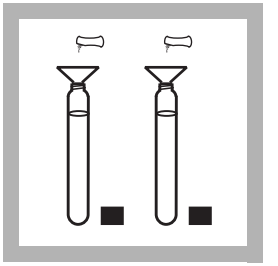


9. Remove the caps from 2 AmVer Diluent Reagent Test 'N Tubes for Low Range Ammonia Nitrogen. Using a pipet, add 2 mL of centrifuged sample into 1 vial. Add 2 mL of centrifuged blank to the other vial. Label the vials appropriately.

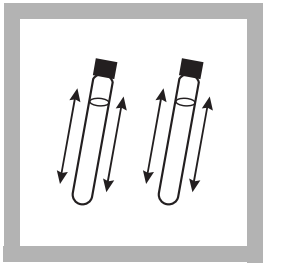
Note: *Pipet carefully to avoid disturbing the sediment.*



10. Using a funnel, add the contents of one Ammonia Salicylate Reagent Powder Pillow to each vial.

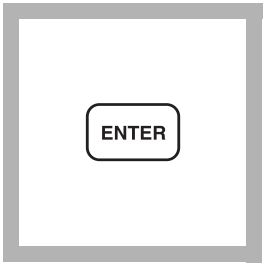


11. Using a funnel, add the contents of one Ammonia Cyanurate Reagent Powder Pillow to each vial.



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

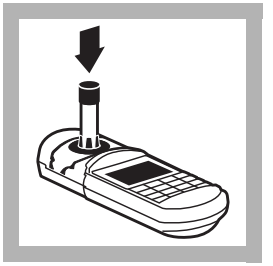
Note: *A green color will develop if inorganic nitrogen is present.*



13. The display will show: **15:00 TIMER 2**

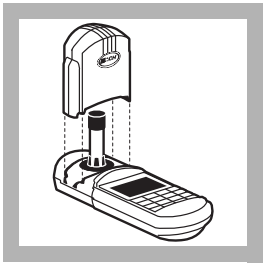
Press: **ENTER**

A 15-minute reaction period will begin.

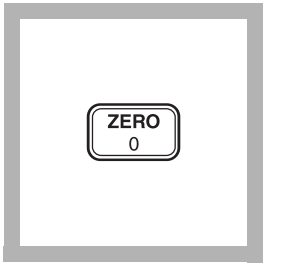


14. After the timer beeps, clean the outside of the vials with a towel. Place the blank in the adapter. Push straight down on the top of the vial until it seats solidly into the adapter. Do not move the vial from side to side as this can cause errors.

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



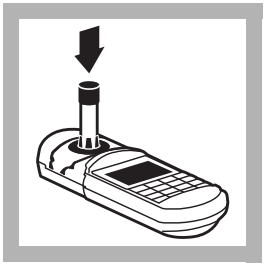
15. Tightly cover the sample cell with the instrument cap.



16. Press: **ZERO**

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

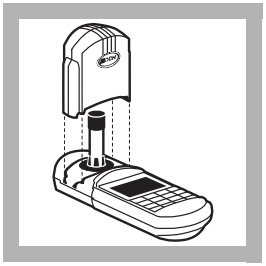
0.0 mg/L N



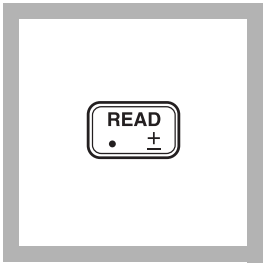
17. Place the prepared sample in the adapter.

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

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



18. Tightly cover the sample cell with the instrument cap.



19. Press: **READ**

The cursor will move to the right, then the result in mg/L total inorganic nitrogen will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

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. 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*.

Accuracy Check

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off a fresh High Range Nitrate Nitrogen PourRite Ampule Standard, 500 mg/L NO_3^--N.

c) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard, respectively, to 3 25-mL mixing cylinders. Mix thoroughly.

d) Analyze each sample as described in the procedure; use a 1-mL aliquot of the prepared sample in Step 5. The nitrogen concentration should increase about 1.8 to 1.9 mg/L for each 0.1 mL of standard added.

e) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

To check accuracy, use a 10.0 mg/L Nitrate Nitrogen Standard Solution listed under Optional Reagents. Alternatively, a 20.0 mg/L nitrate nitrogen standard can be prepared by diluting 2 mL of solution from a PourRite Ampule Standard for High Range Nitrate Nitrogen, 500 mg/L NO_3^--N, to 50 mL with deionized water. Substitute this standard for the sample and perform the test as described. The recovery of the standards should be about 90-95%.

Method Performance

Precision/Accuracy

The total inorganic nitrogen test provides an estimate of the total nitrite, nitrate, and ammonia nitrogen load in water or wastewater samples. This test is most applicable for monitoring an industrial process stream or a wastewater treatment stream where it is important to track the inorganic nitrogen load as it passes through the treatment process. The test exhibits different recoveries of each of the three nitrogen species, as summarized below. This test is not recommended for quantifying only one of the three species. In that case, use a specific procedure for each particular analyte.

Ammonia Nitrogen

In a single laboratory, using a standard solution of 20.0 mg/L NH_3^-N and 2 representative lots of reagent with the instrument, a single operator obtained a mean recovery of 21.3 mg/L with a standard deviation of
± 0.77 mg/L N (replicate number = 7 per reagent lot).

Nitrate Nitrogen

In a single laboratory, using a standard solution of 20.0 mg/L NO_3^-N and 2 representative lots of reagent with the instrument, a single operator obtained a mean recovery of 18.9 mg/L with a standard deviation of
± 0.55 mg/L N (replicate number = 7 per reagent lot).

Nitrite Nitrogen

In a single laboratory, using a standard solution of 20.0 mg/L NO_2^-N and 2 representative lots of reagent with the instrument, a single operator obtained a mean recovery of 14.6 mg/L with a standard deviation of
± 0.77 mg/L N (replicate number = 7 per reagent lot).

Estimated Detection Limit
The estimated detection limit for program 68 is 0.7 mg/L N. For more information on the estimated detection limit, *see Section 1*.

Interferences

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

Species	Level	Effect
Calcium	1000 mg/L as $CaCO_3$	Positive
Manganese (IV)	3 mg/L	Negative
Magnesium	1000 mg/L as $CaCO_3$	Positive
Sulfide	3 mg/L	Negative
Sulfate	250 mg/L	Negative

The following do not interfere below the levels listed:

Species	Level
Al^{3+}	8 mg/L
Ba^{2+}	40 mg/L
Cu^{2+}	40 mg/L
Fe^{3+}	8 mg/L
Zn^{2+}	80 mg/L
F^-	40 mg/L
PO_4^{3-}-P	8 mg/L
SiO_2	80 mg/L
EDTA	80 mg/L

Summary of Method

Titanium (III) ions reduce nitrate and nitrite to ammonia in a basic environment. After centrifugation to remove solids, the ammonia is combined 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.

REQUIRED REAGENTS

Total Inorganic Nitrogen Pretreatment Reagent Set ($TiCl_3$ Reduction) (25 tests) 26049-45
Includes: (1) 26051-50, (1) 2040-59, *(50) TIN Pretreatment Diluent Vials

AmVer™ Reagent Set for Nitrogen, Ammonia, Low Range (25 tests) 26045-45
Includes: (1) 23952-66, (1) 23954-66 , (1) 272-42, *(50) AmVer™ Diluent LR Vials

Description	Quantity Required Per Test	Unit	Cat. No.
Total Inorganic Nitrogen Pretreatment Diluent Vials	2 vials	50/pkg	*
Total Inorganic Nitrogen Reductant Ampules	2 ampules	50/pkg	26051-50
Total Inorganic Nitrogen Pretreatment Base Concentrate	2 mL	50 mL	2040-59
AmVer™ Diluent Reagent, Low Range Vials	2 vials	50/pkg	*
Ammonia Salicylate Reagent Powder Pillows for 5-mL sample	2 pillows	50/pkg	23952-66
Ammonia Cyanurate Reagent Powder Pillows for 5-mL sample	2 pillows	50/pkg	23954-66

REQUIRED APPARATUS

Centrifuge, 115V	1	each	26765-00
Centrifuge, 230V	1	each	26765-02
COD/TNT Vial Adapter	1	each	48464-00
Funnel, micro	1	each	25843-35
Pipet, TenSette®, 0.1 to 1.0	1	each	19700-01
Pipet Tips for 19700-01	2	50/pkg	21856-96
Test Tube Rack	1	each	18641-00

OPTIONAL REAGENTS

Hydrochloric Acid, ACS	500 mL	134-49
Nitrate Nitrogen Standard Solution, 10 mg/L NO_3^--N	500 mL	307-49
Nitrate Nitrogen Standard Solution, PourRite Ampules, 500 mg/L NO_3^--N, 2 mL	20/pkg	14260-20
Sodium Hydroxide Standard Solution, 5.0 N	50 mL SCDB	2450-26
Sodium Thiosulfate Standard Solution, 0.1 N	100 mL MDB	323-32
Wastewater Effluent Standard, Inorganics (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28332-49
Water, deionized	4 L	272-56

* These items are not sold separately. Please order the complete set (cat. no. 26049-45 or 26045-45).

OPTIONAL APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
Cylinder, graduated, mixing, 25 mL		each	20886-40
Flask, volumetric, Class A, 50.0 mL		each	14574-41
pH Indicator Paper, 1 to 11 pH		5 rolls/pkg	391-33
Pipet, volumetric, Class A, 2.0 mL		each	14515-36
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28
PourRite Ampule Breaker		each	24846-00

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

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