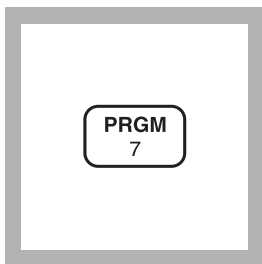


Indophenol Method*

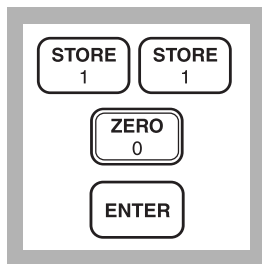
(0–4.50 mg/L Cl₂ and 0–0.50 mg/L NH₃-N)
For finished chloraminated drinking water

Note: For the most accurate chloramine results, determine a reagent blank for each new lot of reagent using deionized water instead of sample. Subtract the blank value from the final chloramine result.



1. Enter the user program number for monochloramine.

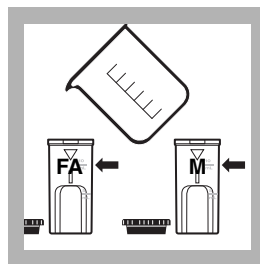
Press: **PRGM**
The display will show:
PRGM?



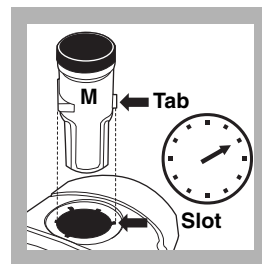
2. Press:
110 ENTER

The display will show
mg/L Cl₂
and the zero icon.

Note: For alternate forms, press the **CONC** key.

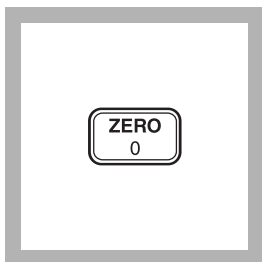


3. Fill two cells with 10 mL of sample
Label one cell “Free Ammonia” and one cell “Monochloramine”.



4. Place the Monochloramine cell into the instrument so that the cell tab is at the two-o'clock position. Make sure the sample cell tab is completely seated in the cell holder slot.

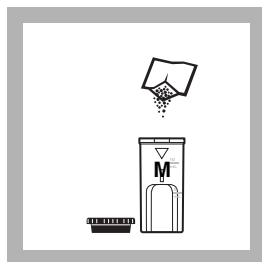
Tightly cover the sample cell with the instrument cap.



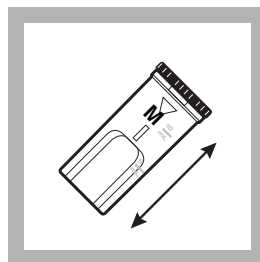
5. Press: **ZERO**

The cursor will move to the right, then the display will show:
0.00 mg/L Cl₂

Remove the cell from the instrument.

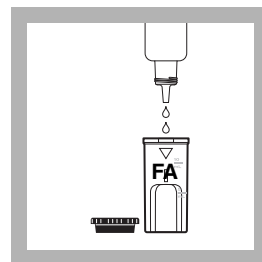


6. Add the contents of one pillow of Monochlor F to the cell for the Monochloramine measurement.



7. Cap the cell and shake for 20 seconds to dissolve the reagent.

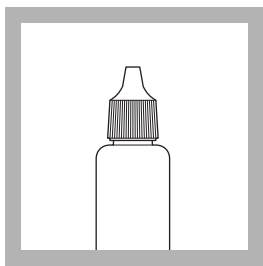
A green color will form if monochloramine is present.



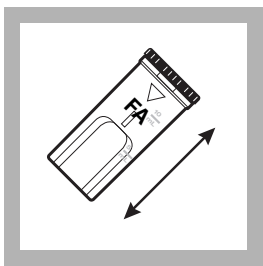
8. Add one drop of Free Ammonia Reagent Solution to the cell for Free Ammonia measurement.

* U.S. Patent 6,315,950

Nitrogen, Free Ammonia and Chloramine (Mono), continued

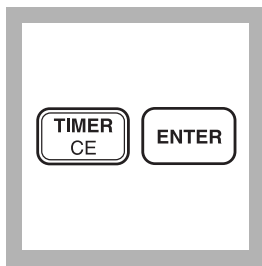


9. Cap the reagent bottle to maintain reagent performance and stability.



10. Cap the cell and mix.

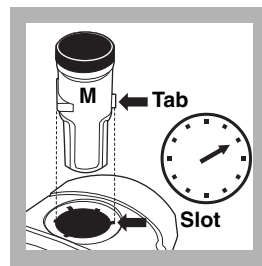
Note: If the sample becomes cloudy by the end of the reaction period, pretreat the sample and retest. See Interferences on page 294.



11. Press: **TIMER ENTER**

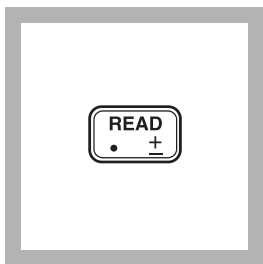
A five-minute reaction period will begin.

Note: The color development time depends on the sample temperature. See Table 1. For accurate results allow the full reaction period to occur.



12. When the timer expires, place the Monochloramine cell into the instrument so that the cell tab is in the two-o'clock position. Make sure the sample cell tab is completely seated in the cell holder slot.

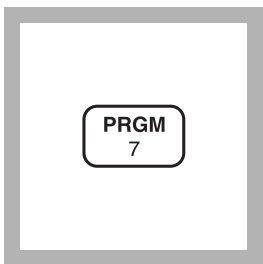
Tightly cover the sample cell with the instrument cap.



13. Press: **READ**

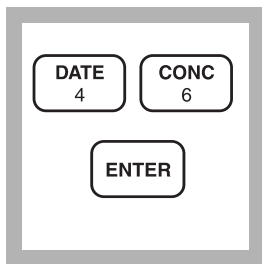
The cursor will move to the right, then the result in mg/L Monochloramine (as Cl_2 or chosen units) will be displayed.

Leave the cell in the instrument.



14. Enter the stored program number for Free Ammonia.

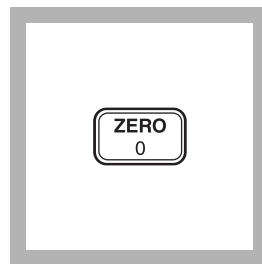
Press: **PRGM**
The display will show PRGM?



15. Press: **46 ENTER**

The display will show $\text{NH}_3\text{-N}$ and the zero icon.

*Note: For alternate forms, press the **CONC** key.*



16. With the Monochloramine sample still in the cell holder, press **ZERO**.

The cursor will move to the right, then the display will show: 0.00 mg/L $\text{NH}_3\text{-N}$.

Remove the cell from the instrument.

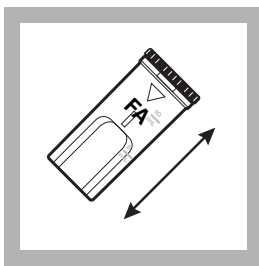
Nitrogen, Free Ammonia and Chloramine (Mono), continued



17. Add the contents of one pillow of Monochlor F to the cell for the Free Ammonia measurement.

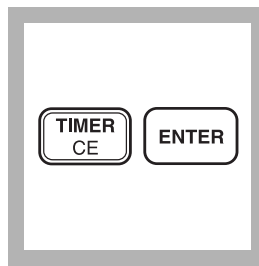
Cap and shake the cell about 20 seconds to dissolve the reagent.

Note: The reaction period indicated in step 11 must be complete before the addition of Monochlor F to the cell for free ammonia measurement.



18. Cap and shake the cell about 20 seconds to dissolve the reagent.

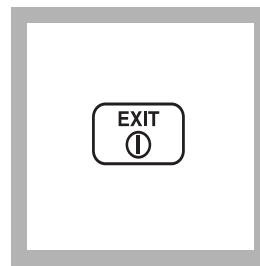
A green color will form if ammonia or monochloramine is present.



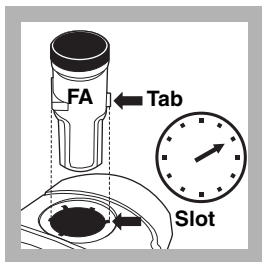
19. Press: **TIMER ENTER**

A five-minute reaction period will begin.

Note: The color development time depends on the sample temperature. See Table 1.

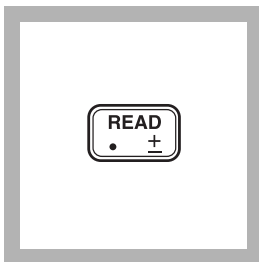


20. After the timer has expired, press: **EXIT**



21. Place the Free Ammonia cell into the instrument so that the cell tab is at the two-o'clock position. Make sure the sample cell tab is completely seated in the cell holder slot.

Tightly cover the sample cell with the instrument cap.



22. Press: **READ**

The cursor will move to the right, then the result in mg/L free ammonia as nitrogen ($\text{NH}_3\text{-N}$) or chosen units will be displayed.

Nitrogen, Free Ammonia and Chloramine (Mono), continued

Sampling and Storage

Collect samples in clean glass bottles. Most reliable results are obtained when samples are analyzed as soon as possible after collection.

Color Development Time

Test results are strongly influenced by sample temperature. **Both reaction periods in the procedure are the same and depend on the temperature of the sample.** The reaction periods indicated in the procedure are for a sample temperature of 18–20 °C (68–73 °F). Adjust both reaction periods according to Table 3.

Table 3 Reaction Period

Sample Temperature		Reaction Periods (Minutes)
° C	° F	
5	40	10
7	42	9
9	48	8
10	50	8
12	54	7
14	58	7
16	61	6
18	68	5
20	73	5
23	75	2.5
25	77	2
>25	>77	2

Interferences

This method is intended for finished, chloraminated drinking water samples that have a measurable combined (total) chlorine disinfectant residual. Samples where the disinfectant residual has disappeared and samples which exhibit a chlorine demand may produce low ammonia test results. Blanks and ammonia standards analyzed without a disinfectant residual must be prepared using high quality, reagent grade water.

The following do not interfere in free ammonia determination when at or below the stated concentration.

Nitrogen, Free Ammonia and Chloramine (Mono), continued

Substance	Level Tested
Aluminum	0.2 mg/L Al
Chloride	1200 mg/L Cl
Copper	1 mg/L Cu
Iron	0.3 mg/L Fe
Manganese	0.05 mg/L Mn
Nitrate	10 mg/L NO ₃ -N
Nitrite	1 mg/L NO ₂ -N
Phosphate	2 mg/L -PO ₄
Silica	100 mg/L SiO ₂
Sulfate	1600 ppm as CaCO ₃
Zinc	5 ppm Zn

Samples containing high levels of both Total Hardness and Alkalinity may become turbid (cloudy) after the addition of the Free Ammonia Reagent Solution. If this occurs by the end of the first reaction period, the sample for Free Ammonia measurement must be pretreated as follows:

Note: The sample for Monochloramine measurement does not need pretreatment.

1. Measure 10 mL of sample into the cell for Free Ammonia measurement.
2. Add the contents of one Hardness Treatment Reagent Powder Pillow (Cat. No. 28823-46) to the sample.
3. Cap the cell and invert until the reagent is dissolved.
4. Remove the cap.

Continue with the analysis at step 2 using the pretreated sample as the Free Ammonia cell.

Accuracy Check (Monochloramine, Program 110)

1. Prepare the following monochloramine standard fresh before use.
2. Add the contents of one Buffer Powder Pillow, pH 8.3 to about 50-mL of organic-free water in a clean 100-mL Class A volumetric flask. Swirl to dissolve the powder.
3. Using a Class A volumetric pipet, transfer 2.00 mL of Nitrogen, Ammonia Standard Solution, 100 mg/L as NH₃-N into the flask.

Nitrogen, Free Ammonia and Chloramine (Mono), continued

4. Dilute to volume with organic-free water, cap and mix thoroughly. This is a 2.00 mg/L buffered ammonia standard.
5. Pipet 50.0 mL of the buffered ammonia standard into a clean 100-mL beaker. Add a stir bar.
6. Obtain a recent lot of Chlorine Solution Ampules, 50–70 mg/L, and note the actual free chlorine concentration for this lot.
7. Calculate the amount of Chlorine Solution to be added to the ammonia standard using the following equation:

$$\text{mL chlorine solution required} = \frac{455}{\text{free chlorine concentration}}$$

8. Open an ampule and, using a glass Mohr pipet, add the calculated amount of Chlorine Solution slowly to the ammonia standard, while mixing at medium speed on a stir plate.
9. Allow the monochloramine solution to mix for 1 minute after all Chlorine Solution is added.
10. Quantitatively transfer the monochloramine solution to a clean 100-mL Class A volumetric flask. Dilute to the mark with organic-free water, cap, and mix thoroughly. This is a nominal 4.5 mg/L (as Cl₂) monochloramine standard.

Use this standard within 1 hour of preparation.

Important Note: Because of the strong buffer used in the preparation of this standard, it cannot be used for accuracy verification of the Free Ammonia test.

Accuracy Check (Free Ammonia Test, Program 46)

Dilution water is required when testing a diluted sample and preparing standard solutions. Dilution water must be free of ammonia, chlorine and chlorine demand. A convenient source is a recirculating, deionizer system with carbon filtration which produces 18 megaohm-cm water.

Standard Additions Method

1. Measure 50 mL of sample into three 50-mL mixing cylinders.
2. Use the TenSette Pipet to add 0.3, 0.6, and 1.0 mL of Ammonium Nitrogen Standard, 10 mg/L as NH₃-N to the three samples. Mix well.

Nitrogen, Free Ammonia and Chloramine (Mono), continued

3. Analyze each spiked sample, following all steps of the Monochloramine and Free Ammonia procedure. The ammonia nitrogen concentration should increase 0.02 mg/L for each 0.1 mL of standard added.
4. If these increases do not occur, see *Standard Additions (Section 1 of the DR/890 Procedures Manual)* for more information.

Standard Solution Method

Prepare a 0.20 mg/L ammonia nitrogen standard by diluting 2.00 mL of the Ammonia Nitrogen Standard Solution, 10 mg/L, to 100 mL with dilution water. Or, using the TenSette Pipet, prepare a 0.20 mg/L ammonia nitrogen standard by diluting 0.4 mL of a Ammonia Nitrogen Voluette Standard Solution, 50 mg/L as $\text{NH}_3\text{-N}$, to 100 mL with dilution water. Analyze the standard solution, following all steps of the Monochloramine and Free Ammonia procedure.

Method Performance

Monochloramine Test

Precision

In a single laboratory, using a monochloramine standard solution of 2.10 mg/L Cl_2 and representative lots of reagent, a single operator obtained a standard deviation of ± 0.12 mg/L Cl_2 .

Estimated Detection Limit

The estimated detection limit for Method 10171 is 0.05 mg/L Cl_2 .

Free Ammonia Test

Precision

In a single laboratory using a solution containing 1.80 mg/L Cl_2 plus 0.20 mg/L ammonia nitrogen ($\text{NH}_3\text{-N}$) and two representative lots of reagent with the DR/890, a single operator obtained a standard deviation of ± 0.01 mg/L N for seven replicates.

Estimated Detection Limit

The estimated detection limit for program 46 is 0.02 mg/L N.

For more information on the estimated detection limit, see *Section 1 of the DR/850 or DR/890 Procedure Manual*.

Nitrogen, Free Ammonia and Chloramine (Mono), continued

Summary of Method

Monochloramine (NH_2Cl) and “free ammonia” (NH_3 and NH_4^+) can exist in the same water sample. Added hypochlorite combines with free ammonia to form more monochloramine. In the presence of a cyanoferrate catalyst, monochloramine in the sample reacts with a substituted phenol to form an intermediate monoimine compound. The intermediate couples with excess substituted phenol to form a green-colored indophenol, which is proportional to the amount of monochloramine present in the sample. Free ammonia is determined by comparing the color intensities, with and without added hypochlorite.

Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the Material Safety Data Sheet (MSDS) for information specific to the reagent used.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Free Ammonia Reagent Set (50 tests) Includes: (1) 28022-99, (1) 28773-36.....			28797-00
Free Ammonia Reagent Solution.....	1 drop	4 mL SCDB.....	28773-36
Monochlor F Reagent Pillows	2 pillows	100/pkg.....	28022-99

REQUIRED APPARATUS

Sample Cell, 1-cm/10-mL, with cap.....	2	2/pkg	48643-02
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OPTIONAL REAGENTS

Buffer, pH 8.3, Powder Pillows	25/pkg		898-68
Chlorine Solution, Voluette [®] Ampule	16/pkg		14268-10
Hardness Treatment Reagent Pillows (1 per test).....	50/pkg		28823-46
Nitrogen Ammonia Standard Solution, 10 mg/L as $\text{NH}_3\text{-N}$	500 mL		153-49
Nitrogen Ammonia Standard Ampule, 50 mg/L as $\text{NH}_3\text{-N}$, 10 mL.....	16/pkg		14791-10
Nitrogen Ammonia Standard Solution, 100 mg/L as $\text{NH}_3\text{-N}$	500 mL		24065-10

Nitrogen, Free Ammonia and Chloramine (Mono), continued

OPTIONAL APPARATUS

Description	Per Test	Unit	Cat. No.
Ampule Breaker Kit		each.....	21968-00
Beaker, 100 mL, Polypropylene.....		each.....	1080-42
Beaker, 100 mL, Glass		each.....	500-42H
Cylinder, 50 mL, mixing		each.....	20886-41
Flask, Volumetric, Class A, 100 mL		each.....	14574-42
Pipet Filler, Safety Bulb		each.....	14651-00
Pipet, TenSette [®] , 0.1 to 1.0 mL.....		each.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg.....		21856-96
Pipet, Mohr, Glass, 10 mL		each.....	20934-38
Pipet, Volumetric, Class A, 2.0 mL.....		each.....	14515-36
Pipet, Volumetric, Class A, 50.00 mL.....		each.....	14515-41
Scissors.....		each.....	28831-00
Stir Bar, Octagonal		each.....	20953-53
Stirrer, Magnetic.....		each.....	23436-00
Thermometer, -10 to 110 °C.....		each.....	1877-01
Wipers, Disposable Kimwipes [®] , 30 x 30 cm, 280/box.....		box.....	20970-01