



✓ Method 8009

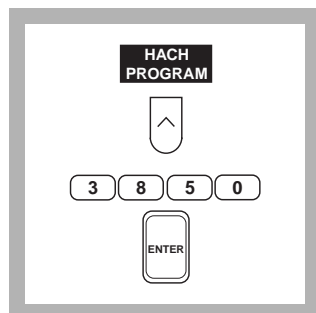
Zincon Method*

(0 to 3.000 mg/L)

Scope and Application: For water and wastewater. Digestion is required for determining total zinc; see the Digestion section following this procedure. USEPA Approved** for wastewater analyses. The estimated detection limit for program number 3850 is 0.009 mg/L Zn.

* Adapted from *Standard Methods for the Examination of Water and Wastewater*.

** Federal Register, 45 (105) 36166 (May 29, 1980)



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

Select the stored program number for zinc (Zn) by pressing **3850** with the numeric keys.

Press: **ENTER**

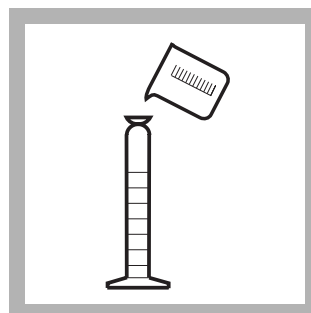
Note: If samples cannot be analyzed immediately, see *Sample Collection, Preservation and Storage* following these steps. Adjust pH of preserved samples before analysis.

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



2. The display will show: **HACH PROGRAM: 3850 Zinc**

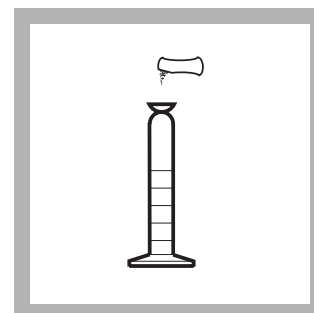
The wavelength (λ), **620 nm**, is automatically selected.



3. Fill a 25-mL graduated mixing cylinder with 20 mL of sample.

Note: Use only glass-stoppered cylinders in this procedure. Rinse with 1:1 hydrochloric acid and deionized water before use.

Note: For proof of accuracy, use a 0.5-mg/L zinc standard solution in place of the sample (see *Accuracy Check*).



4. Add the contents of one ZinconVer 5 Reagent Powder Pillow to the cylinder. Stopper. Invert several times to dissolve the powder completely.

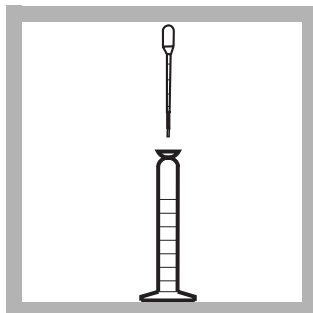
Caution! The reagent in this step contains cyanide and is very poisonous if taken internally or if the fumes are inhaled. Do not add to an acidic sample (<pH 4).

Note: Inconsistent readings may result for low zinc concentrations if all the particles are not dissolved.

Note: The sample should be orange. If the color is brown or blue, dilute the sample and repeat the test. Either the zinc concentration is too high or an interfering metal is present.

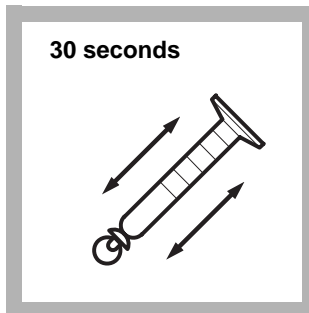


5. Pour 10 mL of the solution into a sample cell (the blank).



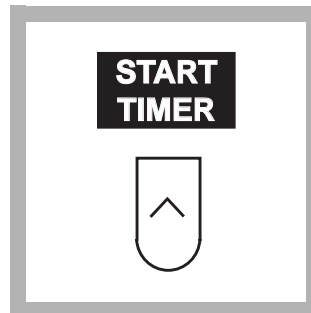
6. Add 0.5 mL of cyclohexanone to the remaining solution in the graduated mixing cylinder.

Note: Use a plastic dropper. Rubber bulbs may contaminate the cyclohexanone.



7. Press the soft key under **START TIMER**. During this time period, stopper the cylinder and shake vigorously for 30 seconds (this is the prepared sample).

Note: The sample will be reddish-orange, brown, or blue depending on the zinc concentration.



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

A 3-minute reaction period will begin.

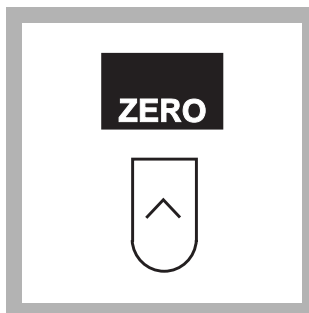
Note: During this time period, complete Step 9.



9. During the reaction period, pour the solution from the cylinder into a sample cell (the prepared sample).



10. When the timer beeps, place the blank in the cell holder. Close the light shield.

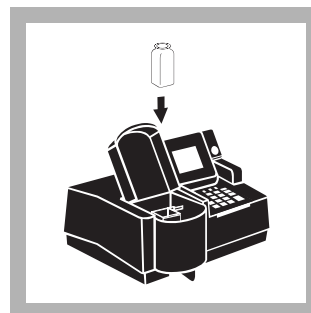


11. Press the soft key under **ZERO**.

The display will show:

0.000 mg/L Zn

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.



12. Place the prepared sample in the cell holder. Close the light shield. Results in mg/L zinc (or chosen units) will be displayed.

Interferences

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

Interfering Substance	Interference Levels and Treatments
Aluminum	Greater than 6 mg/L
Cadmium	Greater than 0.5 mg/L
Copper	Greater than 5 mg/L
Iron (ferric)	Greater than 7 mg/L
Manganese	Greater than 5 mg/L
Nickel	Greater than 5mg/L
Organic Material	Large amounts may interfere.
Highly buffered or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment. Adjust pH to 4–5.

Sample Collection, Preservation and Storage

Collect samples in acid-cleaned plastic or glass bottles. If prompt analysis is impossible, preserve the sample by adjusting to pH 2 or less with nitric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature.

Before analysis, adjust the pH to 4–5 with 5.0 N sodium hydroxide. Do not exceed pH 5 as zinc may precipitate. Correct the test result for volume additions; see Section 1.2.2 *Correcting 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), 50.
- c. Press **ENTER** to accept the default standard concentration (mg/L), 25.
- d. Press the soft key under **ENTRY DONE**.
- e. Snap the neck off a Zinc Voluette® Ampule Standard, 25-mg/L Zn.
- f. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively to three 50-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

Using Class A glassware, prepare a 0.50-mg/L zinc standard solution by pipetting 5.00 mL of Zinc Standard Solution, 100-mg/L, into a 1000-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily. Perform the Zincon procedure as described above.

To adjust the calibration curve using the reading obtained with the 0.50-mg/L Zinc 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.

Digestion

Digestion is required if total zinc is being determined. The following is not the USEPA digestion (see Section 2.1.1 *USEPA Mild Digestion with Hot Plate for Metals Analysis Only* for more information).

- a. If nitric acid has not been added to the sample previously, add 5 mL of concentrated nitric acid to one liter of sample (use a glass serological pipet and pipet filler). If the sample was acidified at collection, add 3 mL of nitric acid to one liter of sample.
- b. Transfer 100 mL of acidified sample to a 250-mL Erlenmeyer flask.
- c. Add 5 mL of 1:1 hydrochloric acid.
- d. Heat sample on a hot plate for 15 minutes at 95 °C. Make sure the sample does not boil.
- e. Filter cooled sample through a membrane filter and adjust the volume to 100 mL with deionized water.
- f. Adjust the pH to 4–5 with 5.0 N sodium hydroxide before analysis (see *Sample Collection, Preservation and Storage* for instructions).

Method Performance

Precision

Standard 0.500 mg/L Zn

Program	95% Confidence Limits
3850	0.495–0.505 mg/L Zn

For more information on determining precision data and method detection limits, refer to Section 1.5.

Estimated Detection Limit

Program	EDL
3850	0.009 mg/L Zn

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 40 CFR part 136, appendix B, see Section 1.5.1.

Sensitivity

Program Number: 3850

Portion of Curve	Δ Abs	Δ Concentration
Entire Range	0.010	0.0132 mg/L

See Section 1.5.3 *Sensitivity Explained* for more information.

Calibration Standard Preparation

To perform a zinc calibration using the Zincon method, prepare calibration standards containing 0.20, 0.50, 1.00, 1.50, 2.00, 2.50, and 3.00 mg/L zinc as follows:

- a. Prepare a 10.00-mg/L Zinc stock working standard. Pipet 10.00 mL of a 1000-mg/L Zinc Standard Solution into a 1-liter Class A volumetric flask using a 10.00-mL Class A volumetric pipet. Dilute this flask to volume with deionized water. Stopper and invert several times to mix.
- b. Pipet 2.00, 5.00, 10.00, 15.00, 20.00, 25.00, and 30.00 mL of the 10.00-mg/L Zinc Stock Working Standard into seven different 100-mL class A volumetric flasks, respectively.
- c. Dilute each flask to volume with deionized water. Stopper each flask and then invert several times to mix.
- d. Using the Zincon 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 standard prepared above.

Summary of Method

Zinc and other metals in the sample are complexed with cyanide. Adding cyclohexanone causes a selective release of zinc. The zinc then reacts with 2-carboxy-2'-hydroxy-5'-sulfoformazyl benzene (zincon) indicator to form a blue-colored species. The blue color is masked by the brown color from the excess indicator. The intensity of the blue color is proportional to the amount of zinc present.

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

ZincoVer 5 reagent contains potassium cyanide. Cyanide solutions are regulated as hazardous wastes by the Federal RCRA. Cyanide should be collected for disposal as a reactive (D003) waste. Be sure that cyanide solutions are stored in a caustic solution with pH >11 to prevent release of hydrogen cyanide gas. See *Section 1* for further information on proper disposal of these materials.

REQUIRED REAGENTS AND STANDARDS

	Cat. No.
Zinc Reagent Set, 20-mL sample size (100 tests = 100 samples + 100 blanks)	24293-00
Includes: (1) 14033-32, (1) 21066-69	

Description	Quantity Required		Cat. No.
	Per Test	Unit	
Cyclohexanone	1 mL	100 mL MDB	14033-32
ZincoVer 5 Reagent Powder Pillows	1 pillow	100/pkg	21066-69

REQUIRED EQUIPMENT AND SUPPLIES

Cylinder, graduated, mixing, 25-mL	1	each	20886-40
DR/4000 1-Inch Cell Adapter	1	each	48190-00

ZINC, continued

OPTIONAL REAGENTS AND STANDARDS

Bleach, household	1 gal.....	obtain locally
Hydrochloric Acid Solution, 1:1 (6.0 N).....	500 mL.....	884-49
Nitric Acid Solution, 1:1	500 mL.....	2540-49
Nitric Acid, ACS	500 mL.....	152-49
Sodium Hydroxide Standard Solution, 5.0 N.....	1 L.....	2450-53
Sodium Hydroxide Standard Solution, 5.0 N.....	59 mL DB.....	2450-26
Sodium Hydroxide Solution, 50%	500 mL.....	2180-49
Water, deionized.....	4 L.....	272-56
Zinc Standard Solution, 100-mg/L.....	100 mL.....	2378-42
Zinc Standard Solution, 1000-mg/L.....	100 mL.....	14177-42
Zinc Standard Solution, 2-mL PourRite® Ampule, 25-mg/L as Zn.....	20/pkg.....	14246-20
Zinc Standard Solution, 10-mL Voluette® Ampule, 25-mg/L Zn	16/pkg.....	14246-10

OPTIONAL EQUIPMENT AND SUPPLIES

Description	Unit	Cat. No.
Ampule Breaker Kit	each.....	21968-00
Aspirator, Nalgene vacuum pump	each.....	2131-00
Beaker, 1000-mL.....	each.....	500-53
Cylinder, graduated, 100-mL	each.....	508-42
Dropper 0.5- and 1.0-mL marks.....	10/pkg.....	21247-10
Filter Discs, glass, 47-mm.....	100/pkg.....	2530-00
Filter Holder, 47-mm.....	each.....	2340-00
Flask, Erlenmeyer, 250-mL.....	each.....	505-46
Flask, volumetric, Class A, 100-mL	each.....	14574-42
Flask, volumetric, Class A, 100-mL	6/pkg.....	14574-72
Flask, volumetric, Class A, 1000- mL	each.....	14574-53
Hot Plate, 3 ½" diameter, 120 VAC.....	each.....	12067-01
Hot Plate, 3 ½" diameter, 240 VAC.....	each.....	12067-02
pH Paper, pH 1.0 to 11.0	5 rolls/pkg.....	391-33
pH Meter, <i>sensio</i> TM 1, portable	each.....	50050-00
Pipet Filler, safety bulb.....	each.....	14651-00
Pipet, TenSette, 0.1- to 1.0-mL	each.....	19700-01
Pipet Tips, for 19700-01 Pipet	50/pkg.....	21856-96
Pipet, serological, 2-mL	each.....	532-36
Pipet, volumetric, Class A, 0.50-mL.....	each.....	14515-34
Pipet, volumetric, Class A, 2.00-mL.....	each.....	14515-36
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, 20.00-mL.....	each.....	14515-20
Pipet, volumetric, Class A, 25.00-mL.....	each.....	14515-40
PourRite Ampule Breaker	each.....	24846-00



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