Zinc

DOC316.53.01476

USEPA[1] Zincon Method[2] — Method 8009

0.02 to 3.00 mg/L Zn — Powder Pillows

Scope and application: For water and wastewater. Digestion is required for a total zinc analysis.

[1] USEPA approved for wastewater analyses 3500 Zn B: Federal Register, 45(105) 36166 (May 29, 1980).
[2] Adapted from Standard Methods for the Examination of Water and Wastewater.

Test preparation

Before starting

Clean all glassware with 6.0 N (1:1) hydrochloric acid, then fully rinse with deionized water to remove contaminants.
Use only glass-stoppered mixing cylinders in this procedure.
Make sure that the dropper that is used in this procedure is plastic. Droppers that have rubber bulbs can contaminate the reagent.
Do not use the Pour-Thru Cell or sipper module (for applicable instruments) with this test.
The reagents that are used in this test contain potassium cyanide. **Keep cyanide solutions at pH > 11 to prevent exposure to hydrogen cyanide gas.** Collect the reacted samples for safe disposal.
Always do tests in sample cells. Do not put the instrument in the sample or pour the sample into the cell holder.
Make sure that the sample cells are clean and there are no scratches where the light passes through them.
Rinse the sample cell and cap with the sample three times before the sample cell is filled.
Make sure that there are no fingerprints or liquid on the external surface of the sample cells. Wipe with a lint-free cloth before measurement.
Cold waters can cause condensation on the sample cell or bubbles in the sample cell during color development. Examine the sample cell for condensation or bubbles. Remove condensation with a lint-free cloth. Invert the sample cell to remove bubbles.
Install the instrument cap over the cell holder before ZERO or READ is pushed.
After the test, immediately empty and rinse the sample cell. Rinse the sample cell and cap three times with deionized water.
Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.
Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

Description	Quantity
Cyclohexanone	0.5 mL
ZincoVer® 5 Reagent Powder Pillow, 20-mL	1
Cylinder, graduated, 25-mL	1
Sample cells, 25-mm (10 mL)	2

Refer to Consumables and replacement items on page 5 for order information.

Sample collection and storage

- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.

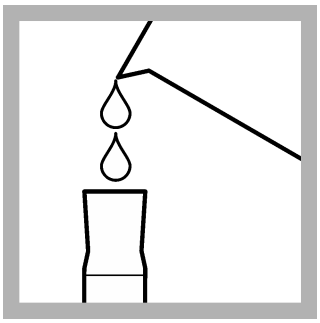
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated nitric acid (about 2 mL per liter). No acid addition is necessary if the sample is tested immediately.
- Keep the preserved samples at room temperature for a maximum of 6 months.
- Before analysis, adjust the pH to 4–5 with 5 N sodium hydroxide solution. Do not exceed pH 5 as zinc can precipitate. Do not add the reagent to samples with a pH less than 4 because there is cyanide in the reagent.
- Correct the test result for the dilution caused by the volume additions.

Powder pillow procedure

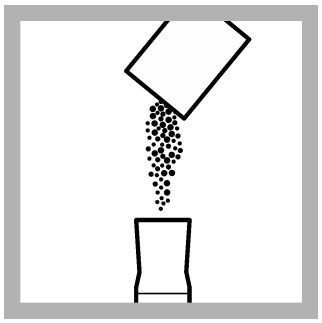


1. Set the instrument to zinc (Zn).

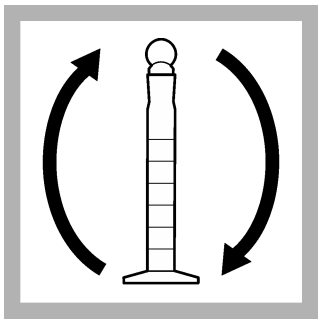
For DR300, push the up arrow button. For PCII, push the menu button, checkmark button, then the menu button again.



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

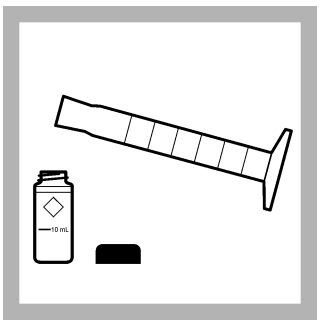


3. Add the contents of one ZincoVer 5 Reagent Powder Pillow to the mixing cylinder.



4. Close the cylinder. Invert the cylinder several times to dissolve the powder completely. Inconsistent readings may result if all the particles are not dissolved.

The sample should be orange. If the sample is brown or blue, the zinc concentration is too high or an interfering metal is present. Dilute the sample and do the test again.



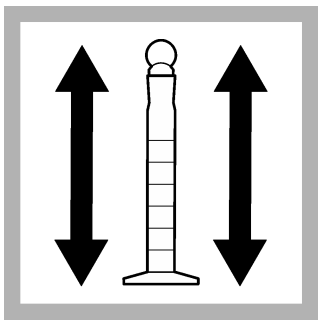
5. Prepare the blank: Fill a sample cell to the 10-mL mark with the solution.



6. Prepared the sample: Use a plastic dropper to add 0.5 mL of cyclohexanone to the solution in the mixing cylinder.



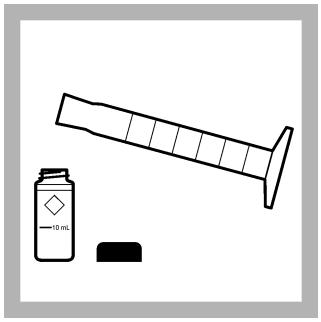
7. Set and start a timer for 30 seconds. A 30-second reaction time starts.



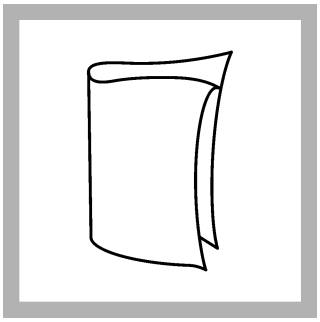
8. During the reaction period, close the mixing cylinder and vigorously shake the prepared sample. The sample becomes reddish-orange, brown or blue. The color is dependent on the zinc concentration.



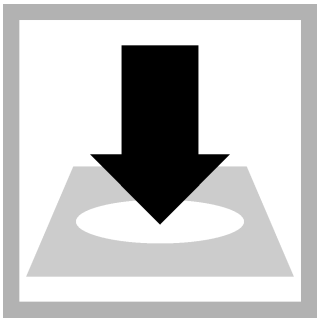
9. Set and start a timer for 3 minutes. A 3-minute reaction time starts. During the reaction period, complete the next step.



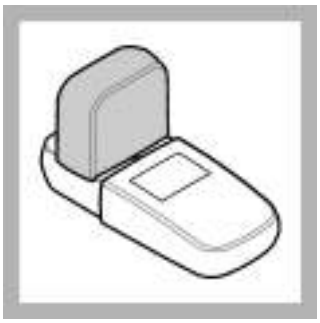
10. Fill a sample cell to the 10-mL mark with the prepared sample solution.



11. When the timer expires, clean the blank sample cell.



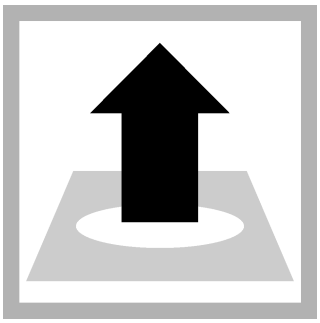
12. Insert the blank into the cell holder. Point the diamond mark on the sample cell toward the keypad.



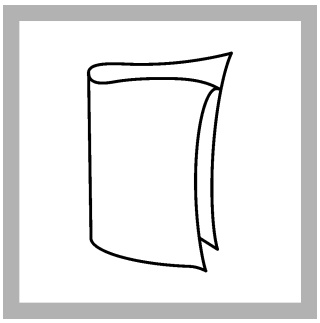
13. Install the instrument cap over the cell holder.



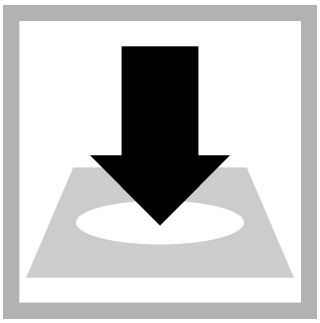
14. Push **ZERO**. The display shows “0.00”.



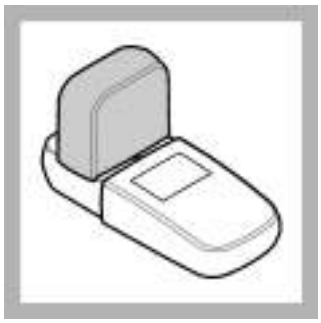
15. Remove the sample cell from the cell holder.



16. Clean the prepared sample cell.



17. Insert the prepared sample into the cell holder. Point the diamond mark on the sample cell toward the keypad.



18. Install the instrument cap over the cell holder.



19. Push **READ**. Results show in mg/L Zn.

Interferences

Interfering substance	Interference level
Aluminum	More than 6 mg/L
Cadmium	More than 0.5 mg/L
Copper	More than 5 mg/L
Iron (ferric)	More than 7 mg/L
Manganese	More than 5 mg/L
Nickel	More than 5 mg/L
Organic Material	Large amounts may interfere. Pretreat the sample with a digestion.

Interfering substance	Interference level
Highly buffered samples or extreme sample pH	Can prevent the correct pH adjustment (of the sample) by the reagents. Sample pretreatment may be necessary. Adjust the pH to 4–5.
Amino-tri(methylene phosphonic acid) (AMP)	Samples that contain AMP cause a negative interference. Digest the sample to remove this interference (use the total phosphorus hot plate digestion, Method 8190). ***Note:*** *Be sure to adjust the pH of the sample after the digestion to pH 4–5 with sodium hydroxide before the zinc analysis.*

Pollution prevention and waste management

Reacted samples contain potassium cyanide and must be disposed of as a hazardous waste. Dispose of reacted solutions according to local, state and federal regulations.

Digestion

For total zinc determinations, the sample must be digested with heat and acid to make sure that all forms of the metal are measured. The steps that follow can be used for a mild digestion.
Note: *The following procedure is the USEPA mild digestion procedure. Refer to the Water Analysis Guide for more digestion procedures.*

1. Add concentrated nitric acid to the sample with a glass serological pipet and pipet filler:
   - If the sample was acidified for preservation, add 3 mL of nitric acid to 1 liter of the preserved sample.
   - If the sample was not acidified for preservation, add 5 mL of nitric acid to 1 liter of sample.
2. Transfer 100 mL of acidified sample to a 250-mL Erlenmeyer flask.
3. Add 5 mL of 1:1 hydrochloric acid.
4. Put the sample on a hot plate at 95 °C (203 °F) until 15–20 mL of the sample remains. Make sure that the sample does not boil.
5. Put the cooled sample through a 0.45-µm filter to remove any insoluble material.
6. Adjust the pH of the digested sample to pH 4–5 with 5.0 N sodium hydroxide. Do not exceed pH 5 as zinc may precipitate.
7. Quantitatively transfer the sample to a 100-mL volumetric flask and dilute to the mark with deionized water.

Accuracy check

Standard additions method

Use the standard additions method to validate the test procedure, reagents and instrument and to find if there is an interference in the sample.

Items to collect:

- Zinc Standard Solution, 10-mg/L Voluette ® Ampule, 25-mL as Zn
- Ampule breaker
- Pipet, TenSette®, 0.1–1.0 mL and tips
- Mixing cylinders, 25-mL (3)

1. Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the standard solution, respectively, to three 25-mL portions of fresh sample. Mix well.
2. Use the test procedure to measure the concentration of each of the spiked samples. Start with the smallest sample spike. Measure each of the spiked samples in the instrument.
3. Compare the expected result to the actual result. The expected zinc concentration increase is 0.1 mg/L for each 0.1 mL of standard that is added.

Standard solution method

Use the standard solution method to validate the test procedure, the reagents and the instrument.

Items to collect:

- Zinc Standard Solution, 100-mg/L
- 100-mL volumetric flask, Class A
- 2-mL volumetric pipet, Class A and pipet filler
- Deionized water

1. Prepare a 2.00-mg/L zinc standard solution as follows:
   a. Use a pipet to add 2.0 mL of 100-mg/L zinc standard solution into the volumetric flask.
   b. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
2. Use the test procedure to measure the concentration of the prepared standard solution.
3. Compare the expected result to the actual result.

   Note: *The factory calibration can be adjusted slightly with the standard calibration adjust option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are small variations in the reagents or instruments.*

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a DR300 and a Pocket Colorimeter II during ideal test conditions. Users can get different results under different test conditions.

Precision (95% confidence interval)
2.00 ± 0.08 mg/L Zn

Summary of method

Zinc and other metals in the sample are complexed with cyanide. The addition of cyclohexanone causes a selective release of zinc. The zinc 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.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Zinc Reagent Set, 20-mL sample size, includes:	—	100 tests	2429300
Cyclohexanone	0.5 mL	100 mL MDB	1403332
ZincoVer® 5 Reagent Powder Pillow, 20-mL	1	100/pkg	2106669

Required apparatus

Description	Quantity/Test	Unit	Item no.
Mixing cylinder, graduated, 25 mL with stopper	1	each	2088640
Sample cells, 10-mL round, 25 mm x 60 mm	2	6/pkg	2427606

Recommended standards

Description	Unit	Item no.
Water, deionized	4 L	27256
Zinc Standard Solution, 100 mg/L	100 mL	237842
Zinc Standard Solution, 10-mg/L Voluette® Ampule, 25-mL as Zn	16/pkg	1424610
Zinc Standard Solution, 1000 mg/L	100 mL	1417742

Optional reagents and apparatus

Description	Unit	Item no.
Flask, Erlenmeyer, 250 mL	each	50546
Hot plate, 4-inch round, 120 V	each	1206701
Hydrochloric Acid Solution, 6.0 N (1:1)	500 mL	88449
Nitric Acid, concentrated	500 mL	15249
Pipet, TenSette®, 0.1–1.0 mL	each	1970001
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	50/pkg	2185696
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800
Pipet, volumetric, Class A, 2 mL	each	1451536
Pipet filler, safety bulb	each	1465100
Flask, volumetric, Class A, 100 mL, glass	each	1457442

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