Zinc

DOC316.53.01145

USEPA[1] Zincon Method[2]

Method 8009

0.01 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

Instrument specific information

The table in this section shows all of the instruments that have the program for this test. Table 1 shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests.

To use the table, select an instrument, then read across to find the corresponding information for this test.

Table 1 Instrument-specific information for powder pillows

Instrument	Sample cell orientation	Sample cell
DR 6000 DR 3800 DR 2800 DR 2700	The fill line is to the right.	2495402
DR 5000 DR 3900	The fill line is toward the user.	
DR 900	The orientation mark is toward the user.	2401906

Before starting

Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.
Clean all glassware with 6.0 N (50%) hydrochloric acid, then rinse thoroughly 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.
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 proper disposal.
Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used and use any recommended personal protective equipment.
Do not use the Pour-Thru Cell or sipper module (for applicable instruments) with this test.
Dispose of reacted solutions according to local, state and federal regulations. Use the Safety Data Sheets for disposal information for unused reagents. Consult the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

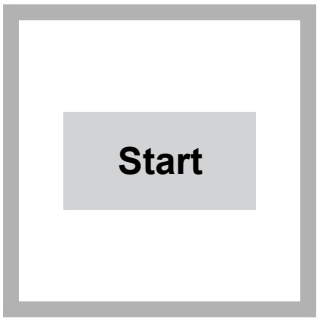
Description	Quantity
Cyclohexanone	0.5 mL
ZincoVer® 5 Reagent Powder Pillow, 20-mL	1
Cylinder, graduated mixing, 25-mL	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument specific information on page 1.)	2

Refer to Consumables and replacement items on page 6 for reorder information.

Sample collection and storage

- Collect samples in clean glass or plastic bottles that have been cleaned with 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 preserved samples at room temperature for a maximum of 6 months.
- Before analysis, adjust the pH to 4–5 with 5.0 N sodium hydroxide standard solution. Do not exceed pH 5 as zinc can precipitate.
- Correct the test result for the dilution from the volume additions.

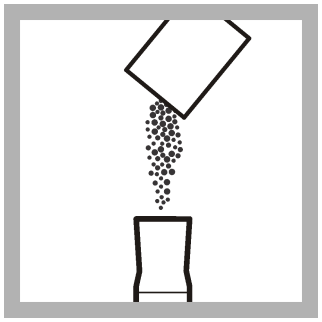
Powder pillow procedure



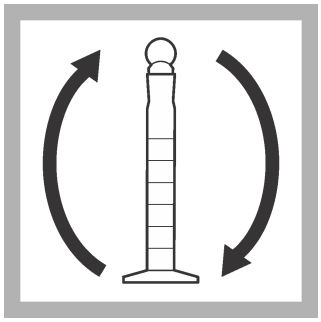
1. Start program **780 Zinc**. For information about sample cells, adapters or light shields, refer to Instrument specific information on page 1.

Note: *Although the program name may vary between instruments, the program number does not change.*

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

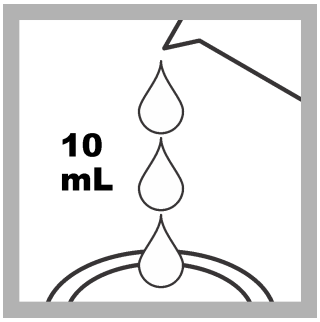


3. Add the contents of one ZincoVer 5 Reagent Powder Pillow to the mixing cylinder.



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.

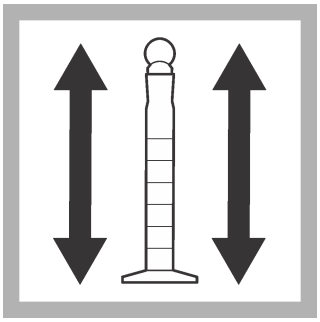
The sample should be orange. If the sample is brown or blue, the zinc concentration is too high or an interfering metal is present. Dilute the sample and repeat the test.



5. Blank preparation: Pour 10 mL of the solution into a sample cell.

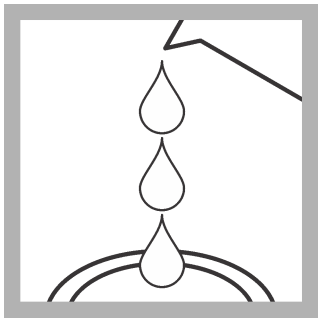
6. Prepared sample: Use a plastic dropper to add 0.5 mL of cyclohexanone to the solution that is still in the mixing cylinder.

7. Start the instrument timer. A 30-second reaction time starts.

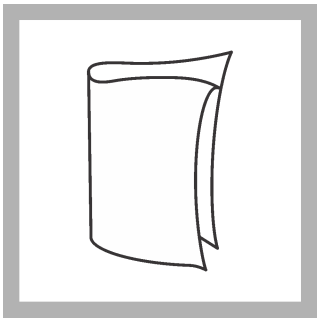


8. During the reaction period, close the mixing cylinder and vigorously shake the prepared sample. The sample shows reddish-orange, brown or blue, depending on the zinc concentration.

9. Start the instrument timer. A 3-minute reaction time starts.
During the reaction period, complete the next step.

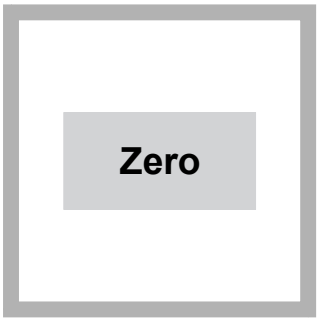


10. Pour the prepared sample solution from the mixing cylinder into a second sample cell.

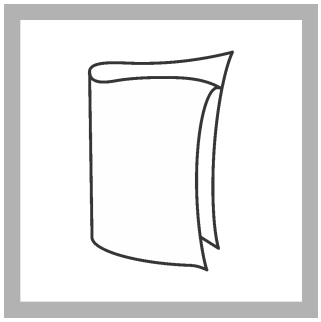


11. When the timer expires, clean the blank.

12. Insert the blank into the cell holder.



13. Push **ZERO**. The display shows 0.00 mg/L Zn.



14. Clean the prepared sample.

15. Insert the prepared sample into the cell holder.

16. 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

Interfering substance	Interference level
Nickel	More than 5 mg/L
Organic Material	Large amounts may interfere. Pretreat the sample with a mild digestion.
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.*

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 (sample spike)

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

Items to collect:

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

1. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
2. Go to the Standard Additions option in the instrument menu.
3. Select the values for standard concentration, sample volume and spike volumes.
4. Open the standard solution.
5. 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 20-mL portions of fresh sample. Mix well.

6. 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.
7. Select **Graph** to compare the expected results to the actual results.

   Note: *If the actual results are significantly different from the expected results, make sure that the sample volumes and sample spikes are measured accurately. The sample volumes and sample spikes that are used should agree with the selections in the standard additions menu. If the results are not within acceptable limits, the sample may contain an interference.*

Standard solution method

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

Items to collect:

- 100 mg/L zinc standard solution
- 1-L volumetric flask, Class A
- 10-mL volumetric pipet, Class A and pipet filler
- Deionized water

1. Prepare a 1.00 mg/L zinc standard solution as follows:
   a. Use a pipet to add 10.00 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 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 slight variations in the reagents or instruments.*

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a spectrophotometer during ideal test conditions. Users may get different results under different test conditions.

Program	Standard	Precision (95% Confidence Interval)	Sensitivity Concentration change per 0.010 Abs change
780	1.00 mg/L Zn	0.97–1.03 mg/L Zn	0.013 mg/L Zn

Summary of method

Zinc and other metals in the sample are complexed with cyanide. Adding 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. The measurement wavelength is 620 nm for spectrophotometers or 610 nm for colorimeters.

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.

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.
Cylinder, graduated mixing, 25 mL with stopper	1	each	2088640

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, 120 V	each	1206701
Hydrochloric Acid, 6.0 N 1:1, 50%	500 mL	88449
Nitric Acid, concentrated	500 mL	15249
Pipet, TenSette®, 0.1–1.0 mL	each	1970001
Pipet tips for TenSette Pipet 1970001	50/pkg	2185696
Ampule Breaker, Voluette® ampules	each	2196800
Pipet, volumetric, Class A, 10-mL	each	1451538
Pipet filler, safety bulb	each	1465100
Flask, volumetric, Class A, 1000-mL	each	1457453

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