

Chlorophosphonazo Colorimetric Method 8 to 1000 µg/L Ca and Mg as CaCO₃ (ULR)

Method 8374
Solution Pillow

Scope and application: For boiler and ultrapure water.




Test preparation

Instrument-specific information

Table 1 shows all of the instruments that have the program for this test. The table also shows the sample cell and adapter requirements.

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

Table 1 Instrument-specific information

Instrument	Adapter	Sample cell
DR 6000	LZV902.99.00020	2410212 
DR 5000	A23618	
DR 3900, DR 3800	—	
DR 2800	LZV585 (B)	
DR 1900	—	

Before starting

For the most accurate magnesium test results, keep the sample temperature between 21–29 °C (70–84 °F).

This test measures any calcium or magnesium contamination in the sample cells. To test cleanliness, run the test multiple times until the results are consistent. Use dedicated sample cells for this analysis.

If the test result is more than 750 µg/L, make a 1:1 dilution of the sample for best accuracy. Use ultra-pure (aldehyde-free) water for the dilution. Do the analysis again on the diluted sample and multiply the result by two.

Use "Alternate Forms" ("Chemical Forms" on some instruments) only when the sample is known to contain only Mg or Ca. This method does not distinguish between the two forms.

As an alternative to the solution pillow, use 1 mL of chlorophosphonazo solution. Use a bottle-top dispenser to add the reagent.

In bright light conditions (e.g., direct sunlight), close the cell compartment, if applicable, with the protective cover during measurements.

Total hardness in mg/L equals mg/L Ca as CaCO₃ plus mg/L Mg as CaCO₃.

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

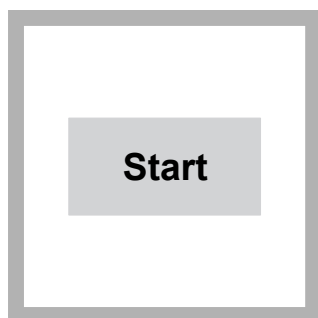
Description	Quantity
ULR Hardness Reagent Set	1
Clippers (shears) to open solution pillows	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	1

Refer to [Consumables and replacement items](#) on page 5 for order information.

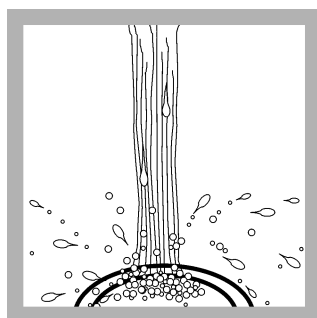
Sample collection

- Collect samples in clean plastic bottles with tight-fitting caps. Do not use glass bottles, which will contaminate the sample.
- Rinse the sample bottle several times with the sample to be collected.
- Seal the bottle to prevent contamination during the transport.
- Analyze the samples as soon as possible for best results.

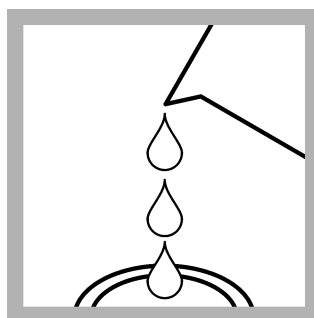
Test procedure



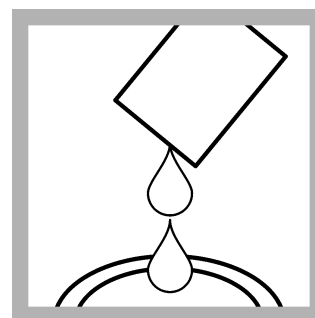
1. Start program 228 Hardness Tot ULR. For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.



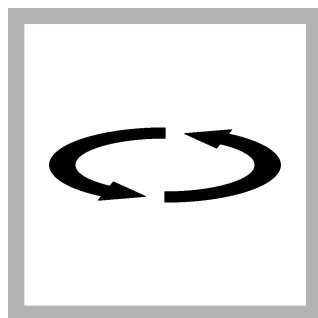
2. Clean a plastic sample cell and the cap three times with the test water. Make sure that the underside of the cap does not come in contact with surfaces that can contaminate it.



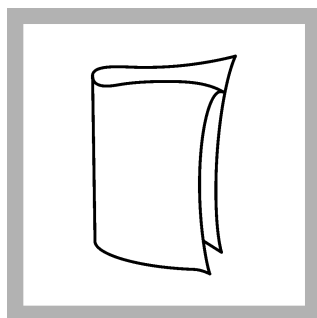
3. Fill the plastic sample cell to the 25-mL mark with sample.



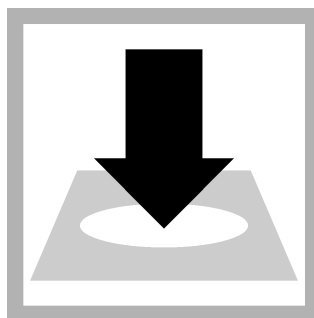
4. Add the contents of one Chlorophosphonazo Solution Pillow to the sample cell. A small quantity of solution that remains in the pillow does not affect results.



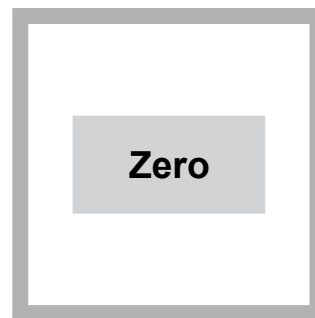
5. Close the sample cell. Swirl to mix.



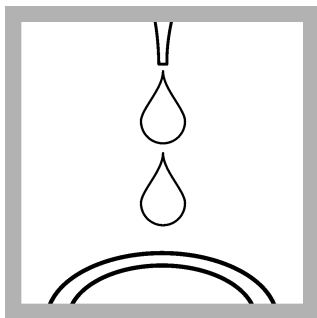
6. Clean the prepared sample cell.



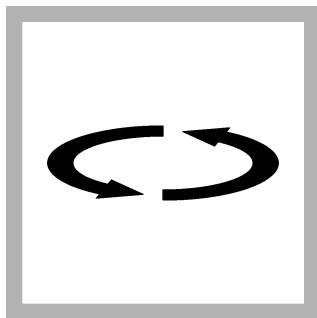
7. Insert the prepared sample into the cell holder.



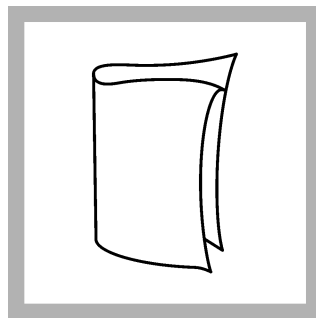
8. Push ZERO. The display shows 0 $\mu\text{g/L}$ CaCO_3 .



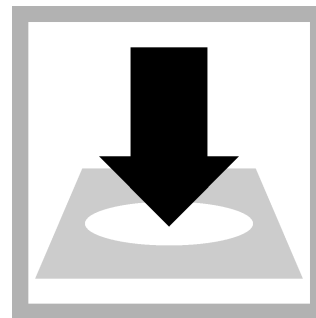
9. Remove the sample cell from the holder. Add 2 drops of CDTA Reagent for Ultra Low Range Hardness. Complete the remaining steps within 1–2 minutes.



10. Close the sample cell. Swirl to mix.



11. Clean the prepared sample cell.



12. Insert the prepared sample into the cell holder.



13. Push **READ**. Results show in $\mu\text{g/L CaCO}_3$.

Interferences

Table 2 shows the results of interference studies that used various hardness standard solutions between 0 and 500 $\mu\text{g/L}$ as CaCO_3 . Various cations and anions were added to the hardness standard solutions at levels in the range applicable for ultra-pure water applications. An ion was considered an interference when the measured hardness concentration changed by $\pm 10\%$.

Table 2 Interfering substances

Interfering substance	Interference level
Aluminum	Negative interference above 150 $\mu\text{g/L}$
Ammonium	No interference at or below 1000 $\mu\text{g/L}$
Copper	Positive interference above 250 $\mu\text{g/L}$
Formaldehyde	No interference at or below 47,000 $\mu\text{g/L}$
Nitrate	Positive interference above 250 $\mu\text{g/L}$
Potassium	No interference at or below 1000 $\mu\text{g/L}$
Silicon	Positive interference above 1000 $\mu\text{g/L}$
Sodium	Negative interference above 79,000 $\mu\text{g/L}$

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:

- 50,000- $\mu\text{g/L}$ (50-mg/L) as CaCO_3 Calcium Chloride Standard Solution
 - Flask, volumetric, 50-mL plastic
 - 20-mL volumetric pipet, Class A and pipet filler safety bulb
 - Pipet, TenSette[®], 0.1–1.0 mL and tips
 - Ultrapure water
1. Prepare a 20,000- $\mu\text{g/L}$ (20-mg/L) hardness standard solution as follows:
 - a. Use a pipet to add 20 mL of a 50 mg/L hardness standard solution into a 50-mL plastic volumetric flask.
 - b. Dilute to the mark with ultrapure water. Mix well. Prepare this solution daily.
 2. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
 3. Go to the Standard Additions option in the instrument menu.
 4. Select the values for standard concentration, sample volume and spike volumes.
 5. Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the prepared standard solution, respectively, to three 25-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, the reagents and the instrument.

Items to collect:

- 0.50-mg/L (500 $\mu\text{g/L}$) as CaCO_3 Calcium Chloride Standard Solution
1. Use the test procedure to measure the concentration of the standard solution.
 2. 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 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 spectrophotometer during ideal test conditions. Users can get different results under different test conditions.

Program	Standard	Precision (95% confidence interval)	Sensitivity Concentration change per 0.010 Abs change
228	500 $\mu\text{g/L}$ CaCO_3	478–522 $\mu\text{g/L}$ CaCO_3	8 $\mu\text{g/L}$ CaCO_3

Summary of Method

Calcium and magnesium react equivalently with the Chlorophosphonazo III indicator to form a complex with color that absorbs light very strongly at 669 nm. One drop of the CDTA reagent breaks up this complex. The decrease in color is proportional to the amount of calcium and magnesium in the sample (as CaCO_3). The measurement wavelength is 669 nm.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Hardness (ULR) Reagent Set, Solution Pillows (100 tests), includes:	—	each	2603100
Chlorophosphonazo Indicator Solution Pillows	1	100/pkg	2589599
CDTA Solution	2 drops	30 mL SCDB	2589723
Sample Cells, 1-inch square polystyrene, 25-mL with caps	1	2/pkg	2410222
OR			
Hardness (ULR) Reagent Set, Bulk Solution (500 tests), includes:	—	each	2603101
Chlorophosphonazo Indicator Solution	1 mL	500 mL	2589549
CDTA Solution	2 drops	30 mL SCDB	2589723
Sample Cells, 1-inch square polystyrene, 25-mL with caps	1	2/pkg	2410222

Required apparatus

Description	Quantity/test	Unit	Item no.
Clippers (shears), to open plastic pillows, stainless steel	1	each	2369400

Recommended standards

Description	Unit	Item no.
Calcium Chloride Standard Solution, 500 µg/L as CaCO ₃	946 mL	2058016
Calcium Chloride Standard Solution, 50 mg/L as CaCO ₃	946 mL	2127716

Optional reagents and apparatus

Description	Unit	Item no.
Flask, volumetric, 50 mL, polypropylene	each	1406041
Pipet, TenSette, 0.1–1.0 mL	each	1970001
Pipet tips for TenSette Pipet, 0.1–1.0 mL	50/pkg	2185696
Pipet, volumetric Class A, 20 mL	each	1451520
Pipet filler, safety bulb	each	1465100



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