DOC316.53.01168

Chelant, Free

Magnesium Chloride Method

0 to 20.0 mg/L as CaCO₃

Method 8352 Digital Titrator

Scope and application: For industrial waters.



Test preparation

Before starting

Before the test procedure, rinse labware with a 1:1 HCl solution to remove hardness on the plastic or glass. Rinse several times with deionized water.

For the best results, measure the reagent blank value for each new lot of reagent. Replace the sample with deionized water in the test procedure to determine the reagent blank value. Subtract the number of digits used for the reagent blank from the number of digits used for the sample titration.

As an alternative to the ManVer 2 Hardness Indicator Powder Pillow, use 4 drops of Hardness 2 Indicator Solution or a 0.1-g scoop of ManVer 2 Hardness Indicator Powder.

The optional TitraStir Titration Stand can hold the Digital Titrator and stir the sample.

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

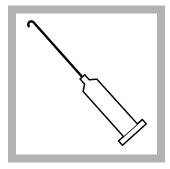
Description	Quantity
Hardness 1 Buffer Solution	2 mL
ManVer 2 Hardness Indicator Powder Pillow	1
Magnesium Chloride Titration Cartridge, 0.0800 M	1
Digital Titrator	1
Delivery tube for Digital Titrator	1
Graduated cylinder, 100-mL	1
Erlenmeyer flask, 125-mL	1

Refer to Consumable and replacement items on page 4 for order information.

Sample collection

- Collect samples in clean glass or plastic bottles.
- Filter samples that are turbid with filter paper and a funnel.

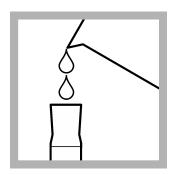
Test procedure



1. Insert a clean delivery tube into the digital titration cartridge. Attach the cartridge to the Digital Titrator.



2. Hold the Digital Titrator with the cartridge tip up. Turn the delivery knob to eject air and a few drops of titrant. Reset the counter to zero and clean the tip.



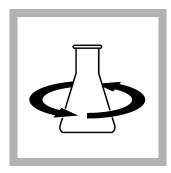
3. Use a graduated cylinder to measure 100 mL of sample.



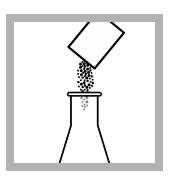
4. Pour the sample into a clean, 125-mL Erlenmeyer flask.



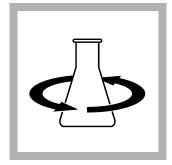
5. Add 2 mL of Hardness 1 Buffer Solution.



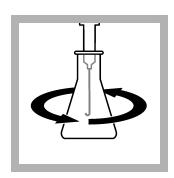
6. Swirl to mix.



 Add the contents of one ManVer 2 Hardness Indicator Powder Pillow.



8. Swirl to mix.
If a blue color forms, free chelant is in the sample. Go to the next step. If a red color forms, the sample has a chelant deficiency.



9. Put the end of the delivery tube fully into the solution. Swirl the flask. Turn the knob on the Digital Titrator to add titrant to the solution. Continue to swirl the flask. Add titrant until the color changes to a red-violet color. Record the number of digits on the counter.



10. Calculate the concentration. Digits used × 0.1 = mg/L free chelant as CaCO₃.

Example: A 100-mL sample was titrated with the 0.0800 M cartridge and the counter showed 120 digits at the endpoint. The concentration is $120 \times 0.1 = 12 \text{ mg/L}$ as $CaCO_3$.

Conversions

To change the units or chemical form of the test result, multiply the test result by the factor in Table 1.

Table 1 Conversions

mg/L chelant (CaCO ₃) to	multiply digits by	Example
mg/L tetrasodium EDTA	0.38	Digits × 0.38 = mg/L as Na ₄ EDTA

Interferences

Interfering substance	Interference level
Orthophosphate	Forms calcium phosphate and causes a slow endpoint. If sufficient time is given to let the calcium phosphate dissolve during the titration, the orthophosphate will not interfere with the test.
Polyphosphates	Interfere directly and are included in the test result.
pH	To analyze chelant residual in boiler water, adjust the pH of the sample before the Hardness 1 Buffer Solution is added in the test procedure as follows:
	 Add 2 drops of Phenolphthalein Indicator Solution to a 100-mL sample. Add 5.25 N Sulfuric Acid Standard Solution 1 drop at a time until the solution changes from pink to colorless. Record the number of drops added. Discard the sample. To a fresh 100-mL sample, add the same number of drops of 5.25 N Sulfuric Acid Standard Solution before the Hardness 1 Buffer Solution is added in the test procedure.

Accuracy check

Standard additions method (sample spike)

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

Items to collect:

- EDTA Standard Solution, 0.035 N
- Pipet, TenSette, 0.1–1.0 mL and pipet tips
- **1.** Use the test procedure to measure the concentration of the sample.
- 2. Use a TenSette pipet to add 0.4 mL of the standard solution to the titrated sample.
- 3. Titrate the spiked sample to the endpoint. Record the number of digits on the counter.
- **4.** Add one more 0.2-mL addition of the standard solution to the titrated sample.
- **5.** Titrate the spiked sample to the endpoint. Record the number of digits on the counter.
- **6.** Add one more 0.2-mL addition of the standard solution to the titrated sample.
- 7. Titrate the spiked sample to the endpoint. Record the number of digits on the counter.
- 8. Compare the actual result to the correct result. The correct result for this titration is 70 digits of the 0.0800 M Magnesium Chloride Titration Cartridge for each 0.4-mL addition of the standard solution. If much more or less titrant was used, there can be a problem with user technique, reagents, apparatus or an interference.

Summary of method

A hardness indicator is added to the sample. The sample is titrated with a standard solution of magnesium chloride at the endpoint pH of 10. The endpoint is determined by a color change from blue to red-violet.

Consumable and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
Hardness 1 Buffer Solution	2 mL	100 mL	42432
ManVer® 2 Hardness Indicator Powder Pillows	1 pillow	100/pkg	85199
Magnesium Chloride Titration Cartridge, 0.0800 M	varies	each	2062501

Required apparatus

Description	Quantity/test	Unit	Item no.
Cylinder, graduated, 100-mL	1	each	50842
Digital Titrator	1	each	1690001
Delivery tube for Digital Titrator, J-hook tip	1	5/pkg	1720500
Flask, Erlenmeyer, 125 mL	1	each	50543

Recommended standards

Description	Unit	Item no.
EDTA Standard Solution, 0.035 N	100 mL	2349932

Optional reagents and apparatus

Description	Unit	Item no.
ManVer 2 Hardness Indicator Powder	113 g	28014
ManVer Hardness Indicator Solution	100 mL	42532
Phenolphthalein Indicator Solution, 5-g/L	100 mL MDB	16232
Sulfuric Acid Standard Solution, 5.25 N	100 mL	244932
Filter paper, 2–3-micron, pleated, 12.5-cm	100/pkg	189457
Funnel, poly, 65-mm	each	108367
Bottle, sampling, with cap, low density polyethylene, 250-mL	12/pkg	2087076
Clippers	each	96800
Water, deionized	500 mL	27249
Pipet, TenSette [®] , 0.1–1.0 mL	each	1970001
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	50/pkg	2185696
Stir bar, octagonal	each	2095352
TitraStir [®] Titration Stand, 115 VAC	each	1940000
TitraStir® Titration Stand, 230 VAC	each	1940010
Delivery tube for Digital Titrator, 90-degree bend for use with TitraStir Titration Stand	5/pkg	4157800