

Phenolphthalein (Total) Acidity¹

0 to 10,000 mg/L as CaCO₃

Method 8010

Buret Titration

Scope and application: For water, wastewater and seawater.

¹ Adapted from *Standard Methods for the Examination of Water and Wastewater*, 23/0 B (4a).



Test preparation

Before starting

Prevent agitation of the sample during collection and mixing to prevent the loss of gases such as carbon dioxide, hydrogen sulfide and ammonia.

As an alternative to the Phenolphthalein Indicator Powder Pillow, use 4 drops of Phenolphthalein Indicator Solution.

Color or turbidity in the sample can make it difficult to see the color change at the endpoint. For these samples, use a pH meter to determine the titration endpoint. The end point for phenolphthalein acidity is pH 8.3.

The optional TitraStir Titration Stand can hold the buret 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

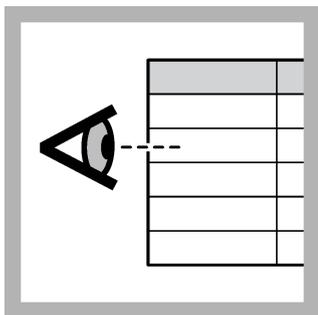
| Description | Quantity |
|---------------------------------------------------------------------------------------------------------------|----------|
| Phenolphthalein Indicator Powder Pillow | 1 |
| Sodium Hydroxide Standard Solution, 0.020 N | varies |
| pH meter and probe (for samples that have a lot of color or turbidity) | 1 |
| Buret, Class A, 50 mL | 1 |
| Graduated cylinder (use a size that is applicable to the selected sample volume), or TenSette pipet with tips | 1 |
| Erlenmeyer flask, 250 mL | 1 |
| Funnel, micro | 1 |
| Support stand with buret clamp | 1 |
| Water, deionized | varies |

Refer to [Consumables and replacement items](#) on page 4 for order information.

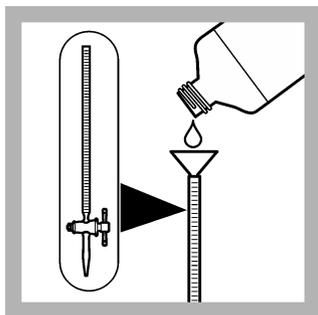
Sample collection

- Collect samples in clean glass or plastic bottles with tight-fitting caps. Completely fill the bottle and immediately tighten the cap.
- Prevent agitation of the sample and exposure to air.
- Analyze the samples as soon as possible for best results.
- If immediate analysis is not possible, keep the samples at or below 6 °C (43 °F) for a maximum of 24 hours.
- Let the sample temperature increase to room temperature before analysis.

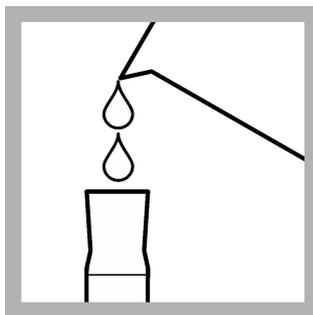
Test procedure



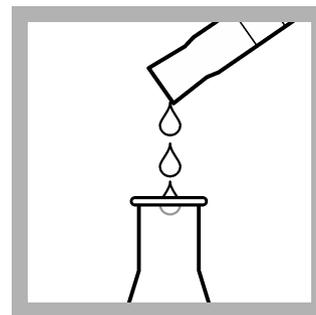
1. Select a sample volume and titrant from [Table 1](#) on page 3.



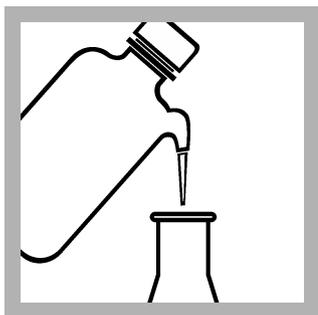
2. Fill a 50-mL buret to the zero mark with the titrant.



3. Use a graduated cylinder or pipet¹ to measure the sample volume from [Table 1](#) on page 3.



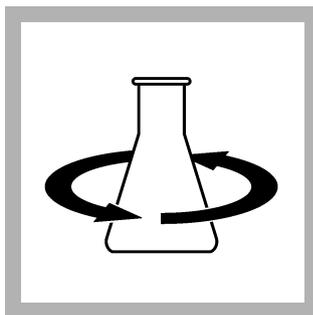
4. Pour the sample into a clean, 250-mL Erlenmeyer flask.



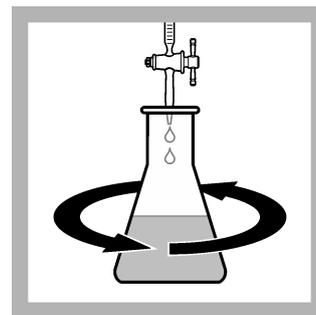
5. If the sample volume is less than 50 mL, dilute to approximately 50 mL with deionized water.



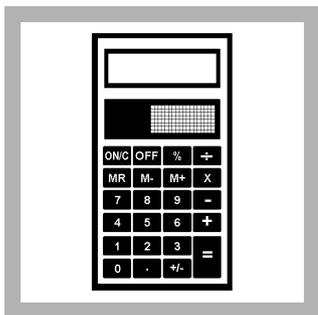
6. Add the contents of one Phenolphthalein Indicator Powder Pillow. The indicator is not necessary if a pH meter is used.



7. Swirl to mix.



8. Put the flask under the buret. Swirl the flask. Add titrant until the color changes to light pink and stays pink for 30 seconds (pH 8.3).



9. Use the multiplier in [Table 1](#) on page 3 to calculate the concentration.
 $\text{mL of titrant} \times \text{multiplier} =$
 mg/L as CaCO_3
phenolphthalein acidity.

Sample volumes and multipliers

Select a range in [Table 1](#), then read across the table row to find the applicable information for this test. Use the multiplier to calculate the concentration in the test procedure.

¹ Titration accuracy has a direct relation to the accuracy of the sample volume measurement. For smaller volumes, it is recommended to use a pipet to increase accuracy.

Example: A 50-mL sample was titrated with 0.020 N titrant and 12 mL of titrant was used at the endpoint. The concentration is $12 \text{ mL} \times 20 = 240 \text{ mg/L}$ as CaCO_3 phenolphthalein acidity.

Table 1 Sample volumes and multipliers

| Range (mg/L) | Sample volume (mL) | Titrant—sodium hydroxide | Multiplier |
|--------------|--------------------|--------------------------|------------|
| 1–1000 | 50 | 0.020 N | 20 |
| 800–2000 | 25 | 0.020 N | 40 |
| 2000–5000 | 10 | 0.020 N | 100 |
| 4000–10,000 | 5 | 0.020 N | 100 |

Conversions

To change the units or chemical form of the test result, multiply the test result by the factor in [Table 2](#).

Table 2 Conversions

| mg/L to... | multiply by... | Example |
|-------------------|----------------|--------------------------------------------------------------|
| Grains per gallon | 0.0584 | $25 \text{ mg/L} \times 0.0584 = 1.46 \text{ grains/gallon}$ |

Interferences

| Interfering substance | Interference level |
|-----------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Chlorine | Chlorine can react with the indicators and cause an incorrect result. Add 1 drop of 0.1 N Sodium Thiosulfate to the sample to remove chlorine before the test is started. |
| Color or turbidity | Color or turbidity can make it difficult to see the color change at the endpoint. Use a pH meter and titrate the samples to a pH of 8.3. |
| Hydrolyzable metals | <p>Pretreat samples that contain hydrolyzable metals (e.g. iron, manganese or aluminum) before the test is started as follows:</p> <ol style="list-style-type: none"> 1. Measure the sample volume into the flask. 2. Titrate the sample with 0.020 Sulfuric Acid Standard Solution until the sample pH is 4 or less. Record the mL of acid added. 3. Add 5 drops of 30% hydrogen peroxide solution. 4. Boil the solution for 2–5 minutes. 5. Let the solution temperature decrease to room temperature. 6. Use this solution as the sample in the test procedure. Titrate the solution until the pH is 8.3. Record the mL of 0.020 N Sodium Hydroxide added. 7. Calculate: (mL sodium hydroxide – mL of sulfuric acid) \times multiplier = mg/L as CaCO_3 phenolphthalein acidity |

Accuracy check

Validate the endpoint color

Prepare a buffer solution that has the correct pH and color at the endpoint to compare with the titrated sample.

1. Add 50 mL of deionized water to a flask.
2. Add one buffer powder pillow and one indicator powder pillow as follows:
 - Phenolphthalein (total) acidity—Add one pH 8.3 buffer powder pillow and one Phenolphthalein Indicator Powder Pillow.

3. Swirl to mix. The buffer solution will have the correct endpoint color.
4. Compare the color of the buffer solution with the color of the sample during the test procedure. Stop the titration when the titrated sample has the same color as the buffer solution.

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:

- Sulfuric Acid Standard Solution, 0.500 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 0.1 mL of the standard solution to the titrated sample.
 3. Titrate the spiked sample to the endpoint. Record the mL of titrant added.
 4. Add one more 0.1-mL addition of the standard solution to the titrated sample.
 5. Titrate the spiked sample to the endpoint. Record the mL of titrant added.
 6. Add one more 0.1-mL addition of the standard solution to the titrated sample.
 7. Titrate the spiked sample to the endpoint. Record the mL of titrant added.
 8. Compare the actual result to the correct result. The correct result for this titration is 2.5 mL of titrant for each 0.1-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.

Titrant test

Identify the strength of the Sodium Hydroxide Standard Solution each month. Sodium Hydroxide Standard Solution slowly absorbs carbon dioxide from the air, which decreases the strength of the solution.

1. Add 20 mL of Sulfuric Acid Standard Solution, 0.02 N to a 250-mL Erlenmeyer flask.
2. Add one Phenolphthalein Indicator Powder Pillow to the flask.
3. Titrate the solution to the endpoint. Record the mL of titrant added.
4. Compare the actual result to the correct result. The correct result for this titration is 20 mL of titrant. If more than 21 mL of titrant was added, discard the titrant. Get new titrant.

Summary of method

A phenolphthalein indicator is added to a fresh portion of sample. Then, the sample is titrated with a sodium hydroxide solution until the indicator changes color at the endpoint pH of 8.3. This titration includes both strong and weak acid species and is a measure of the total acidity. The results are expressed in mg/L as calcium carbonate (CaCO_3) at a specified pH.

Consumables and replacement items

Required reagents

| Description | Quantity/Test | Unit | Item no. |
|---------------------------------------------|---------------|---------|----------|
| Phenolphthalein Indicator Powder Pillows | 1 pillow | 100/pkg | 94299 |
| Sodium Hydroxide Standard Solution, 0.020 N | varies | 1 L | 19353 |
| Water, deionized | varies | 4 L | 27256 |

Required apparatus

| Description | Quantity/test | Unit | Item no. |
|---------------------------------------------------------------------------------------|---------------|--------|----------|
| Buret clamp, double | 1 | each | 32800 |
| Buret, Class A, 50 mL | 1 | each | 2636541 |
| Support stand | 1 | each | 56300 |
| Funnel, micro | 1 | each | 2584335 |
| Graduated cylinders—Select one or more for the sample volume: | | | |
| Cylinder, graduated, 5 mL | 1 | each | 50837 |
| Cylinder, graduated, 10 mL | 1 | each | 50838 |
| Cylinder, graduated, 25 mL | 1 | each | 50840 |
| Cylinder, graduated, 50 mL | 1 | each | 50841 |
| Cylinder, graduated, 100 mL | 1 | each | 50842 |
| Tensette [®] pipets and pipet tips—Select one or more for the sample volume: | | | |
| Pipet, TenSette [®] , 0.1–1.0 mL | 1 | each | 1970001 |
| Pipet tips, TenSette [®] Pipet, 0.1–1.0 mL | varies | 50/pkg | 2185696 |
| Pipet, TenSette [®] , 1.0–10.0 mL | 1 | each | 1970010 |
| Pipet tips, TenSette [®] Pipet, 1.0–10.0 mL | varies | 50/pkg | 2199796 |
| Flask, Erlenmeyer, 250 mL | 1 | each | 50546 |

Recommended standards

| Description | Unit | Item no. |
|------------------------------------------|------------|----------|
| Sulfuric Acid Standard Solution, 0.020 N | 100 mL | 20342 |
| Sulfuric Acid Standard Solution, 0.500 N | 100 mL MDB | 212132 |
| Buffer Powder Pillows, pH 8.3 | 25/pkg | 89868 |

Optional reagents and apparatus

| Description | Unit | Item no. |
|-------------------------------------------------|------------|----------|
| Clippers | each | 96800 |
| Hydrogen Peroxide Solution, 30%, ACS | 473 mL | 14411 |
| Phenolphthalein Indicator Solution, 5-g/L | 100 mL MDB | 16232 |
| Sodium Thiosulfate Standard Solution, 0.1 N | 100 mL | 32332 |
| Stir bar, octagonal | each | 2095352 |
| TitraStir [®] Titration Stand, 115 VAC | each | 1940000 |
| TitraStir [®] Titration Stand, 230 VAC | each | 1940010 |



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HACH COMPANY
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FAX: (970) 669-2932