Carbon Dioxide

Method 8205

Digital Titrator

Sodium Hydroxide Method

10 to 1000 mg/L CO₂

Scope and application: For water and seawater.

Test preparation

Before starting

To prevent agitation of the sample, pour the sample directly into the Erlenmeyer flask. Fill a graduated cylinder with the sample volume of deionized water. Pour the water into the Erlenmeyer flask. Make a mark on the Erlenmeyer flask at the water level to identify the sample volume level.

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 endpoint for phenolphthalein acidity is pH 8.3.

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

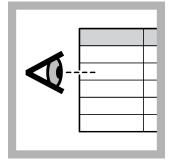
| Description | Quantity |
|--|----------|
| Phenolphthalein Indicator Powder Pillow | 1 |
| Sodium Hydroxide Titration Cartridge (refer to Sample volumes and digit multipliers on page 2) | 1 |
| pH meter and probe (for samples that have a lot of color or turbidity) | 1 |
| Clippers | 1 |
| Digital Titrator | 1 |
| Delivery tube for Digital Titrator | 1 |
| Erlenmeyer flask, 250-mL | 1 |

Refer to Consumables and replacement items on page 3 for order information.

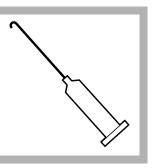
Sample collection

- 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 titration cartridge from Table 1 on page 2.



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



3. 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.



4. Pour the sample volume in Table 1 on page 2 in a 250-mL Erlenmeyer flask. When possible, collect the sample directly in the Erlenmeyer flask to prevent agitation of the sample.



5. Add the contents of one Phenolphthalein Indicator Powder Pillow.



6. Swirl to mix. If a pink color forms, no carbon dioxide is in the sample.



7. 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 from colorless to a light pink color for a minimum of 30 seconds (pH 8.3). Record the number of digits on the counter.



8. Use the multiplier in Table 1 on page 2 to calculate the concentration.
Digits used × digit multiplier = mg/L CO₂.

Sample volumes and digit multipliers

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

Example: A 100-mL sample was titrated with 0.3636 N Sodium Hydroxide Titration Cartridge and the counter showed 250 digits at the endpoint. The concentration is 250 digits \times 0.2 = 50 mg/L CO₂.

| Table 1 | Sample | volumes | and | digit | multipliers | |
|---------|--------|---------|-----|-------|-------------|--|
|---------|--------|---------|-----|-------|-------------|--|

| Range (mg/L as CO ₂) | Sample volume (mL) | Titration cartridge | Digit multiplier |
|----------------------------------|--------------------|---------------------|------------------|
| 10–50 | 200 | 0.3636 N | 0.1 |
| 20–100 | 100 | 0.3636 N | 0.2 |

| Table T Sample volumes and digit multipliers (continued) | | | | |
|--|--------------------|---------------------|------------------|--|
| Range (mg/L as CO ₂) | Sample volume (mL) | Titration cartridge | Digit multiplier | |
| 100–400 | 200 | 3.636 N | 1.0 | |
| 200–1000 | 100 | 3.636 N | 2.0 | |

Table 1 Sample volumes and digit multipliers (continued)

Interferences

| Interfering substance | Interference level |
|-----------------------|--|
| Other acids | Interferes directly and is included in the test result. |
| Color and turbidity | 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 endpoint is pH 8.3. |

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:

- Carbon Dioxide Voluette Ampule Standard, 10,000 mg/L CO₂
- Ampule Breaker
- 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 number of digits on the counter.
- 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 number of digits on the counter.
- 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 number of digits on the counter.
- 8. Compare the actual result to the correct result. The correct result for this titration is 50 digits of the 0.3636 N Sodium Hydroxide Titration Cartridge or 5 digits of the 3.636 N Sodium Hydroxide Titration Cartridge 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.

Summary of method

A phenolphthalein indicator is added to the sample. Carbonic acid formed by carbon dioxide in the sample is titrated with a sodium hydroxide standard solution until the indicator changes color at the endpoint pH of 8.3. Strong acids are not thought to be in the sample or at concentrations levels that will have an effect on the results.

Consumables and replacement items

Required reagents

| Description | Quantity/Test | Unit | Item no. |
|--|---------------|---------|----------|
| Carbon Dioxide Reagent Set (approximately 100 tests) | _ | each | 2272700 |
| Phenolphthalein Indicator Powder Pillows | 1 | 100/pkg | 94299 |
| Sodium Hydroxide Titration Cartridge, 0.3636 N | varies | each | 1437801 |
| Sodium Hydroxide Titration Cartridge, 3.636 N | varies | each | 1438001 |

Required apparatus

| Description | Quantity/test | Unit | ltem no. |
|--|---------------|-------|----------|
| Digital Titrator | 1 | each | 1690001 |
| Delivery tube for Digital Titrator, J-hook tip | 1 | 5/pkg | 1720500 |
| Flask, Erlenmeyer, 125 mL | 1 | each | 50543 |
| Flask, Erlenmeyer, 250 mL | 1 | each | 50546 |

Recommended standards

| Description | Unit | ltem no. |
|--|--------|----------|
| Carbon Dioxide Standard Solution, Voluette [®] Ampule, 10,000 mg/L as CO ₂ , 10 mL | 16/pkg | 1427510 |

Optional reagents and apparatus

| Description | Unit | ltem no. |
|---|------------|----------|
| Phenolphthalein Indicator Solution, 5-g/L | 100 mL MDB | 16232 |
| Ampule Breaker, 10-mL Voluette [®] Ampules | each | 2196800 |
| Buffer Powder Pillows, pH 8.3 | 25/pkg | 89868 |
| 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 |

