

## Ceric Acid Titration Method

Method 8351

100–2500 mg/L as NaNO<sub>2</sub>

Digital Titrator

**Scope and application:** For cooling tower waters.



### Test preparation

### Before starting

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

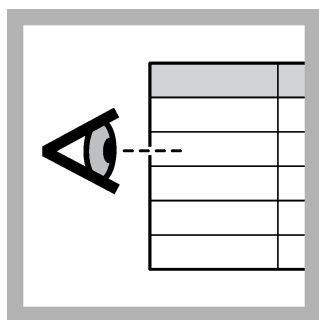
| Description                                                                      | Quantity |
|----------------------------------------------------------------------------------|----------|
| Ferriin Indicator Solution                                                       | 1        |
| Sulfuric Acid Standard Solution, 5.25 N                                          | 1        |
| Ceric Standard Solution Titration Cartridge, 0.5 N                               | 1        |
| Digital Titrator                                                                 | 1        |
| Delivery tube for Digital Titrator                                               | 1        |
| Graduated cylinder (use a size that is applicable to the selected sample volume) | 1        |
| Erlenmeyer flask, 125 mL                                                         | 1        |
| Water, deionized                                                                 | varies   |

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

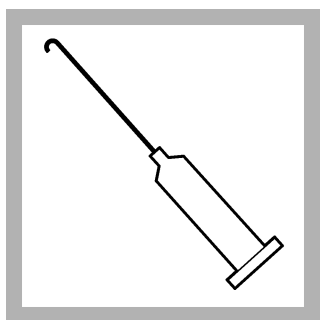
### Sample collection and storage

- Collect samples in clean glass or plastic bottles.
- 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.

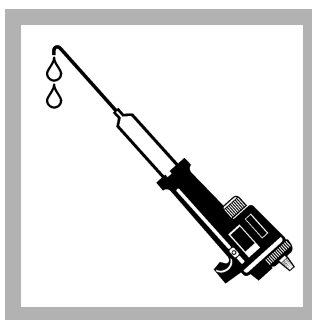
## Test procedure



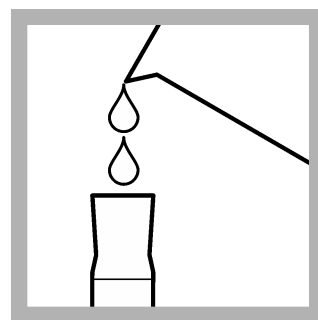
1. Select a sample volume from [Table 1](#) on page 3.



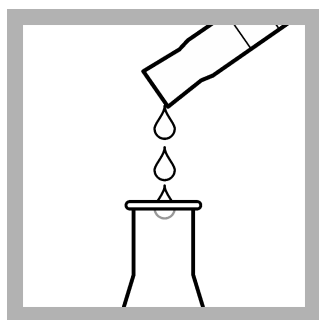
2. Insert a clean delivery tube into the digital titration cartridge. Attach the cartridge to the Digital Titrator.



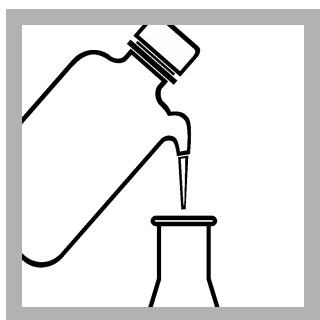
3. Hold the Digital Titrator with the cartridge tip up. Turn the delivery knob to eject air and a few drops of titrant. Reset the counter to zero and clean the tip.



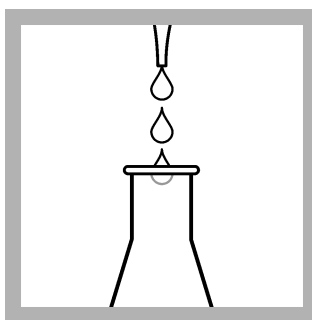
4. Use a graduated cylinder or a pipet<sup>1</sup> to measure the sample volume from [Table 1](#) on page 3.



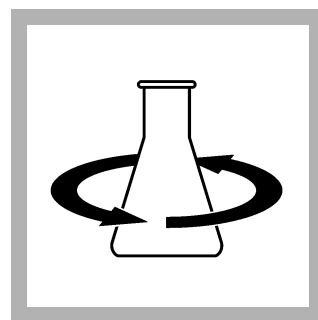
5. Pour the sample into a clean, 125-mL Erlenmeyer flask.



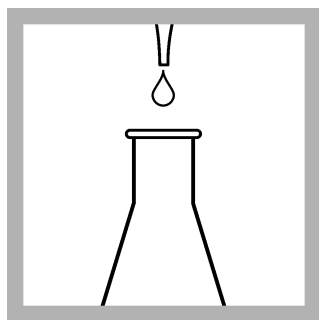
6. Dilute to approximately 75 mL with deionized water.



7. Add 5 drops of 5.25 N Sulfuric Acid Standard Solution.



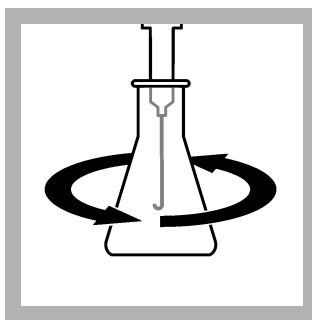
8. Swirl to mix.



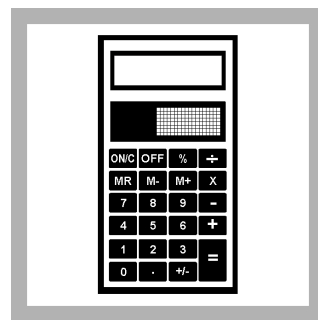
9. Add one drop of Ferroin Indicator Solution.



10. Swirl to mix.



11. 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 orange to pale blue. Record the number of digits on the counter.



12. Use the multiplier in [Table 1](#) on page 3 to calculate the concentration. Digits used  $\times$  digit multiplier = mg/L sodium nitrite as  $\text{NaNO}_2$ .

<sup>1</sup> A pipet is recommended for sample volumes less than 10 mL.

## 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 25-mL sample was titrated with 0.5 N Ceric Standard Solution Titration Cartridge and the counter showed 250 digits at the endpoint. The concentration is  $250 \text{ digits} \times 0.86 = 215 \text{ mg/L}$  sodium nitrite as  $\text{NaNO}_2$ .

**Table 1 Sample volumes and digit multipliers**

| Range (mg/L as $\text{NaNO}_2$ ) | Sample volume (mL) | Digit multiplier |
|----------------------------------|--------------------|------------------|
| 100–400                          | 25                 | 0.86             |
| 400–800                          | 10                 | 2.15             |
| 800–1500                         | 5                  | 4.31             |
| 1500–2500                        | 2                  | 10.78            |

## Accuracy check

### Standard solution method

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

Items to collect:

- Sodium Nitrite, ACS
- 1000-mL volumetric flask, Class A
- 5-mL volumetric pipet, Class A and pipet filler safety bulb
- Deionized water

1. Prepare a 1000-mg/L sodium nitrite standard solution as follows:
  - a. Add 1.000 gram of sodium nitrite to the volumetric flask.
  - b. Dilute to the mark with deionized water. Mix well.
2. Use the test procedure to measure the concentration of the prepared standard solution. Use 5.0 mL of the prepared standard solution.
3. Compare the actual result to the correct result. The correct result for this titration is approximately 1000 mg/L as  $\text{NaNO}_2$ .

### Standardization of the ceric standard solution

The normality of the ceric standard solution can decrease over time. Before use, examine the normality with the standardization procedure. It is recommended to do the standardization procedure monthly.

Items to collect:

- Sodium Thiosulfate Titration Cartridge, 0.200 N
  - 125-mL Erlenmeyer flask
  - Deionized water
1. Use a graduated cylinder to measure 50 mL of deionized water into a 125-mL Erlenmeyer flask.
  2. Use a pipet to add 5 mL of 19.2 N Sulfuric Acid Standard Solution. Swirl to mix.
  3. Insert a clean delivery tube into the Ceric Standard Titration Cartridge. Attach the cartridge to the titrator.
  4. Hold the Digital Titrator with the cartridge tip pointing up. Turn the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.
  5. Put the delivery tube into the solution and swirl the flask. Turn the knob on the titrator to add 200 digits of titrant to the solution.

6. Insert a clean delivery tube into a 0.200 N Sodium Thiosulfate Titration Cartridge. Attach the cartridge to the titrator.
7. Hold the Digital Titrator with the cartridge tip pointed up. Turn the delivery knob to eject a few drops of titrant. Reset the counter to zero and clean the tip.
8. Titrate the solution until the color changes from intense yellow to faint yellow. Record the number of digits on the counter. The correct number of digits for this step is 400–450 digits.
9. Add 1 drop of Ferroin Indicator Solution to the flask. Swirl to mix. The color of the solution changes to faint blue.
10. Continue the titration with Sodium Thiosulfate until the color changes to orange. Record the number of digits on the counter.
11. Divide the number of digits by 500 to calculate the correction factor.
12. Multiply the mg/L sodium nitrite from the titration procedure by the correction factor to get the correct sodium nitrite concentration.

## Summary of method

Ferroin indicator and acid is added to the sample. The sample is titrated with tetravalent cerium ion, which is a strong oxidant. After the cerium oxidizes the nitrite, the indicator is oxidized and causes a color change from orange to pale blue. The quantity of titrant used changes in relation to the concentration of sodium nitrite in the sample.

## Consumables and replacement items

### Required reagents

| Description                                        | Quantity/Test | Unit       | Item no. |
|----------------------------------------------------|---------------|------------|----------|
| Ceric Standard Solution Titration Cartridge, 0.5 N | 1             | each       | 2270701  |
| Ferroin Indicator Solution                         | 1             | 29 mL DB   | 181233   |
| Sulfuric Acid Standard Solution, 5.25 N            | 1             | 100 mL MDB | 244932   |

### 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    |
| Pipet filler, safety bulb                      | 1             | each  | 1465100  |
| Pipet, volumetric, Class A, 2.00 mL            | 1             | each  | 1451536  |
| Pipet, volumetric, Class A, 5.00 mL            | 1             | each  | 1451537  |
| Pipet, volumetric, Class A, 10.0 mL            | 1             | each  | 1451538  |
| Pipet, volumetric, Class A, 20.0 mL            | 1             | each  | 1451520  |

### Recommended standards

| Description                                     | Unit   | Item no. |
|-------------------------------------------------|--------|----------|
| Sodium Nitrite, ACS                             | 454 g  | 245201   |
| Sodium Thiosulfate Titration Cartridge, 0.200 N | each   | 2267501  |
| Sulfuric Acid Standard Solution, 19.2 N         | 100 mL | 203832   |

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**Optional apparatus**

| Description                                                                               | Unit   | Item no. |
|-------------------------------------------------------------------------------------------|--------|----------|
| Ampule Breaker, 10-mL Voluette® Ampules                                                   | each   | 2196800  |
| Pipet, TenSette®, 1.0–10.0 mL                                                             | each   | 1970010  |
| Pipet tips for TenSette® Pipet, 1.0–10.0 mL                                               | 50/pkg | 2199796  |
| 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  |



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