

**Iodate-Iodide Method****Method 8216****4 to more than 400 mg/L as SO<sub>3</sub><sup>2-</sup>****Digital Titrator**

Scope and application: For boiler water.

**Test preparation****Before starting**

Samples must be analyzed immediately after collection and cannot be preserved for later analysis.

Sulfite reacts quickly with oxygen in the air. Shaking or swirling the sample causes low results. Prevent agitation of the sample during the procedure.

As an alternative to the Dissolved Oxygen 3 Reagent Powder Pillow, use 0.5 mL of 19.2 N Sulfuric Acid Standard Solution.

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**

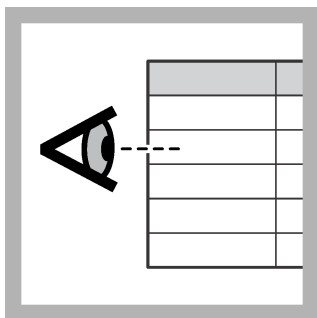
| Description  | Quantity       |
|--|----------------|
| Dissolved Oxygen 3 Reagent Powder Pillows                    | 1              |
| Iodate-Iodide Titration Cartridge, 0.3998 N                  | 1              |
| Starch Indicator Solution                                    | 1 full dropper |
| Clippers   | 1              |
| Digital Titrator   | 1              |
| Delivery tube for Digital Titrator                           | 1              |
| Graduated cylinder (size varies with selected sample volume) | 1              |
| Erlenmeyer flask, 125-mL                                     | 1              |
| Water, deionized   | varies         |

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

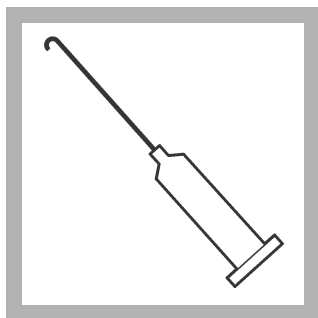
**Sample collection**

- Analyze the samples immediately. The samples cannot be preserved for later analysis.
- Collect samples in clean glass or plastic bottles with tight-fitting caps. Completely fill the bottle and immediately tighten the cap.
- If the sample temperature is more than 50 °C (122 °F), decrease the sample temperature to 50 °C (122 °F) or lower before analysis.
- Prevent agitation of the sample and exposure to air.

## Test procedure



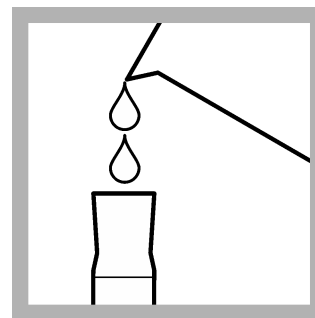
1. Select a sample volume and titration cartridge from [Table 1](#) on page 3.



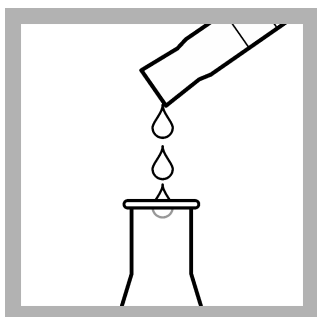
2. Insert a clean delivery tube into the Iodate-Iodide Titration Cartridge. Attach the cartridge to the Digital Titrator.



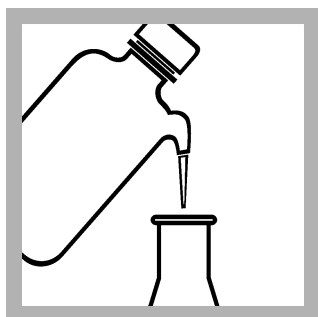
3. Hold the Digital Titrator vertically with the cartridge tip up. Turn the delivery knob to eject air and a few drops of titrant. Reset the counter to zero and clean the tip.



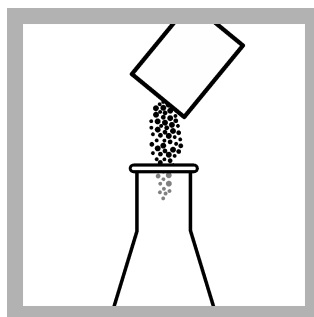
4. Use a graduated cylinder to measure the sample volume from [Table 1](#) on page 3.



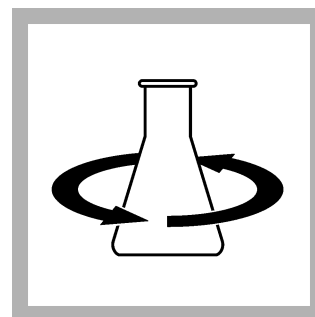
5. Pour the sample into a clean, 125-mL Erlenmeyer flask.



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



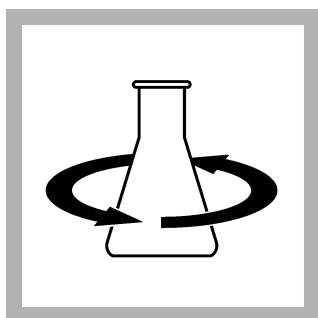
7. Add the contents of one Dissolved Oxygen 3 Reagent Powder Pillow.



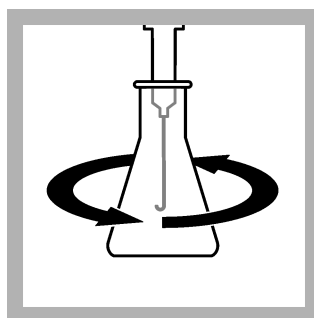
8. Swirl to mix.



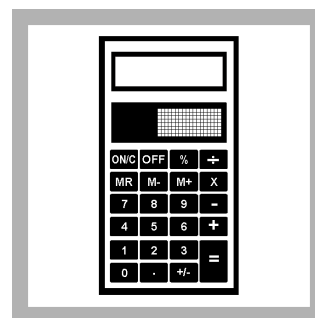
9. Add 1 full dropper of Starch Indicator Solution.



10. Swirl to mix.



11. Put the delivery tube point fully into the solution and swirl the flask. Turn the knob on the Digital Titrator to add titrant to the solution. Continue to swirl the flask and add titrant until the color changes to a permanent blue color. 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 sulfite  $\text{SO}_3^{2-}$

## Sample volumes and digit multipliers

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

**Example:** A 50-mL sample was titrated and the counter showed 250 digits at the endpoint. The concentration is: 250 digits x 0.4 = 100 mg/L sulfite  $\text{SO}_3^{2-}$ .

**Table 1 Sample volumes and digit multipliers**

| Range (mg/L as $\text{SO}_3^{2-}$ ) | Sample volume (mL) | Titration cartridge | Digit multiplier |
|-------------------------------------|--------------------|---------------------|------------------|
| a maximum of 160                    | 50                 | 0.3998 N            | 0.4              |
| 100–400                             | 20                 | 0.3998 N            | 1                |
| 200–800                             | 10                 | 0.3998 N            | 2                |
| more than 400                       | 5                  | 0.3998 N            | 4                |

## 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 sulfite ( $\text{SO}_3^{2-}$ ) to...   | multiply by: | Example   |
|---|--------------|---|
| mg/L bisulfite, hydrogen sulfite ( $\text{HSO}_3^-$ )                               | 1.01         | 200 mg/L $\text{SO}_3^{2-}$ x 1.01 = 202 mg/L $\text{HSO}_3^-$                  |
| mg/L sodium bisulfite, sodium hydrogen sulfite ( $\text{NaHSO}_3$ )                 | 1.30         | 200 mg/L $\text{SO}_3^{2-}$ x 1.30 = 260 mg/L $\text{NaHSO}_3$                  |
| mg/L sodium metabisulfite, sodium pyrosulfite ( $\text{Na}_2\text{S}_2\text{O}_5$ ) | 2.37         | 200 mg/L $\text{SO}_3^{2-}$ x 2.37 = 474 mg/L $\text{Na}_2\text{S}_2\text{O}_5$ |
| mg/L sodium sulfite ( $\text{Na}_2\text{SO}_3$ )                                    | 1.58         | 200 mg/L $\text{SO}_3^{2-}$ x 1.58 = 316 mg/L $\text{Na}_2\text{SO}_3$          |

## Interferences

[Table 3](#) shows the substances that can interfere with this test.

**Table 3 Interferences**

| Interfering substance | Interference level  |
|-----------------------|---|
| Metals                | Some metals, especially copper, catalyze the oxidation of sulfite to sulfate. Immediately add one Dissolved Oxygen 3 Powder Pillow for each liter of sample during sample collection to prevent the interference. |
| Nitrite               | Reacts with sulfite and causes low results.   |
| Organic compounds     | Oxidizable organic compounds can cause high results.  |
| Oxidizable compounds  | Cause high results.   |
| Sulfide               | Causes high results.  |

## 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:

- Sulfite Voluette Ampule Standard, 5,000-mg/L  $\text{SO}_3^{2-}$
- 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 25 digits of the Iodate-Iodide 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.

### Standard solution method

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

Items to collect:

- Sodium Thiosulfate Standard Solution, 0.025 N
  - 250-mL volumetric flask, Class A
  - 10.0-mL volumetric pipet, Class A and pipet filler safety bulb
  - Deionized water
1. Prepare a 40-mg/L sulfite-equivalent standard solution as follows:
    - a. Use a pipet to add 10.0 mL of a 0.025 N sodium thiosulfate standard solution to the volumetric flask.
    - b. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
  2. Use the test procedure to measure the concentration of the prepared standard solution. Use 50-mL of the prepared standard solution.
  3. Compare the actual result to the correct result. If much more or less titrant was used, there can be a problem with user technique, reagents or apparatus.

### Summary of Method

The water sample is acidified and titrated with a potassium iodide-iodate standard solution. The acid releases free iodine, which is reduced to colorless iodide by the sulfite in the sample. When all of the sulfite is gone, the iodine reacts with the starch indicator to form a blue color.

### Consumables and replacement items

#### Required reagents

| Description                                    | Quantity/Test | Unit       | Item no. |
|--|---------------|------------|----------|
| Sulfite Reagent Set (approximately 100 tests): | —             | each       | 2272300  |
| Dissolved Oxygen 3 Reagent Powder Pillows      | 1             | 100/pkg    | 98799    |
| Iodate-Iodide Titration Cartridge, 0.3998 N    | varies        | each       | 1496101  |
| Starch Indicator Solution                      | 1 dropperful  | 100 mL MDB | 34932    |
| Water, deionized                               | varies        | 4 L        | 27256    |

#### Required apparatus

| Description                  | Quantity/test | Unit | Item no. |
|------------------------------|---------------|------|----------|
| Clippers for plastic pillows | 1             | each | 96800    |
| Cylinder, graduated, 5-mL    | 1             | each | 50837    |
| Cylinder, graduated, 10-mL   | 1             | each | 50838    |
| Cylinder, graduated, 25-mL   | 1             | each | 50840    |

**Required apparatus (continued)**

| Description                                    | Quantity/test | Unit  | Item no. |
|--|---------------|-------|----------|
| Cylinder, graduated, 50-mL                     | 1             | each  | 50841    |
| Digital Titrator                               | 1             | each  | 1690001  |
| Delivery tube for Digital Titrator, J-hook tip | 1             | 5/pkg | 1720500  |
| Flask, Erlenmeyer, 125-mL                      | 2             | each  | 50543    |

**Recommended standards**

| Description  | Unit   | Item no. |
|--|--------|----------|
| Sulfite Equivalent Standard Solution, 10-mL Voluette® Ampule, 5,000-mg/L as $\text{SO}_3^{2-}$ | 16/pkg | 2267410  |
| Sulfite Equivalent Standard Solution, 15 mg/L as $\text{SO}_3^{2-}$                            | 500 mL | 2408449  |
| Sodium Thiosulfate Standard Solution, 0.025 N  | 1 L    | 2409353  |

**Optional reagents and apparatus**

| Description   | Unit   | Item no. |
|---|--------|----------|
| Ampule Breaker, 10-mL Voluette® Ampules   | each   | 2196800  |
| Flask, volumetric, Class A, 250-mL  | each   | 1457446  |
| Pipet, volumetric, Class A, 10-mL   | each   | 1451538  |
| Pipet filler, safety bulb   | each   | 1465100  |
| Pipet, TenSette®, 0.1–1.0 mL  | each   | 1970001  |
| Pipet tips for TenSette® Pipet, 0.1–1.0 mL  | 50/pkg | 2185696  |
| Sulfuric Acid Standard Solution, 19.2 N   | 100 mL | 203832   |
| 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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