

Iodate-Iodide Method¹

Method 8071

0–500 mg/L as SO₃²⁻ (or 0 to more than 500 mg/L)

Buret Titration

Scope and application: For boiler water.

¹ Adapted from *Standard Methods for the Examination of Water and Wastewater*.



Test preparation

Before starting

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

Let the sample temperature decrease to 50 °C (122 °F) or less before 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

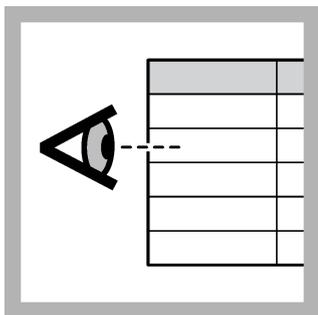
Description	Quantity
Dissolved Oxygen 3 Reagent Powder Pillow	1
Potassium Iodide-Iodate Standard Solution, 0.0125 N	varies
Starch Indicator Solution	1 mL
Buret, Class A, 10 mL	1
Graduated cylinder (use a size that is applicable to the selected sample volume), or TenSette pipet with tips	1
Erlenmeyer 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.

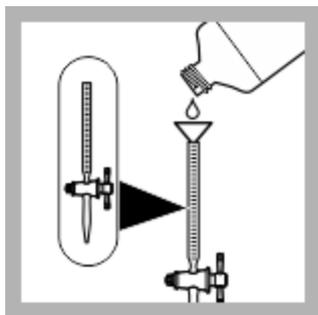
Sample collection

- 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.
- Let the sample temperature decrease to 50 °C (122 °F) or less before analysis.
- Prevent agitation of the sample and exposure to air.

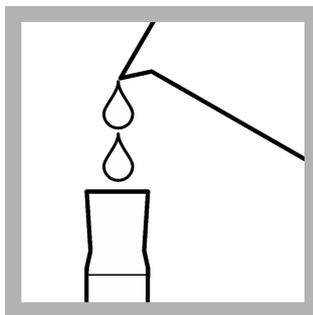
Test procedure



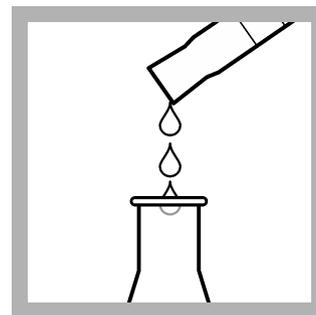
1. Select a sample volume and titrant from [Table 1](#) on page 3.



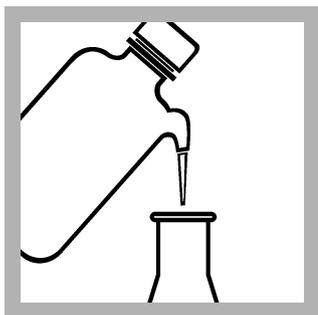
2. Fill a 10-mL buret to the zero mark with the titrant.



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



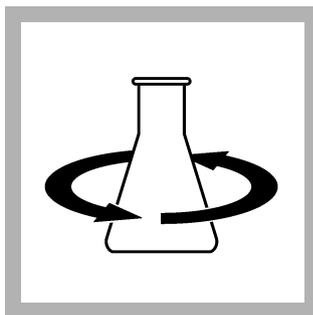
4. Pour the sample into a clean, 250-mL Erlenmeyer flask.



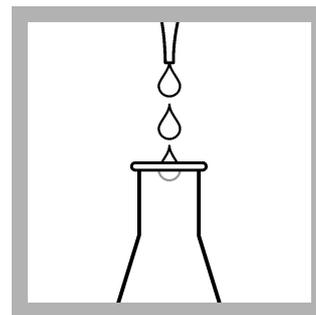
5. If the sample volume is less than 50 mL, dilute to approximately 50 mL with deionized water.



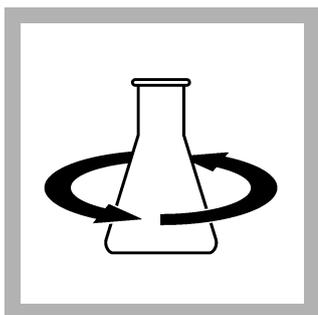
6. Add the contents of one Dissolved Oxygen 3 Powder Pillow.



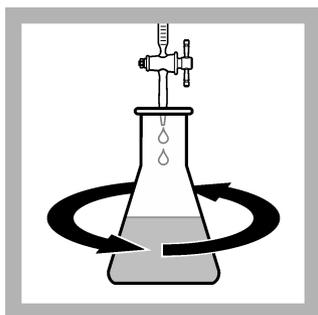
7. Swirl to mix.



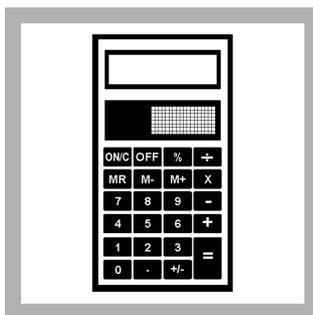
8. Add 1 mL of Starch Indicator Solution.



9. Swirl to mix.



10. Put the flask under the buret. Swirl the flask. Add titrant until the color changes to a permanent blue color.



11. Use the multiplier in [Table 1](#) on page 3 to calculate the concentration. mL of titrant × multiplier = mg/L sulfite SO_3^{2-} .

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 0.0125 N titrant and 6 mL of titrant was used at the endpoint. The concentration is $6 \text{ mL} \times 10 = 60 \text{ mg/L}$ sulfite SO_3^{2-} .

Table 1 Sample volumes and multipliers

Range (mg/L)	Sample volume (mL)	Titrant—potassium iodide-iodate	Multiplier
0–100	50	0.0125 N	10
40–200	25	0.0125 N	20
100–500	10	0.0125 N	50
More than 500	5	0.0125 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 sulfite (SO_3^{2-}) to...	multiply by:	Example
mg/L bisulfite, hydrogen sulfite (HSO_3^-)	1.01	$200 \text{ mg/L } \text{SO}_3^{2-} \times 1.01 = 202 \text{ mg/L } \text{HSO}_3^-$
mg/L sodium bisulfite, sodium hydrogen sulfite (NaHSO_3)	1.30	$200 \text{ mg/L } \text{SO}_3^{2-} \times 1.30 = 260 \text{ mg/L } \text{NaHSO}_3$
mg/L sodium metabisulfite, sodium pyrosulfite ($\text{Na}_2\text{S}_2\text{O}_5$)	2.37	$200 \text{ mg/L } \text{SO}_3^{2-} \times 2.37 = 474 \text{ mg/L } \text{Na}_2\text{S}_2\text{O}_5$
mg/L sodium sulfite (Na_2SO_3)	1.58	$200 \text{ mg/L } \text{SO}_3^{2-} \times 1.58 = 316 \text{ mg/L } \text{Na}_2\text{SO}_3$

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 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 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 1.0 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.

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	2459800
Dissolved Oxygen 3 Reagent Powder Pillows	1	100/pkg	98799
Potassium Iodide-Iodate Standard Solution, 0.0125 N	varies	1 L	1400153
Starch Indicator Solution	1 mL	100 mL MDB	34932
Water, deionized	varies	4 L	27256

Required apparatus

Description	Quantity/test	Unit	Item no.
Buret clamp, double	1	each	32800
Buret, Class A, 10 mL	1	each	2636538
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

Required apparatus (continued)

Description	Quantity/test	Unit	Item no.
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.
Sulfite Equivalent Standard Solution, 10-mL Voluette [®] Ampule, 5,000-mg/L as SO ₃ ²⁻	16/pkg	2267410
Sulfite Equivalent Standard Solution, 15 mg/L as 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
Sulfuric Acid Standard Solution, 19.2 N	100 mL	203832



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