Sulfite DOC316.53.01162

Iodate-Iodide Buret Titration Method¹

0 to 500 mg/L as SO_3^{2-} (or 0 to > 500 mg/L)

Method 8071
Buret Titration

Scope and Application: For boiler water.

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



Test preparation

Before starting the test:

Sulfite is easily destroyed by atmospheric oxygen. Violent shaking or swirling will cause low results.

Analyze samples immediately. Cool hot samples to 50 °C (122 °F) or lower before analysis.

Collect the following items:

Description	Quantity
Dissolved Oxygen Reagent Powder Pillow	1 pillow
Potassium Iodide-Iodate Standard Solution, 0.0125 N	1 bottle
Starch Indicator Solution	1 bottle
Buret, Class A, 10-mL, with support stand	1
Graduated cylinder (see Range-specific information)	1
Erlenmeyer flask, 250-mL	1

See Consumables and replacement items for reorder information.

Buret titration



1. Select a sample volume from the *Range-specific information*.



2. Fill the buret to the zero mark with 0.0125 N Potassium lodide-lodate Standard Solution.



3. Use a graduated cylinder or pipet to measure the sample volume. Add the sample to the Erlenmeyer flask.

If the sample volume is less than 50 mL, gently dilute to approximately 50 mL with deionized water. Do not agitate.



4. Add the contents of one Dissolved Oxygen 3 Powder Pillow and mix gently.

Buret titration (continued)



5. Add one full dropper of Starch Indicator Solution. Swirl gently to mix.



6. Titrate the sample while gently swirling the flask until the color changes to a permanent blue color (end point).

Write down the volume of

titrant that was used.



information to calculate the concentration: mL titrant used x multiplier = mg/L sulfite (SO_3^{2-}) Example: 50 mL of sample was titrated and 5 mL of titrant was used to reach the end point. The concentration is 5 x 10 = 50 mg/L as SO_3^{2-}

7. Use the multiplier in

the Range-specific

Table 395 Range-specific information

Range (mg/L as SO ₃ ²⁻)	Sample volume (mL)	Multiplier
0–100	50	10
40–200	25	20
100–500	10	50
Over 500	5	100

Interferences

Interfering substances lists substances that can interfere with this test.

Table 396 Interfering substances

Interfering substance	Interference level
Nitrite	Nitrite will react with sulfite to cause a negative error in the titration.
Metals	Some metals, especially copper, catalyze the oxidation of sulfite to sulfate. Immediate fixing of sample with the contents of one Sulfamic Acid Powder Pillow per liter of sample will help minimize the interference.
Organic compounds	Oxidizable organic compounds could cause high results.
Oxidizable compounds	Will cause high results.
Sulfide	Will cause high results.

Sample collection, preservation and storage

- Collect samples in clean plastic or glass bottles. Fill completely and cap tightly.
- Avoid excessive agitation or prolonged exposure to air. Complete the test procedure as soon as possible after collection for best accuracy. Samples cannot be preserved or stored.
- Cool hot samples to 50 °C (122 °F) or lower.

Accuracy check

The standard additions method can be used to find if the sample has an interference. The standard solution method can be used to confirm analytical technique and reagent performance.

Standard additions method (sample spike)

Required for accuracy check:

- Sulfite Voluette Ampule Standard, 5,000-mg/L SO₃²⁻
- Ampule breaker
- TenSette Pipet, 0.1–1.0 mL and Pipet Tips
- Pipet filler
- 1. Open the standard solution ampule.
- 2. Use the TenSette Pipet to add 0.2 mL of the standard to the titrated sample. Swirl to mix.
- Titrate the spiked sample to the end point. Write down the amount of titrant that was used to reach the end point.
- 4. Use the TenSette Pipet to add 0.3 mL of standard to the titrated sample. Swirl to mix.
- 5. Titrate the spiked sample to the end point. Write down the amount of titrant that was used to reach the end point.
- 6. Use the TenSette Pipet to add 0.4 mL of standard to the titrated sample. Swirl to mix.
- Titrate the spiked sample to the end point. Write down the amount of titrant that was used to reach the end point.
- 8. Each 0.1 mL of standard that was added will use 1.0 mL of titrant to reach the endpoint. If more or less titrant was used, there can be an interference (see *Interferences*) or the concentration of the titrant has changed (see *Standard solution method*).

Standard solution method

Complete the following test to make sure the concentration is accurate.

Required for accuracy check:

- 0.025 N Sodium Thiosulfate Standard Solution
- 250-mL volumetric flask, Class A
- 10-mL volumetric pipet, Class A
- Use a pipet to add 10.0-mL of 0.025 N Sodium Thiosulfate Standard Solution to a 250-mL volumetric flask. Dilute to the mark with deionized water. The diluted standard is equivalent to 40 mg/L sulfite.
- Use a graduated cylinder to measure 50 mL of the diluted standard. Add the standard to the Erlenmeyer flask.
- Titrate the standard to the end point with the titrant solution. The result should be close to 40 mg/L sulfite.

Summary of method

The water sample is acidified, treated with a starch indicator 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, excess iodine will react with the starch to form a blue color.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
Dissolved Oxygen 3 Reagent Powder Pillows	1 pillow	100/pkg	98799
Potassium Iodide-Iodate Standard Solution, 0.0125 N	varies	1 L	1400153
Starch Indicator Solution	1 mL	100 mL MDB	34932
Reagent set, Sulfite Buret Titration	_	100 tests	2459800

Required apparatus

Description	Quantity/Test	Unit	Catalog number
Buret, Class A, Teflon Stopcock plug, Certified 10-mL	1	each	2636538
Buret Clamp, double	1	each	32800
Clippers, for opening pillows	1	each	96800
Graduated cylinder—select one or more based on range:			
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
Flask, Erlenmeyer, graduated, 250-mL	1	each	50546
Support Stand	1	each	56300
Pipet, volumetric, Class A, 5 mL	1	each	1451537
Pipet, volumetric, Class A, 10 mL	1	each	1451538
Pipet filler, Safety Bulb	1	each	1465100

Recommended standards

Description	Unit	Catalog number
Sodium Thiosulfate Standard Solution, 0.025 N	1 L	2409353
Sulfite Standard Solution, Voluette® Ampule, 5,000-mg/L SO ₃ 10-mL	16/pkg	1426710

Optional reagents and apparatus

Description	Unit	Catalog number
TenSette Pipet, 0.1 to 1.0 mL	each	1970001
Pipet tips	50/pkg	2185696
Ampule breaker	each	2196800

