

Sodium Thiosulfate Method

Method 8211

20 to more than 400 mg/L as CrO_4^{2-}

Digital Titrator

Scope and application: Closed system cooling towers.



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

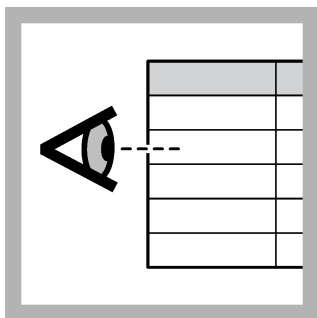
Description	Quantity
Dissolved Oxygen 3 Reagent Powder Pillow	1
Potassium Iodide Powder Pillow	1
Sodium Thiosulfate Titration Cartridge, 0.2068 N	1
Starch Indicator Solution	1 mL
Digital Titrator	1
Delivery tube for Digital Titrator	1
Graduated cylinder (use a size that is applicable to the selected sample volume)	1
Erlenmeyer flask, 125-mL	1
Water, deionized	varies

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

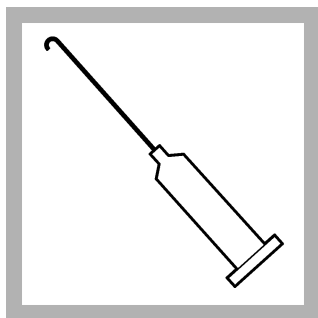
Sample collection

- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.
- Analyze the samples as soon as possible for best results.
- If prompt analysis is not possible, add 1 mL of concentrated sulfuric acid. Swirl to mix.

Test procedure



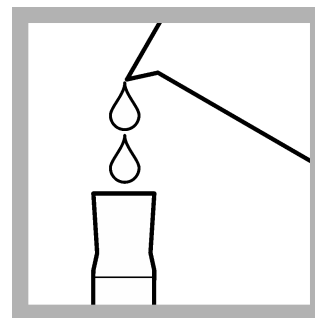
1. Select a sample volume and titration cartridge from [Table 1](#) on page 3.



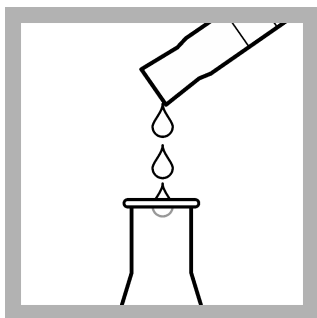
2. Insert a clean delivery tube into the digital titration cartridge. Attach the cartridge to the Digital Titrator.



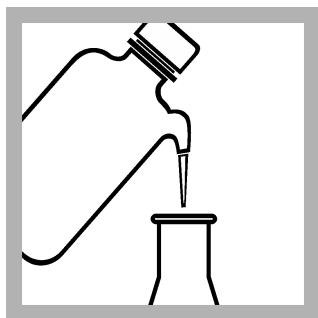
3. Hold the Digital Titrator with the cartridge tip up. Turn the delivery knob to eject air and a few drops of titrant. Reset the counter to zero and clean the tip.



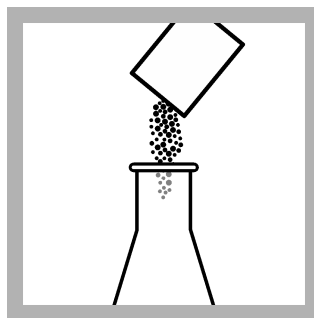
4. Use a graduated cylinder to measure the sample volume from [Table 1](#) on page 3.



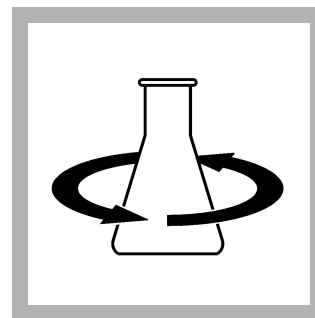
5. Pour the sample into a clean, 125-mL Erlenmeyer flask.



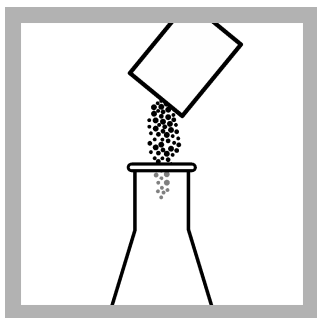
6. If the sample volume is less than 50 mL, dilute to approximately 50 mL with deionized water.



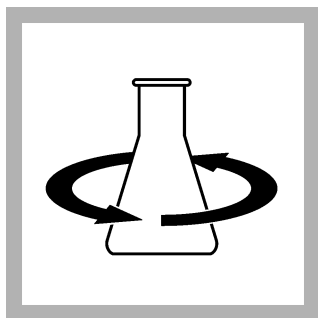
7. Add the contents of one Potassium Iodide Powder Pillow.



8. Swirl to mix.



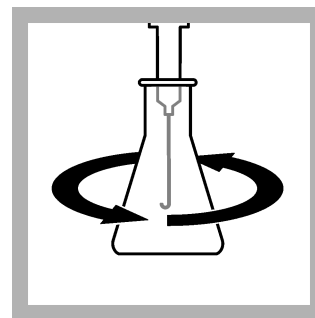
9. Add the contents of one Dissolved Oxygen 3 Reagent Powder Pillow.



10. Swirl to mix.



11. Set and start a timer for 3 minutes. A 3-minute reaction time starts. Do not wait more than 10 minutes.



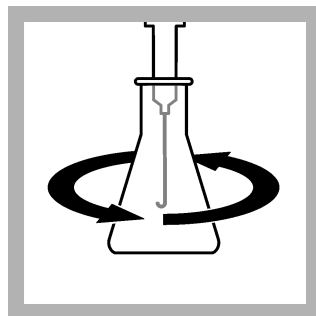
12. Put the end of the delivery tube fully into the solution. Swirl the flask. Turn the knob on the Digital Titrator to add titrant to the solution. Continue to swirl the flask. Add titrant until the color changes to pale yellow.



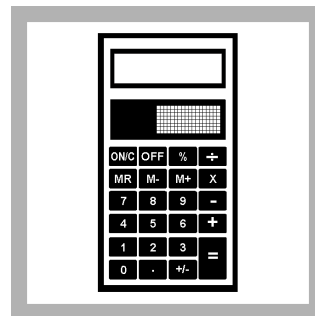
13. Add 1 full dropper of Starch Indicator Solution.



14. Swirl to mix. The color of the solution changes to dark blue.



15. 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 dark blue to colorless. Record the number of digits on the counter.



16. Use the multiplier in [Table 1](#) on page 3 to calculate the concentration. Digits used \times digit multiplier = mg/L chromate as CrO_4^{2-} .

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 50-mL sample was titrated with 0.2068 N Sodium Thiosulfate Titration Cartridge and the counter showed 250 digits at the endpoint. The concentration is $250 \text{ digits} \times 0.2 = 50 \text{ mg/L as } \text{CrO}_4^{2-}$.

Table 1 Sample volumes and digit multipliers

Range (mg/L as CrO_4^{2-})	Sample volume (mL)	Titration cartridge	Digit multiplier
20–80	50	0.2068 N	0.2
50–200	20	0.2068 N	0.5
100–400	10	0.2068 N	1.0
More than 400	5	0.2068 N	2.0

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 chromate (CrO_4^{2-}) to...	multiply results by...	Example
mg/L chromium	0.448	mg/L chromate \times 0.448 = mg/L chromium
mg/L sodium chromate	1.4	mg/L chromate \times 1.4 = mg/L sodium chromate

Interferences

Interfering substance	Interference level
Copper	Causes positive interference. To remove the interference, add one Magnesium CDTA Powder Pillow and then two 1.0-g scoops of Sodium Acetate to the sample in step 7.
Iron, ferric (Fe^{3+})	
Other oxidants	Substances that can oxidize iodide to iodine in acidic conditions cause positive interference.

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:

- Hexavalent Chromium Standard Solution, 1000 mg/L as Cr⁶⁺
 - 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 0.2-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 0.2-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 22 digits of the 0.2068 N Sodium Thiosulfate 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:

- Hexavalent Chromium Standard Solution, 1000 mg/L as Cr⁶⁺
 - 100-mL volumetric flask, Class A
 - 3.0-mL volumetric pipet, Class A and pipet filler safety bulb
 - Deionized water
1. Prepare a 67-mg/L chromate standard solution as follows:
 - a. Use a pipet to add 3.0 mL of hexavalent chromium 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

Chromate in the sample reacts with iodide in acidic conditions to form iodine as triiodide. The addition of starch indicator makes a blue color complex with the iodine. The complex is titrated with sodium thiosulfate to a colorless endpoint. The quantity of titrant used is proportional to the chromate concentration.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
Chromate Reagent Set (approximately 100 tests)	—	each	2272400
Dissolved Oxygen 3 Reagent Powder Pillows	1 pillow	100/pkg	98799
Potassium Iodide Powder Pillows (2x)	1 pillow	50/pkg	2059996

Consumables and replacement items (continued)

Description	Quantity/Test	Unit	Item no.
Sodium Thiosulfate Titration Cartridge, 0.2068 N	varies	each	2267601
Starch Indicator Solution	1 mL	each	34932
Water, deionized	varies	4 L	27256

Required apparatus

Description	Quantity/test	Unit	Item no.
Graduated cylinders—Select one or more for the sample volume:			
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
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

Recommended standards

Description	Unit	Item no.
Hexavalent Chromium Standard Solution, 1000 mg/L as Cr ⁶⁺	100 mL	1466442

Optional reagents and apparatus

Description	Unit	Item no.
Magnesium CDTA Powder Pillows	100/pkg	1408099
Sodium Acetate, trihydrate, ACS	454 g	17801H
Chromium Hexavalent Standard Solution, 50.0-mg/L Cr ⁶⁺	100 mL	81042H
Sulfuric Acid, ACS	500 mL	97949
Bottle, sampling, with cap, low density polyethylene, 250-mL	12/pkg	2087076
Spoon, measuring, 1-g	each	51000
Clippers	each	96800
Flask, volumetric, Class A, 100-mL	each	1457442
Pipet, volumetric, Class A, 3-mL	each	1451503
Pipet filler, safety bulb	each	1465100
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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