Nitrite

DOC316.53.01178

Ceric Acid Titration Method

Method 8351

100 to 2500 mg/L as $NaNO_2$

Digital Titrator

Scope and Application: For cooling tower waters.

Test preparation

Before starting the test:

For added convenience when stirring, use the TitraStir® stirring apparatus.

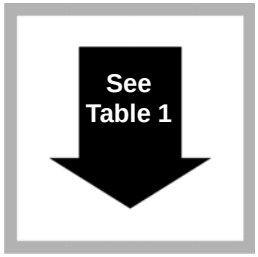
A pipet is recommended for measuring the sample volume when the volume is less than 10 mL.

Collect the following items:

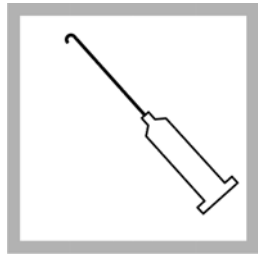
Description	Quantity
Ferroin Indicator Solution	1 bottle
Sulfuric Acid Standard Solution, 5.25 N	1 bottle
Ceric Standard Solution Titration Cartridge, 0.5 N	1 cartridge
Digital titrator	1
Delivery tube for digital titrator	1
Graduated cylinder	1
Erlenmeyer flask, 125-mL	1

See *Consumables and replacement items* for reorder information.

Nitrite



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



2. Insert a clean delivery tube into the titration cartridge. Attach the cartridge to the titrator.

3. 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.

4. Use a graduated cylinder or pipet to measure the sample volume from the *Range-specific information* table in a 125 mL Erlenmeyer flask.

5. Dilute to approximately 75 mL with deionized water.

6. Add five drops of 5.25 N Sulfuric Acid Standard Solution to the flask. Swirl to mix.

7. Add one drop of Ferroin Indicator Solution to the flask. Swirl to mix.

8. Place the delivery tube into the solution and swirl the flask. Turn the knob on the titrator to add titrant to the solution. Continue to swirl the flask and add titrant until the color changes from orange to pale blue.

Write down the number of digits displayed on the counter.

Nitrite (continued)

9. Use the multiplier in the *Range-specific information* table to calculate the concentration:

digits x multiplier = mg/L sodium nitrite ($NaNO_2$)

Example: 25 mL of sample was titrated and 250 digits were used to reach the endpoint. The concentration is 250 x 0.86 = 215 mg/L as $NaNO_2$

Table 255 Range-specific information

Range (mg/L as $NaNO_2$)	Sample volume (mL)	Multiplier
100–400	25	0.86
400–800	10	2.15
800–1500	5	4.31
1500–2500	2	10.78

Sample collection, preservation and storage

Collect samples in clean plastic or glass bottles. Prompt analysis is recommended.

If prompt analysis is not possible, store samples for 24 to 48 hours at 4 °C (39 °F) or lower. Warm to room temperature before analysis. Do not use acid preservatives.

Accuracy check

Standard solution method
Required for accuracy check:

- Sodium Nitrite, ACS
- 1000-mL volumetric flask, Class A

1. Prepare a 1000-mg/L sodium nitrite standard solution as follows. Add 1.000 gram of sodium nitrite to the volumetric flask. Add deionized water to the mark and mix fully.
2. Add 5.0 mL of the solution to an Erlenmeyer flask. Dilute to approximately 75 mL with deionized water and mix fully.
3. Add the sulfuric acid and Ferroin indicator. Swirl to mix.
4. Titrate the standard to the end point with the titration cartridge and calculate the result. The result should be close to 1000 mg/L as $NaNO_2$.

Standardization of ceric standard solution
The normality of the ceric standard solution will sometimes decrease over time. Before use, verify the normality with the following procedure. This standardization should be done monthly.

1. Use a graduated cylinder or pipet to measure 50 mL of deionized water into a 125-mL Erlenmeyer flask.
2. Add 5 mL of 19.2 N Sulfuric Acid Standard Solution. Swirl to mix.
3. Insert a clean delivery tube into a Ceric Standard Titration Cartridge.
4. Hold the Digital Titrator with the cartridge tip pointing up. Turn the delivery knob until a few drops of titrant are expelled. Reset the counter to zero and wipe the tip.
5. Place the delivery tube tip into the solution. While swirling the flask, add 200 digits of Ceric Standard.
6. Insert a clean delivery tube into a 0.200 N Sodium Thiosulfate Titration Cartridge.
7. Hold the Digital Titrator with the cartridge tip pointing up. Turn the delivery knob until a few drops of titrant are expelled. Reset the counter to zero and wipe the tip.
8. Place the delivery tube tip into the solution. While swirling the flask, titrate with the sodium thiosulfate from an intense yellow color to a faint yellow color. Record the number of digits required. This step should require about 400–450 digits of titrant.

9. Add one drop of Ferroin Indicator Solution. Swirl to mix. The solution will turn a faint blue.
10. Continue titrating with the Sodium Thiosulfate Standard Solution from a faint blue to orange color. Record the number of digits required.
11. Divide the number of digits by 500 to calculate the correction factor (number of digits ¸ 500 = correction factor).
12. Multiply the mg/L sodium nitrite from the titration procedure by the correction factor to obtain the correct sodium nitrite concentration.

Summary of method

Sodium nitrite is titrated with tetravalent cerium ion, a strong oxidant, in the presence of ferroin indicator. After the cerium oxidizes the nitrite, the indicator is oxidized and causes a color change from orange to pale blue. The concentration of sodium nitrite is proportional to the amount of titrant.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
Ceric Standard Solution Titration Cartridge, 0.5 N	1 pillow	each	2270701
Ferroin Indicator Solution	1 pillow	29 mL DB	181233
Sulfuric Acid Standard Solution, 5.25 N	1 pillow	100 mL MDB	244932

Required apparatus

Description	Quantity/Test	Unit	Catalog number
Digital Titrator	1	each	1690001
Flask, Erlenmeyer, graduated, 125-mL	1	each	50543
Cylinder, graduated, 25-mL	1	each	50840
Delivery tubes w/ 180° hook	1	each	1720500
Delivery tubes w/ 90° hook	1	each	4157800

Recommended standards

Description	Unit	Catalog number
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

Optional reagents and apparatus

Description	Unit	Catalog number
Stir bar, octagonal 28.6 mm x 7.9 mm	each	2095352
TenSette Pipet, 1.0 to 10.0 mL	each	1970010
TitraStir Stir Plate, 115 VAC	each	1940000
TitraStir Stir Plate, 230 VAC	each	1940010
Water, deionized	500 mL	27249
Thermometer -10 to 225 °C	405 mm	2635700
Volumetric flask, 100 mL	each	1457453
Volumetric pipet, 5 mL	each	1451537
Sampling bottle, 250 mL	each	2087076
Analytical balance	each	2936701
Weighing papers	500/pkg	1473800

FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:
In the U.S.A. – **Call toll-free 800-227-4224**
Outside the U.S.A. – **Contact the HACH office or distributor serving you.**
On the Worldwide Web – **www.hach.com**; E-mail – **techhelp@hach.com**

HACH COMPANY
WORLD HEADQUARTERS
Telephone: (970) 669-3050
FAX: (970) 669-2932

© Hach Company, 2007, 2010, 2012. All rights reserved. Printed in the U.S.A.