# Iron

DOC316.53.01177

## TitraVer Titration Method

**Method 8214**

**10–1000 mg/L as Fe**

**Digital Titrator**

**Scope and application:** For water, wastewater and seawater.

## 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

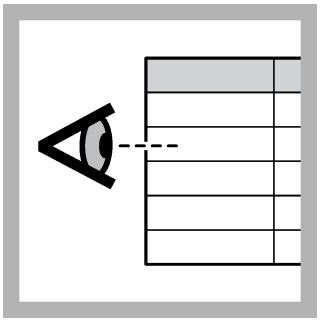
| Description | Quantity |
|---|---|
| Citrate Buffer Powder Pillow | 1 |
| Sodium Periodate Powder Pillow | 1 |
| Sulfosalicylic Acid Powder Pillow | 1 |
| TitraVer Standard Solution Titration Cartridge (refer to Sample volumes and digit multipliers on page 3) | 1 |
| Digital Titrator | 1 |
| Delivery 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.

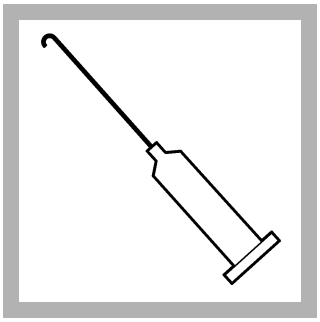
### Sample collection

- Collect samples in clean glass or plastic bottles.

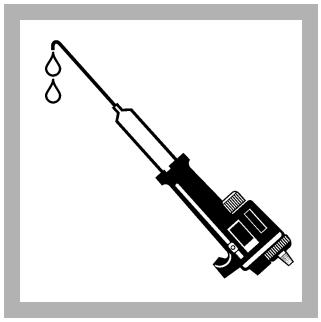
## Test procedure



**1.** Select a sample volume and titration cartridge from Table 1 on page 3.

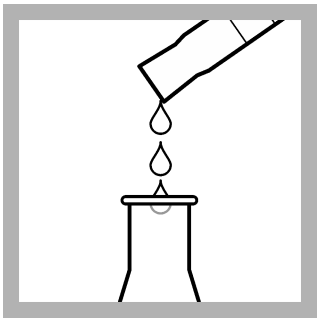


**2.** Insert a clean delivery tube into the digital titration cartridge. Attach the cartridge to the Digital Titrator.

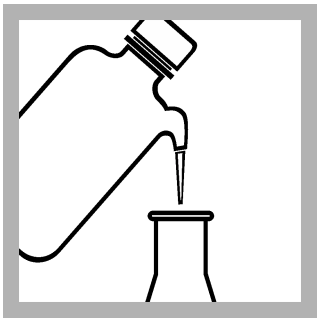


**3.** Hold the Digital Titrator with the cartridge tip up. Turn the delivery knob to eject air and a few drops of titrant. Reset the counter to zero and clean the tip.

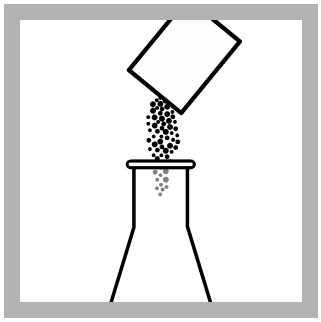
**4.** Use a graduated cylinder or a pipet[1] to measure the sample volume from Table 1 on page 3.



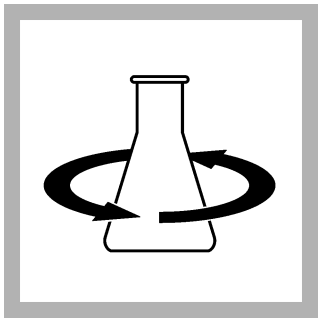
**5.** Pour the sample into a clean, 125-mL Erlenmeyer flask.



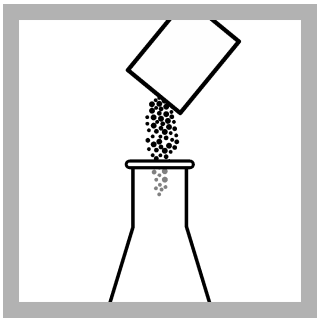
**6.** If the sample volume is less than 50 mL, dilute to approximately 50 mL with deionized water.



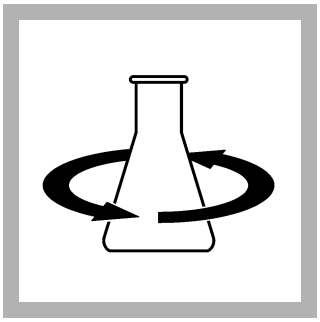
**7.** Add the contents of one Citrate Buffer Powder Pillow.



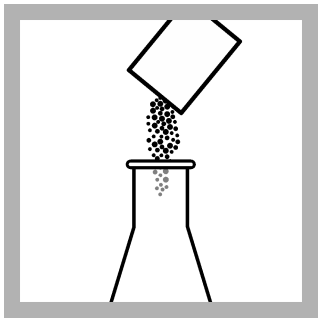
**8.** Swirl to mix.



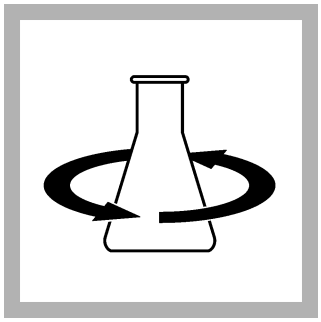
**9.** Add the contents of one Sodium Periodate Powder Pillow.



**10.** Swirl to mix.
The color of the solution changes to yellow if iron is in the sample.

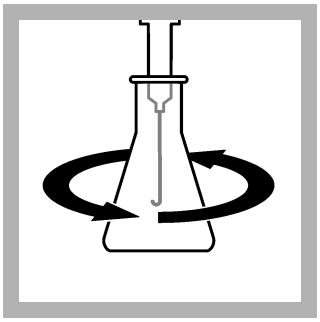


**11.** Add the contents of one Sulfosalicylic Acid Powder Pillow.

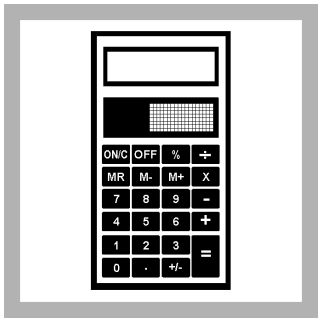


**12.** Swirl to mix.
The color of the solution changes to red if iron is in the sample.

[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.



**13.** 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 red to yellow. Record the number of digits on the counter.




**14.** Use the multiplier in Table 1 on page 3 to calculate the concentration. Digits used × digit multiplier = mg/L Fe.

## 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 0.0716 M TitraVer Standard Solution Titration Cartridge and the counter showed 250 digits at the endpoint. The concentration is 250 digits x 0.1 = 25 mg/L Fe.

**Table 1 Sample volumes and digit multipliers**

| Range (mg/L as Fe) | Sample volume (mL) | Titration cartridge | Digit multiplier |
|---|---|---|---|
| 10–40 | 50 | 0.0716 | 0.1 |
| 25–100 | 20 | 0.0716 | 0.25 |
| 100–400 | 50 | 0.716 | 1.0 |
| 250–1000 | 20 | 0.716 | 2.5 |

## 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:

- Iron Standard Solution, 1000 mg/L as Fe
- 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 0.5 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 0.5-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 0.5-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 10 digits of 0.0716 M TitraVer Standard Solution Titration Cartridge (100 digits of the

0.0716 M titration cartridge) for each 0.5-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.

## Summary of method

Ferrous iron $Fe^{2+}$ is oxidized by sodium periodate to ferric ion $Fe^{3+}$. The ferric ion develops a red complex with sulfosalicylic acid. The red complex is removed by titration with EDTA. Citric acid is used to buffer the solution and to make the ferric ion stable in the solution.

## Consumables and replacement items

### Required reagents

| Description | Quantity/Test | Unit | Item no. |
|---|---|---|---|
| Reagent set, 10–100 mg/L range (approximately 100 tests): | — | each | 2449200 |
| Citrate Buffer Powder Pillows | 1 pillow | 100/pkg | 2081599 |
| Sodium Periodate Powder Pillows | 1 pillow | 100/pkg | 98499 |
| Sulfosalicylic Acid Powder Pillows | 1 pillow | 100/pkg | 2081669 |
| TitraVer Standard Solution Titration Cartridge, 0.0716 M | varies | each | 2081701 |
| Reagent set, 100–1000 mg/L range (approximately 100 tests): | — | each | 2449300 |
| Citrate Buffer Powder Pillows | 1 pillow | 100/pkg | 2081599 |
| Sodium Periodate Powder Pillows | 1 pillow | 100/pkg | 98499 |
| Sulfosalicylic Acid Powder Pillows | 1 pillow | 100/pkg | 2081669 |
| TitraVer Standard Solution Titration Cartridge, 0.716 M | varies | each | 2081801 |

### Required apparatus

| Description | Quantity/test | Unit | Item no. |
|---|---|---|---|
| Graduated cylinders—Select one or more for the sample volume: | | | |
| Cylinder, graduated, 25 mL | 1 | each | 50840 |
| Cylinder, graduated, 50 mL | 1 | each | 50841 |
| Digital Titrator | 1 | each | 1690001 |
| Delivery tube for Digital Titrator, J-hook tip | 1 | 5/pkg | 1720500 |
| Pipet filler, safety bulb | 1 | each | 1465100 |
| Pipet, volumetric, Class A, 20 mL | 1 | each | 1451520 |
| Flask, Erlenmeyer, 125 mL | 1 | each | 50543 |

### Recommended standards

| Description | Unit | Item no. |
|---|---|---|
| Iron Standard Solution, 1000 mg/L as Fe | 100 mL | 227142 |
| Iron Standard Solution, 10 mg/L as Fe | 500 mL | 14049 |
| Iron Standard Solution, 25 mg/L as Fe | 10 mL/16 | 1425310 |
| Iron Standard Solution, 50 mg/L as Fe | 10 mL/16 | 1425410 |
| Iron Standard Solution, 100 mg/L as Fe | 100 mL | 1417542 |

## Optional apparatus

| Description | Unit | Item no. |
|---|---|---|
| Ampule Breaker, 10-mL Voluette® Ampules | each | 2196800 |
| 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 |

**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



04/2015, Edition 8