# **Volatile Acids**

#### **Sodium Hydroxide Method**

#### 100-2400 mg/L CH<sub>3</sub>COOH

Scope and application: For wastewater.

☐ Test preparation

# Before starting

Refer to the *Volatile Acids Procedure, Sample Distillation* in the distillation apparatus documentation to distill the sample. As an alternative, refer to the distillation procedure in *Standard Methods for the Examination of Water and Wastewater*.

The final result is adjusted to give the correct answer based on a 70% correction factor. For higher recoveries, use the esterification method.

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 |
|--|----------|
| Phenolphthalein Indicator Powder Pillows   | 1        |
| Sodium Hydroxide Titration Cartridge, 0.9274 N                                   | 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, 250-mL   | 1        |
| Water, deionized   | varies   |

Refer to Consumables and replacement items on page 3 for order information.

#### Sample collection

Collect samples in clean glass or plastic bottles.

## Method 8218 Digital Titrator

### **Test procedure**



1. Collect 125 mL of distillate.



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



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



4. 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.



5. Use a graduated cylinder or a pipet to measure the sample volume from Table 1 on page 3.



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



7. If the sample volume is less than 150 mL, dilute to approximately 150 mL with deionized water.



**8.** Add the contents of one Phenolphthalein Indicator Powder Pillow.



9. Swirl to mix.



**10.** 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 light pink. Record the number of digits on the counter.



**11.** Use the multiplier in Table 1 on page 3 to calculate the concentration. Digits used × digit multiplier = mg/L Volatile Acids (as acetic acid, CH<sub>3</sub>COOH).

#### 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 150-mL sample was titrated with 0.9274 N Sodium Hydroxide Titration Cartridge and the counter showed 250 digits at the endpoint. The concentration is 250 digits x 1 = 250 mg/L Volatile Acids (as acetic acid,  $CH_3COOH$ ).

| Range (mg/L as CH <sub>3</sub> COOH) | Sample volume (mL) | Digit multiplier |
|--------------------------------------|--------------------|------------------|
| 100–400                              | 150                | 1                |
| 200–800                              | 75                 | 2                |
| 600–2400                             | 25                 | 6                |

Table 1 Sample volumes and digit multipliers

#### Summary of method

The sample is acidified with sulfuric acid and distilled with deionized water. The distillate is titrated to the phenolphthalein endpoint with sodium hydroxide standard.

#### **Consumables and replacement items**

#### **Required reagents**

| Description  | Quantity/Test | Unit    | Item no. |
|--|---------------|---------|----------|
| Volatile Acid Reagent set (approximately 100 tests): | _             | each    | 2460200  |
| Phenolphthalein Indicator Powder Pillows             | 1 pillow      | 100/pkg | 94299    |
| Sodium Hydroxide Titration Cartridge, 0.9274 N       | varies        | each    | 1484201  |
| Water, deionized                                     | varies        | 100 mL  | 27242    |

#### **Required apparatus**

| Description   | Quantity/test | Unit  | ltem 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, 250 mL                                     | 1             | each  | 50546    |

#### **Recommended standards**

| Description  | Unit   | ltem no. |
|--|--------|----------|
| Volatile Acids Standard Solution, 1000 mg/L as Acetic Acid | 100 mL | 1420542  |

#### **Optional apparatus**

| Description   | Unit  | ltem no. |
|---|-------|----------|
| Ampule Breaker, 10-mL Voluette <sup>®</sup> Ampules                                       | each  | 2196800  |
| Stir bar, octagonal   | each  | 2095352  |
| TitraStir <sup>®</sup> Titration Stand, 115 VAC   | each  | 1940000  |
| TitraStir <sup>®</sup> 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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