# **Volatile Acids**

## Sodium Hydroxide Method<sup>1</sup>

# 100–2400 mg/L CH<sub>3</sub>COOH (Acetic Acid)

Scope and application: For water, wastewater and seawater.

<sup>1</sup> Adapted from Standard Methods for the Examination of Water and 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.

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 Standard Solution, 0.100 N	1
Buret, Class A, 50 mL	1
Graduated cylinder (use a size that is applicable to the selected sample volume), or TenSette pipet with tips	1
Erlenmeyer flask, 250 mL	1
Funnel, micro	1
Support stand with buret clamp	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.
- Analyze the samples as soon as possible for best results.
- If immediate analysis is not possible, keep the samples at or below 6 °C (43 °F) for a maximum of 24 hours.
- Let the sample temperature increase to room temperature before analysis.

# Method 8291 Buret Titration

# **Test procedure**







**2.** Select a sample volume and titrant from Table 1 on page 3.



**3.** Fill a 50-mL buret to the zero mark with the titrant.



**4.** Use a graduated cylinder or pipet<sup>1</sup> to measure the sample volume from Table 1 on page 3.



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



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



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



8. Swirl to mix.



**9.** Put the flask under the buret. Swirl the flask. Add titrant until the color changes to light pink and stays pink for 30 seconds.



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

# Sample volumes and multipliers

Select a range in Table 1, then read across the table row to find the applicable information for this test. Use the multiplier to calculate the concentration in the test procedure.

<sup>&</sup>lt;sup>1</sup> 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.

**Example:** A 150-mL sample was titrated with 0.100 N titrant and 12 mL of titrant was used at the endpoint. The concentration is  $12 \text{ mL} \times 86 = 1032 \text{ mg/L}$  Volatile Acids (as acetic acid, CH<sub>3</sub>COOH).

Range (mg/L)	Sample volume (mL)	Titrant—sodium hydroxide	Multiplier
100–400	150	0.100 N	86
200–800	75	0.100 N	172
600–2400	25	0.100 N	516

#### Table 1 Sample volumes and 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	ltem no.
Phenolphthalein Indicator Powder Pillows	1 pillow	100/pkg	94299
Sodium Hydroxide Standard Solution, 0.100 N	varies	1 L	19153

#### **Required apparatus**

Description	Quantity/test	Unit	Item no.
Buret clamp, double	1	each	32800
Buret, Class A, 50 mL	1	each	2636541
Support stand	1	each	56300
Funnel, micro	1	each	2584335
Graduated cylinders—Select one or more for the sample volume:			
Cylinder, graduated, 25 mL	1	each	50840
Cylinder, graduated, 50 mL	1	each	50841
Cylinder, graduated, 100 mL	1	each	50842
Cylinder, graduated, 250 mL	1	each	50846
Pipet, TenSette <sup>®</sup> , 0.1–1.0 mL	1	each	1970001
Pipet tips, for TenSette <sup>®</sup> Pipet, 1.0–10.0 mL	1	50/pkg	2199796
Flask, Erlenmeyer, 250 mL	1	each	50546



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