

# Acetic Acid, Glacial

CH<sub>3</sub>COOH

Formula Wt 60.05

CAS No. 64-19-7

## GENERAL DESCRIPTION

*Typical appearance:* liquid with a sharp odor

*Analytical use:* aqueous and nonaqueous acid–base titrations

*Change in state (approximate):* boiling point, 118 °C; freezing point, 16 °C

*Aqueous solubility:* miscible with water

pK<sub>a</sub>: 4.8

## SPECIFICATIONS

Assay .....	≥99.7% CH <sub>3</sub> COOH
	<i>Maximum Allowable</i>
Color (APHA) .....	10
Dilution test .....	Passes test
Residue after evaporation .....	0.001%
Acetic anhydride [(CH <sub>3</sub> CO) <sub>2</sub> O] .....	0.01%
Chloride (Cl) .....	1 ppm
Sulfate (SO <sub>4</sub> ) .....	1 ppm
Heavy metals (as Pb) .....	0.5 ppm
Iron (Fe) .....	0.2 ppm
Substances reducing dichromate .....	Passes test
Substances reducing permanganate .....	Passes test
Titration base .....	0.0004 meq/g

## TESTS

**Assay and Acetic Anhydride.** Analyze the sample by gas chromatography using the general parameters cited on page 80. The following specific conditions are also required.

*Column:* Type I, methyl silicone

Measure the area under all peaks, and calculate the area percent for acetic acid and acetic anhydride. Correct for water content.

**Color (APHA).** (Page 43).

**Dilution Test.** Dilute 1 volume of the acid with 3 volumes of water and allow to stand for 1 h. The solution should be as clear as an equal volume of water.

**Residue after Evaporation.** (Page 25). Evaporate 100 g (95 mL) to dryness in a tared, preconditioned dish on a hot plate (≈100 °C), and dry the residue at 105 °C for 30 min.

**Chloride.** (Page 35). Dilute 10 g (9.5 mL) with 10 mL of water, and add 1 mL of silver nitrate reagent solution. Prepare a standard containing 0.01 mg of chloride ion (Cl) in 20 mL of water, and add 1 mL of silver nitrate reagent solution. Evaporate the solutions to dryness on a hot plate (≈100 °C). Dissolve the residues with 0.5 mL of ammonium hydrox-

ide, dilute with 20 mL of water, and add 1.5 mL of nitric acid. Any turbidity in the solution of the sample should not exceed that of the standard.

**Sulfate.** To 48 mL (50 g) of sample, add 2 mL of 1% sodium carbonate solution, and evaporate to dryness on a hot plate ( $\approx 100\text{ }^{\circ}\text{C}$ ). Dissolve the residue in 1.0 mL of 10% hydrochloric acid and 15 mL of water. Dilute to 25 mL with water. For the control, take 0.10 mg of sulfate ion ( $\text{SO}_4$ ) in 20 mL of water, add 1.0 mL of 10% hydrochloric acid and dilute to 25 mL. To sample and control solutions, add 1 mL of 12% barium chloride solution. Compare after 10 min. Sample turbidity should not exceed that of the control solution.

**Heavy Metals.** (Page 36, Method 1). To 40 g (38 mL), add about 10 mg of sodium carbonate, evaporate to dryness on a hot plate ( $\approx 100\text{ }^{\circ}\text{C}$ ), dissolve the residue in about 20 mL of water, and dilute with water to 25 mL.

**Iron.** (Page 38, Method 1). To 50 g (48 mL), add 10 mg of sodium carbonate, and evaporate to dryness. Dissolve the residue in 2 mL of hydrochloric acid, dilute with water to 50 mL, and use the solution without further acidification.

**Substances Reducing Dichromate.** To 10 mL, add 1.0 mL of 0.1 N potassium dichromate, and cautiously add 10 mL of sulfuric acid. Cool the solution to room temperature, and allow to stand for 30 min. While the solution is swirled, dilute slowly and cautiously with 50 mL of water, cool, and add 1 mL of freshly prepared 10% potassium iodide reagent solution. Titrate the liberated iodine with 0.1 N sodium thiosulfate, using starch as the indicator. Not more than 0.40 mL of the 0.1 N potassium dichromate should be consumed (not less than 0.60 mL of the 0.1 N sodium thiosulfate should be required). Correct for a complete blank.

**Substances Reducing Permanganate.** Add 40 g (38 mL) of the sample to 10 mL of water. Cool to  $15\text{ }^{\circ}\text{C}$ , add 0.30 mL of 0.1 N potassium permanganate, and allow to stand at  $15\text{ }^{\circ}\text{C}$  for 10 min. The pink color should not be entirely discharged.

**Titration Base.** To 25 g (24 mL) add 0.10 mL of a solution of 1 g of crystal violet (or methyl violet) in 100 mL of glacial acetic acid. The color should be violet. Titrate the solution with 0.1 N perchloric acid in glacial acetic acid to a green color. Not more than 0.10 mL should be required.