

PERMANGANATE FADING FOR ACETIC ACID

UOP Method 969-00

SCOPE

This qualitative method is for determining the fading of a permanganate solution when it is added to concentrated acetic acid. It is a measure of the impurities in acetic acid that are oxidized by potassium permanganate. Permanganate fading indicates the presence of reducing compounds, in particular aldehydes, at the ppm level. This method is applicable to samples from the Acetica acetic acid process.

OUTLINE OF METHOD

The acetic acid sample is placed in a color comparison Nessler tube and is immersed in a constant temperature bath. When the sample reaches the specified temperature, 20°C, a permanganate solution is added. The color of the solution is observed after a 2-hour period. The presence or absence of any remaining pink color is noted.

APPARATUS

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used, unless stated otherwise.

Balance, readability, 1-mg

Bath, jar, Nalgene, 4-1/2 gal., Fisher Scientific, Cat. No. 11-823-16

Bath, cooler, -25 °C to 40 °C, Fisher Scientific, Cat. No. 13-265-172

Bottle, amber, one liter, Fisher Scientific, Cat. No. 03-326-2R

Clamp, water bath, 3 prong, for suspending Nessler tubes, Fisher Scientific, Cat. No. 15-445-70

Clamp, water bath, small, for suspending thermometer, Fisher Scientific, Cat. No. 15-445-80

IT IS THE USER'S RESPONSIBILITY TO ESTABLISH APPROPRIATE PRECAUTIONARY PRACTICES AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE. EFFECTIVE HEALTH AND SAFETY PRACTICES ARE TO BE FOLLOWED WHEN UTILIZING THIS PROCEDURE. FAILURE TO UTILIZE THIS PROCEDURE IN THE MANNER PRESCRIBED HEREIN CAN BE HAZARDOUS. MATERIAL SAFETY DATA SHEETS (MSDS) OR EXPERIMENTAL MATERIAL SAFETY DATA SHEETS (EMSDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).

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Column, chromatographic for filtering 150 mL, Fisher Scientific, Cat. No. K420300-0023

Controller, temperature, Cryotol, for use with cooler bath, Fisher Scientific, Cat. No. 13-265-85

Cylinder, graduated, 500-mL, Fisher Scientific, Cat. No. 08-552G

Flask, volumetric, 1000-mL, Fisher Scientific, Cat. No. 10-210-5G

Glass wool, for filtering, Fisher Scientific, Cat. No. 11-390

Pipet, Mohr, 0.2 mL with 0.01 mL graduation, Fisher Scientific, Cat. No. 13-678-92A

Pipet, volumetric, Class A, 1-, 4- and 20-mL, Fisher Scientific, Cat. Nos. 13-650-2B, 2E, and 2N, respectively

Thermometer, for water bath, Fisher Scientific, Cat. No. 14-983-10B, two required

Timer, clock, Fisher Scientific, Cat. No. 06-660

Tube, color comparison, Nessler with cap stopper, 50-mL, Fisher Scientific, Cat. No. 07-059A

Weighing paper, 3x3 inch, Fisher Scientific, 09-898-12A

REAGENTS AND MATERIALS

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Potassium permanganate, 99+%, Fisher Scientific, Cat. No. P287-500

Potassium permanganate solution, 0.02-*N*. Weigh 0.632 g of potassium permanganate to the nearest 1 mg onto weighing paper. Transfer quantitatively with water into a 1000-mL volumetric flask containing approximately 200 mL of water. Dilute to volume with water and mix well. The solution should be prepared weekly and stored in an amber bottle. If the solution is to be kept for more than a day, filter the solution through a chromatographic column that has been packed with glass wool to remove any MnO₂.

Sulfuric acid, Fisher Scientific, Cat. No. A-300-212

Water, distilled, test as follows to confirm suitability: Use a graduated cylinder to transfer 500 mL of water into a 1000-mL volumetric flask. Add by pipet 1 mL of concentrated H₂SO₄ and 0.15 mL of 0.02-*N* KMnO₄. Mix well. Transfer 50 mL of this solution into a Nessler tube by filling tube to the mark. Allow the solution to stand at room temperature for 2 hours. The pink color must not completely disappear.

PROCEDURE

1. Assemble the water bath as per manufacturer's recommendations.

2. Set the temperature controller to 20 °C.
3. Immerse the thermometer into the water bath and secure with a small clamp. Allow the bath to stabilize at the specified temperature.
4. Pipet 4 mL of the sample and 20 mL of water into a 50-mL Nessler tube and mix. Place the tube in the constant temperature bath and secure with a clamp.
5. Maintain the water level in the bath approximately 25 mm below the top of the Nessler tube.
6. Ensure that the sample in the tube has reached and stabilized at 20°C. Use a thermometer to check the temperature of the solution.
7. Remove the Nessler tube containing the sample from the water bath. Add, by pipet, 1.0 mL of the 0.02-*N* KMnO₄ solution. Stopper the tube, invert to mix the contents and return to the bath.
8. Start the timer.
9. At the end of two hours, remove the tube and note the color by viewing downward through the tube against a white background from which diffused white light is reflected.

REPORT

Report results as “pass” if the pink color persists at the end of 2 hours at 20°C, or “fail” if the pink color has disappeared.

NOTE

Substances reacting with potassium permanganate reduce it to manganese dioxide, which colors the solution yellow. The color of the test solution in the presence of reducing agents changes from pink-orange to yellow-orange. The permanganate fading can be used to judge the presence of oxidizable materials.

PRECISION

No statement is made about the precision of assessing this test method, since the results of the method are pass-fail.

TIME FOR ANALYSIS

The elapsed time for one analysis is 2.3 hours. The labor requirement is 0.3 hours.

SUGGESTED SUPPLIER

Fisher Scientific, 711 Forbes Ave., Pittsburgh, PA 15219 (412-562-8300) www.fishersci.com