

ALCOHOL IMPURITIES IN ACETONE

UOP Method 806-84

SCOPE

This qualitative method is for determining alcoholic impurities in acetone. No color production after 15 minutes indicates that alcohols, if present, are lower than approximately 30 mass-ppm.

OUTLINE OF METHOD

A potassium dichromate solution is added to the acetone sample and the color of the solution is observed over a 15-minute period. The time at which a blue or violet color appears is reported.

APPARATUS

Balance, readability, 0.1-mg

Cylinder, graduated, 100-mL

Flask, volumetric, 100-mL

Pipets, Mohr, 1- and 3-mL

Test tube, Pyrex, 10-mL

REAGENTS AND MATERIALS

All reagents shall conform to specifications established by the Committee on Analytical Reagents of the Americal Chemical Society where such specifications exist, unless otherwise specified. References to water mean distilled or deionized.

Potassium dichromate solution, (Agulhon's reagent). Dissolve $0.5 \text{ g} \pm 1 \text{ mg}$ of potassium dichromate in $22 \pm 0.5 \text{ mL}$ of water in a 100-mL volumetric flask. Dilute to volume with concentrated, 69-71%, nitric acid. Mix well by shaking to effect solution.

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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PROCEDURE

Into a clean dry test tube, pipet 3.00 mL of potassium dichromate solution. Add 1.00 mL of the acetone sample with another pipet, and swirl to mix. Allow the mixture to stand at room temperature for 15 minutes. Periodically observe the solution to determine the time at which a blue or violet color appears, and record this time.

CALCULATIONS

Report, to the nearest 0.25 minutes, the time required for the appearance of a blue or violet color. If no blue or violet color appears after 15 minutes, report "greater than 15 minutes". A color change from orange to yellow or green may be noted but should not be reported.

PRECISION

Based on 6 replicate determinations of a blend containing 35 mass-ppm ethanol in acetone, the estimated standard deviation (esd) for the elapsed time to obtain a color change at the 12.25 minute level was calculated to be 0.4 minutes. Duplicate results by the same operator should not differ by more than 1.3 minutes (95% probability) at the stated level.

TIME FOR ANALYSIS

The elapsed time and labor requirement are identical, 0.33 hour.