

## READILY CARBONIZABLE SUBSTANCES (Colorimetric)

### PRINCIPLE

This method describes a test for various impurities called readily carbonizable substances. It can be used when a spectrometer is not available. The quality of the sample is determined by the color that results from the treatment of the sample being tested with concentrated sulfuric acid in a strictly defined manner. Carbohydrates, such as glucose, are largely responsible for the yellow color developed in citric acid during this test. The carbohydrates dehydrate to form furfural species that can subsequently undergo condensation with citric acid to produce colored condensation products. This colored solution is then visually compared with Matching Fluid K.

### SCOPE

This method applies to citric acid final product samples, both liquids and solids.

### SPECIAL APPARATUS

1. Water or oil bath set at 90°C ( $\pm 1^{\circ}\text{C}$ )
2. Test tubes (22x175 mm as per USP)
3. Test tube rack
4. Analytical balance
5. Vortexing mixer (VWR Vortexer)
6. Timer
7. Sonicator bath
8. Volumetric pipet, 1.0 mL and 10 mL

### REAGENTS

95% minimum Reagent Grade Sulfuric Acid (low Nitrate less than 1 ppm maximum)

Matching Fluid K (See USP reference for preparation.)

**READILY CARBONIZABLE SUBSTANCES — continued****SAFETY PRECAUTIONS**

1. Sulfuric acid is considered to be a particularly hazardous substance by the Hazard Evaluation for Highly Hazardous Substances. Review the Material Safety Data Sheet (MSDS) to familiarize yourself with the hazards associated with this chemical. Some symptoms of overexposure are as follows:
  - inhalation - severe irritation or burns of respiratory system
  - skin/eyes - physical contact may cause severe second and third degree skin burns
2. Mists containing sulfuric acid are known as human carcinogens by IARC.
3. To adequately control the health hazards associated with this chemical:
  - use only in a fume hood (ensure fume hood is working prior to use)
  - use proper gloves (latex gloves for use up to 5 minutes, neoprene/latex gloves for up to 1 1/2 hours, Stanzoil Neoprene for up to 8 hours) and splash-proof safety glasses
  - properly dispose of any generated waste
  - decontaminate designated area upon completion
4. Stanzoil Neoprene gloves, chemical-resistant apron and splash-proof safety goggles must be worn when changing out the concentrated sulfuric acid bottle.
5. Whenever possible, complete the portion of this procedure on which you are working without disruption. Should an interruption occur, immediately remove your gloves to avoid contamination of other objects.
6. The test tube racks which are used have been known to deteriorate over time under the harsh acidic conditions of this test; check the integrity of the rack prior to each use.
7. Perform this procedure carefully, keeping the safety of yourself and others top priority at all times.

**READILY CARBONIZABLE SUBSTANCES — continued****PROCEDURE**

\*\*\*\*\*Familiarize yourself with precautions before proceeding\*\*\*\*\*

Transfer 1.0 g of powdered Anhydrous Citric Acid to a 22- × 175-mm test tube previously rinsed with 10 mL of sulfuric acid TS and allowed to drain for 10 minutes. Add 10 mL of sulfuric acid TS, agitate until solution is complete, and immerse in a water bath at  $90 \pm 1^\circ$  for  $60 \pm 0.5$  minutes, keeping the level of the acid below the level of the water during the entire period (Note 1). Cool the tube in running water, and transfer the acid to a color-comparison tube. This solution is compared to a similar volume of Matching Fluid K (Note 2) in a matching tube. The tubes should be observed vertically against a white background.

**CALCULATION**

Record color darker or lighter than Matching Fluid K.

**NOTES**

1. Both the weight of the sample used and the reaction time are important since the reaction of readily carbonizable substances with sulfuric acid is non-linear. After 1 hour, promptly remove the samples from the  $90^\circ\text{C}$  water bath and cool to quench the reaction. Prolonged reaction times will result in high RCS results.
2. The matching of the sample tube and the tube of Matching Fluid K should take place at  $25^\circ\text{C}$ , since the color of the Matching Fluid K solution is color dependent.

**REFERENCE**

USP 29/NF 24, 2<sup>nd</sup> Supplement, 2006.

**METHOD HISTORY**

Lactic Acid, Readily Carbonizable Substances, (Colorimetric) (L-3), Date of Acceptance 9-08-2006.