

CHLORINATION COLOR OF PHENOL BY SPECTROPHOTOMETRY

UOP Method 803-84

SCOPE

This method is for determining the color of phenol after exposure to chlorine gas as a qualitative measure of certain impurities such as 2,3-benzofuran in phenol. The lower limit of detection is approximately 5 mass-ppm.

OUTLINE

Ten grams of melted phenol is slowly reacted with chlorine until a weight increase of 5.5 - 6.0 g is attained. The maximum absorbance is measured over the range of 450 - 550 nm using a spectrophotometer.

APPARATUS

Adapter, reducing, F joints, top outer 14/20, bottom inner 24/40, SGA Scientific Inc., Cat. No. A-1816, or equivalent

Balance, readability, 0.01-g

Bath, to heat water to 55 C

Beaker, 400-mL

Cells, 1- and 10-mm, glass or silica

Condenser, micro, air, F joints 14/20, 200-mm length, SGA Scientific Inc., Cat. No. JM-3290, or equivalent

Flask, distilling, round bottom, 100-mL, single neck having a side inlet tube (with rubber connector that is included with the flask), and thermometer well, joint F 24/40, SGA Scientific Inc., Cat. No. JF-3230, or equivalent

Flowmeter, soap film, 100-mL, Alltech Associates, Inc., Cat. No. 4045, or equivalent

Pipets, disposable, capillary, Pasteur, 145-mm length, Sargent-Welch Scientific, Cat. No. S-69647-A, or equivalent

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).

Spectrophotometer, capable of measuring absorbance over 450-550 nm range, Beckman, Model 35, or equivalent

Thermometer, -20 to 102 C, ASTM 12-C, Sargent-Welch Scientific, Cat. No. S-80725, or equivalent

Tubing, tygon, 3/16-inch ID, 5/16-inch OD, Sargent-Welch Scientific, Cat. No. S-73651-KC, or equivalent (3 meters required)

Valve, for chlorine gas cylinder, monel, inlet connection CGA-110, Matheson, Model 60L, or equivalent

REAGENTS AND MATERIALS

All reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, when such specifications are available, unless otherwise specified.

Chlorine gas, cylinder, lecture bottle, 99.9% ultra high purity, Matheson, or equivalent.

CAUTION: Use only in a hood or well ventilated area.

PROCEDURE

Warm the phenol sample in a hot water bath at 55 C to obtain a fluid portion. *CAUTION*: Phenol causes burns and is absorbed through the skin. In the event of contact, wash area with large quantities of water. Ingestion or inhalation can cause dizziness, nausea and fainting. Mix well and promptly weigh 10 ± 1 g into the distillation flask. Fit the air condenser to the flask and insert the thermometer into the thermowell. Insert a glass pipet through the rubber connector on the side inlet tube and into the flask so that the tip of the pipet extends below the surface of the phenol. Connect the pipet to the chlorine cylinder with a short length of tygon tubing.

Working in a well ventilated hood, adjust the chlorine flow rate to 40 ± 5 mL/minute as measured with the flow meter and begin to bubble the gas through the melted phenol. After the initial rapid temperature rise, maintain the reaction temperature at 80 ± 3 C by periodically immersing the flask in a beaker of cold water. From time to time remove the chlorine line, condenser and thermometer from the flask, wipe dry and weigh. When a weight increase of 5.5-6.0 g has been achieved, generally between 45-60 minutes, allow the chlorinated phenol to cool to room temperature. Transfer the cooled sample to a 10-mm glass or silica cell and immediately scan the spectrum over the 450-550 nm range using water in the reference cell.

RESULTS

Report the maximum absorbance and the corresponding wave length in the range of 450-550 nm using a 10 mm path length cell. If extreme color degradation causes the spectrophotometer to read off scale, the absorbance can be found by transferring the sample and water reference from the 10-mm to a 1-mm cell and multiplying the resulting absorbance by ten.

PRECISION

Based on 6 replicate determinations of a pure phenol sample, the estimated standard deviation (esd) for maximum absorbance at the 0.145 level in the 450-550 nm range was calculated to be 0.007. Duplicate results by the same operator should not differ by more than 0.03 (95% probability) at the stated level.

TIME FOR ANALYSIS

The elapsed time and labor requirement are identical, 2.5 hours.

SUGGESTED SUPPLIERS

Alltech Associates, Inc., 2051 Waukegan Rd., Deerfield, IL 60015
Beckman Instruments, Inc., 2500 Harbor Blvd., Fullerton, CA 92634
Matheson, P. O. Box 96, Joliet, IL 60434
Sargent-Welch Scientific Co., 7300 N. Linder Ave., Skokie, IL 60077
SGA Scientific Inc., 188 Industrial Dr., Elmhurst, IL 60126