



A Honeywell Company

Ferrox Test for Organic Oxygen Compounds in Propylene Dichloride and Carbon Tetrachloride

UOP Method 712-71 (2006)

Scope

This is a simple test to determine whether organic oxygen compounds are present in propylene dichloride (1,2-dichloropropane) and carbon tetrachloride in concentrations greater or less than 0.1 wt-% organic oxygen, using propylene glycol as the organic oxygen reference. Nitrogen and sulfur compounds interfere, while diaryl ethers give a false negative test. These are not serious limitations, since none of these classes of compounds is likely to be present in commercial propylene dichloride or carbon tetrachloride.

Reference

Davidson, D., *Ind. Eng. Chem., Anal. Ed.*, **12**, 40 (1940)

Outline of Method

“Ferrox” paper, filter paper impregnated with ferric thiocyanate, is brought into contact with the sample. Ferric thiocyanate is an intensely colored compound which is highly soluble in organic oxygen compounds. The color is leached from the paper to an extent depending on the amount and type of oxygen compounds present.

The sample is decanted into small test tubes for visual comparison with a standard solution of propylene glycol in purified propylene dichloride made up to contain 0.1 wt-% organically combined oxygen. Alternatively, the sample may be diluted with benzene and the absorbance at 460 nm measured spectrophotometrically. If the sample is more intensely colored than the standard by either visual or spectrophotometric examination, the sample is considered to contain more than 0.1 wt-% organic oxygen and vice versa.

Apparatus

Balance, analytical

Column, liquid chromatographic. Prepare from a 500-mm length of 17-mm OD glass tubing.

Cuvettes, 10-mm light path, for spectrophotometer, optional, see *Outline of Method* and *Procedure*

Glassware, miscellaneous, normally available in an analytical laboratory

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Oven, for use at 150°C

Pipets, volumetric, 2-mL

Spectrophotometer, capable of measuring absorbance at 460 nm, optional, see *Outline of Method and Procedure*

Test tubes, 10 x 75-mm and 15 x 125-mm

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 exist, unless otherwise specified. References to water mean deionized or distilled water.

Benzene

Ferric chloride

Ferrox paper. Dissolve 1 g of ferric chloride in 10-mL of methanol. Dissolve 1 g of potassium thiocyanate in a separate 10-mL portion of methanol. Combine the solutions, and filter to remove the precipitated potassium chloride. Dip the filter paper in the filtrate and allow it to air dry. More than one dipping may be necessary to produce a product having a greenish sheen. Cut the dried paper into small squares (5-10 mm) and store in a well-stoppered container, protected from lengthy exposure to light. Under such conditions, the ferrox paper may be preserved for months.

Filter paper

Methanol

Organic oxygen standard solution. Use specially purified propylene dichloride (see below) to prepare a solution containing 0.1% organic oxygen (0.238% propylene glycol) by weight. This is the standard solution against which samples of the propylene dichloride are to be measured. In preparing the solution, dilution from higher concentrations is not practical due to the low solubility of propylene glycol in propylene dichloride.

Potassium thiocyanate

Propylene dichloride (1,2-dichloropropane)

Propylene dichloride (1,2-dichloropropane), specially purified. Dry 30 g of silica gel for 24 hours at 150°C. Add this to the chromatographic column in 5-g portions, tapping with a short piece of rubber tubing to settle the column. Percolate 150 mL of propylene dichloride through the column and collect the effluent.

Propylene glycol

Silica gel, 60-200 mesh, chromatographic grade

Water, deionized or distilled

Procedure

Pipet 2 ml of the sample and 2 ml of the standard solution into separate 125-mm test tubes and add a square of the ferrox paper to each tube. Allow these to stand for 10 minutes with occasional swirling and decant the liquids into separate 10 x 75-mm test tubes. Observe the colors from above, looking down the length of the tubes.

For spectrophotometric measurement, pipet 2 mL of the sample into a 125-mm test tube and add a square of ferrox paper. Allow to stand for 10 minutes with occasional swirling, add 1 mL of benzene and mix. Decant the colored solution into a 10-mm cuvette and measure the absorbance at 460 nm using water as the reference liquid. Treat the 0.1 % organic oxygen standard in the same way.

Report whether or not the color of the sample determined by either of these procedures is *darker* than that of the standard.

The same procedure, reagent purification, and preparation of the standard solution is followed where carbon tetrachloride samples, instead of propylene dichloride, are to be tested for the presence of organic oxygen compounds.

Bias

To check the reliability of the test with various organic oxygen-containing compounds, several of these were used to prepare solutions containing 0.1 wt-% organically combined oxygen in propylene dichloride which had not been treated with silica gel. "Blanks" of untreated and gel-treated propylene dichloride were tested along with these solutions by the spectrographic method, as tabulated below.

<u>Compound</u>	<u>Oxygen in Solution, wt-%</u>	<u>Absorbance at 460nm</u>
Isopropyl alcohol	0.091	1.5+
Propylene glycol	0.093	0.52
Acetone	0.104	0.47
Ether	0.106	0.42
Ethylene chlorohydrin	0.094	0.32
Isobutyraldehyde	0.105	0.57
1,2-Dimethoxyethane	0.105	0.17
Propylene dichloride untreated blank	-	0.12
Propylene dichloride blank treated with silica gel	-	0.04

Thus, in the situation calling for 0.1 wt-% oxygen in propylene glycol or carbon tetrachloride, it can be seen that the choice of propylene glycol will permit only about one-third the specified amount of simple alcohols like isopropyl alcohol, but permits more than 0.1 wt -% oxygen in the form of polyfunctional compounds like chlorohydrins and dialkoxyalkanes.