



Standard Test Method for Determination of Carbonyls In C₄ Hydrocarbons¹

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1. Scope

1.1 This test method covers the determination of carbonyls (ketones and aldehydes) in C₄ hydrocarbons. This test method was tested on polymerization-grade 1,3-butadiene.

1.2 The applicable range for this test method is 0 to 50 mg/kg carbonyls calculated as acetaldehyde.

1.3 Other C₄ hydrocarbons and their mixtures besides polymerization-grade 1,3-butadiene could be tested using this same test method. However, the precision section of this test method covers only carbonyls in applicable range as listed in 1.2, as found in polymerization-grade 1,3-butadiene.

1.4 The values stated in SI units are to be regarded as the standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 484 Specification for Hydrocarbon Dry Cleaning Solvents²

D 1193 Specification for Reagent Water³

E 1 Specification for ASTM Thermometers⁴

3. Summary of Test Method

3.1 A measured amount of sample is added to an alcoholic hydroxylamine hydrochloride solution that has been adjusted to a given coloration using either alcoholic acid or base. The carbonyls will react with the hydroxylamine hydrochloride releasing an equivalent amount of hydrochloric acid which is then back-titrated to the original coloration. A blank containing only methanol and sample is titrated and the sample's results are calculated using the blank adjustment. Results are reported as milligrams per kilogram carbonyls as acetaldehyde.

4. Significance and Use

4.1 The determination of the carbonyl content of polymerization-grade 1,3-butadiene is necessary, since in some polymerization reactions, the presence of carbonyls in excess over some specified amount can have a deleterious effect upon the polymer properties or the reaction itself, or both.

5. Apparatus

5.1 *Bunsen Valves*—A device constructed so that when used with an Erlenmeyer flask, the sample vapors can exit the flask while protecting the flask's liquid contents. See Fig. 1 for details.

5.2 *Cooling Coil*—Prepare a cooling coil by winding about 10 to 15 cm of seamless copper tubing (about 6-mm diameter) on a short length of pipe (about 1.5 to 2.0-cm diameter), allowing sufficient length of tubing at the end of the coil to connect it to the sample source. Attach a valve at a point that would not extend more than 8 cm above the surface of the cooling bath liquid. To the valve, attach a 6 to 8 cm length piece of tubing bent downward so that the hydrocarbon liquid can be directed into the receiving container.

5.3 *Dewar Flask*—The Dewar flask must be of sufficient volume to completely immerse the main portion of the cooling coil except for the extremities necessary for receiving and delivering the sample through the coil.

5.4 *Erlenmeyer Flasks*. 250-mL capacity.

5.5 *Volumetric Flasks*. 1-L capacity. These flasks should be Class A glassware.

5.6 *Graduated Cylinders*—100-mL capacity, glass cylinders, graduated in 1 or 2-mL divisions.

5.7 *Microburets*. 2.00 or 5.00-mL capacity. The microburets should be Class A glassware with 0.01 or 0.02-mL divisions or less. It is advisable to have the buret's tip end equipped with a syringe needle to dispense very small drops of titrant.

5.8 *Sample Cylinders*—These should be of sufficient volume to give the required amount of sample for testing. Stainless steel cylinders equipped with needle valves should be used. It is suggested that a 500-mL-capacity cylinder be the minimum size to be used for butadiene.

5.9 *Thermometer*—For observing temperatures below -45°C . The Low Cloud and Pour Point Thermometer, conforming to the requirements for ASTM Thermometer 6C, as prescribed in Specification E 1, is satisfactory. Thermometer 6C has a range from -80 to $+20^{\circ}\text{C}$.

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.D0.04 on C₄ Hydrocarbons.

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² Discontinued; see 1983 Annual Book of ASTM Standards, Vol. 05.01.

³ Annual Book of ASTM Standards, Vol 11.01

⁴ Annual Book of ASTM Standards, Vol 14.03.