Designation: D5303 – 92 (Reapproved 2012)

Standard Test Method for Trace Carbonyl Sulfide in Propylene by Gas Chromatography[1]

This standard is issued under the fixed designation D5303; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of traces of carbonyl sulfide (COS) in propylene. It is applicable to COS concentrations from 0.5 to 4.0 mg/kg (parts per million by mass). See Note 1.

NOTE 1—The lower limit of this test method is believed to be below 0.1 mg/kg, depending on sample size and sensitivity of the instrumentation being used. However, the cooperative testing program was conducted in the 0.5 to 4.0 range due to limitations in preparing commercial test mixtures.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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.* Specific hazards statements are given in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3609 Practice for Calibration Techniques Using Permeation Tubes

D4468 Test Method for Total Sulfur in Gaseous Fuels by Hydrogenolysis and Rateometric Colorimetry

E840 Practice for Using Flame Photometric Detectors in Gas Chromatography

3. Summary of Test Method

3.1 A procedure is given for removing a sample from the sample cylinder, separating COS from propylene, detecting COS, calibrating the detector, quantitating COS content in the sample, and assaying the gas standard. General comments and recommended techniques are given.

3.2 A relatively large volume of sample is injected into a gas chromatograph having a single packed column, operated isothermally at 10 to 50°C, that separates COS from propylene. COS is detected with a flame photometric detector.

3.3 Calibration data, based on peak areas, are obtained using a known gas standard blend of COS in the range expected for the sample. The COS peak area in the sample is measured and the concentration of COS calculated.

3.4 The COS gas standard blend is assayed prior to use for calibration.

4. Significance and Use

4.1 In processes producing propylene, COS usually remains with the C_3 hydrocarbons and must be removed, since it affects product quality. COS acts as a poison to commercial polymerization catalysts, resulting in deactivation and costly process downtime.

4.2 Accurate gas chromatographic determination of trace COS in propylene involves unique analytical problems because of the chemical nature of COS and idiosyncracies of trace level analyses. These problems result from the reactive and absorptive nature of COS, the low concentration levels being measured, the type of detector needed, and the interferences from the propylene sample matrix. This test method addresses these analytical problems and ways to properly handle them to assure accurate and precise analyses.

4.3 This test method provides a basis for agreement between two laboratories when the determination of trace COS in propylene is important. The test method permits several calibration techniques. For best agreement between two labs, it is recommended that they use the same calibration technique.

5. Interferences

5.1 Hydrogen sulfide (H_2S) or sulfur dioxide (SO_2) can be present in the propylene and must be separated from COS. (See Note 2.)

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.D0.03 on Propylene.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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