



Standard Test Method for Ethylene, Other Hydrocarbons, and Carbon Dioxide in High-Purity Ethylene by Gas Chromatography¹

This standard is issued under the fixed designation D2505; 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 carbon dioxide, methane, ethane, acetylene, and other hydrocarbons in high-purity ethylene. Hydrogen, nitrogen, oxygen, and carbon monoxide are determined in accordance with Test Method [D2504](#). The percent ethylene is obtained by subtracting the sum of the percentages of the hydrocarbon and nonhydrocarbon impurities from 100. The method is applicable over the range of impurities from 1 to 500 parts per million volume (ppmV).

1.2 *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.* For some specific hazard statements, see Section [6](#).

1.3 The values stated in acceptable metric units are to be regarded as the standard. The values in parentheses are for information only.

2. Referenced Documents

2.1 ASTM Standards:²

[D2504](#) Test Method for Noncondensable Gases in C₂ and Lighter Hydrocarbon Products by Gas Chromatography

[D4051](#) Practice for Preparation of Low-Pressure Gas Blends

[E260](#) Practice for Packed Column Gas Chromatography

[F307](#) Practice for Sampling Pressurized Gas for Gas Analysis

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.D0.02](#) on Ethylene.

Current edition approved May 1, 2010. Published May 2010. Originally approved in 1966. Last previous edition approved in 2004 as D2505 – 88 (2004)^{E1}. DOI: 10.1520/D2505-88R10.

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.

3. Summary of Test Method

3.1 The sample is separated in a gas chromatograph system utilizing four different packed chromatographic columns with helium as the carrier gas. Methane and ethane are determined by using a silica gel column. Propylene and heavier hydrocarbons are determined using a hexamethylphosphoramide (HMPA) column. Acetylene is determined by using, in series, a hexadecane column and a squalane column. Carbon dioxide is determined using a column packed with activated charcoal impregnated with a solution of silver nitrate in β,β'-oxydipropionitrile. Columns other than those mentioned above may be satisfactory (see [5.3](#)). Calibration data are obtained using standard samples containing the impurities, carbon dioxide, methane, and ethane in the range expected to be encountered. Calibration data for acetylene are obtained assuming that acetylene has the same peak area response on a weight basis as methane. The acetylene content in a sample is calculated on the basis of the ratio of peak area of the acetylene peak to the peak area of a known amount of methane. Calculations for carbon dioxide, methane, and ethane are carried out by the peak-height measurement method.

4. Significance and Use

4.1 High-purity ethylene is required as a feedstock for some manufacturing processes, and the presence of trace amounts of carbon dioxide and some hydrocarbons can have deleterious effects. This method is suitable for setting specifications, for use as an internal quality control tool and for use in development or research work.

5. Apparatus

5.1 Any chromatographic instrument with an overall sensitivity sufficient to detect 2 ppmV or less of the compounds listed with a peak height of at least 2 mm without loss of resolution.

5.2 *Detectors—Thermal Conductivity*—If a methanation reactor is used, a flame ionization detector is also required. To determine carbon dioxide with a flame ionization detector, a methanation reactor must be inserted between the column and

the detector and hydrogen added as a reduction gas (see Test Method D2504, Appendix X1, Preparation of Methanation Reactor).

5.3 Column—Any column or set of columns can be used that separates carbon dioxide, methane, acetylene and C₃ and heavier compounds. There may be tailing of the ethylene peak but do not use any condition such that the depth of the valleys ahead of the trace peak is less than 50 % of the trace peak height. (See Fig. 1 for example.)

5.4 Recorder—A recorder with a full-scale response of 2 s or less and a maximum rate of noise of ± 0.3 % of full scale.

5.5 Gas-Blending Apparatus—A typical gas-blending apparatus is shown in Fig. 2. A high-pressure manifold equipped with a gage capable of accurately measuring ethylene pressures up to 3.4 MN/m² gage (500 psig) is required. Other types of gas-blending equipment, such as described in Practice D4051, can be used.

NOTE 1— Practice E260 contains information that will be helpful to those using this method.

6. Reagents and Materials

6.1 Copper or Aluminum, or Stainless Steel Tubing, 6.4-mm (1/4-in.) outside diameter, and nylon tubing, 3.2-mm (1/8-in.) outside diameter.

6.2 Solid Supports—Crushed firebrick or calcined diatomaceous earth, such as Chromosorb P,³ 35 to 80-mesh and 80 to 100-mesh. Other supporting materials or mesh sieves can be satisfactory.

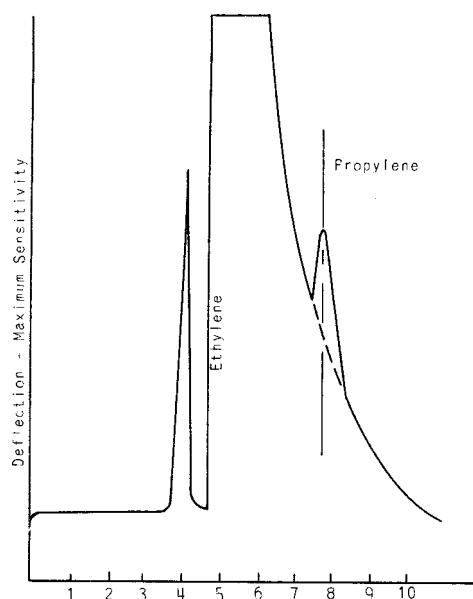


FIG. 1 Typical Chromatogram for Propylene

³ The sole source of supply of the apparatus is available from the Celite Division, Johns Mansville Co., New York, NY. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee¹, which you may attend.

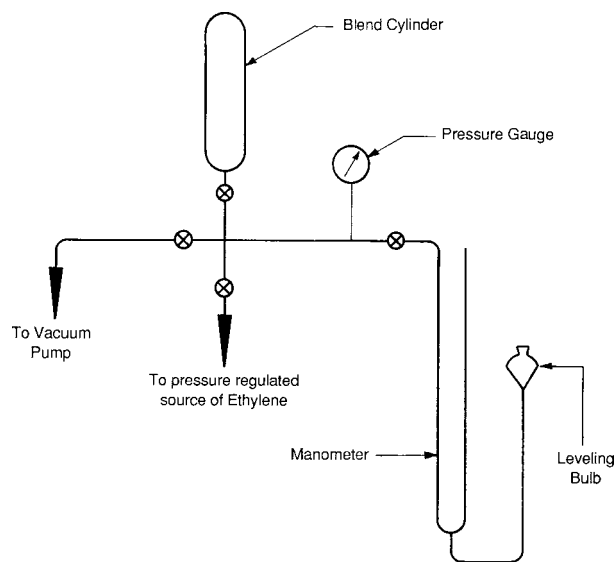


FIG. 2 Gas-Blending Manifold

6.3 Active Solids—Activated carbon, 30 to 40-mesh,⁴ silica gel, 100 to 200-mesh.⁵ Other sizes may be satisfactory.

6.4 Liquid Phases—Hexamethylphosphoramide (HMPA)⁶, hexadecane,⁶ Squalene,⁶ silver nitrate, and β,β' -oxydipropionitrile.⁷ Other liquid phases may be satisfactory. (**Warning**—Combustible solvents. See A1.7.) (**Warning**—HMPA may be harmful if inhaled. Causes irritation. A potential carcinogen (lungs). See A1.5.)

6.5 Helium. (**Warning**—Compressed Gas, Hazardous Pressure. See A1.2.)

6.6 Hydrogen. (**Warning**—Flammable Gas, Hazardous Pressure. See A1.6.)

6.7 Acetone. (**Warning**—Extremely Flammable. See A1.1.)

6.8 Gases for Calibration—Pure or research grade carbon dioxide, methane, ethane, acetylene, ethylene, propane, and propylene. Certified calibration blends are commercially available from numerous sources and may be used. (**Warning**—Flammable Gases, Hazardous Pressure. See A1.2 and A1.3.)

6.9 Methanol. (**Warning**—Flammable. Vapor Harmful. See A1.4.)

⁴ A fraction sieved in the laboratory to 30 to 40 mesh from medium activity charcoal, 20 to 60 mesh, sold by Central Scientific Co., 1700 Irving Park Road, Chicago, IL 60613, has been found satisfactory for this purpose. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee¹, which you may attend.

⁵ The sole source of supply of the apparatus known to the committee at this time is Silica gel Code 923 available from the Davison Chemical Co., Baltimore, Md. 21203. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee¹, which you may attend.

⁶ The sole source of supply of the apparatus known to the committee at this time is available from the Fisher Scientific Co., St. Louis, MO. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee¹, which you may attend.

⁷ β,β' -oxydipropionitrile, sold by Distillation Products Industries, Division of Eastman Kodak Co., Rochester, NY, has been found to be satisfactory. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee¹, which you may attend.

NOTE 2—The use of copper tubing is not recommended with samples containing acetylene as this could lead to the formation of potentially explosive copper acetylide.

7. Sampling

7.1 Samples should be supplied to the laboratory in high pressure sample cylinders, obtained using the procedures described in Practice F307, or similar methods.

8. Preparation of Apparatus

8.1 *Silica Gel Column*—Dry the silica gel in an oven at 204°C (400°F) for 3 h, cool in a desiccator, and store in screw-cap bottles. Pour the activated silica gel into a 0.9-m (3-ft) length of 6.4-mm (¼-in.) outside diameter copper or aluminum tubing plugged with glass wool at one end. Tap or vibrate the tube while adding the silica gel to ensure uniform packing and plug the top end with glass wool. Shape the column to fit into the chromatograph.

8.2 *Silver Nitrate— β,β' -Oxydipropionitrile—Activated Carbon Column*—Weigh 10 g of β,β' -oxydipropionitrile into a brown 125-mL (4-oz) bottle. Add 5 g of silver nitrate (AgNO_3) crystals. With occasional shaking, dissolve as much AgNO_3 as possible, and allow the bottle to stand overnight to ensure saturation. Prepare this solution fresh, as required. Without disturbing the crystals at the bottom of the bottle, weigh 2.5 g of supernatant AgNO_3 solution into a 250-mL beaker and add 50 mL of methanol. While stirring this mixture, slowly add 22.5 g of activated carbon. Place the beaker on a steam bath to evaporate the methanol. When the impregnated activated carbon appears to be dry, remove the beaker from the steam bath and finish drying in an oven at 100 to 110°C for 2 h. Plug one end of a 4-ft (1.2-m) length of 6.4-mm (¼-in.) outside diameter aluminum or stainless steel tubing with glass wool. Hold the tubing vertically with the plugged end down and pour freshly dried column packing into it, vibrating the column during filling to ensure uniform packing. Plug the top end with glass wool and shape the tubing so that it may be mounted conveniently in the chromatograph.

8.3 *Hexamethylphosphoramide Column (HMPA)*—Dry the 35 to 80-mesh inert support at 204°C (400°F). Weigh 75 g into a wide-mouth 500-mL (16-oz) bottle. Add 15 g of HMPA to the inert support and shake and roll the mixture until the support appears to be uniformly wet with the HMPA. Pour the packing into a 6-m (20-ft) length of 6.4-mm (¼-in.) outside diameter copper or aluminum tubing plugged at one end with glass wool. Vibrate the tubing while filling to ensure more uniform packing. Plug the top end of the column with glass wool and shape the column to fit into the chromatograph.

8.4 *Hexadecane-Squalane Column*—Dissolve 30 g of hexadecane into approximately 100 mL of acetone. Add 70 g of 80 to 100-mesh inert support. Mix thoroughly and pour the mixture into an open pan for drying. The slurry should be stirred during drying to ensure uniform distribution. When the acetone has evaporated, add a portion of the packing to a 7-m (25-ft) length of 3.2-mm (⅛-in.) outside diameter nylon tubing which has been plugged at one end with glass wool. Vibrate the column while filling to ensure more uniform packing. Fill the column with packing to only 4 m (15 ft) of the length of the column. Fill the remainder of the column with squalane

packing prepared in the same manner as the hexadecane packing. Plug the open end of the tubing with glass wool and shape the column to fit into the chromatograph with the hexadecane portion of the column at the front end of the column. The column shall be purged under test conditions (no sample added) until a constant baseline is obtained.

NOTE 3—Columns made with liquid phases listed above were used satisfactorily in cooperative work. Other columns may be used (see 5.3).

9. Calibration

9.1 Preparation of Standard Mixtures:

9.1.1 *Preparation of Concentrate*—Prepare a concentrate of the impurities expected to be encountered. A certified calibration blend containing the expected impurities can be obtained and used as the concentrate. An example of a satisfactory concentrate is given in Table 1. The concentrate can be prepared using the gas blending manifold as shown in Fig. 2 or using a similar apparatus as follows: Evacuate the apparatus and add the components in the order of increasing vapor pressure; that is, propylene, carbon dioxide, ethane and methane. Record the increase in pressure on the manometer as each component is added. Close the reservoir and evacuate the manometer after each addition.

9.1.2 *Dilution of Concentrate*—Dilute the concentrate with high-purity ethylene in a ratio of approximately 1:4000. This can be done by adding the calculated amount of the concentrate and high purity ethylene to an evacuated cylinder using the gas-blending apparatus (Fig. 2). Use a source of high-pressure, high-purity ethylene equipped with a needle valve and a pressure gage capable of accurately measuring the pressure of the blend as the ethylene is added to the cylinder containing the concentrate. Add the calculated amount of ethylene; warm one end of the cylinder to ensure mixing of the blend. Allow the temperature to reach equilibrium before recording the final pressure on the cylinder. Prepare at least three calibration samples containing the compounds to be determined over the range of concentration desired in the products to be analyzed.

9.2 *Calculation of Composition of Standard Mixtures*—Calculate the exact ratio of the concentrate dilution with ethylene by correcting the pressure of the ethylene added for the compressibility of ethylene (Table 2). Multiply the dilution ratio or factor by the percentage of each component present in the original concentrate (Table 1). These calculations give the amount of each component that has been added to the high-purity ethylene blend stock. The actual composition of the final blend must be ascertained by making corrections for the impurities present in the high-purity ethylene used for the blend stock. The amount of correction is determined by making chromatograph runs on the high-purity ethylene and measuring the peak heights of the impurities. These peak heights will be

TABLE 1 Suggested Composition of a Concentrate of Impurities Used in Preparing Standard Mixtures for Calibration Purposes

Component	Percent
Carbon dioxide	10
Methane	45
Ethane	25
Propylene	20

TABLE 2 Supercompressibility of Ethylene

NOTE—The trace component, A, in parts per million volume of finished blend, is determined as follows:

$$A = [(1\ 000\ 000 \times Z_b \times P_a)/(Z_a \times P_f)] + B$$

where:

Z_a = value of Z for observed partial pressure and temperature of the trace component added,

P_a = partial pressure of trace component, psia (mm Hg $\times$ 0.01934),

Z_b = appropriate value of Z for final observed temperature and pressure of ethylene blend,

P_f = final observed pressure (psia) at observed temperature, and

B = concentration, mols per million of trace component present in diluent ethylene.

For synthetic pressures below 200 psia (1380 kN/m²), Z usage is not significant.

Pressure, psia (kN/m ² absolute)	Values of Z					
	15°C	20°C	25°C	30°C	35°C	40°C
15 (103)	0.98	0.99	0.99	0.99	0.99	0.99
100 (690)	0.95	0.96	0.96	0.96	0.96	0.97
200 (1380)	0.90	0.91	0.92	0.92	0.92	0.93
300 (2068)	0.85	0.86	0.87	0.88	0.89	0.89

used in adjusting the calibration factors described in 9.3. Since peak height is very sensitive to changes in conditions, it is extremely important in correlating peak heights obtained in making calibrations, calibration adjustments, and final impurity determinations that these values be obtained under the same GLC column operating conditions in all cases.

9.3 Determination of Calibration Factors—Chromatograph the standard blend and the high-purity ethylene blend stock by each of the procedures given in Section 10.

9.3.1 Calculate calibration factors for carbon dioxide, methane, ethane, propylene, and heavier hydrocarbons as follows:

$$F = C/(S - B) \quad (1)$$

where:

F = mol percent per unit of peak height,

C = concentration of component added to the high-purity ethylene blend stock, mol %,

S = mm peak height of component in standard mixture, and

B = mm peak height of component in high-purity ethylene blend stock, mm.

9.3.2 Calculate calibration factor for acetylene as follows:

$$Fa = C/(Sa - Ba) \quad (2)$$

where:

Fa = weight percent per unit area,

C = concentration of methane added to the high-purity ethylene blend stock in weight percent,

Sa = area of methane peak in standard mixture, and

Ba = area of methane peak in high-purity ethylene blend stock.

10. Procedure

10.1 Methane and Ethane—Typical operating conditions for the analyses for methane and ethane are summarized in Table 3. Slowly flush the sample to be analyzed through the gas sample valve on the chromatograph until all extraneous vapor has been purged from the sample loop. Turn the gas valve to introduce the sample into the carrier gas stream. Record the deflection of each component peak at the minimum attenuation or greatest sensitivity for maximum peak height. Fig. 3 shows a typical chromatogram obtained with the procedure and operating conditions as outlined. Measure the height of each peak from the baseline in millimetres. Both peak height and peak area need to be measured for methane since the area will be used for preparation of an acetylene calibration curve.

10.2 Carbon Dioxide—Typical operating conditions for the analysis for carbon dioxide are given in Table 3. Flush the sample to be analyzed through the gas sample valve on the chromatograph until all extraneous vapor has been purged from the sample loop. Turn the gas valve to introduce the sample into the carrier gas stream. Record the carbon dioxide peak at the greatest sensitivity for maximum peak height. Measure the height of each peak from the baseline in millimetres.

NOTE 4—The elution order for this column is as follows:

Material	Approximate Time, min
Air	2
Methane	3
Carbon dioxide	7
Ethylene	11

10.3 Propylene and Heavier—Typical operating conditions for the procedure for propylene and heavier components are given in Table 3. Flush the sample to be analyzed through the

TABLE 3 Operating Conditions

	Methane and Ethane	Carbon Dioxide	Propylene and Heavier	Acetylene
Column packing	silica gel	AgNO ₃ -oxydipropionitrile on support	HPMA on support	hexadecane-squalane on support
Column dimensions	0.9 m (3 ft) by 6.4 mm (¼ in.)	1.2 m (4 ft) by 6.4 mm (¼ in.)	6 m (20 ft) by 6.4 mm (¼ in.)	6 m (25 ft) by 3.2 mm (⅛ in.)
Sample volume	1 mL	25 mL	1 mL	0.25 mL
Carrier gas	helium	helium	helium	helium
Carrier pressure	20 lb	20 lb	10 lb	
Temperature	50°C	35°C	30°C	ambient
Detector	thermal conductivity	thermal conductivity	thermal conductivity	hydrogen flame ionization

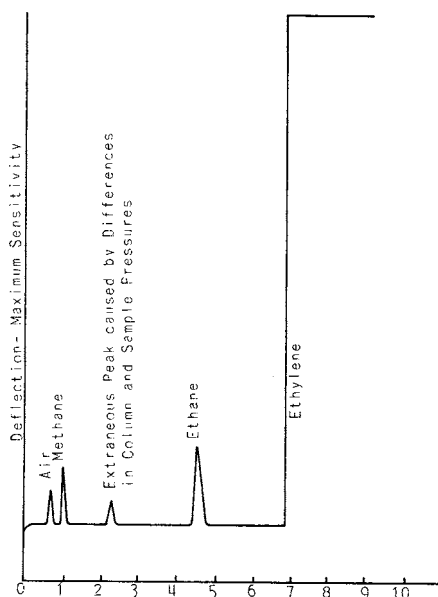


FIG. 3 Typical Chromatogram for Air, Methane, and Ethane

gas sample valve on the chromatograph until all the extraneous vapor has been purged from the sample loop. Turn the gas valve to introduce the sample into the carrier stream. Record the peaks of each component at maximum sensitivity for maximum peak height. Fig. 1 shows a typical chromatograph obtained with the procedure and conditions described. Measure the height of each peak from the baseline as formed by the tailing of the ethylene peak.

10.4 Acetylene—Typical operating conditions for the analysis for acetylene are given in Table 3. Flush the sample to be analyzed through the gas sample valve on the chromatograph until all extraneous vapor has been purged from the sample loop. Operate the gas valve to introduce the sample into the carrier gas stream. Measure the peak areas of the methane and acetylene peaks. Fig. 4 shows a typical chromatograph obtained with the procedure and conditions outlined.

11. Calculation

11.1 Carbon Dioxide, Methane, Ethane, Propylene, and Heavier—Calculate the mol percent of each component present in the sample as follows:

$$C = D \times F \quad (3)$$

where:

C = concentration of component, mol% ,

D = peak height of the component, mm, and

F = calibration factor of component as determined in 9.3.1.

11.2 Acetylene—Calculate the mol percent of acetylene in the sample as follows:

$$C = A \times Fa \times (28/26) \quad (4)$$

where:

C = concentration of acetylene, mol% ,

A = area of the acetylene peak, and

Fa = area calibration factor as determined in 9.3.2.

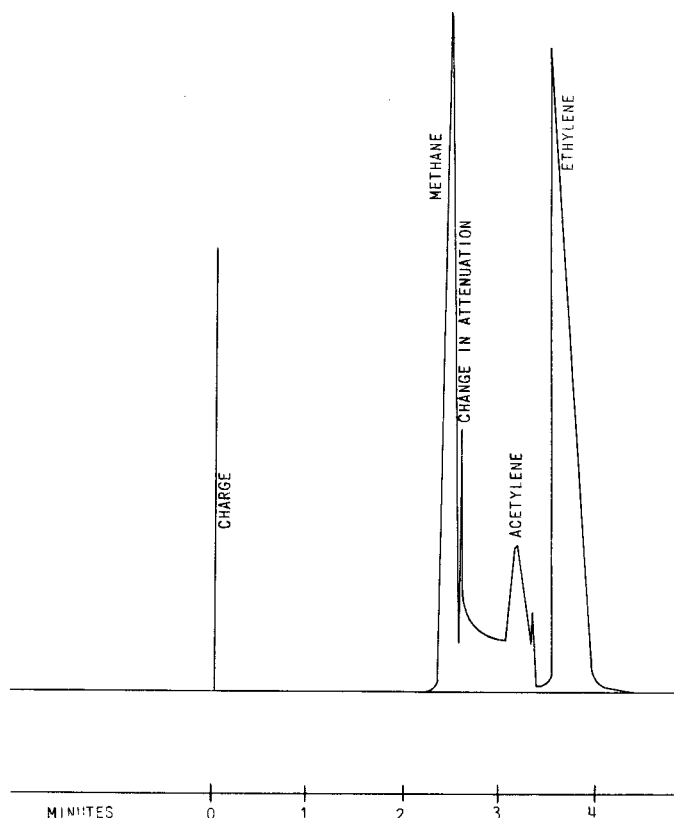


FIG. 4 Typical Chromatogram for Acetylene as a Trace Impurity in Ethylene

11.3 Ethylene—Calculate the mol percent of ethylene in the sample by adding the concentration of all impurities and subtract from 100.

12. Precision and Bias

12.1 The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

12.1.1 Repeatability—The difference between successive test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, and in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

Component	Range	Repeatability
Ethylene	99.80–99.99 mol %	0.006 mol %
Methane	1–150 ppmV	3 ppmV
Ethane	1–1500 ppmV	43 ppmV
Propylene	1–15 ppmV	3 ppmV
Propane	1–15 ppmV	2 ppmV
Acetylene	1–20 ppmV	1 ppmV
Carbon dioxide	1–10 ppmV	1 ppmV

12.1.2 Reproducibility—The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material would, in the long run, and in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

Component	Range	Reproducibility
Ethylene	99.80–99.99 mol %	0.1 mol %
Methane	0–150 ppmV	34 ppmV



Ethane	0–500 ppmV	72 ppmV
Ethane	500–1500 ppmV	290 ppmV
Propylene	0–15 ppmV	11 ppmV
Propane	0–15 ppmV	7 ppmV
Acetylene	0–20 ppmV	6 ppmV
Carbon dioxide	0–10 ppmV	4 ppmV

12.2 *Bias*—The bias of the procedure in this test method has not yet been determined but is now under consideration by the responsible subcommittee.

13. Keywords

13.1 carbon dioxide; ethane; ethylene; gas chromatography; hydrocarbons; methane

ANNEX

(Mandatory Information)

A1. WARNING STATEMENTS

A1.1 Acetone

Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Vapors may spread long distances and ignite explosively.
Avoid build-up of vapors and eliminate all sources of ignition, especially non-explosion proof electrical apparatus and heaters.
Avoid prolonged breathing of vapor or spray mist.
Avoid contact with eyes and skin.

A1.2 Compressed Gas

Keep container closed.
Use with adequate ventilation.
Do not enter storage areas unless adequately ventilated.
Always use a pressure regulator. Release regulator tension before opening cylinder.
Do not transfer to cylinder other than one in which gas is received. Do not mix gases in cylinders.
Do not drop cylinder. Make sure cylinder is supported at all times.
Stand away from cylinder outlet when opening cylinder valve.
Keep cylinder out of sun and away from heat.
Keep cylinder from corrosive environment.
Do not use cylinder without label.
Do not use dented or damaged cylinder.
For technical use only. Do not use for inhalation purposes.

A1.3 Flammable Gas

Keep away from heat, sparks and open flame.
Use with adequate ventilation.
Never drop cylinder. Make sure cylinder is supported at all times.
Keep cylinder out of sun and away from heat.
Always use a pressure regulator. Release regulator tension before opening cylinder.
Do not transfer cylinder contents to another cylinder. Do not mix gases in cylinder.
Keep cylinder valve closed when not in use.
Do not inhale.
Do not enter storage areas unless adequately ventilated.

Stand away from cylinder outlet when opening cylinder valve. Keep cylinder from corrosive environment.
Do not use cylinder without label.
Do not use dented or damaged cylinder.
For technical use only. Do not inhale.

A1.4 Methanol

May be fatal or cause blindness if swallowed or inhaled.
Cannot be made non-poisonous.
Keep away from heat, sparks, and open flame.
Keep container closed.
Avoid contact with eyes and skin.
Avoid breathing of vapor or spray mist.
Use with adequate ventilation.
Do not take internally.

A1.5 Hexamethyl Phosphoramide

A potential carcinogen (lung).
Avoid breathing vapor or mist.
Avoid contact with skin, eyes, and clothing.
Use with adequate ventilation.
Keep container closed when not in use.
Wash thoroughly after handling.

A1.6 Hydrogen

Keep away from heat, sparks, and open flame.
Use with adequate ventilation.
Never drop cylinder. Make sure cylinder is supported at all times.
Keep cylinder out of sun and away from heat.
Always use a pressure regulator. Release regulator tension before opening cylinder.
Do not transfer cylinder contents to another cylinder. Do not mix gases in cylinder.
Keep cylinder valve closed when not in use.
Do not inhale.
Do not enter storage areas unless adequately ventilated.
Stand away from cylinder outlet when opening cylinder valve.
Keep cylinder from corrosive environment.

A1.7 n-Hexadecane

Keep away from heat, sparks, and open flame.
Keep container closed.

Use with adequate ventilation.
Avoid breathing vapor or spray mist.
Avoid prolonged or repeated contact with skin.

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