



Standard Guide for Analysis of Propylene Concentrates¹

This standard is issued under the fixed designation D 5273; 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 guide lists the major grades of propylene concentrates produced in North America. It includes possible components and test methods, both ASTM and other, either actually used, or believed to be in use, to test for these properties. This guide is not intended to be used or construed as a set of specifications for any grade of propylene concentrate.

1.2 The values stated in SI units are to be regarded as the 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.*

2. Referenced Documents

2.1 ASTM Standards:

- D 2163 Test Method for Analysis of Liquefied Petroleum (LP) Gases and Propene Concentrates by Gas Chromatography²
- D 2384 Test Methods for Traces of Volatile Chlorides in Butane-Butene Mixtures²
- D 2504 Test Method for Noncondensable Gases in C₂ and Lighter Hydrocarbon Products by Gas Chromatography²
- D 2505 Test Method for Ethylene, Other Hydrocarbons, and Carbon Dioxide in High-Purity Ethylene by Gas Chromatography²
- D 2712 Test Method for Hydrocarbon Traces in Propylene Concentrates by Gas Chromatography²
- D 3227 Test Method for Thiol Mercaptan Sulfur in Gasoline, Kerosine, Aviation Turbine, and Distillate Fuels (Potentiometric Method)²
- D 3246 Test Method for Sulfur in Petroleum Gas by Oxidative Microcoulometry³
- D 3700 Practice for Obtaining LPG Fluid Samples Using a Floating Piston Cylinder³

D 4178 Practice for Calibrating Moisture Analyzers³

D 4468 Test Method for Total Sulfur in Gaseous Fuels by Hydrogenolysis and Rateometric Colorimetry⁴

D 4629 Test Method for Trace Nitrogen in Liquid Petroleum Hydrocarbons by Syringe/Inlet Oxidative Combustion and Chemiluminescence Detection³

D 4864 Test Method for Determination of Traces of Methanol in Propylene Concentrates by Gas Chromatography³

3. Terminology

3.1 Definitions:

3.1.1 *outgassing, n*—practice of removing a portion of liquid contents from a conventional sampling cylinder after filling to provide expansion room.

3.1.2 *propylene concentrate, n*—hydrocarbon product containing more than 50 % propylene.

3.1.2.1 *Discussion*—Grades of propylene concentrates listed in this guide are: polymer, 99.0 % minimum propylene content; chemical, 92.0 %; and refinery, 60 %.

3.2 Symbols:

3.2.1 *AgDDC, n*—silver diethyldithiocarbamate.

3.2.2 *GC, n*—gas chromatograph.

3.2.3 *GC-AED, n*—gas chromatography atomic emission detector.

3.2.4 *GC-ECD, n*—gas chromatography electron capture detector.

3.2.5 *GC-FPD, n*—gas chromatography flame photometric detector.

3.2.6 *GC-PID, n*—gas chromatography photoionization detector.

3.2.7 *GC-SCD, n*—gas chromatography sulfur chemiluminescent detector.

3.2.8 *IC, n*—ion chromatography.

3.2.9 *ICP-MS, n*—inductively coupled plasma-mass spectrometry.

3.2.10 *LPG or LP gases, n*—liquefied petroleum gas.

4. Significance and Use

4.1 This guide is intended to provide information on the likely composition of propylene concentrates and on probable ways to test them. Since there are currently no ASTM test

¹ This guide is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.D0.03 on Propylene.

Current edition approved Dec. 10, 2002. Published March 2003. Originally approved in 1992. Last previous edition approved in 1997 as D 5273 – 92(1997).

² *Annual Book of ASTM Standards*, Vol 05.01.

³ *Annual Book of ASTM Standards*, Vol 05.02.

⁴ *Annual Book of ASTM Standards*, Vol 05.06.

methods for determining all components of interest, this guide provides information on other potentially available test methods.

4.2 Although this guide is not to be used for specifications, it can provide a starting point for parties to develop mutually agreed upon specifications which meet their respective requirements. It can also be used as a starting point in finding suitable test methods for determining various components of propylene.

5. Sampling

5.1 *General*—Sample propylene concentrates are to be analyzed for trace components by a technique that minimizes or eliminates losses of light components and concentration of heavy ones. The sections below list some different sampling methods and principles. However, it is not the intent of this guide to list procedures that are applicable to all sampling situations. It is strongly recommended that samples be obtained under the supervision of a person with wide knowledge and experience in sampling olefinic liquified petroleum gases. Also, even though this guide does not address the location of a sampling point in a line or vessel, the importance of the proper sampling location cannot be over-emphasized.

5.2 *Floating Piston Cylinder*—Test Method D 3700 meets the criterion of minimizing or eliminating loss of light compounds and concentration of heavy ones. However, some labs have safety codes preventing use of rupture-disc piston containers. Alternative procedures must be used in these labs.

5.3 *Conventional Outgassing Method*—The widely used outgassing technique (that is, the practice of removing a portion of the fluid contents from a conventional sampling cylinder after

filling in order to provide expansion room) causes a loss of light components into the vapor space. Subsequent handling to recapture these light ends in the liquid phases of the sample, such as repressurization of the cylinder contents with an inert gas, will not completely effect their recovery, especially the permanent gases. However, the loss is not significant to some users.

5.4 *Vaporization Methods*—Vaporization of the sample, either at the source or in the lab prior to analysis, can cause loss of heavier components, if present, and concentration of lighter ones. Test Method D 2712 describes a low pressure vaporization sampling technique that is suitable to determine trace compounds through butadiene.

5.5 Reactive and Polar Components:

5.5.1 Determination of reactive components, such as certain sulfur compounds and arsine, is generally believed to require special sample containers, such as TFE-fluorocarbon lined cylinders, or containers that have been specially passivated.

5.5.2 It is very difficult to obtain a valid sample to determine traces of polar compounds, such as water and ammonia, in the lab. On-line analyzers, if available, or sorption of the analyte at the sample source for subsequent lab analysis, are believed to yield the most accurate results.

6. Composition and Test Methods

6.1 Table 1 indicates possible composition ranges and ASTM test methods for different grades of propylene concentrates. Table 2 lists other test methods known or believed to be in use.

TABLE 1 Possible Composition of Propylene Concentrates

Component	Polymer Grade	Chemical Grade	Refinery Grade	Test Method
Propylene, % mass	99.0 +	92 to 99	60 to 90	D 2163
Propane, % mass	0.1 to 1.0	1 to 8	10 to 35	D 2163
Methane, mg/kg	<2 to 100	<10 to 1000	<10 to 1000	See Table 2
Ethane, mg/kg	<2 to 200	<10 to 2000	<100 to 100 000	See Table 2
Acetylene, mg/kg	<1 to 10	<2 to 100	...	D 2712
Ethylene, mg/kg	<2 to 100	<2 to 100	<50 to 5000	D 2712
Cyclopropane, mg/kg	<1 to 10	<2 to 500	<2 to 500	See Table 2
Methylacetylene, mg/kg	<1 to 10	<2 to 100	<10 to 1000	D 2712
Propadiene, mg/kg	<1 to 10	<2 to 100	<10 to 1000	D 2712
Butenes, mg/kg	<1 to 20	<2 to 200	<100 to 10 000	D 2712
Butanes, mg/kg	<2 to 50	<10 to 1000	<100 to 15 000	See Table 2
1,3 butadiene, mg/kg	<1 to 10	<2 to 50	<2 to 100	D 2712
C5s, mg/kg	<1 to 10	<2 to 100	<2 to 100	See Table 2
C6s, and heavier, mg/kg	<1 to 10	<2 to 100	<2 to 100	See Table 2
Benzene, mg/kg	<0.1 to 20	<0.1 to 10	...	See Table 2
H ₂ , mg/kg	<1 to 10	<2 to 100	...	See Table 2
O ₂ , mg/kg	<1 to 10	<2 to 100	...	See Table 2
CO, mg/kg	<1 to 10	<2 to 100	...	See Table 2
CO ₂ , mg/kg	<1 to 10	<2 to 100	<2 to 100	See Table 2
N ₂ , mg/kg	<2 to 40	<10 to 1000	...	See Table 2
H ₂ S, mg/kg	<0.1 to 20	<1 to 20	...	See Table 2
C ₂ S, mg/kg	<1 to 10	<1 to 10	<1 to 50	See Table 2
Total S, mg/kg	<1 to 10	<1 to 10	<10 to 250	See Table 2
Mercaptan, S, mg/kg	<1 to 5	<1 to 10	<1 to 10	See Table 2
Water, mg/kg	<1 to 25	<2 to 100	<2 to 100	See Table 2
Total nitrogen, mg/kg	<1 to 5	<1 to 10	...	See Table 2
Total chloride, mg/kg	<1 to 5	<1 to 10	...	See Table 2
Methanol, mg/kg	<1 to 5	<1 to 10	...	D 4864
Other alcohols, mg/kg	<1 to 5	<1 to 10	...	See Table 2
Total oxygenates, mg/kg	<1 to 5	<1 to 10	...	See Table 2
Arsine, mg/kg	<0.1 to 1	<0.1 to 1	<0.1 to 1	See Table 2
Total hydrides, mg/kg	<0.1 to 1	<0.1 to 1	<0.1 to 1	See Table 2

TABLE 2 Propylene Test Methods (Non-ASTM)

Components	Possible Test Methods
Methane, ethane, cyclopropane, butanes, and C5s C6s and heavier	An adaptation of Test Method D 2712 is used by some labs. Others use a GC wide-bore capillary method. GC wide-bore capillary or packed high temperature columns, or both, are used by some.
Benzene	Capillary or packed column GC methods.
H ₂ , N ₂ , O ₂ , CO	An adaptation of Test Method D 2504 is used by some.
CO ₂	An adaptation of Test Method D 2505 is used by some.
Carbonyl sulfide	A GC-FPD method is currently undergoing ASTM cooperative testing. Other methods used in the industry are GC-PID, GC-conductivity, GC-SCD, UOP-212, and GC in series with a Test Method D 4468-type analyzer.
Hydrogen sulfide	Same as listed above for COS, except there are no methods currently undergoing ASTM testing.
Total sulfur	Some labs use an adaptation of Test Method D 3246; others use Test Method D 4468 with an oxy-hydrogen pyrolyzer.
Mercaptan sulfur	Some methods used are: UOP 212; GC-FPD; caustic absorption/potentiometric titration analysis by Test Method D 3227.
Water	Obtaining a valid sample for lab analysis is extremely difficult. Instead of a lab method, an ASTM study group developed in 1982 a standard practice for calibrating moisture analyzers, Practice D 4178. Several types of portable and on-line analyzers are available.
Total nitrogen (bound)	An adaptation of Test Method D 4629 is used by some labs; others use microcoulometry.
Total chlorides	An adaptation of Test Methods D 2384 may be used by some labs; others use reductive microcoulometry.
Arsine	Some methods known to be in use are: AgDDC absorption/colorimetric finish AgDDC absorption/GFAAS finish direct GC-ECD method direct GC-PID method
Total hydrides	MDA scientific toxic gas analyzer
Ammonia	Some methods in use are: acid absorption/Nessler finish acid absorption/specific ion electrode acid absorption/IC finish MDA tape method
Other oxygenates	GC methods; GC-AED, colorimetric methods
Other alcohols	GC methods, both capillary and packed column. Variation of Test Method D 4864

6.2 Listing of any given component in Table 1 does not mean that the component will be present in all, or even any, propylene products. Inclusion in the list is definitely not a recommendation that all propylene products should be tested for the component.

7. Keywords

7.1 propylene; propylene product concentrations; propylene test methods

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