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Standard Guide for Analysis of Propylene Concentrates[1]

This standard is issued under the fixed designation D5273; 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 covers a list of 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 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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D2163 Test Method for Analysis of Liquefied Petroleum (LP) Gases and Propene Concentrates by Gas Chromatography[3]

D2384 Test Methods for Traces of Volatile Chlorides in Butane-Butene Mixtures

D2504 Test Method for Noncondensable Gases in C_2 and Lighter Hydrocarbon Products by Gas Chromatography

D2505 Test Method for Ethylene, Other Hydrocarbons, and Carbon Dioxide in High-Purity Ethylene by Gas Chromatography

D2712 Test Method for Hydrocarbon Traces in Propylene Concentrates by Gas Chromatography

D3227 Test Method for (Thiol Mercaptan) Sulfur in Gasoline, Kerosine, Aviation Turbine, and Distillate Fuels (Potentiometric Method)

D3246 Test Method for Sulfur in Petroleum Gas by Oxidative Microcoulometry

D3700 Practice for Obtaining LPG Samples Using a Floating Piston Cylinder

D4178 Practice for Calibrating Moisture Analyzers

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

D4629 Test Method for Trace Nitrogen in Liquid Petroleum Hydrocarbons by Syringe/Inlet Oxidative Combustion and Chemiluminescence Detection

D4864 Test Method for Determination of Traces of Methanol in Propylene Concentrates by Gas Chromatography

3. Terminology

3.1 *Definitions:*

3.1.1 *outaging*, *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 *Abbreviations:*

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.

[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.

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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.

[3] Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

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 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 liquefied 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 overemphasized.

5.2 *Floating Piston Cylinder*—Test Method D3700 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 Outaging Method*—The widely used outaging 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 D2712 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. Online 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.

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

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	D2163
Propane, % mass	0.1 to 1.0	1 to 8	10 to 35	D2163
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	...	D2712
Ethylene, mg/kg	<2 to 100	<2 to 100	<50 to 5000	D2712
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	D2712
Propadiene, mg/kg	<1 to 10	<2 to 100	<10 to 1000	D2712
Butenes, mg/kg	<1 to 20	<2 to 200	<100 to 10 000	D2712
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	D2712
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 10	<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_2S, mg/kg	<0.1 to 10	<1 to 20	...	See Table 2
C_OS, 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	...	D4864
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	An adaptation of Test Method D2712 is used by some labs. Others use a GC wide-bore capillary method.
C6s and heavier	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 D2504 is used by some.
CO_2	An adaptation of Test Method D2505 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 D4468-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 D3246; others use Test Method D4468 with an oxy-hydrogen pyrolyzer.
Mercaptan sulfur	Some methods used are: UOP 212; GC-FPD; caustic absorption/potentiometric titration analysis by Test Method D3227.
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 D4178. Several types of portable and online analyzers are available.
Total nitrogen (bound)	An adaptation of Test Method D4629 is used by some labs; others use microcoulometry.
Total chlorides	An adaptation of Test Methods D2384 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 D4864