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THE LEADER IN ENVIRONMENTAL TESTING

Removal of Dissolved Gases in Crude Oil and Classification

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- Introduction
- ND Rule Requirements
- DOT/PHMSA Requirements – Classification
- Analytical Measurements
- Methodologies for Classification
- Vapor Pressure Measurements
- Future Methods

- Crude Oil Extraction
- Crude Oil Separation
- Additives
- Conditioning (removal of dissolved gases)
- Custody Transfer

- **Unrefined Petroleum Products** offered in Transport must be conditioned to remove the dissolved gases (April 1, 2015)
- The Vapor Pressure must be less than 13.7 psi prior to custody transfer
- Four Part Conditioning Rule
 - A Gas Liquid Separator and/ Emulsion heater - treater
 - Ample capacity
 - Effectively separates into Gaseous and Light Hydrocarbons
 - Must be operated within manufacturers recommended operating limits
 - 50 psi – 110⁰ F
 - >50 psi – 110⁰ F
 - Any other pressure and temperature must show that VP is not >13.7 psi
 - Any other methodologies must be approved by the Commission
 - Vapor Pressure Method ASTM 6377 (latest edition)(did not define conditions)

- **Applies to all modes of transport other than traditional pipelines**
- **“§173.41 Sampling and testing program for unrefined petroleum-based products.**
- (a) *General. Unrefined petroleum-based products offered for transportation must be properly classed and described as prescribed in §173.22*, in accordance with a sampling and testing program, which specifies at a minimum:
 - (1) **A frequency of sampling and testing** that accounts for any appreciable variability of the material (*e.g.*, history, temperature, method of extraction [including chemical use], location of extraction, time of year, length of time between shipments);
 - (2) **Sampling prior to the initial offering** of the material for transportation and when changes that may affect the properties of the material occur (*i.e.*, *mixing* of the material from multiple sources, or further processing and then subsequent transportation);
 - (3) **Sampling methods** that ensure a representative sample of the entire mixture, as offered, is collected;

- Testing methods that enable classification of the material under the HMR;
- (5) **Quality control measures for sample frequencies;**
- (6) **Duplicate sampling methods or equivalent measures for quality assurance;**
- (7) **Criteria for modifying the sampling and testing program;** and
- (8) Testing or other **appropriate methods** used to identify properties of the mixture relevant to packaging requirements (*e.g.*, compatibility with packaging, identifying specific gravity for filling packages).
- (b) *Certification.* Each person who offers a hazardous material for transportation shall certify, as prescribed by §172.204 of this subchapter, that the material is offered for transportation in accordance with this subchapter, including the requirements prescribed by paragraph (a) of this section.

- (c) *Documentation, retention, review, and dissemination of program.* The sampling and testing program must be documented in writing (*i.e.* hardcopy or electronic file thereof) and must be retained for as long as the sampling and testing program remains in effect, or a minimum of one year. The sampling and testing program must be reviewed at least annually and revised and/or updated as necessary to reflect changed circumstances. The most recent version of the sampling and testing program must be available to the employees who are responsible for implementing it. When the sampling and testing program is updated or revised, all employees responsible for implementing it must be notified, and the most recent version must be made available.
- (d) *Access by DOT to program documentation.* Each person required to develop and implement a sampling and testing program must maintain a copy of the sampling and testing program documentation (or an electronic file thereof) that is accessible at, or through, its principal place of business, and must make the documentation available upon request at a reasonable time and location to an authorized official of the Department of Transportation.
- [80 FR 26746, May 8, 2015]”

Table 1—Criteria for Assignment of PG for a Class 3 Flammable Liquid ¹

Packing Group	Flash point (closed-cup)	Initial boiling point (IBP)
I		≤35 °C (95 °F)
II	<23 °C (73 °F)	>35 °C (95 °F)
III	≥23 °C, ≤60 °C (≥73 °F, ≤140 °F)	>35 °C (95 °F)

1) This table is for informational purposes only and does not provide legal advice on compliance with regulations.

- GPA 2174 – Floating Piston Cylinder (FPC)
- ASTM D3700 – Floating Piston Cylinder
- ASTM – D8009 – Syringe style Manual Piston Cylinder (MPC); Syringe style FPC
- ASTM D7975 – Syringe Style MPC
- ASTM 4057 – Boston Round Sample Bottle



Images from: Welker, Parkes Scientific

- ASTM D93 or D56 – Flash Point (FP)
- ASTM D86 – Initial Boiling Point (IBP)
- New Guidance for IBP noted in API RP 3000
 - API RP 3000 -sec 5.6.3.2 alternate methods
 - GPA 2177 M - GC (TCD/FID)
 - D7900 (GC/FID)
 - Determination of IBP using D7169 (Definition of cut point)
- Newer Method ASTM D8003 GC FPC or MPC (C1-C24)
- IBP Calculator (Envantage software)
- ASTM D6377 – Total Vapor Pressure **NOT** Reid Vapor Pressure (RVP)

IBP for C1 – C4 hydrocarbons

- C1 = -259 °F (Methane)
- C2 = -127 °F (Ethane)
- C3 = -44 °F (Propane)
- iC4 = 11 °F (Iso-butane)
- C4 = 31 °F (n-butane)
- C5 = 97 °F (n-pentane)



Image from: http://www.paclp.com/Lab_Instruments/OptiDist

TABLE 2 Sampling, Storage, and Sample Conditioning

		Group 1	Group 2	Group 3	Group 4
Temperature of sample bottle	°C	<10			
	°F	<50			
Temperature of stored sample	°C	<10 ^A	<10	ambient	ambient
	°F	<50 ^A	<50	ambient	ambient
Temperature of sample after conditioning prior to analysis	°C	<10 ^B	<10 ^B	Ambient or 9 to 21°C above pour point ^C	Ambient or
	°F	<50	<50	Ambient or 48 to 70°F above pour point ^C	Ambient or
If sample is wet		resample	resample	dry in accordance with 7.5.3	
If resample is still wet ^D		dry in accordance with 7.5.2			

ASTM D86 IBP

- Sampling Methods (FPC, MPC, Boston Round)
- Measurement Techniques
- ASTM D6377 - XX



Images from: Grabner Instruments

- ASTM 6377 – XX
- D8003
- D8009
- H₂S requirements (UOP, ASTM)

1. Kesavalu M. Bagawandoss, “Sampling and Analysis in light of the PHMSA/DOT Final Rule”, COQA Meeting, New Orleans, LA, Oct., 2015.
2. ASTM D86 -12.
3. GPA 2177 -13.
4. GPA 2103 -03.
5. ASTM D7900 -13.
6. ASTM D7169 -11.
7. ASTM D6377 – 16.
8. Classifying and Loading of Crude Oil into Rail Tank Cars, ANSI/API Recommended Practice 3000, First Edition, September 2014.
9. 26706 Federal Register / Vol. 80, No. 89 / Friday, May 8, 2015 / Rules and Regulations.
10. http://www.ecfr.gov/cgi-bin/text-idx?SID=a9a6d66aef54e0dac28bcca51dfda688&mc=true&node=pt49.2.173&rgn=div5#se49.2.173_141
11. <http://www.gpo.gov/fdsys/pkg/FR-2015-05-08/pdf/2015-10670.pdf>

THANK YOU !!!

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