PROCEDURE FOR THE DETERMINATION OF DISTILLATION POINTS OFLIQUID FUELS BY AUTOMATED DISTILLATION

SOP MV-FUELS-128
Version 2.1
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1 Introduction

1.1 This document describes the standard operating procedure (SOP) for measuring the distillation temperatures of liquid fuels using an automated distillation instrument.

1.2 This SOP covers gasoline, diesel fuel, aviation fuel, kerosene, and similar petroleum products.

1.3 This SOP is based on American Society for Testing and Materials (ASTM) standard method D86-99a$^{\epsilon 1}$.

2 Method

2.1 A 100 mL sample is introduced into the instrument's flask.

2.2 The sample is distilled under specific conditions depending on its characterization.

2.3 Vapor temperature and condensate volume are periodically measured. These data are used to calculate results.

3 Instrumentation

3.1 Automated distillation instrument that meets ASTM D86-99a$^{\epsilon 1}$ specifications and California Air Resources Board (CARB) regulation requirements.

3.2 Data acquisition system associated with or provided by the manufacturer of chosen instrument running under Microsoft Windows.

3.3 125 mL distillation flask

3.4 100 mL graduated cylinder appropriate for use with 3.1 above

4 Reagents

4.1 Toluene, A.C.S. reagent grade or better.

4.2 Mesitylene, (1,3,5-trimethylbenzene) A.C.S. reagent grade or better.

4.3 Pentane, for general lab use.

5 Preparation of Instrument

5.1 Follow the manufactures manual on instrument warm up.

5.2 The correct distillation method is selected within the software. Method D86 123 1 is used for gasolines with Reid vapor pressure (RVP) greater than 9.5. Method D86-123-2 is used for gasolines with RVP less than 9.5.

5.3 The initial heater temperatures, switching time, and final heat adjustment time are selected by the operator based on the sample RVP and any previous distillation analyses performed (see 7.11).

6 Calibration

6.1 Refer to manufacture's recommendation for calibration: temperature measurement and volume measurement.

6.2 Temperature calibration is performed by distilling pure toluene on the instrument comparing the result with the historical reference value of 228.4°F. Any observed difference between the two readings is used by the software to calibrate the instruments temperature sensor.

6.3 Temperature calibration is carried out at least once every six months or whenever the temperature sensor is changed.

6.4 Volume calibration is performed by following instructions on proper instrument manual and software by the manufacturer.

6.5 Volume calibration is carried out at least once every six months or whenever the volumetric flask is changed.

7 Procedure

7.1 A piece of soft, lint-free cloth attached to a metal wire is used to clean the instrument's condenser tube. It is inserted into the receiver tube and pulled gently through the distilling compartment.

7.2 The sample is chilled to a temperature between 32°F and 50°F.

7.3 The graduated cylinder and distillation flask are chilled to a temperature between 55°F and 65°F.

7.4 The sample is poured into the graduated cylinder. The bottom of the

meniscus should line up with the 100 mL mark.

7.5 The sample is transferred as completely as possible from the graduated cylinder to the distillation flask.

7.6 The distillation flask is centered on the auto distillation instrument heating plate with the side arm connected to the inlet of the condenser tube.

7.7 The temperature sensor is inserted into the distillation flask. The sensor's measuring element must be exactly level with the lower inside edge of the flask's side arm.

7.8 A metallic drop plate is inserted into the top of the graduated cylinder for certain types of auto distillation instrument. The cylinder is inserted under the outlet of the condenser tube. A piece of insulating material cut to fit the cylinder is used as a cover.

7.9 The distillation is started and monitored via the instrument software.

7.10 The following parameters must be met for a distillation to be considered valid:

7.10.1 Time to first drop: 5 to 10 minutes (5 to 15 minutes for diesel)

7.10.2 Time from first drop to 5% recovered: 60 to 100 seconds (N/A for diesel)

7.10.3 Distillation rate: 4 to 5 mL/min

7.10.4 Time from final heat adjustment to end point: 0 to 5 minutes

8 Quality Control

8.1 According to ASTM D86-99a$^{\epsilon 1}$, if any of the parameters in 7.10 is not met, the distillation run is not considered valid. However, in practice, failure to meet all these criteria does not make a significant difference in the data obtained. The acceptability of data obtained from an invalid run is determined by the client.

8.2 In order to obtain data which can meet ASTM D86-99a$^{\epsilon 1}$ requirements, repeated runs with different initial temperatures, switching times, heater curve modifications, and final heat adjustment volumes are used.

8.3 At least once every six months, the distillation temperature of pure toluene is determined and compared with the historical reference value of 228.4°F. Any

difference between the two values is used by the instrument software to correct the automated instrument's reading.

8.4 Repeatability and Reproducibility is given by the following equations:

	Repeatability (°F)	**Reproducibility (°F)**
20-70% collected	r = 2.2 + 1.42S	R = 5.2 + 3.97S
90-95% collected	r = 2.0 + 1.08S	R = 3.6 + 2.53S

Where S is the average slope (or rate of change)

S is calculated with the following equations:

S= (T60-T40)/20 for T50

S= (T90-T80)/10 for T90

9 Safety

9.1 Prepare fuel samples and standards under a fume hood.

9.2 Wear safety glasses and disposable gloves when handling fuels or solvents.

9.3 Fuels and solvents may be harmful or fatal if ingested or inhaled.

9.4 All fuels and solvents should be treated as extremely flammable and explosive.

10 References

10.1 "Standard Test Method for Distillation of Petroleum Products (Designation D86-99a$^{\epsilon 1}$)," Annual Book of ASTM Standards, Vol 05.01.

10.2 User Manuals for instruments and software from the manufacturer.

11. Revision History

11.1 Revision 1.1: Effective date: 10/1/1997.

11.2 Revision 1.2: Effective date: 4/1/2000.

Significant changes:

Section 8.3 modified to include mesitylene distillation, which had been inadvertently omitted.

11.3 Revision 1.3 Effective date: 7/1/2001.

Significant changes:

All references to MP 626 changed to MP 626/7/8 to reflect the presence of different models of the instrument.

References to ASTM method updated to D86-99a$^{\epsilon 1}$ to reflect regulatory changes.

Requirements in section 7.10 updated to reflect the new version of D86.

Calibration frequency changed to once per six months to mirror the requirements of D86.

11.4 Revision 1.4 Effective date: 4/1/2002.

Significant changes:

References to mesitylene distillation removed and all references to manual distillation removed.

Section 5.1 updated to reflect newer instrument models.

Section 7.1 changed to reflect current practice, as advised by the instrument manufacturer.

11.5 Revision 1.5 Effective date: 7/1/2014.

Significant changes:

Added Optidist to reflect the presence of different models of the instrument. Repeatability, reproducibility, and slope equations added. Safety section added.

11.6 Version 2.0 Effective date: 5/1/2017.

Significant changes:

Items 3.1 and 3.2 were revised to remove the specifications on brand names of instruments.

Items 4.2 and 4.3 were added. Items 5.1, 6.1, 6.4, 7.6, 7.8 and 10.2 were revised to remove instrument specifications.

11.7 Version 2.1 Effective date: 12/1/2019

SOP format updated for ADA compliance