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APPROVAL AUTHORITY		DATE
Andrew D. Sullivan	Instructor	August 18, 2015

REVISION HISTORY			
Revision	Description of Change	Author	Effective Date
01	Initial Release	Andrew D. Sullivan	August 18, 2015
02	Fixed guide to pressure correction in step 26.6	Andrew D. Sullivan	September 13, 2017

REFERENCE DOCUMENTS	
Document	Title
ASTM D86	Standard Test Method for Distillation of Petroleum Products at Atmospheric Pressure

1.0 Purpose

This document describes the standard operating procedure (SOP) for measuring the distillation temperatures of liquid fuels using the K45000 Koehler manual apparatus for Distillation of Petroleum Products at Atmospheric Pressure.

2.0 Scope

This procedure is specific for group 1 and 2 liquids per the ASTM D86 test method. This includes gasoline and kerosene with a FBP less than 480°F.

3.0 Health, Safety, and the Environment

This standard test method requires heating highly flammable liquids to high temperatures. An accident can cause a fire. Keep all non-necessary flammable materials away from the testing area. Have an extra fire extinguisher at the testing location. Verify the safety shower is functional. Everyone in the area is to be wearing a flame retardant lab jacket at a minimum. Never sit down near hydrocarbons where a spill could result in burning liquids spilling on legs. A fire

watch needs to be stationed with a fire extinguisher to monitor and respond from a safe location.

Special care must be taken if a mercury thermometer is used. Mercury is a hazardous material and must be managed properly. If a mercury thermometer is broken, collect the mercury using pipettes into a sealed jar.

4.0 Special Material and Equipment

- K45000 Koehler Front View Distillation Apparatus
- Distillation Flask, Type B, 125mL
- Graduated Cylinder, Type B, 100mL
- Receiver Cooling Bath Jar
- ASTM 7F Low Distillation thermometer, 30 to 580°F (or equivalent)
- Flask Support Board, Type B, 1 1/2" Dia. Hole
- Top silicone plug
- Side silicone plug
- Ice
- Stopwatch (an ordinary watch works, but not as well)

5.0 Definitions and Acronyms

Following is a list of key definitions and acronyms needed to perform this procedure. A more exhaustive listing is in the ASTM method referenced.

ASTM - American Society for Testing and Materials

ASM D86 - Standard Test Method for Distillation of Petroleum Products at Atmospheric Pressure

IBP - initial boiling point. The corrected thermometer reading that is observed at the instant the first drop of condensate falls from the lower end of the condenser tube.

Dry point - Thermometer reading that is observed at the instant the last drop of liquid evaporates from the lowest point in the distillation flask.

FBP - final boiling point. The maximum corrected thermometer reading obtained during the test.

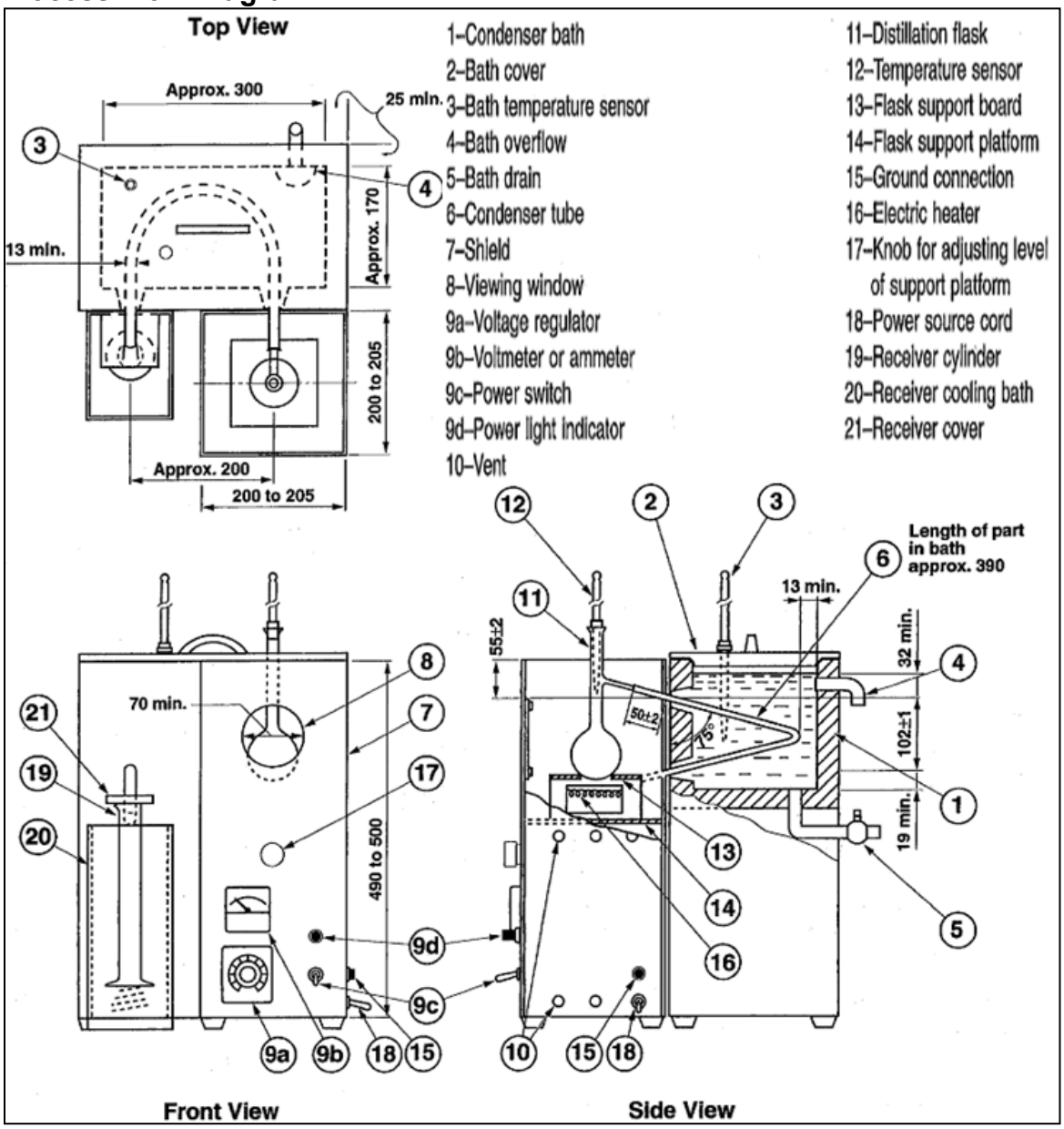
Decomposition - Cracking of a molecule yielding smaller molecules with lower boiling points than the original molecule. Indications of thermal decomposition

are evolution of fumes and erratic temperature readings that usually decrease after any attempt is made to adjust heat.

6.0 Overview

A 100 mL sample is introduced into the instrument's flask. The sample is distilled under specific conditions depending on its characterization. Vapor temperature and condensate volume are periodically measured. These data are used to calculate results.

7.0 Process Flow Diagram



8.0 Roles and Responsibilities

Technicians Shall:

- Follow all laboratory rules and safety practices.
- Properly wear and use all safety equipment as set out in this procedure and standard lab rules.
- Notify the instructor or supervisor of non-routine issues that pose a possible safety concern.
- Conduct a pre-task hazard assessment for all jobs before they are started, and request assistance from the instructor if there are causes for concern from the standpoint of exposure to a hazardous material, a physical exposure hazard, or an ergonomic hazard.

9.0 Procedure

COMMUNICATION WITH IS ESSENTIAL FOR SAFE OPERATIONS Deviations of this procedure must be RISK ASSESSED, DOCUMENTED, and APPROVED by a Supervisor		Initial, date, & time
1.	PRINT your group's names here.	
2.	READ this procedure completely before starting. You need to be familiar with the big picture to be effective.	
3.	PREPARE a quality PTHA before starting. You can't be a great operator until this step is automatic.	
4.	SIGN OUT glassware needed for this lab. It is special and expensive.	
5.	USE a piece of soft, lint-free cloth attached to a metal wire to clean the instrument's condenser tube. It is inserted into the receiver tube and pulled gently through the distilling compartment.	
6.	CHILL the sample to 50°F or cooler.	
7.	CHILL the graduated cylinder and distillation flask to a temperature between 55°F and 65°F.	
8.	CHARGE the cooling bath with ice water to hold 32-34°F.	

WARNING Don't pour material from a large container into a graduated cylinder. Many containers such as metal sample cans, cans with bung openings, and glass jars don't pour well. A large flammable fluid spill may result. Use a pipette in the sink for safe transfer.		
9.	TRANSFER the sample into the graduated cylinder. The bottom of the meniscus should line up with the 100 mL mark.	
10.	TRANSFER the sample as completely as possible from the graduated cylinder to the distillation flask.	
11.	VERIFY a distillation flask support board with a 38 mm hole is positioned on the heater.	
12.	POSITION distillation flask on the support board and heater with a snug fitting stopper sealing the vapor tube to the condenser tube.	
	12.1. ADJUST the flask in a vertical position so that the vapor tube extends into the condenser tube for a distance from 25 to 50 mm.	
	12.2. ADJUST the flask support board to fit it snugly against the bottom of the flask.	
13.	ADD a boiling chip to the distillation flask. A small chip of porous ceramic works well.	
WARNING ASTM thermometers contain mercury. Use safe thermometer handling practices that you have been trained on. Clean up procedures are provided in safety section above.		
14.	INSERT an ASTM 7F thermometer (30 to 580°F) into the distillation flask with snug fitting stopper. The top of the mercury bulb must be level with the lower inside edge of the flask's side arm.	
15.	PLACE the receiving cylinder that was used to measure the sample, without drying the inside of the cylinder, into its temperature-controlled bath under the lower end of the condenser tube.	

	15.1. VERIFY the end of the condenser tube is centered in the receiving graduated cylinder and extends inside the cylinder a distance of at least 25 mm, but not below the 100-mL mark.	
	15.2. To reduce evaporation loss of the distillate, cover the receiving cylinder with the fiber receiver cover that fits the condenser tube snugly.	
16.	APPOINT a fire watch with a charged fire extinguisher to monitor and respond instantly should a fire start. They are responsible for making sure all the safety steps detailed above are followed. The individual may rotate shifts.	
17.	START the distillation by applying heat at a setting of 50%.	
18.	USE the attached table to collect data.	
19.	RECORD the IBP to the nearest 1°F. This is the thermometer reading at the instant the first drop of condensate falls from the lower end of the condenser tube.	
20.	MOVE the receiving cylinder so that the tip of the condenser touches its inner wall. This prevents splashing which make reading the level difficult.	
21.	ADJUST heating so that the time interval between the first application of heat and the IBP is 5-10 minutes.	
22.	ADJUST heating so that the time from IBP to 5% recovered is 60-100 seconds.	
23.	ADJUST heating so that the rate of condensation from 5% recovered to 5 mL residue in the flask is 4 to 5 mL per min.	
24.	ADJUST heating so that the time from 5 mL of liquid residue in the flask to the FBP is 5 minutes or less. NOTE: 5 ml remaining in the distillation flask corresponds to 93.5 ml in the receiving cylinder based on 1.5 ml holdup.	
	CAUTION Heating a dry distillation flask may break it.	

25.	IMMEDIATELY STOP heat when the FBP or Dry Point has been observed.	
26.	ALLOW condenser to drain until recovery is constant within 0.1 ml for 2 minutes.	
27.	PERFORM the following calculations:	
	27.1. RECORD the volume in the receiving cylinder as the percent recovery.	
	27.2. MEASURE the volume remaining in the distillation flask using a 5 ml graduated cylinder to the nearest 0.1 mL as the percent residue.	
	27.3. RECORD total recovery as the sum of the percent recovery and the percent residue.	
	27.4. RECORD the percent loss by subtracting the percent total recovery from 100.	
	27.5. RECORD atmospheric pressure for the location. You may use 683 mm Hg for Billings which excludes normal barometric variations.	
	27.6. CORRECT temperature readings to 760 mm Hg using the attached table or equation from the test method. The equation might be easier and is: $T_{corrected} = T_{raw} + 0.00012\ (760 - P)\ (460 + T_{raw})$ Where P is the barometric pressure at the time of the test in mmHg. For Billings you can use 683 mm Hg. As an example, consider a point at 400°F. $T_{corrected} = 400 + 0.00012\ (760 - 683)\ (460 + 400) = 407.9°F$	
28.	PREPARE an Excel graph of your results with the percent recovered on the X-axis. Each person needs to do this independently.	

29.	RETURN the glassware you signed out and have instructor verify the lab has been appropriately cleaned and cared for.	
30.	RETURN this signed off procedure and graphs to your instructor.	

END OF PROCEDURE

Data Collection Form:

	Time (m:s)	Heat Setting (%)	Rate (ml/min)	Raw Temp (°F)	Press Correction (°F)	Final Temp (°F)
Heat On						
IBP						
5 ml						
10 ml						
20 ml						
30 ml						
40 ml						
50 ml						
60 ml						
70 ml						
80 ml						
90 ml						
95 ml						
FBP						

Percent Recovery:

Percent Residue:

Percent Total Recovery:

Percent Loss:

Temperature Correction Table

TABLE 6 Approximate Thermometer Reading Correction

Temperature Range		Correction[A] per 1.3 kPa (10 mm Hg) Difference in Pressure	
°C	°F	°C	°F
10–30	50–86	0.35	0.63
30–50	86–122	0.38	0.68
50–70	122–158	0.40	0.72
70–90	158–194	0.42	0.76
90–110	194–230	0.45	0.81
110–130	230–266	0.47	0.85
130–150	266–302	0.50	0.89
150–170	302–338	0.52	0.94
170–190	338–374	0.54	0.98
190–210	374–410	0.57	1.02
210–230	410–446	0.59	1.07
230–250	446–482	0.62	1.11
250–270	482–518	0.64	1.15
270–290	518–554	0.66	1.20
290–310	554–590	0.69	1.24
310–330	590–626	0.71	1.28
330–350	626–662	0.74	1.33
350–370	662–698	0.76	1.37
370–390	698–734	0.78	1.41
390–410	734–770	0.81	1.46

[A] Values to be added when barometric pressure is below 101.3 kPa (760 mm Hg) and to be subtracted when barometric pressure is above 101.3 kPa.