

CHAPTER 3

EXPERIMENTATION

3.1 PHYSICAL PROPERTIES OF HIGH SPEED DIESEL

3.1.1 Standard Test Method for Kinematic Viscosity (ASTM D 445-94)

This test method specifies a procedures for the determination of the kinematic viscosity of liquid petroleum products by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer. The values stated in IS units are to be regarded as the standard.

Procedure

Adjust and maintain the viscometer bath at 40 °C. Select a clean, dry, calibrated viscometer having a range covering the estimated kinematic viscosity. The flow time shall not be less than 200s and the longer time than 1000s. Start the automated apparatus in accordance with the maunfature's instructions. The apparatus shall follow the procedural details.

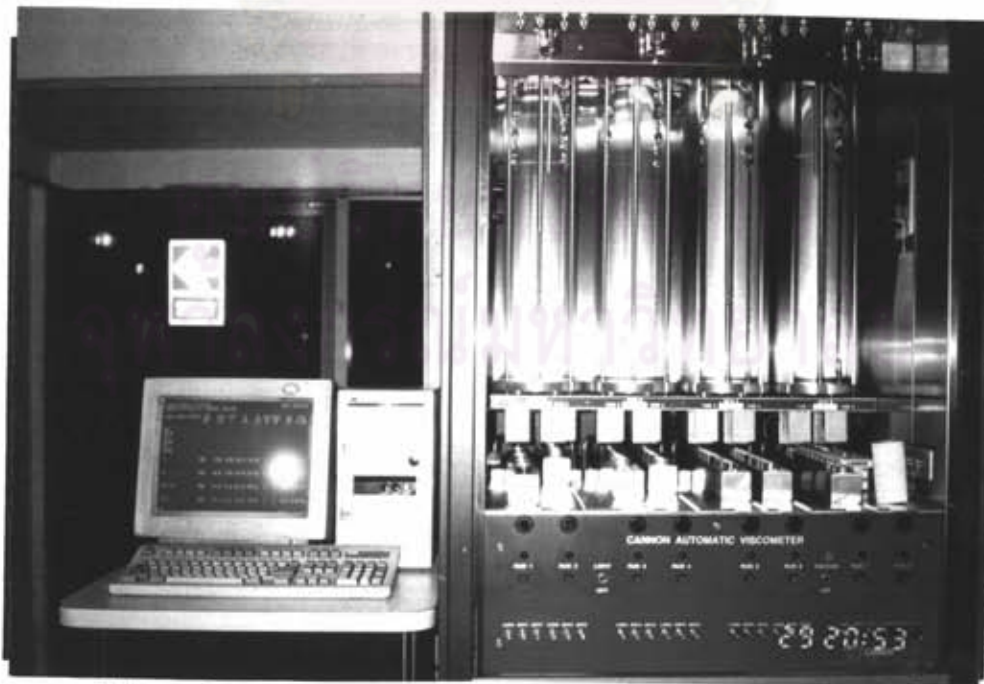


Figure 3.1 Cannon Automatic Viscometer (model CAV 4).

3.1.2 Standard Test Methods for Flash-Point by Pensky-Martens Closed Cup Tester (ASTM D 93-94)

This test method describes the determination of the flash point of petroleum products by automated by Pensky-Marteni Closed Cup Apparatus.

Procedure

Fill the test cup with the test specimen to the filling mark inside of the test cup. Place the test cover on the test cup and place the assembly into the apparatus. Insert the temperature measuring device into its holder. Start the automated apparatus in accordance with the maunfature's instructions. The apparatus shall follow the procedural details.



Figure 3.2 iSL-Automated Flash point (model PMFP 93).

3.1.3 Standard Test Method for Pour Point of Petroleum Products (ASTM D 97-93)

This test method is intended for use on any petroleum product.

Procedure

Pour the specimen into the test jar to the level mark. Close the test jar with the cork carrying the high-pour thermometer. Adjust so the thermometer and the jar are coaxial, and the thermometer bulb is immersed so the beginning of the capillary is 3 mm below the surface of the specimen. Place the disk in the bottom of the jacket. Place the gasket around the test jar, 25 mm from the bottom. Insert the test jar in the jacket.

Pour point are expressed in integers that are positive or negative multiples of 3 °C. Begin to examine the appearance of the specimen when the temperature of the specimen is 9°C above the expected pour point (estimated as a multiple of 3°C). At each test thermometer reading that is a multiple of 3°C below the starting temperature remove the jar from the jacket. To remove condensed moisture that limits visibility wipe the surface with a clean cloth moistened in alcohol (ethanol or methanol). Tilt the jar just enough to ascertain whether there is a movement of the specimen in the test jar. The complete operation of removal, wiping, and replacement shall require not more than 3 s. Continue in this manner until a point is reached at which the specimen shows no movement when the test jar is held in a horizontal position for 5 s. Record the observed reading of the test thermometer.

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3.1.4 Standard Test Method for Sulfur in Petroleum Products by Energy-Dispersive X-Ray Fluorescence Spectroscopy (ASTM D4294-90)

Procedure

Prepare the sample cell and fill with sample to a minimum depth of 3 mm. Provide adequate head space. Standards-Obtain four readings on each standard using the recommended counting time for the instrument (typically acceptable counting times are 50 to 300 s). Immediately repeat the procedure using freshly prepared cells and fresh portions of samples. From the data obtained, calculate the average reading for each sulfur concentration. Prepare a calibration graph from the averaged results.

Samples for analysis-Before filling in the cell. Fill the cell to the required depth and ensure there are no air bubbles between the window and the liquid. Obtain two consecutive counts using the recommended counting time for the instrument on a portion of the sample. Calculate the average count for the sample.



Figure 3.3 Oxford X-ray Fluorescence Analyzer (model LAB-X 1000).

3.1.5 Standard Test Method for Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption (ASTM D 1319-95)

This test method is for determining hydrocarbon types over the concentration range from 5 to 99 volume % aromatics, 0.3 to 55 volume % olefins, and 1 to 95 volume % saturates in petroleum fractions.

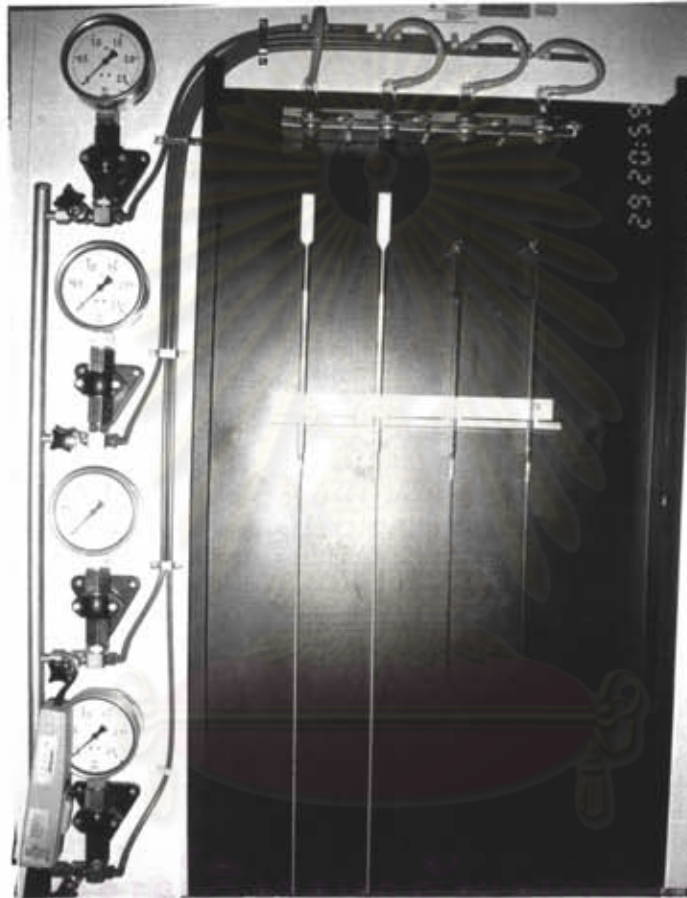


Figure 3.4 Adsorption Columns

Procedure

Packed silica gel into column. Attach the filled column to the apparatus assembly in the darkened room or area fasten the lower end of the column to the fixed rule with a rubber band. Chill the sample and a hypodermic syringe to 2 to 4°C. Draw 0.75 ± 0.03 -mL of sample into the syringe and inject the sample 30 mm below the surface of the gel in the charger section. Fill the charger section to the spherical joint with

isopropyl alcohol. Connect the column to the gas manifold and apply 14kPa gas pressure for 2.5 min to move the liquid front down the column. Increase the pressure to 34 kPa gage for another 2.5 min. After the red, alcohol-aromatic boundary has advanced 350 mm into the analyzer section, make a set of readings by quickly marking the boundary of each hydrocarbon-type zone observed in ultraviolet light, in the following sequence. For the nonfluorescent saturate zone, mark the front of the charge and the point where the yellow fluorescence first reaches its maximum intensity; for the upper end of the second, or olefin zone, mark the point where the first intense blue fluorescence occurs; finally, for the upper end of the third, or aromatic zone, mark the upper end of a reddish or brown zone. With colorless distillates, the alcohol-aromatic boundary is clearly defined by a red ring of dye. However, impurities in cracked fuels often obscure this red ring and give a brown coloration, which varies in length, but which shall be counted as a part of the aromatic zone, except that when no blue fluorescence is present, the brown or reddish ring shall be considered as part of the next distinguishable zone below it in the column. If the boundaries have been marked off with index clips, record the measurements.

When the sample has advanced another 50 mm down the column, make a second set of readings by marking the zones in the reverse order as described in 5 so as to minimize errors due to the advancement of boundary positions during readings. If the marking has been made with a glass-writing pencil, two colors can be used to mark off each set of measurements and the distances measured at the distances measured at the end of the test with the analyzer section lying horizontally on the bench top. If the boundaries have been marked off with index clips, record the measurements.

Erroneous results can be caused by improper packing of the gel or incomplete elution of hydrocarbons by the alcohol. With precision bore columns, incomplete elution can be detected from the total length of the several zones, which must be at least 500 mm for a satisfactory analysis. With standard wall tubing, this criterion of total sample length is not strictly applicable because the inside diameter of the analyzer section is not

the same in all columns. Release the gas pressure and disconnect the column. To remove used gel from the precision bore column, invert it above a sink and insert through the wide end a long piece of No. 19-gage hypodermic tubing with a 45° angle tip. By means of 6-mm outside diameter copper tubing at the opposite end for attaching a rubber tube, connect to a water tap and flush with a rapid stream of water. Rinse with residue-free acetone and dry by evacuation.

Calculation

1. For each set of observations calculate the hydrocarbon types to the nearest 0.1 Volume percent as follows.

$$\text{Aromatics, \%volume} = (L_a/L) \cdot 100 \quad (1)$$

$$\text{Olefins, \%volume} = (L_o/L) \cdot 100 \quad (2)$$

$$\text{Saturates, \%volume} = (L_s/L) \cdot 100 \quad (3)$$

Where: L_a = length of the aromatic zone, mm,
 L_o = length of the olefine zone, mm,
 L_s = length of the saturate zone, mm, and
 L = sum of $L_a + L_o + L_s$.

Average the respective calculated values for each type and report. If necessary, adjust the result for the largest component so that the sum of the components is 100%.

2. Equations 1, 2, and 3 calculate concentrations on an oxygenate-free basis and are correct only for samples that are composed exclusively of hydrocarbons. For samples that contain oxygenated blending components, the above results can be corrected to a total sample basis as follows:

$$C = C \cdot \frac{100-B}{100} \quad (4)$$

where:

C = concentration of hydrocarbon type (% volume) on a total sample basis,

C = concentration of hydrocarbon type (% volume) on an oxygenate-free basis, and

B = concentration of total oxygenate blending components (% volume) in sample as determined by Test Method D 4815, or GC/OFID of equivalent.

• 3.1.6 Standard Test Method for ASTM Color of Petroleum Products (ASTM Color Scale) (ASTM D 1500-91)

This test method covers the visual determination of the colour of a wide variety of petroleum products. This test method reports results specific to the test method and recorded as “ASTM Colour”.

Procedure

Place a sample container or containers, filled to a depth of at least 50 mm with distilled water in the compartment or compartments, of the colorimeter through which the standard glasses will be observed. Place the sample in its container in the other compartment. Cover the containers to exclude all exterior light. Switch on the light source and compare the color of the sample with that of the standard glasses. Determine for two field comparators which glass matches the color of the sample; or if an exact match is not possible, then use that glass which possesses the next darker color.



Figure 3.5 Colorimeter

3.1.7 Standard Test Method for Density of Liquid Petroleum Products by Hydrometer (ASTM D 1298-85)

Procedure

Transfer the sample to a clean hydrometer cylinder without splashing, to avoid the formation of air bubbles. Place the cylinder containing the sample in a vertical position in a location free from air currents.

3. Lower the hydrometer gently into the sample. Continuously stir the sample with the thermometer record the temperature of the sample to the nearest 0.25°C (0.5°F) and then remove the thermometer.

4. To convert corrected values to standard temperature, use the following from the Petroleum Measurement Tables 53 A or 53 B to obtain density 15 °C .

3.2 OXIDATION REACTION OF SULFUR COMPOUND IN HIGH SPEED DIESEL

Reagent and Material

1. Hydrogen peroxide solution 30 %, puriss grade.
2. tert-Butyl hydroperoxide solution 70 % in water, puriss grade.
3. Acetic acid, puriss grade; > 99.5 %.
4. Ethanol 97 %, puriss grade.
5. Methanol 97 %, puriss grade.
6. Purity of water.
7. Magnesium sulfate anhydrous (MgO_4S), purum grade; >98 %.
8. Peracetic acid 40%

Apparatus

1. Temperature-controlled bath.
2. Motor and Wire stirrer, a piece of stiff iron wire of about 0.9 mm in diameter (No-20 Bds) 250 mm long.
3. Thermometer, ASTM 12C.
4. Dropper Pipet, provided with a rubber bulb.
5. Analytical Balance, capable of reproducing weights to 0.1 mg.
6. Separatory Funnel, 500 ml.
7. Centrifuge tube, 100 ml.

3.2.1 Oxidation Reaction by Hydroperoxide

Procedure

1. Mix high speed diesel 200 g. and solvent 200 g. in reactor which be controlled 90°C in water bath.
2. Stir with speed 1600 cycles per minute.
3. After the solution reaches 90°C , add dropwise of hydroperoxide within 30 minutes and continue stir 60 minutes and then allowed to cool down to room temperature.
4. The top was separated, washed successively with water and centrifuged.
5. Add Magnesium Sulfate 1 g. to above high speed diesel then centrifuge for taking water away.

3.2.2 Oxidation Reaction by Peroxyacid

Procedure

1. Mix high speed diesel 200 g. and solvent 200 g. in reactor which be controlled 60°C in water bath.
2. Stir with speed 1600 cycles per minute.

3. After the solution reaches 90°C , add dropwise of peroxyacid within 30 minutes and continue stir 45 minutes and then allowed to cool down to room temperature.
4. The top was separated, washed successively with water and centrifuged.
5. Add Magnesium Sulfate 1 g. to above high speed diesel then centrifuge for taking water away.

3.3 SOLVENT EXTRACTION OF SULFUR COMPOUND

Reagent and Materials

1. Ammonia Solution 25 %.
2. Acetic acid, puriss grade; > 99.5 %.
3. Methanol 97 %, puriss grade.
4. Ethanolamine ($\text{CH}_2\text{.OHCH}_2\text{NH}_2$); > 97 %.

Apparatus

1. Vibration machine.
2. Separatory Funnel, 500 ml.
3. Centriifuge tube, 100 ml.

Procedure

1. Mix high speed diesel 200 g. and solvent in ratio in separation flask.
2. Set separation flask with vibration machine and start with speed 60 pulse per minute for 20 minutes.
3. Separate high speed diesel layer and then centrifuge.
4. Extract again with distillated water for more 2 times.
5. Add Magnesium Sulfate 1 g. to above high speed diesel.