

## **CHAPTER III**

### **EXPERIMENTAL**

#### **3.1 Apparatus, Chemicals and Materials**

##### **3.1.1 Apparatus**

**1. Gas Chromatograph**

HP 5890 Series II, Hewett Packard, USA.

**2. High vacuum pump**

MFG Corporation, Michigan. USA.

**3. Soxhlet**

**4. Rotary evaporator**

Eyela Rotary Vacuum Evaporator, Tokyo Rikakikai Co., Ltd.  
Tokyo, Japan.

**5. Water vacuum pump**

Eyela Rotary Vacuum Evaporator, Tokyo Rikakikai Co., Ltd.  
Tokyo, Japan.

**6. Water bath**

Thermolyne, Lybron Corporation, Subsidiary of Sybron Corporation  
Dubuque, Iowa. USA.

**7. Diesel engine**

ISUZU 4JA 1L model, Direct Injection 2500 cc. Turbo.

##### **3.1.2 Chemical and Material**

**1. Cyclohexane**

AR grade

**2. Standard Polycyclic Aromatic Hydrocarbons**

Supelco Co., Ltd. 16 mixture PAHs from EPA 610.

3. Fusel oil

Chemical Waste from the fermentation Industries

4. Glass-microfibre filter

Whatman model GF/F, 7.0 cm. Diameter

5. Flexible Polyurethane Foam

0.41 g/cm<sup>3</sup> density



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### 3.2 Test Engine

ISUZU 4JA 1L model, Direct Injection, 2500 cc. turbo was used in this project. The specification of the engine are shown in Table 3.1

**Table 3.1** Characteristics of diesel engine

Model	4JA 1L
Bore (mm)	93.0
Stroke (mm)	92.0
Displacement (cc)	2,499.00
Compression Ratio	18.5
Cylinders	4
Injection	Direct Injection
Power output (Kw/rpm)	58/3,900
Torque (Nm/rpm)	176/1,800
Low Idle Speed (rpm)	750

### 3.3 Purification of fusel oil

Black-brown fusel oil from the fermentation industries was decolorized by activated charcoal and followed by vacuum filtration. The filtrate was distilled by normal atmospheric distillation until light brown liquid started to come out of the distilling flask.

### 3.4 Engine operation and Collected exhaust sampling

The engine was preconditioned for half an hour at the beginning of each test and then held for 20 minutes at each test condition to establish equilibrium

before the exhaust gases were sampled. Engine conditions were fixed at no load condition and speed was varied at 800, 1600, 2400 rpm, respectively.

Collected exhaust samplings were performed according to by the EPA 1988 Transient FTP for heavy-duty diesel engines which were analyzed for PAHs by using a dilution technique[13] as shown in Figure 3-1

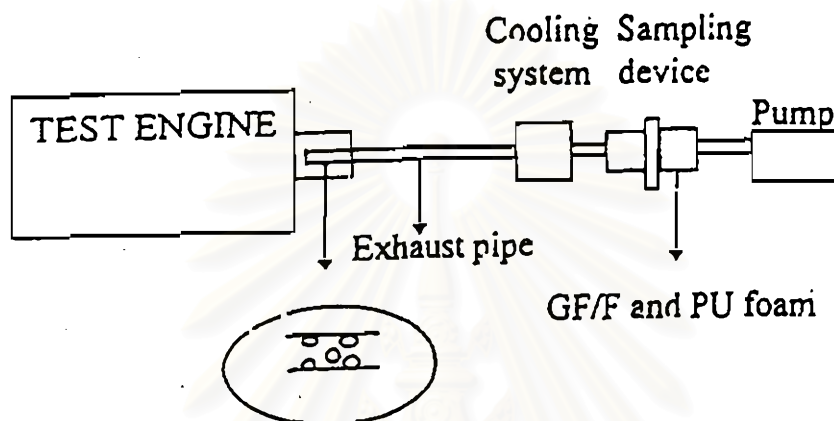


Figure 3-1 Sampling system

Exhaust samplings were taken from the exhaust pipe via a 5/8 -in i.d. stainless-steel probe. The exhaust emission was passed through a cooling system, sampling device and pump, respectively. In sampling device, whatman glass fiber GF/F grade filtration paper and flexible polyurethane foam were used as filters[18] which trapped exhaust particulates and semi-volatile exhaust samples. The samples were kept in dark and stored in refrigerator until extraction.

### 3.5 Sample Analysis

#### 3.5.1 Soxhlet extraction

The glass fiber filter and polyurethane foams exposed to the exhaust were extracted with 300 mL cyclohexane for 12 hours by soxhlet extraction. The cyclohexane soxhlet extracts were allowed to evaporate by rotary evaporation until the volume was less than 10 mL and then transferred to a 10 mL volumetric flask. The remaining extract was rinsed with a small amount of cyclohexane and this solution was added to the volumetric flask and stocked in refrigerator until subjected to GC analysis.

#### 3.5.2 Gas Chromatograph

PAHs were identified and quantified by GC using standard PAHs from Supelco Co., Ltd. The GC conditions to analyze the PAHs samples are listed below [18].

Column	DB-5 HT capillary column length 25 m, ID 0.20 mm., film thickness 0.33 $\mu\text{m}$ .
Carrier Gas	Helium
Carrier Gas Flow Rate	1.6 mL/min at 80 $^{\circ}\text{C}$
Split Flow Rate	60 mL/min
Purge Flow Rate	2.6 mL/min
Column Head Pressure	28 psi
Oven	80 $^{\circ}\text{C}$ (1 min) to 200 $^{\circ}\text{C}$ rate 15 $^{\circ}\text{C}/\text{min}$ 200 $^{\circ}\text{C}$ to 310 $^{\circ}\text{C}$ rate 3 $^{\circ}\text{C}/\text{min}$
Injector Temperature	250 $^{\circ}\text{C}$
Injector	Splitless mode

Sample volume	1 $\mu$ L on-column injector
Detector	Flame Ionization

Before a sample was injected, the GC was preconditioned for an hour at oven temperature of 250 °C, to flush contaminants from the column. PAHs were identified by comparing their retention time with these reference standards. Quantitative were carried out by comparing the integrated peak area of samples to the corresponding standard calibration curve.

### 3.6 Data Evaluation

PAHs mixture for EPA 610 was used as the standard for establishing the calibration curve. The chromatographic system was used by the normal standard technique[19] which prepare calibration standards at a minimum of five concentration levels by adding volumes of one or more stock standards to a 10 mL volumetric flask with cyclohexane.

The standard were analyzed by GC using the same condition employed for all samples. Retention time and response peak area of each component were recorded and the calibration curves were established by plotting the responding peak area against the concentration of standard PAHs. All the calibration curves are shown in Appendix A

Linear equation of standard calibration standard substances	Linear equations
Acenaphthene	$Y = 397452X - 466713$
Acenaphthylene	$Y = 376833X - 893392$
Anthracene	$Y = 715446X - 11810$
Benzo(a)anthracene	$Y = 443087X - 14806$
Benzo(b)fluoranthene	$Y = 432597X - 3566.4$
Benzo(k)fluoranthene	$Y = 280421X - 7611.9$

Benzo(g,h,i)perylene	$Y = 503244X - 46306$
Benzo(a)pyrene	$Y = 434222X - 11171$
Chrysene	$Y = 515528X - 2190.3$
Dibenz(a,h)anthracene	$Y = 433492X - 65346$
Fluoranthene	$Y = 1E+06X - 22402$
Fluorene	$Y = 434295X - 84477$
Indeno(1,2,3-c,d)pyrene	$Y = 305657X - 13930$
Naphthalene	$Y = 293254X - 3657$
Phenanthrene	$Y = 640119X - 24585$
Pyrene	$Y = 898707X - 634.88$

Where

Y	=	Peak Area of Chromatogram
X	=	Concentration (ppm)

The quantity of each PAH was found in the diesel exhaust can be evaluated by the following method[17].

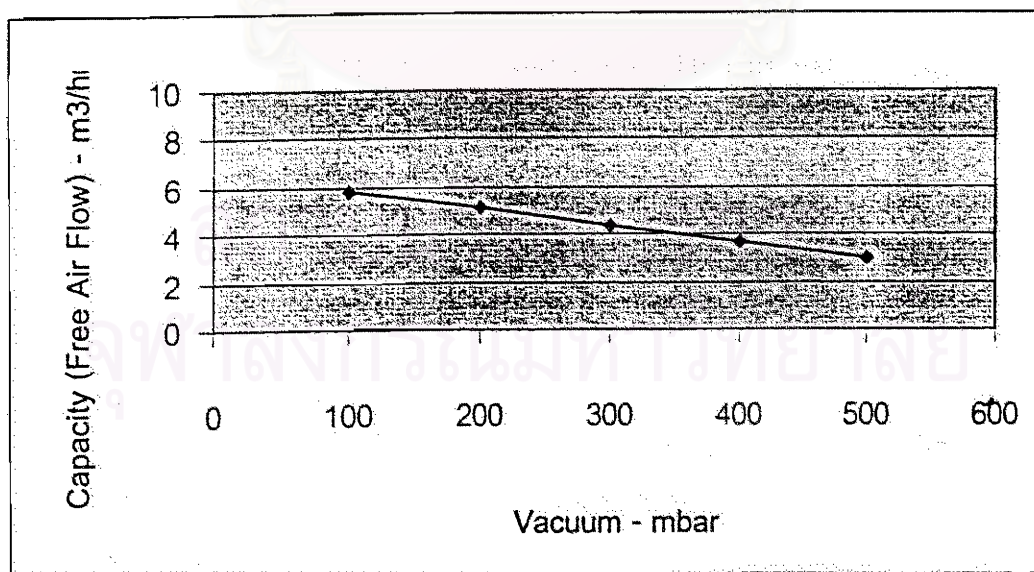


Fig 3.2 Pressure performance curve for measuring volume of exhaust emission

The linear equation of this curve is :

$$F = (-6.97 \times 10^{-3} \times P) + 6.49$$

where

$$F = \text{Flow rate of exhaust ( m}^3/\text{h)}$$

$$P = \text{Pump pressure ( mbar )}$$

Volume of exhaust emission

$$V = F \times T$$

where

$$V = \text{Volume of exhaust gas (m}^3\text{)}$$

$$T = \text{Time of sampling (hr)}$$

The concentration of each PAH in 1  $\mu\text{L}$  injected sample was calculated by substituting the response peak area in the corresponding linear equations of the standard calibration curve. The quantity of PAHs per cubic meter of exhaust was calculated by the equation

$$Q = (C \times E)/V$$

where

$$Q = \text{Quantity of PAHs 1 m}^3 \text{ exhaust}(\mu\text{g/m}^3)$$

$$C = \text{Concentration of PAHs in } \mu\text{L injected sample } (\mu\text{g/mL})$$

$$E = \text{Final volume (mL) of extract sample before GC injection}$$

Example: Calculation of pyrene (ppm) in exhaust samples of diesel engine

$$\text{Pump pressure (P)} = 40.000 \text{ mbar}$$

$$\text{Time of sampling(T)} = 20 \text{ min}$$

$$\text{Peak area of chromatogram(A)} = 20,000$$

$$\text{Final volume of extract sample(E)} = 10 \text{ mL}$$

$$F = (-6.97 \times 10^{-3} \times 40.000) + 6.49$$



$$\begin{aligned} &= 6.22 \text{ m}^3/\text{h} \\ V &= 6.22 \times (20/60) \\ &= 2.07 \text{ m}^3 \end{aligned}$$

Substitute  $Y = 20,000$  in the linear equation of pyrene calibration curve

$$\begin{aligned} X &= (20,000 + 634.88)/898707 \\ &= 2.3 \times 10^{-2} \text{ ppm} \\ Q &= [(2.3 \times 10^{-2}) \times (10)]/2.07 \\ &= 0.11 \text{ } \mu\text{g}/\text{m}^3 \end{aligned}$$



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