CHAPTER III

MATERIAL AND METHOD

3.1 INTRODUCTION

This research was conducted to determined chemical fingerprints of RON 95 commercial petrol (Petronas, Shell and BHP) in the Klang Valley area. This research is experimental in form with emphasis in laboratory analysis. To ensure understanding of how the research was done, the steps composed of two major phases. The first phase was to determine the chemical fingerprint for RON 95 petrol. While, phase two was to identify the differences in petrol composition of RON 95 petrol from different service station for each petrol supplier. This study was conducted by taking samples from five different stations of three major petrol supplies in the Klang Valley. A standardized sampling technique was adopted during the collection of the fuels.

3.2 ASSUMPTIONS

There are some assumptions made in this study, which were:

i. Sampling was done in the morning to avoid the volatilization of the petrol.

ii. Glass bottle and volumes of the petrol were standardized for all service stations.

iii. Selected petrol stations in Kuala Lumpur area were the ones closest to the analysis laboratory (UKM KL)

iv. All samples analysis was conducted at room temperature.

3.3 MATERIAL

3.3.1 Materials and chemicals

RON 95 petrol was purchased from five different selected service stations of Petronas, Shell and BHP within three a days period. HPLC grade hexane was used as a diluent solvent. The chemicals were obtained from Fisher Scientific (UK). Equipment used for sample analysis had included Glass bottle 500 ml (Schott Duran, UK), 500 ml beaker (Schott Duran, UK), universal bottle, 100 ml measuring cylinder, 100 µl micropipette (Pipetman Gilson, USA), and vials of 1.5 ml (Fisher Scientific, UK).

3.3.2 Instrument

HP 7890A gas chromatography (Agilent, USA) with flame ionization detector was used in this analysis. The analysis on the GC was carried out on a HP-5 capillary column (30m x 0.32mm ID x 0.25 µm, HP-5 J&W Scientific®). Ultrapure helium was used as the gas carrier.

3.4 METHOD

3.4.1 Sampling

First phase of this experimental research refers to the specific objective of to the first three. First, experimental was done to determine the chemical fingerprints of RON 95 petrol from three commercial brands. Samples were collected from selected service station that was located nearby to the laboratory in UKM KL. The detail of the selected petrol (Petronas, Shell and BHP) service station is shown in Table 3.1. The second

phase of experiment had involved the use of the GC-FID data obtained from samples in 1st phase.

Table 3.1: Selected service station for Petronas, Shell and BHP RON 95 petrol

No	Sample	Date Collected	Location	Appearance
1	Petronas A	2 Feb 2012	Jln. Tun Razak	Light Yellow
2	Petronas B	2 Feb 2012	Jln. Raja Muda Abdul Aziz	Light Yellow
3		2 Feb 2012	Jln. Genting Klang	Light Yellow
4	Petronas C	2 Feb 2012	Jln. Sentul	Light Yellow
5	Petronas D	2 Feb 2012	Bt. 3, Jln. Gombak	Light Yellow
6	Petronas E	31 Jan 2012	Jln. Pahang	Light Yellow
7	Shell A	2 Feb 2012	Jln. Tun Razak	Light Yellow
8	Shell B Shell C	2 Feb 2012	Jln. Tunku Abd. Rahman	Light Yellow
9	Shell D	2 Feb 2012	Jln. Gombak	Light Yellow
10	Shell E	2 Feb 2012	Jln. Genting Klang	Light Yellow
11	BHP A	2 Feb 2012	Tmn. Titiwangsa	Light Brown
12	BHP B	2 Feb 2012	Jln. Genting Klang	Light Brown
13	BHP C	2 Feb 2012	Setapak	Light Brown
14	BHP D	2 Feb 2012	Bt. 3 Jln. Gombak	Light Brown
15	BHP E	2 Feb 2012	Jln. Sentul	Light Brown

3.4.2 Sample preparation

A volume of 500 ml petrol samples were collected from 15 service stations in the Klang Valley area over a period of three days (Table 3.1). The samples were then stored in glass bottle covered by aluminum foil at room temperature. Samples were then prepared for GC-FID analysis by diluting an aliquot of the gasoline with hexane in a ratio 1:4 (Sandercock & Pasquire 2004). To ensure no loss of compounds within the samples, dilution was done just prior to analysis. Sample was shaken for 1 minute. Then 100 µl of the diluted samples were then transferred into a chromatography vial and capped. Each sample was extracted in triplicate.

3.4.3 Gas Chromatography – Flame Ionization Detector (FID)

All analysis was performed using an Agilent 7890A Agilent gas chromatography with flame ionization detector (Agilent, USA). The GC-FID was equipped with a HP-5 capillary column (30m x 0.32mm ID x 0.25 µm, HP-5 J&W Scientific®). For analysis, a single temperature ramp was used with split injection (100:1) at 250°C. The detertor was set at 250°C. The initial temperature was 35°C which was held for 15 min. The temperature was increased from 35°C to 200°C at the rate of 2°Cmin^{-1}. This was followed up by holding time 5 min. The total run time was 102.5 min. The injection volume was 1.0 µl and helium was used as a carrier gas at 30 ml/min. The carrier gas had a constant flow rate of 1.2ml/min.

Table 3.2: The sequence of samples injected into the GC-FID

No.	Sample	Description
1	Hex	Hexane
2-4	Petronas A	Petrol from Petronas service station
5-7	Petronas B	Petrol from Petronas service station
8-10	Petronas C	Petrol from Petronas service station
11-13	Petronas D	Petrol from Petronas service station
14-16	Petronas E	Petrol from Petronas service station
17-19	Shell A	Petrol from Shell service station
20-22	Shell B	Petrol from Shell service station
23-25	Shell C	Petrol from Shell service station
26-28	Shell D	Petrol from Shell service station
29-31	Shell E	Petrol from Shell service station
32-34	BHP A	Petrol from BHP service station
35-37	BHP B	Petrol from BHP service station
38-40	BHP C	Petrol from BHP service station
41-43	BHP D	Petrol from BHP service station
44-46	BHP E	Petrol from BHP service station

3.4.4 Statistical analysis

Prior to the statistical analysis, resolution of the data has been reduced from 0.01 second to 0.05 second to form a standard block retention time. This standard retention

time were selected because there are a lot of data derived from GC-FID analysis and the other hand all data will be more manageable.

All GC-FID data has been analysis by using chi-square test, principle component analysis (PCA) and QUEST modeling analysis. Firstly, data was performed a chi-square test to identify and observe the peak frequencies for all three brands petrol based on their location of service station. Both of the chi-square test and PCA analysis was done using SPSS Statistics version 20 software (IBM, USA).

PCA analysis is a descriptive method to reduce the dimension of factors number pertinent variables. This method has been done by changing data set to the main components were arranged in descending order to the most relevant probability data. Based on GC-FID data set, an assessment of the probability of the most the relevant peaks can be identified by PCA. List of standard retention times were arranged in ascending order with their matching peak area. After PCA were carried out, the main component of the projection can be obtained. Each major component are associated with the eigenvalue. The eigenvalue represent the uniqueness of the data. The higher eigenvalue give more variation (Doble, P. et al. 2003).

A standard retention time and corresponding peak area were horizontally arranged in ascending order in SPSS 20. First of all, on the analyze menu, dimension reduction were chosen and followed by factor analysis. Then, on the descriptive menu, univariate descriptive, initial solution, coefficients, KMO and Bartlett's test of sphericity and anti-image were selected. Next, correlation matrix, unrotated factor solution, scree plot and based on eigenvalue greater than 1 were chosen in the extraction menu while on the rotation menu, varimax and rotated solution were selected. Finally, exclude cases listwise and sorted by size on the options menu. PCA

will provide a result of total variances explained and matrix components for the results interpretation.

The third statistical analysis has been used in this study is QUEST modeling. This model was selected to identify the brand of the petrol based on the standard retention time from the PCA analysis. Before execute all data, first of all on the reduction menu, variable, location, replicate and injection were selected. Then, on the type menu, all input from company which is representing the brand of petrol were chosen. After that, on the partition menu, analysis was set at 50% of the data for training, 40% for testing and 10% of the data for the validation. All data were randomly selected by the software. QUEST modeling will provide a result of percentage for the three partitions above.