CHAPTER III

EXPERIMENTAL

3.1 Apparatus and instruments

1. High Vacuum pump

   Edward's model E2M2 20035

2. Non-Dispersive Infrared Analyser (NDIR)

   Thechno test model 483

3. Timing Light

   Techno test model 136.

4. High performance Liquid Chromatography.

   Waters 800.

3.2 Reagents and Their Purification.

1. Formaldehyde

   Analysis grade (36%); Merck

2. Acetaldehyde

   Analytical grade (99.5%); Mecrk

3. Acetone

   Analytical grade (99%); Merck

4. Propionaldehyde

   Analytical grade (85%); Merck

5. 2-Butanone

   Analytical grade (98%); Merck

6. Crotonaldehyde

Analytical grade (98%); Merck

7. Benzaldehyde

Analytical grade (99%); Merck

8. Acetonitrile

HPLC grade (99.99%); Merck

9. 2,4-Dinitrophenylhydrazine

Analytical grade (99%); Merck

10. Dispersant Additive for Gasoline

LZ 8195; Lubrizol

3.3 Engine Description.

The specifications of the test engine are shown in Table 3.1. Prior to testing, oil filter and engine lubricant oil were changed. PTT VSH Performa lubricating oil was used throughout the study. The test engine was run for 50 minutes with the new lubricating oil before sampling commenced.

Table 3.1 Description of the test engine.

Make/Style	Toyota Corolla
Year	1982
Number of cylinder	4
Displacement volume	1500
Fuel system	Carburettor
Emission system	Exhaust Gas Recirculation (EGR)

3.4 Fuel Description.

The test fuels used in this study were unleaded gasoline base fuel (ULG 98), delivered from Petroleum Authority of Thailand (PTT),and blended unleaded gasoline base fuels with a dispersant additive. The dispersant was commercially LUBRIZOL LZ8195 package at treat rates of 5 dosages, 300, 400, 500, 600, and 700 ppm (v/v). The test fuels were summarised in Table 3.2.

Table 3.2 Description of fuel

1. Unleaded gasoline base fuel	-
2. Unleaded gasoline base fuel blended with LZ8195 (dispersant)	300 ppm
3. Unleaded gasoline base fuel blended with LZ8195 (dispersant)	400 ppm
4. Unleaded gasoline base fuel blended with LZ8195 (dispersant)	500 ppm
5. Unleaded gasoline base fuel blended with LZ8195 (dispersant)	600 ppm
6. Unleaded gasoline base fuel blended with LZ8195 (dispersant)	700 ppm

3.5 Analysis of CO and HC in Exhaust Emission. (22)

The CO and HC in exhaust emission were measured with a Non-Dispersive Infrared Analyser (NDIR). The tests were at least done for five times at each condition of engine (timing and speeds) and fuel that was blended with dispersant at each concentration

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3.6 Analysis of individual aldehydes and ketones in exhaust emission.(21)

Measurement of individual aldehydes and ketones measurement were made, by passing exhaust emission through 2,4-dinitrophenylhydrazine reagent. Aldehydes and ketones have a carbonyl group that reacts with 2,4-dinitrophenylhydrazine to give 2,4-dinitrophenylhydrazone derivatives, which precipitated separately from solution. The residue was filtered and dried, then dissolved in 20 ml of acetonitrile. All of samples were analysed by HPLC.

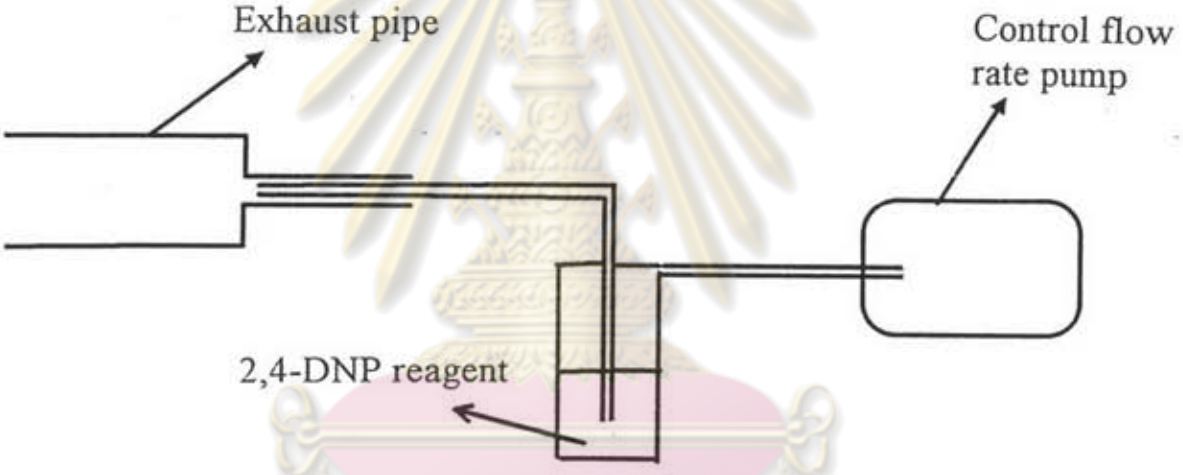

Figure 3.1 Schematic of sample collection for analysis.

3.7 HPLC Condition

The HPLC analyses were performed on waters 800 HPLC, equipped with a UV detector.. The instrument conditions are reported in Table 3.3

Table 3.3 Summary of HPLC Condition.

Column:			
Dimension	125x40 mm		
Pack. Mat.	Nucleosil 100 C-18		
Condition:			
Time (min)	Flow rate (ml/sec)	(%) Acetonitrile	(%) H_2O
initial	0.5	67	33
8	0.8	67	33
9	0.8	85	15
15	1.0	100	0
18	1.0	85	15
19	1.0	67	33
20	1.0	67	33
UV-detector 365 nm			

3.8 Preparation of Solution and Standard.

The reagent, a 0.25 M. solution of 2,4-dinitrophenylhydrazine may be used for the preparation of derivatives of carbonyl compounds. 25 g. of 2,4-dinitrophenylhydrazine was dissolved in 300 ml of 95% sulphuric acid in a 600 ml beaker on steam bath,after it was diluted the solution with 200 ml of 70 % ethanol, allow to stand and filter through a sintered glass funnel.

Standard solutions of aldehydes and ketones were prepared by injecting 0.5 ml of the aldehydes and ketones into vial containing the derivatizing agent. The hydrazones precipitated from solution. The residue was filtered and dried, then dissolved in 20 ml acetonitrile.. The

concentrations of the stock solution were calculated from the density of the aldehydes and ketones

3.9 Compound Identification.

The individual aldehydes and ketones were analyzed as 2,4-dinitrophenylhydrazone derivatives, that could be identifies by comparing theis HPLC retention times with these obtained from known 2,4-dinitrophenylhydrazone derivatives of formaldehyde, acetaldehyde, acetone, propionaldehyde crotonaldehyde, 2-butanone, and benzaldehyde. The HPLC chromatogram of 2,4-dinitrophenylhydrazone derivative standards is shown in Figure 3.2. The identification of the components is presented in Table 3.4

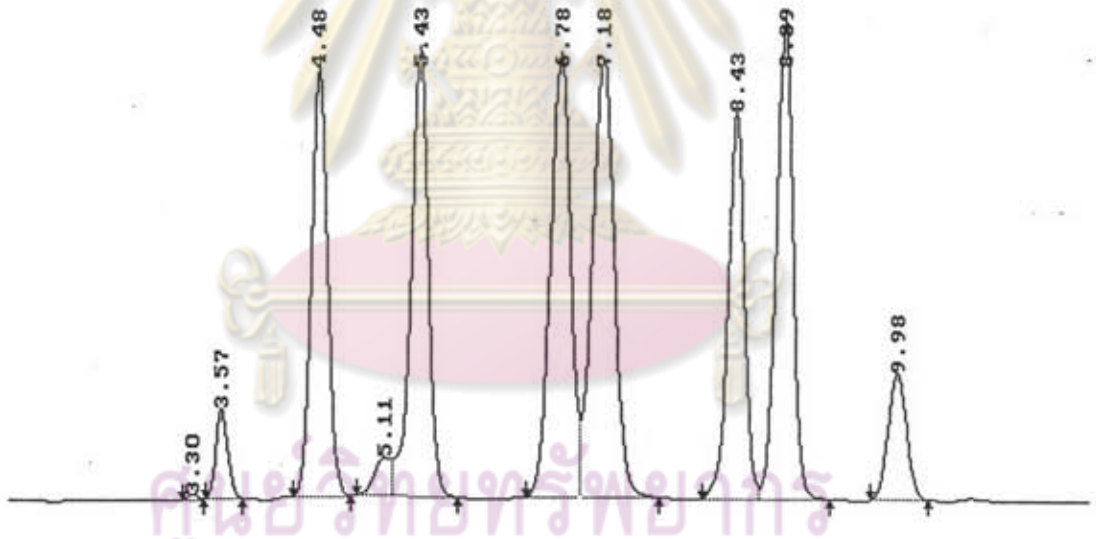

Figure 3.2 HPLC Chromatogram of 2,4-dinitrophenylhydrazone derivative of standards

Table 3.4 Retention time of standards

2,4-dinitrophenylhydrazone derivative of standards.	Retention time (min)
Formaldehyde	4.48
Acetaldehyde	5.43
Acetone	6.78
Propionaldehyde	7.18
Crotonaldehyde	8.43
2-Butanone	8.89
Benzaldehyde	9.98

3.10 .Calibration and calculation of aldehyde and ketone concentrations

Standard solutions were analysed, under the same HPLC condition and during the same time period as the sample, to construct the calibration curve (Appendix B).

The concentration of the analytes in the exhaust emission can be expressed in mg/m^3 by the follow in equation.

A = Air volume sample (m^3) = F x $t/10^3$

F = flow rate (l/min)

t = time (min)

B = Concentration of sample (mg/20ml)

= C x $20/10^3$

C = concentration of sample (ppm)

20 = volume of sample (ml)

mg/m^3 = Concentration of sample/Air volume of sample