

CHAPTER III
EXPERIMENTAL

3.1 Chemicals

The chemicals used in this experiment are listed in Table 3.1.

Table 3.1 List of selected hydrocarbons in this experiment

Chemicals	Brand	Purity
<i>n</i> -pentane (C ₅ H ₁₂)	Labscan	99% min, AR
<i>n</i> -hexane (C ₆ H ₁₄)	Labscan	99% min, AR
<i>n</i> -heptane (C ₇ H ₁₄)	J.T. Baker	98.5% min, AR
<i>n</i> -octane (C ₈ H ₁₆)	Labscan	99% min, AR
<i>n</i> -decane (C ₁₀ H ₂₂)	Fluka	purum ≥ 98.0% (GC)
3-methylpentane (C ₆ H ₁₄)	Fluka	purum ≥ 99.0% (GC)
2,2,4-trimethylpentane (C ₈ H ₁₆)	Labscan	99.5% min, AR
Toluene (C ₇ H ₈)	Labscan	99.9% min, AR
Dichloromethane (CH ₂ Cl ₂)	Merck	99.5% min, AR
Methanol (CH ₃ OH)	Labscan	99.8% min, AR
Ethanol (C ₂ H ₅ OH)	Merck	99.5% min, AR
Acetone (C ₃ H ₆ O)	Labscan	99.5% min, AR
Elemental mercury (Hg ⁰)	Wendt-Chemie	99.9995%

N-pentane, *n*-hexane, *n*-heptane, *n*-octane, *n*-decane, 3-methylpentane, and 2,2,4-trimethylpentane (isooctane) were used in the solubility study. Other organic solvents were used to re-dissolve the fine black particles occurred from unexpected change of mercury drop in 3-methylpentane.

3.2 Sample Preparation

The solubility experiment was performed by shaking the 1-dram glass vial containing hydrocarbon solvent (2.5 ml) and a drop of elemental mercury (approx. 2 mm) in a shaker immersed in a controlled-temperature water bath with a temperature controller with $\pm 0.01^\circ\text{C}$ accuracy. The shaking speed of 55 revolutions per minute in a reciprocal way was controlled by a regulator voltage at $\pm 0.5\text{ V}$. The picture of the experimental apparatus is shown in Figure 3.1.

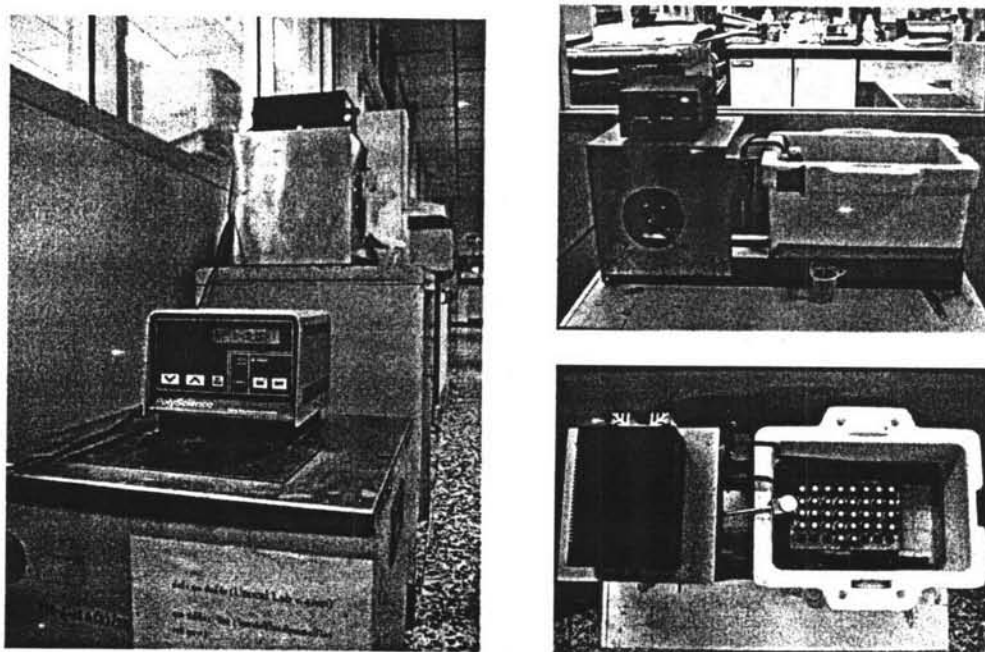


Figure 3.1 Experimental apparatus for mercury solubility study.

3.3 Equilibration Time Determination

The purpose of this experiment was to find the time for mercury to reach its equilibrium in solvents. The representative solvents for normal- paraffins and branched- paraffins in this experiment were *n*-octane and 2,2,4-trimethylpentane (isooctane), respectively. The equilibrating temperature was set at 5°C due to the fact that temperature is one of the factors controlling the rate of dissolution and the longer time is required to reach the solubility equilibrium at low temperature than

high temperature. The obtained result was set as an equilibration time for the remaining experiments.

3.4 Effect of Mercury Concentration in Headspace and Suspended Mercury on Solubility Study

3.4.1 Effect of Mercury Concentration in Headspace on Solubility Study

This experiment was aimed to measure the mercury concentration in vapor phase over the liquid level because 1-dram vials containing half the amount of hydrocarbon solvent were used throughout this research. *N*-pentane and *n*-hexane were selected to determine the mercury concentration above liquid phase due to their highest vapor pressure among the solvents used in this work. Equilibrating temperature at 40°C was used to study because high temperature generates large amount of vapors easily. The vapor phase over the solvent was sampled to check the mercury concentration by mercury analyzer (model SP-3D, Nippon Instruments Corporation).

3.4.2 Effect of Suspended Mercury on Solubility Study

In addition to study the effect of mercury concentration in headspace, fine and suspended metal mercury may exist during shaking and affect the solubility data. A test for proving the presence of fine and suspended metal mercury was performed. *n*-Heptane solution, which was equilibrated at 25°C with metal mercury as described in the sample preparation step, was analyzed for mercury concentration before and after filtering through 0.22 μm filter.

3.5 Mercury Solubility Study

Mercury solubility in hydrocarbon solvents study was conducted by shaking the sample at the desired temperature until it reached the equilibrium (more than 20 hours). The studied temperatures were 5, 15, 25, and 40°C. The experiments were separated into two parts as follows.

3.5.1 Single Solvent System

The elemental mercury was equilibrated in each solvent individually.

3.5.2 Mixed Solvent System : Simulated Condensate

The experiment was carried out in the same way as the single solvent system, but the mixture of hydrocarbons was used to simulate the actual condensate instead. The break down components of simulated condensate were obtained from the company. However, the ratio of mixing was based on the available hydrocarbon solvents previously studied in the solubility experiment. Table 3.2 presents the composition of simulated condensate.

Table 3.2 Composition of simulated condensate

Composition	Percent by Weight (%)
<i>n</i> -pentane (C ₅ H ₁₂)	16.388
<i>n</i> -hexane (C ₆ H ₁₄)	8.760
cyclohexane (C ₆ H ₁₂)	6.704
<i>n</i> -heptane (C ₇ H ₁₄)	6.120
methylcyclohexane (C ₇ H ₁₄)	8.651
toluene (C ₇ H ₈)	4.844
<i>n</i> -octane (C ₈ H ₁₈)	2.446
2,2,4-trimethylpentane (C ₈ H ₁₈)	6.003
<i>o</i> -xylene (C ₈ H ₁₀)	4.788
<i>n</i> -decane (C ₁₀ H ₂₂)	35.296
<i>Total</i>	<i>100</i>

3.6 Hysteresis Study on Mercury Solubility

Hysteresis study of mercury solubility was performed by heating the solution to above the desired temperature and cooling down to the desired temperature. Solvent with metallic mercury was heated and maintained at 40°C for

more than 20 hours, followed by decreasing temperature to a desired temperature at 25, 15, and 5°C. Then, the solution was left equilibrating for more than 20 hours before sampling to analysis. Hysteresis study was conducted both in a single solvent system and a mixed solvent system (simulated condensate).

3.7 Mercury Concentration Measurement

Approximate 60-120 μL of mercury solution was sampled from each vial by a microsyringe, injected to the sample boat containing the additives, and weighed on a 4-digit balance with its precision within ± 0.3 mg. The sample amount was recorded in the computer of mercury analyzer (model SP-3D, Nippon Instruments Corporation) for automatic analysis of the mercury concentration following the method UOP 938-00.

The sample was decomposed by heating in the mercury atomizer unit. The mercury vapor in the gaseous product was then collected by amalgamating with gold inside the first mercury collector at 150°C to prevent the adsorption of the combustion products. After that, the amalgamated mercury was heated to generate mercury vapor and again re-amalgamated on a second collector. Upon completion of the two-step amalgamation process, the mercury was released in the form of elemental mercury by heating the collector at 700°C and carried to an absorption cell in the mercury detector. The atomic mercury vapor was detected at 253.7 nm absorption wavelength. After introducing the sample into the combustion chamber, all steps were done automatically by the computer of mercury analyzer.