

## CHAPTER III EXPERIMENTAL

### 3.1 Chemicals

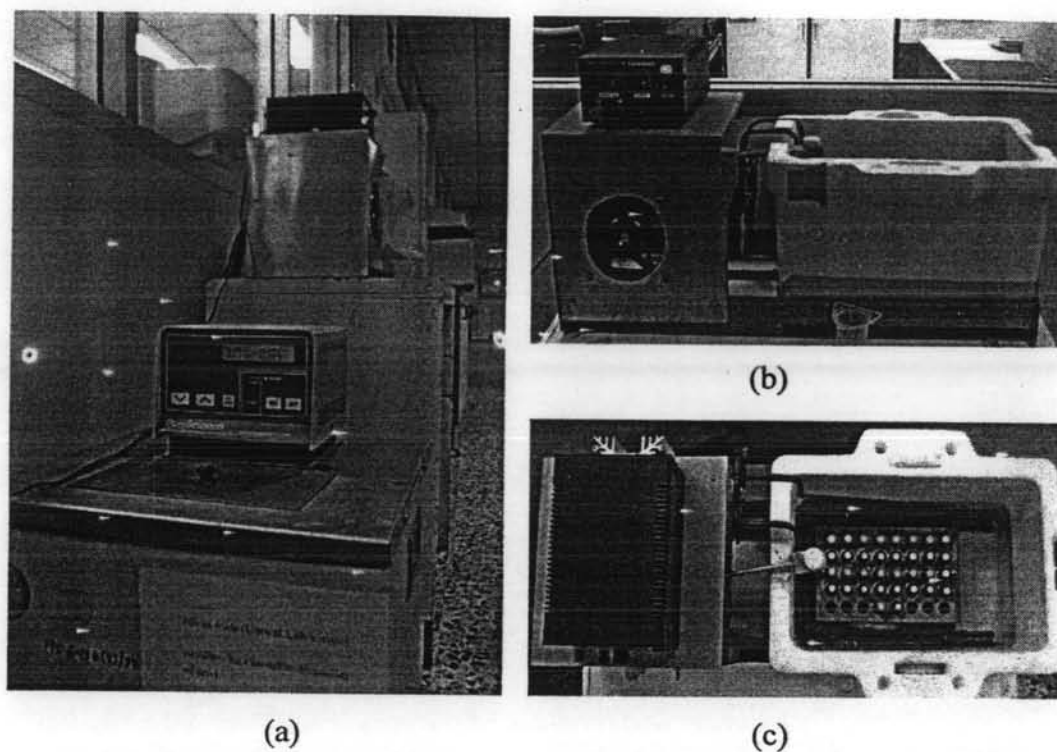
The hydrocarbons used in this study were cyclic aliphatic and aromatic hydrocarbons namely cyclohexane, methylcyclohexane, toluene, *o*-xylene and ethylbenzene. Two systems were prepared, single solvent systems and simulated crude oil. All solvents were analytical grade and 99.9995 % elemental mercury was used. The detail of these solvents and mercury are tabulated in Table 3.1.

**Table 3.1** Specification of hydrocarbons and mercury in this study

Solvent	Brand name	% Purity
Cyclohexane (C <sub>6</sub> H <sub>12</sub> )	Labscan	99.00
Methylcyclohexane (C <sub>7</sub> H <sub>14</sub> )	Labscan	98.00
Toluene (C <sub>7</sub> H <sub>8</sub> )	J.T.Baker	99.90
<i>o</i> -Xylene (C <sub>8</sub> H <sub>10</sub> )	Fluka	98.00
Ethylbenzene (C <sub>8</sub> H <sub>10</sub> )	Fluka	98.00
Elemental mercury	Wendt-Chemie	99.9995

### 3.2 Sample Preparation

Samples were prepared by dropping elemental mercury (about 2 mm size) using a plastic disposable pipette into 2.5 ml solvent in a teflon-septa sealed glass vial. The vials were shaken in a shaker bath at constant speed of 55 rpm in a reciprocal manner at a controlled temperature. The shaking speed was controlled by a regulator with a good precision control of  $\pm 0.5$  V. The temperatures were in the range of 5 to 40 °C with precision  $\pm 0.01$  °C. The setup of shaker and the temperature controlled bath is illustrated in Figure 3.1.



**Figure 3.1** The experimental apparatus: (a) the controlled-temperature waterbath, (b) and (c) the shaking device.

### 3.3 Equilibration Time Determination

The equilibration time was determined before studying the solubility. Methylcyclohexane and toluene representing cyclic aliphatic and aromatic, respectively were selected and investigated for the mercury concentration as a function of time at the lowest temperature (5°C). The obtained results were used in the solubility study in all experiments both increasing and decreasing temperatures.

### 3.4 Mercury Solubility in Single Solvent

The solubility of mercury in single solvents was studied at operating temperatures of 5, 15, 25 and 40 °C for over 20 hours. In the case that the mercury analyzer was not ready, the equilibrated solutions were left in the controlled-temperature water bath longer than the equilibration time. In this experiment, two

possible factors were suspected to affect the accuracy of mercury solubility, i.e. mercury in headspace and suspended mercury. To check mercury in the vapor phase, the highest vapor pressure of cyclohexane was equilibrated at 40°C for 20 hours. For suspended mercury in the liquid phase, the equilibrated solution was filtered through a filtering medium 0.22 micron before performing mercury analysis.

### 3.5 Mercury Solubility in Simulated Crude Oil

The procedure conducted was the same as the single solvent study but solvent mixture was used instead. The simulated crude oil was designed in such a way that it resembled the actual crude oil composition and the weight fraction is shown in Table 3.2.

**Table 3.2** The composition of simulated crude oil

Component	% by Weight
<i>n</i> -Pentane (C <sub>5</sub> H <sub>12</sub> )	2.98
<i>n</i> -Hexane (C <sub>6</sub> H <sub>14</sub> )	1.84
Cyclohexane (C <sub>6</sub> H <sub>12</sub> )	1.39
<i>n</i> -Heptane (C <sub>7</sub> H <sub>16</sub> )	1.90
Methylcyclohexane (C <sub>7</sub> H <sub>14</sub> )	1.37
Toluene (C <sub>7</sub> H <sub>8</sub> )	0.43
2,2,4 Trimethylpentane (C <sub>8</sub> H <sub>18</sub> )	1.87
<i>n</i> -Octane (C <sub>8</sub> H <sub>18</sub> )	1.11
<i>o</i> -Xylene (C <sub>8</sub> H <sub>10</sub> )	0.38
<i>n</i> -Decane (C <sub>10</sub> H <sub>22</sub> )	86.73
Total	100

### 3.6 Hysteresis Study

Hysteresis was studied in both the single solvent systems and simulated crude oil. The mercury drop in hydrocarbon solvent was firstly equilibrated at high temperature over 20 hours and then equilibrated at decreasing temperature for another 20 hours. The equilibrated temperatures were as follow: 40°C to 25°C, 25°C to 15°C and 15°C to 5°C. It was necessary to use the same samples for the whole run.

### 3.7 Mercury Measurement

Mercury solubility in liquid phase was analyzed by mercury analyzer NIC (SP-3D) followed the UOP 938-00 method. The sample solution (50-100  $\mu$ l) was withdrawn using a microsyringe through the septum seal, and then transferred to a sample boat containing NIC additives. The sample was weighed on a 4-digit balance with the precision of  $\pm 0.3$  mg and quickly introduced into the NIC mercury atomizer unit to be decomposed by heating. The mercury vapor in the combustion gas is then collected by amalgamation on a gold collector at 150 °C to prevent the adsorption of the combustion gas. Subsequently, the amalgam is heated to release mercury vapor to a second gold collector for the second amalgamation. After two-step amalgamation is complete, the second amalgam is heated to 700 °C to release elemental mercury vapor to absorption cell for atomic absorption at wavelength of 253.7 nm. For the measurement of mercury in vapor phase (headspace), the gas tight syringe was used to withdraw the vapor ( $300 \pm 5 \mu$ l) above the liquid in the vial and subsequently injected into the gold amalgam trap to collect mercury in gas phase. After that, the amalgam tube was heated to release mercury and then subjected to repeat the same analytical steps as for the liquid sample. The analytical calibration curve in the range of 0-20 ng and 0-200 ng of NIC was performed weekly with a mercury standard solution ( $\text{Hg}(\text{NO}_3)_2$ ,  $1000 \pm 5 \mu\text{g/L}$ ). The linearity of 0.9995 fitting coefficient with precision of 4.4% at 20 ppb level is obtained. Then, the calibration curve is checked by conostan mercury standard ( $100 \pm 1$  ppm by weight). All analysis was done 3 times.