CHAPTER III
EXPERIMENTAL

3.1 Materials and Equipment

3.1.1 Chemicals

3.1.1.1 Aromatic Hydrocarbons

Benzene (C_6H_6) of minimum 99.8% purity with density of 0.878 g/ml was obtained from Scharlau Chemie.

Toluene ($C_6H_5CH_3$) of 99.5% purity with density of 0.867 g/ml was obtained from LabScan Asia CO, Ltd.

p-Xylene ($C_6H_4(CH_3)_2$) of 99% purity with density of 0.861 g/ml was obtained from BDH Laboratory Supplies.

3.1.1.2 Normal Paraffin Hydrocarbon

n-Hexane ($CH_3(CH_2)_4CH_3$) of 96 % purity with density of 0.659 g/ml was obtained from Scharlau Chemie.

3.1.1.3 Solvents

Sulfolane of was purchased from Fluka Co., Ltd. with 98.0% purity and density of 1.27 g/ml.

Dimethylmethylsulfoxide (DMSO) was purchased from Fluka Co., Ltd. with 99.9% purity and density of 1.100 g/ml.

3-Methoxypropionitrile (3MOPN) was purchased from Fluka Co., Ltd. with 99% purity and density of 0.939 g/ml.

Ethylene carbonate (EC) was purchased from Fluka Co., Ltd. with 99% purity and density of 1.33 g/ml.

Ethylene glycol (EG) was purchased from Fluka Co., Ltd. with 98.0% purity and density of 1.113 g/ml.

3.1.1.4 Others

Acetone (CH_3COCH_3) of 99.5% purity was obtained from LabScan Asia CO, Ltd.

Sodium sulfate anhydrous (Na_2SO_4) was purchased from Fluka Co., Ltd.

These chemicals were employed with no further purification.

3.1.2 Equipment

Gas Chromatograph (GC/ FID) was supplied by Agilent Technologies, 6890N model, and serial number is US1026136. The gas chromatography is equipped with a flame ionization detector (FID).

Coulometer was supplied by Metrohm, the 737KF model Coulometer was used to analyze the water contents in the solvents.

Stirrer/ Heaters were obtained from Mivaris and were used at 807 rounds per minutes. The heater function is not included in the experimental part.

Water pumps were obtained from March Manufacturing, Inc. They were used for water circulating to control the extraction temperature.

Controlled-temperature water bath was supplied by Memmert and was used to heat the circulated water.

Mercury Thermometers were in the range of 0 ° to 200 °C

Syringes were supplied by Agilent Technologies as a 10 μl syringe for the injection of the gas chromatography. The 100 μl syringes were supplied by SGE.

3.2 Experimental Procedures

3.2.1 Determination and Preparation of Water Contents in the Solvents

The water content in the solvents causes changing in the capacity and selectivity of the solvent extraction so the solvents were prepared so that they contained the same amount of water. The water contents were analyzed by using the coulometric Karl Fischer titration method with a Metrohm 737KF Coulometer. Because the water contents were very low, sampling vials and syringes had to be desiccated before each test. After that, sodium sulfate anhydrous was used.

3.2.2 Sample Preparation

The extraction feed were made up of n-hexane as paraffin and varied aromatics of benzene, toluene, and *p*-xylene to determine the effect of each aromatic on the solvents. The compositions of the extraction feed were made up of aromatic of 0.57 mass fraction and n-Hexane of 0.43 mass fraction as referred to the composition of naphtha reformate used in the extraction by tetraethylene glycol (Al-Sahhaf and Kapetanovic, 1996).

3.2.3 Determination of Critical Solution Temperature

The miscibility was measured by completely mixing a known volume of solvent with a known volume of feed by a magnetic stirrer, followed by heating up gradually to meet the point where the turbidity of the mixture disappeared. The temperature was read. The heating was then stopped with continued stirring until it became turbid. Temperature reading was made once the mixture became turbid. These two readings should not differ more than 1°C. The critical solution temperatures were used to set the operating temperature of the extraction that should be 25 °C lower than the critical solution temperature.

3.2.4 Solvents

The solvents used in this study are single solvent and mixed solvents:

3.2.4.1 Single Solvents

- Sulfolane
- Dimethylsulfoxide (DMSO)
- 3-Methoxypropionitrile (3MOPN)
- Ethylene carbonate (EC)
- Ethylene glycol (EG)

3.2.4.2 Mixed Solvents

- Dimethylsulfoxide (DMSO)/Ethylene carbonate (EC)
- 3-Methoxypropionitrile (3MOPN)/Ethylene glycol (EG)

3.2.5 Batch Extractions

The extraction of BTX aromatics was established to study the effects of 3 key parameters. They are; the solvent-to-feed ratio, operating temperature, and solvent choice. The batch extractions were carried out with 3 ratios of the solvent-to-feed ratio and 3 different operating temperatures for each solvent or solvent pair. The equipment set up was made up of an Erlenmeyer flask and a separatory funnel with a transparent heating jacket made from acrylic resin with circulating temperature-controlled-water to control the temperature both for the extraction and the separation process.

For different solvent-to-feed ratios, the extractions were carried out the volume of the feed and the solvent for each batch as shown in Table 3.1.

Table 3.1 Batch-extraction volume used for different solvent-to-feed ratios

Solvent-to-feed Ratio	Solvent (ml)	Feed (ml)	Total (ml)
1:1	20	20	40
2:1	30	15	45
3:1	30	10	40

Before the extraction run, the feed and the solvent were put to the extraction temperature for at least half an hour to make sure they were close to extraction temperature. After mixing, the mixture was stirred continuously during extraction. The mixture was then transferred to a separatory funnel placed in a controlled-temperature jacket with using the same circulating-water source used in the extraction process as shown in Figure 3.1. At the completion of settling process of 3 hours, the top and bottom phase were collected as the raffinate and extract phase respectively to calculate the mass balance of the sample as shown in Appendix B.

3.2.6 Extraction Analysis

The extraction feed, extract phase, and raffinate phase, obtained after settling, were analyzed by using gas chromatograph with FID detector. The amount

of aromatics and nonaromatics were used to calculate the solvent capacity and selectivity to compare the efficiency of the solvents.

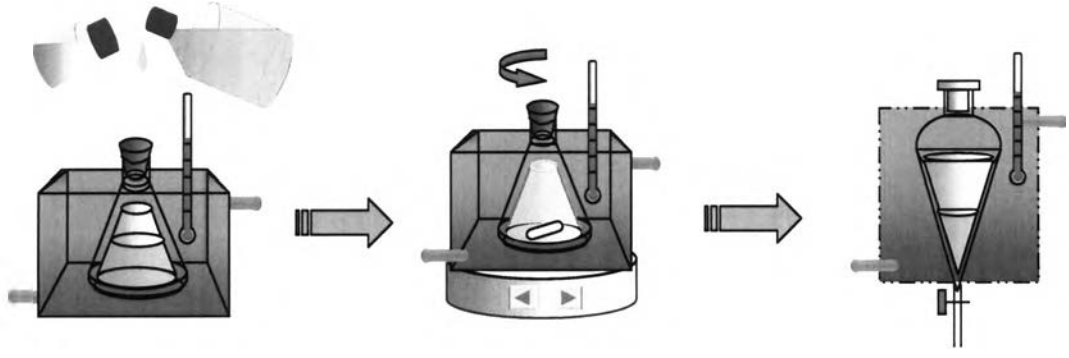

Figure 3.1 Batch aromatics-extraction set up.

Raw data, calibration of each hydrocarbon, and a sample of calculation are complied in Appendices A, B, and C, respectively.