

## CHAPTER III

### EXPERIMENTAL

#### 3.1 Materials

##### 3.1.1 Chemicals

- *m*-chloronitrobenzene 99.0 % purity, Italmar
- *p*-chloronitrobenzene 99.5 % purity, Italmar
- *n*-hexane 99.0 % purity, Acros

##### 3.1.2 Adsorbents

- Alkali and alkali earth ion-exchanged faujasite zeolites were obtained from UOP, A Honeywell Company, USA.
- Chemical compositions: KY ( $K_{51}Na_2(AlO_2)_{53}(SiO_2)_{139}$ )

#### 3.2 Equipment

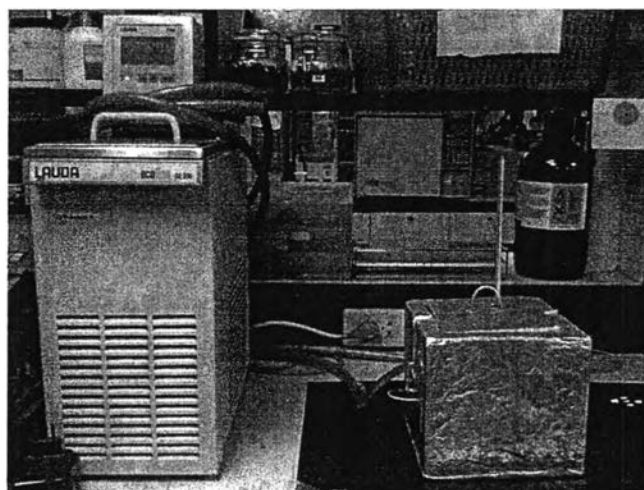
- Agilent 6890N series gas chromatograph (GC) equipped with a SUPELCOWAX<sup>TH</sup> capillary column and a flame ionization detector (FID)
- A cooling water bath
- A crystallization unit
- A heater
- An insulated chamber

#### 3.3 Methodology

##### 3.3.1 Effects of Feed Composition on *m*- and *p*-CNB Crystallization

- The feed solution was seven grams of *m*- and *p*-CNB with different *m*-CNB compositions consisting of below the eutectic, at the eutectic, and above the eutectic composition.
- Melted the solid mixture to obtain a homogeneous solution.

- Cooled the solution by the cooling water to the crystallization temperature, at which the crystal was initially formed.
- Collected the crystals wash, and dissolve with hexane.
- The dissolved crystals were analyzed for the CNB composition by the GC.

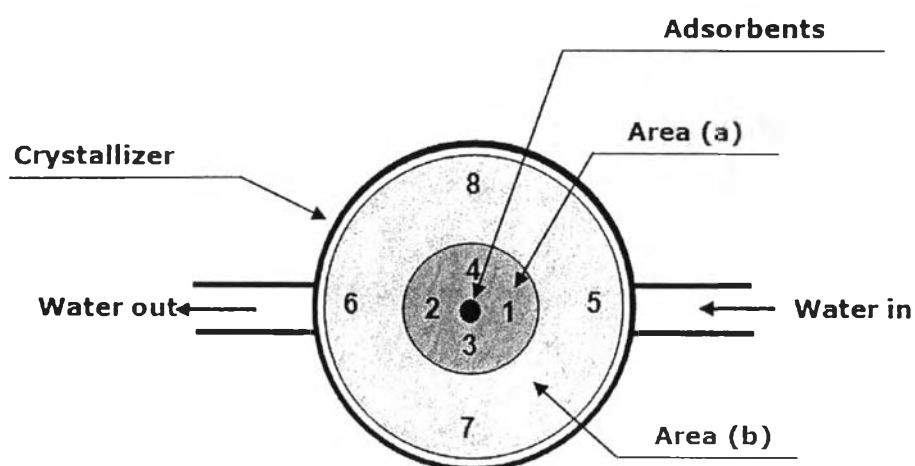


**Figure 3.1** Crystallization unit.

### 3.3.2 Effects of KY Zeolite on the Crystallization of *m*- and *p*-CNB

- A phase diagram study with the presence of KY zeolite on the crystallization was studied by using the feed solution of seven grams of *m*- and *p*-CNB with different *m*-CNB compositions below the eutectic, at the eutectic, and above the eutectic composition.
- Melted solid mixture to obtain a homogeneous solution.
- Calcined KY zeolite at 350 °C for an hour.
- Added five grains of KY zeolite at the center of the CNB mixture in the crystallizer and collected to check the composition after adding KY zeolite by the GC.
- Cooled the solution by the cooling water to the crystallization temperature, at which the crystal was initially formed.

- Collected the crystals from 8 positions in two areas, area (a) and area (b), as shown in Figure 3.2, washed, and dissolved with hexane.
- The dissolved crystals were analyzed for the CNB composition by the GC.



**Figure 3.2** Locations where crystals are collected for studying effect of the number of adsorbents on the crystallization.