

# CHAPTER III

## MATERIALS AND METHODS

### 3.1 Materials

#### 3.1.1 Fluorescent lamp samples

The 36 watt, used fluorescent lamps 4-foot long with a 1-inch diameter were used as the samples in this study. These lamps were obtained from a household hazardous waste exchange project generated by the Nakorn Khon Kaen Municipality. Dust and stains were removed by washing the lamps with a detergent solution followed by rinsing in tap water. The aluminum caps were carefully removed using a pair of needle-nosed pliers.

#### 3.1.2 Air sampling devices

In this study, the commercial sampling tubes, Anasorb®C 300 from SKC, Inc., recommended by OSHA and NIOSH, were applied to collect the mercury vapor emitted from crushed used fluorescent lamps. These tubes were 6 mm.O.D.x 70 mm. long and each could contain 200 mg of solid sorbent. The composition of this sorbent was mainly composed of oxides of manganese and copper (OSHA, 1991). To draw air through the sorbent tube, a personal sampling pump, Universal sample pump No.224-PCXR8, SKC, Inc., was employed. A low flow holder connected with the sampling pump was used to adjust the air flow rate to about 200 ml/min as recommended by OSHA and NIOSH (NIOSH, 1994). The air collection devices are shown in Figure 3.1.

#### 3.1.3 Crushing unit

A 230 liter HDPE drum available in the market was selected as the lamp residue container. A 1 mm. thick HDPE sheet was formed to serve as a hopper for collecting the lamp residue. A 5 mm. thick steel sheet was modified as the lid of the drum. A 6 cm. O.D.x 9 cm. long auger blade was made of steel. To drive the blade, a ¼ power horse motor was designed as its power source. A 1.5 inch diameter

pipe with a cap was used as the chute to insert the lamps into the unit. Several types of rubber were applied to seal the unit.



**Figure 3.1** Air sampling devices

### **3.1.4 Chemical spraying device**

A 3-liter commercial pressure sprayer was adapted for chemical spraying in this study. To spray tiny drops of sodium sulfide solution into the crushing unit, the nozzle of the sprayer was adjusted and fixed on the unit. Before spraying, the piston of the sprayer was slid up and down to produce high pressure in the bottle containing the solution. The solution was then sprayed when the spray valve was opened.

### **3.1.5 Instruments**

#### **3.1.5.1 Microwave digester**

To determine the amount of mercury in the lamp residue, the microwave digester model MARS X®, CEM corporation was applied to digest the lamp residue using method SW486-3051.

### 3.1.5.2 Rotary agitator

In the leachate extraction procedure, the rotary agitator (Overhead mixer) model Rotax 6.8 of VELP was employed to overturn the bottles containing the mixture of waste and synthetic acid rain extraction fluid.

### 3.1.5.3 Atomic absorption spectrophotometer

The atomic absorption spectrophotometer (hydride system) model FIMS 400, Perkin Elmer as shown in Figure 3.2 was used to analyze the concentration of mercury in all samples. The detection limit of this instrument is 0.01  $\mu\text{g Hg/l}$ .



**Figure 3.2** Atomic absorption spectrophotometer

## 3.2 Concept of the used fluorescent lamps management

The used fluorescent lamps consist of both recyclable and non-recyclable components. First, the aluminum caps of the lamps were removed for recycling. Next, the decapped lamp was ground by the crushing unit. After crushing, mercury vapor emitted from the crushed lamp was controlled by sodium sulfide spraying into the crushing unit. Finally, the lamp residue was solidified by cement. A diagram of the used lamp management is shown in Figure 3.3

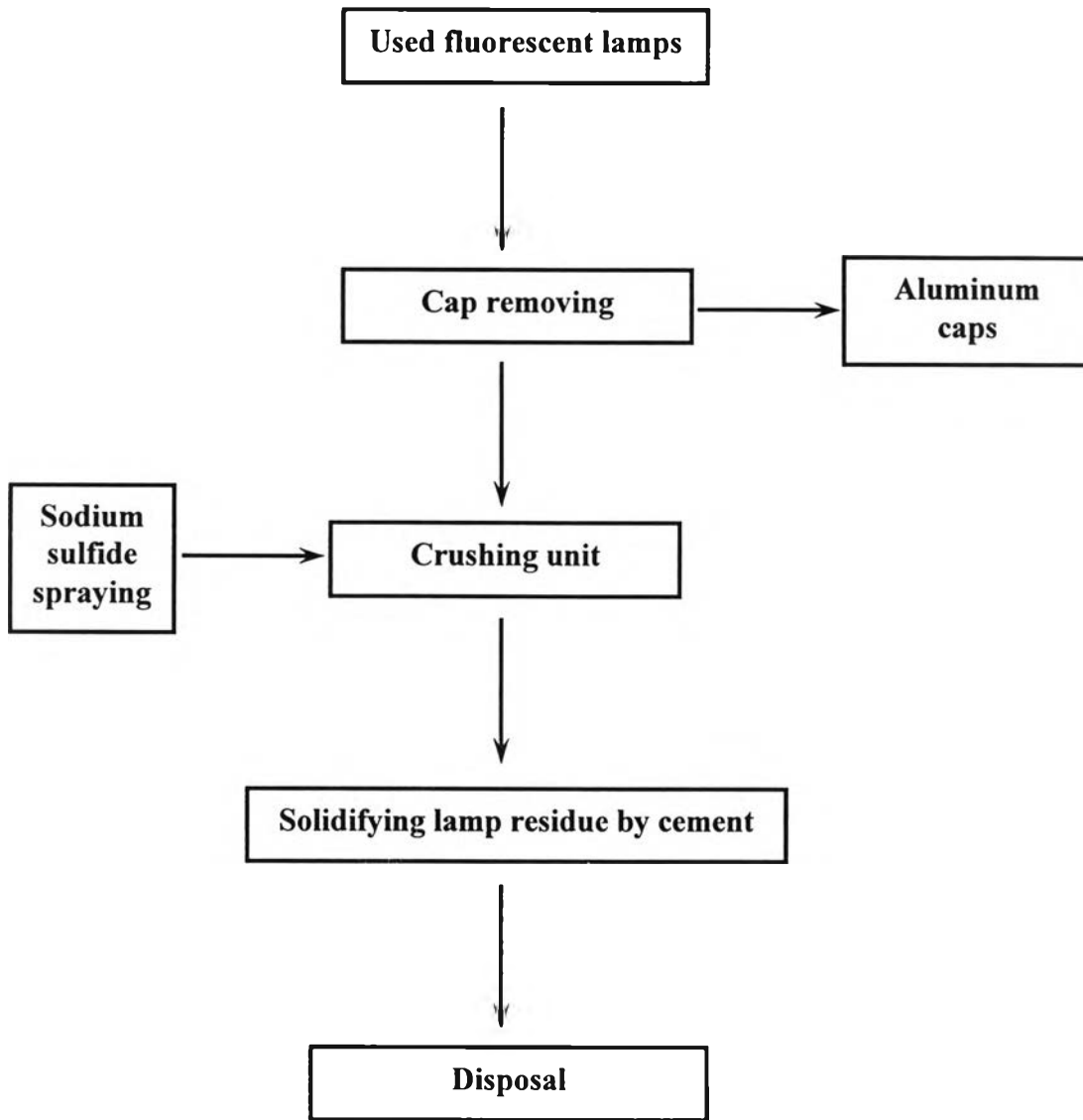


Figure 3.3 Diagram of the used fluorescent lamp management

### **3.3 Experimental procedures**

#### **3.3.1 Developing a fluorescent lamp crushing unit**

##### **3.3.1.1 Criteria for constructing a crushing unit**

- 1) The crushing unit must be a simple unit to be easily operated and moved.
- 2) The crusher can be separated from a container to use with other containers in the same size.
- 3) The crusher must support a 4-foot long, straight fluorescent lamp with 1-inch diameter (T-8 lamp) or smaller.
- 4) The crusher must be designed to reduce the volume of a used lamp as much as possible.
- 5) Every spare parts of the crushing unit must be available in the market.
- 6) The drum container must be in good condition to prevent mercury release into environments.
- 7) The drum container must make of plastics to reduce the contamination of mercury from crushed lamps and resist corrosive substances.
- 8) The capacity of the container must not be less than 200 liters.
- 9) Gaps on the crushing unit must be sealed to prevent the leakage of mercury vapor.

##### **3.3.1.2 Testing the crushing unit**

The crushing unit was tested by crushing the used lamps to find out the lamp feed rate. After crushing, the volume of each crushed lamp was measured to find out the reduction in lamp volume.

#### **3.3.2 Release of mercury from the crushed lamp**

In this step, there were three experiments.

##### **3.3.2.1 Mercury vapor sampling and preparation**

All mercury vapor samplings were collected following the OSHA's method ID-140. A sampling tube was connected to the pump using an adjusted low flow flexible tube. The sampling pump was calibrated at the flow rate of about 200 ml/min. And then the sorbent tube connected with the calibrated pump was inserted into the port. Next, a used fluorescent lamp was inserted and crushed by crushing

unit. The air inside the unit was sampled for 15 minutes. After the sampling was completed, the both ends of the sample was capped by the plastic end caps and placed in the plastic bag.

For sample preparation, the sampling tube was cut and broken to open the tube. The sorbent was carefully transferred from the tube and placed into a 50-ml volumetric flask. Then 2.5 ml of concentrated  $\text{HNO}_3$  followed by 2.5 ml concentrated  $\text{HCl}$  were added to the volumetric flask. Next, the flask was swirled gently and occasionally for 1 hour. The deionized water was carefully added into the flask to a 50-ml total volume. The solution in the flask was filtered to separate the insoluble residue. Before analysis, the amount of mercury in the sample was adjusted to the standard by diluting 1 ml of sample with 99 ml of mixed acid ( $\text{HCl}$  (5%v/v) and  $\text{HNO}_3$ (5%v/v)) in a 100-ml volumetric flask.

### **3.3.2.2 Sample preparation of mercury in lamp residue**

To make the sample homogeneous, the lamp residue from 3.3.2.1 was pulverized prior to use for analysis of mercury in the lamp residue and mercury leachability test. To analyze total mercury in the lamp residue, a microwave digestion following Method SW846-3051, was applied. About 0.5 g of the pulverized sample was weighed and placed into a microwave vessel followed by 10 ml of concentrated  $\text{HNO}_3$ . Next, the sample was digested by the microwave unit for 10 minutes. After cooling, the supernatant was filtered with a 2.5  $\mu\text{m}$  pore size filter on a 50-ml volumetric flask. The volume of the supernatant solution was made up to 50 ml by deionized water. And then 1 ml of the sample was transferred to a 100 ml volumetric flask and diluted with a mixed acid solution of  $\text{HCl}$  (5%v/v) and  $\text{HNO}_3$  (5%v/v) to fit with the calibration curve range.

### **3.3.2.3 Leachate extraction procedure**

The leachate extraction procedure following the Notification of Ministry of Industry No.6,1997 was modified for the mercury leachability tests. 15 g of the pulverized lamp residue was placed in a 1000 ml polypropylene bottle. Then 300 ml of synthetic acid rain extraction fluid or leachant with a pH of 5 was added into the bottle. The leachant consisted of distilled water mixed with the solution

of sulfuric acid and nitric acid in the ration of 80 to 20 by weight. The bottle was rotated by the overhead mixer with a revolution rate of 30 rounds per minute (rpm.) at room temperature. After being rotated for 18 hours, the leachate was filtered with a 0.7  $\mu\text{m}$  pore size glass fiber filter. After filtration, 1 ml of leachate was transferred to a 100 ml volumetric flask and diluted with a mixed solution of HCl (5%v/v) and  $\text{HNO}_3$  (5%v/v) to fit with the calibration curve range.

### **3.3.3 Sodium sulfide spraying**

The used fluorescent lamps were crushed in each crushing unit. In order to minimize mercury vapor, the sodium sulfide solution at the amounts of 1, 1.25, 1.5, 1.75, 2.00 and 2.5 times the stoichiometric amount was individually sprayed for each experiment. After chemical spraying, the sorbent tube was used to collect mercury vapor and the sample was analyzed following the OSHA's method ID-140 as explained in Section 3.3.2.1.

### **3.3.4 Stabilization/solidification of mercury-containing lamp residue**

The fluorescent lamp residue that was sprayed with each concentration of sodium sulfide (as mentioned in Section 3.3.3), was then weighed, and mixed with cement to achieve the waste/cement ratios of 0.5, 0.75 and 1.00. Then water was introduced into each mixture to obtain the water/cement ratio of 0.5. Next, the mixture was mixed to make the component homogeneous and placed into the block and cured for 7 days. After curing, the solidified waste samples were ground and sifted through a sieve with a mesh hole size of 9.5 millimeters. Fifteen grams of solidified waste passing the screen was mixed with 300 ml of synthetic acid rain extraction fluid in a 1000 ml polypropylene bottle. The bottle was rotated by the overhead mixer with a revolution rate of 30 r.p.m. at room temperature. After rotation for 18 hours, the leachate was filtered with a 0.7  $\mu\text{m}$  pore size glass fiber filter. After filtration, 1 ml of leachate was transferred to a 100 ml volumetric flask and diluted with a mixed solution of HCl (5%v/v) and  $\text{HNO}_3$  (5%v/v) to fit with the calibration curve range.

### 3.4 Analysis of mercury

The flow injection analysis mercury hydride system was used for mercury determination in this study. First, a 500  $\mu\text{l}$  of sample solution was filled into the carrier solution, 3% (v/v) hydrochloric acid (HCl) solution. By the carrier fluid stream, the sample was carried to the manifold which acts as the reaction cell. Next, the reducing agent, 0.2 % (w/v) sodium borohydride ( $\text{NaBH}_4$ ) in 0.05% (w/v) sodium hydroxide (NaOH) solution, was placed and mixed with the sample in the acid carrier fluid in the manifold. And then the reaction between mercury and the reducing agent took place, reducing the ionic mercury to metallic mercury. Finally, mercury in vapor phase was transferred by a carrier gas (argon gas in this analysis) to a cell where the absorption of mercury was measured.

