

III. EXPERIMENTAL

All chemicals employed were of reagent grade. No further purification of these compounds was attempted.

The distilled water used through-out this study was prepared by distilling the deionized water which passed through a set of five ion-exchanger columns (Amberite 200 in Cation Column 1, Zerolit 525 in Cation Column 2, Amberite IRA 402 in Anion Column 3, Amberite IRA 401 in Anion Column 4 and the mixed cation anion exchanger in the last column).

Atomic absorption analysis for cadmium, copper, lead, manganese, nickel and zinc in airborne particulates, lignite and laterite soil was obtained with an Atomic Absorption/Emission Spectrophotometer, Model IL 551, Instrumentation Laboratory, Inc. The IL 551 is a single channel, double beam AA/AE Spectrophotometer with a built in microcomputer which displays all required analytical information on a 23 x 17 cm cathode-ray tube screen.

3.1 Lignite

3.1.1 Sampling

Lignite samples were obtained in August, October and December, 1983. The lignite sample, about 1 kg was collected by the composite method at 5-minute interval from the conveyor belt which moved from the crushing unit to the Mae Moh Power Plant Units 1 to 3. The composite sample was filled into a clean plastic bag, and the bag was sealed. After collection, the samples were taken to a laboratory and put into a pulverizer where the samples were ground into powder. The samples were ready for preparation of sample solutions.

3.1.2 Analysis (2)

The fine-ground lignite sample was passed through a 100-mesh

sieve. The sample size was reduced to about 10 g by the process of mixing and partitioning (using the "quarter" system). This sample was dried at 105° C for 1 hour, placed in the desiccator and weighed about 6 g (accurately to 0.1 mg) in an opened 25-cm³ crucible. The crucible was placed in a cold muffle furnace. The temperature was set to attain 300° C in about 1 hour and then 500° C for an additional 2 hours.

The sample was stirred every hour until no carbonaceous materials remained. The ash sample was placed in a 125-cm³ plastic bottle with a screw cap. Then, 3 cm³ of aqua regia solution and 5 cm³ of hydrofluoric acid were added to the sample solution. The bottle was tightened and placed on a water-bath for at least 2 hours. After that, 50 cm³ of saturated boric acid solution was added to the resultant solution, and the solution was cooled to room temperature. The volume was adjusted to 100 cm³ by the addition of the distilled water. The sample was stored in a polyethylene bottle. Blank solution was prepared in the same manner but without the lignite sample. This solution was ready for determining cadmium, copper, lead, manganese, nickel and zinc by atomic absorption spectrophotometric technique.

3.2 Laterite soil

3.2.1 Sampling

In December 1983, laterite soil sampling was performed at station L1 - L8 and H1 - H8 as shown in Figure 3. The sample about 1 kg was collected from the surface to 2-3 cm depth. At each station, the samples were taken from three areas (the distance between each area is about 10 m) by digging the soil and screening it to remove leaves, roots, gravels, etc. Such samples were filled into a clean plastic bag, and the bag was sealed. After collection, the samples were taken to a laboratory and put into a pulverizer where the samples were ground into powder. These samples were ready for preparation of sample solutions.

Location of the airborne particulate and laterite soil sampling stations
(see Figure 3)

Station	Location
L1	about 0.5 km north of the power plant
L2	about 0.5 km northeast of the power plant
L3	about 0.5 km east of the power plant
L4	about 0.5 km southeast of the power plant
L5	about 0.5 km south of the power plant
L6	about 0.5 km southwest of the power plant
L7	about 0.5 km west of the power plant
L8	about 0.5 km northwest of the power plant
H1	The forest, about 2 km north of the power plant
H2	Mae Moh Meteorological Substation No. 1
H3	Blast Hole Drill Office
H4	Mae Moh Meteorological Main Station
H5	Mae Moh EGAT School
H6	The entrance gate of Mae Moh Project
H7	Ban Na Pom
H8	Ban Huai Rak Mai (Resettlement area)

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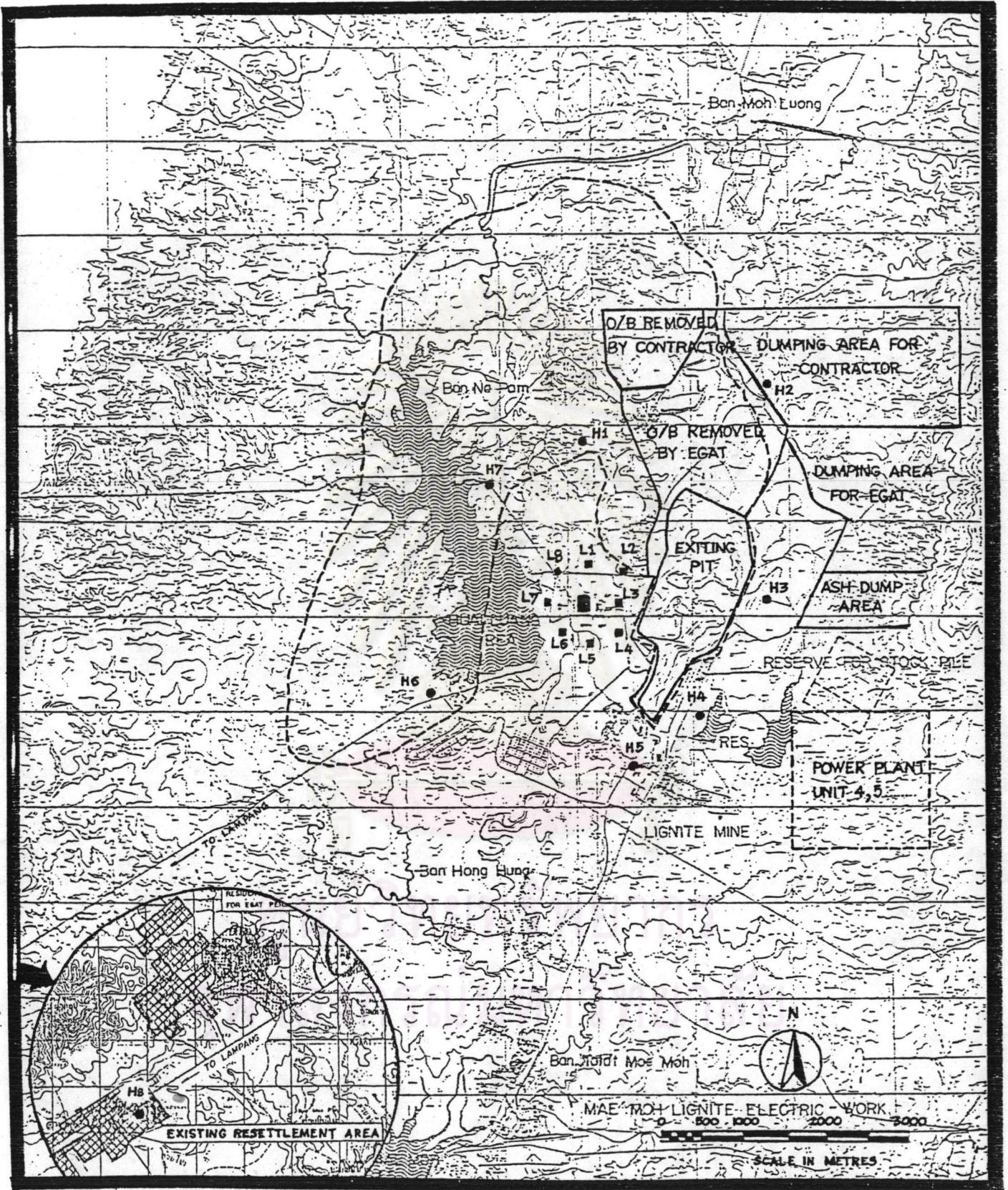


Figure 3 Location of airborne particulate and laterite soil sampling stations

3.2.2 Analysis (3)

The fine laterite soil sample was passed through the 100-mesh sieve. The sample size was reduced to about 5 g by the process of mixing and partitioning (using the "quarter" system). This sample was dried at 105° C for 2 hours and placed in the desiccator and weighed about 1 g. (accurately to 0.1 mg) in an opened 50-cm³ platinum crucible. Then, 5 cm³ of concentrated nitric acid and 2 cm³ of concentrated perchloric acid were added to the crucible. The soil-acid mixture was heated on a hot plate until fumes of perchloric acid appeared. The crucible was cooled to room temperature and then, 5 cm³ of concentrated hydrofluoric acid was added to the soil-acid mixture. The crucible was placed in a sand-bath, heated to 200° C - 225° C and the content was evaporated to dryness. The crucible was removed from the sand-bath and when it was cooled, 5 cm³ of concentrated hydrochloric acid and 5 cm³ of distilled water were added. The crucible was heated on the hot plate until the solution boiled gently. When the residue completely dissolved in hydrochloric acid. The solution was filtered into a 25 cm³ volumetric flask. The filter paper was washed with distilled water and the solution was diluted to the volume. Blank solution was prepared in the same manner but without the soil sample. This solution was ready for determining cadmium, copper, lead, manganese, nickel and zinc by atomic absorption spectrophotometric technique.

3.3 Airborne particulates

The samplings of airborne particulates were based on seasonal wind pattern. They were performed in August, representing the southwest monsoon, in October, representing the transition period and in December, representing the northeast monsoon. The airborne particulates were measured by using gravimetric method. Since there were only two High Volume Air Samplers available, the Low Volume Air Sampler was chosen for sampling the airborne particulates at the distance of 0.5 km from the Power Plant and the High Volume Air Samplers were used for sampling the airborne particulates at the distance of 2 km from the Power Plant.

3.3.1 High volume airborne particulates

3.3.1.1 Sampling

3.3.1.1.1 Station location

The sampling stations were located in the areas as shown in Figure 3. The sampling stations which were designated as H1 through H7 were located at the distance of 2 km from the Power Plant Units 1 to 3. Only station No. H8 was located at the resettlement area (Ban Huai Rak Mai), about 9 km to the southwest of the Plant. This station was used to represent background heavy metal concentration.

3.3.1.1.2 Apparatus

Total suspended particulates were collected on a 8 in. x 10 in. glass fiber filter of 0.3 μm pore size with an collection efficiency of more than 95 percents by the High Volume Air Samplers (Sierra Instruments, Inc. Model 305-2000x and General Metal Works, Inc. GMWL-2000H) as shown in Figure 4. The flow rate of air drawn through the sampler was measured by a rotameter with scale 0-70 cubic foot per minute. (see Figure 5). Before sampling, the flow rate of this instrument was calibrated with an orifice calibration unit. (see Figures 6 and 7)

3.3.1.1.3 Method (51)

The sheet of glass fiber filter was dried in a desiccator with anhydrous silica gel for about 24 hours, weighed accurately to 0.1 mg and kept in a clean plastic bag. When an airborne particulate sampling was desired, the weighed filter was placed in the High Volume Air Sampler. The sampler was turned on, and the time was recorded. The flow rate of ambient air drawn through the filter was measured by the calibrated rotameter. The sampling period for this collection was about 24 hours. After the sampling was completed. Then the filter was weighed again.

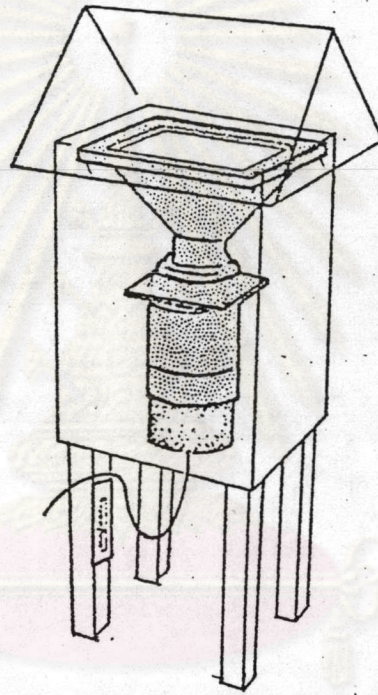


Figure 4 High Volume Air Sampler

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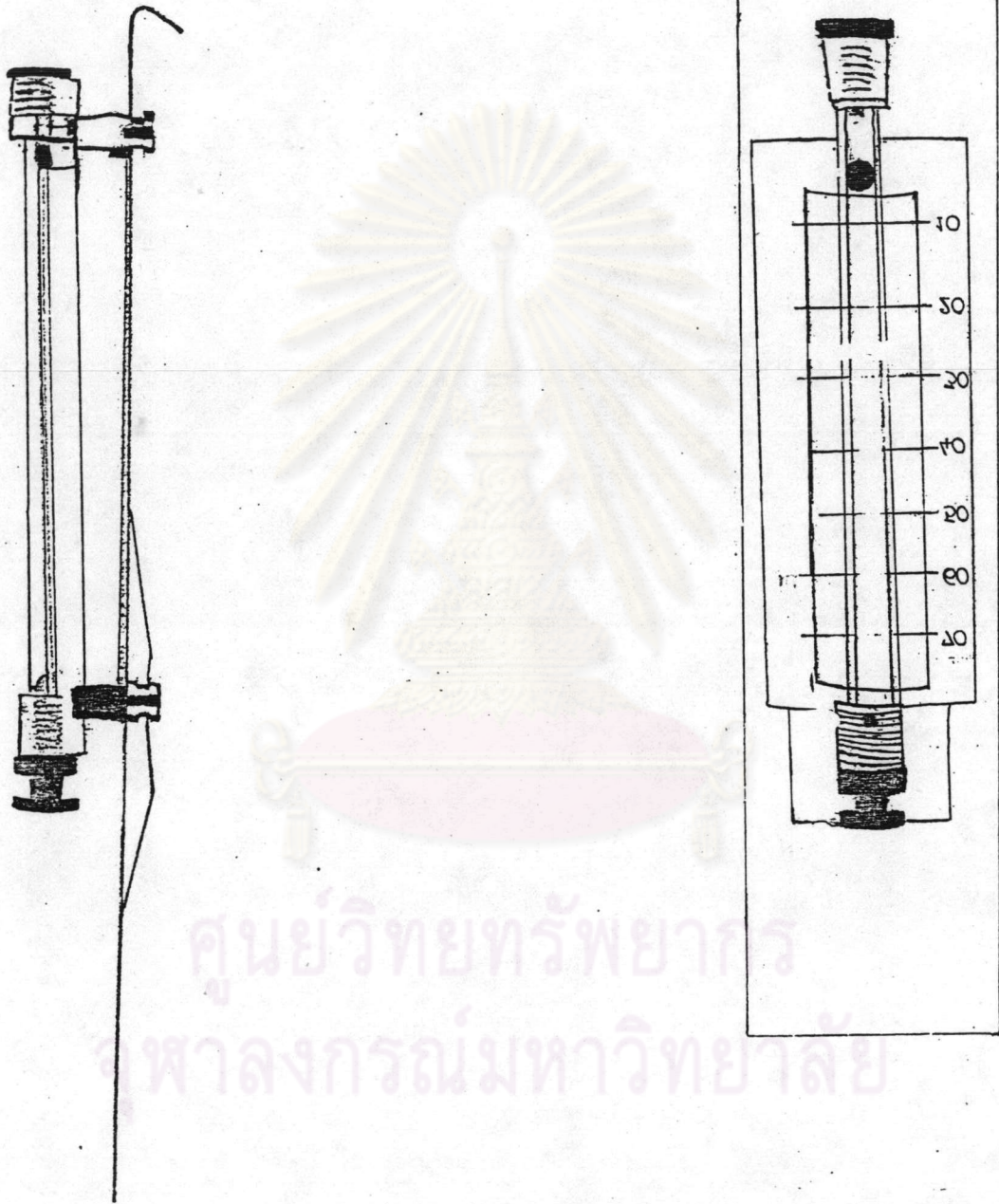


Figure 5 High volume rotameter

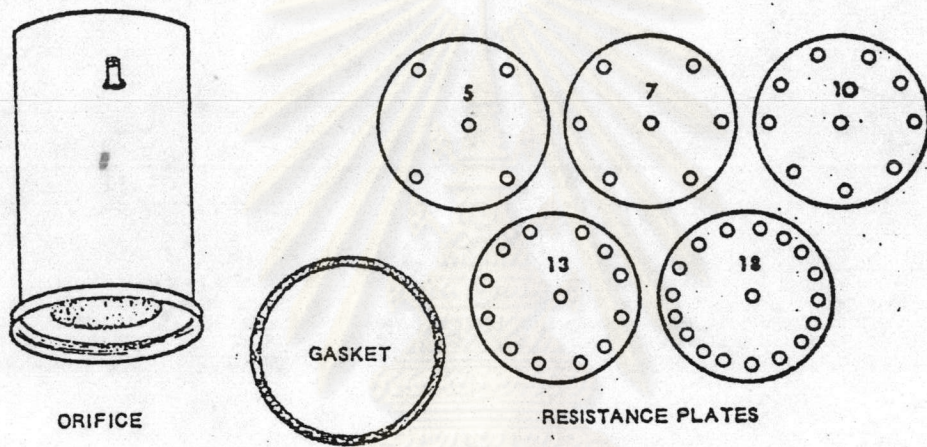


Figure 6 Orifice calibration unit

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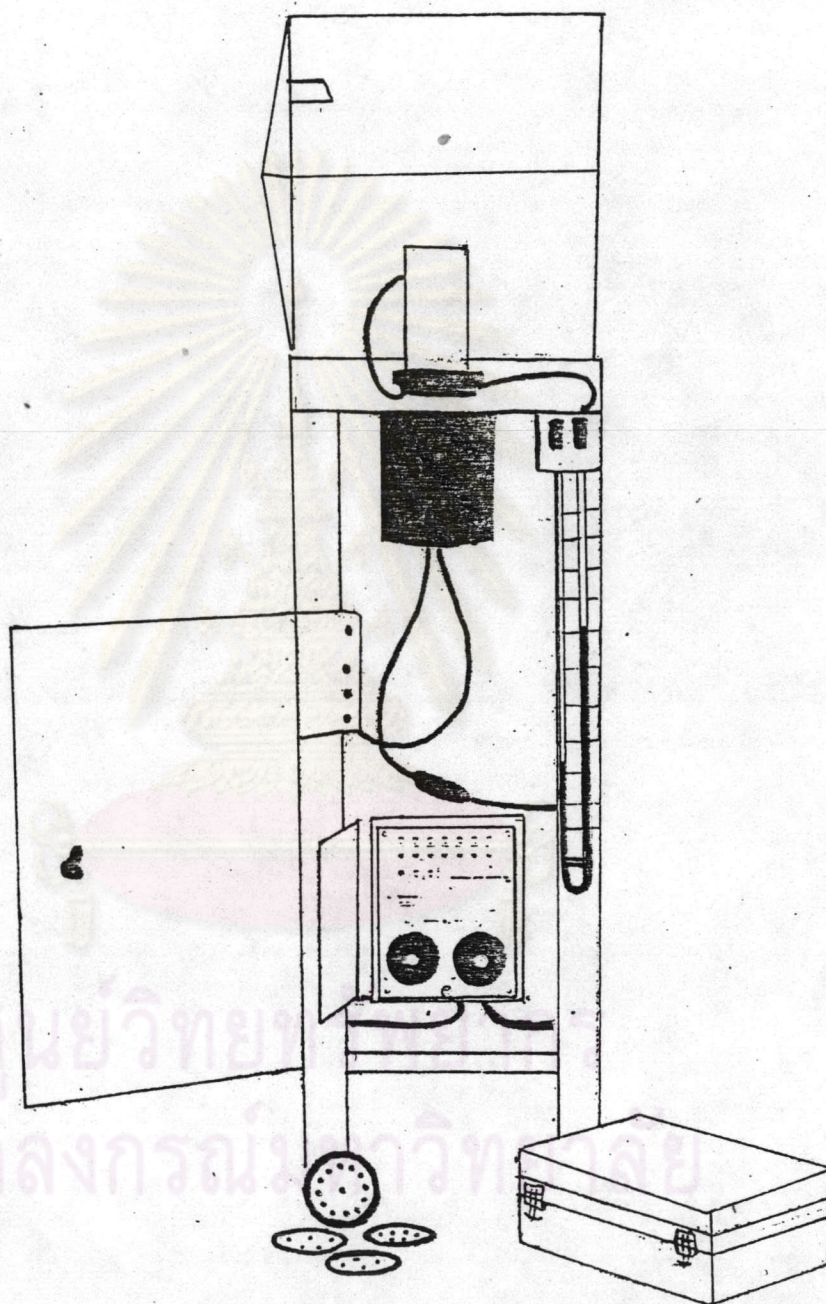


Figure 7 High Volume Air Sampler and orifice assembled for calibration with rotameter

The difference between the weights of the filter before and after sampling was the total weight of total suspended particulates.

3.3.1.2 Analysis (39-43,48)

The sheet of 8 in. x 10 in. glass fiber filter, of which a 7 in. x 9 in. section was exposed on the High Volume Air Sampler, was cut to pieces and placed in a 25 cm³ crucible, and then, the crucible was placed in a cold muffle furnace. After that, the temperature was raised to 500^o C and held at this temperature for 1 hour. The ashed filter was placed in a glass thimble which was placed in a soxhlet extraction tube. A 250-cm³ round bottom flask with 24/40 female joint was charged with 32 cm³ of 19 percents hydrochloric acid and 128 cm³ of 40 percents nitric acid. The flask was attached to the soxhlet extraction tube, and the tube was equipped with an Allihn condenser. The sample was extracted at the temperature of the boiling acid for 3 hours. Then, the extraction tube and condenser was removed from the flask. The extracted liquid was concentrated to 10 cm³ on the hot plate and allowed to cool and stood overnight. The residue was quantitatively transferred to a 100-cm³ graduated centrifuge tube, washed and rinsed several times with 1:10 hydrochloric acid and diluted to 25 cm³. Following the dilution, the sample was centrifuged at 2000 RPM for about 30 minutes and the supernatant liquid was decanted into a glass bottle that was then capped. Blank solution was prepared in the same manner but a new sheet of glass fiber filter was used instead. This solution was ready for determining cadmium, copper, manganese, nickel, lead and zinc by atomic absorption spectrophotometric technique.

3.3.2 Low volume airborne particulates

3.3.2.1 Sampling

3.3.2.1.1 Station location

The sampling stations were located at the distance of 0.5 km

from the Plant and were designated as L1 through L8 as shown in Figure 3.

3.3.2.1.2 Apparatus

Total particulate matters were collected on a 37 mm - diameter Millipore membrane of 0.22 μm pore size by a Bendix Personal Air Sampler, Model C-115 as shown in Figure 8. The flow rate of the instrument was calibrated with a calibrated rotameter.

3.3.2.1.3 Method (1)

A sheet of 37 - mm diameter Millipore membrane, pore size 0.22 μm with filter paper (Whatman No. 5) was dried in the desiccator with anhydrous silica gel for about 24 hours weighed accurately to 0.1 mg and put in a clean plastic holder by facing the Millipore membrane outside for collecting airborne particulates. This collector was ready for air sampling. When an airborne particulate sampling was desired, the collector was connected to the Bendix Personal Air Sampler by mean of a piece of rubber tubing. After the collector was set at the level of 1 m. above ground, the plastic cover of the collector was opened, then the sampler was turned on and the sampling time was recorded. The flow rate of the ambient air drawn through the collector was maintained at 1.6-2.0 liters per minute. The sampling interval for this collection was 2-3 hours. After sampling, the pieces of Millipore membrane with filter paper were dried in the desiccator for 24 hours. Then, the pieces of Millipore membrane with filter paper were weighed again. The difference between the weight of the Millipore membrane with filter paper before and after sampling was the total weight of total particulate matters.

3.3.2.2 Analysis (1)

The collected filter was wet ashed with 20 cm^3 of a mixture of nitric acid and perchloric acid (ratio 9:1) in a 50 - cm^3 beaker. After the filter had been completely ashed. The acid solution was boiled down to the appearance of fumes of perchloric acid. The residue was washed and rinsed several times with 1:10 hydrochloric acid. Then, 1.2 cm^3 of 1:1

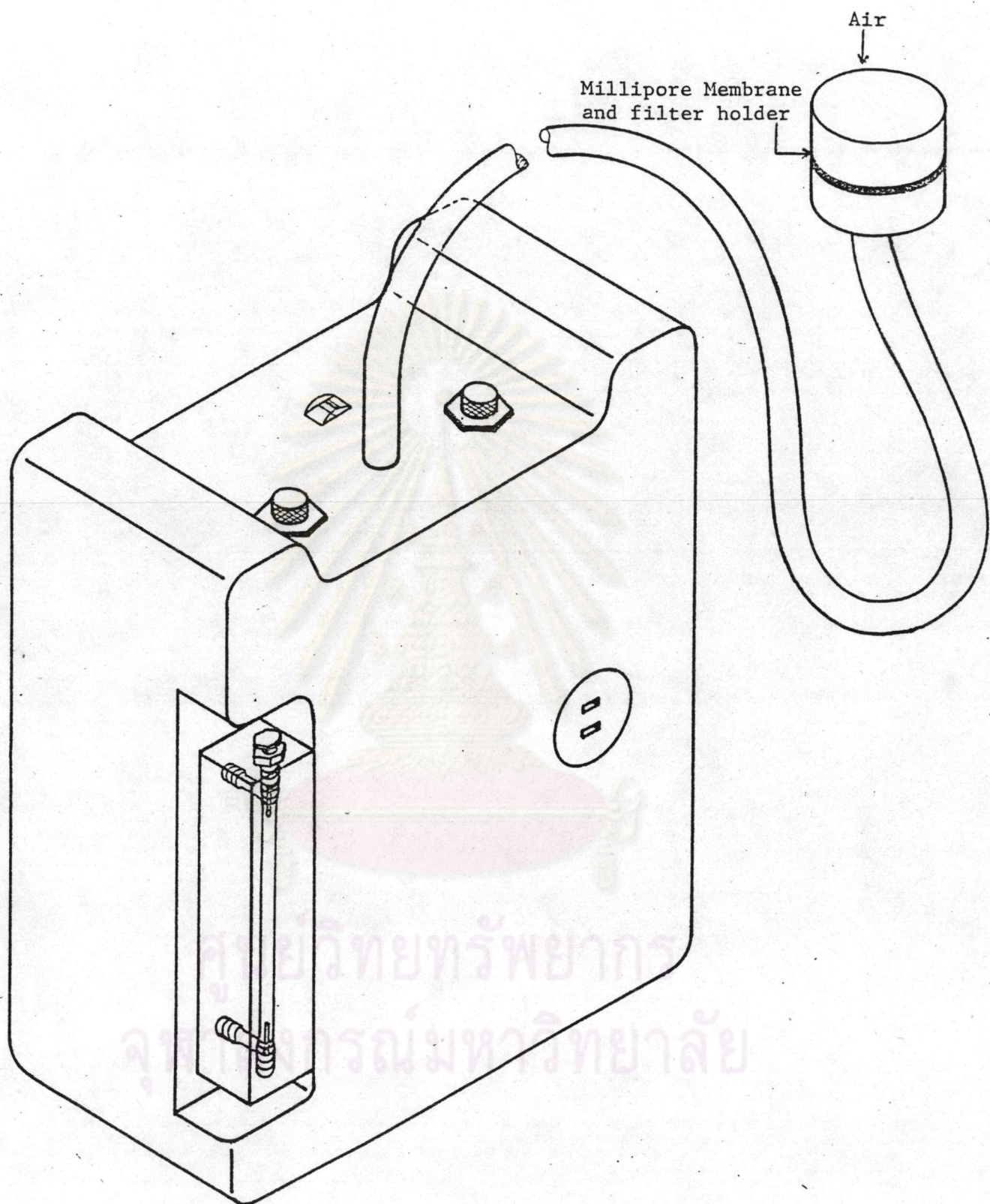


Figure 8 Bendix Personal Air Sampler with an airborne particulates collector

hydrochloric acid was added in the solution and the solution was diluted to 25 cm^3 with 1:10 hydrochloric acid in the volumetric flask. Blank solution was prepared in the same manner but a new piece of Millipore membrane with filter paper was used instead. This solution was ready for determining cadmium, copper, lead, manganese, nickel and zinc by atomic absorption spectrophotometric technique.

3.4 Flame atomic absorption spectrophotometric analyses

3.4.1 Stock solution

The ALKO standard $1,000 \mu\text{g}/\text{cm}^3$ solution of each metal species was employed.

3.4.2 Standard solution

Series of standard solutions containing $0.10 \mu\text{g}/\text{cm}^3$ to $3.00 \mu\text{g}/\text{cm}^3$ of cadmium, copper, lead, manganese, nickel and zinc were prepared by successive dilution of the $1,000 \mu\text{g}/\text{cm}^3$ stock solution with 1:10 hydrochloric acid.

3.4.3 Blank solution

The blank solution was 1:10 hydrochloric acid.

3.4.4 Preparation of calibrating high volume sample solutions

The calibrating sample solutions were prepared as mentioned in 3.3.1.2, except that each heavy metal in the range of $0.10 \mu\text{g}/\text{m}^3$ to $3.00 \mu\text{g}/\text{cm}^3$ were added to each clean sheet of glass fiber filter.

3.4.5 Analysis of the sample

Aliquots of the sample, calibrating sample and blank solution were subjected to the atomic absorption analysis. The concentrations in $\mu\text{g}/\text{cm}^3$ of the sample, calibrating sample and blank were directly read from

the screen of the atomic absorption spectrophotometer. The corrected concentration was obtained by subtracting the blank concentration from the sample and calibrating sample concentration.

The concentration of each heavy metal in the high volume air sample was determined by comparing the corrected concentration of the sample with the calibrating sample concentration of that heavy metal.



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