

CHAPTER 3

GEOCHEMICAL PROCEDURES



3.1 Geochemical Sampling Techniques

3.1.1 Active Stream Sediment Sampling

Active stream sediment samples were collected from accessible streams and creeks. Sample of 200-300 gm of fine-grained sediments are normally adequate for utilization. However, it may be necessary to collect 300-500 gm or more of relatively coarse-grained sediments in order to obtain sufficient portion of fine material for use in chemical analysis. Stream sediment samples were taken, at every interval of 150-200 m along the stream, either by hand or wooden scoop from just below the top of the fine-grained layer. All samples were collected from, if possible, the middle of stream, the active stream bed, or the accumulations of the fine material beneath boulders. The sample were taken as close to the centre of the stream channel as possible within a distance of 10 m around each sample site. Wet samples collected were then placed in plastic envelopes with ensured sample numbers and sample locations. The wet samples in the plastic envelopes may be dried simply under the sunlight at a base camp. This would prevent some chemical deterioration related to bacterial activity that might occur under the oxygen-poor environment. Organic materials that are likely to contain erratic metal concentration or cultural noise are avoided during sample collection. Also every effort was taken in order to avoid sampling collapsed bank material of local origin as this is not representative

sediment of the drainage basin. Any information which may be valuable in the interpretation phase of the survey were recorded, such as geologic units, stream characteristics, topography, composition, and color of sediment. Therefore, the field data cards modified by Smith (1973) were used to record field information. Advantages of using these field data cards are that no observations are omitted or forgotten and the data are computerized rather easily. The field data coding form is given in Appendix A.

3.1.2 Soil Sampling

Soil samples were taken from both banks of stream. The purpose and usefulness of soil profile sampling are aimed to point out the exact location of the sources of anomalies. A complete investigation starts with the collection of a series of samples along the vertical cross section of the soil profile from top soil to depth of approximately 80 cm. Fig. 3.1 shows the distribution of Cr, Ni, Co and Cu contents from soil samples at various depth of soil profile in ultramafic terrain. The fine-grained soil samples were collected at approximately 15-20 m away from both banks at which the stream sediment sampling positions are located. The samples were taken from B-horizon of soil profile by a mattock at the depth of 20-30 cm. 100-300 gm of the fine-grained soil samples and 300-500 gm of coarser material are adequate for subsequent use. The samples are contained in plastic envelopes with sample numbers and locations. The soil sampling information are recorded just like those of stream sediment sampling. Some other information such as, the color change in color of soil, change in the constitution of soil, the presence of organic matter, and possible contamination from any sources, change in zone being sampled and its thickness are also added. The important information were

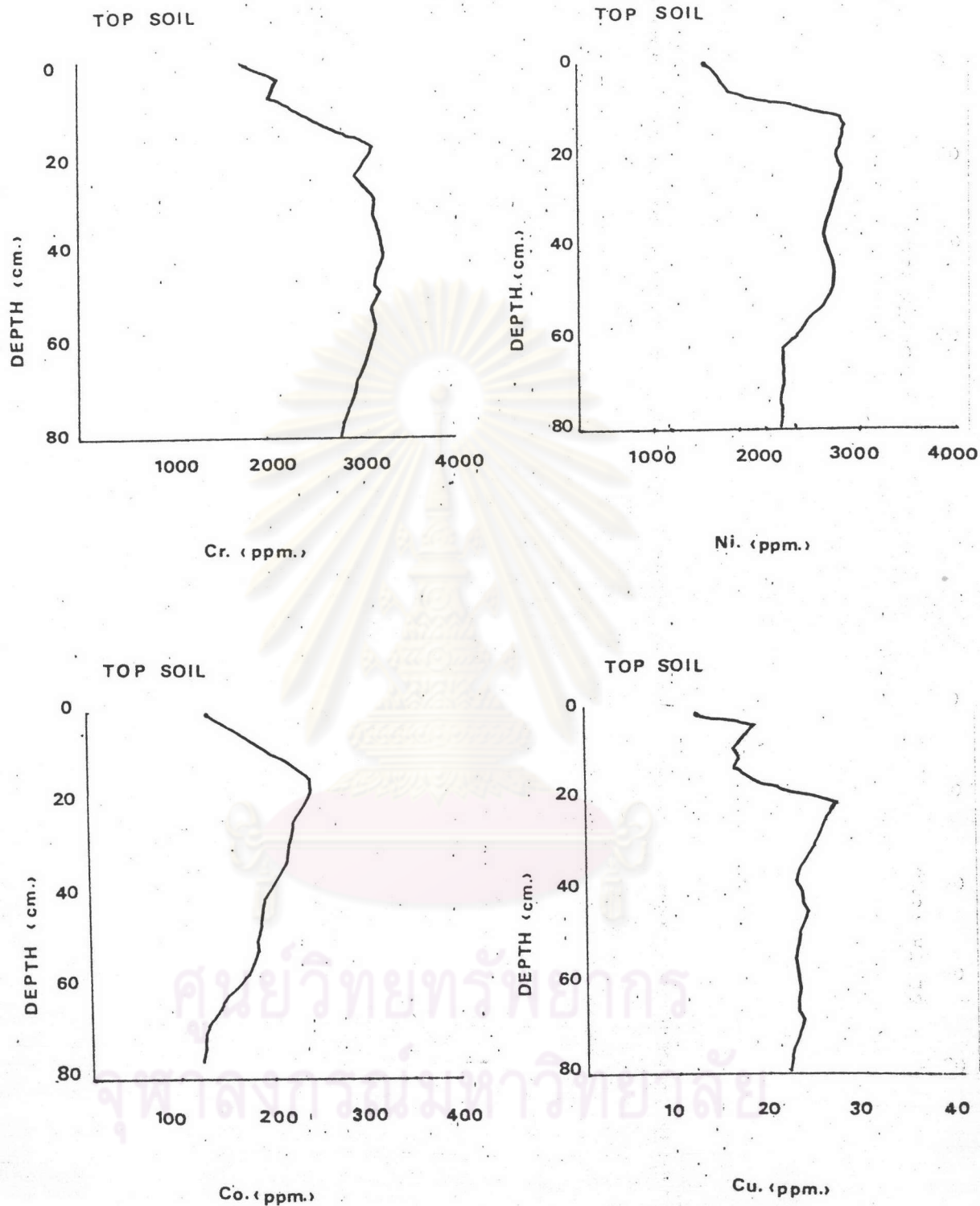


Fig. 3.1 Distribution of chromium, nickel, cobalt and copper in soil profile in ultramafic terrain, Amphoe Wang Nam Yen, Changwat Prachin Buri.

recorded using the field data coding form that was used for the stream sediment sampling.

3.2 Laboratory Techniques

3.2.1 Preparation of Sample for Analysis

It is necessary to apply some kinds of treatments to the soil and stream sediment sample prior to chemical analyses. The main purposes of these treatments are aimed : to remove water which would interfere with subsequent processing; to separate the to separate the proper fractions for analysis such as sieving samples to a certain size; to homogenize samples in order to eliminate variations; and to grind samples to analytical fineness. The preparation of samples must be taken to avoid contamination and labelling errors, the data obtained will be worthless if it is not prepared correctly. The main processes for sample preparation will be described and the flowchart summarized the treatment stage for soil and stream sediment samples is shown in Fig.3.2.

3.2.1.1 Drying : Drying is necessary because damp samples can not be properly sieved. At first, samples are dried by exposing to sunlight at the base camp where each is poured from its envelopes and spread out onto heavy kraft paper. After a day or more, the dried samples are stored in plastic envelopes again, and then the samples must be treated by being laid out in pre-numbered watch glass in the oven (about 90^o C) for drying.

3.2.1.2 Crushing : The purpose of crushing is to reduce the size of lumps. After drying, any harden cakes of fine-grained material must be slightly crushed in a mortar. Dried sandy or silty samples require very little crushing before sieving.

3.2.1.3 Sieving : After crushing stream sediment and soil samples generally pass through the minus 80 mesh stainless steel sieve. The sieved portion has been obtained for further analysis, the remained portion is poured back into a labelling plastic bag. After sieving, the sieve should be dusted and cleaned with a brush. The remaining unsieved portions are also kept in sealed labelling plastic bag in case the sieved samples are lost or damaged.

3.2.1.4 Splitting : It is necessary to obtain a small representative portion from a large sample collected portion. The procedure is simply gently pour the rolled sample onto the plastic plate in a circular shape and then split it into four equal quarters using a clean spatula and then remove to alternate quadrants and mix them. Similarly mix the other two alternate quadrants. This results in two representative splits of the sample. About 50 gm of sieved and splitted weight of each sample are required for analysis

3.2.2 Analytical Methodology

All the analytical methods currently use for determination of elements, generally, are described by Ward (1969). Atomic absorption spectrophotometric method is used to determine Cr, Ni, Co, and Cu. The elements are analysed by using a Varian-Techtron model AA 6. The method involves digesting samples of 0.5 gm with nitric acid (5 ml) at 90 C for an hour, fuming, and finally take the sample into solution. The volume of solution is adjusted to 10 ml. with deionized water, mix thoroughly and filter. Portions of sample solution are aspirated into air acetylene flame of an atomic absorption spectrophotometer. Appropriate hallow cathode lamp is used for each element and standard solutions containing known concentrations of each element determined are used for calibration. The estimation of the result is zeroing

instrument with 1:1 nitric acid and atomize the supernatant liquid or filtrate in an atomic absorption spectrophotometer. Read the absorbance and convert to concentration in atomized solution as directed below for the particular instrument. Calculate result as follow:

$$\text{ppm} = (\text{microgram/ml in solution}) \times 10$$

Setting up and alignment of the AAS unit should be done according to the instrument manuals. The actual measurements start once the instrument have been set to their maximum sensitivities. The operating conditions for instrument are given below:

	Cr	Ni	Co	Cu
Lamp current	5 mA	5 mA	5 mA	3 mA
Fuel	acetylene	acetylene	acetylene	acetylene
Support	air	air	air	air
Flame stoichiometry	reducing	oxidizing	oxidizing	oxidizing
Wavelength	357.9 nm	232.0 nm	240.7 nm	324.7 nm
Optimum Working range	2-8 $\mu\text{g/ml}$	3-12 $\mu\text{g/ml}$	3-12 $\mu\text{g/ml}$	2-8 $\mu\text{g/ml}$

In analyses by atomic absorption spectrophotometer for various elements, where the values are very high, the solutions must be diluted ten times, one hundred times or more if necessary, to bring the values within the scale. The solutions are diluted with 1:1 nitric acid.

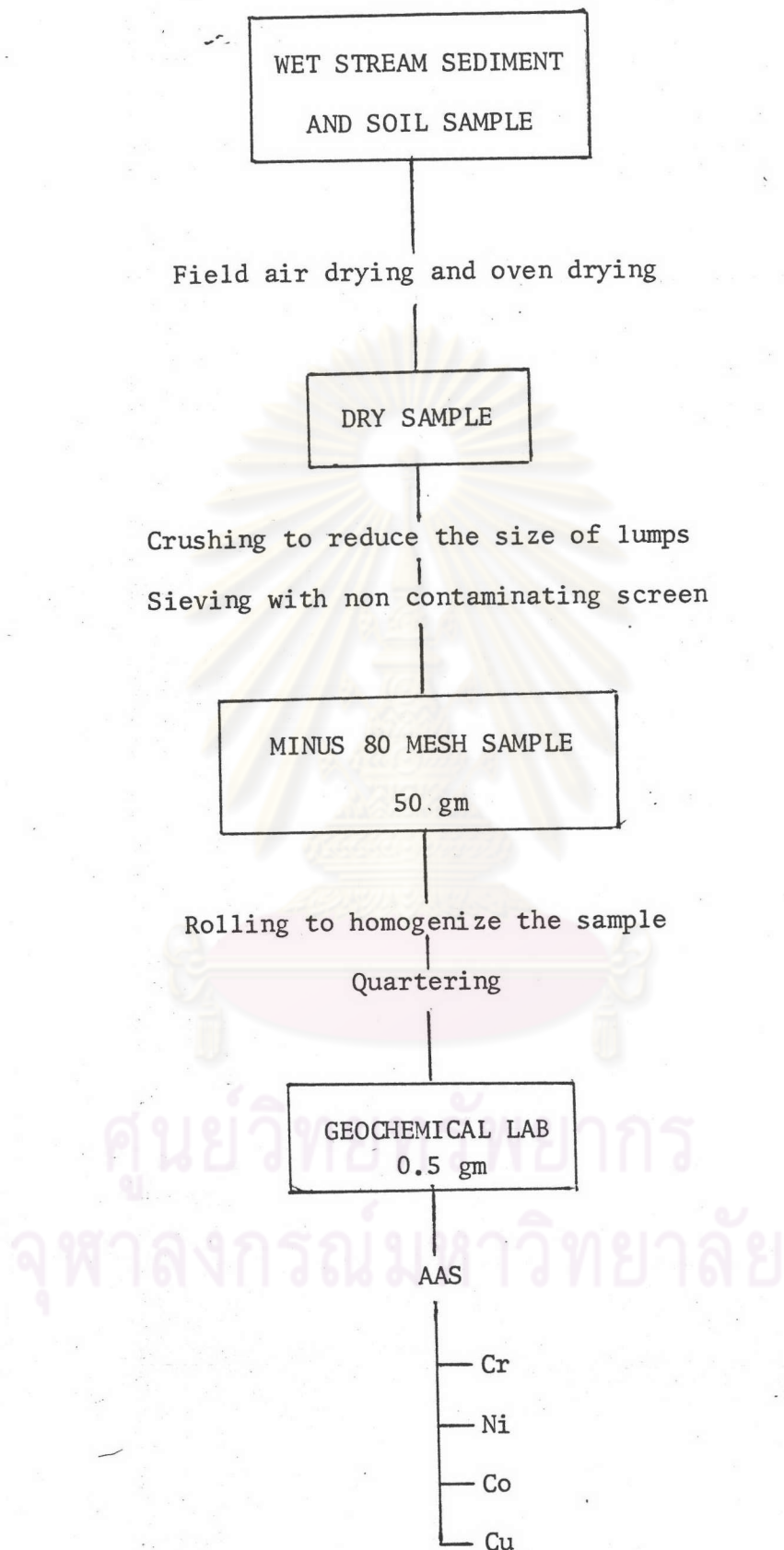


Fig. 3.2 Sample preparation scheme for stream sediment and soil samples