

CHAPTER II

METHODOLOGY

A detailed method of study is shown as the flow chart (Figure 2.1). It consists of the following topics.

2.1 Field Study

2.1.1 Field investigation

This step was carried out specifically for mapping the intrusive rocks distribution in the study area and compared with the report of Jungyusuk et al. (1995) in order to find out field relationship and collecting samples for further study in laboratory which has done during March 2002 and March 2003. The detailed geologic map was prepared from the result of the field evidences and modified after Jungyusuk et al. (1995). The intrusive rocks were categorized based on field evidences into four types, namely gabbro, diorite, quartz diorite and hornblende-biotite granodiorite, whereas Jungyusuk et al. (1995) have reported only two types, quartz diorite and hornblende-biotite granodiorite.

2.1.2 Sample collection

Altogether 49 rock samples were collected which included 12 samples of gabbro, 14 samples of diorite, 11 samples of quartz diorite and 12 samples of hornblende-biotite granodiorite. The samples were collected to represent the variation in each rock type. Their location are given in the map of Figure 2.2.

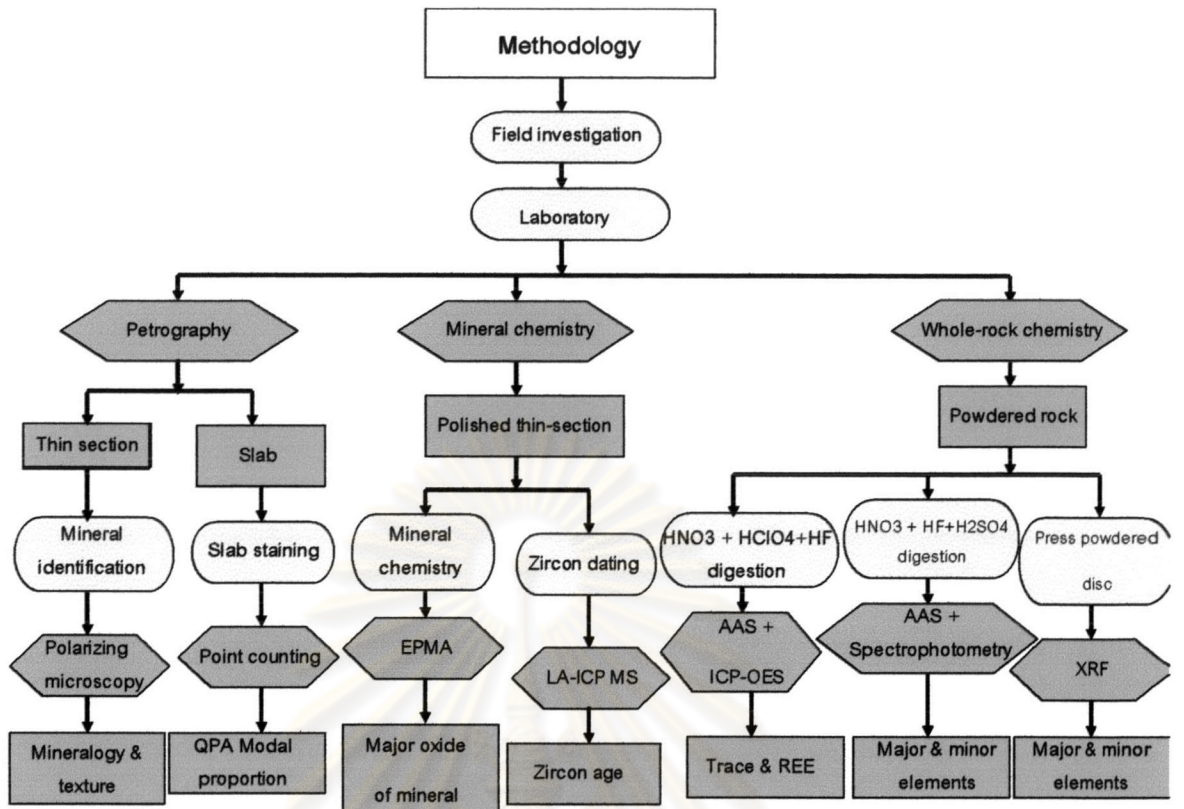


Figure 2.1 Showing methodology in this study.

Sample location map

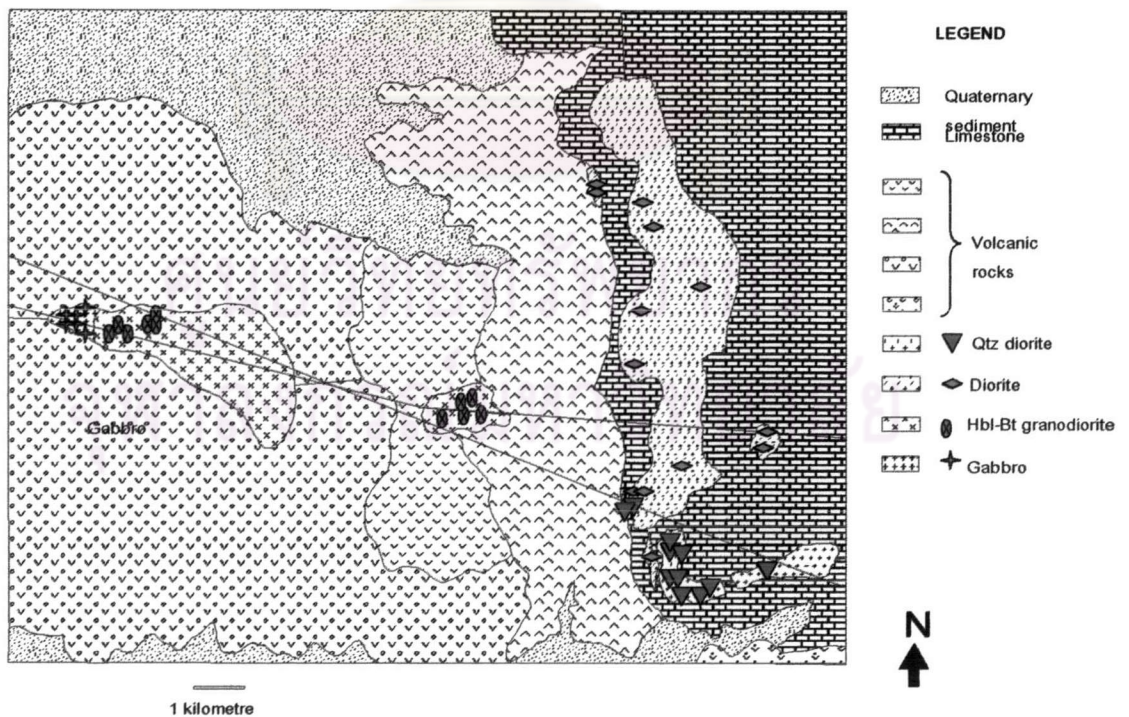


Figure 2.2 Sample location map.

2.2 Laboratory study

2.2.1 Petrography

2.2.1.1 Thin Section

The rock samples were slab-cut and prepared as thin section. The thin section was usually set at 0.03 mm thick and covered with a covered glass. These thin sections were used for mineral identification and textural study by a polarizing microscope. The text books include An introduction to the rock-forming minerals (Deer et al., 1966), Atlas of igneous rocks and their textures (MacKenzie et al., 1982), Petrography of Igneous and Metamorphic Rocks (Philpotts, 1989), Rock-forming Minerals in Thin Section (Pichler, 1997) were used for mineral and texture identifications. Consequently, those textures were interpreted after Hibbard (1995) and Bard (1987).

2.2.1.2 Slab Staining

Two slabs of hornblende-biotite granodiorite and five slabs of pyroxene diorite were stained by Amaranth and Sodium cobaltinitrite method, after Hutchison (1974) and the details are described in Appendix A.

2.2.2 Polished Section for Mineral Chemistry study

2.2.2.1 Mineral Chemistry by EPMA

For mineral composition determination, two samples of each rock type were prepared as polished thin section. The un-covered slightly thick sections were polished by 12, 6, 3 and 1 and 0.3 μm -sized diamond pastes. The polished sections were later analyzed specifically on pyroxene, hornblende and plagioclase by an electron probe micro-analysis (EPMA).

Eight samples, two samples per rock type, were selected for quantitative analysis using a JEOL Electron Probe Micro-Analyser (EPMA) model JXA-8100 located at the Department of Geology, Faculty of Science, Chulalongkorn University. Conditions for analysis and standard materials used in this study are given in Table 2.1 and Table 2.2, respectively.

Table 2.1 Conditions for electron probe micro-analysis in this study.

Spectrometer	Crystal	Elements	Counting time for peak, sec.	Counting time for background, sec.
2	TAP crystal	Al, Si, Na and Mg	30	10
3	PET crystal	K, Ti, Ca and Ba	30	10
4	LiF crystals	Fe	30	10

Table 2.2 Standard materials, for EPMA, used in this study.

Standard	Element
Corundum	Al
Wollastonite	Si and Ca
Periclase	Mg
Pyrophanite	Ti
Barite	Ba
Fyalite	Fe

2.2.3 Zircon Dating

In addition, in order to calculate the time of emplacement of the rock. two grains of zircon in gabbro were *in situ* determined its isotopic composition of U, Th and Pb by laser ablation ICP-MS, HP 4500 (Series 300) at the Macquarie University, Australia for age dating. The operating conditions for U-Pb analysis is shown in Table 2.3.

Table 2.3 Operating conditions for U, Th and Pb isotopic analysis by laser ablation ICP-MS.

forward power	1350 kW
gas flow rate	plasma Ar 13 L/min
auxiliary Ar	0.8 L/min
carrier He	ca. 1 L/min
Degree of defocusing	200 micron above sample
incident pulse energy	0.5-0.9 mJ.

2.2.4 Whole Rock Analysis

Total numbers of 20 rock samples from four rock types in the study area were selected for whole-rock analysis. Firstly, about 1-2 kg of each sample was crushed by a jaw crusher to about 1 – 1.5 cm pieces. In order to avoid weathering effect, such weathered pieces were removed before sub-sampling by the quartering method. Secondly, the samples were pulverized by a tungsten carbide disc mill into size of minus 200 mesh. All powdered-rock samples were dried at 105 °C for 2 hours to remove the moisture before keeping in glass containers.

2.2.4.1 Major and Minor Elements Analysis

In this study, major and minor elements were carried out by two methods including wet analysis and X-ray fluorescence spectrometer (XRF). Wet analysis using the two solutions method, solution A and B (after Shapiro, 1975), was done at the Department of Geology, Chulalongkorn University, while the XRF method was done at the Center of Scientific Instruments of Chulalongkorn University. All the major and minor elements content were reported as oxide.

a) Wet Chemical Analysis

According to the two solutions method, solution A was prepared by fusing the powdered rock samples with NaOH and dissolved in diluted HCl and used for SiO₂ and Al₂O₃ determinations, whereas solution B was prepared by decomposing the powdered rock samples by the acid mixture of HF, HNO₃ and H₂SO₄ and were used for the determination of total iron, MgO, CaO, TiO₂, P₂O₅, MnO, Na₂O and K₂O. The detail of preparation of the two solutions method are described in Appendix A. The determination of SiO₂, Al₂O₃, total iron, TiO₂, P₂O₅ and MnO were analyzed by the spectrophotometer, while MgO, CaO, Na₂O and K₂O were determined by Atomic absorption spectrometer at the Department of Geology, Chulalongkorn University. FeO was analyzed separately by decomposing the powdered rock samples by HF-H₂SO₄ and titrated with dichromate solution using diphenylamine sulfonic acid as the indicator (Sarvu, 1927). All of these analyses were carried out by calibrated with the USGS reference materials including G-2, GSP-1, BHVO-1 and QLO-1. The detail of the conditions of these analyses are summarized in Tables 2.4 and 2.5.

Table 2.4 Methods and wavelengths used in major and minor element determinations by spectrophotometer.

Element	Method	wavelength
SiO ₂	Molybdenum blue method	640 nm.
Al ₂ O ₃	calcium aluminum alizarin red-S complex	475 nm
TiO ₂	Tiron (disodium -1, 2-dihydroxybenzene-3, 5-disulfonate)	430 nm
P ₂ O ₅	Molybdovanadophosphoric acid complex	420 nm
MnO	permanganate complex	525 nm
Total iron (as Fe ₂ O ₃)	Orthophenanthroline complex	555 nm

Table 2.5 Methods and wavelength used in major and minor element determinations by Atomic Absorption Spectrometer.

Element	Method	Wavelength, nm
MgO	Air-acetylene flame	285.0
CaO	Air-acetylene flame	442.7
Na ₂ O	Air-acetylene flame	589.0
K ₂ O	Air-acetylene flame	766.5

b) X-Ray Fluorescence Spectrometry (XRF)

The same set of powdered rock samples were prepared as pressed pellets by using HBO₃ (boric acid) as the binding material at the ratio of sample : binder of 8:2 and mixed in the tungsten carbide mill and pressed at the pressure of 400 kg/cm² for 1 minute. The analyses were done for major and minor elements by XRF model PW2400 Phillips by using SemiQ software at the Scientific and Technological Research Equipment Center of Chulalongkorn University by the following conditions (Table 2.6).

Table 2.6 Conditions used in major and minor element determinations by XRF.

Element	Crystal	Voltage, kV	Current, mA
K ₂ O, CaO and TiO ₂	LiF200	30	80
P ₂ O ₅	Ge 111	24	100
SiO ₂	PE 002	24	100
Al ₂ O ₃	PE 002	24	100
Na ₂ O and MgO	PX 1	24	100
MnO	LiF 220	55	45

c) Determination of Loss on Ignition

Transfer about 5 g. of sample to a platinum crucible, without cover. Then place the crucible into an electric muffle furnace and allow the temperature to rise slowly. When the temperature reaches 500 °C, cover the crucible and continue heating to 1,000 °C for 60 minutes.

When the furnace is cool down, place the platinum crucible in a desiccator, weigh the crucible and repeat weighing until constant weight. Then calculate the weight loss at 1,000 °C.

2.2.4.2 Trace Elements Analysis

The powdered rock samples were prepared by dissolving 0.5 g of the powdered rock samples in the acid mixture of conc. HNO₃, HF and HClO₄ and diluted to 50 ml and was used for analyses by both AAS and ICP-OES for trace element and REE, respectively.



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a) Atomic absorption spectrometry (AAS)

Trace elements : Cu, Pb, Zn, Ba, Rb and Sr were analyzed by the staff of Rock and Mineral Resources Analysis Group, *Mineral Resources Analysis and Identification Division*, Department of Mineral Resources, Thailand, by the Atomic absorption spectrometer (AAS); Varian Spectra 800. Their concentrations were determined from the calibration graph, prepared from the standard solutions for AAS of each element and evaluated the reliability of the analyses by analysing the reference rock material; Sy-3 which was prepared by the Canadian Centre for Mineral and Energy Technology. The detail of the conditions of these analyses are summarized in Table 2.7.

Table 2.7 conditions of trace element analyses by AAS at Department of Mineral Resources.

Element	Method	wavelength
Cu	Air-acetylene flame	327.4 nm.
Pb	Air-acetylene flame	283 nm.
Zn	Air-acetylene flame	307.6 nm.
Ba	Acetylene –nitrous flme	553.6 nm.
Rb	Air-acetylene flame	780 nm.
Sr	Acetylene –nitrous flame	460.7 nm.

b) ICP-OES

The other trace elements namely Nb and Y and rare earth elements ; La, Ce, Pr, Nd, Sm, Eu, Gd, Dy and Yb were analyzed by Inductively Coupled Plasma Spectrometer (ICP-OES), Varian-Vista MPX at the National Waste and Disaster Disposal Management College. The calibration curve of each element was prepared from the standard solution for ICP-OES and evaluated the reliability of the analyses by analyzing the reference rock material; GSP-2 which was prepared by USGS. The detail of the conditions of these analyses are summarized in Table 2.8.

Table 2.8 Conditions of trace element analyses by ICP-OES at the National Waste and Disaster Disposal Management College.

Element	Wavelength, nm
Ce	418.65
Dy	353.17
Eu	420.50
Gd	342.24
Hf	264.14
La	33.74
Nb	269.70
Nd	401.22
Pr	390.84
Rb	421.55
Sm	359.25
Sr	407.77
Y	371.02
Yb	328.93