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APPENDIX

Analytical techniques

Methods and Techniques of chemical analysis of rocks for major element oxides and trace elements employ the X-ray fluorescence method, modified Penfield method and classical volumetric method.

Description of Sample preparation and analysis techniques

Rock crushing

1. The rock sample is coarse-crushed into chips by jaw crusher.

2. Dust is removed by blower.

3. Chips with xenolith and perhaps with altered surface are removed by hand pick.

4. 100 gm. of chips of crushed sample was then pulverized by a Tungsten-Carbide barrel on a Tema rotary crusher for 3 minutes. The finest of powder sample should be reduced to 200-250 mesh.

5. The fine powder is homogenized by mixing on glazed paper and sampling by cone splitting.

This powder is used for the determination of major and trace element by x-ray fluorescence, minerals by x-ray diffraction and ferrous iron by the classical volumetric method.

Preparation of glass bead

1. Take 4 gm. of powder sample in an oven for two hours at $120^{o}c$.

2. Weigh the dry sample (The different weight is H_2O^-)

3. Weigh the powder sample exactly 1.5 gm. in a platinum crucible (platinum/gold 95/5%). Add 7.5 gm. of a mixture of anhydrous spectromelt (mixing the equal amount of lithium metaborate and lithium tetraborate flux to the sample with the ratio 5:1) and mix sample and spectromelt with a glass rod in the platinum crucible.

4. Put the platinum crucible on a Harzog burner for ten minutes at $1100^{o}c$.

5. Take the bead out of the platinum crucible and write down the sample number after cooling has completed.

The glass bead was homogenized by the way of this method and used for the determination of major element oxides and trace elements by x-ray fluorescence.

X-ray Fluorescence Method

The x-ray fluorescence spectrometry is used for determining the element that present in a rock sample. The principle of this method is that when an element is a multi component sample is bombarded with primary x-ray photon, secondary x-ray photons characteristic of this element are produced. These secondary of fluorescent x-rays are then dispersed according to their wave lengths. This is achieved by using a crystal of known d-spacing where diffraction takes place according to Bragg's relationship $n\lambda = 2d\sin\theta$ where λ is the wavelength, d is the spacing between the planes of the crystal.

The intensity of the diffracted fluorescent radiation is then measured, and the concentration of the element producing this radiation can be found, since the intensity is proportional to concentration.

The sample analysis was carried out on a philips (PW. 1400) automic x-ray spectometer with computerized system at Analytical Laboratory Section, Geological Survey Division; Department of Mineral Resources. The technique used in this work is the fusion method for glass-bead sample. The homogeneity and the surface of samples are to be in great case.

The regression analysis was applied also with the matrix corrections, these are carried out on onlined-phillips computer.

Determination of Ferrous Iron

Ferrous iron was determined by classical technique. The sample powder is treated with hot hydrofluoric and sulphuric acids. The contents of the platinum crucible are then transferred to a beaker containing a mixture of sulphuric, phosphoric and boric acids. The mixture is then titrated with potassium dichromate, until the indicator, sodium diphenylamine sulphonate assumes a permanent purple color. Fe_2O_3 was calculated from total Fe_2O_3 and FeO by using the relationship : Fe_2O_3 = total Fe_2O_3 - (FeO x 1.1113). This method is fast and direct, no comparisons are made with other rocks. The presence of sulphides or organic matter renders and accurate determination of ferrous iron impossible. None of these components present any serious problem in any of rocks analysed.

Determination of Combined Water (H_2O^+)

The combined water (H_2O^+) of the rock samples were determined by a modified Penfield Method. The procedure of the analytical - tecnique is as follows:

1. Place 3 gm. of sodium tungstate flux and 1.000 gm. of the sample in a porcelain crucible and mix them thoroughly with a glass rod. Using a long-stemmed funnel, completely transfer this mixture into the bulb of a dried Penfield tube. Close the tube with a capillary stopper.

2. Horizontally insert the bulb of the Penfield tube, with a strip of damp cloth spirally wrapped around the middle sector, into the open flame from a bunzen burner. Slowly rotate the tube while heating the bulb at red-heated for 30 minutes. Keep the cloth wet throughout the heating period.

3. Heat the juncture of the tube until it softens. Pull off the bulb with iron tongs, and heat the sealed end of the tube for a few seconds to round of the sharp tip.

4. Let the tube cool, unwrap the damp cloth, and blot the water from the outside of the tube with a towel. Remove the stopper and weight the tube (tube weight plus weight of combined water).

5. Dry the tube in an oven at 110^{o}c for 1 hour. Let the tube cool and weight of the tube during drying.

Condition for X-ray Fluorescence Analysis for Major Element

Element		Filter	Collimater	Detector	Crystal	Upper level	Lower Level	KV	mA	Angle	+OFFS
SiO_2	KA	NO	C	F	PE	75	25	50	60	109.210	2.00
TiO_2	KA	NO	F	F	LIF-200	75	27	50	60	86.140	.00
Al_2O_3	KA	NO	C	F	PE	75	25	50	60	145.130	2.00
Fe_2O_3	KA	NO	F	F	LIF-200	75	20	50	60	57.520	2.00
MnO	KA	YE	F	F	LIF-200	75	17	50	60	62.970	2.00
MgO	KA	NO	C	F	TLAP	80	20	50	60	45.170	2.00
CaO	KA	NO	C	F	LIF-200	75	25	50	60	113.090	2.00
Na_2O	KA	NO	C	F	TLAP	80	20	50	60	55.100	3.00
K_2O	KA	NO	C	F	LIF-200	75	25	50	60	136.690	2.00
P_2O_5	KA	NO	F	F	Ge	75	25	50	60	141.040	3.00
S	KA	NO	C	F	Ge	75	25	50	60	110.690	2.00

Collimater

F = Fine

C = Coarse

Detector

F = Flow

+OFFS

-OFFS = offset angle from the goniometer.

Condition for X-ray Fluorescence Analysis for Trace Element

Element		Filter	Collimater	Detector	Crystal	Upper level	Lower level	KV	mA	Angle	+OFFS
Ba	LA	NO	F	F	LIF-200	75	27	50	60	87.170	2.00
Nb	KA	NO	F	FS	LIF-200	75	25	75	40	21.400	1.60
Pb	LA	NO	F	FS	LIF-200	70	25	75	40	33.930	1.50
Rb	KA	NO	F	FS	LIF-200	75	25	75	40	26.620	1.00
Sr	KA	NO	F	FS	LIF-200	75	25	75	40	25.150	1.00
Zn	KA	NO	F	F	LIF-200	75	24	60	50	41.800	1.00
Zr	KA	NO	F	FS	LIF-200	75	25	75	40	22.550	.80

Collimater

F = Fine

Detector

F = Flow

FS = Flow and Scintillation

+OFFS

-OFFS

= offset angle from the goniometer

BIOGRAPHY

Ms. Suporn Boonsue was born in Bangkok, Thailand, on September 28, 1959. In 1979 she graduated with Bachelor of Science in Geology from Chiang Mai University, Chiang Mai, Thailand. During the following years she was attached to physics Section, Geological Survey Division, Department of Mineral Resources, she is assigned to work on the rocks and minerals identification.