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Chemical Composition of Korean Geological Reference Materials

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1. Introduction

Geological reference material (below geo-standard) is essential for analytical research and has been widely used for a chemical analysis to obtain accurate geochemical data. Besides, it is useful to evaluate the accuracy of the analysis, to develop and improve analytical techniques. There is a trend that demand for calibration increases as various analytical instruments and techniques are newly invented and applied [1,2,3]. The geo-standards, which are the targets of a geological survey such as rocks, ore, minerals, soils, and sediments, contain various elements in high concentration [3].

In this study, we selected granite as a candidate for the Korean geo-standard, and as a preliminary study, we analyzed major elements and rare earth elements (REE) by using X-ray Fluorescence (XRF) and Prompt Gamma-ray Activation Analysis (PGAA), respectively.

Precision and accuracy of the analytical techniques are evaluated by using geo-standards supplied by USGS (US Geological Survey) and GSJ (Geological Survey of Japan). Moreover, we checked the homogeneity of the samples, which is defined by the concentration of elements of interest and their average values, absolute and relative standard deviations and so on.

2. Materials and Methods

2.1 Sample selection and mineralogy

For the selection of a candidate of Korean geo-standard, following conditions should be satisfied: 1) it represent the Korean rocks, 2) its mineral and chemical compositions are homogeneous, 3) it does not include any geological structures like fissure or fold, and 4) fresh rock is available in a large volume.

Based on above conditions, we selected Jurassic granite from Geochang-gun province, Gyeongsang-namdo (KJG-2) (Fig. 1). It belongs to the central part of the Yeongnam Massif and biotite granite with porphyritic texture formed in the Mesozoic Jurassic. It consists of quartz, plagioclase, alkali-feldspar, biotite along with accessory minerals such as muscovite, magnetite, zircon, apatite and so on.

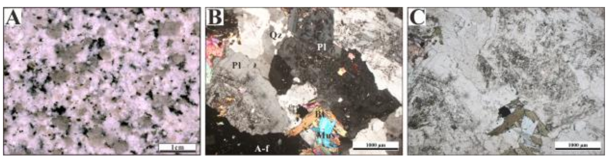

Figure 1. Photograph showing of the KJG-2. A, rock slap; B, crossed polarized light; C, plane polarized light. Abbreviation: Qz, quartz; Pl, plagioclase; A-f, alkali feldspar; Bt, biotite; Mus, muscovite; opq, opaque.

2.2 Sample preparation

When analyzing the chemistry of geological materials, massive samples are pulverized into fine powders using various crushers [3]. A sample was prepared as below procedures [4] : break a slab of fresh rock with 30×30×2 cm into 5 to 10 cm using a rock-hammer; crush broken slabs into small pieces of 1 to 2 cm using a jaw crusher (Retsch; DE/BB-0); mill the pieces of the rock in a disc mill (J-NDM 95); make fine powders using mortar grinder with balls made of agate mortar (RM 200); screen the fine powders with a stainless steel sieve of 200 mesh (75 ㎛); mix the resultant powders well before packing them in glass bottles.

Figure 2. Procedures for sample preparation

2.3 XRF and PGAA methods

XRF is currently one of the most widely used analytical techniques in the determination of the major elements and REE in the rock samples. Large numbers of precise analyses can be made in a relatively short time [5]. XRF was performed using an instrument (BRUKER S8 TIGER) at the Center for Research Facilities of Gyeongsang National University, and PGAA was carried out at NIST Center Neutron Research in US and analytical conditions are presented in Table 1.

Table 1. The analytical condition of PGAA at NIST.

weight	Average Neutron Flux	Irradiation time	Counting time	Neutron Source
496.17 mg	8.8 x 10^7n/cm^2.s	10,000 sec	1,000 sec	Cold-Neutron

3. Results and Discussion

The accuracy of XRF analysis was evaluated by using geo-standards. The samples of USGS and GSJ geo-standards were also prepared in the same way and analyzed using XRF method. The accuracy was given as a deviation of the measured concentration values from the recommended ones. The measured values are in good agreement with the recommended ones except for MnO, MgO and TiO_2.

The mean, variance, standard deviation and relative standard deviation (RSD) of the major elements of the KJG-2 are summarized in Table 2. The analytical results are obtained by measuring each sample in 8 times, and RSD is calculated to be 0.43 to 3.3%. RSD (or coefficient of variation) is an indicator of the precision of the method, meaning that the larger the value of RSD, the greater the deviation of the analysis values relatively. In general, when the RSD is within 3%, it can be said that the accuracy of analysis is sufficiently high.

Table 2. XRF results (major elements) for KJG-2.

KJG-2					
	Av.	**Unc.**	**V**	**s**	**RSD**
SiO_2	71.22	0.25	0.43	0.66	0.93
TiO_2	0.28	0.001	5.00E-06	2.00E-03	0.71
Al_2O_3	15.99	0.051	0.018	0.134	0.84
Fe_2O_3	1.74	0.005	0	0.013	0.75
MnO	0.05	0.0001	1.00E-07	4.00E-04	0.8
MgO	0.44	0.002	2.00E-05	0.005	1.14
CaO	2.53	0.004	1.00E-04	0.011	0.43
Na_2O	4.23	0.005	2.00E-04	0.014	0.33
K_2O	3.43	0.009	5.00E-04	0.023	0.67
P_2O_5	0.09	0.001	8.00E-06	0.003	3.33

Av. Average value (wt.%) Unc. Uncertainty (±)
V Variation s Standard deviation
RSD Relatively Standard Deviation (%)

Table 3 shows a list of PGAA result for twenty-two minor and trace elements in KJG-2. Minor elements such as Cl, In and Sm are contained less than 100 μg/g. Whereas the concentration of minor elements like Fe, Sr, Nb and Th are greater than 5,000 μg/g. In particular, C, I and U are contained at a high concentration of 29,600 ±5,020 μg/g, 14,900±3,360 μg/g, and 24,800±1,580 μg/g, respectively.

In order to determine standard value of geo-standard, it is necessary to cross-check data that obtained using other techniques.

Table 3. PGAA result of KJG-2.

Nuclide	**Conc. [μg/g]**	**Unc. [μg/g]**	**Nuclide**	**Conc. [μg/g]**	**Unc. [μg/g]**
13-C	29600	5020	116-In	16	1
36-Cl	610	6	116-Sn	874	969
46-Sc	198	14	128-I	14900	3360
54-Cr	301	23	141-Ce	8670	1220
57-Fe	9020	315	150-Sm	3	0
59-Ni	291	17	175-Yb	378	44
67-Zn	1380	525	183-W	1940	453
78-Se	120	14	192-Ir	798	28
80-Br	905	79	204-Tl	1520	407
88-Sr	5050	240	233-Th	4810	989
94-Nb	3310	357	239-U	24800	1580

Based on the results of the preliminary studies in this study, the feasibility of development as a Korean geo-standard of granite by comparing the results obtained by geochemical data with various analytical techniques at other institutions like Neutron Activation Analysis (NAA), Inductively Coupled Plasma Mass spectrometry (ICP-MS).

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