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Elemental analysis of granite by instrumental neutron activation analysis (INAA) and X-ray fluorescence analysis (XRF)

A. El-Taher [a,b,*]

[a] Physics Department, Faculty of Science, Al-Azher University, Assuit 71452, Egypt
[b] Physics Department, Faculty of Science, Qassium University, Buraydah 51452, Saudia Arabia

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ABSTRACT

The instrumental neutron activation analysis technique (INAA) was used for qualitative and quantitative analysis of granite samples collected from four locations in the Aswan area in South Egypt. The samples were prepared together with their standards and simultaneously irradiated in a neutron flux of 7×10^{11} n/cm^2 s in the TRIGA Mainz research reactor. Gamma-ray spectra from an hyper-pure germanium detector were analyzed. The present study provides the basic data of elemental concentrations of granite rocks. The following elements have been determined Na, Mg, K, Fe, Mn, Sc, Cr, Ti, Co, Zn, Ga, Rb, Zr, Nb, Sn, Ba, Cs, La, Ce, Nd, Sm, Eu, Yb, Lu, Hf, Ta, Th and U. The X-ray fluorescence (XRF) was used for comparison and to detect elements, which can be detected only by XRF such as F, S, Cl, Co, Cu, Mo, Ni, Pb, Se and V. The data presented here are our contribution to understanding the elemental composition of the granite rocks. Because there are no existing databases for the elemental analysis of granite, our results are a start to establishing a database for the Egyptian granite. It is hoped that the data presented here will be useful to those dealing with geochemistry, granite chemistry and related fields.

1. Introduction

Nuclear analytical methods have been successfully applied for the determination of a great variety of elements in environmental, biological and geological samples. Neutron activation analysis techniques have been improved and have become an excellent tool for such purposes (Hassan et al., 1981, 1982, 1983; Duffey et al., 1970; Senftle et al., 1971; Henkelmann and Born, 1973; Clayton et al., 1983; Zaghloul et al., 1993; De Sena et al., 1995; El-Taher, 2010a, 2010b, 2010c, 2010d, 2010e). Actually different techniques could be used for estimating the trace, minor and major elements of these environmental samples, which are considered as complex samples. The major advantages of NAA are (a) the relative freedom from matrix effects and interferences, (b) high accuracy and (c) very low zero blank contributions. Because nuclear reactions and decay processes are virtually unaffected by chemical and physical structures of the material during and after irradiation, the composition of the matrix has little influence on the induced activity put this technique in the forefront (Brodsky, 1986; Esse and Hopke, 1986; Zaghloul and El-Abbady, 1988; Gordus, 1995; De Sena et al., 1995; Descantes et al., 2001; IAEA, 2001; Scheid et al., 2009; El-Taher, 2010a, 2010b, 2010c, 2010d, 2010e; Hasler, 1993; Vereijke, 1992).

The elemental composition is basic for clarifying the chemical origin of rock formations. The application of INAA in geology permits the correlation of the elemental composition of productive and non-productive layers with either the presence or absence of radioactive goods for nuclear analysis isotopes (Montero et al., 2000). The concentration of elements in Egyptian granite is poorly known, however natural radioactivity levels in Egyptian granite have been described by several authors (Saied et al., 1994; Abdel Hady et al., 1994; Kamal, 1998; El-Shershaby, 2002; Walley El-Dine et al., 2001; Arafa, 2004; El-Taher et al., 2004, 2007; El-Arabi, 2007; El-Arabi et al., 2008; Medhat, 2009).

Granites mainly consist of coarse grains of quartz, potassium feldspar and sodium feldspar and other common minerals such as mica and hornblende. Typical granites are chemically composed of 75% silica, 12% aluminum, less than 5% potassium oxide, less than 5% soda, as well as by lime, iron, magnesia and titania in smaller quantities (Tzortzis et al., 2003). In terms of natural radioactivity, granites exhibit an enhanced elemental concentration of uranium and thorium compared with the very low abundance of these elements observed in the mantle and the crust of the Earth (Faure, 1986).

Since granite was widely used as the building material and because radiation exposure of the population can be increased appreciably by the use of building materials containing above-normal levels of natural radioactivity, it is, therefore important to measure the concentrations of major, minor and trace elements in granite rocks collected from four locations in Aswan area in south

* Corresponding author at: Physics Department, Faculty of Science, Qassium University, Buraydah 51452, Saudia Arabia.
E-mail address: Atef_Eltaher@hotmail.com

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Egypt, in addition to rare earth elements and the natural radionuclides uranium and thorium. These will be available for subsequent evaluations of the possible future environmental contamination due to human activity. This research is our contribution to the understanding of the elemental composition of the granite rock and also to establishing a database.

2. Experimental procedure

2.1. Samples preparation and irradiation

Twenty granite rock samples were collected from four locations in Aswan area as follows: samples collected from Wadi El-Allaqi and Gabel Ibrahim Pacha represent old granite (El-Amin et al., 1987; Mansour, 1991) and samples collected from El-Shalal and Sehyel Island represented young granite (El-Shazly, 1954). The samples have been prepared as finely ground homogenous material. They were crushed to a diameter range of less than 125 μm and greater than 63 μm. The crushed samples were dried at 105 °C to constant weight. Polyethylene capsules filled with 100 mg of powder samples were then irradiated with the standard reference material by thermal neutrons at the University of Mainz Triga research reactor (100 KW_{th}) with a flux of 7×10^{11} n/cm^2 s. Table 1 shows the irradiation cycles. The elemental concentrations in our samples were quantitatively determined by comparison with activities of Dolerite WSE reference material, which was activated simultaneously. The data were collected for various measurements after appropriate cooling times. Table 2 shows the radioisotopes used to calculate the concentration of the analyzed elements as well as their nuclear data (El-Taher et al., 2003; El-Taher, 2007; El-Taher, 2010a, 2010b, 2010c, 2010d, 2010e). Table 3 shows chemical composition of Dolerite WSE and Microgabro PMS standard reference materials.

2.2. Instrumentation

The applied gamma-ray spectrometer consists of a HPGe detector with its electronic circuit. The detector has the following specifications: energy resolution (FWHM) at 1.33 MeV ^{60}Co is 1.70 keV peak to Compton ratio ^{60}Co is 65.2, relative efficiency at 1.33 MeV ^{60}Co is 29.2%, energy resolution (FWHM) at 1.22 MeV ^{57}Co is 686 eV, Operation bias voltage is +2000 dc. The detector is connected to the following components: preamplifier, amplifier, ADC converter and MCA. The measurements were performed and analyzed using the Intergamma Software produced by Intertechnique Deutschland GmbH, Mainz, Germany. The electronic dead time in all measurements was less than 10% and was automatically corrected by the Intergamma software.

2.3. X-ray fluorescence (XRF)

XRF is one of the most important techniques for the analysis of metals and trace elements, which is independent of the chemical form of the element, as INAA. X-rays emitted from an ionized atom have energies characteristic of the element involved and the intensity of an X-ray is proportional to the concentration of an

Table 1
Data for irradiation, cooling, counting times and elements determined.

Irradiation time	Decay time	Counting time	Elements determined
1 min	5 min	4 min	Mg, Ti
5 min	1 h	15 min	Mn, Na, K
6 h	2 d	1 h	Ga, La, Sm, U
6 h	14 d	8 h	Sc, Cr, Fe, Co, Zn, Zr, Rb, Nb, Sn, Ba, Ce, Nd, Eu, Yb, Cs, Lu, Hf, Ta, Th

Table 2
Activation and measurement conditions for detected elements.

Element	Activation product	Energy (keV)	$T(1/2)$	Detection limits (μg/g)
Al	^{28}Al	1179	2.2 m	35
As	^{76}As	559	26.3 h	0.3
Ba	^{131}Ba	496	11.8 d	16.4
Ca	^{49}Ca	3984	8.7 m	10
Ce	^{141}Ce	145	32.5 d	0.3
Co	^{60}Co	1332	5.3 yr	0.2
Cr	^{51}Cr	320	27.7 d	0.5
Cs	^{134}Cs	604	2 yr	0.07
Eu	^{152}Eu	1408	13.3 yr	0.09
Fe	^{59}Fe	1099	44.5 d	75
Ga	^{72}Ga	834	14.1 h	0.6
Hf	^{181}Hf	428	42.4 d	0.06
K	^{42}K	1524	12.4 h	90
La	^{140}La	1596	40.3 h	0.4
Lu	^{177}Lu	208.4	161 d	0.01
Mg	^{27}Mg	1014	9.5 m	1.7
Mn	^{56}Mn	846	2.6 h	0.01
Na	^{24}Na	1369	15 h	1.2
Nb	^{95}Nb	765	35 d	2.2
Nd	^{147}Nd	531	11 d	3.8
Rb	^{86}Rb	1077	18.7 d	2.6
Sc	^{46}Sc	889	38.8 d	0.007
Sm	^{153}Sm	103	46.3 h	0.03
Sn	^{117}Sn	158	13.6 d	9.6
Ta	^{182}Ta	1221	115 d	0.11
Th[a]	^{233}Pa	312	27 d	0.2
Ti	^{51}Ti	320	5.8 m	0.4
U[a]	^{239}NP	106	2.4 d	0.3
V	^{52}V	1434	3.4 m	0.003
Yb	^{169}Yb	198	32 d	0.05
Zn	^{65}Zn	115	244 d	3.2
Zr	^{95}Zr	756	64 d	19

Table 3
Approximate chemical composition of Dolerite WSE and Microgabro PMS standard reference materials.

Element	WSE (%)	PMS (%)	Element	WSE (ppm)	PMS (ppm)
SiO_2	51	47	**Ho**	1	0.5
Al_2O_3	14	17	**La**	30	3
Fe_2O_3	13	10	**Lu**	0.4	0.2
MnO	0.2	0.15	**Mo**	4	2
MgO	5.5	9.5	**Nb**	20	2
CaO	9	12.5	**Nd**	3.5	6
Na_2O	2.5	2	**Ni**	55	110
K_2O	1	0.1	**Pb**	15	3
Ti_2O	2.5	1	**Pr**	8	1
P_2O_5	0.3	<0.1	**Rb**	30	<2
			Sb	<0.5	<0.5
	ppm	**ppm**	**Sc**	30	35
As	1	1	**Sm**	9	2
Ba	350	150	**Sn**	20	5
Be	1	1	**Sr**	420	300
Ce	60	7	**Ta**	1.2	0.2
Co	45	50	**Tb**	1	0.3
Cr	100	350	**Th**	3	0.3
Cs	<1	<1	**Tm**	0.5	0.2
Cu	65	60	**U**	1	0.1
Dy	6	2	**V**	350	190
Er	3	1	**W**	1	1
Eu	2	1	**Y**	30	15
Ga	20	15	**Yb**	3	1
Gd	4	2	**Zn**	120	60
Hf	6	1	**Zr**	200	40