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Original Article

A study of neutron activation analysis compared to inductively coupled plasma atomic emission spectrometry for geological samples in Iran



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ABSTRACT

Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) is widely used for the determination of trace elements in geological samples in Iran. In this paper, we have calculated the detection limits of neutron activation analysis (NAA) for some of the common elements in such samples utilizing the ORIGEN and MCNP codes and verified the simulations using the experimental results of three soil standard reference materials, namely, G02.SRM, G18.SRM, and G28.SRM. The results show that while the detection limit of ICP-AES method is usually in the mg/kg range, it is represented to the μ g/kg range for most of the elements of interest using the NAA method, and the simulations can be verified in a tolerance range of 20%.

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1. Introduction

Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) is an analytical technique used for the detection of trace metals. It is a type of emission spectroscopy that uses the inductively coupled plasma to produce excited atoms and ions that emit electromagnetic radiation at wavelengths characteristic of a particular element [1–3]. The intensity of this emission is indicative of the concentration of the element within the sample. The ICP-AES method is extensively used to determine the amount of trace metal or non-metal elements in geological samples in Iran [4]. The detection limits for some of the common elements determined by this method are listed in Table 1 [5].

As an alternative analysis method, NAA is a highly sensitive method for the accurate determination of elemental concentrations

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in a material. The NAA method is based on the detection and measurement of characteristic gamma rays emitted from radioactive isotopes produced in the sample upon irradiation with neutrons of a nuclear reactor. Samples with unknown elemental concentrations are irradiated with thermal neutrons in a nuclear reactor. Neutrons are absorbed in the nuclei of constituent atoms and later these nuclei emit specific gamma rays regarding to the characteristic of the particular elements. The emitted radiations could be considered as 'fingerprints' for the elements and the amount of radioactivity produced is directly proportional to the amount of its parent isotope in the sample [6,7].

While NAA is a well-known method for determining the elemental composition of geological samples [8—10], it is not a very common one in Iran. The goal of this paper is to show that the NAA could be considered as a proper method to determine the amount of elements in geological samples in Iran regarding the accessibility to the neutron sources of Isfahan Miniature Neutron Source Reactor (MNSR) or the Tehran Research Reactor (TRR).

The detection limit of the NAA technique for the selected elements of Table 1 is investigated in this paper by using the MCNP $\,$

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Table 1Detection limits of ICP-AES method for main evaluated elements in geological samples.

Element	Na	As	Cu	K	La	Mn	Cs	Cr	Sb	Rb	Sc	Zn
Detection Limit (mg/kg)	25	25	2.5	50	5	2.5	20000	2.5	25	25	2.5	2.5

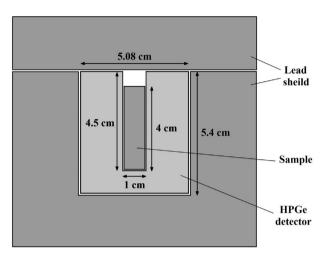


Fig. 1. Detector setup for the NAA spectroscopy.

[11] and ORIGEN codes [12] and the results are evaluated employing the G02.SRM, G18.SRM, and G28.SRM via simulation and experiment and the IAEA soil-7 standard material [13] via simulations.

2. Materials and methods

2.1. Detection limit

In the process of neutron activation analysis, the sample is irradiated in a reactor core with thermal neutrons flux, Φ (n/cm^2 s) for an irradiation time t_i (s). After the irradiation, the sample is cooled for a decay time of t_d (s), and then it is placed in front of a detector. The total counts under the peak during the counting time of t_c is [6]:

$$C = \frac{\varepsilon(E_{\gamma}, g)WA\theta\sigma(E_{a})\Phi(E_{a})\gamma(1 - e^{-\lambda t_{i}})e^{-\lambda t_{d}}(1 - e^{-\lambda t_{c}})}{M\lambda}$$
(1)

where C, counts registered; W, weight of target element; t_i , irradiation time; t_d , decay time; t_c , counting time; E_a , energy of activating particles; A, Avogadro's number; M, atomic weight; θ , target isotope abundance; λ , decay constant; γ , gamma-ray branching ratio; E_{γ} , gamma-ray energy; $\Phi(E_a)$, flux of activating particles; $\sigma(E_a)$, reaction cross-section; E_a , counting geometry factor; E_a , E_a , detector efficiency. For the multiplication of the cross-section and neutron flux, the sum of the multiplication of the cross-section and the fracture of the neutron flux for each energy group is used.

When the background counts under each photo peak is determined, the minimum mean number of counts under the photo peak needed to ensure a false negative rate no larger than 5% is obtained using the "Currie's equation" [14].

$$N_D = 4.65\sqrt{N_B} + 2.71\tag{2}$$

where N_D is the minimum mean number of counts required under the photo peak and N_B is the background counts under the photo peak. For obtaining the detection limit, N_D from Eq (2) is substituted for C in Eq (1) and the equation rearranged in term of mass.

2.2. Simulations

Simulations are performed employing MCNP 4C [11] and ORI-GEN 2.1 [12] codes and then compared to the experimental results for a sample inserted completely inside the detector's hole in order to maximize the solid angle. Activation process is simulated by ORIGEN and the deposited energy inside the detector has been scored using FT8 pulse height tally of MCNP. The thermal.lib and gxuo2brm.lib data libraries are used for ORIGEN simulations. The

Table 2 Nuclear data for the elements [14].

Nuclide	Isotopic abundance	Radionuclide	Thermal cross-section (10 ⁻²⁴ cm ²)	Half-life of produced isotope	Gamma ray Energy (keV)	Gamma ray yield
²⁷ Al	1	²⁸ Al	0.23	134 s	1779	1
⁷⁵ As	1	⁷⁶ As	3.86	26.33 h	559	0.45
⁶⁵ Cu	0.31	⁶⁶ Cu	2.48	306 s	1039	0.09
⁴¹ K	0.0673	⁴² K	1.45	12.36 h	1524	0.18
¹³⁹ La	0.999	¹⁴⁰ La	9.34	1.68 days	1596	0.954
²⁶ Mg	0.11	²⁷ Mg	0.0372	568 s	1014	0.28
⁵⁵ Mn	1	⁵⁶ Mn	13.2	2.58 h	847	0.989
²³ Na	1	²⁴ Na	0.53	14.97 h	2754	1
³⁰ Si	0.031	³¹ Si	0.108	2.62 h	1266	0.0007
⁴⁸ Ca	0.00187	⁴⁹ Ca	1.12	523 s	3084	0.92
⁵⁸ Fe	0.0028	⁵⁹ Fe	1.31	44.4 days	1291	0.43
					1099	0.56
³⁷ Cl	0.24	³⁸ Cl	0.423	2230 s	2167	0.424
					1642	0.319
⁸¹ Br	0.49	⁸² Br	2.58	1.47 days	776	0.835
¹²¹ Sb	0.57	¹²² Sb	6.33	2.7 days	564	0.71
¹³³ Cs	1	¹³⁴ Cs	30.7	752.31 days	604	0.976
⁵⁰ Cr	0.043	⁵¹ Cr	15.2	27.66 days	320	0.1
⁴⁵ Sc	1	⁴⁶ Sc	26.3	83.8 days	889	0.999
⁶⁴ Zn	0.49	⁶⁵ Zn	0.726	244.2 days	1115	0.506
⁸⁵ Rb	0.72	⁸⁶ Rb	0.494	18.64 days	1076	0.09

Table 3 Proposed irradiation, decay, and counting times for isotopes to be measured.

Radionuclide	Irradiation time	Decay time	Counting time
⁶⁶ Cu	60 s	60 s	300 s
³⁸ Cl, ⁵⁶ Mn	600 s	600 s	600 s
⁷⁶ As, ²⁴ Na, ¹⁴⁰ La, ⁴² K, ⁸² Br, ¹²² Sb, ⁸⁶ Rb	1 h	1 day	1 h
¹³⁴ Cs, ⁵¹ Cr, ⁴⁶ Sc, ⁶⁵ Zn	6 h	3 days	12 h

thermal.lib is associated to thermal neutrons and gxuo2brm.lib is associated to photons. The NAA system setup is composed of a rabbit system sending the sample into the reactor core and holds it there for irradiation and then brings it back to the detection laboratory for gamma spectroscopy. Fig. 1 shows the detector setup for the gamma spectroscopy.

The sample is a cylindrical polyethylene container with the diameter of 9 mm and the height of 4 cm which goes directly inside the detector. The approximate weight of a geological sample is 11 g. The common compounds of a geological sample are; SiO_2 , Al_2O_3 , CaO, Fe_2O_3 , and MgO. The amount of each compound in the sample may vary depending on the geological site. For this study, the sample is assumed to be containing 20% SiO_2 , 20% Al_2O_3 , 20% Fe_2O_3 , 20% MgO, and 20% CaO. The sample also contains the trace metal or non-metal elements of Table 1.

Each of these elements, when irradiated with the neutron, can absorb a neutron. When the produced radioisotope is not stable, it may undergo a disintegration process which can result in one or more gamma rays. Table 2 shows the specifications of absorbing and produced isotopes and emitted gamma rays for the elements to be measured [15]. Each one of these elements has an optimum irradiation time, decay time, and counting time under given neutron flux and sample mass. The elements of interest for this study are categorized in four different groups based on the isotope's thermal cross section and half-life of the produced nuclei. For elements with short half-life or high thermal absorption cross sections, the required irradiation time is short. If the thermal absorption of the isotope is low or the half-life of the produced nuclei is long, the irradiation time should be chosen long enough so the number of produced nuclei could reach the saturation amount for maximum activation. The decay and counting times depend mostly on the half-life of the produced nuclei. For a produced nuclei with short half-life, the decay and counting times should be short, otherwise the activation is significantly reduced during the decay time and the background count will increase during a long counting time. For longer half-lives, we can have a longer decay time to minimize the background radiation caused by other activated isotopes without considerable reduction of the activity of the element to be measured and we need a longer counting time to have a larger

Table 4Total background counts in the photo peak margin and the detection limit for each element to be measured.

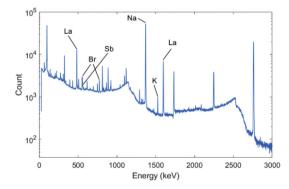
Element	Total N_B in the photo peak margin	Detection limit
Na	5.72E+07	4.30 μg/kg
As	2.27E+09	28.60 μg/kg
Cu	6.87E+08	0.69 mg/kg
K	1.46E+09	1.20 mg/kg
La	8.64E+07	2.50 μg/kg
Mn	1.15E+10	12.50 μg/kg
Cs	7.22E+10	81.90 μg/kg
Br	1.34E+09	44.20 μg/kg
Cl	4.98E+08	0.17 mg/kg
Cr	9.69E+09	0.21 mg/kg
Sb	2.77E+09	57.40 μg/kg
Sc	3.31E+10	2.40 μg/kg
Zn	4.50E+10	1.70 mg/kg
Rb	1.66E+09	11.60 mg/kg

photo-peak in the spectrum. The proposed irradiation, decay and counting times for the elements to be measured are listed in Table 3. In this study, the thermal neutron flux is assumed to be 10^{12} (n/cm^2 s).

The activation process of the elements to be measured is simulated using ORIGEN [12] and the results are imported to MCNP [11] as the radiation source to simulate the detector response.

In order to determine the detection limit of each element, the background count in the photo peak margin of each gamma ray needs to be calculated. As the first step, the activation process of the sample inside the reactor core is simulated employing the ORIGEN code [12]. The simulation is done for all elements listed in Table 1. For each element, the total activity of the sample compounds is calculated during the counting time using the data listed in Table 3.

At the next step, the results obtained by the ORIGEN [12] are imported to the MCNP code [11] in order to simulate the pulse height spectrum of the HPGe detector via the F8 and FT8 pulse height tallies. The GEB option is used in the MCNP [11] code for determination of the photo-peak margin for each one of the trace elements and parameters A, B and C are calculated through curve fitting on the Full Width at Half Maximum at different energies for



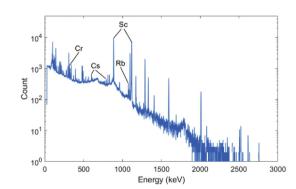


Fig. 2. The recorded spectrum of the G02.SRM standard sample for the measurement times of 1500 s after the decay time of 118 h (left) and measurement time of 3600 s after the decay time of 330 h (right).

Table 5Experimented and simulated mean concentration of trace elements in the G18.SRM standard sample.

Element	Cr	Sc	Rb	La	K	Na
Actual value (mg/kg)	100	2.6	32.0	10.0	10210	3784
Experiment (mg/kg)	108	1.8	28.7	8.5	8834	3211
Experiment relative error (%)	8	31.0	10.3	15.0	13	15
Simulation (mg/kg)	102	3.1	25.9	9.4	10070	3965
Simulation relative error (%)	3	19.2	19.1	6.0	1	5

our detector. At the end, the detection limit of the intended element is computed using Eqs. (1) and (2).

2.3. Experiments

Isfahan Miniature Neutron Source Reactor (MNSR) is a 30 kW pool-type light water research reactor. It has the thermal neutron flux of 10^{12} (n/cm^2 s) at full power [16]. It is designed based on the Canadian SLOWPOKE reactor and considered as an excellent tool for Neutron Activation Analysis.

The HPGe coaxial detector has a cylindrical geometry with the

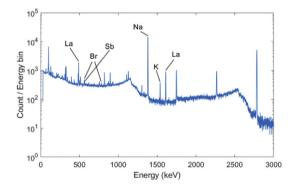
diameter of 5.08 cm and the height of 5.4 cm [17]. The detector has a well tube to hold the sample. This hole has a diameter of 1 cm and depth of 4.5 cm (See Fig. 1). The rest of PC based gamma-ray spectroscopy system consists of a high voltage (HV) power supply, an amplifier, an 8 k multi-channel analyzer (MCA), emulation software and a micro-computer for spectrum data collection and evaluation. The detector operates on a bias voltage of (–Ve) 3000 V, and has a resolution of 2.1 keV (FWHM) for the ⁶⁰Co gamma-ray energy of 1332 keV.

In order to verify the simulations, NAA experiments are performed on the GO2.SRM, G18.SRM, and G28.SRM soil standard reference materials at the Isfahan Miniature Neutron Source Reactor (MNSR). In the experiments, the mass of the GO2.SRM, G18.SRM, and G28.SRM samples are 102.7 mg, 110.5 mg, and 110.1 mg, respectively, and the neutron flux is $5 \times 10^{11} \, (n/cm^2 \, s)$. Because of using soil standard reference materials, it was no necessary to use any special sample preparation method except the routine provisions.

With the intention of minimizing the uncertainties and errors in the experimental data, we used a relative method to calibrate the gamma spectrometry setup. In this method, we first irradiated a sample of GO2.SRM soil standard reference material inside the

Table 6Experimented and simulated mean concentration of trace elements in the G28.SRM standard sample.

Element	Cr	Cs	Sc	Rb	La	K	Sb	Na
Actual value (mg/kg)	58.4	9.4	25.6	97.3	67.8	24488	1.8	35685
Experiment (mg/kg)	68.0	8.0	28.0	82.0	58.0	22402	2.0	32942
Experiment relative error (%)	16.4	15.5	9.4	15.7	14.4	9	8.1	8
Simulation (mg/kg)	59.8	6.5	30.3	78.8	63.7	24209	2.4	37243
Simulation relative error (%)	2.4	30.8	18.4	19.0	6.0	1	28.1	4



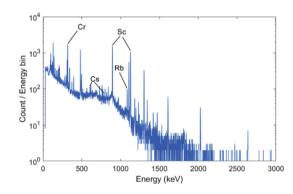
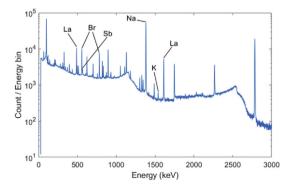


Fig. 3. The recorded spectrum of the G18.SRM standard sample for the measurement times of 1500 s after the decay time of 118 h (left) and measurement time of 3600 s after the decay time of 330 h (right).



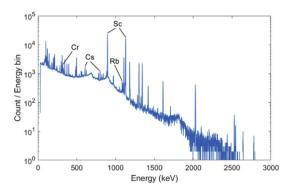
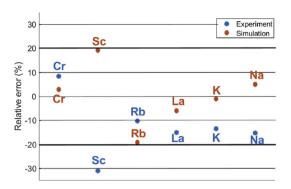


Fig. 4. The recorded spectrum of the G28.SRM standard sample for the measurement times of 1500 s after the decay time of 118 h (left) and measurement time of 3600 s after the decay time of 330 h (right).



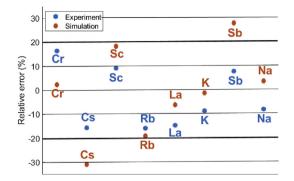


Fig. 5. Relative error of the experiment and simulation results for the G18.SRM (left) and G28.SRM (right) standard samples.

Table 7Reported and simulated mean concentration of trace elements in the IAEA soil-7 standard sample.

Element	Na	K	Sc	Cu	Cl	Mn	La
Report (mg/kg) [9] Simulation (mg/kg) Relative error (%) Element Report (mg/kg) [9] Simulation (mg/kg)	2480 2539 2 As 13.8 18.2	12200 11543 5 Rb 53.0 35.2	8.4 9.1 8.3 Zn 103.0 95.5	11.1 10.1 9.0 Sb 1.6 1.7	1600 1866 17 Br 6.7 8.5	635 652 3 Cr 61.0 52.1	28.9 26.0 10.0 Cs 5.6 6.8
Relative error (%)	31.9	33.6	7.3	6.3	26.9	14.6	21.4

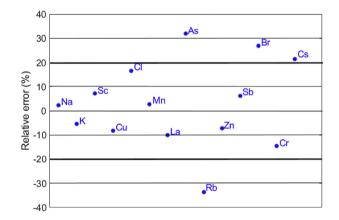


Fig. 6. Relative error of the simulation results for the IAEA soil-7 standard material.

reactor for 270 min and then measured its radiated spectrum for two periods of 1500 s and 3600 s after decay times of 118 h and 330 h, respectively. The first spectrum is used to calibrate the setup

for detection of La, K, Br, Sb, and Na and the second one for Cr, Cs, Sc, and Rb. Fig. 2 shows the recorded spectrum of the G02.SRM sample for the two measurement times. The photo-peaks corresponding to each of the interesting elements are indicated in the figure.

After the calibration, we measured the amount of these elements in the G18.SRM and G28.SRM soil standard reference materials while keeping the irradiation, decay and measurement times, sample size, sample distance to the detector, spatial angle, and other possible error producing parameters the same as the one for G02.SRM sample.

3. Results and discussions

The simulated detection limits for the intended elements in the geological sample are listed in Table 4. Since ORIGEN solves the equations analytically, the uncertainty of the simulations is only due to the MCNP code. 50 million particle histories are tracked in the MCNP code and the uncertainty of the simulations is obtained less than 0.05%. The results show that while the detection limit of ICP-AES method is usually in the mg/kg range, it is represented to the $\mu g/kg$ range for most of the elements of interest using the NAA method, emphasizing the validity of NAA method for determination of 14 elements in geological samples.

The results of the NAA experiments on the G18.SRM and G28.SRM soil standard reference materials are compared to the simulation results of these samples. The results are listed in Table 5 and Table 6. Fig. 3 and Fig. 4 show the recorded spectrums of the G18.SRM and G28.SRM samples, respectively. The spectrums are recorded for the measurement times of 1500 s and 3600 s after the decay times of 118 h and 330 h, respectively, identical to the G02.SRM sample. The relative error (equal to simulated or measured value of the element minus the actual value of the

Table 8Comparison between the detection limit of the NAA and ICP-AES methods for the elements to be measured.

Element	Detection limit of NAA method	Detection limit of ICP-AES method [5]			
Na	4.3 μg/kg	25 mg/kg			
As	28.6 μg/kg	25 mg/kg			
Cu	0.69 mg/kg	2.5 mg/kg			
K	1.2 mg/kg	50 mg/kg			
La	2.5 μg/kg	5 mg/kg			
Mn	12.5 μg/kg	2.5 mg/kg			
Cs	81.9 μg/kg	20000 mg/kg			
Br	44.2 μg/kg	n/a			
Cl	0.17 mg/kg	n/a			
Cr	0.21 mg/kg	2.5 mg/kg			
Sb	57.4 μg/kg	25 mg/kg			
Sc	2.4 μg/kg	2.5 mg/kg			
Zn	1.7 mg/kg	2.5 mg/kg			
Rb	11.6 mg/kg	25 mg/kg			

element in sample divided by the actual value) of the results is illustrated in Fig. 5. As depicted in the figure, the relative error of the simulation and experimental results of the G18.SRM and G28.SRM soil standard reference materials is below 20% for most of the elements. The simulation method used for the geological sample and the soil standard reference materials is the same. Hence, it is concluded that the simulated detection limits of the elements in the geological sample are in a tolerance range of 20%. We also employed the IAEA soil-7 [13] standard material as another benchmark for our simulations. The results are listed in Table 7. The relative error for these results is also depicted in Fig. 6. Again, the relative error of the simulation results is below 20% for most of the elements.

Table 8 compares the obtained detection limits of the NAA method with that of the ICP-AES. As seen, the detection limit of the NAA method is far better than the one of the ICP-AES for most of the intended elements. Therefore, it seems that the NAA method could be proposed as an alternative method for determination of 14 elements in geological samples in Iran, regarding the availability of Tehran research reactor or the NAA center of Esfahan as a reliable, easy to use, and high flux thermal neutron source.

4. Conclusions

This study draws on the applicability of Neutron Activation Analysis (NAA) in contrast to Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) for determination of trace elements in geological samples in Iran, given the fact that Isfahan Miniature Neutron Source Reactor (MNSR) or Tehran Research Reactor (TRR) are available as a reliable, easy to use, and high flux thermal neutron source.

For this study, the activation of a usual geological sample is simulated utilizing the ORIGEN code [12]. The sample contains SiO_2 , Al_2O_3 , Fe_2O_3 , MgO, and CaO as bulk material with trace amounts of Na, Cu, Mn, As, Cs, Br, Cl, Rb, Zn, Sc, Sb, Cr and K. Simulation results show that the NAA method can have lower detection limits in comparison to the ICP-AES method for some of the elements in the geological samples.

In order to verify our simulations, we performed the analysis of the G02.SRM, G18.SRM, and G28.SRM soil standard reference materials at the Isfahan Miniature Neutron Source Reactor. The relative error of the simulation and experimental results of the G18.SRM and G28.SRM soil standard reference materials is below 20% for most of the elements. The simulations are also verified using the reported results for the IAEA soil-7 standard material [13]. Again, the relative error of the simulation results is below 20% for most of the elements. The simulation method used for the geological sample and the soil standard reference materials is the same. Hence, it is concluded that the simulated detection limits of the elements in the geological sample in a tolerance range of 20%.

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