Evaluation for Accuracy and Applicability of Instrumental Neutron Activation Analysis of Geological Materials on Nigeria Nuclear Research Reactor-1(NIRR-1)

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Abstract

Evaluation of the composition of some geo-standard reference materials; Fly ash 1633b, SO-2, SARM-1, SARM-52, W-2, DNC-1, BIR-1, AGV-1, IAEA SL-3, and IAEA Soil-7 following instrumental neutron activation analysis on NIRR-1 Nuclear Reactor to ascertain accuracy and applicability of the method in the Centre for Energy Research and Training (CERT), Ahmadu Bello University, Zaria, Nigeria was performed. The results showed an insignificant maximum deviation error of 0.304 to 0.393% in all the geo-standard reference materials. The analysis of 15 elements in all geo-standards observed, provided some level of assurance in analysing geological materials of varying matrices using instrumental thermal neutron activation analysis (ITNAA) on NIRR-1 for some elements. The dendrogram from the cluster analysis of the values for; Mn, Sc, Rb, and Yb revealed no statistically significant difference. These results showed a general agreement between the certified or literature values and those obtained using the present irradiation and counting schemes in CERT for the ten geo-standard reference materials and are thus can be referred to as established elements in geological studies using the present experimental set-up in CERT.

Keywords: INAA, geological material, geo-standard reference material, relative standard deviation, cluster analysis, dendrogram, CERT, NIRR-1.

1.0 INTRODUCTION

The accurate determination of trace concentrations of elements in complex matrices such as rocks, soils and sediments are very important in both mineral exploitations and soil fertility mapping. Methods that can be used for the analysis of such materials are among others, atomic absorption spectrometry (AAS), inductively coupled plasma-atomic emission spectrometry (ICP-AES) or mass spectrometry (ICP-MS), all of which require dissolution and sometimes subsequent chemical treatment of the sample to ensure full dissolution with the inherent risk of contamination.

However, a method with widest acceptance and unique advantages in areas such as sensitivity, speed, precision, cost, low matrix effects, and preservation of sample known as Instrumental Neutron Activation Analysis (INAA) has been used extensively to determine the concentration of elements in some varieties of geological standard materials available in centre for energy research and training, CERT-Nigeria.

Instrumental Neutron Activation Analysis (INAA) as an analytical technique involving the use of neutrons usually sourced from nuclear reactors have been exploited by analysts especially in the analysis of geological materials since the discovery of the method by Hevesy *et al.*, (1936). The exploitation of INAA technique commenced in the Centre for Energy Research and Training (CERT), Ahmadu Bello University, Zaria, using the Miniature Neutron Source Reactor (MNSR), code-named Nigeria Nuclear Reactor-1 (NIRR-1), the 14-MeV Neutron Generator and a 5 Ci ^{241}Am/Be source with the analysis of trace elements in geological samples specifically relating to soil fertility and solid mineral studies have been studied by Onojah *et al.,* (1995).

However, the acquisition of a Miniature Neutron Source Reactor (MNSR), Nigeria Research Reactor – I (NIRR-1) was commissioned for operation in February 2004, revolutionized the analysis of solid samples of geological origin in CERT and Nigeria. Early works using this facility demonstrated that the data being generated ought to be accompanied with high level of accuracy and must in fact be an unavoidable requisite. In addition, the obvious objectives for INAA usage including sample matrix compatibility with the natural reference standards (i.e. the secondary reference material) and adequate robustness for use in routine work (Oladipo, 1987), it is important and indeed a must that the analyst consistently estimates the degree of uncertainty associated with data suites being generated (Angel Rids *et al*., 1998).

Control samples otherwise referred to as geo-standards should have a high degree of similarity to the actual samples being analyzed, in order to draw reliable conclusions on the performance of the measurement system. Control samples must be so homogeneous and stable at any given time. Quality control samples can be purchased as certified reference materials (CRMs) or may be prepared in-house (Oladipo *et al*., 1989). In the latter case, sufficient quantities should be prepared to allow the same samples to be used over a long period of time.

Scientist in this area carried similar work employing different nuclear techniques revealed that the level of accuracy in analysis of geological samples and ceramic (pottery) is controlled by the accuracy obtained in measurement of different standard materials (Molnar et al., 1993; De Corte *et al.*, 2004). CERT is presently a leading centre of excellence in the use of the nuclear analytical facilities and techniques in Nigeria. It is aware of the responsibility attached fact that, by and large, the data being generated from its facilities will eventually form basis for many decisions. Therefore accuracy must be a watchword of all analysts involved. Such a vision can be sustained only if adequate quality assurance program is established. CERT has provided an in-house (self) assessment of adherence to accuracy policy using well characterized international reference materials which have been treated as representing the unknown geological samples of slightly different matrices. The analysis of quality control samples (i.e. geo-standard reference materials) with construction of quality control charts has been suggested as a way to build in quality checks on results as they are being generated (P. Bode, 2010)

The aim of this investigation involves a total evaluation for accuracy of the relative INAA method currently being employed in the analysis of geological samples in CERT. Essentially, the procedure will involve analyzing a set of geological reference materials of varying matrices and the results compared with literature or the certified values. A selection of well characterized international reference standards that approximate the matrices of different geological samples encountered in real samples will be accommodated in the investigation. This method has proven quite useful in evaluating quality assurance problems

2.0 EXPERIMENTAL

2.1 Samples Evaluation

The geological reference standards used in this investigation were chosen with two criteria in mind. The first was connected to the enormous variability of samples commonly referred to as geological; undoubtedly, there will be also variable reproducibility in their matrices. Efforts were made to accommodate most geo-standard reference materials that are employed in the analysis of geological samples on NIRR-1 using relative INAA method. The idea of multi-standards permitted a good test of the robustness of the method of choice of the INAA technique against matrix interferences. The second criterion was that the selected geo-standards should imitate the real geological samples in composition. Therefore, geo-standards whose compositions range from low to high concentrations in the elements of interest were selected. These factors as well as availability of the identified standards guided the final choice of the standards are listed in Table 1.

2.2 Sample Preparation and Analysis

From each reference standard about 150-200 mg sample were weighed into pre-cleaned, polyethylene sample sheets that were then sealed. Multi-element analysis of each reference sample and a selected standard (for the short irradiations) were performed using neutron activation analysis. The samples were irradiated using Nigeria Research Reactor-1 (NIRR-1) at a neutron flux of 2.5 x 10^{11} n/cm^2 s in the outer irradiation channels. Long-lived irradiations involved neutron irradiation of a batch of reference samples and standards for 6hrs at 5.0 x 10^{11}n/cm^{2}sec in the inner irradiation channels using the same facility.

Essentially, each reference sample or standard underwent two irradiations procedure descript in a work performed by Jonah *et al.,* 2005 (i.e short and long irradiations). For the short irradiation, each sample or standard were irradiated for 1 minute, allowed to decay for a few minutes, followed by ten minutes counting on a HPGe detector coupled to its associated electronics. Nuclides with short-lives analysed during this first short count included sodium, calcium, aluminium, magnesium, vanadium and titanium. These samples and standards were recounted after two to three hours decay to analyse those nuclides of intermediate half-lives. The elements in this group included sodium, potassium, manganese and dysprosium. For the long-lived irradiations, first counting exercise began four days after irradiation, each sample or standard were counted for thirty minutes to analyse those nuclides with half-lives mainly in the order of hours or few days.

The same batch of samples/standards were recounted for one hour each after nine to ten days decay in order to analyse those nuclides with half-lives in the order of days and years.

A total of 25 elements were determined. For many of these elements, the limits of detection with INAA were below experimentally detectable values. These dataset were ultimately reduced to 18 elements for each of the eight geo-standard reference materials analysed. Those elements retained were sodium, magnesium, aluminium, calcium, potassium, vanadium, titanium, iron, lanthanum, samarium, ytterbium, scandium, cobalt, rubidium, caesium, barium, europium and hafnium.

3.0 RESULTS AND DISCUSSION

The determinations of 18 elements in geological matrices were obtained accurately. The nuclides used in the determination are given in Table 2 along with necessary experimental conditions. The values obtained in the present study were compared with the certified values in Table 3 (a to f). In most cases, the agreement between the results of this study and those of the literature or certified was excellent with maximum deviation error of 0.304% in k for SARM-52, 0.326% in Ca for SARM-52, 0.327% in Rb for IAEA SL-3, 0.380% in Rb for SARM-52, and 0.393% in Eu for IAEA SL-3. It was observed that the accuracy recorded varied with the geological matrix, for example, perfect agreement was recorded for Na in Fly ash 1633b (bituminous coal), Fe in SARM-1 (granite), Sc in IAEA-SL-3 (sediment standard) and Co in SARM-52 (synite).

There were discrepancies in some elements; an example is the Mg value in the Fly ash 1633b.This discrepancy could possibly be sourced to the high Al concentration in the standard. The value of Mg may therefore require correction from spectral interference caused by threshold nuclear reaction $^{27}Al(n, p)^{27}Mg$ on $^{26}Mg(n, \gamma)^{27}Mg$. Other elements which showed discrepancies include Ca in SARM-52, Cs in AGV-1, Eu in Fly ash 1633b and IAEA SL-3, Hf in W-2 and Ba in W-2. Figure 1 shows the dendrogram obtained from the cluster analysis of the data pooled from the certified values and the results of this work using Minitab Version 15 on Windows. The dendrogram essentially summarised observations from the comparison between this work and the certified values of the geo-standards employed. While elements Na, K, Mg, Al, Ca, V (all major elements in geological materials), Mn, Sc, Rb, and Yb revealed no statistically significant difference, Co and Eu were slightly different. Results obtained for Fe were generally significantly different from the certified values. The results of those elements that showed discrepancy between the certified values and results of this work

4.0 CONCLUSION

The results of this work have provided an assurance on quality for 15 elements in geological materials of varying matrices using instrumental thermal neutron activation analysis (ITNAA). It is believed this number can be increased through radiochemical thermal neutron activation analysis (RTNAA) method.

The results obtained in this work assure that the INAA technique being employed for analysis of geological materials is fit for the purpose. Results of this investigation have shown that no single geo-standard reference material can provide alone the accuracy required in the relative INAA method of analytical geochemistry. Though may be difficult in practice, numerous geo-standard reference materials are needed to ensure comprehensive quality control. Since no single standard showed an all-round high accuracy for all the elements of interest to this work, routine analysis of geological materials requires the availability of well-characterized internationally-recognized geochemical reference materials that approximate the matrices commonly encountered (Oladipo *et al.*, 1989).

It is also grossly inadequate to limit accuracy checks on data being generated by a laboratory to its performance in the inter-laboratory network participation which occur through inter-comparison exercise only rather through a regular and comprehensive routine geo-standard reference material analysis.

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Table 1: Description of the Geo-Standards Analysed

Certified Geo-standard Reference Material	Description of the Geo-standard
IAEA Soil – 7	Well – characterised International Atomic Energy Agency-produced topsoil standard.
Andesite, AGV-1	AGV-1 is an andesite rock from Oregon, U.S Geological Survey.
DNC-1	A dolerite collected from a quarry in North Carolina and prepared by the U.S. Geological Survey.
W-2	A diabase collected from a quarry in Virginia produced by the U.S. Geological Survey.
SARM-1 and SARM-52	SARM-1 is granite while SARM-2 is a syenite, both standards are produced under the broad name South African Reference Material by South African Bureau Standards.
SO-2	It is a B-horizon soil reference standard produced at Ontario, Canada.
Fly-Ash 1633b	A bituminous coal fly ash produced by NIST in the USA.
IAEA-SL-3	A lake sediment reference material produced by the IAEA, Vienna.
BIR-1	An Iceland basalt reference standard produced by the US Geological Survey.

Table 2: Irradiation and Counting Conditions and some Nuclear Parameters of Radionuclides of Interest

Element	Radionuclides	Half-life	Peak-line (Kev)	Conditions*
Al	^{28}Al	2.23m	1778.8	a
As	^{76}As	26.3h	559.1	c,d
Ba	^{131}Ba	11.7d	496	d
Ca	^{49}Ca	8.72m	3084	a
Co	^{60}Co	5.27y	1173, 1332.5	d
Cr	^{51}Cr	27.7d	320	d
Cs	^{134}Cs	2.06y	604, 796	d
Dy	^{165}Dy	2.35h	94.7	b
Eu	^{152}Eu	12.4y	121.8, 1407.9	d
Fe	^{59}Fe	44.6d	1099, 1291.6	d
Hf	^{181}Hf	42.4d	482	d
K	^{42}K	12.36h	1524.7	b,c
La	^{140}La	40.23h	487.6, 1596	c,d
Mg	^{27}Mg	9.5m	1014	a
Mn	^{56}Mn	2.582h	846.6, 1811	b
Na	^{24}Na	15.02h	1368.5, 2754	b,c
Rb	^{86}Rb	18.65d	1076.6	d
Sb	^{122}Sb	2.72d	564	c,d
Sc	^{46}Sc	83.8d	889, 1120	d
Sm	^{153}Sm	46.Sh	103	c
Ta	^{182}Ta	115d	1221	d
Th	^{233}Pa	27.0d	311.9	c
Ti	^{51}Ti	5.76m	320	a
U	^{239}Np	2.35d	106, 277.60	c
V	^{52}V	3.76m	1434	a

Conditions*

a= (t_{irr} = 1 min, t_{decay} = <5% dead time, t_{count} = 10min, geometry = 5 cm)

b= (t_{irr} = 1 min, t_{decay} = 120 – 180 min, t_{count} = 10 min, geometry = 1cm)

c= (t_{irr} = 1 min, t_{decay} = 3-4 d, t_{count} = 30 min, geometry = 5cm)

d= (t_{irr} = 1 min, t_{decay} = 9-10d, t_{count} = 1 h, geometry = 1 cm)

Table 3: Results obtained in the present study (NIRR-1(Found)) compared with the certified values (a to f)

(a)

Standard Ref. Material	Na			Mg			Al			Ca		
	Certified Value	NIRR-1 (Found)	Deviation Error (%)	Certified Value	NIRR-1 (Found)	Deviation Error (%)	Certified Value	NIRR-1 (Found)	Deviation Error (%)	Certified Value	NIRR-1 (Found)	Deviation Error (%)
Fly Ash 1633b	0.201	0.20±0.06	0.004	0.482	1.22±0.2	-1.531**	15.05	14.48±0.012	0.037	1.51	1.41±0.10	0.066
SO-2	1.90±0.05	1.87±0.02	0.015	0.54±0.03	0.505±0.38	0.064	8.07±0.18	7.28±0.01	0.097	1.96±0.10	1.61±0.09	0.178
SARM-1	No Info.	2.73±0.01	-	No info	0.71	-	6.39	6.35±0.03	0.006	0.56	0.47±0.23	0.160
SARM-52	No Info	0.053±0.13	-	0.36	BDL	-	4.96	4.74±0.01	0.044	0.26	0.175±0.33	0.326
W-2	1.63	1.65±0.02	-0.012	3.84	3.91±0.08	-0.018	8.17	7.61±0.01	0.068	7.76	7.00±0.04	0.097
DNC-1	1.402	1.51±0.02	-0.077	6.11	6.16±0.05	-0.008	9.70	9.22±0.01	0.049	8.21	6.91±0.05	0.158
BIR-1	1.34	1.45±0.02	-0.082	5.85	5.00±0.08	0.145	No info	8.31±0.02	-	9.51	8.97±0.05	0.056
AGV-1	3.16	3.36±0.01	-0.063	0.92	1.33±0.32	-0.445	9.07	8.80±0.02	0.029	3.53	3.10±0.08	0.121
IAEA SL-3	0.669	0.727±0.03	-0.086	2.70	2.90±0.07	-0.074	2.45	2.48±0.02	-0.012	11.1	10.9±0.04	0.018
IAEA-Soil-7	0.240	0.277±0.09	-0.154	1.13	0.583±0.12	0.484	4.70	4.84±0.01	-0.029	16.3	16.1±0.03	0.012

(b

Standard Ref. Material	Ti			V			Fe			La		
	Certified Value	NIRR-1 (Found)	Deviation Error (%)	Certified Value	NIRR-1 (Found)	Deviation Error (%)	Certified Value	NIRR-1 (Found)	Deviation Error (%)	Certified Value	NIRR-1 (Found)	Deviation Error (%)
Fly Ash 1633b	0.791	0.742±0.07	0.061	295.7	259.8±0.03	0.120	7.78	7.31±0.01	0.060	94	88.8±0.01	0.055
SO-2	0.86±0.02	0.65±0.07	0.244	64±10	51.3±0.07	0.191	5.56±0.16	5.38±0.02	0.032	No info	48.5±0.01	-
SARM-1	No Info	BDL	-	No info	5.44±0.66	-	1.01	1.01±0.09	0	109	106±0.01	0.027
SARM-52	0.78	0.58±0.08	0.250	346	298.8±0.02	0.136	No info	16.1±0.01	-	No info	17.9±0.01	-
W-2	0.64	0.48±0.09	0.250	260±12	235±0.03	0.096	7.57	7.01±0.01	0.073	10±0.59	11.6±0.09	-0.160
DNC-1	0.288	0.312±0.15	-0.083	148±8.3	144±0.04	0.027	6.97	7.61±0.01	-0.091	3.6±0.30	3.54±0.05	0.016
BIR-1	0.575	0.59±0.12	-0.026	310±11	293±0.03	0.055	7.90	9.73±0.02	-0.231	0.63±0.07	0.684±0.22	-0.085
AGV-1	0.63	0.681±0.12	-0.081	120	106±0.07	0.116	4.46	3.88±0.03	0.130	38±2	36.7±0.01	0.034
IAEA SL-3	0.261	0.283±0.16	-0.084	-	27.2±0.08	-	No info	1.06±0.07	-	22.5	20.6±0.01	0.084
IAEA-Soil-7	0.30	0.34±0.13	-0.133	66	79.4±0.05	-0.203	2.57	2.84±0.02	-0.105	28	BDL	-

(c)

Standard Ref. Material	Sm			Yb			Sc		
	Certified Value	NIRR-1 (Found)	Deviation Error (%)	Certified Value	NIRR-1 (Found)	Deviation Error (%)	Certified Value	NIRR-1 (found)	Deviation Error (%)
Fly Ash 1633b	20	20.9±0.05	-0.045	(7.6)	8.17±0.04	-0.075	(41)	39.3±0.01	0.041
SO-2	No Info	12.1±0.05	-	No Info	3.44±0.08	-	No Info	12.6±0.01	-
SARM-1	15.8	19.8±0.05	-0.253	14.2	13.3±0.03	0.063	No Info	0.459±0.06	-
SARM-52	4.24±0.05	BDL	-	No Info	1.45±0.15	-	No Info	BDL	-
W-2	-	-	-	2.1±0.2	1.83±0.18	0.128	-	-	-
DNC-1	No Info	1.53±0.05	-	2. ±0.1	1.93±0.14	0.035	31±1.0	33.1±0.01	-0.064
BIR-1	(1.1)	1.01±0.05	0.081	1.7±0.1	1.99±0.13	-0.170	44±1	46.9±0.01	-0.065
AGV-1	5.9±0.4	6.37±0.05	-0.079	1.72±0.2	1.47±0.2	0.145	12±1	12.4±0.01	-0.033
IAEA SL-3	3.83	4.27±0.05	-0.114	1.89	2.19±0.11	-0.158	3.91	3.91±0.01	0
IAEA-Soil-7	5.1	5.96±0.06	-0.168	2.4	3.02±0.07	-0.258	8.3	BDL	-

(d)

Standard Ref. Material	Co			Rb			Cs		
	Certified Value	NIRR-1 (Found)	Deviation Error (%)	Certified Value	NIRR-1 (Found)	Deviation Error (%)	Certified Value	NIRR-1 (found)	Deviation Error (%)
Fly Ash 1633b	(50)	44.9±0.01	0.102	(140)	111±0.07	0.207	(11)	12.13±0.06	-0.102
SO-2	9±2	8.07±0.05	0.103	78±6	70.0±0.09	0.102	No Info	BDL	-
SARM-1	No Info	1.334±0.19	-	325±3	314±0.02	0.033	No Info	BDL	-
SARM-52	81±1.1	81.0±0.01	0	20±4	27.6±0.26	-0.38	No Info	BDL	-
DNC-1	57±2.2	60.3±0.01	-0.057	4.5±4	BDL	-	No Info	BDL	-
BIR-1	52±2	55.0±0.01	-0.058	No Info	BDL	-	No Info	BDL	-
AGV-1	15±1.2	16.1±0.02	-0.073	67±1	62.9±0.08	0.061	1.3±0.1	0.928±0.34	0.286
IAEA SL-3	No Info	2.88±0.05	-	38.8±2	26.1±0.10	0.327	1.38±0.2	1.43±0.18	-0.036
IAEA Soil-7	8.9±1.1	10.4±0.03	-0.168	51±1	59.6±0.09	-0.168	5.4±0.1	6.10±0.06	-0.129
Fly Ash 2689	(48)	46.3±0.01	-	No Info	137±0.07	-	(11)	12.0±0.06	-0.091

(e)

Standard Ref. Material	Ba			Eu			Hf		
	Certified Value	NIRR-1 (Found)	Deviation Error (%)	Certified Value	NIRR-1 (Found)	Deviation Error (%)	Certified Value	NIRR-1 (found)	Deviation Error (%)
Fly Ash 1633b	709±14	686	0.032	(4.1)	2.96±0.04	0.278	6.8	7.58±0.05	-0.114
SO-2	966±67	1029±0.04	-0.065	No Info	3.27±0.05	-	No Info	23.8±0.02	-
SARM-1	No Info	70.7±0.27	-	0.35±0.02	BDL	-	No Info	BDL	-
SARM-52	No Info	BDL	-	No Info	0.28±0.25	-	No Info	BDL	-
W-2	170±10	105±0.37	0.382	1.0±0.06	0.79±0.08	0.210	2.6±0.18	1.74±0.15	0.331
DNC-1	118±11	120±0.32	-0.016	0.59±0.03	0.54±0.17	0.084	No Info	1.82±0.20	-
AVG-1	1230±16	1033±0.05	0.160	1.60±0.1	1.32±0.06	0.175	5.1±0.4	5.32±0.05	-0.043
IAEA SL-3	No Info	140±0.20	-	0.66±0.1	0.40±0.12	0.393	9.1	9.07±0.03	0.003
IAEA-Soil-7	159±15	127±0.23	0.201	-	-	-	-	-	-
BIR-1	-	-	-	0.55±0.05	0.44±0.16	0.200	0.6±0.08	BDL	-

No Info means there is no information about this element in the certificate that accompanied the standard ** Result requires correction from spectral interference caused by the nuclear reaction $^{27}Al(n, p)^{27}Mg$ on $^{26}Mg(n, \gamma)^{27}Mg$. BDL means value was below the detection limit. Results are in ppm (ugg^{-1}) unless otherwise stated,

$$\text{Deviation Error}(\%) = \left(\frac{Certified\ value - Experimental\ value}{Certified\ value}\right)\%$$

(f)

Standard Ref. Material	K		
	Certified Value	NIRR-1 (Found)	Deviation Error (%)
Fly Ash 1633b	1.95	2.019	0.035
SO-2	2.45	2.055	0.163
SARM-1	4.14	3.36	0.188
SARM-52	0.21	0.146	0.304
W-2	0.52	0.37	0.288
DNC-1	0.194	BDL	-
AVG-1	2.42	2.079	0.141
IAEA SL-3	0.8740	0.836	0.043
IAEA-Soil-7	1.21	1.205	0.004
BIR-1	-	-	-