

Neutron-induced prompt gamma-ray analysis of standard reference materials of international atomic energy agency and tannery sediment of Bangladesh

Shaikh A. Latif¹, Oura Y.², Katada M.², Islam M. Amirul³, Khandaker M. Uddin⁴, Ibrahim M. Mehedi^{1,5}

¹Faculty of Engineering, King Abdulaziz University, Jeddah 21589, Kingdom of Saudi Arabia

²Department of Chemistry, Graduate School of Science, Tokyo Metropolitan University, Hachioji, Tokyo 192-0397, Japan

³Institute of Nuclear Science and Technology, Atomic Energy Research Establishment, Savar, Dhaka, Bangladesh Atomic Energy Commission

⁴School of Healthcare and Medical Sciences, Sunway University, 47500, Kuala Lumpur, Malaysia

⁵Center of Excellence in Intelligent Engineering Systems (CEIES), King Abdulaziz University, Jeddah, Saudi Arabia

Abstract— The aim of this study is to determine the multi-elements in International Atomic Energy Agency (IAEA) standard reference material and tannery sediment of Bangladesh, non-destructively with high accuracy. To achieve this goal, neutron-induced prompt gamma-ray analysis (PGA) was applied to the samples. Elemental concentrations in all samples were calculated using comparison method. This work shows that multi-elements (B, Al, Si, Cl, K, Ca, Ti, Cr, Mn, Fe, Sm and Gd) in IAEA standard reference material (IAEA-SRM-Soil-7) was easily and promptly determined by PGA with high accuracy ($\pm 10\%$ in most elements). This study presents that the elements B and Si are determined non-destructively only by PGA. In the present research, the concentration of Cr in the tannery sediment was found (1.45 ± 0.04) % and it is very consistent (within 5.2%) with the instrumental neutron activation analysis (INAA) value. This study reveals that PGA is a suitable method for rapid identification of the environmental pollution problem due to higher levels of Cr arises from tannery industries as well as other sources of Bangladesh. Finally, it is concluded that PGA is the best choice for multi-elements determinations in environmental samples.

Keywords— PGA • IAEA-SRM • Tannery sediment • Multi-elements • Toxic chromium

I. INTRODUCTION

Neutron-induced prompt gamma-ray analysis (PGA) was first introduced as a practical method during the 1960's [1-3]. After that PGA has been using for multi-element analysis into different fields of study. In geologic and meteoritic materials numerous elements (H, B, N, Si, S, Ca, Cd and Gd) can be non-destructively determined by PGA, most of which are often difficult or impossible to be measured by instrumental neutron activation analysis (INAA) [4]. Due to this reason PGA can be used as a complementary analytical tool to INAA [5]. Recently, an overview for the determination of elemental concentrations in several matrices especially in forensic samples are illustrated by Acharya R and Pujari PK [6].

This study involves the determination of multi-elements in the International Atomic Energy Agency geological standard

reference materials (soils and sediments) as well as environmental polluted samples (e.g. tannery sediment) of Bangladesh. International Atomic Energy Agency standard reference materials (IAEA-SRM) are widely used in the different laboratories of the world especially in its member states as a standard for the quality assurance of the analytical results. Since Bangladesh is one of the member states of IAEA, neutron activation analysis (NAA) laboratory at the institute of nuclear science and technology (INST) as well as other laboratories of Bangladesh Atomic Energy Commission (BAEC) usually receives several standards from IAEA and uses for various research purposes. The quality control of the analytical results are very important. In these cases standard reference materials plays a vital role. So standard reference materials should be selected very carefully for achieving perfect and reliable results. Small amounts of standards are to be used as it is very expensive. From many years before IAEA geological standard reference materials are being used as standard for many research purposes in the different laboratories of the world. But in the IAEA-SRM-Soil-5, Soil-7 and lake sediment SL-1 and SL-3 some major, minor and trace constituents have not certified [7-10]. Latif SA et al. [11] shows that Si can be easily determined non-destructively in meteorites especially when small amounts are desirable. Other elements (B, Ca, Ti, S) can also be determined with high accuracy in meteorites. The applicability of PGA for analyzing multi-elements (B, Na, Mg, Al, Cl, K, Ca, Ti, Mn, Fe, Sm, and Gd) in irregularly shaped large meteorite samples with good accuracy and precision are described by Latif SA et al. [12]. So, this study emphasis for the non-destructive determination of the non-certified major, minor and trace elements in that SRM's using highly sophisticated PGA facility of JRR-3M reactor at the Japan Atomic Energy Agency (JAEA).

The tannery industries of Bangladesh are discharging liquid wastes directly to the environment without effluents treatment. Due to this problem the contamination levels in the environment increasing rapidly. The adjacent area of tannery industries and river in which the effluents comes directly from industries through drains are highly polluted. It's mainly due to chromium

and other toxic elements. Because chromium is used as a reagent for lather processing in the tannery industries. Chromium is a toxic element and it causes adverse effects on human health. To create the awareness of the public it is necessary to use a rapid and reliable method for quantification of toxic elements in the environment especially chromium. So this study also focuses on the applicability of PGA for the determination of multi-elements in environmental polluted samples (e.g. tannery sediment) of Bangladesh.

II. PRINCIPLES OF PGA

PGA is based on the detection of capture gamma-rays emitted by a target material while it is being irradiated with neutrons. Neutrons of any energy may be used for PGA. When a neutron interacts with the target nucleus via a non-elastic collision, a compound nucleus forms in an excited state. The excitation energy of the compound nucleus is due to the binding energy of the neutron with the nucleus. The excited compound nucleus will almost instantaneously (within 10-14 s) de-excite into a more stable configuration through emission of one or more characteristic prompt gamma rays. In many cases, this new configuration yields a radioactive nucleus which may undergo delayed emission of gamma-rays, with a decay rate (half-life) characteristic of radioactive nucleus. The prompt gamma-rays are used in PGA, whereas the delayed gamma-rays are used in NAA.

In PGA, nearly every neutron capture yields gamma-rays that are potentially usable for analysis of the target element. However, the energy spectrum of capture gamma-rays is fairly complex for most nuclides. The analytical methodology of PGA is similar to INAA, except of course, no decay corrections are necessary.

Three kinds of neutron sources are used for PGA: nuclear reactors, radioisotope neutron sources, and neutron generators with accelerators. Among these, the nuclear reactor is the primary choice as the neutron source for PGA because it provides the highest neutron flux and hence the highest analytical sensitivities. Typically, the sample will not acquire significant long-lived radioactivity and may be removed from the facility and used for other purposes.

Reactor-based PGA can be performed by: (1) irradiating the sample inside of the reactor while measuring the prompt gamma-rays with a detector positioned outside the reactor, or (2) irradiating the sample outside of the reactor with a guided neutron beam while measuring the prompt gamma-rays with a detector positioned near the sample. Method (1) provides the highest neutron flux but suffers from low analytical sensitivity due to the low gamma-ray detection efficiency caused by the detector being placed far from the sample. The operating environment is also more cumbersome to use and more hazardous to personnel. Method (2) provides greater gamma-ray detection efficiency because the detector can be located closer to the sample. That, along with recent improvements in the design of cold and thermal neutron guided beams and an easier to use operating environment, makes it the method of choice.

For the quantitative analysis of elements in geological samples by PGA, comparative (multi-standard) or external and internal mono-standard methods can be used. In comparative

method, one or more standard samples containing known amounts of all of the elements to be determined are run. The element standards can artificially made from high-purity elements and compounds, or they can be rock powders of standardized composition. Samples and standards must be irradiated in the neutron beam and counted under identical conditions.

In order to avoid the procedure of irradiating several standards, which consumes precise 'beam time' the external mono-standard method can be used [13]. In this method, a single element standard is periodically run in order to 'calibrate' the PGA setup. All element concentrations are determined using experimentally derived factors relating those elements with the single element calibration.

In order to avoid problems with neutron absorption and scattering within the sample, and the attenuation of prompt gamma-rays by the sample itself, the sample sizes must generally be kept small, in the two aforementioned methods. An internal mono-standard method was proposed by Sueki K et al. [14] for multi-element PGA analysis of relatively large samples (i.e. larger than the beam cross-sectional area and the effective neutron penetration depth). This method uses a single element of known composition in the sample as a standard. Other elemental concentrations are calculated from experimentally determined factors relating those elements to the standard element. In the present research work, small geological samples were analyzed by the comparison method.

III. EXPERIMENTAL

A. Sample preparation:

The samples used in this study are listed in Table 1. All of the samples (except tannery sediment) were originally supplied as powder. The sediment sample collected from the tannery industry area of Bangladesh was at first naturally dried in a clean room and then it was again dried in an oven at a temperature of about 500C until they attained constant weight. An agate mortar was used to make powder samples from the dried sediments for ensuring the homogeneity. The IAEA-SRM-Soil-7 was an aliquot of a homogeneous powder prepared by IAEA [8]. Standard reference material coal fly ash 1633b was a homogeneous powder prepared by the National Institute of Standards and Technology (NIST), Gaithersburg, USA. The Allende sample was an aliquot of a homogeneous 'standard' powder prepared by E. Jarosewich at the Smithsonian Institute, USA from several hundred grams of the meteorite. An aliquot of each sample (100-250 mg) was heat-sealed between two layers of fluorinated ethylene propylene (FEP) film. The cross-sectional size of each sample was approximately 1.5 cm × 1.5 cm.

A basaltic rock powder, JB-1 (250.9 mg), prepared by the Geological Survey of Japan [15] was used as a standard sample for determining all elements except B, Mg, S, Cl, Cr, Co and Ni. Reagent grade chemical standards B (solution, 52.89 μ g), Mg (133.91 mg), Na₂SO₄ (230.44 mg), NH₄Cl (100.96 mg), Cr (322.65 mg), Co (36.90 mg) and Ni (82.53 mg) were used as standards for B, Mg, S, Cl, Cr, Co and Ni respectively. The standard samples were also heat-sealed in FEP film.

Table 1 List of samples used in this study

Sample name	Group/Type	Mass (mg)
IAEA-SRM-Soil-7	Soil	107.7
NIST-SRM-1633b	Coal fly ash	134.4
Allende (meteorite)	CV3 (chondrite)	256.6
Tannery sediment	Sediment	202.0

B. Sample irradiation and counting:

The samples and standards were irradiated in the thermal neutron guided beams of the JRR-3M reactor facility [16, 17]. The neutron flux was $1.1 \times 10^8 \text{ n.cm}^{-2}.\text{s}^{-1}$ at the sample irradiation position [18]. The IAEA-SRM-Soil-7 and NIST-SRM-1633b were irradiated two times; the other samples were irradiated only one time. In all cases the beam cross-sectional size ($2.0 \text{ cm} \times 2.0 \text{ cm}$) was always greater than those of the samples. The samples were suspended in the beam using Teflon string, and were irradiated for durations ranging from 5000-8000 seconds. The samples were irradiated in a He gas environment in order to minimize attenuation of the beam intensity due to atmospheric scattering and to prevent production of prompt γ -rays from atmospheric N_2 . All samples were counted during the irradiation using a high-resolution HPGe γ -ray detector (30% relative efficiency) equipped with a Compton-suppression system and a pulse height analyzer system controlled by a personal computer. The Ge detector was located 24.5 cm away from the sample and with its axis perpendicular to the beam. After completion of the samples counting the background counts were measured.

Table 2 The prompt gamma-ray energies used in this study for PGA analyses of the listed elements

Element	Energy	γ -ray intensity (I_γ , %)
B	478	2.59
Na	871	21.92
Mg	2828	42.51
Al	1779*	100
Si	1273	15.93
S	841	75.55
Cl	1951	21.72
K	770	51.48
Ca	1942	72.55
Ti	1382	69.08
Cr	835	26.86
Mn	314	6.76
Fe	352	11.7
Co	277	19.94
Ni	465	12.98
Sm	333	98.9
Gd	1187	10.83

*Delayed γ -ray used for Al because I_γ is 100%, and its half-life is short (2.24 minute)

C. Data analysis:

The γ -rays spectrums were saved in the floppy disc and the software program SPEC anal analyzed the peak areas of the specified γ -rays. The prompt γ -rays used in this study are shown in Table 2. Only the delayed γ -ray 1779 keV was used for the determination of Al concentration in the sample. In this study the elemental concentrations in all samples were calculated using the comparison method. With the comparison method, an element (x) concentration is determined in an unknown sample by comparing the sample data with that for a standard sample of known concentration of that element (x). Assuming the neutron beam flux remains constant, the concentration of element (x) in the unknown (unk) and standard (std) samples is related by the following equation:

$$\frac{n_{unk}^x}{n_{std}^x} = \frac{A_{unk}^x}{A_{std}^x} \times \frac{t_{std}^{irrad}}{t_{unk}^{irrad}}$$

where n is the number of atoms, A is the photo-peak area, t is the irradiation time. One disadvantage of this method is the that it requires careful monitoring of the neutron beam flux. Yonezawa C et al. [18] reported that the cold neutron flux variation during one reactor operation cycle (26 days), which includes the uncertainty due to the counting geometry, to be 5.9%. Strictly speaking, it depends on the conditions of the reactor operation. In the background spectrum, a peak at 1979 keV was detected. The background counts (0.0839 cps) at 1779 keV peak were subtracted from the total counts of the samples. Therefore, the background corrected net cps in the sample was used for the Al concentration calculation.

IV. RESULTS AND DISCUSSION

A. Accuracy of the analytical results:

In order to evaluate the accuracy of PGA method the standard powder of stony meteorite Allende (CV3, chondrite: a chondrite is a kind of cosmic sedimentary rock formed in the original solar cloud or nebula of dust and gas from which our present solar system is formed) and NIST standard reference material NIST-SRM-1633b were analyzed. The analytical results are shown in Table 3 and Table 4. Figure 1 shows that the concentrations of multi-element (B, Mg, Al, Si, S, Cl, K, Ca, Ti, Cr, Mn, Fe, Co, Ni, Sm and Gd) determined in the Allende meteorite are generally in good agreement (<10% relative error) with those reported in the Literatures [19-23] and Latif SA [24]. The concentration of Cl determined in Allende meteorite is agreement ($\pm 20.9\%$) with the higher range of the literature value. The determined elemental concentrations for NIST-SRM-1633b [25] are also generally in good agreement (<10%

relative error) with the certified values in most cases (Table 4). The concentrations of Na determined in all samples using PGA are notable exceptions. The Doppler-broadened B 478 keV peak overlaps the peak of Na at 472 (γ -ray intensity I_γ , 60%) keV. Another peak of Na at 871 (I_γ , 22%) keV in the sample matrix is not good shape, it is also broadened, may be the oxygen peak at 871 (I_γ , 100%) keV overlaps it. Probably it is the cause of very high concentrations of Na determined in the sample using at 871 keV peak. Finally the concentrations of Na determined in all the samples using PGA is not included due to very notable exceptions. In this study it is shown that the accuracy of the analytical results for most of the investigated elements is very high. It reveals that PGA could be a good and reliable methods for analyzing the standard reference materials and other samples.

Table 3 Elemental concentrations for the Allende meteorite determined by PGA using thermal neutron irradiation

Element	Concentration	Lit. mean or range [†]	\pm RSD (%)
B ppm	1.14 \pm 0.22	1.1*	4.1
Mg %	13.0 \pm 1.1	14.8 \pm 0.1	12.1
Al %	1.64 \pm 0.15	1.74 \pm 0.04	5.7
Si %	16.45 \pm 0.97	16.0 \pm 0.1	2.8
S %	2.07 \pm 0.08	2.10 \pm 0.03	1.44
Cl ppm	387 \pm 38	218-320	20.9
K ppm	>507	330 \pm 87	
Ca %	1.82 \pm 0.10	1.84 \pm 0.05	0.94
Ti ppm	852 \pm 74	900 \pm 50	5.4
Cr ppm	3868 \pm 256	3640	6.3
Mn %	0.14 \pm 0.02	0.145 \pm 0.004	3.5
Fe %	24.4 \pm 0.5	23.6 \pm 0.1	3.5
Co ppm	742 \pm 46	662 \pm 5	12.1
Ni %	1.43 \pm 0.04	1.42 \pm 0.02	0.6
Sm ppm	0.32 \pm 0.07	0.31 \pm 0.02	1.96
Gd ppm	>0.563		

[†]Literature data [19-23], *Ref [24], RSD relative standard deviation

Table 4 Comparison of elemental concentrations for the NIST-SRM-1633b (coal fly ash) determined by PGA using thermal neutron irradiation with the certified values unless otherwise indicated. The number of replicate samples run is given in parentheses

Element	Concentration	Lit. value [†]	±RSD (%)
B ppm	73.2±0.9 (2)	-	-
Mg %	<3.11 (2)	0.482±0.008	
Al %	14.75±0.36 (2)	15.05±0.27	2
Si %	20.56±1.21 (2)	23.02±0.08	10.7
Cl ppm	<151 (2)	-	-
K %	1.83±0.05 (2)	1.95±0.03	6.3
Ca %	1.58±0.12 (2)	1.51±0.06	4.7
Ti %	0.754±0.016 (2)	0.791±0.014	4.7
Cr ppm	<1060 (2)	198.2±4.7	-
Mn ppm	<460 (2)	131.8±1.7	-
Fe %	7.71±0.17 (2)	7.78±0.23	1
Sm ppm	17.8±0.3 (2)	20 [‡]	11
Gd ppm	17.9±1.1 (2)	13 [‡]	37

[†]Literature value certified [25], [‡]Non-certified, RSD relative standard deviation

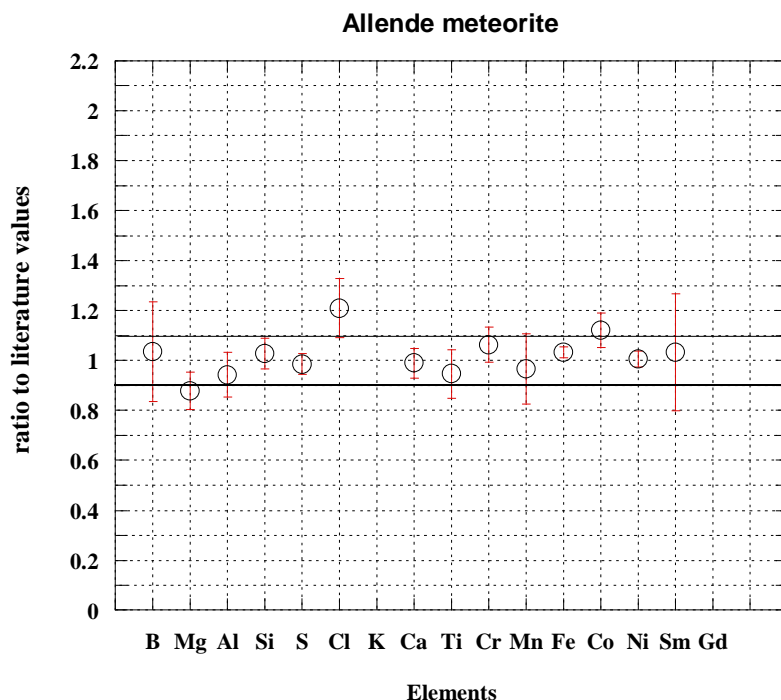


Fig. 1 Comparison of elemental concentrations determined in the Allende meteorite with literature values

B. Multi-element analysis in the tannery sediment of Bangladesh:

The concentrations of multi-elements (B, Al, Si, Cl, K, Ca, Ti, Cr, Mn, Fe, Sm and Gd) were determined in this study by PGA using thermal neutron beams. The analytical results are shown in Table 6. It is seen that the concentration of toxic element Cr is very high and is very consistent (within 5.2%) with the INAA value [26]. This study again reveals that the elevated concentration of Cr in this sample is due to anthropogenic

contamination. This study also shows that the higher levels of Cr can be easily determined in sediments by PGA using thermal neutron beams.

CONCLUSION

This study shows that multi-elements (B, Al, Si, Cl, K, Ca, Ti, Cr, Mn, Fe, Sm and Gd) in IAEA-SRM-Soil-7 can easily non-destructively determined by PGA with high accuracy. The literature values [8] for most of the elements mentioned above

of the IAEA-SRM-Soil-7 sample are non-certified except Cr, Mn and Sm. This study reports the concentration of the elements mentioned above for the certification especially for B, Si and Gd. The elements B and Si are determined non-destructively only by PGA. Silicon was determined with good accuracy by PGA. Therefore, PGA analysis of Si would seem to be good alternative to other methods (e.g., wet chemistry, X-ray fluorescence). This is especially true with SRM analyses, where the available sample mass is often small (<1g) and non-destructive analysis is preferred.

Since PGA is an online processes and multi-element character then it is easier-to-use effectively for determining the elevated levels of toxic element chromium as well as other elements in tannery sediments of Bangladesh. It should be mentioned here that lower concentration of Cr (<1000 ppm) in geological sample could not determine by PGA using thermal neutron beams. Probably using cold neutron beams PGA lower concentration (<1000 ppm) of chromium in geological samples can be measured. This study shows that PGA is a suitable method for the rapid identification of the environmental pollution problem due to higher levels of chromium arises from tannery industries as well as other sources of Bangladesh.

Table 6 Elemental concentrations for the tannery sediment of Bangladesh determined by PGA using thermal neutron irradiation

Element	Concentration
B ppm	26.6±0.5
Mg %	n.d
Al %	1.63±0.17
Si %	28.9±1.5
Cl ppm	4645±113
K %	0.75±0.04
Ca %	4.6±0.2
Ti ppm	1347±74
Cr %	1.45±0.04; (1.53±0.02) †
Mn ppm	n.d
Fe %	3.25±0.12
Sm ppm	2.64±0.13
Gd ppm	1.94±0.36

†INAA value [26], n.d not determined

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