

# URANIUM ISOTOPIC ANALYSIS OF DEPLETED URANIUM IN PRESENCE OF OTHER RADIOACTIVE MATERIALS BY USING COAXIAL AND PLANAR Ge DETECTORS

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## ABSTRACT

This paper deals with the determination of isotopic abundance of depleted uranium (DU) employing two different  $\gamma$ -ray spectrometric methods. One is the multi-group  $\gamma$ -ray analysis (MGA) method using the low energy region ( $< 300$  keV) with a planar Ge detector intrinsically calibrated with  $\gamma$ - and X-rays of uranium without use of standards. The MGA method is quicker and more practical for situ applications, however, it has been previously demonstrated by author [1] that relative abundances of DU obtained by MGA method are not consistent with the declared values for the DU samples in the presence of the actinides such as  $^{232}\text{Th}$ ,  $^{237}\text{Np}$ ,  $^{233}\text{Pa}$  and  $^{241}\text{Am}$ . The main reason for these erroneous results has already been proposed to be the interference of the  $\gamma$ - and X-rays of uranium with those emissions of the other actinides in the 80-130 keV region. The latter method is the absolute  $\gamma$ -ray analysis method (AGA), which uses the  $\gamma$ -rays of energies in the range of 130-1001 keV region for uranium but it requires an accurate absolute efficiency calibration with use of radionuclide standards. For the application of the AGA method, a high efficient coaxial Ge detector was calibrated by using the multi-radionuclide standards. It is emphasized that less intense but cleaner gamma peaks at 163.3 keV (5.08 %) and 205 keV (5.01%) of  $^{235}\text{U}$  are preferably used for the determination of  $^{235}\text{U}$  abundance in the AGA method, although there are more intense peaks of  $^{235}\text{U}$  observed in the  $\gamma$ -ray spectra such as 143.8 keV (10.76%) and 185.7 keV (57.2%) of  $^{235}\text{U}$ . Because both 143.8 keV and 185.7 keV peaks affected seriously due to interferences with 143.3 keV (0.44%) of  $^{237}\text{Np}$  and with 186.2 keV (3.51%) of  $^{226}\text{Ra}$ . Additionally, in the higher energy region (above 130 keV) of the spectra, it was chosen the analytical peaks: 258.2 keV (0.076%), 742.8 keV (0.106%), 766.3 keV (0.294%) and 1001 keV (0.837%)  $\gamma$ -rays of  $^{234\text{m}}\text{Pa}$  ( $^{238}\text{U}$ ) for the determination of  $^{238}\text{U}$ . However, the 1001 keV peak among them is preferably chosen for the isotopic abundance analysis of  $^{238}\text{U}$ . On the other hand, in the low energy region, the most intense peak of  $^{234}\text{Th}$  ( $^{238}\text{U}$ ), 63.3 keV (4.8%) has not been considered for the analysis because it has a fully multiplet (62.86 keV + 63.29 keV) and includes the interference peaks of the 62.70 keV (1.5%) of  $^{234}\text{Pa}$ , the 63.81 keV (0.263%) of  $^{232}\text{Th}$  and the 63.90 keV (0.011%) of  $^{237}\text{Np}$ , when the presence of other radioactive materials along with DU. It is clear that the AGA method needs more tedious and laborious work, however, it results in more accurate results for  $^{235}\text{U}$  isotopic analysis of DU samples. The results for the abundances measured from the AGA method are compared with those of MGA method when DU samples measured along with other radioactive materials.

## 1. INTRODUCTION

Recent developments in semiconductor detector technologies have opened new possibilities for measurement of  $\gamma$ -ray and X-ray radiations which are used to detect, locate and identify radiation sources and discriminate between items of commerce, naturally occurring radioactive material (NORM), technologically enhanced radioactive material (TENORM) and special nuclear material (SNM) such as  $^{235}\text{U}$ -enriched uranium or plutonium. Besides, the forensics applications employ frequently the nuclear analytical techniques such as X-ray fluorescence (XRF), proton-induced X-ray emission spectrometry (PIXES), alpha spectrometry (AS) and gamma

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ray spectrometry (GRS). Thus, the nuclear analytical techniques can provide additional evidence to clarify criminal cases, as a complementary tool to conventional investigation methods such as inorganic mass spectrometry [Inductively Coupled Plasma Mass Spectrometry (ICP-MS) or Laser ablation ICP-MS), Thermal Ionization Mass Spectrometry (TIMS), Accelerator Mass Spectrometry (AMS), Glow Discharge Mass Spectrometry (GDMS), Secondary-Ion Mass Spectrometry (SIMS)], X-ray Diffraction (XRD), Optical Microscopy (OM), Electron Microscopy (EM). These methods can analyze nature, use and origin of nuclear materials through determination of radioisotopes, isotopic and mass ratios, material age, impurity content, etc., which can be used to produce “nuclear fingerprint” of a suspect material. A well-characterized “nuclear fingerprint” enables the accurate tracing of minute quantities of radioactive and nuclear materials. Of these materials, depleted uranium (DU) is also used in many peaceful and military applications owing to its very high density ( $19.3 \text{ g cm}^{-3}$ ). For instance, it is well suited for offensive use in armour-piercing ammunition. DU-ammunitions were used in Kosovo by NATO during the Balkan conflict. Tanks and aircraft can fire the DU-ammunition with a calibre of 100-200 mm and 25-30 mm, respectively. The weight of the DU penetrators fired by aircraft is approximately 300g [2].

As it is known, DU material can be either (1) by-product of natural uranium (NU) enrichment process, called “clean DU”, or (2) the depleted uranium derived from reprocessing of spent nuclear fuel, called “dirty DU”. During the chemical reprocessing, the trans-uranium and trans-plutonium elements which are the most toxic elements, remain partially in the uranium fraction and they can be found both in the enriched uranium and in dirty DU because the “dirty DU” contains some radioactive products ( $^{237}\text{Np}$ ,  $^{233}\text{Pa}$ ,  $^{239+240}\text{Pu}$ ,  $^{241}\text{Am}$  and  $^{236}\text{U}$ ) and even some fission products.

Historically, mass spectrometers as non-nuclear method have widely been used to measure uranium isotopic abundances [3]. In nuclear applications, for example, during tracking of illicit traffic and seizing nuclear contraband materials, the GRS method is more preferential for determination of the isotopic abundance (e.g., enrichment or depletion degree of  $^{235}\text{U}$  atoms) within scope of internationally safeguards or security activities for increased homeland protection. Because GRS method is the most practical one among other nuclear methods, such as AS, delayed neutron counting and neutron activation analysis (NAA) followed by high resolution GRS. In this context, it is more practical one for situ applications is the MGA method because it does not require use of any calibration reference materials. Performance of the applicability of MGA method for depleted and natural and enriched uranium isotopic analysis has already been tested [4,5] and its major and minor deficiencies are described in detail [6]. Further, in the presence of some actinides ( $^{232}\text{Th}$ ,  $^{237}\text{Np}$ ,  $^{233}\text{Pa}$  and  $^{241}\text{Am}$ ), it has been demonstrated previously by author [1] that relative abundances of DU and NU obtained by MGA method using the  $\gamma$ - and X-rays of uranium in the 80-130 keV region are not consistent with the declared values. The main reason for these erroneous results has already been proposed to be the interference of the  $\gamma$ - and X-rays of uranium with those emissions of actinides in the 80-130 keV region, and it is suggested that it be used two detector mode (planar and coaxial HPGe) covering both low energy region (below 300 keV) and higher energy region of up to 1200 keV of the uranium  $\gamma$ -ray spectra [1]. Therefore, the objective of the present work is to determine uranium isotopic abundances in some DU samples by employing two different gamma-spectrometric approaches, called the AGA method for high energy region and MGA method for low energy region. For this aim, firstly, a p-type coaxial HPGe detector covering the energy region of up to 1200 keV was calibrated with the multi-nuclide source and then, the obtained gamma-ray spectra were analyzed for both “clean DU” and simulated “dirty DU” samples. Secondly, a planar Ge detector equipped with a special U-Pu Digital Analyzer were used for the same samples by MGA method. A sophisticated gamma spectroscopy software with MGA method has been tested for the determination of uranium isotopic abundance analysis from gamma-ray data collected in the low energy region. Finally, the results obtained by two different gamma spectrometric methods are compared with each other.

## 2. FUNDAMENTALS OF URANIUM ISOTOPIC ANALYSIS METHODS

First, the absolute (direct)  $\gamma$ -ray analysis (AGA) method for uranium that can be used the  $\gamma$ -rays of energies in the range of 130-1001 keV region, has been applied to determine uranium isotopic abundances. From the activities of  $^{235}\text{U}$  and  $^{238}\text{U}$  being measured with a Ge detector calibrated with multi-radionuclide standards. If the component,  $^{234}\text{U}$  isotope in the uranium,  $N^{24} = 0.0054\%$  is assumed to be neglected for simplicity due to

percentage of very low isotope content, the isotopic abundance of  $^{235}\text{U}$  isotope,  $h_a$  in uranium sample can be defined simply as [7]:

$$h_a = \frac{N^{25}}{N^{24} + N^{25} + N^{28}} \cong \frac{1}{1 + k \cdot \left( A_U^{28} / A_U^{25} \right)} \quad (1)$$

where  $N^{25}$  is the number of  $^{235}\text{U}$  atoms,  $N^{28}$  is the number of  $^{238}\text{U}$  atoms,  $k = \lambda^{25} / \lambda^{28} = 6.348394$  is the ratio of decay constants,  $A_U^{25}$  is the measured activity of  $^{235}\text{U}$ , and  $A_U^{28}$  is the measured activity of  $^{238}\text{U}$ . For the measurement of uranium activities, the photopeak efficiency of a  $\gamma$ -ray counting system with a HPGe detector as a function of photon energy is given by:

$$\varepsilon(E_i) = \frac{N_p^i / t_c}{P_i \cdot A_r \cdot D} \quad (2)$$

Where  $N_p^i$  is the net peak count for  $i$ -th peak of interest,  $P_i$  is the  $\gamma$ -ray emission probability at energy  $E_i$ ,  $A_r$  is the activity of radionuclide used, and  $t_c$  is the counting time (live time) and  $D$  is the decay correction factor,  $D = \exp(-0.693\lambda t_r)$  corrected for the true time,  $t_r$  which is elapsed time from the certificate date of the nuclide to the beginning of measurement.

The latter method is multigroup analysis (MGA) method for uranium (MGAU) that employs generally X- and  $\gamma$ -rays in the 80-130 keV region of a  $\gamma$ -ray spectrum taken from uranium without use of radionuclide standard sources or nuclear reference materials. The fundamentals of MGA methodology has not been explained here since the considerations on the descriptions of peak shapes, efficiencies, geometry, absorbing material effect and background subtraction of the MGA methodology is described in detail [8,9,10]. However, it can be expressed briefly that the MGA method for the determination of the isotopic ratio is to measure basically the peak intensity of two or more peaks from  $\gamma$ -rays of nearest energies but arising from different isotopes. Because the  $\gamma$ -ray emission probabilities and half-lives are known, the isotopic ratios of two different atoms can be calculated if relative detection efficiencies for the peaks of interest can be estimated. It is worth noting that in a sophisticated MGA code, relative detection efficiencies are determined intrinsically in the same  $\gamma$ -ray spectrum by an iterative procedure [11].

### 3. EXPERIMENTAL

In this study, a coaxial p-type high efficient (120.8%) HPGe detector, called setup 1 for AGA method, and a planar LEGe detector called setup 2 for MGA method were used in turn for uranium isotopic analysis. The HPGe detector with a 0.5 mm thick Al window was equipped with a 16 K ADC conversion gain/MCA channels digital signal analyzer(DSA-1000) supported by PC Genie-2000 gamma software. Its measured energy resolution was 1.95 keV at 1332.5 keV ( $^{60}\text{Co}$ ) The other technical specifications for this HPGe detector are given in Table 1.

Table 1. Planar LEGe detector configuration for for MGA method for uranium analysis

	Brand	Model Number	Active Diameter (mm)	Thickness (mm)	Active Surface (mm <sup>2</sup> )	Resolution @50 Kcps for a shaping time of 2μsec	
S E T U P  2	Canberra	GL0515R <sup>1)</sup>	25	15	500	Isotope	
						<sup>57</sup> Co	
						Energy (keV)	
						122	
						FWHM* (keV)	
0.597							
FWTM** (keV)							
1.092							
	Canberra	IN2K	U-Pu Inspector 2000 with a special U-Pu Software (MGA/MGAU Ver.2.1) supported by PC Genie Gamma Software. * FWHM: Full Width at Half Maximum, ** FWTM: Full Width at Tenth Maximum.				

<sup>1)</sup> In this model, the end cap window can be used with a filter consisting of three tin (Sn) discs, each is approximately 0.8 mm in thickness. These discs are all attached to an Al-cover with a pivot that allows to be individually rotated into place in front of the detector. It has also a multi-attitude type cryostat.

A planar LEGe detector with a 0.5 mm thick-Al window was chosen to have adequate resolution and good efficiency for low-energy  $\gamma$ -rays. The principal technical specifications for the planar LEGe detector are given in Table 2, The LEGe detector has a measured resolution of 0.597 keV at 122 keV. Its planar Ge crystal has an active diameter of 25 mm, active surface area of 500 mm<sup>2</sup> and crystal thickness of 15 mm. A digital MCA system was used as a single instrument, a portable U-Pu inspector-2000 that includes a high voltage power supply, a spectroscopy amplifier, a digital stabilizer having the capability both zero and gain stabilization, and a digital signal processor (DSP) with a 16 K ADC conversion gain/MCA channels for pulse height analysis. Each spectrum was collected in the live-time mode. Dead times varied typically between 0.1% and 2.5% for all measurements. The measurements with both the coaxial HPGe and planar LEGe detector were carried out at three different detector-sample distances of 0, 5,10 and 15 cm.. But, to avoid or minimize true coincidence summing effects existed in close geometry measurements, the measurements performed only at a sample-detector distance of d<sub>2</sub>=10 cm were reported here for the comparison of those results for both Ge detectors. The measurement periods were chosen different periods yielding better than 1% of counting statistics.

Table 2. A coaxial Ge detector configuration for AGA method for uranium analysis

	Brand	Model Number	Active Diameter (mm)	Thickness (mm)	Resolution and Efficiency		
					Isotope	<sup>57</sup> Co	<sup>60</sup> Co
S E T U P  1	Canberra	GC11021	82	85.5	Energy (keV)	122	1332
					FWHM* (keV)	1.08	1.95
					FWTM** (keV)	3.69	3.69
					Peak/Compton ratio	-	85.7:1
					Relative efficiency	-	120.8
	Canberra	DSA-1000	Digital signal analyzer (DSA-1000) supported by PC Genie Gamma Software. * FWHM: Full Width at Half Maximum, ** FWTM: Full Width at Tenth Maximum.				

The depleted uranium U<sub>3</sub>O<sub>8</sub> samples containing 0.441% <sup>235</sup>U isotope were measured in this study, which are filled as powder uranium in a plastic vials with 1 mm wall thickness. A set of five <sup>237</sup>Np foils (coded as JK, JL, JS, JX and GU in No. 23 foil set from IAEA) having a total activity of 120 kBq, a 37 kBq <sup>241</sup>Am-gamma point source, ThO<sub>2</sub> (obtained from Merck) having a total activity of 44.1 kBq, and Th(NO<sub>3</sub>)<sub>4</sub> (obtained from the Radiochemical Centre, Amersham) having a total activity of 33.3 kBq were used for test measurements in this study.

The certified multinuclide standard sources containing <sup>241</sup>Am, <sup>109</sup>Cd, <sup>57</sup>Co, <sup>123m</sup>Te, <sup>51</sup>Cr, <sup>113</sup>Sn, <sup>137</sup>Cs, <sup>88</sup>Y and <sup>60</sup>Co radionuclides spiked in a sand matrix were obtained by Isotope Products Inc., traceable to NIST. The heights of the used uranium samples were same with those calibration source heights, which are d<sub>1</sub>=0.5 cm and 2.5 cm as is seen in Fig.1.

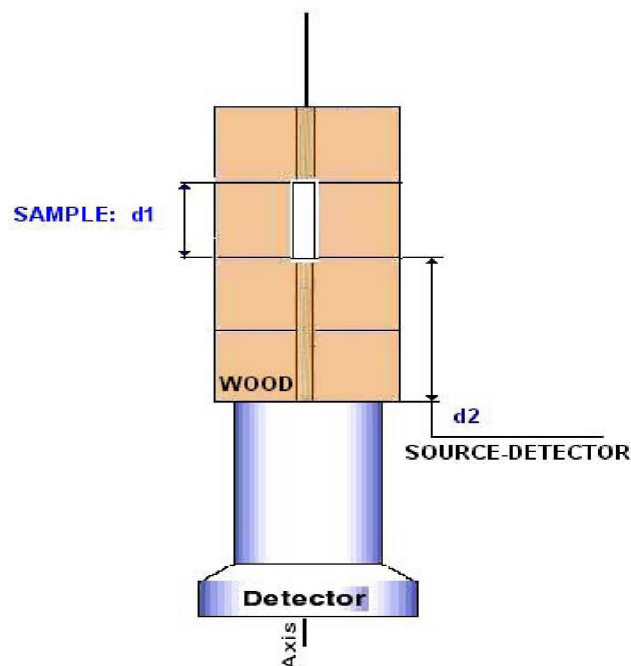


Fig.1. Sample-detector configuration  
 ( $d_1$ :sample height,  $d_2$ : source-to-detector distance)

The individual uranium samples to be measured was placed at a particular distance far from endcap of the detector by fixing a sample holder made of wood. The  $\gamma$ -ray spectra are automatically acquired and stored with the help of a  $\gamma$ -ray spectroscopy program (Canberra Genie 2000). The stored  $\gamma$ -ray spectra can also later be analyzed with MGA for uranium software supported by Genie-2000. The uranium samples were measured three times in the detectors for each counting period predetermined.

#### 4. RESULTS AND DISCUSSION

In this study, a coaxial HPGe detector described as Setup1 was calibrated at source-to detector distance of 10 cm using the mixed-standard sources containing  $^{241}\text{Am}$ ,  $^{109}\text{Cd}$ ,  $^{57}\text{Co}$ ,  $^{123\text{m}}\text{Te}$ ,  $^{51}\text{Cr}$ ,  $^{113}\text{Sn}$ ,  $^{137}\text{Cs}$ ,  $^{88}\text{Y}$  and  $^{60}\text{Co}$  radionuclides. As seen in Fig. 1, the source heights of the used sources in the vials,  $d_1$  were chosen to be 0.5 cm and 2.5 cm, respectively. Then, the efficiency calibrations for the present HPGe detector-source geometry shown in Fig. 1 were carried out for two different heights of the vials. An example of the full energy peak efficiency curve obtained for the sample measuring geometry (Setup1), shown in Fig.2, which indicates the energy range of up to 1200 keV and the experimental points for the used nuclides, marked with the experimental uncertainties. The experimental efficiencies were fitted to the logarithmic functions. In general, the fitted curve is satisfactorily accurate of better than 2-2.5% on the whole energy range.

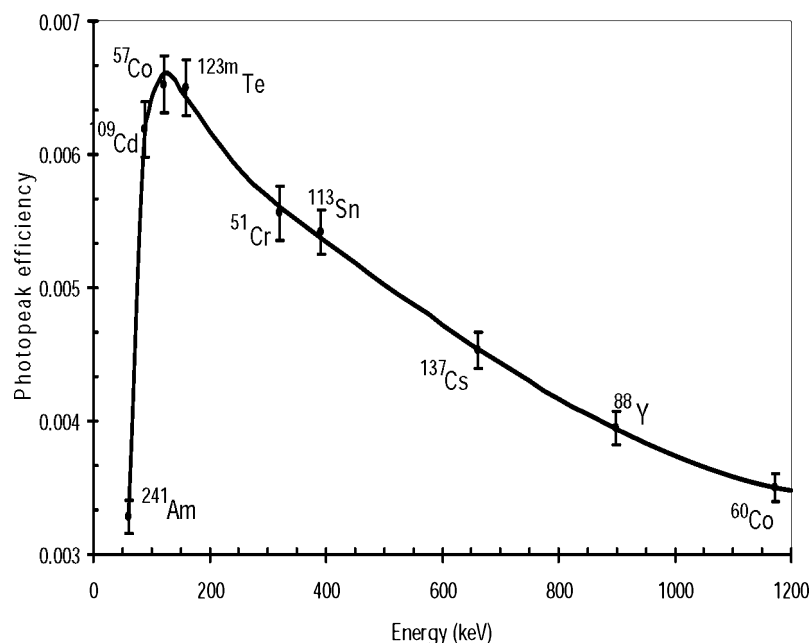


Fig. 2. Full energy peak efficiency for in the energy range of 40 to 1200 keV for the setup 1 measuring geometry

After efficiency calibration of the HPGe detector for setup1, the individual depleted uranium samples prepared from depleted  $U_3O_8$  powder, which are same heights in the vials were measured with a coaxial HPGe detector. Then, other radioactive materials above mentioned are placed close to the samples and the measurements were repeated. The obtained gamma-ray spectra were analyzed by a gamma spectroscopy software (Canberra Genie 2000) using with interactive peak fitting module in order to deconvolute the interference peaks such as (766+768 keV) from the daughters of  $^{238}U$ . To avoid the possible interference peaks in the low-energy region (below 300 keV), less intense but cleaner gamma peaks at 163.3 keV (5.08 %) and 205 keV (5.01%) of  $^{235}U$  in the same spectrum are preferably used for the determination of  $^{235}U$  although there are more intense peaks of  $^{235}U$  in the observed spectra such as 143.8 keV (10.76%) and 185.7 keV (57.2%). Because both 143.8 keV and 185.7 keV peaks affected somehow due to the interferences with 143.3 keV (0.44%) of  $^{237}Np$  and with 186.2 keV (3.51%) of  $^{226}Ra$ . Similarly, the 258.2 keV (0.076%), the 742.8 keV (0.106%), the 766.3 keV (0.294%) and the 1001.03 keV (0.837%) peaks from  $^{234m}Pa$  daughter of  $^{238}U$  in the high energy region (up to 1200 keV) is mainly preferred to determine  $^{238}U$  isotopic abundances in DU samples.

Additionally it is emphasized for the AGA method that the 1001 keV peak is preferably chosen for the isotopic abundance analysis of  $^{238}U$ . On the other hand, in the low energy region, the more strong 63.3 keV (4.8%) peak of  $^{234}Th$  which is daughter of  $^{238}U$ , should not be considered to be a good analytical peak since it has a fully multiplet (62.86 keV + 63.29 keV) and includes also the interferences of the 62.70 keV (1.5%) peak of  $^{234}Pa$ , the 63.81 keV (0.263%) peak of  $^{232}Th$  and the 63.90 keV (0.011%) peak of  $^{237}Np$ , and it is very close to 59.6 keV peak ( $^{241}Am$ ) in the presence of other radioactive materials along with DU.

From setup 2 with a LEGe detector, a set of measurement results for determination relative abundances of uranium isotopes for different counting times of 1 to 24 h were also obtained by MGA method. The results for the individual measurements of depleted uranium samples with and without other radioactive materials are given in Table 3. The measurement uncertainties are based on  $\pm 1\sigma$  confidence interval. The estimated uncertainties are expressed as percentages associated with the measured results in Table 3.

Table 3. Abundance results for  $^{235}\text{U}$  in DU sample with and without other radioactive materials

			AGA Method		MGA Method	
			HPGe Detector		LEGe Detector	
Sample	Sample height $d_1$ (cm)	Sample to detector distance $d_2$ (cm)	Measured* $^{235}\text{U}$ %	% Diff.**	Measured* $^{235}\text{U}$ %	% Diff.**
$\text{U}_3\text{O}_8$	0.5	10	$0.439\pm 2.3$	0.3	$0.458\pm 1.0$	4.1
$\text{U}_3\text{O}_8$	2.5	10	$0.482\pm 2.5$	9.6	$0.430\pm 2.8$	2.3
$\text{U}_3\text{O}_8 + ^{241}\text{Am} + (^{237}\text{Np}/^{233}\text{Pa})$	2.5	10	$0.495\pm 2.2$	12.5	Not determined	-
$\text{U}_3\text{O}_8 + ^{241}\text{Am} + (^{237}\text{Np}/^{233}\text{Pa}) + \text{Th}(\text{NO}_3)_4$	2.5	10	$0.482\pm 4.6$	9.6	$17.775\pm 0.6$	$\approx 1700$

\*: The uncertainty (%) associated with the measured abundance is given as the plus and minus percentage following the measured value.

\*\* : %Difference =  $100 \times (\text{measured} - \text{declared}) / \text{declared}$ , and it means mean difference percentage from three measurements.

The AGA method for isotopic analysis of uranium provided more accurate results for the abundance of  $^{235}\text{U}$  in DU with the percentage difference of 0.3-9.6% from its declared value and their precisions are also high, i.e. below 2.5%. It is worth noting that the observed results for  $^{235}\text{U}$  abundance in Table 3 are still meaningful even if other radioactive materials are counted along with DU.

In contrary to the AGA method, the MGA method provides a relative abundance of  $^{235}\text{U}$  for DU estimates that are in statistical control with about a  $\pm 5\%$  uncertainty budget. However, when presence of other radioactive materials, this method does either not provide any measured result for  $^{235}\text{U}$  abundance or results in an incorrect value, which is higher 1700% of its declared value. That is, MGA method does not work at all for case of the presence of additional radioactive isotopes. As it has been previously demonstrated by author [1] that relative abundances of DU obtained by MGA method that uses the very complex  $\gamma$ - and X-rays of uranium in the 80-130 keV region are not consistent with the declared values for the DU samples in the presence of other radioactive materials, especially the actinides such as  $^{232}\text{Th}$ ,  $^{237}\text{Np}$ ,  $^{233}\text{Pa}$  and  $^{241}\text{Am}$ . The main reason for these erroneous results has already been proposed to be the interference of the  $\gamma$ - and X-rays of uranium with those emissions of actinides in the 80-130 keV region [1].

## 5. CONCLUSIONS

When the results for the abundances of  $^{235}\text{U}$  determined from the AGA method are compared with those of MGA method in presence of other radioactive materials along with DU, the AGA method is a more reliable method for uranium isotopic analysis. The AGA method employs also non-destructive  $\gamma$ -ray measurements but in this work they were performed using a high efficient coaxial Ge detector, thus giving higher precisions. Since the detector was calibrated with use of a set of radioactive standards, the AGA method needs more tedious and laborious work, however, it provided accurate results for the isotopic analysis of DU samples. It is suggested that some additional test measurements be also performed using filters such as tin interposed between the source sample and the front of detector window in order to improve the measured abundance values in observing the effect of filter on the peak to Compton continuum ratio due to scattered radiation in absorber material. However, the interference peaks needs to take into account in the analysis after treatment of delicately de-convolution of the

peaks by non-linear least squares method, especially in the low energy region. Accordingly, when the presence of other additional radioactive isotopes in uranium samples such as “dirty DU”, uranium isotopic analysis is still problematic and nondestructive methods have still some major deficiencies.

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