



²³⁵U isotopic characterization of natural and enriched uranium materials by using multigroup analysis (MGA) method at a defined geometry using different absorbers and collimators

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Abstract. Characterization of nuclear materials is an important topic within the context of nuclear safeguards, homeland security and nuclear forensics. This paper deals with the performance of multigroup gamma-ray analysis (MGA) method using the X- and γ -rays in the 80–130 keV region and enrichment meter principle (EMP) based on the analysis of 185.7 keV peak for a certain geometry using different absorbers and collimators. The results from MGA and those of EMP are compared. In particular, the effect of aluminum/lead absorbers and lead collimator on the enrichment determination of ²³⁵U in natural and low enriched samples is investigated in a given source-detector geometry. The optimum diameter/height ratio for the Pb-collimator is found to be $D_c/H_c = 1.4$ – 1.6 in the chosen geometry. In order to simulate the container walls, ten different thicknesses of Al-absorbers of 141 to 840 mg·cm⁻² and six different thicknesses of Pb-absorbers of 1120–7367 mg·cm⁻² are interposed between sample and detector. The calibration coefficients (% enrichment/cps) are calculated for each geometry. The comparison of the MGA and EMP methods shows that the enrichment meter principle provides more accurate and precise results for ²³⁵U abundance than those of MGA method at the chosen geometrical conditions. The present results suggest that a two-step procedure should be used in analyses of uranium enrichment. Firstly MGA method can be applied *in situ* and then EMP method can be used at a defined geometry in laboratory.

Key words: nuclear safeguards • enrichment measurements • uranium • multigroup analysis • enrichment meter principle

Introduction

Recent developments in semiconductor detector technologies have opened new possibilities for the measurement of γ -ray and X-ray radiations, which are used to detect, locate, and identify radiation sources [1]. The safeguards and forensics applications employ frequently the nuclear analytical techniques such as X-ray fluorescence, proton-induced X-ray emission spectrometry, neutron radiography, alpha spectrometry, and gamma-ray spectrometry (GRS). These techniques may be used to identify the nature, use, and origin of nuclear materials through the determination of radioisotopes, isotopic and mass ratios, material age, impurity content, etc., which can be used to produce ‘nuclear fingerprint’ of a suspect material [2].

Nowadays, in many nuclear applications, the main aim is to get the accurate information in a short period without destructing the material. This requires a nondestructive measurement technique such as GRS. It has become more preferential for the determination of the isotopic abundance (e.g., enrichment or depletion degree of ²³⁵U atoms) within the context of international safeguards and security activities for increased homeland protection due to its ease of use, portability, noninvasive nature and speed [3, 4]. It also allows analysts or first respond-

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ers on site to measure and identify the radiation sources and materials. Measurement of uranium isotopic abundances with GRS can be done using different approaches [5]. The so-called enrichment meter principle is based on 185.7 keV peak of ^{235}U [6]. The multigroup gamma-ray analysis (MGA) method is based on the X- and γ -ray peaks in the 80–130 keV region [7]. A third approach is based on the measurement of γ -ray peaks in the energy region of 144–1001 keV [4, 8]. Therefore, the determination of relative abundances of uranium isotopes can be performed by using either a suitable γ -ray detector calibrated with the use of a set of radionuclide standards or an intrinsically calibrated low energy high resolution Ge detector [9, 10]. MGA method was first formulated and established by Gunnink *et al.* [11] with *in situ* applications in mind. In fact, MGA methodology can easily be applied not only to a nondestructive analysis of fissile ^{235}U isotope but also to characterize plutonium isotopes in the samples. The performance testing of MGA method for uranium was demonstrated by several works [12–14] for plutonium and uranium (depleted, natural, or enriched) having different physical and chemical forms and isotopic conditions. It was shown that MGA provides very rapid assay results when a suitable planar/coaxial Ge or a room temperature CdZnTe semiconductor detector is used. Although the adequacy of MGA method for uranium is confirmed, the validation tests show also a number of major and minor deficiencies of the method, which are described in detail [8]. MGA method has found widespread application in numerous fields such as nuclear safeguards, export/import material control, technological process measurements, waste characterization, tracking of nuclear materials issued in illicit trafficking, and homeland security activities against terrorist threats and attacks [15].

Performance of the applicability of MGA method for depleted, natural, and enriched uranium isotopic analysis has already been tested [7, 8]. Additionally, it has been demonstrated previously by Yücel [16] that uranium abundances in depleted and natural uranium obtained by MGA method in the presence of some actinides are not consistent with the declared values. The reason for these discrepancies in the measured results is proposed to be the interference of the γ - and X-rays of uranium with the X- and γ -ray emissions from actinides in the 80–130 keV region. Another reason is the existence of nearby objects containing radioactive and nuclear material. In some cases a series of closely spaced samples must be analyzed separately for proper characterization or the enriched material distribution in a large sample may be needed. The interferences can be eliminated by using collimators around the detector and/or by using shielding materials in front of these surrounding objects.

The isotopic abundance is investigated with two approaches in this study, namely; MGA method and EMP method. Of these two methods, the enrichment meter principle (EMP) requires the sample to have quasi-infinite thickness and a defined solid angle. A collimator is needed to define this solid angle. We

determined the optimum dimensions for such a collimator used with a Ge detector to measure uranium isotopic abundances and applied MGA and EMP methods to certified reference nuclear materials (EC-NRM 171) and other certified natural uranium bearing ore materials (CRMs).

Measurements were done with a series of absorber thicknesses that may be used to mask other nuclear or radioactive materials around the sample or a part of the sample or may be used as a nuclear material container. Finally, the results obtained with the two methods are compared.

Fundamentals of uranium isotopic analysis methods

For the determination of ^{235}U isotopic abundance, the isotopic abundance of ^{235}U in the sample, h_a can be formulated as $h_a = \frac{^{25}\text{N}}{^{24}\text{N} + ^{25}\text{N} + ^{28}\text{N}}$, where ^{25}N is the number of ^{235}U atoms and ^{28}N is the number of ^{238}U atoms [17]. However, for simplicity, the percentage of ^{234}U atoms can be ignored in this definition since $^{24}\text{N} = 0.0054\%$ is very low. ^{235}U isotopic abundance can then be determined from the measured activities of ^{235}U and ^{238}U . The ratio of decay constants is $k = \lambda^{25}/\lambda^{28} = 6.348394$.

The descriptions of peak shapes, efficiencies, geometry, absorbing material effect and background subtraction considerations of the MGA methodology are described in detail in several different works [12–14]. MGA method uses X- and γ -rays in the 80–130 keV region of a γ -ray spectrum of uranium sample. The determination of the uranium isotopic ratio is to measure basically the intensity of two or more peaks from γ -rays of very close energies arising from different isotopes. Since 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 [15].

The EMP may also be used to determine the ^{235}U enrichment in a uranium sample. This method relies on the assumption that the enrichment ratio is linearly proportional to the count rate of only 185.7 keV peak of ^{235}U . At least two reference materials with sufficiently different certified enrichment values are used to determine the proportionality constant. The enrichment of an unknown sample may then be analyzed by interpolation or extrapolation. There are some important assumptions for the application of this method. First of all, the calibration standards and the sample is assumed to have quasi-infinite thickness so the self-absorption does not play a role on count rates. This corresponds to a minimum thickness of 2.67 cm for UO_2 and 2.72 cm for U_3O_8 with bulk density $2.0 \text{ g}\cdot\text{cm}^{-3}$. Another factor is the container material and thickness. The calibration standards and the sample should ideally be in containers of same material and have same thickness; otherwise a correction should be applied. Also one should be certain that there is no significant amount of ^{226}Ra in the sample since this radionuclide also emits γ -ray (exactly, 186.211 keV), which is very close to 185.7 keV

Table 1. The specifications for the detector and data acquisition system

Brand	Model	Active diameter [mm]	Thickness [mm]	Active surface [mm ²]	Absorbing layers	Resolution		
						Time constant amplifier (shaping time)	6 μs @ Input count rate = 1 kcps	2 μs @ Input count rate = 30 kcps
ORTEC	SGD-GEM-3615P4 p-type, coaxial	37.7	16.4	1000 nominal	1.27 mmAl, 0.7 mm inactive Ge	⁵⁷ Co [keV]	122	122
						FWHM [keV]	0.522	0.602
						⁶⁰ Co [keV]	1332.5	1332.5
						FWHM [keV]	1.60	1.87
						FWTM/FWHM at 1332.5 keV	1.80	–

Ortec Uranium Analysis Software U235View supported by MAESTRO/Gamma Vision Gamma Acquisition software. Canberra U-Pu Software (MGA/MGAU Ver. 2.1) supported by PC Genie-2000 Gamma Acquisition Software.

peak of ²³⁵U, thus leading to an overestimation of the enrichment value.

Experimental

In this study, the p-type coaxial high purity Ge detector operating through a digital signal analyzer (Ortec DSPEC-Jr.2.0 supported by Gamma Vision Software from Ortec) is used. The technical specifications for the detector and its data acquisition system are given in Table 1. The resolution of the detector is 0.522 keV at 122 keV (⁵⁷Co) at 1 kcps (kilocounts per second). The Al end-cap thickness is 1.27 mm. Its Ge crystal has a nominal active surface area of 1000 mm² with a diameter of 37.7 mm and crystal thickness of 16.4 mm. Before all measurements, the detector is in energy and efficiency calibrated using a multinuclide gamma calibration source containing ²⁴¹Am, ¹⁰⁹Cd, ⁵⁷Co, ¹³³Ba radionuclides for which the energy range covers up to about 310 keV. Each spectrum is collected in the lifetime mode. Digital multichannel analyzer (MCA) system is a single instrument that includes a full 16K-channel MCA memory/conversion gain for pulse height analysis, a spectroscopy grade amplifier, a digital stabilizer having the capability both zero and gain stabilization, and a digital signal processor. A positive detector-interface module is used to provide a high voltage bias. Two different software for the application of MGA method were used in this work. One is MGAU program (purchased from Canberra) and the other

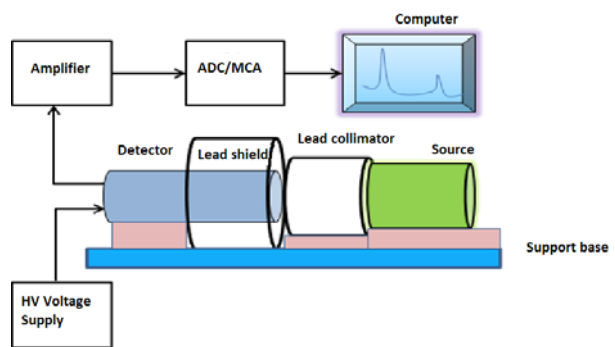


Fig. 1. Experimental setup used in uranium measurements.

is U235View program (purchased from Ortec) as described in Table 1.

The experimental setup used in the measurements is illustrated in Fig. 1. Sample to detector distance is set to 7.5 cm. Dead times varied between 0.25 and 3.85% for all measurements.

A set of certified nuclear reference materials (NRMs) purchased from EC JRC-IRMM (Belgium) is used to calibrate the detector for enrichment meter method. The γ-ray spectra of the NRMs and CRMs obtained from CANMET (Canada) were the performance testing of the method in a given source-detector geometry with use of different absorbers and collimator conditions. This set is composed of U₃O₈ powder encapsulated hermetically in aluminum housing at different isotopic enrichments. The properties of the sources are given in Table 2.

Table 2. Isotopic contents of uranium certified reference materials and standard nuclear materials

Reference sample ID ^{1,2,3}	Uranium amount [wt%]	Isotope content [atom%]		Isotope content [mass%]	
		²³⁵ U/U	²³⁸ U/U	²³⁵ U/U	²³⁸ U/U
		CRM BL-5	7.1 ± 0.1	0.7204 ± 0.0006	99.2742 ± 0.0010
CRM CUP-2	75.4 ± 0.2	0.7204 ± 0.0006	99.2742 ± 0.0010	0.7114 ± 0.0006	99.2650 ± 0.0010
NRM171-071	84.5 ± 0.3	0.7209 ± 0.0005	99.2738 ± 0.0002	0.7119 ± 0.0005	99.2828 ± 0.0002
NRM171-194	84.5 ± 0.3	1.9664 ± 0.0014	98.0159 ± 0.0009	1.9420 ± 0.0014	98.0406 ± 0.0009
NRM171-295	84.5 ± 0.3	2.9857 ± 0.0021	96.9826 ± 0.0015	2.9492 ± 0.0021	97.0196 ± 0.0015
NRM171-446	84.5 ± 0.3	4.5168 ± 0.0032	95.4398 ± 0.0016	4.4623 ± 0.0032	95.4950 ± 0.0016

¹ Canadian certified reference materials (CRMs) were obtained from CANMET-Mining and Mineral Sciences, Canada.

² A set of standard reference nuclear materials (NRM 171) was obtained from EC JRC-IRMM, Belgium. The abundances ²³⁴U/U and ²³⁶U/U in NRM 171 are not given in this table since they are out of the scope of this study.

³ These quoted uncertainties in CRMs are given within ± 1 s but the NRMs within ± 1.96 s.

Table 3. The calibration constants for enrichment meter method for different collimator diameters and absorber thicknesses

Al absorber thickness [mm]	Calibration constant [% enrichment/cps] for 185.7 keV peak Collimator hole diameter, D_c				
	15 mm	20 mm	25 mm	30 mm	50 mm
0	0.5230 ± 0.0322	0.2742 ± 0.0118	0.1656 ± 0.0054	0.1195 ± 0.0033	0.0445 ± 0.0008
0.522	0.5587 ± 0.0367	0.2703 ± 0.0114	0.1687 ± 0.0056	0.1224 ± 0.0034	0.0446 ± 0.0008
0.630	0.5321 ± 0.0340	0.2764 ± 0.0120	0.1704 ± 0.0057	0.1221 ± 0.0034	0.0447 ± 0.0008
0.800	0.5307 ± 0.0333	0.2799 ± 0.0122	0.1725 ± 0.0058	0.1229 ± 0.0035	0.0447 ± 0.0008
0.956	0.5496 ± 0.0350	0.2848 ± 0.0126	0.1735 ± 0.0054	0.1232 ± 0.0035	0.0454 ± 0.0008
1.215	0.5411 ± 0.0340	0.2873 ± 0.0128	0.1752 ± 0.0060	0.1224 ± 0.0034	0.0463 ± 0.0008
1.574	0.5572 ± 0.0358	0.2926 ± 0.0131	0.1762 ± 0.0060	0.1241 ± 0.0035	0.0460 ± 0.0008
1.933	0.5607 ± 0.0364	0.2913 ± 0.0129	0.1801 ± 0.0062	0.1275 ± 0.0037	0.0469 ± 0.0008
2.389	0.5777 ± 0.0386	0.2946 ± 0.0132	0.1819 ± 0.0042	0.1280 ± 0.0037	0.0471 ± 0.0008
2.426	0.5787 ± 0.0380	0.2947 ± 0.0132	0.1814 ± 0.0064	0.1296 ± 0.0037	0.0473 ± 0.0008
3.111	0.5836 ± 0.0385	0.3007 ± 0.0136	0.1868 ± 0.0066	0.1333 ± 0.0039	0.0487 ± 0.0009

Table 4. ^{235}U enrichment values of reference material EC-NRM171-071 with enrichment reference value (0.7209 ± 0.0002)% ^{235}U for collimator with 15 mm hole diameter

Al absorber thickness [mm]	^{235}U enrichment meter method [%]	^{235}U View (MGA software by Ortec) [%]	MGAU (MGA software by Canberra) [%]
0	0.7092 ± 0.0135	0.6760 ± 0.0951	0.6890 ± 0.0347
0.522	0.7107 ± 0.0136	0.7150 ± 0.0954	0.7260 ± 0.0364
0.630	0.7163 ± 0.0137	0.6800 ± 0.0981	0.7190 ± 0.0362
0.800	0.7109 ± 0.0136	0.7450 ± 0.0846	0.7480 ± 0.0346
0.956	0.7040 ± 0.0137	0.7350 ± 0.0962	0.7780 ± 0.0348
1.215	0.7122 ± 0.0137	0.7160 ± 0.0932	0.7460 ± 0.0361
1.574	0.7085 ± 0.0139	0.7110 ± 0.0995	0.7370 ± 0.0367
1.933	0.7058 ± 0.0140	0.7080 ± 0.1003	0.7490 ± 0.0371
2.426	0.7194 ± 0.0143	0.7150 ± 0.0996	0.7070 ± 0.0388
3.111	0.6907 ± 0.0139	0.7330 ± 0.1001	0.7510 ± 0.0379

Results and discussion

The γ -ray spectra of CRM and NRM uranium samples were analyzed with interactive peak fitting module to deconvolute the interference peaks in the same spectrum. The calibration constants for the EMP based on 185.7 keV peak count rates are calculated for different collimator diameters and for different absorber thicknesses. As an example, the measured results of ^{235}U for the natural uranium sample (NRM171-071) are given in Table 3 and Fig. 2.

As shown in Table 3 and Fig. 2, the calibration constants based on 185.7 keV peak differed at each counting geometry. Similarly, measurement periods also influenced counting statistics. Results for relative abundances of uranium isotopes for counting periods of 1800, 3600, 7200 and 60 000 s were obtained from individual measurements of natural and low enriched uranium samples and count rates between 0.2 to 9 cps were observed. As an example, the mean results of ^{235}U abundance for the natural uranium sample of the reference source set for the collimator with 15 mm hole diameter are given in Table 4. The coverage factor for the measurement uncertainties is 2. Graphical representation of the enrichment level for different absorber thicknesses for EC-NRM171-071 is also shown in Fig. 3.

The enrichment ratio is not significantly affected by the aluminum absorber thickness up to 0.5 cm.

However, the counting statistics definitely worsened with increasing absorber thickness. This leads to the conclusion that one needs to increase the counting time of a sample in a relatively thick container to get a reliable enrichment value with an acceptable uncertainty which in turn poses a problem for *in situ* enrichment determination. As a consequence, such samples with thick containers may be required to be moved into the laboratory or taken out of the container for a proper analysis.

Hole diameter-height (D_c/H_c) ratio of a collimator is also important since the collimator is used to restrict the solid angle to a small area of the sample which ensures the requirements for the proper application of enrichment meter method are met. One of these requirements is the source to be quasi-infinite both in thickness and surface area. When performing measurements on a large sample one may need to know the enrichment ratio at a particular position or a series of samples close to one another may be needed to be measured. Another conclusion that can be drawn from the results is that there is an optimum condition on the geometrical properties of the collimator. The collimator height is set to 2.5 cm in our experiments. The hole diameters are chosen as 1.5, 2.0, 2.5, 3.0, 5.0 cm. Measurement results show that the hole diameter-height ratio of the collimator should ideally be around 1.5. There is a compromise between the counting statistics

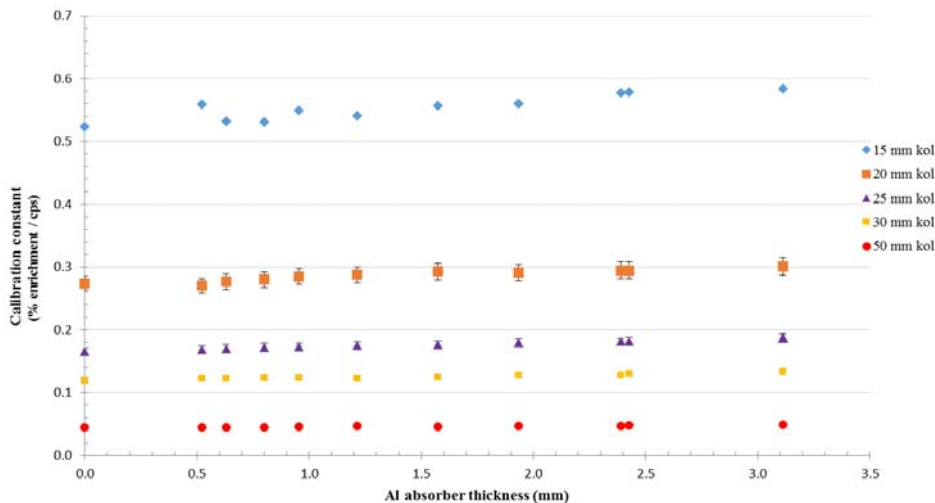


Fig. 2. Calibration constants for enrichment meter method for different absorber thicknesses.

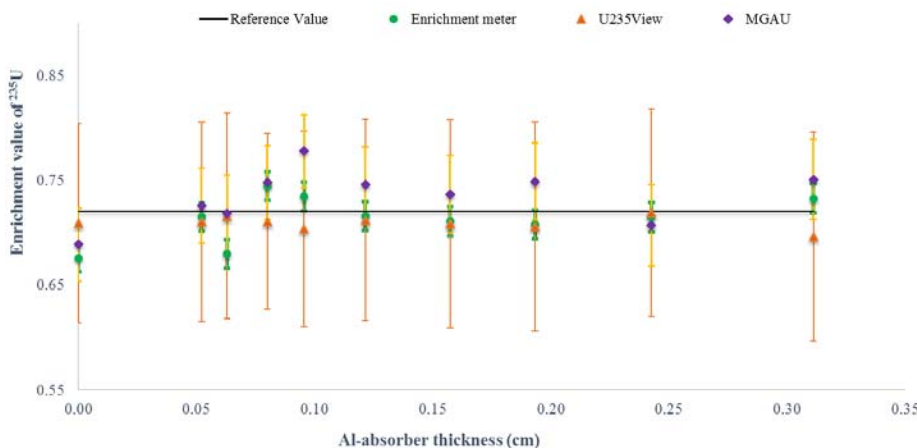


Fig. 3. Enrichment value of ²³⁵U for the natural uranium reference material EC-NRM171-071 with different methods and absorber thicknesses.

which gets better with increasing hole diameters and quasi-infinite source assumption which holds true for smaller internal diameters. The optimum ratio gives sufficiently good counting rates thus lowering uncertainties while keeping the quasi-infiniteness assumption as close to reality as possible.

Finally, one may claim that the enrichment meter method gives better results with lower uncertainties than the MGA method. This is easily seen from Fig. 3. However the need to calibrate the detector with reference sources having similar geometry and material composition to the sample is a major drawback. Another disadvantage is the time required for a proper analysis. Repeated and numerous measurements made in this study show that at least 2-h counting period is needed for the enrichment meter period for acceptable accuracy and uncertainty.

Conclusions

Portable MCA system can be equipped with a planar Ge detector or a p-type coaxial Ge detector. Such systems can give the analysts more accurate results from the lower part of a γ -ray spectrum (below 300 keV) using MGA methodology for determin-

ing U or Pu isotopic abundances. With the MGA method, it is difficult to accomplish better than 12% uncertainty in natural uranium samples even for relatively long measurement periods which are too lengthy for safeguards verification (about 30 to 120 min decision time). Despite major and minor deficiencies or limitations of the MGA method, it can be still improved for more accurate natural uranium analysis taking into account some aspects related to the analytical peaks used in MGA methodology. Similar measurements may also be done with the enrichment meter method based on 185.7 keV peak as long as certain geometrical conditions are met. The results indicate that the enrichment meter method gives better results with lower uncertainties than the MGA method.

As a conclusion, we suggest that a two-step procedure in the analysis of uranium enrichment should be used. First, MGA method is applied *in situ*. Second, in cases where there is a suspicion or a detailed analysis is needed it may be necessary to take samples to the laboratory and apply enrichment meter method for a more decisive result. More detailed and cumbersome methods such as ICP-MS are also an option if the analysis laboratory is equipped with such equipment.

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