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ANALYSIS OF INTERFERENCES FROM FULL ENERGY PEAKS IN GAMMA SPECTROMETRY OF NORM AND TENORM SAMPLES

by

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A considerable number of primordial radioisotopes are present in almost all the samples extracted from the Earth's crust, such as oil, rock, soil or other materials. Their concentrations are often determined by gamma spectrometry. Although the relative concentrations of isotopes often fluctuate within a narrow range, it is not always the case. Some natural materials (such as naturally occurring radioactive material) show unusual activity ratio between ²³⁸U and ²³²Th, while technologically processed materials (technologically enhanced naturally occurring radioactive material) might also introduce significant disequilibrium in radioactive chains. Knowing that primordial radioisotopes emit in total more than a thousand gamma and characteristic X-ray photons and that many of them interfere with each other, a question arises whether for some activity ratios commonly used photopeaks become useless for quantitative analysis, due to interferences with other photopeaks. A computer program was developed in order to calculate full energy photon interferences for any chosen photopeak. The calculations are based on the inputs in the form of isotope activities and detector calibration equations and its characteristics are presented in this paper.

Key words: gamma spectrometry, interference, NORM, TENORM, radioactivity

INTRODUCTION

NORM and TENORM are abbreviations for naturally occurring radioactive material and technologically enhanced naturally occurring radioactive material, respectively. While the natural radioactivity is present everywhere, in some cases human activities create greater chances for the exposure of general population to particular natural radioactive material. TENORM is a NORM material in which the natural concentrations of radioactive materials are modified by some technological process (oil extraction, combustion, water treatment in water plants, *etc*.) [1].

Gamma spectroscopy is often used to examine radio active content of natural materials, in every day practices as well as in science. Materials extracted from the Earth's crust are more or less isolated from the atmosphere, so the content of artificial (and cosmogenic) radionuclides is usually very low, meaning that they can be classified as NORM materials. Primordial radionuclides 238 U, 232 Th, and 235 U with progenies, as well as $40K$ are always present in some quantities and all contribute to the gamma radiation levels.

There are three principal problems in assigning the physical meaning to detector signals generated by photons in gamma spectroscopy. First, not all the photons will deposit all of their energy in the detector [2]. This problem was addressed in a number of papers and thus will not be further analyzed in this paper. Second, even if the first problem were somehow avoided, a finite resolution of the detector remains a problem: the photons that are within a certain energy window cannot be distinguished. In the case of samples containing several radionuclides with many gamma photons, the spectrum can become very complex and many photons can fall within a detector full width at half maximum (FWHM) at certain energy. Quantitative analysis of such photopeaks can be a troublesome job and includes using of deconvolution software, estimating interferences based on other photopeaks of the same radionuclide or quantitative analysis by other methods [2]. Another approach might be radioche- mical sepa-

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ration of radionuclides [3]. Finally, several photons may interact with the sensitive volume of the detector during the time interval shorter than the resolving time of the detector, and then the effect of summing will occur. This problem was also analyzed in literature [4] and is usually insignificant for low count rates typical for environmental samples, so it would not be discussed further.

Quantitative gamma analysis of NORM and TENORM materials is often performed without radiochemical separation. Even in the case of high resolution germanium detectors (which have much better resolution compared with NaI(Tl) detectors), photopeaks that are used in quantitative analysis can interfere with other photopeaks. This means that several photopeaks are so close that they cannot be resolved by the detector system, and an overall photopeak is detected instead. The contribution to the over all photopeak by all the photons except for the photon being analyzed is considered to be interference. In this paper, the interferences were calculated for most of the commonly used photopeaks and for several realistic cases of natural radioactivity content. In some cases present in the environment, the isotope activities deviate significantly from the "normal" cases and interferences might mask important photopeaks to such extent to render them completely useless for quantitative analysis. Knowing these cases is particularly important for the laboratories that perform routine gamma spectroscopic analysis.

Samples with roughly equal activity of 238 U and 232 Th are commonplace [5]. If radioactive equilibrium is assumed as well as natural uranium isotopic composition, it is possible to calculate emission and detection rates for all the emitted photons, along with the interferences that they cause to the photons important in quantitative analysis. In this case, all the important photons are either nearly free of interferences, or the extent of those interferences is well known. However, environmental samples such as zircon ore with ²³⁸U to 232 Th activity ratio of 10:1, or phosphate rock samples with 238 U to 232 Th activity ratio of 1:3 are not rare, and even higher imbalances can be found. Radioactive chains might also be significantly disturbed, especially in TENORM materials. In these cases, some low yield photons that can be normally neglected during gamma spectroscopic analysis make significant contribution to the spectrum, and in some cases they might even completely mask photons that are normally free from interferences.

An extensive literature research resulted in a set of important photons commonly used in quantitative gamma spectroscopic analysis. A further research resulted in some extreme cases of significant disruption of radioactive equilibrium and/or extreme values for the 238 U to 232 Th ratio. In the last step, a computer program was developed and used to analyze all the collected data.

MATERIALS AND METHODS

A computer program INCA (interference calculator) currently under development was used for the calculations. The program uses a custom photons database that includes the photons of all isotopes listed in tab. 1. For simplicity, only photons with emission probability higher than 0.1% were included in the database. Important gamma photons in the context of this paper are photons commonly used in quantitative analysis of NORM and TENORM materials. In order to determine such photons, an extensive literature research was made $[6-13]$. All the selected photons are listed in tab. 1, and the papers in which they were used are indicated with x. The values for photon energies and yields, for branching ratios and other data are obtained from BIPM monograph [14]. The program inputs are the detector energy and efficiency calibration data and the specific activities of all radionuclides existing in a sample. The interferences can be estimated for real samples before the gamma spectrometry measurement is started if the calculations are performed with expected or typical values of activities for the type of material (phosphate, zircon ore, *etc*.) that is being measured. The program combines the input data with the data from the photon database to calculate the number of detected photons for each gamma line and isotope for specific sample. Then, a set of photons selected by the user is checked for full energy peak interferences. The user is free to choose the energy window within which he wishes to investigate for possible interferences. This window can be the detector FWHM or FWTM calculated from detector calibration curves or an arbitrary energy window defined by the user.

A hypothetical "typical" case was created in order to have a standard or reference sample, suitable for comparison with TENORM material spectra. For this pur pose, UNSCEAR report for year 2000 was used [5]. According to this report, the values around 500 Bq/kg, 40 Bq/kg, and 40 Bq/kg can be considered as very likely activities of $40K$, $238U$, and $232Th$ in soil around the world, respectively. For this typical sample, radioactive equilibrium was further assumed. The case of the "typical sample" is important for the illustration of how the interferences might significantly differ if radioactive equilibrium is disturbed or the 238 U to 232 Th ratio takes extreme values.

The other cases were chosen in such fashion that the gamma emitting isotopes relative activities are as different from the reference case as possible.

The first case was taken from [15]. Phosphogypsum produced in a sulphuric acid process is doubly interesting: ²³²Th activity is much lower than ²³⁸U activity, and ²²⁶Ra is concentrated 4 times compared to 238 U. Since only the activities for 238 U, 226 Ra, 232 Th, and 210 Pb were given, it was assumed that the other radioisotopes are in equilibrium

Reference		[6]	$[7]$	[8]	$[9]$	[10]	$[11]$	$[12]$	$[13]$
Isotope	Energy [keV]								
40 _K	1460.822	$\mathbf X$	$\mathbf X$	$\mathbf X$			$\mathbf X$	$\mathbf X$	$\mathbf X$
$\sqrt[234]{\text{Th}}$	63.3				$\mathbf X$	$\mathbf X$	$\mathbf X$		
234 Th	92.8					$\mathbf X$			
$\overline{^{234m}}Pa$	1001.026					$\mathbf X$	$\mathbf X$		
234 U	53.2					$\mathbf X$			
230 Th	67.67					$\mathbf X$			
$\overline{^{226}Ra}$	186.211				$\mathbf X$	$\mathbf X$			
214 Pb	295.224		$\mathbf X$			$\mathbf X$	$\mathbf X$	$\mathbf X$	
214Pb	351.932		$\mathbf X$			$\mathbf X$	$\mathbf X$	$\mathbf X$	$\mathbf X$
214Bi	609.312		$\mathbf X$			$\mathbf X$	$\mathbf x$	$\mathbf X$	$\mathbf X$
214Bi	1120.287	$\mathbf X$					$\mathbf X$	$\mathbf X$	
^{214}Bi	1764.494	$\mathbf X$	$\mathbf X$	$\mathbf X$			$\mathbf X$		
210Pb	46.539				$\mathbf X$	$\mathbf X$			
$\overline{^{228}}$ Ac	338.32		$\mathbf x$			$\mathbf x$	$\mathbf X$	$\mathbf X$	
$\overline{^{228}}$ Ac	911.196		$\mathbf X$			$\mathbf X$	$\mathbf X$	$\mathbf X$	\mathbf{X}
^{228}Ac	968.96					$\mathbf X$			
224 Ra	240.986					$\mathbf X$			
$^{212}\mathrm{Pb}$	238.632		$\mathbf X$			$\mathbf X$		$\mathbf X$	$\mathbf X$
$\overline{^{208}\text{T}}$	583.187		$\mathbf X$			$\mathbf X$		$\mathbf X$	
208 Tl	2614.511	$\mathbf X$	$\mathbf X$	$\mathbf X$					
235 U	143.767				$\mathbf X$		$\mathbf X$		
235 U	163.356						$\mathbf X$		
$\overline{^{235}}$ U	185.72				$\mathbf X$				

Table 1. List of photons commonly used in gamma spectroscopic analysis of natural radionuclides

with their parents, and that the uranium isotopic composition is natural.

In second case, a sample with naturally depleted uranium (DU) was considered. $235U$ isotopic fraction as low as 0.44% was found in samples from Oklo mine $[16]$. The reason for the disturbance of isotopic composition is the natural nuclear reactor that was active in Oklo at least 2 billion years ago. Radioactive equilibrium has been achieved again since. This is a hypothetical sample. It was assumed that it is completely analogous to the reference case, with the exception of uranium composition. ²³⁸U and ²³²Th activities were set to 40 Bq/kg, 40 K activity to 500 Bq/kg and the other isotopes activities were calculated having in mind altered uranium isotopic composition.

Activities for the third case were obtained from [17]. Phosphate rock activities were interesting because 232 Th activity was three times higher than 238 U activity. Radioactive equilibrium was also disturbed to some extent. ^{235}U chain activities were calculated assuming natural isotopic composition.

The last case was Zircon ore. Zircon is an interesting example of NORM material. ²³⁸U activities are typically higher up to an order of magnitude than ²³²Th activities, and $40K$ activities are usually lower than either 238 U or 232 Th activities [11, 18]. The case that was studied was of a zircon ore originating from the USA. 238 U chain activities were around 8 times higher than 232 Th chain activities, with some equilibrium disturbances. ²³⁵U chain activities were calculated assuming natural isotopic composition.

For all the samples, several assumptions were made: all the radionuclides are in radioactive equilibrium with their respective parents, unless another activity is specified. 238 U and 235 U activities are calculated according to natural isotopic composition, except for the Oklo mine case. In all the cases, concentrations of all the isotopes were calculated according to these ad hoc rules, but the $40K$ concentration is set to 0 if the data is not available.

Since all the calculations require a known detector efficiency and FWHM, depending on energy, or otherwise set energy window, an actual detector calibration equations were used. Because of the very rich spectrum, the detector that was chosen is an extended range germanium detector, with 105% relative efficiency and FWHM of 1.03 keV at 122 keV and FWHM of 2.04 keV at 1332 keV. For calibration purposes, IAEA spiked soil sample was used. Admittedly, three of the samples that were studied have somewhat different matrix and density then the soil sample used for calibration, so the detector efficiency will be different. However, using of the same calibration equation is justified by the fact that the interferences that are calculated depend on the ratio of the count rates of the interfering photopeaks and do not depend on the absolute value of efficiency. Density effects on efficiency will have the same influence on all the interfering photons, because the efficiency will drop linearly with density [19]. Matrix effects will not be significant, especially considering that the photons that interfere with each other have similar energies. Any uncertainties introduced this way will be small compared to the other sources of uncertainties, above all, values for activities of the samples.

RESULTS AND DISCUSSION

The activities of all the radioisotopes of all the samples were calculated where these activities were not previously available, and are presented in tab. 2. Isotopes were grouped to indicate sub-chains within the main radioactive chains. Sub-chains were identified according to the time needed for radioactive equilibrium to be achieved between two subsequent radionuclides.

The interferences were calculated using INCA program for 23 peaks for 4 samples and the reference sample. The energy window used is calculated from the calibration formula for an extended range germanium detector. The example of calculated values for FWTM is 3.05 keV at 100 keV and 4.65 keV at 1 MeV. Two tables are given, one for the energy window equal to 1 FWHM and another for the energy window equal

to 1 FWTM (tabs. 3 and 4). Detection rates are also calculated based on the actual detector calibration and the known activities. The interferences thus calculated are approximate and the calculations are valid given that the following assumptions are true:

- only the isotopes listed in tab. 2 are present in the sample,
- contribution of gamma photons and characteristic $\overline{}$ X-rays with yield lower than 0.1 per 100 disintegrations is negligible, and
- $\frac{1}{2}$ bremsstrahlung, Compton continuum, X-ray escape peaks, single and double escape peaks and electron escape peaks are deducted from the spectrum or are negligible to begin with.

A conservative approach during gamma-ray spectrometry is that two lines can be resolved if their separation is higher than \sim 1 FWHM. It is clear from tab. 3 that many photons have a significant and variable interference from other photons within the energy window of 1 FWHM. For the energy window of 1 FWTM, interferences are higher, as expected.

The photons in these tables can be divided in three groups: photons with significant and variable interferences, photons with significant but constant interferences and photons with negligible interferences. The first group is the most interesting for the topic of this paper.

As can be seen from the tables, a large interference is present in case of 224 Ra in all the samples. Also, a considerable interference can be found at the 186 keV photopeak that belongs to 226 Ra and 235 U, which was expected. Three photons are especially interesting: 208 Tl photon at 583 keV and ²¹²Pb photon at 239 keV have significant interferences when the window is set to 1 FWTM, and the extent of interferences is very much dependent on the case that is examined. However, if the window is set to 1 FWHM, no interferences are recorded. ²²⁸Ac photon at 338 keV has significant interferences at both energy window settings, and they are affected by the case that is studied. All these lines are often used for quantitative analysis.

The reason for such behaviour of these photopeaks can be found by analysing other photons with similar energies, which is another option available in the INCA program. Within 1 FWTM of the $212Pb$ photon at 239 keV, there are the contributions of two other photons. One is emitted by $227Th$, and the other by $22\overline{4}Ra$. $224Ra$ and $212Pb$ are in the same radio active chain, and the radioisotopes between them have short half-lives (3.6 days at most). Since most of the laboratories wait up to 30 days to perform the measurements, it is very likely that $2^{12}Pb$ and $2^{24}Ra$ activities are equal within the experimental error margin. Th-227, on the other hand, belongs to ²³⁵U chain. As the ²³⁵U activity is connected to ²³⁸U activity in most of the samples, so is the 227 Th activity. This photon proves to be the reason for the difference in interferences to ²¹²Pb photon between the cases.

If the same process is repeated with 208 Tl photopeak at 583 keV, it can be seen that in its vicinity there are 3 photopeaks. Again, one photon is emitted by an isotope in the same radioactive chain, 228 Ac, and its contribution to the photopeak should not vary by much. Another photon is emitted by 234 Pa, and due to its low yield and low 234 Pa activity due to the branching ratio, it is insignificant. The third photon is emitted by $2^{14}Pb$, which belongs to $2^{38}U$ radioactive chain. Its contribution to the overall photopeak ranged from under 1% to over 30% .

Finally, ²²⁸Ac photopeak at 338 keV also has 3 photopeaks in its vicinity. One of them also belongs to 228 Ac, and hence does not represent any problems. The other is a low yield photon of 231 Pa, and can be ne-

glected in all the cases. The third photon belongs to 223 Ra and is shifted less than 0.1 keV from the 228 Ac photon. These two peaks are irresolvable by conventional gamma spectrometers. It also proves that this photon's contribution to the overall photopeak can vary by the factor 10 at least. In the different cases studied, it was less than 1% and more than 10% .

Similar discussion can be repeated for all the listed photons, or any other photon that is available to the INCA program through the database.

It is also interesting to identify the photons such as ²²⁸Ac photon at 969 keV, which has almost constant interferences for all the cases studied. Other photons have no interferences within used energy windows. It is impractical and even impossible to predict and analyse all the possible scenarios, but it was shown conclusively that performing quantitative gamma analysis of NORM and TENORM materials might be erroneous without estimating the contribution of the photons with similar energies.

CON CLU SIONS

In this paper, it was shown that some photons commonly used for NORM and TENORM quantitative analysis can get interferences so large that they become useless for the analysis, only by varying isotopes concentrations. This is true even if a germanium detector with FWHM of 1.03 keV at 122 keV and FWHM of 2.04 keV at 1332 keV was used. The influence of interferences becomes much more significant if a NaI(Tl) detector is used, due to its inferior resolution.

Five different cases were studied, all of which represent realistic examples of what any laboratory can come across. It was shown that not only in cases when the equilibrium in radioactive chains is disturbed, but also when the ratio between 232 Th and 238 U changes, unexpected interferences may occur. ²³²Th and 238 U activity ratio can vary in NORM materials by more than one order of magnitude, which could cause some important photons to be completely masked.

While the analyzed cases illustrate the change in interferences, they are far from the most extreme. Another important addition to this work may be identification of summation and escape peaks that might interfere with photopeaks commonly used in quantitative analysis. Also, NORM and especially TENORM samples can be contaminated by artificial or cosmogenic radionuclides. If the study was extended to the photopeaks of these radionuclides, additional valuable data might have been acquired.

AC KNOWL EDG EMENT

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AUTHOR CONTRIBUTIONS

Theoretical analysis was carried out by M. Z. Živanović and M. J. Anagnostakis. Computer code INCA was written by M. Z. Živanović. Literature research was carried out by M. Z. Živanović and J. D. Nikolić. Calculations were performed by M. Z. Živanović and A. I. Apostol. Figures were prepared by M. Z. Živanović and J. D. Nikolić. All authors discussed the results and participated in the writing of the manuscript. Research was co-ordinated by M. J. Anagnostakis.

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АНАЛИЗА ИНТЕРФЕРЕНЦИЈА ФОТОПИКОВА У ГАМА **CHEKTPOMETPILIII NORM II TENORM Y3OPAKA**

Значајан број примордијалних радионуклида налази се у скоро свим узорцима пореклом из Земљине коре, било да се ради о нафти, камену, земљишту или неком другом материјалу. Њихове концентрације се често одређују гама спектрометријски. Иако се релативне концентрације радионуклида углавном крећу у релативно уском опсегу, то није увек случај. Неки природни материјали (NORM) садрже ²³⁸U и ²³²Th са неуобичајеним односом активности, а технолошки измењени материјали (TENORM) могу такође имати радиоактивне низове у значајној неравнотежи. Ако се зна да примордијални радионуклиди емитују више од 1000 гама фотона и карактеристичних Х-зрака и да многи од њих међусобно интерферирају, поставља се питање да ли за неке односе концентрација често коришћени фотони постају неупотребљиви за квантитативну анализу. У циљу налажења таквих случајева развијен је рачунарски програм који може да рачуна интерференције које потичу од фотона који су предали целу енергију детектору, за било који одабрани фотон. Израчунавања се заснивају на унетим активностима изотопа и калибрационим једначинама детектора.

K*льучне речи: дама сиекииромешрија, иншерференција, NORM, TENORM, радиоакиивности*